Food Bioactive Ingredients

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Esra Capanoglu *Editors*

Retention of Bioactives in Food Processing

Springer

Food Bioactive Ingredients

Series Editor

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The Food Bioactive Ingredients Series covers recent advances and research on the science, properties, functions, technology, engineering and applications of food bioactive ingredients and their relevant products. The series also covers health-related aspects of these bioactive components, which have been shown to play a critical role in preventing or delaying different diseases and to have many health-improving properties. The books in this series target professional scientists, academics, researchers, students, industry professionals, governmental organizations, producing industries and all experts performing research on functional food development, pharmaceuticals, cosmetics and agricultural crops.

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Preface

Bioactive compounds and nutraceuticals which are known for their positive health effects can be lost during processing and storage of food products. It is a fact that most foods are exposed to processing to increase their shelf life and edibility while maintaining their sensory and nutritional properties as well as ensuring their microbial safety. Conventional processing methods are the most widely used techniques but may have several disadvantages such as decreasing the nutritional quality of foods, long processing times, high temperature and indeed high energy uses. In this sense, novel non-thermal food processing technologies including high pressure processing and ultrasonication, and novel thermal food processing technologies including microwave, and Ohmic heating have been started to become widespread.

The scope of this book is to provide a critical evaluation of the effects of conventional and novel non-thermal and thermal food processing techniques as well as emerging technologies such as nanotechnology on the retention and bioaccessibility of bioactive compounds in food materials. Researchers working in the field of antioxidants, phytochemicals, *in vitro* bioaccessibility and *in vitro* and *in vivo* bioavailability studies, novel technologies and functional foods, along with academicians and lecturers (especially on food analytical methods) can use this book as a source for their lectures. Also, nutritionists, food and pharmaceutical industrialists, experts working in the fields of biochemistry, packaging, engineering, processing, etc. can find this book helpful for their studies.

In Part I, Chap. 1 gives an overview of food bioactive compounds and their health-promoting features, while Chap. 2 gives an outlook of different food processing technologies. Part II covers the influence of conventional processes on food bioactive compounds including frying, baking and cooking processes (Chap. 3), chilling, freezing and thawing processes (Chap. 4), drying processes (Chap. 5), canning processes (Chap. 6), juice processing (Chap. 7), extrusion processes (Chap. 8), fermentation and germination processes (Chap. 9), extraction processes (Chap. 10) and modified atmosphere packaging (Chap. 11). Part III addresses the influence of novel thermal processes on food bioactive compounds, namely microwave heating (Chap. 12) and Ohmic heating (Chap. 13). Finally, the influence of novel non-thermal processes on food bioactive compounds has been described in Part IV

which includes irradiation processes (Chap. 14), high pressure processing (Chap. 15), ultrasonication (Chap. 16), membrane separation processes (Chap. 17), ozonation and plasma processing (Chap. 18) and nanotechnology processes (Chap. 19).

Herein, the editors would like to thank all the contributors of the book for their collaboration and efforts in bringing together different subjects dealing with the influence of different food processing technologies on the retention of bioactive compounds. Their acceptance of our invitation in these critical and pandemic times are highly appreciated. Also, it is necessary to express our sincere thanks to all the editorial staff at Springer for their help and support throughout the project. Finally, special acknowledgement is to our family for their understanding and encouragement during the editing of this great project.

Gorgan, Iran — Seid Mahdi Jafari
Istanbul, Turkey — Esra Capanoglu

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Seid Mahdi Jafari received his PhD in Food Process Engineering from the University of Queensland (Australia), in 2006. Now, he is a full-time professor at GUASNR (Iran), part-time professor at UVigo (Spain), adjunct professor at SINANO (China) and adjunct professor at Hebei Agricultural University (China). He has published >420 papers in international journals (h-index = 72 in Scopus) and 80 book chapters/36 books with Elsevier, Springer and Taylor & Francis. Selected achievements:

- One of the top 1% world scientists by Thomson Reuters (2015)
- One of the top national researchers by the Iranian Ministry of Science, Research, and Technology (2017)
- One of the world's highly cited researchers by Clarivate Analytics (Web of Science), in 2018, 2019 and 2020
- A top reviewer in the field of agricultural and biological sciences by Publons (2018 and 2019)

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Esra Capanoglu is a Professor of Istanbul Technical University (ITU), Food Engineering Department in Turkey. She finished her PhD in 2008 at ITU and worked as a Post-Doc in RWTH Aachen University, Germany for 2 years. During her PhD thesis, she worked in the laboratories of Plant Research International (PRI), The Netherlands for 6 months and in IPK Leibniz Institute, Gatersleben, Germany for another 6 months with a DAAD scholarship. She works on antioxidants and phenolic compounds in foods and on their in vitro bioaccessibility/bioavailability. She also focuses on the effects of processing, encapsulation and matrix on these bioactives. She has published two books, several chapters, more than 175 papers in peer-reviewed journals and has taken part in several international (EU 6th Frame, EU 7th Frame, COST, Joint Research Projects, etc.) and national projects (TUBITAK, TAGEM, ISTKA, etc.). She is an Assoc. Editor in ACS Omega and Editorial Advisory Board Member of Journal of Agricultural and Food Chemistry (JAFC), Journal of Berry Research, eFOOD, Frontiers in Nutrition, Turkish Journal of Agriculture and Forestry and several others. Istanbul Technical University, Istanbul, Turkey

Part I
Introduction to Bioactive Compounds and Food Processes

Chapter 1
An Overview of Food Bioactive Compounds and Their Health-Promoting Features

Senem Kamiloglu, Esra Capanoglu, and Seid Mahdi Jafari

1.1 Introduction

Bioactive compounds have been identified as health-promoting therapeutic agents that stimulate the production of new supplements and functional food products. Interest in bioactive compounds continues to expand, assisted by new technological advances and ongoing research initiatives to identify the functions and future uses of bioactive compounds, as well as public interest and consumer needs. Consequently, bioactive compounds that can be utilized as ingredients of functional foods, supplements or nutraceuticals are obtained and characterized by the food industry (Đorđević et al. 2015).

According to the most recent data, non-communicable diseases including cardiovascular diseases, diabetes, cancers, neurodegenerative diseases and chronic respiratory diseases caused 71% of the 56.9 million global deaths (Countdown 2018). The factors leading to occurrence of these diseases include the combined effect of

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unhealthy diets, low physical activity and use of alcohol as well as smoking. In particular, poor diet is the major risk factor as it is linked to one in five deaths (Afshin et al. 2019). In accordance with this fact, epidemiological studies indicate that the so-called Mediterranean diet that is rich in bioactive compounds may be preventive against diet related degenerative diseases (Mentella et al. 2019; Câmara et al. 2021).

Food bioactive compounds include a wide variety of components, often produced as plant secondary metabolites and utilized in various functions, such as competition, protection, attraction and signaling. Food bioactive compounds can be classified as polyphenols, carotenoids, glucosinolates, vitamins, phytosterols, triterpenes, alkaloids, capsaicinoids, polysaccharides, polyunsaturated fatty acids and bioactive peptides, among others (Table 1.1). The health beneficial effects of these bioactive compounds are attributed to their antioxidant, anti-inflammatory, antimicrobial, antiatherogenic, antithrombotic, cardioprotective and vasodilator properties (Câmara et al. 2021).

As food bioactive compounds present in nature are very diverse in terms of source, structure, and biological effects, it is not possible to explore all in detail. Considering that, in this chapter, we briefly focus on *in vivo* evidence of the health beneficial effects of mentioned food bioactive compounds.

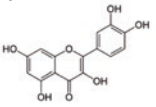
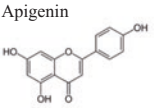
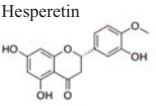
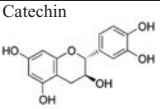
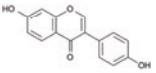
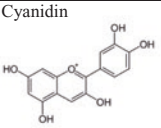
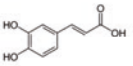
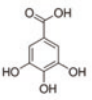
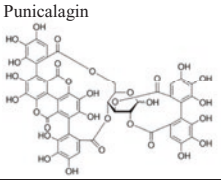
1.2 Polyphenols

Polyphenols are secondary plant metabolites consisting of large class of compounds, which can be classified as flavonoids, phenolic acids, tannins, stilbenes, and lignans (Durazzo et al. 2019). Expanding evidence from epidemiological studies as well as clinical trials and meta-analyses propose that a diet rich in polyphenols can decrease the risk of chronic diseases, thus improving human health (Costa et al. 2017). These potential positive health effects are related to antioxidant, anti-inflammatory (Zhang and Tsao 2016), antimicrobial (Daglia 2012) and anticancer (Niedzwiecki et al. 2016) properties of polyphenols. In the following sections, the potential health promoting effects of different polyphenols are discussed.

1.2.1 Flavonoids

Flavonoids are low molecular weight compounds, comprising of 15 carbon atoms, arranged in a C_6–C_3–C_6 configuration. The flavonoid structure comprised of 2 aromatic rings, joined by a 3-carbon bridge, usually in the form of a heterocyclic ring. Alterations in the substitution patterns of this heterocyclic ring result in six different subclasses, namely, flavonols, flavones, flavanones, flavanols, isoflavones, and anthocyanidins (Balasundram et al. 2006). Differences within each group originate from the variation in number and arrangement of the hydroxyl groups and their extent of alkylation and/or glycosylation (Grootaert et al. 2015; Pandey and Rizvi 2009; Spencer et al. 2008).

Table 1.1 Classification of some important bioactive compounds

Class	Sub-class		Example	Dietary sources
Polyphenols	Flavonoids	Flavonols	Quercetin 	
		Flavones	Apigenin 	
		Flavanones	Hesperetin 	
		Flavanols	Catechin 	
		Isoflavonoids	Daidzein 	
		Anthocyanidins	Cyanidin 	
	Phenolic acids	Hydroxycinnamic acids	Caffeic acid 	
		Hydroxybenzoic acids	Gallic acid 	
	Tannins	Hydrolyzable tannins	Punicalagin 	

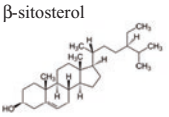
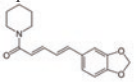
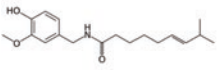
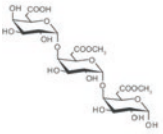
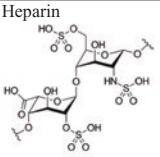
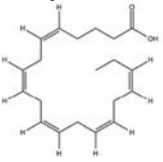
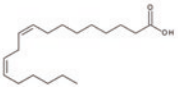
(continued)

Table 1.1 (continued)

		Condensed tannins	Procyanidin B2	
	Stilbenes		Resveratrol	
	Lignans		Secoisolariciresinol	
	Others		Curcumin	
Carotenoids	Xanthophylls		Lutein	
	Carotenes		β-carotene	
Vitamins	Water-soluble vitamins		Vitamin C	
	Fat-soluble vitamins		Vitamin E	
Glucosinolates			Glucoraphanin	
Triterpenes	Squalene derivatives		Squalene	
	Saponins		Soyasaponin I	

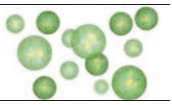
(continued)

Table 1.1 (continued)

Phytosterols		β-sitosterol 	
Alkaloids	Piperidines	Piperine 	
	Methylxanthines	Caffeine 	
Capsaicinoids		Capsaicin 	
Polysaccharides	Homoglycans	Pectin 	
	Heteroglycans	Heparin 	
Polyunsaturated fatty acids	Omega-3 fatty acids	Eicosapentaenoic acid (EPA) 	
	Omega-6 fatty acids	Linoleic acid 	

(continued)

Table 1.1 (continued)

Bioactive peptides	Animal protein sources	Milk-derived bioactive peptides	
	Plant protein sources	Seed-derived bioactive peptides	
	Marine protein sources	Microalgae-derived bioactive peptides	

1.2.1.1 Flavonols

Flavonols are the most common subgroup of flavonoids. Berries, broccoli, kale, cabbage and onion are among the well-known dietary sources of flavonols. The main dietary flavonol aglycones are quercetin, kaempferol and myricetin, which are usually present as *O*-glycosides (Kamiloglu et al. 2020a). Among flavonols, **quercetin** has attracted particular attention due to its potential use in the prevention of various diseases, in particular cardiovascular diseases and cancers. Cardioprotective effects of quercetin include inhibition of low-density lipoprotein (LDL) oxidation, endothelium-independent vasodilator effects, decrease of adhesion molecules and other inflammatory markers, the protective effect on endothelial function and nitric oxide under oxidative stress, prevention of neuronal oxidative and inflammatory damage and platelet antiaggregant effects (Patel et al. 2018). Similarly, quercetin inhibits the propagation of various types of cancers, such as colon, breast, lung, prostate, liver and cervical cancers (Liu et al. 2017; Rauf et al. 2018). The anticancer effects of quercetin are exerted through various mechanisms including promotion of the loss of cell viability, autophagy and apoptosis through the modulation of Wnt/β-catenin, phosphatidylinositol 3-kinase (PI3K)/protein kinase B (Akt)/mechanistic target of rapamycin (mTOR), and mitogen-activated protein kinase (MAPK)/extracellular signal-regulated protein kinase (ERK)1/2 pathways (Reyes-Farias and Carrasco-Pozo 2019).

Kaempferol is also reported to reduce the risk of chronic diseases. For instance, it may improve the antioxidant defense of body against free radicals, which foster the initiation of cancer. At the molecular level, kaempferol modulates key elements in cellular signal transduction pathways linked to apoptosis, angiogenesis, inflammation, and metastasis (Chen and Chen 2013). Furthermore, kaempferol was also found to be effective in prevention and treatment of inflammatory diseases including atherosclerosis, arthritis and allergies (Devi et al. 2015a). In addition, several *in vivo* studies acknowledged the bone-protecting property of kaempferol and kaempferol-containing plants (Trivedi et al. 2008; Nowak et al. 2017; Adhikary et al. 2018; Wong et al. 2019). Similarly, **myricetin** has been reported to possess

several biological activities including antioxidant (Henneberg et al. 2013), antiviral (Pasetto et al. 2014), anti-inflammatory (Kim et al. 2009), antidiabetic (Zamora-Ros et al. 2014), and anticancer (Devi et al. 2015b) effects, as well as protective effects against Alzheimer's disease (Hamaguchi et al. 2009). On the other hand, the *in vivo* protective effects of myricetin have been reported to be limited due to its poor bioavailability (Duthie and Morrice 2012; Park et al. 2016).

1.2.1.2 Flavones

Flavones consist primarily of apigenin and luteolin glycosides (Manach et al. 2004). Glycosylated form of **apigenin** is present in fruits (e.g., oranges), vegetables (e.g., celery, parsley, onions), herbs (e.g., chamomile, thyme, basil, oregano) and beverages (e.g., tea, wine, beer) (Hostetler et al. 2017). Human studies monitoring apigenin supplementation suggested therapeutic effects of this compound on Alzheimer's disease (De Font-Reaulx and Dorazco-Barragan 2010), insomnia (Zick et al. 2011), osteoarthritis (Shoara et al. 2015) as well as anxiety disorder and depression (Amsterdam et al. 2012; Mao et al. 2016). Similarly, animal models involving apigenin reported improved effects on cancer (Shukla et al. 2014), diabetes (Malik et al. 2017), Alzheimer's disease (Liang et al. 2017), amnesia (Liu et al. 2011) and depression (Weng et al. 2016). The different mechanisms underlying the potential therapeutic actions of apigenin were discussed in a recent review (Salehi et al. 2019).

Major dietary sources of **luteolin** include celery, spinach, some varieties of peppers and lettuce (Seelinger et al. 2008). Luteolin has been shown to possess antioxidant activity through reduction of oxidative stress induced damage (Zhang et al. 2017) and anti-inflammatory effects by reducing endotoxin induced uveitis (Dirscherl et al. 2010). Moreover, luteolin also exerted anti-cancer properties through reduction of colon cancer tumors (Lin et al. 2008). Other therapeutic activities include neuroprotective, antidepressant and antiviral effects (Theoharides 2009; Fan et al. 2016; Lin et al. 2016). In addition, luteolin *C*-glycosides were also reported to show antioxidant, antiviral and hepatoprotective activities (Manzoor et al. 2019).

1.2.1.3 Flavanones

Hesperetin, **naringenin** and **eriodictyol** are the major flavanone aglycones, which are abundantly present in citrus fruits (Manach et al. 2004). Research has demonstrated that flavanones, in particular hesperidin and its aglycone hesperetin, possess antioxidant and anti-inflammatory properties. The ability of flavanones to control inflammation is related to their antioxidant activity as well as their capability of inhibiting enzymes required for cellular signal transduction and activation (Barreca et al. 2017). Another health promoting effect of citrus flavanones include cardioprotective properties which is primarily related to enhanced production and release of nitric oxide (Liu et al. 2008). Flavanones also inhibit the growth of tumor and induce

cancer cell apoptosis (Benavente-Garcia and Castillo 2008). Moreover, flavanones may also reduce the level of cholesterol due to the resemblance of their structure to a commonly used statin, i.e., the drug capable of inhibition of cholesterol biosynthesis (Mollace et al. 2011). Furthermore, it has been reported that flavanones possess stronger antimicrobial effect compared to flavonols and flavones (Moon et al. 2013). In addition, flavanones may also act as inhibitors of acetylcholinesterase, which is the major target in the treatment of Alzheimer's disease (Barreca et al. 2017).

1.2.1.4 Flavanols

Epicatechin and catechin are the major flavanols that are present in fruits, chocolate, red wine, and tea (Manach et al. 2004). Major physiological functions and regulating mechanisms of flavanols, in particular **epicatechin**, include antioxidant (Chang et al. 2014), anti-inflammatory (Vazquez-Prieto et al. 2012), antidiabetic (Álvarez-Cilleros et al. 2018), anticancer (Siddique et al. 2012), cardioprotective and neuroprotective (Shaki et al. 2017; Leonardo et al. 2013) activities as well as enhanced skeletal muscle performance (Li et al. 2019). Antioxidative properties are related to the activation of nuclear factor-like 2/anti-oxidant response element (Nrf2/ARE) pathway, reduction of reactive oxygen species (ROS) and preventing the generation of oxidative stress. Anti-inflammatory activity of flavanols is provided through inhibition of nuclear factor kappa B/tumor necrosis factor alpha (NF-κB/TNF-α) and stimulation of the phosphatidylinositol 3-kinase/protein kinase B (PI3K/AKT) pathway. Similarly, antidiabetic action is related to the regulation of islet signaling and resistance and stimulation of secretion of insulin, whereas anticancer activity is provided via inhibition of DNA polymerase α and induction of cell division arrest in G2 phase. Moreover, NO-, Nrf2 and mitochondria-mediated protection mechanisms were related with the cardioprotective effects of flavanols. Furthermore, neuroprotective effects include stimulation of nuclear factor-like 2/heme oxygenase 1 (Nrf2/HO1) pathway and synergistic action with drugs. In addition, enhanced muscle performance involves the promotion of myogenic differentiation, mitochondrial synthesis and capillary angiogenesis (Qu et al. 2021).

1.2.1.5 Isoflavonoids

Soy and soy products are the primary dietary sources of isoflavones, which include three main molecules: **daidzein**, **genistein** and **glycitein** (Manach et al. 2004). These compounds are known for their beneficial effects in cardiovascular health (Cano et al. 2010), bone health (Taku et al. 2010) and hormone regulation. In particular, isoflavonoids have been reported to possess estrogenic or anti-estrogenic function depending on the hormone level in the body (Pilsakova et al. 2010). Isoflavonoids are also demonstrated to reduce the cancer risk owing to their antioxidant and anti-tumorogenic effects such as prevention of carcinogenesis pathway through inhibition of protein tyrosine kinase (PTK), stopping tumor cell growth by

suppressing DNA replication and various growth factors, and controlling of enzyme activities on signal transduction pathway of carcinogenesis (Ko 2014). Moreover, isoflavonoids have been shown to prevent diabetes via modulation of carbohydrate metabolism, activation of gene expression through stimulation of peroxisome proliferator-activated receptors (PPARs) (α, γ), regulation of hyperglycemia, reduction of insulin resistance, and modification of adipocyte differentiation and tissue metabolism (Ahmed et al. 2020).

1.2.1.6 Anthocyanins

Anthocyanins are water-soluble pigments responsible for the red, purple, and blue color of many plant tissues and fruits. Although they are also present in vegetables, roots, cereals, and legumes, these compounds are often associated with fruits. Anthocyanins are present as glycosides of their respective aglycones, called as anthocyanidins. There are about 17 anthocyanidins that have been detected. However, in nature, only 6 anthocyanidins including **cyanidin**, **malvidin**, **delphinidin**, **peonidin**, **pelargonidin**, and **petunidin**, are widely present (Kamiloglu et al. 2015). Several epidemiological studies and clinical trials indicated that anthocyanins have important roles in the prevention of several degenerative diseases, in particular, cardiovascular disease, type 2 diabetes, certain types of cancers, cognition, and vision (Wallace and Giusti 2019). Anthocyanins have been suggested to exert anticancer (Bunea et al. 2013), anti-inflammatory (Taverniti et al. 2014; Kamiloglu et al. 2017), neuroprotective (Strathearn et al. 2014), anti-obesity and antidiabetic effects (Li et al. 2013a; Takikawa et al. 2010). Many signaling pathways such as MAPK, NF-κB, AMP activated protein kinase, and Wnt/β-catenin, and some essential cellular processes, including apoptosis, autophagy, cell cycle, and biochemical metabolism, are involved in the health promoting effects of anthocyanins (Li et al. 2017). On the other hand, the low bioavailability of anthocyanins is an important limiting factor in accomplishing their beneficial effects (Fernandes et al. 2014).

1.2.2 Phenolic Acids

Phenolic acids can be categorized as hydroxycinnamic and hydroxybenzoic acids, with different molecular skeleton based on cinnamic and benzoic acids, respectively (Song et al. 2020). Hydroxycinnamic acids are more prevalent in nature than the hydroxybenzoic acids, and often occur in conjugated forms. Many fruits, vegetables, grains, spices and beverages contain phenolic acids (Rashmi and Negi 2020).

Hydroxycinnamic acids include caffeic, ferulic, *p*-coumaric, and sinapic acids having a three-carbon side chain (C_6–C_3) (Balasundram et al. 2006). Some health benefits of caffeic acid include anti-atherosclerosis (Vinson et al. 2001), antimicrobial (Magnani et al. 2014), and anticancer (Balupillai et al. 2015) effects. Furthermore, ferulic acid has been reported to possess anti-inflammatory (Chen

et al. 2010), neuroprotective (Mhillaj et al. 2018), anti-obesity and antidiabetic effects (Naowaboot et al. 2016). Similarly, *p*-coumaric acid has anti-cancer properties (Hudson et al. 2000), whereas sinapic acid showed anti-inflammatory (Yun et al. 2008) and antimicrobial (Engels et al. 2012) features in addition to anticancer effects (Hudson et al. 2000). Chlorogenic acid is also one of the most common hydroxycinnamic acids with quinic acid that is present in tea and green coffee. Chlorogenic acid is a biologically active compound that exerts a variety of essential and therapeutic functions including antioxidant, anti-inflammatory, antimicrobial, cardioprotective, hepatoprotective and central nervous system stimulator properties. Furthermore, chlorogenic acid could regulate lipid metabolism and glucose in both healthy and genetically metabolic associated disorders (Naveed et al. 2018).

Hydroxybenzoic acids are compounds having C_6–C_1 structure, with gallic, protocatechuic, vanillic, and syringic acids being the most common examples (Balasundram et al. 2006). Gallic acid is reported to show anticancer, antimicrobial and cardioprotective effects (Sourani et al. 2016; Kim et al. 2006; Zanwar et al. 2014). Similarly, protocatechuic acid also possessed health beneficial effects including anti-inflammatory and anticarcinogenic features (Anter et al. 2011; Wang et al. 2010). Syringic acid is another important hydroxybenzoic acid that contributes to the health promoting effects of phenolic acids, in particular to antidiabetic properties (Muthukumaran et al. 2013).

1.2.3 Tannins

Tannins can be classified into two groups: (i) hydrolyzable tannins and (ii) condensed tannins, also called as proanthocyanidins or catechin tannins. Hydrolyzable tannins can be further classified into two groups: (i) gallotannins, providing sugar and gallic acid on hydrolysis, and (ii) ellagitannins, which yield not only sugar and gallic acid but also ellagic acid upon hydrolysis (Smeriglio et al. 2017). Tannins are reported to be present in legumes, beans and berries, and in particular grape seed proanthocyanidins attracted great attention due to their beneficial health properties (Unusan 2020). The bioactive properties of grape seed **proanthocyanidins** include antimicrobial (Ghouila et al. 2017), anticancer (Chen et al. 2014), cardioprotective (Quesada et al. 2012), antiobesity (Pascual-Serrano et al. 2018), anti-neurodegenerative (Lian et al. 2016), eye-protective (Jia et al. 2011), anti-aging (Jiao et al. 2017) and anti-osteoarthritis (Toker et al. 2018) properties. In addition to proanthocyanidins, ellagitannins also recently attracted attention. It has been reported that ellagitannins are converted to their metabolites by the action of gut microbiota (Garcia-Villalba et al. 2017). The microbial metabolites called urolithins are reported to exert anti-inflammatory, antiproliferative, and antiaging properties (Djedjibegovic et al. 2020).

1.2.4 Stilbenes

Stilbenes consist of two phenyl moieties bound by a two-carbon methylene bridge (Pandey and Rizvi 2009). The major component of stilbenes is *trans*-resveratrol, which is present in more than 70 plant species, including grapes, berries, and peanuts (Ignat et al. 2011; Toaldo et al. 2016). It has been suggested that **resveratrol** could play a potential protective role against cardiovascular diseases, certain types of cancer, diabetes, and neurological disorders. Cardioprotective effects of resveratrol include (i) improvement in left ventricular systolic and diastolic function, and flow-mediated dilation (FMD) levels, (ii) reduction of LDL cholesterol levels (Magyar et al. 2012), (iii) decrease in expression of endothelial cell intercellular adhesion molecule (ICAM), vascular cell adhesion molecule (VCAM), interleukin (IL)-8, plasma interferon gamma (IFN-γ) and insulin levels (Agarwal et al. 2013), (iv) reduction of diastolic blood pressure (Biesinger et al. 2016), and (v) decrease in C-reactive protein (CRP) release from the liver (Bo et al. 2013).

In vivo studies investigating the putative cancer chemopreventive properties of resveratrol reported that this polyphenol has the potential to interact with all 3 main phases of carcinogenesis, i.e., initiation, promotion and progression (Elshaer et al. 2018). In particular, chemopreventive role of resveratrol in colorectal and skin cancers are well-documented (Jang et al. 1997; Tessitore et al. 2000; Schneider et al. 2001; Soleas et al. 2002; Afaq et al. 2003; Reagan-Shaw et al. 2004; Nguyen et al. 2009; Patel et al. 2010; Howells et al. 2011). Several mechanisms have been proposed to explain the chemopreventive properties of resveratrol including suppression of inflammation, regulation of drug-metabolic enzymes and acting as a phytoestrogen. On the other hand, the low bioavailability of resveratrol remains to be a limiting factor for usage of this polyphenol as a therapeutic agent (Elshaer et al. 2018).

Resveratrol was also suggested as a possible adjuvant for the treatment of diabetes. Accordingly, daily administration of resveratrol has been shown to decrease glycated hemoglobin (HbA1c) levels, total cholesterol, total protein and systolic blood pressure, and improve the glycemic control (Bhatt et al. 2012). On the other hand, few studies showed that resveratrol did not show any effects on secretion of insulinotropic polypeptide (GIP) and glucagon-like peptide 1 (GLP-1) hormones (Thazhath et al. 2016; Brasnyó et al. 2011). Therefore, the efficiency of resveratrol in improving glycemic control may be questionable (Berman et al. 2017). Clinical trials investigating the efficiency of resveratrol on treatment of neurological disorders including Alzheimer's disease and brain ischemic stroke have shown that resveratrol could reduce the levels of matrix metalloproteinase (MMP) 9, cerebrospinal fluid (CSF) beta amyloid (Aβ) 42 and Aβ40, indicating decreased accumulation of pro-inflammatory cytokines and Aβs in the brain (Turner et al. 2015; Moussa et al. 2017; Chen et al. 2016). Thus, although metabolized quite rapidly, it has been reported that significant amounts of resveratrol and its metabolites were able to cross the blood–brain barrier (Berman et al. 2017).

1.2.5 Lignans

Lignans are formed through oxidative dimerization of two phenylpropane units. The primary dietary source of lignans is linseed, which contains **secoisolariciresinol** and a small amount of **matairesinol** (D'Archivio et al. 2007). Lignans are reported to exert antioxidant, anti-inflammatory and antimicrobial activities (Rodríguez-García et al. 2019). Regarding anti-inflammatory activity, lignans reduced the production of pro-inflammatory cytokines and suppressed the generation of nitric oxide (Chun et al. 2014), and concerning the antimicrobial activity, lignans showed antibacterial activity against Gram-positive bacteria via alteration of biofilm formation, bacteria metabolites, membrane receptors and ion channels (Nor Azman et al. 2018). Human studies concerning lignan bioactivity revealed that these polyphenols lowered the risk of heart disease, menopausal symptoms, osteoporosis and breast cancer (Rodríguez-García et al. 2019).

1.2.6 Other Polyphenols

In addition to the polyphenols discussed above, there are some other compounds, such as curcumin and oleuropein that have been studied extensively for their health promoting effects. **Curcumin** is a yellow-colored hydrophobic polyphenol derived from the rhizomes of turmeric (*Curcuma longa* L.), a plant species belonging to *Zingiberaceae* family. The medicinal use of this plant has been documented in traditional Chinese medicine for at least 2500 years (Kocaadam and Şanlier 2017). Curcumin consumption may exert beneficial effects against several chronic diseases including fatty liver disease (Panahi et al. 2017), arthritis (Nakagawa et al. 2009), metabolic syndrome (Yang et al. 2014), depression (Esmaily et al. 2015), obesity and over-weight (Ganjali et al. 2014), cancer (Ryan et al. 2013), diabetes (Chuengsamarn et al. 2012), inflammatory bowel disease (Suskind et al. 2013), skin disorders (Panahi et al. 2017), and symptoms of premenstrual syndrome (Khayat et al. 2015). Although the use of curcumin against several chronic diseases is promising, its poor bioavailability remains to be a limiting factor as a therapeutic agent (Mantzorou et al. 2018). **Oleuropein** is the ester of elenolic acid and hydroxytyrosol, which is responsible for the bitterness of olives (*Olea europaea*) (Keceli et al. 2017). Oleuropein aglycone is the main phenolic compound present in extra virgin olive oil, which is derived from the deglycosylation of oleuropein (Xu et al. 2018). Some of the biological activities related to oleuropein aglycone include antioxidant (Pérez-Bonilla et al. 2014), antinflammatory (Campolo et al. 2013), antihyperglycemic (Rigacci and Stefani 2015) and lipid lowering effects (Oi-Kano et al. 2017). Furthermore, oleuropein was also effective against breast cancer (Menendez et al. 2008) and Alzheimer's disease (Casamenti et al. 2015).

1.3 Carotenoids

Carotenoids are the most widespread and important group of lipophilic pigments, providing the distinctive yellow, orange, and red colors of many fruits and vegetables including carrots, pumpkin and tomatoes. Humans do not synthesize carotenoids; therefore, they have to be obtained via diet or supplementation (Eggersdorfer and Wyss 2018). Most carotenoids composed of 8 isoprene units with a 40-carbon skeleton. Their basic structures typically comprise of a polyene chain at both ends of the polyene chain with 9 conjugated double bonds and an end group (Maoka 2020). Owing to the presence of these conjugated double bonds, carotenoids can undergo isomerization to *cis-trans* isomers (Rao and Rao 2007). The all-*trans* isomer is the most stable and dominant form, whereas *cis* form of carotenoids exists in tissues and blood (Stahl and Sies 2005). Carotenoids are divided into two groups according to their chemical composition: xanthophylls and carotenes (Rehman et al. 2020). The potential health promoting effects of these groups are presented below.

1.3.1 Xanthophylls

Xanthophylls consist of diverse compounds, which include hydroxyl, aldehyde, epoxy, carboxylic acid, and keto groups. The most important members of xanthophylls are astaxanthin, β-cryptoxanthin, lutein, and zeaxanthin (Rodriguez-Amaya 2015). Xanthophylls have been studied for their biological effects in a variety of diseases including neurologic, ophthalmologic, oral, allergic and immune diseases. **Astaxanthin** exerted beneficial effects in early-stage cancers, possessed anti-allergic activity against the contact dermatitis and showed skin protecting effects against ultraviolet light injury. **β-cryptoxanthin** was reported to be effective in prevention of bone loss through osteoblastic bone formation and inhibition of osteoclastic bone resorption. **Lutein** and **zeaxanthin** prevented the progression of age-related macular degeneration, and they also showed promising effects on uveitis, scleritis, retinitis pigmentosa, glaucoma, cataracts, retinal ischemia and choroideremia (Aziz et al. 2020).

1.3.2 Carotenes

The major compounds of the carotenes include β-carotene, α-carotene, and lycopene, which constitute of carbon and hydrogen atoms. In particular, the bioactive properties of lycopene are reported in detail. Recently, the protective effects of **lycopene** in cancer, cardiovascular, and neurodegenerative diseases have been reviewed in detail. Lycopene can reduce the increased levels of proinflammatory mediators

and prevent NF-κB activation through modulation of oxidative stress. Furthermore, lycopene can also act as a precursor for several oxidative cleavage products and metabolites that can interact with multiple transcription factors to overexpress antioxidant and cytoprotective phase II enzymes and other growth-stimulating proteins for increased neuroprotection (Saini et al. 2020). Similarly, **β-carotene** and other carotenoids are reported to possess provitamin A activity, modulate lipoxygenase activity and activate certain genes responsible cell to cell communication. On the other hand, carotenoids may have adverse effects when taken in high dose by high risk population, e.g., smokers (Paiva and Russell 1999).

1.4 Vitamins

There are 13 vitamins that play a significant role in human nutrition. Based on their solubility, vitamins can be categorized into two main groups. Fat soluble group includes vitamin A (**retinol**), vitamin D (**cholecalciferol**), vitamin E (**tocopherol**) and vitamin K (**phylloquinone**). Water soluble vitamins consist of vitamin C and vitamin B group, which includes vitamin B1 (**thiamine**), vitamin B2 (**riboflavin**), vitamin B3 (**niacin**), vitamin B5 (**pantothenic acid**), vitamin B6 (**pyridoxine**), vitamin B7 (**biotin**), vitamin B9 (**folic acid**), and vitamin B12 (**cobalamins**) (Ball 2004; Fatima et al. 2019). Intake of vitamins may prevent against various diseases including beriberi, anemia, neurological diseases, oral lesions, and pellagra (Kamiloglu et al. 2020b). In particular, vitamin C and vitamin E are reported to show significant antioxidant activity (Rock et al. 1996). The major dietary sources of **vitamin C** are citrus fruits such as oranges as well as green leafy vegetables. Deficiency of vitamin C leads to scurvy, which mainly affects malnourished elderly people (Rani et al. 2019). As an antioxidant, vitamin C provided protection against oxidative stress-induced cellular damage by scavenging of reactive oxygen species and by neutralization of lipid hydroperoxyl radicals. Vitamin C also plays a role in the function of endothelial nitric oxide synthase through recycling the tetrahydrobiopterin, which is related to arterial elasticity and blood pressure regulation (Traber and Stevens 2011). Vitamin E has been shown to be effective against several chronic diseases including cancer, ageing, arthritis and cataracts. Platelet hyper-aggregation, leading to atherosclerosis may also be prevented by vitamin E supplementation. In addition, vitamin E also reduces the production of prostaglandins such as thromboxane, which cause platelet clumping (Rizvi et al. 2014).

1.5 Glucosinolates

Glucosinolates are a large group of sulfur-containing compounds present in the *Brassicaceae* plants including broccoli, cabbage, cauliflower, rapeseed, mustard, and horseradish (Bischoff 2021). The common structure of glucosinolates comprises of a β-D-thioglucose group, a sulphonated oxime moiety and a variable side chain derived from methionine, tryptophan or phenylalanine (Mithen et al. 2000). When glucosinolate containing plants are consumed without processing, myrosinase enzyme hydrolyzes glucosinolates into different metabolites such as isothiocyanates, nitriles, oxazolidine-2-thiones, and indole-3-carbinols. However, in case these plants are consumed after cooking process, myrosinase is inactivated and glucosinolates could partially be absorbed in their intact form through the gastrointestinal mucosa (Prieto et al. 2019). The biological activity of glucosinolates is mainly linked with their hydrolytic products, in particular isothiocyanates. These metabolites are reported to exert antifungal, antibacterial, bioherbicidal, biopesticidal, antioxidant, antimutagenic and anticarcinogenic properties (Vig et al. 2009).

1.6 Triterpenes

Triterpenes and their functionalized form triterpenoids can be classified in 18 subclasses including squalene derivatives, lanostanes, holostanes, cycloartanes, cucurbitanes, dammaranes, euphanes, tirucallanes, tetranortriterpenoids, quassinoids, lupanes, oleananes, friedelanes, ursanes, hopanes, serratanes, isomalabaricanes and saponins. Among the triterpenes, **squalene derivatives** and **saponins** are the most well-known ones (Hill and Connolly 2020). Olive oil is one of the main sources of squalene (Pacetti et al. 2019), whereas saponins can be found in various legumes such as soybean (Berhow et al. 2020), peanut (Kuang et al. 2017) and lentils (Navarro del Hierro et al. 2018). In the literature, anti-cancer effects of triterpenoids have been well-documented (Li et al. 2013b; Wu et al. 2013; Du et al. 2014; Yan et al. 2014; Kamble et al. 2014; Salvador et al. 2014; Zhang et al. 2014). Other biological properties of triterpenoids include anti-infammatory (Jeong and Bae 2014), antiviral (Paduch and Kandefer-Szerszen 2014) and neuroprotective (Ruszkowski and Bobkiewicz-Kozlowska 2014) activities.

1.7 Phytosterols

Phytosterols are plant sterols derived from triterpenes. Although **sitosterol**, **stigmasterol** and **campesterol** are the most common plant sterols, so far more than 250 different plant sterols have been identified (Nes 2011). Free and conjugated sterols are the two common forms of phytosterols. Conjugated form includes steryl esters,

steryl glycosides, and acylated steryl glycosides (Feng et al. 2020). The main sources of phytosterols are vegetable oils, fruits and cereals (Piironen and Lampi 2004) as well as microalgae, which has been identified as a novel source of phytosterols (Francavilla et al. 2012). Clinical trials have confirmed that a diet rich in phytosterols, in particular sitosterol, stigmasterol and campesterol, has a lowering effect on LDL cholesterol (Moreau et al. 2018). Other main bioactive properties of phytosterols include protection against nonalcoholic fatty liver disease (Plat et al. 2014; Chen et al. 2015; Song et al. 2017; Feng et al. 2018; Shahi et al. 2018), obesity (Fukuoka et al. 2014; Bhaskaragoud et al. 2016; Lambert et al. 2017; Kurano et al. 2018; Li et al. 2018) and inflammatory bowel disease (Cheon et al. 2006; Islam et al. 2008; Lee et al. 2012; Sánchez-Fidalgo et al. 2013; Aldini et al. 2014; Kim et al. 2014).

1.8 Alkaloids

Alkaloids are natural compounds that have a ring structure and a nitrogen atom as their most common feature (Koleva et al. 2012). Depending on their chemical structures and sources, alkaloids are divided into several classes such as **isoquinolines** (e.g., biberine, morphine, montanire, salsoline, galantamine), **indoles** (e.g., geissospermine), **pyrroloindoles** (e.g., physostigmine), **piperidines** (e.g., piperine, lobeline), **aporphines** (e.g., nantenine), **pyridines** (e.g., nicotine, arecoline), **methylxanthines** (e.g., caffeine, theobromine), **vincas** (e.g., vinpocetine), **lycopodiums** (e.g., huperzino A), **indole β-carbolines** (e.g., harmine) and **erythrine byproducts** (e.g., (+)-erythravine) (Hussain et al. 2018). In the literature, particularly the medicinal and health-promoting effects of piperidines (Haq et al. 2020) and methylxanthines (Martínez-Pinilla et al. 2015; Franco et al. 2013) are highlighted. Black pepper is the major source of piperine (Stojanović-Radić et al. 2019), whereas coffee, tea, cacao, maté and guarana are rich in caffeine and theobromine (Ashihara et al. 2017; Bartella et al. 2019). Some biological effects of alkaloids include analgesic (e.g., morphine) (Singh et al. 2017), anticancer (e.g., berberine) (Xu et al. 2019), antihyperglycemic (e.g., piperine) (Atal et al. 2012), antiarrhythmic (e.g., quinidine) (Vitali Serdoz et al. 2019) and antibacterial (e.g., ciprofloxacin) (Zhang et al. 2018) effects.

1.9 Capsaicinoids

Capsaicin is one of the most well-known capsaicinoid, which is present in red chili peppers and responsible for its pungency flavor. In addition to capsaicin, several other distinct naturally occurring capsaicinoids including dihydrocapsaicin, nordihydrocapsaicin, homocapsaicin, and homodihydrocapsaicin are also present in peppers (Lu et al. 2020). Beneficial roles of capsaicinoids has been reported against

obesity, cardiovascular diseases, gastrointestinal conditions, cancer, neurogenic bladder and dermatologic conditions (Bley et al. 2012; Sharma et al. 2013). In particular, potential role of capsaicinoids in the weight management is well-documented (Ludy et al. 2012). Regular consumption of capsaicinoids has been shown to reduce the abdominal adipose tissue levels, appetite and energy intake. Although the exact mechanism of action is not clear, it is reported that these effects may be caused by stimulation of the TRPV1 receptor (Whiting et al. 2012; McCarty et al. 2015). On the other hand, even though the intake of capsaicin appears to be a safe practice, further studies should be carried out to understand the safety of regular long-term consumption (Tremblay et al. 2016).

1.10 Polysaccharides

Polysaccharides are present in several parts of fungi, plants, algae, animals, and bacteria, and play important roles in many physiological functions (Zong et al. 2012). They are composed of monosaccharide units linked with glycosidic bonds (Mizrahy and Peer 2012). According to their chemical structure, polysaccharides are divided into two groups: (i) **homopolysaccharides**, which are homoglycans consisting of the same monosaccharides and (ii) **heteropolysaccharides** that are heteroglycans consisting of different monosaccharides (Mohammed et al. 2021). Polysaccharides can also be classified according to their origin as those derived from plants (e.g., pectin, inulin, xylans, β-glucan, arabinans, gums), animals (e.g., chondroitin sulfate, chitin, chitosan, heparin, and hyaluronan), bacteria (e.g., exopolysaccharides, capsular polysaccharides, and peptidoglycans), algae (e.g., alginate, agar, carrageenan) and fungi (Ullah et al. 2019). Studies reported that polysaccharides exert a wide range of biological effects including antioxidant (Zhu et al. 2017), anti-inflammatory (Wijesekara et al. 2011), antimicrobial (Zhang et al. 2015), anti-tumour (Casu et al. 2010), anti-obesity (El Khoury et al. 2012), hypocholesterolemic (Behera and Ray 2016), immune modulatory (Loh et al. 2017) and gastro- and neuroprotective activities (Chawla and Patil 2010; Xie et al. 2015).

1.11 Polyunsaturated Fatty Acids

Polyunsaturated fatty acids (PUFAs), including omega-3 and omega-6, are fatty acids that contain more than one double bond in their backbone. Among PUFAs, omega-3 PUFAs attracted particular attention. Omega-3 PUFAs contain a double bond at the third carbon from the methyl end of the chain. The main omega-3 PUFA forms include **α-linolenic acid** (ALA) (C18:3*n*3), **eicosapentaenoic acid** (EPA) (C20:5*n*3) and **docosahexaenoic acid** (DHA) (C22:6*n*3) containing 18, 20 and 22 carbon atoms, respectively (Comunian and Favaro-Trindade 2016). Major dietary sources of omega-3 PUFAs are cold water fish including tuna, salmon, and anchovy

as well as plant seeds such as rapeseed, soybeans, flaxseed, walnuts and chia. While omega-3 PUFAs from plant sources are rich in ALA, omega-3 PUFAs from animal sources contain more EPA and DHA, which possess higher bioactive properties. Besides plants and animals, microalgae are also a good source of omega-3 PUFA, in particular DHA (Hernandez 2014). Several large scale, randomized clinical trials have shown that dietary intake of omega-3 PUFAs possess cardioprotective effects (Endo and Arita 2016). Moreover, omega-3 PUFAs may also prevent obesity related metabolic changes through modulation of lipid metabolism, promotion of adipogenesis and alteration of epigenetic mechanisms (Albracht-Schulte et al. 2018). Omega-3 PUFAs are effective in the management of neurodegenerative diseases such as Parkinson's and Alzheimer's disease in their early stages (Avallone et al. 2019). Another important role of consumption of omega-3 PUFAs is the reduced risk of premature births and improved intellectual development of the fetus during pregnancy (Yashodhara et al. 2009).

1.12 Bioactive Peptides

Bioactive peptides are peptide sequences within a protein that exert beneficial effects on body functions and/or positively influence human health, beyond their known nutritional value (Kitts and Weiler 2003). Majority of bioactive peptides are composed of 2–20 amino acids and in general contain hydrophobic amino acids (Möller et al. 2008). Bioactive peptides are released as a result of enzymatic proteolysis of proteins through gastrointestinal digestion or *in vitro* hydrolysis using proteolytic enzymes. In addition, they can also be liberated during food processing such as cooking and fermentation (Daliri et al. 2017). Another method that is used to produce bioactive peptides is the fermentation of protein of interest with microorganisms, such as bacteria, yeasts, or fungi, to hydrolyze the peptides using microbial enzymes. Proteins from milk (i.e., whey, casein), eggs and meat are the most commonly utilized animal protein sources for the production of bioactive peptides. Plant protein sources include cereals (e.g., wheat, rice, oat, sorghum, etc.), legumes (e.g., soy, lentil, pea, peanut, chickpea, lupin, etc.) and seeds (quinoa, chia, amaranth, hemp, flaxseed etc.), among others. Besides animal and plant proteins, marine sources such as fish, crab, microalgae, and seaweed, are also used to produce bioactive peptides (Chakrabarti et al. 2018). Studies carried out with various bioactive peptides indicated that these compounds exert anticancer (Hung et al. 2014), antimicrobial (Aissaoui et al. 2017), antihypertensive (García-Tejedor et al. 2014), cholesterol-lowering (Marques et al. 2015) and antidiabetic (Siow and Gan 2016) effects.

1.13 Conclusion

In this chapter, we reviewed the health-promoting features of bioactive compounds including polyphenols, carotenoids, vitamins, glucosinolates, triterpenes, phytosterols, alkaloids, capsaicinoids, polysaccharides, polyunsaturated fatty acids and bioactive peptides. Although more scientific evidence is required in order to make dietary recommendations, there is enough evidence to suggest the consumption of food products rich in bioactive compounds. This refers to a diet rich in fruits, vegetables, grains, nuts and oils which may prevent the occurrence of chronic diseases and hence promote human health. On the other hand, there are several factors that may affect the content and bioavailability of these compounds. One of the factors that has a significant influence on bioactive compounds is the effect of food processing. In the following chapters of this book, effects of different food processing methods, including conventional and novel thermal and non-thermal processes, on the retention of bioactive compounds will be discussed in detail.

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Chapter 2
Different Food Processing Technologies: A General Background

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2.1 Brief Overview of Food Processing Technologies

2.1.1 Classification and Development of Food Processing Technology

Foods have been heat-treated since our ancestors learned to master fire for cooking purposes. After that, many different technologies are used in food processing and a number of them have been applied successfully for thousands of years (e.g. drying, baking, pressing, fermentation), some for several centuries (e.g. canning, freezing, evaporation), and some of which emerged only half a century (e.g. extrusion, extraction, novel processes). Over the last decades, there have been a global surge in the demand for healthy foods with fresh-like characteristics; thus continuous efforts have been made to improve and optimize food processing technologies. In spite of their age, all of these food processing technologies have gone through considerable developments and improvements and aim to make the final product more stable and attractive in flavour, appearance, taste and consistency. But, until the last centuries,

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none of the gourmets were aware of the fact that besides transforming the food material, cooking process also makes their foods more digestible, microbiologically safer and more or less nutritive depending on the selected cooking technology. The selected food processing technology is an important factor affecting not only the food composition, but also the intake of bioactive compounds under normal dietary conditions.

The novel food processing technologies are usually classified as thermal and non-thermal, which are different from the conventional processing methods. The term "novel", from the food industry perspective, does not mean that the concept is necessarily new. For instance, the effect of high-pressure processing (HPP) was discovered in the late 19th century and the concepts of pulsed electric field (PEF) and accompanying patents have been reported for more than 50 years. So, novel will refer to technologies that have recently been developed to a point of having a practical value for some products and commercially viable within the usual cost constraints of the food industry. There is a wide range of raw materials (e.g. vegetables, fruits, meat, etc.), and various processing techniques including all the above-mentioned traditional and novel processing methods.

With the availability of modern technology, it is now possible to pay more attention to food processing issues. Nowadays, most of the consumed food can be purchased at the local supermarket as fresh raw products such as meat, fruit, fish, etc. or as manufactured products after industrial processing (canned meat, dried fish, packed fruit, etc.). It is estimated that 80–90% of these consumed foods are semi-processed and therefore it makes sense to consider the beneficial or detrimental effects of processing on safety and quality (Jongen et al. 2005). In addition, both food types are usually submitted to culinary treatments which will transform the selected food into a cooked dish ready to eat. Therefore, it is necessary to discuss the effects of novel or conventional food technologies on beneficial compounds of foods.

2.1.2 Thermal Processes

Traditionally, thermal processing is one of the most commonly used methods in food industry to improve food quality and prolong the shelf life of foods. It is economical in industrial application and can effectively guarantee the food safety in terms of microbiological quality. In addition to the conventional heating methods, other heating methods which mainly use electric physical heating means, include Ohmic heating, microwave heating, radio frequency (RF) heating and so on. Compared with the indirect heating method, the novel heating technologies have the advantages of easy operation, less pollution, high heat utilization rate and good food quality.

The reason why thermal treatment is effective is that the reaction rate increases with the increase of temperature. Chemical, biochemical or microbial reactions increase with the temperature, but beyond a certain temperature, enzymes and

microorganisms will lose their activity. Particularly under severe conditions, it may induce several chemical and physical changes that impair the organoleptic properties and may reduce the content or bioavailability of some bioactive compounds. On a macro level, it will lead to the deterioration of food quality, including the change of color and taste, the loss of aroma and the change of texture (Rawson et al. 2010, 2011a; Patras et al. 2010). Therefore, novel technologies have the potential of retaining nutritional and organoleptic characteristics of food products in a better way. Because of increasing consumers' demand of having ready to eat foods and with technological developments, food materials are processed with different processing methods and stages so that they become hygienic with prolonged shelf-life having higher digestibility of nutrients. These thermal processes cause several biological, chemical and physical modifications leading to sensory, nutritional and textural changes (Kumar et al. 2016; Van Boekel et al. 2010).

2.1.3 Non-thermal Processes

In order to overcome limitations due to the use of thermal processes and meet the consumer demand for processed foods with better characteristics, food researchers are working to further develop new mild non-thermal technologies. Among them, many emerging technologies have shown good potential in obtaining foods with high quality and nutritional value; increasing the sustainability of the food chain by improving the recovery of high added value components; optimizing the biological and sensory quality characteristics of food and avoiding harmful substances produced during thermal processing (Elezmartinez et al. 2006; Nowacka et al. 2014; Heinz and Buckow 2010; Gallo et al. 2018). Therefore, non-thermal physical processing technologies have been deeply studied in recent years. Emerging non-thermal food processing technologies include HPP, radiation, membrane technology, PEF, ultrasonic wave, ozonation, plasma and nano-based technology. Most of the non-thermal food processing technologies can maintain fresh quality, nutritional value and safety of foods and have been widely recognized. Non-thermal processing technology doesn't only play the role in sterilization and enzyme inactivation, but also improve the functional properties or nutritional value of foods. Especially with the rise of nanotechnology in recent years, effect of nano-based processing methods on food quality and functional characteristics is a frontier research direction (Chap. 19). Table 2.1 summarizes the advantages and disadvantages of these typical non-thermal processes in comparison to thermal processes.

Table 2.1 Advantages and disadvantages of different food processing technologies

Process	Advantage	Disadvantage	Conditions	Comments	References
Thermal processes	Safety Nutritional value largely maintained Inactivation of anti-nutritional factors and some allergens Inactivation of enzymes Sensory attractiveness Simple operation Cost effective	Formation of undesired compounds (e.g. acrylamide) Loss of freshness and related sensory attributes	Pasteurization: 60–70 °C Sterilization: 110–150 °C	Inactivation of cells Inactivation of spores Formation of desired flavour and texture (e.g. Maillard reaction)	Rawson et al. (2011a), Van Boekel et al. (2010)
Irradiation	Nutritional value largely maintained Use of room temperature or low temperature	Sensory changes frequently Enzymes and microbial toxins are not inactivated Radioactivity is harmful to human body	Sterilization: 25–50 kGy	Treatment of packaged food Strong dose limits	Lacroix and Ouattara (2000), Farkas (1998)
High pressure processing	Safety No formation of undesired compounds Nutritional value largely maintained Retention of freshness Physical modification	Desirable flavour compounds generated by heat are not formed	Pasteurization: 600 MPa, ambient temperature (batch process) Sterilization: 600 MPa, 121.1 °C rapid, homogenous heating and cooling due to adiabatic heating (batch process)	Inactivation of vegetative micro-organisms and spores Possible inactivation of enzymes, viruses and prions (p, T dependent) Treatment of packaged food Cold storage (41 °C) or storage at ambient temperature	Heremans (2002), Mujicapaz et al. (2011)

(continued)

Table 2.1 (continued)

Process	Advantage	Disadvantage	Conditions	Comments	References
Pulsed electric field	Safety No formation of undesired compounds Gentle processing, retention of freshness Cell disintegration Improvements of mass transfer processes Physical modification	Spores are not inactivated Desirable flavour compounds generated by heat are not formed Inactivation of anti-nutritional factors not achieved No inactivation of enzymes	Cell disintegration: 1–5 kV/cm 1–10 kJ/kg Non-thermal pasteurization: 25–40 kV/cm 50–200 kJ/kg Short processing times Continuous operation	Intensity-dependent occurrence of electrochemical and thermal side effects Refrigerated storage of products required	Teissie et al. (2005), Zulueta et al. (2010), Corrales et al. (2008)
Ultrasound treatments	Nutritional value largely maintained (especially heat sensitive substances) Physical modification Suitable for combination with other methods Environmentally friendly technology	Insufficient sterilization efficiency Induce lipid degradation, produce unpleasant odors High energy consumption	Power ultrasound: 20–100 kHz High-frequency ultrasound: 100 kHz–1 MHz	Blunt enzymes Cannot achieve satisfactory sterilization	Nowacka et al. (2017), Zheng and Sun (2006), Inguglia et al. (2018)
Membrane processing	Safety No formation of undesired compounds	Desirable flavour compounds generated by heat are not formed Inactivation of anti-nutritional factors not achieved	Microfiltration: 0.1–1 µm Ultrafiltration: 0.001–0.1 µm Nanofiltration: 1–2 nm	Only applicable for liquids Expensive for complete products Added value for separation of ingredients (e.g., lactoferrin)	Castromunoz et al. (2017), Conidi et al. (2014)

(continued)

Table 2.1 (continued)

Process	Advantage	Disadvantage	Conditions	Comments	References
Plasma Processing	Low temperature treatment Less damage to nutrients Maximum preservation of the original sensory characteristics Energy efficient	Additional safety measures are needed to destroy and discharge these gases Desirable flavour compounds generated by heat are not formed Low penetrability, unable to kill bacteria in food	Pulses of high voltage 20–80 kV/1–100 kHz Pulsed ultraviolet-light: Pulse width < 2 ms Cumulative level <12 J/cm^2	Need to be precisely controlled Not suitable for high fat food, can cause oxidation Expensive for complete products	Cullen et al. (2010), Palumbo (2007)

2.2 Effect of Different Food Processing Technologies on Food Bioactives

Processing of food products may have positive or negative effects on bioactive compounds as do the conditions during its passage along the gastrointestinal tract (Wang and Bohn 2012). In fact, processing techniques are becoming more sophisticated and diverse in response to the growing demand for high quality foods, and the impact of food processing on the bioactive compounds has been indicated not to be a simple cause-effect relationship. So, efforts have been made during processing to comprehend and reduce the adverse impact of applied technology on key bioactive components of food which is critical from the nutritional quality point of view. In the following sections, we will briefly discuss the influence of different food processes and more details have been provided in the next relevant chapters.

2.2.1 Conventional Processes

Traditional processing technologies including thermal treatments, baking, drying, freezing and fermentation are used to preserve manufactured foods. On the other hand, cooking or other food preparation steps, i.e. pretreatments on raw material, may be applied to obtain foods. For example, thermal treatment is an important food preservation unit operation which includes pasteurization and sterilization together with other methods such as steaming, boiling, roasting and so on. It can be applied to either solid or liquid products; to prepare the product (i.e. cooking), to develop

Table 2.2 Selected examples on the influence of different processing technologies on the content of bioactives in food products

Product	Process	Conditions	Parameters affected	References
Fruit (açai species, euterpe oleracea and precatoria)	Bleaching	80 °C/1–60 min	(—) Non-anthocyanin polyphenolics (flavone glycosides, flavonol derivatives and phenolic acid concentrations, etc.) (↓) Anthocyanins (↓) Antioxidant capacity	Pacheco et al. (2009)
Puree (tomato and carrot)	Thermal	70 °C/2 min	(↓) Ascorbic acid (—) Total phenolic (—) Total carotenoids	Patras et al. (2009a)
	HPP	400–600 MPa/15 min/20 °C	(↓) Ascorbic acid (—) Total phenolic (↓) Total carotenoids (400–500 MPa) (↑) Total carotenoids (600 MPa)	
Fruit (Indian gooseberry)	Drying	65 and 75 °C/7, 10 and 13 kPa	(↓) Ascorbic acid (↓) Chlorophyll	Methakhup et al. (2005)
Puree (sweet cherry)	Fermentation (lactic acid bacteria)	60 days/4 °C	(—) Total phenolic (↑) Free phenol (↑) Anthocyanins (↑) Antioxidant activity	Cagno et al. (2011)
Juice (black mulberry)	Microwave heating	300 W/95–140 min/100, 38.5, and 7.3 kPa	(↑) Anthocyanin (—) Antioxidant capacity	Fazaeli et al. (2013)
Vegetables (Broccoli)	Microwave cooking	500–1000 W/ 5 min	(—) Vitamin C, (↑) Vitamin E (↑) β-carotene, (↑) Lutein (↑) Glucosinolates (↑) Flavonoids and (↑) Phenolic acids	Lopezberenguer et al. (2007), Zhang and Hamauzu (2004)
	Boiling	5 min	(↓) Vitamin C, (↑) Vitamin E (↑) β-carotene (↑) Lutein (↓) Glucosinolates (↓) Flavonoids and (↓) Phenolic acids	Zhang and Hamauzu (2004)
Vegetables (Artichoke heads)	Ohmic heating	24 V/cm/80 °C	(↑) Peroxidase inactivation (↑) Polyphenol oxidase inactivation (↓) Total protein (↓) Total polyphenolic	Guida et al. (2013)

(continued)

Table 2.2 (continued)

Product	Process	Conditions	Parameters affected	References
Grain (Maize)	Infrared heating	110–140 °C/ wavelength ranging (1.8–3.4 μm)	(↓) Total phenolic (↑) Hydroxymethylfurfural	Žilic et al. (2013)
Condiments (paprika)	Radio frequency heat treatment	13.5 MHz/95, 105, 115 °C	(—) β-carotene (↓) Total carotenoids (—) α-tocopherol (—) Total Tocopherols (—) Vitamin C (—) ASTA value	Molnar et al. (2018)
Condiments (paprika)	Microwave heating	800 W/10 min/80 °C	(—) β-carotene (↓) Total carotenoids (—) α-tocopherol (↓) Total Tocopherols (—) Vitamin C (—) ASTA value	Molnar et al. (2018)

Where (↓) refers to decrease in the level
(↑) refers to increase in the level
(—) refers to no significant change in the level

desired flavours, aroma and color components (i.e. Maillard reaction), modify the food structure, or to preserve or sterilize the food by heat induced inactivation of microorganisms, toxins and enzymes (Cilla et al. 2017). Conventional thermal processing methods also include dehydration and canning (whole or in pieces). In addition, foods, particularly fruits and vegetables, are widely processed into juice, smoothies, soups, nectar, etc. (Rawson et al. 2011a). Table 2.2 summarizes some of the studies on the effect of thermal processing methods on bioactive components in different foods. However, despite the improvement in food safety achieved with thermal treatments, there is often a loss in the content of bioactive compounds and change in the bioaccessibility and/or bioactivity of these bioactives and nutrients which are essential for the human diet.

2.2.1.1 Blanching

Blanching is a process that contributes to the inactivation of enzymes, thereby preserving the color and nutrients of the product. Blanching can be done in many ways, including water, steam, vacuum steam, canning, and hot air. The most common method of water blanching is applied at 75–95 °C (1–10 min). Blanching is often used as a pre-treatment of many processing techniques, such as freezing and drying. However, due to the applied heat, it can lead to the loss of heat-sensitive compounds. There are some papers on different fruits including pineapple chunks and papaya which reported to lose their pigments and turn into white at a certain blanching temperature and time. There is also loss of heat-sensitive phytochemicals (carotenoids, anthocyanins, ascorbic acid) in the blanched fruits of mango, acai fruit, pineapple, and papaya (Pacheco et al. 2009; Patras et al. 2009a). In another study,

degradation of anthocyanins during heat treatment at 80 °C for 1–60 min was investigated in cupuaçu (*Theobroma grandiflorum*) nectar, and ascorbic acid was observed to degrade to dehydroascorbic acid (Vieira et al. 2000).

2.2.1.2 Drying

Drying process helps to extend the shelf-life foods and reduce their volume (Prakash et al. 2004) (Chap. 5). A number of dehydration procedures are employed for fruits and vegetables such as solar drying, heated air drying, microwave drying, osmotic air drying, foam-mat, spray and freeze drying. Even though drying increases the shelf life of the fruits, it takes a very long time even at high air temperatures, and may affect the content of bioactive compounds such as ascorbic acid, polyphenols, and carotenoids. During drying, as a result of some redox reactions, polyphenols with an intermediate oxidation state can exhibit a higher radical scavenging activity than non-oxidised polyphenols. High temperature drying could further cause the formation of Maillard reaction products, and these antioxidants have been shown to have synergistic effects (Fu 2004; Papetti et al. 2002; Piga et al. 2003).

Hot air drying is usually accompanied by the degradation of polyphenols, but the breakdown of polyphenols has been shown to depend on the food substrate and processing conditions. In addition, this process increases or decreases the antioxidant activity of vegetables, depending on the nature of the substrate. Some novel drying methods (microwave, vacuum drying, etc.) resulted with better color and texture than the same fruits dried by traditional convective methods (Yongsawatdigul and Gunasekaran 1996). Studies on drying of guava, kiwi, pineapple, etc., showed that ascorbic acid retained at a higher rate under the conditions of low-temperature super-hot steam drying and vacuum drying, while the retention rates were lower during direct sun drying and indirect sun drying (Nicoli et al. 2000; Methakhup et al. 2005; Uddin et al. 2002; Kong et al. 2010). On the other hand, carotenoids were found to be more stable during drying than anthocyanins. In addition, gallic acid and total hydrolytic bacteriocins were relatively stable during the drying process (Saxena et al. 2012).

2.2.1.3 Fermentation

Fermentation is a non-thermal process in which chemical changes caused by enzymes produced by bacteria, or yeasts are observed. As one of the oldest known food preservation technologies, this process involves the action of the required microorganisms or their enzymes on food ingredients, causing biochemical changes and leading to significant changes in food (Chap. 9). During fermentation, carbohydrate energy sources in foods, such as lactose in milk, are converted to lactic acid, as in yoghurt and cheese. Yeasts (typically of the species *Saccharomyces*) convert glucose to ethanol and carbon dioxide during bread making, and in the production of alcoholic beverages (Vogel et al. 2011). The benefits of fermentation are

shelf-life extension, improvement of the nutritive value of the food, enhancing the taste and digestibility of foods and indeed improved food safety e.g. by low pH and elimination of anti-nutrients during the fermentation process (Van Boekel et al. 2010).

For the substances with antioxidant activity, the acidic conditions produced during fermentation are more conducive to their preservation. For example, the amount of ascorbic acid in fermented tomato juice was 120–140 mg/L, which was much higher than that the control group (60–90 mg/L) (Cagno et al. 2009). Fermentation can produce some phenolesterases (such as ferulic acid esterase) that hydrolyze some bound phenols, release a large number of organic acids, and prevent the degradation of phenols, thus improving antioxidant activity. In addition, some phenolic acid decarboxylases can be produced that result in conversion between phenolic substances (Szwajgier et al. 2012). For example, after 2 weeks of fermentation with *Leuconostoc*, the total phenol content was determined to be three times higher compared to pre-fermentation (Kusznierewicz et al. 2008). It was also found that the content of free phenols increased after fermentation, mainly anthocyanins content increased after fermentation, and the antioxidant activity also increased (Cagno et al. 2011). In another study, after 72 h of fermentation of red onion with *Lactobacillus plantarum* S1, the amount of quercetin diglycoside decreased from 58.3% to 18.3%, while quercetin monoglycoside amount increased from 41.6% to 59.7%, and the latter had higher antioxidant activity compared to the former (Bisakowski et al. 2007). During fermentation, microorganisms such as lactic acid bacteria can also transform the components of original food matrix into more valuable bioactive substances. For example, monosaccharides in food can be converted into extracellular polysaccharides with anti-tumor activity to improve nutritional value, and L-glutamic acid can be converted into γ-aminobutyric acid with antihypertensive activity (Ruasmadiedo et al. 2002; Di Cagno et al. 2010). After microbial fermentation, some active macromolecular substances are transformed into small molecular substances which can be directly absorbed by animal intestines, which contribute to metabolic absorption.

2.2.2 Novel Thermal Processes

2.2.2.1 Microwave Heating

Microwave heating uses electromagnetic waves with a wavelength of 1 mm−1 m that pass-through food materials and cause their molecules to oscillate, generating heat (Chap. 12). The polar molecules in food rotate with the rapid change of the polarity of high-frequency electromagnetic field. The heat generated by friction between molecules, results in a thermal effect on food. In addition, high-speed molecular oscillation excites polar molecules to constantly change their orientation to produce a non-thermal effect. Microwave heating technology has a high heating rate as it does not need the heat conduction process. The polar molecules usually

rotate 9.15×10^8 times or 2.45×10^9 times in 1 s, which shows that the heating rate is very fast. The interaction between microwave electromagnetic field and foods has two distinct effects. On the one hand, microwave energy is transformed into the heat energy of material heating. On the other hand, there are interactions between bioactive components (such as proteins and enzymes) or mixtures (such as bacteria and molds) in materials, so that their biological activities can be inhibited or stimulated (depending on the frequency and intensity of microwave electromagnetic field, etc.). The former is called the heating effect of microwave on materials, and the latter is called non-thermal effect or biological effect.

Microwave heating treatment of blueberry juice was reported to take place in a short period of time, result in lower loss of phenols and color, which can maximize the quality of food materials (Elik et al. 2016). Microwave heating also results in selective heating. Food materials with different properties have different absorption degrees of microwaves, and the higher the water content, the faster the heating rate of food materials. At different operating pressures (12, 38.5 and 100 kPa), microwave heating was found to be superior to conventional heating in terms of color retention, anthocyanin and total phenol contents, and antioxidant activity of concentrated juice. Under the condition of evaporation pressure of 38.5 kPa, using higher microwave power (600 W instead of 450 W) was reported to protect the quality characteristics of products in a better way. It was indicated that microwave heating could also reduce the degradation of anthocyanins in juice, and the anthocyanin content and color could be maintained better at a lower pressure of 7.3 kPa (Schnepf and Driskell 1994; Fazaeli et al. 2013). In addition, the retention rate of bioactive substances (vitamin C, vitamin B6, minerals) in vegetables including cruciferous vegetables, potatoes, corn, and peas by microwave heating was higher compared to that of conventional heating methods such as steaming and boiling. The loss of glucosinolates during microwave cooking was not found to be significant, and the content of glucosinolates was reported to remain unchanged (Gliszczynskaswiglo et al. 2006; Lopezberenguer et al. 2007; Zhang and Hamauzu 2004).

2.2.2.2 Ohmic Heating

Ohmic heating is also known as Joule heating. Its mechanism of action is based on the conductivity of food materials (Chap. 13). Its conductivity mode depends on the directional movement of electrolyte solution ions or molten electrolytes in food materials (Reznick 1996). Most food materials contain ionizable acids or salts, and show certain electrical resistance or electrical impedance characteristics. When the two ends of food materials are applied with electric field, the resistance of food materials can generate heat under the action of current flowing into the food materials, so that the materials can be heated. Ohmic heating technology can be widely used in various processes including food sterilization, enzyme inactivation, blanching, thawing, fermentation and other processing technology (Sensoy and Sastry 2004). Compared with the traditional heating method, Ohmic heating is more suitable for high-temperature instantaneous heating of high protein raw materials, high

viscosity raw materials, solid-liquid mixed food, and can maximize the freshness and flavor of food (Wang and Sastry 2002). The electrical conductivity of food materials containing non-conductive substances such as fat, air, ethanol, bone, and ice is low, while that of milk, fruit and juice, vegetables, pickled food, olive, protein and egg yolk is higher. Therefore, temperature, food composition, material microstructure, electrolyte concentration, particle size, species, quantity, field strength and so on will affect the characteristics (Jaeger et al. 2016; Choi et al. 2015; Tian et al. 2016).

Ohmic heating can be used for sterilization of fruit juices and dairy products, blanching of fruits, vegetables and grains, peeling of fruits and vegetables, and extraction of pigments from plants. It has been reported that ohmic heating has several advantages over traditional hot water blanching, including less nutrient extraction, high efficiency, and can also improve product quality and maintain the color and nutritional value of food. When Ohmic heating technology is applied to blanching apple pieces, the loss of vitamin C in the raw material is about 20% (Guida et al. 2013). The inactivation effect of ohmic heating on polyphenol oxidase and peroxidase is better than that of traditional blanching method, which can be ascribed to its ability to heat food products quickly and uniformly, thus leading to a mild thermal treatment (Guida et al. 2013; Leizerson and Shimoni 2005; Castro et al. 2004).

2.2.2.3 Infrared Heating

The principle of infrared (IR) heating is to transfer energy by IR radiation heat transfer process. The application of IR heating technology in food processing is mainly by far-IR radiation, because most of the components of food absorb IR radiation mainly in the far-IR band (Krishnamurthy et al. 2008). When IR wavelength radiated by IR radiation source is consistent with the wavelength of the heated object, the heated object absorbs a large amount of IR energy, which makes the atoms and molecules inside the object resonate, thus friction and heat are generated between each other, so that the temperature of the heated object is increased and the object is heated quickly and effectively. IR drying technology is mainly used in the processing of dehydrated fruits and vegetables, such as potatoes, onions, apples and other fruits and vegetables. IR heating is also used to inactivate enzymes in fruits, vegetables and grains, which can improve their quality and prolong their shelf life (Khamis et al. 2011).

For grain products, IR heating is reported to improve flavor and digestibility, enhance rheological properties and antioxidant capacity of bakery products (Mcallister and Sultana 2011; Žilic et al. 2010). IR heating treatment in 60s could inactivate 95.5% of lipoxygenase in soybean, and the quality of soybean itself will not be affected compared with the traditional enzyme-inactivation process (Kouzehkanani et al. 1982). On the other hand, IR heating may result in the loss of heat-sensitive nutrients including amino acids and phenolic compounds (Žilic et al. 2006, 2013).

2.2.2.4 Radio Frequency and Dielectric Heating

RF is non-ionizing electromagnetic waves with the frequency range between 1 and 300 MHz and wavelength of up to 11 m (Guo et al. 2011). Both RF and microwave heating process belong to dielectric heating process, and its principle can be simply described as the process of converting electromagnetic energy into heat energy in the material (Hou et al. 2016). In the process of dielectric heating, due to the different heating mechanism, food in electromagnetic field is generally dominated by the rotation of polar molecules or ion conduction. RF heating process mainly generates heat by the rotation of ions, and the constant change of electric field leads to the ions always moving in the direction with opposite charges, resulting in the continuous collision and friction heat generation between ions (Guo et al. 2008). The factors that affect RF heating are mainly related to the characteristics of food materials including structure and water content, among which water content is the most important factor affecting dielectric properties and improving heating uniformity (Sacilik and Colak 2010). In addition, the heating conditions such as frequency, density, temperature and other factors also affect the RF efficiency.

The use of RF as a heating source in food drying is advantageous due to its potential for rapid and constant heating rates compared with conventional heating methods (Marra et al. 2009). This is mainly because of the advantages of overall heating and large penetration depth. RF drying can reduce the phenomenon of food surface crusting and improve the mass transfer efficiency. It is suitable for removing water that is difficult to remove in the slow down stage of traditional drying process, significantly shorten the drying time and result in dried food products with better quality (Wang and Tang 2001; Hou et al. 2015).

In addition, RF technology combined with hot air drying can result in more efficient food drying processes (Hassan et al. 2019). The drying time of nuts and grains by hot air and RF heating is 35–50% shorter than that of hot air alone, and the peroxide value and free fatty acid value are lower, and hence the product quality is better. The research on the effect of RF heating on the quality of corn and almond proved that the quality characteristics of almond were improved after RF treatment (Yang et al. 2018; Wang et al. 2014; Li et al. 2017). Even, RF heating can improve the oil holding and emulsifying properties of corn flour, improve the nutritional value of corn flour, and increase the protein solubility of corn flour, especially when the temperature is up to 55 °C (Hassan et al. 2016). The bioactive substances (β-carotene, α-tocopherol, vitamin C) content of capsicum powder treated at frequency of 13.5 MHz and temperature of 95 °C, 105 °C and 115 °C, respectively, were reported to remain almost unchanged (Molnar et al. 2018).

2.2.3 Novel Non-thermal Processes

2.2.3.1 Irradiation

Irradiation is generally divided into ionizing radiation and non-ionizing radiation. Ionizing radiation includes gamma ray, high energy electron beam and X-ray, while non-ionizing radiation includes ultraviolet ray, infrared ray, etc. (Piyasena et al. 2003; Farkas 1998). Food irradiation technology involves treating food with ionizing or non-ionizing radiation to inactivate bacteria, viruses and insects. It is a kind of food processing technology to prolong the storage time and improve the quality of food (Chap. 14). In ionizing radiation, the penetrating ability of X-ray, α-ray, β-ray and γ-ray is different. Although X-rays are more penetrating than α-rays and β-rays, their application in destroying foodborne microorganisms is limited due to the difficulty of focusing these rays on food (Lacroix and Ouattara 2000). The penetration of α and β-rays is low enough to preserve food. In contrast, gamma rays have a high penetrability. The dose of ionizing radiation that results in formation of positive and negative charges in food can be used to eliminate food borne microorganisms. Non-ionizing radiation is electromagnetic radiation that does not carry enough energy/quantum to ionize atoms or molecules (Rawson et al. 2011a).

Food irradiation was reported to have minor effects on the flavor, color, nutritional properties, taste and other quality attributes of food (Alothman et al. 2009a, b). However, the level of modification (in terms of flavor, color, nutrients, taste, etc.) may vary depending on the raw materials used, the radiation dose and the type of radiation source used such as gamma, X-ray, ultraviolet, and electron beam (Bhat and Sridhar 2008). Radiation can result in DNA damage and impair the reproductive capacity and other functions of cells (Deruiter and Dwyer 2002). By adjusting the radiation dose, food can be pasteurized to reduce or eliminate food borne pathogens. According to the report, gamma radiation and ultraviolet radiation was applied to inactivate microorganisms in fruits, and the effect of irradiation on bioactive compounds was reported to be minor, because the temperature rise of food during processing was not significant.

In the experiment of low dose irradiation (1–3.1 kGy), no significant effect was observed in the content of total phenolics in fruits and vegetables (Alighourchi et al. 2008; Keyser et al. 2008). However, when the dose exceeded 1.5 kGy, the content of ascorbic acid in fruits and vegetables decreased. There was no significant change in carotenoid content. The contents of total anthocyanins and single anthocyanins in pomegranate juice decreased significantly after irradiation with high dose (3.5–10 kGy) (Reyes and Cisneroszevallos 2007). The effect of irradiation on anthocyanins depends on the properties of anthocyanins. For example, diglycoside is relatively stable to irradiation dose compared with monosaccharide. Generally speaking, the reduction of antioxidant compounds is attributed to the formation of radiation-induced degradation products or the formation of free radicals (Sajilata and Singhal 2006; Wong and Kitts 2001). On the other hand, UV irradiation of plant tissues has been shown to have a positive interaction, indicating an increase in

enzymes responsible for flavonoid biosynthesis. In addition to abiotic stress, it also affects phenolic metabolites and induces anthocyanin biosynthesis (Lopezrubira et al. 2005). It was reported that the contents of total phenolics, flavonoids and vitamin C in food were significantly decreased by UV treatment (0.5–14 J/m^2), while anthocyanins and antioxidant activities had no significant changes (Gonzalezaguilar et al. 2007; Alothman et al. 2009a, b). The contents of total phenolics and flavonoids in some foods (such as guava and banana) were reported to increase significantly with the increased processing time.

2.2.3.2 High Pressure Processing

HPP technology is a well-developed non-thermal food processing and preservation technology. It uses 100–1000 MPa pressure to process food to achieve the purpose of sterilization, and enzyme inactivation. In HPP, a liquid (oil or water) is used as pressure conducting medium to pressurize the sample, and high pressure is applied uniformly and instantaneously through food materials (Heremans 2002). The biggest advantage of this method is that the pressure at a given position and time is the same in all directions, the pressure is transmitted uniformly through the pressure transmission medium, and is not affected by the geometric characteristics of food (Oey et al. 2008a) (Chap. 15). In order to achieve sterilization effect, high hydrostatic pressure (HHP) needs to be combined with high temperature (Mujicapaz et al. 2011). HHP treatment is widely used in juice, beverage, fruit, vegetable, meat, fish and seafood products (Heinz and Buckow 2010; Pereira and Vicente 2010).

HPP mainly acts on the non-covalent bonds of food components, such as hydrogen bonds, hydrophobic bonds and ionic bonds. It can minimize the loss of quality and has little impact on the natural color, flavor, taste and texture of food. Meanwhile, it can also reduce the loss of polyphenols and other bioactive plant components during processing (Matser et al. 2004; Tadapaneni et al. 2014). Among the studies on the effects of HHP on nutrients and bioactive substances in food, the most abundant research is on vitamin C (Patras et al. 2009b). Vitamin C is highly sensitive to heat and can degrade rapidly during heat treatment, so it can be used as an index of nutritional quality of products. However, the decrease of vitamin C was observed in different food products treated with HHP and traditional heat treatment methods respectively. Compared with the corresponding heat treatment, HHP can reduce the loss of vitamin and improve its content during storage (Oey et al. 2008b). Table 2.3 lists some reports on the effect of HHP on vitamin content in different food matrices compared with traditional heat treatment methods. Some studies also showed that the content of vitamin C in pomegranate did not change after being treated at 600 MPa and 25 °C for 15 min (Yen and Lin 1996). The retention rate of ascorbic acid in pressure treatment (500 MPa/35 °C/5 min) was higher than that of traditional pasteurized orange juice (80 °C/30s) (Polydera et al. 2004). After treatment with 600 MPa for 15 min, the loss of vitamin C in tomato was only 7%, while that of heat treatment was 54% (Patras et al. 2009c). Compared with the heat-treated sample, the loss of vitamin C in orange juice treated with 400 MPa for 1 min at 40 °C was

Table 2.3 Effect of high pressure processing (HPP) on bioactive content of different food products

Bioactive compound	Product	Conditions	Effect	References
Vitamin C	Tomato purees	400–600 MPa 20 °C 15 min	7% decrease in vitamin C after HHP compared with thermal processing (−54%)	Patras et al. (2009c)
Vitamin C	Litchi based mixed fruit beverage	200–600 MPa 30–70 °C 0–20 min	Loss of 3–27% against 42% of pasteurized product	Jayachandran et al. (2015)
Vitamin C	Pomegranate	600 MPa 25 °C 15 min	No changes after HHP	Yen and Lin (1996)
Vitamin C	Orange juice	400 MPa 40 °C 1 min	8% lower loss of vitamin C compared with thermal treated sample	Plaza et al. (2006)
Vitamin A	Orange juice	400 MPa 40 °C 1 min	Loss of vitamin A 38.74%	Sanchez-Moreno et al. (2005)
β-carotene and lycopene	Tomato pulp	HPP: 500–700 MPa, 30 °C, 10 min Pressure-assisted thermal processing (PATP): 500–700 MPa, 100 °C, 10 min Thermal processing: 0.1 MPa, 100 °C, 10 min	12% increases in lycopene extractability. The bioaccessibility of carotenoids was not significantly different among the treatments (Retention: 60–95%)	Gupta et al. (2011)
Anthocyanin (Delphinidin-3-rutinoside, D3R)	Blackcurrant	200–800 MPa, 18–22 °C 15 min	Greater stability at 800 MPa for D3R	Kouniaki et al. (2004)
Anthocyanin (Pelargonidin-3-glucoside, P3G)	Strawberry	200–800 MPa, 18–22 °C 15 min	Greater stability at 800 MPa for P3G	Zabetakis et al. (2000)

reduced by 8% (Plaza et al. 2006). Luo et al. observed that HHP at 50 MPa for 1 min allowed to maintain ascorbic acid content in green plum while heating reduced it by 17% and the stability of vitamin C during high pressure treatment mainly depends on the concentration of oxygen produced by the aerobic degradation pathway (Luo et al. 2019). In general, the loss of vitamin C varies with the presence of endogenous oxidants such as matrix, enzyme or metal ions, and the level of applied pressure.

In a study on the total antioxidant capacity of orange juice treated with different processing techniques, compared with the traditional high temperature pasteurization treatment, the total antioxidant retention rate of the orange juice treated with

HPP under different storage conditions was higher (Tiwari et al. 2009). Other studies have shown that the bioactive components of strawberry were stable under the pressure of 400–600 MPa (Mcinerney et al. 2007). Although the anthocyanin content decreased slightly, the anthocyanin content was hardly affected compared with the traditional heating method. The contents of anthocyanins in juice and jam (strawberry juice, jam, blackcurrant juice, raspberry juice and black berry puree) treated with high pressure (600 MPa) at ambient temperature were reported to be stable (Aaby et al. 2007; Kouniaki et al. 2004; Garciapalazon et al. 2004; Zabetakis et al. 2000). Compared with heat treatment under environmental pressure, the degradation rate of total anthocyanins in blueberry juice treated by pressure and temperature was reported to be slightly faster during storage (Suthanthangjai et al. 2004). With regard to other bioactive substances, various studies have indicated that high pressure can increase the permeability of plant cells, promote the extractability of vitamin A and flavonols, carotenoids and lycopene, which in turn results in improved bioavailability (Sanchezmoreno et al. 2005; Phunchaisri and Apichartsrangkoon 2005).

2.2.3.3 Pulsed Electric Field, Pulsed X-Ray and Pulsed UV Processes

PEF is a processing method that repeatedly applies high voltage and short pulse to the flowing materials of two electrodes, in order to inactivate enzymes and microorganisms, improve the safety and stability of food, and extend the shelf life (Teissie et al. 2005; Tylewicz et al. 2017). The typical PEF treatment system of pumping products is shown in Fig. 2.1. One of the main differences in the use of PEF lies in

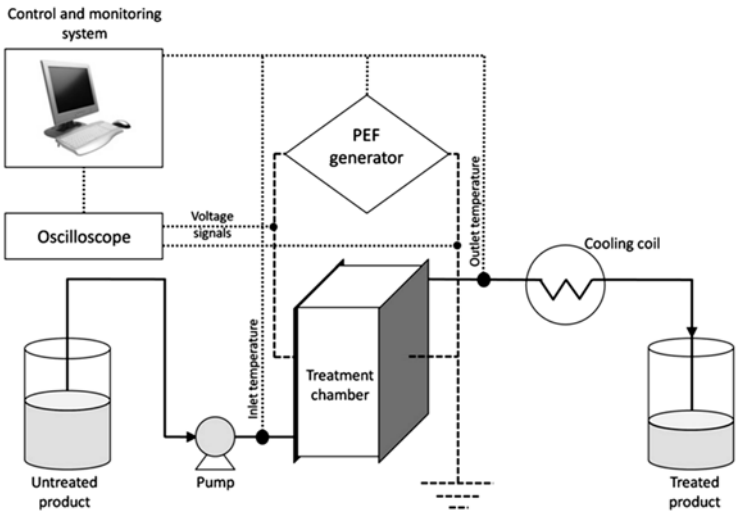

Fig. 2.1 Schematics of a PEF processing system for food products. Adapted from Soliva-Fortuny et al. (2009)

the strength of the electric field used. High-intensity electric fields (15–40 kV/cm, 5–100 pulses, 40–700 μs, 1.1–100 Hz) are more effective in inactivating microorganisms, while low and moderate electric fields (0.6–2.6 V/cm, 5–100 pulses, short processing time in $10^{-4} - 10^{-2}$ s; 1 Hz) have been successfully used to enhance mass transfer in solid foods (Zulueta et al. 2010; Corrales et al. 2008).

PEF technology has been widely studied in food preservation and processing. PEF treatment, especially at low and medium strength, has several advantages including increased extraction rate, reduced treatment time and saving energy (Barba et al. 2015; Tylewicz et al. 2016; Donsi et al. 2010). Several studies have shown that PEF can better retain and/or extract bioactive compounds. For example, PEF treated apples (Dziadek et al. 2019), mangoes (Kumar et al. 2019), and orange juice (Sanchezmoreno et al. 2005; Elezmartinez et al. 2006) were reported to contain higher amounts of vitamin C. In addition, the shorter the duration of PEF treatment, the higher the retention rate of vitamin C, which is also found in the study of PEF treated juice. In general, longer PEF treatment durations may result in reduced vitamin C retention in the product due to heating. Long term exposure can also produce free radicals, which accelerate the degradation of vitamin C.

It was shown that changes in polyphenol content due to PEF treatment was similar to the trend observed in vitamin C. Some authors observed that, the contents of flavanone and total isoflavones remained unchanged at 35 kV/cm (750 and 4 ms bipolar pulses at 800 Hz), while the contents of hesperidin increased in PEF treated juice (35 kV/cm, 4 ms bipolar pulses at 200 Hz, for 800 or 1400 ms) (Torofunes et al. 2015; La Pena et al. 2011). The total phenol content of orange, kiwi fruit, pineapple juice and soybean milk were not affected by PEF (La Pena et al. 2010a, b). However, some experiments have shown that some high-intensity PEF (field strength 25–35 kV/cm, frequency 50–250 Hz, pulse width 1–7 μs and treatment time 50–2050 μs) can increase the loss of vitamin C and increase the lycopene retention rate (87.6–121.2%). The increase of lycopene content may be due to the increase of cell permeability induced by PEF, resulting in the release of intracellular pigment (lycopene) (Omsoliu et al. 2009; Guderjan et al. 2005). On the other hand, anthocyanins in PEF treated foods were reported to be stable (Altuntas et al. 2010).

The application of PEF can also improve the extraction efficiency of bioactive compounds from various by-products and wastes in food processing line. Unlike the PEF application for preservation purposes, the electric field intensity for improving the extraction yield of food metabolites is generally in the range of 1–10 kV/cm (Adeomowaye et al. 2000; Lopez et al. 2009). Under these electric field intensities, the inactivation of microorganisms is negligible for most species, and the degree of cell perforation is often reversible. For example, an increase in the production of active compounds was reported for apple, orange peel, grape, carrot, fresh tea, blackberry, blueberry and coffee bean shell, etc. (Luengo et al. 2013; Corrales et al. 2008; Pataro et al. 2017; Elbelghiti et al. 2007; Belghiti and Vorobiev 2004; Lopez et al. 2009; Liu et al. 2019b; Barbosapereira et al. 2018). PEF is also used in the treatment of animal derived food by-products including meat processing wastes and fish by-products), which can significantly increase the extraction efficiency and

improve the sustainability of the production process compared with the traditional methods.

2.2.3.4 Ultrasound Treatments

Ultrasound is a mechanical wave (16 kHz–100 MHz) whose frequency exceeds the range of human hearing (Mcclements 1995). According to the frequency and productivity of ultrasonic wave, it can be divided into two ranges: low frequency high field intensity (frequency 16–100 kHz, field strength 10–1000 w/cm^2) and high frequency low field strength (frequency 100 kHz–1 MHz, field strength<1 w/cm^2). Ultrasonic with high frequency and low field strength is mainly used to analyze the physical and chemical properties of food, such as hardness, sugar content and acidity of food. Low frequency and high field strength ultrasound can produce hole effect, which can change the physical and chemical characteristics of food materials through the strong pressure and shear force generated in the medium, thus affecting the quality of food (Nowacka et al. 2017; Zheng and Sun 2006) (Chap. 16). In food technology, it can be used for various processes based on heat and mass transfer (e.g., drying, freezing and thawing, extraction, filtration, crystallization or emulsification). In the recent 10 years, high-power ultrasound has been successfully applied to inactivate food enzymes and microorganisms (Inguglia et al. 2018), becoming an alternative to traditional heat treatment methods such as pasteurization. Ultrasonic treatment alone or combined with heating and/or pressure application is an effective tool for microbial inactivation and extraction of bioactive substances.

The advantages of sonication include shorter processing time and lower energy consumption. In addition, there is evidence that ultrasound can improve the nutritional quality of products (Zenker et al. 2003). Figure 2.2 shows two known ultrasonic degradation pathways of ascorbic acid. Compared with heat treatment, ultrasonic treatment has little effect on ascorbic acid content in fruit juice processing, and improves the stability during storage. This positive effect of ultrasound is believed to be due to the effective removal of occlusive oxygen from juice (Knorr et al. 2004), which is a key parameter affecting ascorbic acid retention. Compared with the convective air drying, the vitamin C retention rate of carrot in US assisted drying (20 °C for 120 min) was higher (82–92%) (Frias et al. 2010). The bioavailability of vitamin B and vitamin C could be improved by ultrasonic assisted drying of cherry, tomato, cashew and apple pomace (Fernandes et al. 2016; Pingret et al. 2012). In addition, ultrasound treatment can increase the extraction rate of bioactive compounds by about 6% and 35% (Vilkhu et al. 2008), depending on the food

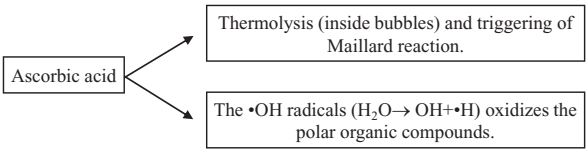

Fig. 2.2 Two pathways for sonication degradation of ascorbic acid

matrix and specific operating parameters (temperature, amplitude) (Rawson et al. 2011b).

2.2.3.5 Membrane Separation Processes

Membrane separation and filtration is an efficient fluid separation technology. Membrane filtration does not only remove turbidity components such as protein, pectin and tannin, but also reduce the microbial load of yeast and bacteria, and the scheme of a pressure-driven membrane process is shown in Fig. 2.3. Microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis have been widely used in food processing industry, especially in the clarification of natural products (such as natural extracts, juice, dairy products, beverages, etc.) (Castromunoz et al. 2017; Destani et al. 2013; Daufin et al. 2001; Girard and Fukumoto 2000) (Chap. 17). Compared with traditional separation methods, membrane technologies have many advantages, including mild temperature and pressure operation conditions, use of no chemical reagents or solvents, high selectivity, less treatment steps and low energy consumption. This also enables it to better maintain the functional characteristics of food, and reduce product pollution (Conidi et al. 2014). In recent years, these technologies have been more widely used in the treatment of agricultural food by-products (wastewater, leftovers, discarded food), as well as the separation and recovery of high value-added compounds (including bioactive extracts), so as to reduce the pollution in the production process, improve the utilization rate and prolong the sustainability (Castromunoz et al. 2018; Galanakis 2013).

MF is the most commonly used technology for clarification of industrial fruit juices including juices of lemon (Chornomaz et al. 2013), tomato (Razi et al. 2012), pomegranate (Conidi et al. 2017), cactus (Castromunoz et al. 2015), grape

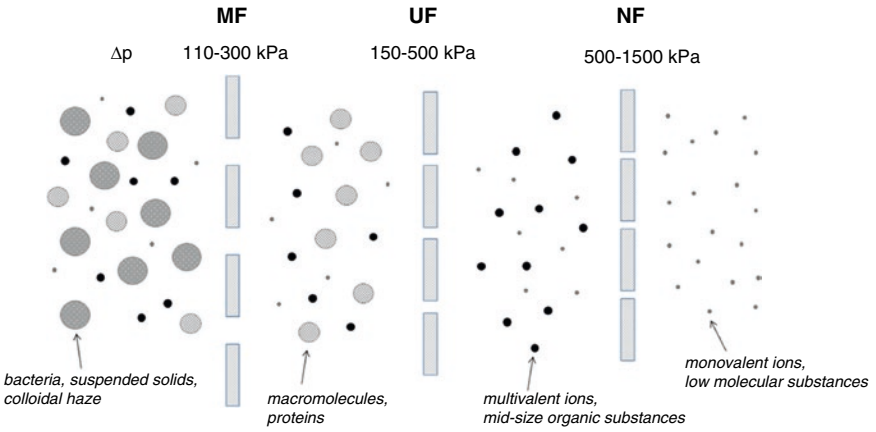

Fig. 2.3 Schematic of pressure-driven membrane processes. Adapted from Castro-Munoz et al. (2020)

(Cancinomadariaga et al. 2012) and pineapple (Laorko et al. 2010). Studies have shown that the juice clarified by MF and UF membrane is rich in antioxidant compounds (e.g. polyphenols, vitamin C), sugar, amino acids and minerals (Castromunoz et al. 2019). However, some studies showed that the pH value of clarified blueberry juice treated by MF didn't change significantly compared with the original juice. But the viscosity, content of soluble solids and proteins decreased by 49.01%, 17.27% and 0.34%, respectively, which may be related to the retention of some solids through MF membrane. Therefore, membrane filtration may still slightly reduce the nutrients in foods. The results showed that application of MF on pomegranate juice had no effect on turbidity, but reduced the contents of total anthocyanins and antioxidant activities of pomegranate juice. Compared with MF process, membrane fouling in UF process is more serious (Mirsaeedghazi et al. 2012). NF membrane is mainly used to concentrate juice, regulate sugar concentration and separate phenolic compounds from sugar (Wei et al. 2007; Garciamartin et al. 2010).

The application of membrane treatment on the recovery of high-value components in functional food and health product industries mainly focuses on carbohydrates, lipids, proteins and secondary components. In general, these processes can provide good recoveries for several bioactive compounds (Akin et al. 2012). For example, MF technology can recover from 47% to 100% of anthocyanins, glutamine, isopropanol, proline, betaine, isobutylene, sugar, galacturonic acid and some phenolic compounds (Conidi et al. 2017). UF and NF membranes were used to separate bioactive compounds from clarified pomegranate juice. The retention of osmotic fractions rich in phenolic compounds and sugars were obtained by concentration/infiltration. The yields of polyphenols and anthocyanins were 84.8% and 90.7%, respectively. Ultrafiltration and membrane electrodialysis can also be used to enrich active peptides within a specific molecular weight range (Korhonen and Pihlanto 2006; Shin et al. 2017).

2.2.3.6 Ozonation, and Plasma Processing

As a kind of preservation technology, ozone can inhibit microorganisms by penetrating into their cell wall, and is suitable for most food borne pathogens. Ozone has been used in the treatment of drinking water for a long time. Its inhibition effect on bacterial cell wall is stronger than that of chlorine gas. The first application of ozone in food processing includes washing foods with ozone water (kiwi fruit, apple, citrus, etc.), which can effectively inhibit bacteria and prolong the shelf life (Cullen et al. 2010) (Chap. 18). Gaseous ozone can prolong the shelf life of meat products and eggs. In 2001, food and drug administration (FDA) of Unites States approved the application of gaseous and liquid ozone as antimicrobial agents in food processing and storage. Generally, ozone concentrations of 0.15–5.0 ppm have been shown to inhibit the growth of spoilage bacteria and yeasts (Palumbo 2007).

Because of its strong oxidation activity, ozone has a certain impact on the quality of food, the content of bioactive substances, especially antioxidant substances. However, this effect varies depending on the chemical composition of the food,

dose of ozone, the type and duration of use (Cullen et al. 2009; Karaca and Velioglu 2007). Therefore, there are often inconsistent conclusions and even contradictory results. For example, some studies have compared the ozone storage and air storage of kiwifruit (the gas phase ozone concentration is 4 mg/h when the temperature is 0 °C and the humidity is 90–95%). The results showed that there was no significant change in ascorbic acid content of kiwifruit during the storage period of 7 months (Barboni et al. 2010). It was also reported that the content of ascorbic acid increased and the content of anthocyanins decreased after ozone treatment. However, the contents of ascorbic acid in pineapple, banana and guava were significantly decreased by ozone treatment, but the contents of total phenolics and flavonoids in pineapple and banana increased significantly after 20 min of ozone exposure. However, it is certain that overuse of ozone may lead to negative effects such as oxidation, discoloration, and undesirable odor on the food surface (Khadre et al. 2001).

Cold plasma (CP) processing is a new non-thermal processing technology which can inactivate the microorganisms on the surface of fresh agricultural products and can be used as a pretreatment of raw materials. It is based on the ionization of gas mixtures, resulting in excited molecules, ions, electrons and free radicals coexisting with electromagnetic radiation (ultraviolet and visible light) (Misra et al. 2016). CP can effectively inhibit bacteria, yeasts, molds and other harmful microorganisms, even spores that are very difficult to inactivate. CP has been used for biological purification and disinfection of medical devices, water, air, food and living tissues. It should be noted that CP has a higher degradation efficiency for pesticides, which may be due to the formation of a large amount of ozone after CP treatment. Ozone can react with water to produce peroxides and hydroxyl radicals to degrade pesticides.

In fact, the high oxidation capacity of CP itself has been proved to change some physicochemical properties of food ingredients, such as starch and protein, and also affect bioactive substances. Some studies have indicated that the contents of total phenolics and flavonoids in green leafy vegetables, apples and white grapes, as well as the antioxidant activities, are decreased after CP treatment compared with heat treatments (Lacombe et al. 2015; Sarangapani et al. 2017; Pankaj et al. 2017). On the other hand, some short-term CP treatments may have a promoting effect. The increase in the content of certain phenolic compounds in foods (apple, blueberry) is attributed to the physiological response promoted by stress in tissues, which increases the activity of phenylalanine aminotransferase (Tappi et al. 2018; Jiang et al. 2012; Alothman et al. 2010). However, CP treatment had little effect on vitamin C content of some foods (kiwifruit and radish sprouts) (Sarangapani et al. 2017; Ramazzina et al. 2015; Oh et al. 2017). However, CP had a significant effect on the anthocyanin content. The anthocyanin content decreased proportionally with the treatment time, and the anthocyanin content in fruits treated with 80 kV and 1 min decreased by 45.85% (Sarangapani et al. 2017; Lacombe et al. 2015). This is mainly due to the oxidative cleavage of chromophores caused by ozone and other free radicals produced by CP, resulting in significant loss of anthocyanins.

2.3 Effects of Food Processing Technologies on Phenolic Compounds

Phenolics and flavonoids widely exist in the plant kingdom. They are found in almost all edible plants expressing shikimic acid metabolic pathway in trace forms. Flavonoids are commonly found in cereals and herbs (lemon, olive, grape, etc.), and phenolic compounds are even more widespread. As shown in Table 2.4, different processing methods have an impact on the content and bioavailability of these antioxidants. Therefore, in this section the effects are discussed in detail according to different treatments/processes.

2.3.1 Pre-treatment

Before food processing, foods are usually washed or soaked, i.e. soaking beans overnight. It was reported that in this case, the loss of total phenolics was about 2%, the loss of isoflavones was as high as 12%, and p-coumaric acid, ferulic acid and erucic acid were detected in the soaking water (Luthria and Pastorcorrales 2006; Wang and Murphy 1996). In addition, food may be mechanically processed, such as chopping and crushing. This process usually leads to the loss of juice (including active substances), and it also affects the enzyme activity, leading to the conversion of some active substances. Chopping onion bulbs and soaking them for 60 min resulted in a 2.3% reduction in quercetin 3,4-diglucoside and 0.8% increase in quercetin 4-glucoside, possibly due to the activation of a glycosidase activity during chopping (Makris and Rossiter 2001). Chopped asparagus significantly reduced the content of rutin (18.5%), but no quercetin was detected.

2.3.2 Thermal Treatments

In the process of temperature rise, polyphenols, flavonoids and other antioxidants seem to go through a dual process. On one hand, the active substances in foods will be lost, especially by blanching, boiling and other heating methods. On the other hand, due to the decomposition of natural antioxidants, other new active products will appear (Buchner et al. 2006). For example, heat treatment can strongly degrade rutin and quercetin (one of the products of protocatechuic acid cracking reaction) under weak alkalinity and oxidation conditions, and they split through different decomposition modes, and rutin will undergo 3-glucoside. The increase of total phenolics and total flavonoids was observed in the process of cooking green tea, heating grape juice, orange juice, and beans, which may be due to the degradation of the protein complexes (Xie et al. 2013; He et al. 2016; Hithamani and Srinivasan 2014). A more specific example is that when the juice was heated at a relatively

Table 2.4 Effect of different processing methods on the content and bioaccessibility of phenolics

Parameter	Product	Process conditions	Effect	References
Total polyphenols and flavonoids content	Broccoli	Steaming (10 min) Boiling (5 min)	Steaming: Total polyphenols (160%), flavonoids (150%) and phenolic acids (130%) Boiling: Flavonoids 28% and phenolic acids 48%	Gliszczynskaswiglo et al. (2006)
Total phenolics and anthocyanin content	Strawberry Blackberry	HPP (400, 500, 600 MPa/15 min/10–30 °C)	Phenols: Strawberry: (600 MPa increased 9.8%) Blackberry (600 MPa increased 5.0%) Anthocyanin: No significant difference	Patras et al. (2009b)
The extraction rates of total polyphenols	Orange peel	PEF: (Specific energy: 0.06–3.77 kJ/kg, pulses width: 3 μs; frequency: 1 Hz; E = 1, 3, 5 and 7 kV/cm	Increased by 20%, 129%, 153% and 159% with 1, 3, 5 and 7 kV/cm application	Luengo et al. (2013)
Total phenolic content	Grape juice	Ultrasonic treatment (20 kHz)	Increased by 114.3%	Ashokkumar et al. (2008)
Bioaccessibility of total phenolics and flavonoids	Red tomatoes	Cooking (70–80 °C/5–70 min)	No significant difference	Kamiloglu et al. (2014)
Bioaccessibility of total polyphenols and flavonoids	Cereal: Finger millet and pearl millet	Roasting (150 °C, 5 min), pressure cooking, boiling (5, 10, 15 min) and microwave heating	Finger millet: Pressure cooking improved Pearl millet: In addition to roasting, other processing methods improved	Hithamani and Srinivasan (2014)

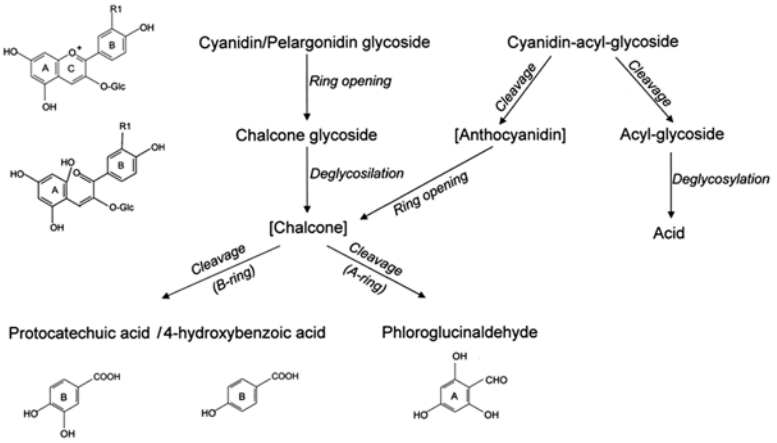

Fig. 2.4 Proposed mechanism for thermal degradation of anthocyanins (Adapted from Rawson et al. 2011a)

lower temperature (44 °C), the total polyphenols, antioxidant activity and anthocyanins increased. However, with the increase of temperature (55 °C), all bioactive components including total flavonoids were decreased. Figure 2.4 shows the possible degradation patterns of anthocyanins in foods and the formation of various intermediates. Researchers reported similar results (Rawson et al. 2011b); with the increase of processing temperature (25–45 °C), the total phenolic content of watermelon juice was decreased. Bioactive compounds are directly affected by processing temperature and processing time (Abid et al. 2014).

2.3.3 Pressure Related Treatment

Pressure related technology has a positive significance for the retention of antioxidant components such as phenolics and flavonoids. It may increase the catalytic activity of the enzymes, increase the reaction rate, and enhance the secretion of enzymes in cells. Many oxidases (such as polyphenol oxidase) use phenols as substrates, so these enzymes can reduce the antioxidant activity of phenolic compounds (Rossle et al. 2010). The results showed that phenolic compounds were not destroyed at low pressure (<400 MPa), and higher pressure (600 MPa) retained higher total phenolic content than 400 MPa ($P < 0.05$). Compared with the conventional heat treatment, the HHP treated samples maintained significantly higher contents of total phenolics, anthocyanins and ascorbic acid at 400–600 MPa (Patras et al. 2009b). It seems that phenolic compounds are relatively resistant to pressure. HPP assisted extraction can obtain higher yield of phenolic compounds, higher antioxidant and anticancer activities in a short time. The reason for this increase is the deprotonization of charged groups, the destruction of salt bridges, and the destruction of water bonds on the cell

membrane, resulting in high permeability. Another reason is that the dielectric constant of water decreases, which leads to the decrease of the polarity of the medium and may increase the content of phenolics and antioxidants (Hurtadofernandez et al. 2010).

2.3.4 Electric Fields Related Treatment

The electric field treatment can promote the permeability of plant cells, and the space formed on the cell membrane will promote the extracellular mass transfer and increase the release of polyphenols. Compared with heat treatment, higher concentrations of phenolic acids (chlorogenic acid) and flavonols (quercetin) were observed in fruits and vegetables treated by PEF (Odriozolaserrano et al. 2008). In addition, the effects of different electric field energy treatments on polyphenols and flavonoids were significantly different. The total phenolic content of orange juice samples treated with high energy was higher than that of orange juice samples with low energy treatment. The extraction rates of total polyphenols were increased by 20%, 129%, 153% and 159% by PEF at 1, 3, 5 and 7 kV/cm, respectively (Luengo et al. 2013). However, with the increase of energy, the content of polyphenols also decreased. Under the conditions of 25 kV/cm, 30 kV/cm and 0.2 μs (pulse rise time), total phenolic content in apple juice decreased significantly compared with the control sample, and the similar phenomenon was found in strawberry treatment (Aguilarrosas et al. 2007; Odriozolaserrano et al. 2008). This is due to the high residual activities of peroxidase and polyphenol oxidase in fruits treated with high energy PEF (e.g., strawberry, apple, peach) and oxygen exposure in the pipeline, which can affect the degradation of polyphenols in PEF processed foods (Giner et al. 2002).

2.3.5 Other Treatments (Ultrasound, Microwave)

The content of total phenolics and flavonoids in the samples treated by ultrasound were observed to generally increase. There is evidence that grapefruit juice treated by ultrasonic wave has higher DPPH radical scavenging activity and antioxidant capacity (Alighourchi et al. 2013). Due to the formation of free OH* radicals and the increase of hydroxylation, the concentration of single phenolic compounds (such as flavonoids) may increase. Ultrasonic treatment of grape juice can increase the total phenolic content by 114.3% (Ashokkumar et al. 2008). There is also evidence that phenolic compounds can bind to other compounds (polysaccharides, proteins) on the cell wall (Lieu and Le 2010). The basic principle of increasing phenolic content involves the cavitation process in food ingredients and the pressure exerted in the process, which leads to the destruction of the outer layer of cells and the release of bound polyphenols from food. Another reason is that the hydroxyl functional groups are added to the aromatic compounds of polyphenols due to ultrasonic treatment.

The energy of microwave is absorbed by food. Under the action of alternating electromagnetic field, ions and polar molecules in food system rotate and collide to produce heat needed for cooking. This energy can heat the solvent more quickly and effectively, so it is also used for assisted extraction. The study on green tea showed that microwave treatment increased the contents of total phenolics and catechins, and prevented the combination of catechins and polyphenols with leaf matrix, thus increasing the content of catechins in green tea. The extraction efficiency of caffeine and polyphenols can be improved by using this method (Pan et al. 2003). The phenolic contents in the product was 297, 329 and 342 μg/g dry weight (dw), respectively, when the products were treated with microwave of different power (125, 250 and 250 W) for 5 min. These results indicate that higher content of phenolic compounds can be obtained by increasing microwave power at a certain time and power (Hayat et al. 2010). However, in plant tissues, microwave treatment causes local temperature rise, leading to tissue rupture, which makes phenolic compounds migrate to the surrounding solvent. Long time microwave treatment can release bound phenols during the processing of citrus peel (Jeong et al. 2004). With the increase of microwave power and irradiation time, the bound phenolic acids and glycoside bound phenolic acids are decomposed during microwave heat treatment. On the whole, the content of free and bound phenolic acids decreased with the increase of microwave power and treatment time.

2.4 Effects of Food Processing Technologies on Bioactive Polysaccharides

Bioactive polysaccharides are high molecular polymers with various physiological activities. They are widely distributed in animals, plants, microorganisms and fungi, as important bioinformatics macromolecules in living organisms. Bioactive polysaccharides have immunomodulatory, antitumor, antiviral, antioxidant, hypoglycemic and other functions, and have been applied in many industrial fields such as functional foods, cosmetics, and pharmaceutical products (Huang et al. 2012; Liu et al. 2015). In this section, the effects of different processing parameters (designing different processing technologies) on the structure and functional properties of active polysaccharides are introduced.

2.4.1 Pre-treatment

In the extraction of animal and plant polysaccharides, the raw materials are usually pretreated (crushed and extruded) to improve the yield. Among them, extrusion treatment can improve the yield of active polysaccharides more than the physical crushing. This is because extrusion can destroy the tissue and cell structure of the

raw material, while the physical crushing only divides the fruiting body into small particles, and the tissue morphology does not change. Under the condition of 95 °C (1–6 h), the extraction rate of polysaccharide from *Tremella fuciformis* treated by extrusion was 40–63% higher than that of crushing *Tremella fuciformis* (Gu et al. 2011).

2.4.2 Thermal Treatments

Heat treatment first affects pectin, which is due to their elimination of beta and pectin methyl esterase being almost ubiquitous in plants. Studies showed that the impurities of pectin were reduced after the blanching treatment. The chemical composition, main chain structure and side chain structure of pectin were changed, and the effect of high-temperature short-time treatment on the structure was less than that of low-temperature long-time treatment (Luo et al. 2008; Lo et al. 2002; Bao and Chang 1994). Taking pear as an example, pectin was partially degraded during cooking, while xylose and cellulose were not affected. Therefore, it has been reported that the pectin content in cooking water can be increased by 400% after boiling for 1 h compared with the level after boiling for 20 min (Renard 2005). Treatment with heat at different temperatures (40, 90 °C) and/or calcium immersion ($CaCl_2$) affected the texture of lotus roots with a decrease in the content of galactose, rhamnose and arabinose. The $CaCl_2$ immersion with blanching at 40 °C resulted in a higher hardness compared to the control (blanching alone or calcium immersion), mainly due to the formation of a calcium pectin network, which safeguarded the integrity of the cell wall. The cell wall of the lotus root was correlated with the degree of esterification of pectin (Zhao et al. 2016). In addition, blanching treatment affects the molecular weight distribution and viscosity of polysaccharides (Nyman et al. 1994). Svanberg et al. (1995) studied the changes of molecular weight distribution and viscosity of water-soluble dietary fiber of carrot after several pretreatment methods, such as freezing, blanching, soaking, microwave and boiling water cooking. The results showed that the polymerization degree and viscosity of carrot water-soluble dietary fiber were closely related to the treatment methods, and boiling water cooking and blanching treatment reduced the polymerization degree and viscosity of water-soluble dietary fiber. Other studies have shown the antioxidant activity of polysaccharides *in vitro* and indicated that the ability to inhibit the growth of breast cancer are reduced after heat treatment (Radzki et al. 2016).

Different pasteurization conditions (temperature: 65, 75, 85 °C, time: 15, 25 min) have effects on aloe acetyl mannan and cell wall polysaccharide (Rodriguezgonzalez et al. 2011). The deacetylation of acetyl mannan occurs in the sterilization process, and the galactose on the side chain is removed, resulting in the formation of a new hydrogen bond between the mannan oligosaccharide chain and the long chain of mannan. On one hand, slight degradation of pectin was observed at 65 °C, whereas the significant reduction of pectin (mainly high galacturonic acid) observed for samples treated at 85 °C may be due to their thermal degradation. After different

pasteurization conditions, structural changes of aloe polysaccharide lead to different physical and chemical properties. The swelling capacity, water holding capacity and liposuction capacity of aloe polysaccharide treated at 65 °C and 75 °C were enhanced, while those of aloe polysaccharide treated at 85 °C were significantly decreased (Rodriguezgonzalez et al. 2011; Rodriguezgonzalez et al. 2012). Polygalacturonic acid in green bean pods was partially degraded during sterilization, and the degree of acetyl substitution did not change significantly (Stollesmits et al. 1995). It is speculated that this may be due to the β-elimination reaction between the highly methylated and polygalacturonate fractions of pectin. In the process of thermal sterilization, the main change in polysaccharides is that the branch chain or substituent may be removed, and the main chain may be degraded, so the molecular weight and viscosity of the polysaccharide will be reduced (Kok et al. 1999). In the process of baking, frying and other high-temperature processing, Maillard reaction will occur, the amino group of protein and the carbonyl group of polysaccharides will be covalently linked, and the polysaccharide will be changed greatly during this reaction. This is a wide topic with too many details, and will not be discussed in this chapter.

2.4.3 Pressure Related Treatment

High hydrostatic pressure (50–1000 MPa) is a common method for processing foods rich in polysaccharides (Mcinerney et al. 2007). The high hydrostatic pressure treatment of plant gel showed that, with the increase of pressure, the content of polysaccharides in the gel increased and the antioxidant activity was decreased (Vegagalvez et al. 2014). The results showed that the average molecular weight of fucoidan decreased by 8–31%. With the increase of enzyme concentration, the contents of sulfate, fucose and galactose decreased, while the content of glucose increased (Park et al. 2012). After 200 MPa high pressure treatment, the glass transition temperature of glycan was decreased from 75 to 65, the gel strength decreased, and the breaking force and fracture constant decreased. However, the crosslinking degree of glycans increased with the increase in pressure to 600 MPa, and the crystallinity of the gel network structure increased, but the glass transition temperature did not change.

Dynamic high-pressure micro jet is a new food processing technology. It combines high-pressure technology with homogenization technology, through high-speed collision of materials in the oscillating reaction chamber, high-frequency oscillation, instantaneous pressure drops and cavitation. The effects of high-pressure microfluidization on the structure and functional activity of polysaccharides are currently a research hotspot, including xanthan gum (Laneuville et al. 2013), pectin (Chen et al. 2012) and plantago seed polysaccharide (Hu et al. 2013). Dynamic high pressure micro fluidization technology can break the molecular chain of polysaccharides and degrade them under high pressure. It will also change the

monosaccharide composition, particle size and molecular aggregation state of polysaccharides, and improve the antioxidant activity (Tsai et al. 2009).

2.4.4 Drying Related Treatments

Polysaccharide rich raw materials usually contain a lot of moisture. Drying technology (natural drying, hot air drying, vacuum drying, freeze-drying, spray drying) is conducive to its packaging, storage and transportation. Within different drying methods, vacuum drying had better effect on physicochemical properties and antioxidant activity of polysaccharides. The results showed that all kinds of drying methods had little effect on the primary structure of polysaccharides, but had certain effects on the chemical composition (contents of sugar, protein and uronic acid) of polysaccharides, and had great influence on antioxidant activity. Different drying methods had no significant effect on the primary structure of bergamot polysaccharide, because the UV and Fourier transform infrared (FTIR) spectroscopy of the samples were similar (Wu 2015). But different drying methods will affect the yield of crude polysaccharide, as well as the protein and ash contents. Among them, the molecular weight distribution of vacuum freeze-drying samples is narrower, and the reduction ability and free radical scavenging ability are stronger. On the contrary, drying method affected the monosaccharide composition and uronic acid content of epimedium polysaccharide (Nep and Conway 2011). The polysaccharide sample of vacuum freeze-drying had the highest content of uronic acid and the strongest antioxidant capacity. In addition to antioxidant activity, drying technology also affects other activities of polysaccharides. Although drying did not change the carbohydrate content in yeast cell wall and the chemical structure of β-D-glucan, it increased the TNF-α induced activity of β-D-glucan in mouse macrophage model. It is suggested that the increase of purity of β-D-glucan after drying may enhance the immune activity of β-D-glucan of *Saccharomyces cerevisiae* (Liepins et al. 2015).

2.4.5 Other Treatments (Ultrasound, Microwave)

Ultrasonic wave can break the polysaccharide chain, destroy the polymer network structure, weaken the intermolecular interaction, reduce the viscosity resistance of polysaccharide, and enhance the swelling capacity and solubility. After ultrasonic treatment, the viscosity of the adhesive can be reduced to one thousandth compared to that of before. Ultrasonic degradation of polysaccharides makes their structure loose, and cavitation effect can reduce the activation energy of chemical reaction, thus promoting the chemical reaction (Chen et al. 2014). Ultrasonic assisted extraction can accelerate the dissolution rate of polysaccharides to a certain extent, and affect the galacturonic acid content and esterification degree of pectin (Zhang et al. 2015). However, the main characteristics of the primary structure (main chain

connection, main monosaccharide composition, etc.) of polysaccharides will not be changed (Guo et al. 2014; Yan et al. 2015).

Microwave technology is most commonly used in the extraction of polysaccharides. Due to the high-energy effect of microwave, the temperature of the extracted material rises rapidly, and the local high temperature and pressure will be generated in the instant of water vapor to dissolve the polysaccharide molecules. Increasing the extraction temperature through external heating is more conducive to the dissolution of polysaccharide molecules. In addition, the polysaccharides obtained by microwave treatment had good antioxidant and antibacterial activities (Tsubaki et al. 2016). The results showed that the contents of protein, carbohydrate and molecular weight of the polysaccharides extracted by microwave were higher than those extracted by traditional methods.

2.5 Effects of Food Processing Technologies on Essential Fatty Acids and Sterols

Omega-6 (ω-6) and omega-3 (ω-3) polyunsaturated fatty acids (PUFAs) are essential lipid bioactive substances for the human body. For example, α-linolenic acid (ALA), eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) are essential PUFAs for human beings (Whelan and Rust 2006). Among them, the main dietary sources of ω-6 fatty acids include cereals, vegetable oils and baked foods, and the main sources of ω-3 fatty acids are fish oil and rapeseed oil. ALA is widely found in many plant products, and the best source of EPA and DHA is cold water oily fish. Phytosterols are also natural active substances derived from lipids, which are distributed in most plants and widely exist in flowers, fruits, leaves, seeds and rhizomes of plants (Johansson and Hoffmann 1979). Known as the "key to life", it has very important physiological functions, such as lowering cholesterol, anti-cancer, maintaining the stability of biological internal environment, controlling the metabolism of glycogen and minerals, regulating stress response, etc. (Moreau et al. 2002). Cereals and oils are abundant natural sources of phytosterols. They exist in four different forms: free or esterified to fatty acids, sugar groups and phenolic acids (Normen et al. 2002).

2.5.1 Pre-treatment

Lipids containing PUFAs and their esters are easily oxidized, a free radical chain mechanism called autoxidation. In general, storage of fat products results in a decrease in the content of PUFA components, depending on storage conditions. Refrigeration and freezing are common storage procedures and most fatty acids (especially PUFAs) in fish meat gradually decrease at −10 °C (Eun et al. 1994). In

addition, the food containing sterol will have oxidation reaction during processing and storage, and enzymes may also induce the production of sterol oxidation products. After 18 weeks of storage at 4 and 20 °C, the content of oxidized sterols increased by 35% and 100%, respectively. Lipoxygenase catalyzes the oxidation of polyunsaturated fatty acids, which are the key enzymes in this pathway. Their existence is considered to be the main reason for blanching before refrigeration. Some authors pointed out that lipoxygenase and peroxidase extracted from sweet corn were completely inactivated within 9 min and 15 min after steam blanching, thus protecting PUFAs (Dornenburg and Davies 1999).

2.5.2 Thermal Treatments

Thermal treatment of lipids usually involves oxidation process, and sometimes produces toxic volatile and nonvolatile compounds. PUFAs are more easily oxidized than monounsaturated fatty acids during heating. Blanching of meat products results in a decrease in PUFA content, an increase in free fatty acid levels, and a significant increase in peroxide value (Fox et al. 1994). Damage to PUFAs can lead to the production of primary and secondary lipid oxidation products, leading to browning, formation of fluorescent compounds, flavor changes, and loss of essential nutrients (Lubis and Buckle 2007). The volatile compounds (*n*-alkanes, 2-alkenaldehyde, 1-alkanol and alkyl furanane) in the flavor extract of cooked steak were increased by 4 times. The effects of different heating methods on fatty acids were also different. When processing eggs rich in ω-3, lipid oxidation increased by 2–9 times. Moreover, the oxidation level of PUFAs in cooked eggs (30.4%) was higher than that in scrambled eggs (Cortinas et al. 2003). The results of experiments on many kinds of fish (trout, silver carp and cod) showed that the total amount of PUFA has no obvious change, and EPA and DHA are also stable in the cooking process. However, frying can lead to 30–40% loss of fish oil (Mendez 1992).

2.5.3 Pressure Related Treatment

High pressure is increasingly used to improve the quality and functional properties of lipids and meat products and extend shelf life. However, it has been found that meat is more susceptible to lipid oxidation under high pressure, which leads to deterioration and change of fatty acid composition (Orlien et al. 2000; He et al. 2012). Under 300 MPa, the lipid oxidation of pork treated with pressure was slightly affected, but it was increased at higher pressure (Cheah and Ledward 1996). When the ambient pressures was 400 MPa or higher, the oxidation rate of cod muscle increased (Angsupanich and Ledward 1998). The results showed that the oxidation rate of chicken breast muscle treated with 500 MPa pressure had no effect on the oxidation rate (Beltran et al. 2003). Lipid oxidation was induced by high pressure

treatment, and fatty acid composition was changed by PUFA oxidation. High pressure (> 200 MPa) could decrease the ratio of PUFAs/saturated fatty acids, the value of ω-6/ω-3 PUFAs and the content of DHA in yak meat. High pressure (>400 MPa) and temperature lead to the increase of thiobarbituric acid reactive substances of yak body fat, and finally lead to the change of fatty acid composition (Wang et al. 2013).

2.5.4 Frying Related Treatment

Frying temperature and frying method can affect the oil or fat used as cooking medium and the oil composition of fried food because of the important lipid exchange between the two components. In general, oxidation of one component induces oxidation of another. The thermal stress of edible oil rich in PUFAs (at 180 °C for 30–90 min) decomposes its conjugated diene hydroperoxide precursor to produce high content of *n*-alkanes, *trans*-2-alkenals and alkanes, 4-dienes and 4-hydroxy-*trans*-2-enals, while only low concentrations of aldehydes are produced in oils with low PUFAs content, indicating that the higher the degree of unsaturation, the lower the stability during frying (Claxson et al. 1994). Increases in saturated and unsaturated aldehydes were observed as lipid oxidation products in cooking oils. Frying creates an exchange between fat and frying medium, resulting in significant changes in fatty acid composition and the ratio of ω-6/ω-3 in oily fish (Sanchezmuniz et al. 1992).

Almonds are rich in fatty acids, of which oleic acid (62.9%) and linoleic acid (20.0%) are the most important. At certain temperature (150 or 180 °C) and time (5, 10 or 20 min), not only the unsaturated fatty acids (linoleic acid, oleic acid and oleic acid), but also the saturated fatty acids (palmitic acid and stearic acid) were increased. Baking at higher temperature (200 °C) for a long time (10 or 20 min) may lead to the degradation of fatty acids, especially unsaturated fatty acids. The highest oil yield (56.3% of roasted almonds), unsaturated fatty acids (89.4% of crude oil) and saturated fatty acids (7% of crude oil) were obtained when roasted at 180 °C for 20 min. Frying in a rapeseed oil/palm oil mixture produced the highest amount of sterol oxidation products (Dutta 1997).

2.5.5 Other Physical Treatments

It has been suggested that the high energy level involved in the microwave process will promote the oxidation of PUFAs and promote the formation of conjugates and polymers in a few minutes, because the microwave forces the molecules to vibrate strongly, especially the hydrogen atoms near the unsaturated center in the active methylene group (Farag et al. 1992). According to some studies, microwave heating did not lead to significant loss of polyunsaturated fatty acids. Only some studies showed that

the percentage of PUFAs in soybean and egg yolk samples decreased after microwave treatment (Murcia et al. 1992). In addition, when corn oil and palm oil were heated for 5, 10 and 15 min, some triglycerides were hydrolyzed to free fatty acids. It was observed that the increase of lipid oxidation resulted in a decrease in the percentage of PUFAs and an increase in saturated fatty acids (Matalgyto and Alkhalifa 1998). In addition, heating of sunflower oil, soybean oil, and peanut oil resulted in a general decrease in PUFAs with prolonged heating time (Hassanein et al. 2003). There is also evidence that microwave treatment (120 min, 170 °C and specific interval) has no significant effect on sterol content of the five commonly used oils (Albi et al. 1997).

2.6 Effects of Food Processing Technologies on Carotenoids and Retinol

Carotenoids are yellow, orange or red pigments with symmetrical tetraterpene skeleton as the basic structure. They are hydrophobic compounds and widely exist in vegetables (lettuce, pepper, spinach, etc.) and fruits (carrots, tomatoes, oranges, etc.). Astaxanthin is also present in animal tissues (fish, eggs, milk), but is usually converted to retinol or vitamin A (β-carotene, α-carotene and β-cryptoxanthin). During storage and processing, other carotenoid derivatives will also be produced due to hydrogenation and oxidation (Ruizrodriguez et al. 2008).

2.6.1 Pre-treatment

Pretreatment, such as washing, peeling, cutting, drying, and storage in the refrigerator or at room temperature may have a negative impact on the final carotene content of the prepared dishes. Chopping vegetables can promote the loss of vitamin A and carotene. Boiling the whole chicken liver caused 5% loss in retinol content. If the liver is chopped, the rate of loss will increase by 8% (Sungpuag et al. 1999). In industrial production, lycopene content increased temporarily due to temperature rise during extrusion (160 °C), but decreased due to decomposition after processing (Tonyali et al. 2016). Generally, mechanical destruction or homogenization before processing can enlarge the area of digestive enzymes, so it is a favorable way to release carotenoids from food matrix.

2.6.2 Thermal Treatments

During processing, the content of carotenoids and vitamin A depends on the chemical properties of raw materials. Even under similar processing conditions, the contents of carotenoids and vitamin A may vary with the substrate involved. Some

studies suggest that heating reduces the total amount of carotenoids, while others do not observe significant changes, or believe that heat treatment increases the concentration of carotenoids. As early as 1977, some scientists thought that the extraction of soluble solids was the possible reason for the obvious increase of carotenoid content during processing of vegetables. The effect of heating may come from the presence of dietary fat (oil), which promotes the release of carotenoids in a hydrophobic environment. In addition, the increase of enzyme activity is also one of the reasons, because they may catalyze the degradation of specific protein carotenoid aggregates (Johnson 2000). Some experiments have shown that the carotene in cooked broccoli increased by 31%. In cooked cauliflower, the increase of β-carotene (38%) was higher than that of lutein (24%) (Weesie et al. 1995). However, the contents of *cis*-β-carotene and lutein increased by 28% and 2% respectively, while the content of β-carotene decreased by 7%. The contents of carotene and lutein increased in broccoli and spinach cooked and boiled (Gliszczynskaswiglo et al. 2006). Cooking increased the total content of carotenoids, especially trans-β-carotene, but had little effect on lutein. Compared with different heating methods, steam blanching seems to have better retention of carotene content and better sensory quality than water blanching (Sungpuag et al. 1999), and blanching (100 °C for 5 min) had better retention than frying (180 °C for 2 min).

Thermal processing can destroy the structure of food, resulting in structural changes of carotenoids, mainly isomerization (*cis*/*trans*), change its solubility, resulting in micellization and formation of different carotenoid by-products (Victoriacampos et al. 2013). During normal blanching and cooking, β-carotene is more vulnerable to heat damage than α-carotene and lutein is more susceptible than carotene. In freshly harvested cauliflower, cabbage, spinach, carrot, pumpkin, sweet potato, and red pepper, all trans carotenes were converted into plastids with low vitamin A activity. The decrease of carotene bioavailability was found in different kinds of food, such as cucurbit and tomato (Ryan et al. 2008), cauliflower (Osullivan et al. 2010), drinks (Rodriguezroque et al. 2015), orange juice (Victoriacampos et al. 2013), etc.

2.6.3 Pressure Related Treatment

In general, pressure treatment may reduce the loss of natural pigments compared to steam cooking (La Cruzgarcia et al. 1997). The retention of β-carotene in green leafy vegetables is about 73–84% under pressure cooking, and adding some antioxidant spices such as turmeric can improve this retention (Gayathri et al. 2004). The bioavailability of carotenoids was increased in carrot, amaranth and mung bean (Mcinerney et al. 2007; Knockaert et al. 2011). In contrast, a decrease in the bioavailability of carotenoids was found in some vegetables (cauliflower, tomato) with stronger cell structure (Mcinerney et al. 2007; Knockaert et al. 2011). This is mainly due to changes in the microstructure of the pulp, resulting in the formation of a fiber network that makes lycopene difficult to be absorbed by digestive enzymes and bile

salts (Colle et al. 2010). In the presence of structural barriers, HPP affects carotenoids by increasing the release of carotenoids and improving bioavailability. In the absence of these barriers, the soluble and insoluble phases determine the carotenoid bioavailability (Palmero et al. 2014).

2.6.4 Thermal Treatments

Baking and frying can reduce the content of vitamin A, and since the high temperature process is more severe than boiling, so the proportion of reducing carotenoids is higher than boiling. For example, in an electric oven at 200 °C (20–45 min), the degradation rate of carotenoids in sweet potato was higher than that of boiling in water (10–30 min). Fried eggs (200 °C, 2 min) lost 43% of the original retinol content than raw eggs, which was 32% higher than that of boiled eggs. The retention rate of lycopene was only 25–37.3% when the tomato was roasted at 177–218 °C. The longer the baking time, the greater the loss was, which was consistent with the performance of frying (Mayeaux et al. 2006). According to an *in vitro* digestion experiment, stir frying can improve the bioavailability of pumpkin, carrot and amaranth leaves (53, 63, 192%), thus promoting the intake of carotenoids (Goni et al. 2006).

2.6.5 Other Physical Treatments (Microwave, Ultrasound, Pulsed Electric Field)

Microwave heating is less destructive than cooking. The loss of total carotenoids in cauliflower was about 23%. The contents of β-carotene and violaxanthin decreased during conventional cooking and microwave cooking, while lutein level increased in both cooking methods. This may be due to the conversion of cis isomer of lutein to trans form, which has been noticed during microwave cooking of broccoli (Updike and Schwartz 2003). Lycopene was seriously affected by high power microwave, and only 64.4% of lycopene remained after heating for 1 min (Mayeaux et al. 2006).

The bioavailability of carotenoids can be reduced by high intensity PEF treatment. The bioavailability of the processed drinks (milk, soybean milk juice) were improved compared with the untreated drinks. As for the effect of antioxidant activity, PEF increased the antioxidant activity compared with untreated samples (Rodriguezroque et al. 2015). In addition, ultrasonic treatment did not change the total lycopene content in tomato pulp (Anese et al. 2013), but led to a significant decrease in bioavailability, which may be due to the formation of a stronger network to embed lycopene in the substrate (Anese et al. 2013). When adding milk and lipase before digestion *in vitro*, lycopene did not show any difference in bioavailability compared with the control group (Anese et al. 2015).

2.7 Effects of Food Processing Technologies on Vitamins

2.7.1 Tocopherols

Tocopherol belongs to vitamin E which is an important antioxidant and its main source is fats, oils and cereals. It can react with free radicals in cell membrane and other lipid environments and inhibit free radicals, thus preventing PUFAs from being damaged by lipid oxidation (Bramley et al. 2000). In the process of storage, taking wheat germ rich in tocopherol as an example, the oxidation of oil was increased, and tocopherol content was decreased with the storage time. The first-order kinetic degradation of tocopherols in wheat germ during different storage periods is shown in Fig. 2.5, and the rate constant K of β-tocopherol was increased with the increase of temperature (Capitani et al. 2011).

The loss of tocopherol during cooking in water ranges from 10% to 20%, depending on the type of food and processing time. In the cooking of some cereals and legumes, grain losses ranged from 22% to 55% for cereals and from 9% to 59% for legumes (Leskova et al. 2006; Atienza et al. 1998). However, the decrease of α-tocopherol and γ-tocopherol was not significant. In rabbit meat, boiling reduced tocopherol content by 39%, which was higher than frying (12%) or baking (14%) (Dal Bosco et al. 2001).

Tocopherol is a fat-soluble vitamin; the use of other fats in stews and frying may promote its degradation, similar to unsaturated fatty acids as described earlier. If the

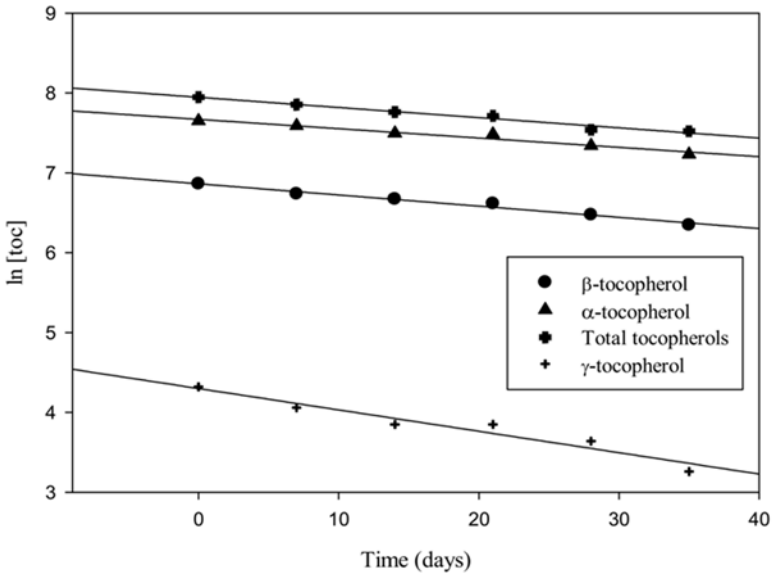

Fig. 2.5 First-order kinetic degradation of tocopherols in wheat germ during different storage periods (Adapted from Capitani et al. 2011)

frying process is short, for example when frying salmon, the use of corn oil and partially hydrogenated vegetable oil did not have a negative impact on tocopherol content (Alsaghir et al. 2004). During continuous frying process, the reuse of oil reduces the tocopherol level. α-tocopherol was oxidized more easily than other isomers when potato was fried with rapeseed. After 4 or 5 cycles, α-tocopherol was reduced by 50%. β- and γ-tocopherol decreased similarly after 7 or 8 cycles (Gordon and Kourimska 1995). High γ-tocopherol content was found in fried beef slices with corn oil (Saghir et al. 2005). Compared with corn oil or partially hydrogenated vegetable oil, olive oil had higher final α - tocopherol content.

High temperature had significant effect on the antioxidant activity of tocopherol. The antioxidant activity of α-tocopherol in lard was basically unchanged in the range of 80–110 °C, and decreased with the increase of temperature when it was higher than 110 °C. The effects of temperature on the antioxidant activities of α-tocopherol and δ-tocopherol were different. The activity of δ-tocopherol was about twice as much as that of α-tocopherol at 80 °C, but it was almost the same at 130 °C ($P < 0.05$) (Reblova 2006). In addition, the researchers confirmed that tert butyl hydroquinone and butylated caffeic acid could effectively protect the tocopherols, especially δ-tocopherol, in oils and fats (Liu et al. 2019a). When microwave heating egg cake, the tocopherol in egg yolk was significantly reduced (up to 50%). Yoshida and Kajimoto (2010) reported that microwave treatment of soybean seeds resulted in about 40% loss of tocopherol. The order of stability during microwave heating was $\beta > \gamma > \alpha$ tocopherol (Yoshida and Kajimoto 2010).

2.7.2 Folates

Folic acid, a derivative of pteridine, is a group vitamin B found in green leafy vegetables, animal liver and yeast. Folic acid deficiency is directly related to neural tube malformation, megaloblastic anemia, cleft lip and palate, depression, tumor and other diseases (Braun 1996). It was reported that the average loss rate of folic acid in industrial processing (industrial cooking) was 32.2%.

Folic acid is a water-soluble compound. When food is immersed in water at room temperature or boiling temperature, folic acid will be lost and a large amount of folic acid can be found in water (Arcot et al. 2002). The retention rate of folic acid in cooked broccoli was only 44%, and that in chickpea was 51%. The effects of different processing methods on folic acid in spinach showed that cooking and blanching caused the most significant loss of folic acid in spinach, which were 37% and 56% respectively. Steaming and baking had the least effect on the loss of folic acidin spinach, which was about 9%. The folate in spinach can be effectively preserved by freezing and cold storage, and the rate of loss was less than 25% (Na et al. 2018). No significant loss of 5-methyltetrahydrofolate was found in juice stored at temperatures between 20 and 22 °C for less than 3 months. However, the loss of this vitamin has been reported in juice stored for 9 months (Kapoor et al. 2018). The retention rate of total folic acid in meat and poultry was 55–70% when boiling and

stewing, and 60–90% when baking and baking (Leskova et al. 2006). Soaking time was positively correlated with the loss of folic acid, and the degree of loss was affected by pH value, oxygen content, metal ions and antioxidant levels (Ruizrodriguez et al. 2008). The addition of reducing agent in food increased the retention of folic acid during hot processing, while the presence of metal (Fe^{2+}) increased the loss of folic acid (Leskova et al. 2006).

The loss of folic acid during processing is mainly due to their solubility in water, while the decrease in folic acid content during frying and baking is attributed to the thermal instability of folic acid. Depending on the differences in matrices, the loss during processing is different. The loss of folic acid during bread baking reached about 12–25% (Kariluoto et al. 2004). The content of folic acid in vegetables was almost unchanged when it was fried (Gujska and Majewska 2005). The loss of folic acid caused by frying was about 41%.

The stability of folic acid to high temperature (>65 °C) combined with high pressure (800 MPa)/heat treatment (20–65 °C) was much higher than that of 5-methyltetrahydrofolate. High temperature (60 °C) and HPP had little effect on folic acid, but the degradation rate constant of 5-methyltetrahydrofolate increased with the increase of pressure (Nguyen et al. 2003). The content of folic acid in peas heated by microwave was reduced by 75%, which was close to the loss rate of pressure evaporation (73%).

2.8 Conclusions

According to the current understanding, high temperature treatment usually negatively affects the content of bioactive compounds, and the degradation mechanism of bioactives is numerous, complex and even unknown. In the traditional processing methods, the decomposition rate of fat-soluble compounds such as carotenoids, tocopherols, polyunsaturated fatty acids and phytosterols is lower in the traditional processing methods involving water than the methods using oil as the cooking medium. In contrast to non-polar compounds, the contents of phenols, folates and other polar compounds are reduced during the process of boiling, which is first due to leaching, and then due to their thermal instability. New non-thermal technologies contribute to the sustainability of food production and show higher efficiency than traditional processing methods. For example, ultrasonic treatment may improve the bioavailability of polyphenols, and PEF can improve the bioavailability and antioxidant activity of carotenoids, vitamin C and phenolic compounds. It shows great potential for improving the biological activity of bioactive compounds or realizing the value of by-products by extracting bioactive substances. Until now, the information about the effects of some novel technologies on specific bioactive ingredients is still very little, and the results of the same processing conditions are not suitable for the study of each food matrix. It is also necessary to better understand the complex physicochemical mechanism of processing technology and its impact on food processing and bioactive characteristics, and more research in this field is needed.

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Part II
Influence of Conventional Processes on Food Bioactive Compounds

Chapter 3
Influence of Frying, Baking and Cooking on Food Bioactives

Knut Franke, Fabrice Tonfack Djikeng, and Tuba Esatbeyoglu

3.1 Introduction

Food is generally processed for consumption or preservation. Among various processing methods, those using dry and wet heat are the most popular ones. They are generally employed to transform raw ingredients to finished or ready-to-eat products with good stability, sensory properties and digestibility applied either at home or by the food industry (Al-Juhaimi et al. 2018). Cooking, frying, boiling, blanching, steaming, roasting, baking, drying, pasteurization and sterilization among others are the main methods used for food processing (Akdaş and Bakkalbaşı 2017). These procedures influence the shelf-life, safety, sensory, physical, and chemical properties of foods (Rehman et al. 2003). Some of these changes induced by food processing are desired such as increased shelf-life due to microbial and enzymatic inactivation, increased availability of nutrients, increased digestibility, and improved taste, texture, flavor, odor, color and edibility. In this way, consumers' satisfaction and well-being are addressed as well as the economic stability and growth of the industry. Processing can also lead to a better bioaccessibility of active compounds, reaching the intestinal tract where they are absorbed, metabolized and transferred to target tissues (Ifie and Marshall 2018).

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However, processing can result in modification of physicochemical, nutritional and sensory properties of foods (Al-Juhaimi et al. 2018). Processing methods can significantly affect food ingredients and products by decreasing their nutritional value through reactions such as non-enzymatic browning, oxidation, leaching or thermal degradation and negatively affecting organoleptic properties (Veda et al. 2010). At a certain extent, these changes can make food inadmissible by consumers.

It is well known that the majority of foods contains bioactive compounds such as vitamins, minerals, polyphenols (e.g. flavonoids, tannins), phenolic acids, stilbenes, glucosinolates, alkaloids, polyunsaturated fatty acids (PUFA), phytosterols, carotenoids, terpenes, etc. (Lattimer and Haub 2010; Markowiak and Slizewska 2017; Cao et al. 2019; Scolaro et al. 2020). Their chemical structures, occurrence and health benefits are clearly described in the literature (Septembre-Malaterre et al. 2018; Walia et al. 2019). Unlike essential macro- and micronutrients (such as fat, protein, carbohydrates, mineral elements, and vitamins), the body can function properly without them. However, their consumption has a positive impact on physiological or cellular activities in humans and animals (Walia et al. 2019). Because of their potential biological effects in humans, it is important that changes in their contents during processing should be evaluated to better assess the dietary and functional properties of the processed foods.

Based on the reasons mentioned above, this chapter presents the existing reports on the impact of cooking, frying and baking on polar and non-polar bioactive compounds of foods.

The most important polar and non-polar compounds, which can be considered to possess bioactivity in human nutrition, are the polar polyphenols, phenolic acids, glucosinolates and Vitamin C and the non-polar PUFA, vitamin E (tocopherols and tocotrienols), phytosterols, and carotenoids. After a brief introduction to each of the bioactives, frying, cooking, and baking as typical food preparation processes and their general effect on these substances will be discussed considering available research data found in literature.

3.2 Retention of Polar Food Bioactives

As mentioned above, the retention of bioactives during frying, cooking, and baking will be discussed separately for polar and non-polar substances, because they differ in their stability within the food matrices as well as in the interaction with the surrounding heating medium, e.g. frying oil or water. This part of the chapter is focused on the polar or water-soluble bioactives that are polyphenols including flavonoids, hydroxybenzoic acids, hydroxycinnamic acids, glucosinolates and water-soluble vitamins (ascorbic acid, folate).

Polyphenols, also known as polyhydroxyphenols, are compounds with at least one aromatic ring containing hydroxyl groups (Quideau et al. 2011; El Gharras 2009). Polyphenols are among the most widespread plant secondary metabolites (Rasouli et al. 2017). More than 8000 different phenolic compounds have been

found in the plant kingdom (Kabera et al. 2014; Santini et al. 2013). Polyphenols have simple and high molecular weight structures (El Gharras 2009). They help plants fight against external aggressors of different origin. However, it is important to note that each species displays a set of phenolic compound structures with different intra- and inter-cellular distribution. Various polyphenols have been reported to possess interesting biological properties, i.e. antioxidant, anticancer, anti-inflammatory, antimicrobial, and antibacterial activity, etc., that make them beneficial for human health (Cory et al. 2018). Some examples to polyphenols identified in plants can be listed as quercetin, kaempferol, catechin, tyrosol, oleuropein or rutin.

Glucosinolates (*S*glucopyranosyl thiohydroximates; GSL) are important sulfur-containing anionic hydrophilic plant secondary metabolites in cruciferous vegetables (Blažević et al. 2020). Glucosinolate-rich plants are cabbage, e.g. white cabbage, Chinese cabbage, broccoli, mustard, horseradish, watercress, horseradish, capers, and radishes (Riso et al. 2014). When these plants are chewed, cut, or otherwise damaged, glucosinolates are released and isothiocyanates and other degradation products are produced (Ishida et al. 2014). The decomposition products of glucosinolates usually contribute to their unique taste (Vig et al. 2009). Glucosinolates are reported to possess beneficial roles for human health, since they show protective effects against cancer and cardiovascular diseases (Traka 2016).

Water-soluble vitamins are almost non-toxic, but their deficiency can lead to nutritional disorders (Zhang et al. 2018). Vitamin C, also known as ascorbic acid (AA), is a lactone of polyhydroxycarboxylic acid and has an enediol structure. In nature, it exists mainly as L-isomer and the content of D-isomer is very low. It is also widely used as an antioxidant in the food industry to inhibit enzymatic browning of fruits and vegetables. With respect to vegetables, green pepper, tomato, cauliflower and various dark leafy vegetables, and among fruits, citrus, lemon, jujube, hawthorn and kiwi, are rich in vitamin C. As a powerful dietary antioxidant, vitamin C is well known for its health-promoting effects (i.e. antioxidant and anti-inflammatory activity) (Pullar et al. 2017).

During frying, cooking and baking of foods, the retention of these polar bioactives can significantly be affected by the heat that breakdown molecules with low thermal stability or facilitate the release of bioactives bound to cell wall or to other molecules. In the same line, leaching of polar bioactives from the food into water can contribute to variation in bioactive retention.

3.2.1 Influence of Frying on Polar Bioactive Retention

Frying is an old and popular cooking method which normally takes about 5–10 min or more depending to the food product being processed and involves high temperatures such as 150–200 °C (Combe and Rossignol-Castera 2010). It increases the palatability of food, generates pleasant organoleptic properties (flavor, color and odor) and enables crust formation (Aladedunye and Przybylski 2009). The oil acts

as a heat transfer medium. The process has a preserving function due to destroying of microorganisms and enzymes and due to reducing water activity of the food products (Bordin et al. 2013). Additionally, the frying fat becomes part of the final product substantially contributing to taste and flavor, but may also deliver bioactives to the product (Chiou et al. 2012). Vice versa, bioactives can migrate into the frying oil.

The effect of frying is not always beneficial. High processing temperatures promote lipid oxidation that leads to rancidity of foods. In the same line, lipophilic vitamins are destroyed due to their low thermal stability (see next section), same with some labile bioactive compounds. These changes in foods and oil depend on factors such as the nature of oil used, surface/volume ratio, oxygen availability, temperature, heating process and level of immersion of the food (Bordin et al. 2013). However, the longer the oil is used, the greater adverse reactions may occur, which might result in the release of possible toxic substances into the frying oil and food (Del Ré and Jorge 2006).

It is important to note that during frying processes, the temperature distribution within the food is heterogeneous. The highest temperature is observed at the periphery of the food while the center of the food containing relatively higher amounts of water shows lower temperatures (100–105 °C), making nutrients and bioactives breakdown more intense at the periphery (Da Silva and Moreira 2008; Moreira et al. 1999).

Different types of frying processes can be distinguished: deep-fat frying, shallow-fat frying, stir frying and pan frying. Each process has a different impact on the nutrients and bioactive compounds of foods due to their varying processing conditions.

Deep-fat frying is a cooking process of foods which requires their immersion in hot oil at temperatures varying between 165–190 °C. During deep-fat frying, heat is transferred by convection from the oil onto the food surface and from the surface to the center of the product by conduction (Oke et al. 2018). The chemical reactions that take place in oil are hydrolysis, oxidation and polymerization.

In a study carried out by Kumar et al. (2017), the change in phytoceuticals concentration during deep-fat frying of snacks with nutra-coconut oil (NCO, mix of coconut oil and solvent-extracted flaxseed oil) showed that the total phenolic content significantly decreased. The degradation of these molecules correlated with frying temperature and time. The degradation was significantly higher in pure coconut oil compared to that in NCO. This finding indicated that despite their decomposition, phytoceuticals have good protective effects on the oil. A reduction in phenolic antioxidants generally implies a decrease in antioxidant potential. In many studies, a significant correlation between the phenolic concentration and antioxidant efficacy of plant extracts was demonstrated (Gertz et al. 2000). Gomez-Alonso et al. (2003) evaluated the changes in phenolic composition and antioxidant activity of virgin olive oil during deep-fat frying. Their results showed that after 10 min of frying at 180 °C, the amount of dihydroxyphenol components was dropped to 50–60% of the original value. After six frying processes under similar conditions, only about 10% of those bioactives remained. However, they noticed that tyrosol stability as

well as that of its derivatives linked to the aldehydic and dialdehydic forms of elenolic acid was good after twelve frying operations.

In a study investigating the effect of different cooking methods on nutritional and physicochemical characteristics of selected vegetables, Miglio et al. (2008) showed that deep-fat frying of carrots, courgettes and broccoli in peanut oil at 170 °C led to a significant decrease or total destruction in chlorogenic and ascorbic acids; while the other active principles such as *p*-coumaric acid, caffeic acid increased. The decrease in some bioactives was justified by their labile character at high temperature, while the increase in others such as caffeic acid can be attributed to breakdown of chlorogenic acid. It has been reported that during frying, chlorogenic acid can be hydrolyzed into caffeic and quinic acids, therefore increasing their concentrations (Miglio et al. 2008).

In another report, Barakat (2014) investigated the effect of frying on nutritional and bioactive compounds of ovo-vegetarian diets and noticed that the ascorbic acid and total phenolic content of all the six diets formulated from various vegetables, such as cauliflower (*Brassica oleracea* var.), pea (*Pisum sativum* L.), green zucchini (*Cucurbita pepo* L.), taro corms (*Colocasia esculenta* L.), green bean (*Phaseolus vulgaris* L.) and green spinach (*Spinacia oleracea* L.), were significantly reduced during deep-fat frying (3–5 min at 180–190 °C). The reduction was more than 70% for ascorbic acid, while it was only 5–10% for total phenolic compounds.

Shallow-fat frying is an oil-based cooking technique suitable for foods with large surface/volume ratio. Here, the food touches the bottom of the pan during the whole process and is cooked in an oil reaching half of its thickness. The heat is transferred to the bottom surface by conduction from the hot surface of the pan (Oke et al. 2018). The oil layer is generally irregular, due to shape of the product fried. This together with water evaporation from the hot food causes temperature variations.

Very few studies are available on the impact of shallow-fat frying on polar food bioactives. In one study, the impact of shallow frying on the antioxidant potential and concentration of phenolic compounds' for selected vegetables was assessed (Gitanjali et al. 2004). The outputs of investigations exhibited a significant reduction in the evaluated parameters for spinach, amaranth and potato while the same parameters increased in carrot, tomato and eggplant. The decrease in phenolic compounds was justified by thermal degradation. In another study, Kalogeropoulos et al. (2007) investigated the retention and distribution of polyphenols during shallow frying. The polyphenols retention percentage in food ranged from 25 to 70%. The loss in antioxidants was attributed to their low stability at high temperature.

Stir frying is a cooking method originated from China. Here, the ingredients are fried in a small quantity of hot oil while being stirred. A traditional round-bottom cast iron or carbon steel pan called wok is heated to high temperatures. With this technique, dishes are cooked extremely quickly within minutes. Azizah et al. (2009) demonstrated that stir frying of pumpkin for 2, 4 and 6 min results in 18–54% losses in phenolic compounds indicating their instability under heat (Crozier et al. 1997; Zhang and Hamauzu 2004). In a similar study, Akdaş and Bakkalbaşı (2017) showed that stir frying for 5 min using pre-heated oil significantly reduced the total phenolic

(determined using the Folic-Ciocalteu assay) and ascorbic acid contents of kale from 20,868 to 12,677 mg GAE/kg dry weight (DW) resulting in a retention of 60.7%, and from 4918 to 2218 mg/kg (45.1% retention), respectively. In the same report, high losses in flavonols were detected in stir frying (79.7% for quercetin, 81.5% for kaempferol and 81.3% for total flavonoids). The antioxidant capacity tested using the TEAC (Trolox Equivalent Antioxidative Capacity) method showed a drop from 91.9 to 61.1 mmol Trolox eq/g. The authors justified the decrease in phenolic bioactives and antioxidant potential of kale by the selected processing parameters and their levels.

In a study investigating the effect of food processing methods on bioactive compounds of cauliflower, Afaf (2015) showed that total phenolic and flavonoid contents of cauliflower were significantly lowered during stir frying at 160 °C. The total phenolic, flavonoid and vitamin C contents of raw cauliflower were 763 mg GAE (gallic acid equivalent)/100 g, 236 mg CE (catechin equivalent)/100 g and 63.4 mg/100 g DW, respectively. After frying, these values dropped to 613 mg GAE/100 g, 198 mg CE/100 g and 43.4 mg/100 g DW, respectively. Similar data were obtained by Ahmed and Ali (2013) who demonstrated that after stir frying of cauliflower for 4 min 30 s at 140 °C the following phenolic compounds were significantly destroyed: gallic acid (from 11.9 to 3.50 mg/100 g), pyrogallol (from 18.9 to 12.5 mg/100 g), protocatechuic (from 192.4 to 52.4 mg/100 g), chlorogenic acid (from 25.83 to 10.45 mg/100 g), quercetin (from 202.5 to 58.7 mg/100 g), coumaric acid (from 6.94 to 2.10 mg/100 g) and kaempferol (from 25.9 to 11.8 mg/100 g) analyzed by HPLC in raw and processed samples. Other bioactives such as catechin, catechol, rutin, naringenin, rosmarinic acid, vanillic acid, syringic acid and cinnamic acid who were initially present in the raw cauliflower were no longer detected after stir frying. However, the caffeic acid content increased after frying. During stir frying, the breakdown of chlorogenic acid into quinic and caffeic acid can take place explaining the increase in caffeic acid concentration in cauliflower (Miglio et al. 2008). It is important to note that the loss of polyphenols can also be related to covalent binding established between oxidized phenols and proteins or amino acids. It can also be due to the polymerization of the oxidized phenols (Friedman 1996). Fauzan et al. (2020) reported that stir frying for 10 min at 105 °C significantly increased the total phenolic concentration of several green leafy vegetables, such as spinach, water spinach, cassava leaves, katuk leaves, long bean leaves, pumpkin leaves, squash leaves, green mustard, radish and papaya leaves, while it induced a high loss in vitamin C content. However, the authors did not explain the increase in phenolic compounds, but explained the drop in vitamin C by the degradation effect of heat.

Similar to stir frying, **pan frying** is characterized by the use of a very small quantity of oil. The oil is used here just as a lubricant (McGinnis 2006). Here, the food is flipped at least once to ensure that both sides are properly cooked. It requires lower heat to avoid burning of the periphery of the food. Gunathilake et al. (2018) studied the effect of cooking methods on the polyphenol and antioxidant capacity of some edible leaves. Results showed that pan frying at 170 °C reduced the polyphenol content of *Olax zeylanica*, *Cassia auriculata*, *Sesbania grandiflora*, *Gymnema*

lactiferum, *Pedulis* and *Centella asiatica* by 80.7%, 18.9%, 75.2%, 75.8%, 76.3% and 20.6%, respectively. Frying decreases the phenolic concentration of leafy vegetables, probably by leaching into oil or deterioration due to high processing temperatures. Han and Xu (2014) investigated the impact of some home cooking techniques on bioactives of amaranth and showed that pan frying at 232 °C for 4 min had no effect on the total polyphenol content of amaranth (1.94 mg/g for both raw and pan-fried products). However, it led to the significant reduction in anthocyanins (50.9 and 6.15 μg/g for raw and fried products, respectively) and ascorbic acid content (1.23 and 1.02 mg/g for raw and fried products, respectively). The loss in ascorbic acid and anthocyanin contents was explained by thermal destruction.

Laleh et al. (2006) showed that temperature increase is associated with greater degree of destruction in anthocyanins of *Berberis* species due to the hydrolyzation of the free glycoside structure.

3.2.2 *Influence of Dry-Heat Cooking Processes on Polar Bioactive Retention*

Baking refers to the process of dehydrating, drying, and hardening materials by dry heat under the ignition point of the materials and is usually applied in an oven. Baked products (especially bread) are a common and important staple food. Due to the high temperature above 100 °C to 400–500 °C and the loss of water, baking has a huge impact on content and structure of bioactives and nutrients in the food. High temperatures required by this technique can affect the nutrient and bioactives present in foods. Yang et al. (2016) investigated the influence of baking on some physical and nutritional characteristics of potato tubers. Potatoes were baked in an electric oven at 250 and 220 °C for 60 and 65 min, respectively. They found that the retention of total phenolic compounds after baking at 250 °C for 60 min was significantly higher compared to that of the ones baked at 220 °C for 65 min in all the tested cultivars except Kennebec. The authors attributed this to the elevated reactivity of phenolic molecules that undergo several reactions during processing that are related to the cultivars and cooking conditions. Investigations carried-out by Navarre et al. (2010) showed that the concentration of extractable phenolic antioxidants increases when the food is baked for 30 min at 375 °C. On the other hand, Perla et al. (2012) reported a 54% loss in total phenolic content after 60 min baking at 204 °C. The influence of baking and addition of olive leaf extract and inulin particles on oleuropein retention and accessibility in a food matrix was assessed by Pacheco et al. (2018). They found that baking reduced oleuropein for about 23%. This was justified by the high solubility of oleuropein which could be increased by raising the temperature. Therefore, mobility of the molecules and compound diffusion through the matrix can also increase due to higher solubility. Reports show that different interactions can be established between these molecules and starch, which might facilitate compound retention in such a matrix. These interactions may occur

through the establishment of low energy bonds such as hydrogen bonds, whereby hydroxyl or carbonyl groups found in the structure of phenolic compounds may interact with hydroxyl groups of the starch and of the water (Zhu 2015; Pacheco et al. 2018). Such an interaction can be established between oleuropein and the matrix through hydrogen bonds prolonging its stability and increasing its retention percentage. The increase in flavonol concentration of about 25% during oven baking of onions was reported by Lombard et al. (2005). In the same line, it was demonstrated that cooking beans for more than 2 h before baking them reduced folic acid retention by 50% (USDA 2007). McDougall et al. (2010) studied the effect of various cooking regimes on rhubarb's polyphenols and registered a dramatic decrease in the relative amount of anthraquinone aglycones between 5- and 10-min baking which was accompanied by a decrease in concentration of anthraquinone glycosides and the putative anthraquinone dimer derivatives. However, after 20 min baking, there was a slight recovery in the abundance of some anthraquinone derivatives which was explained by the increase in extraction of these compounds from the tissues or by their degradation into dimeric anthraquinone molecules.

In baked foods such as bread and biscuits, the retention rate of total polyphenols ranged from 20 to 65% (Žilić et al. 2016; Alvarez-Jubete et al. 2010; Perla et al. 2012). However, the retention rate of total polyphenols in extra virgin olive oil (EVOO) and virgin olive oil (VOO) after baking at 180 °C for 90 min was reported to be up to 77–89%, respectively (Goulas et al. 2015). In contrast, Rodriguez-Mateos et al. (2014) and Zambrano-Moreno et al. (2015) showed that baking maintained the total polyphenol content of wild blueberry, organic eggplant and conventional eggplant unchanged or even increased to 120%. Regardless of the difference in the food substrate, the occurrence of the above phenomenon seems to be related to the baking temperature.

Fruits such as blueberries, strawberries, etc. are often used in bakery products to enrich the taste and aesthetics. Some by-products of the fruit industry such as grape skin, dried apple peel powder, raspberry and cranberry pomace powder are also added. Such fruits are usually rich in polyphenols, especially anthocyanins. However, studies have confirmed that anthocyanins are unstable and easily decompose (Mildner-Szkudlarz et al. 2016; Rupasinghe et al. 2008). Mildner-Szkudlarz et al. (2016) showed that the average recovery rate of anthocyanins in muffins with blueberries was 22%. The recovery rate of cyanidin-3-*O*-galactoside in muffins with dried apple peel powder was reported to be 20% (Rupasinghe et al. 2008). Karakaya et al. (2016), Li et al. (2011) and Górnaś et al. (2015) showed that the retention rate of total anthocyanin was up to 63–98%. When cookies with blue maize were baked at 179 °C for 4 min, the loss of anthocyanin was about 12% (Li et al. 2011). In the study of Karakaya et al. (2016), the anthocyanin retention rates of buns, biscuits and breadsticks baked with grape skin were shown to be 95, 98 and 63%, respectively. According to Rodriguez-Mateos et al. (2014), the possible reason is the influence of pH value. Acidic baking powder containing citric acid or lactic acid lowers the pH and better retains the polyphenols. Alkaline baking powder such as baking soda and soda increases the pH value of the entire system and causes a greater loss of polyphenols (Rupasinghe et al. 2008). Among them, catechins,

total phenolic acid and chlorogenic acid are also affected and the content is reduced. Anthocyanins degrade during processing into their aglyca (Markakis 1974). Cyanidin-3-*O*-galactoside is most affected with a retention rate of 20% (Rupasinghe et al. 2008), and the retention rates of malvidin, peonidin and petunidin derivatives are all around 75% (Rodriguez-Mateos et al. 2014). Mildner-Szkudlarz et al. (2016) also reported an average recovery percentage of 53 and 156 of flavonols and ellagic acid, respectively, in muffins supplemented with raspberry and cranberry pomace powder. They found that ellagic acid was less affected by lower baking temperature while anthocyanins (only 22% retention) and flavonols where significantly influenced. In view of bringing in clarification to the changes observed, the authors mentioned the strong interaction between ellagic acid and the cell wall (Rommel and Wrolstad 1993). They also specified that the breakdown of ellagitannins to release hexahydroxydiphenic acid and their transformation into ellagic acid can also explain its increase (Bobinaite et al. 2012).

Green tea extracts are rich in flavonoids and phenolic acids. Adding green tea extract to biscuits can improve their health properties (Sharma and Zhou 2011). Studies have shown that large amount of catechins is lost during the baking process. After baking at 160 °C for 10 min, the retention rate of epigallocatechin gallate (EGCG) was about 20.0% and that of epicatechin gallate (ECG) 27.5%. The loss in catechins is due to the alkaline pH, the interaction of catechins with certain ingredients in the dough, the isomerization or oxidation and degradation of catechins during the baking process (Sharma and Zhou 2011; Wang and Zhou 2004).

In muffins, quercetin glycosides were significantly lost during baking (about 39–45%). This can be explained by the fact that quercetin glycosides can get thermally hydrolyzed and deglycosylated into the aglycon quercetin (Rupasinghe et al. 2008; Rohn et al. 2007). During baking process, 3,4′-*O*-quercetin diglucoside can also be converted into 4′-*O*-quercetin glucoside. According to Lombard et al. (2005) the ratio of 3,4′-*O*-quercetin diglucoside/4′-*O*-quercetin glucoside changed from 1:1.3 to 1:1.7 in baked onion, showing a 40% conversion. For the total flavonoids, the retention rate in roasted potatoes and roasted corn was about 45% (Perla et al. 2012; Žilić et al. 2016), while in onions, the concentration increased to 106% due to evaporation of water (Lombard et al. 2005).

The loss in vitamins such as folic acid during baking was shown to be minimal compared to other cooking methods such as boiling (Keagy et al. 1975). The retention rate of folate in grilled fish and shellfish was about 80–90% (Bergström 1994).

3.2.3 Influence of Processes Based on Water as Heat Transfer Medium on Polar Bioactive Retention

This section reviews the thermal cooking methods using water as heat transfer medium, including boiling and steaming. Boiling is a moist-heat cooking technique in which food is cooked for a relatively long time by immersing food in boiling

water (100 °C). Steaming is one of the natural and healthy ways of all the conventional methods to cook the food (Saikia and Mahanta 2013). It appears that the impact of wet-thermal processing is related to the degree of exposure to heat and water, more precisely, the exposure time and contact with liquid water (Volden et al. 2009). The effects of these traditional cooking methods on polyphenols and glucosinolates can follow the order: boiling > blanching > steaming. In some cases, the ranking of boiling and blanching may change, but steaming seems to be the best way to retain bioactive substances.

Steaming is carried out by continuously boiling water to evaporate it into steam. The steam brings heat to nearby food, thereby cooking the food. Food is kept separately from boiling water but in direct contact with steam. Afaf (2015) showed that steaming of cauliflower florets led to a significant drop in vitamin C, total phenolic and flavonoid contents from 63.4 to 40.1 mg/100 g, 763 to 542 mg GAE/100 g and 236 to 197 mg CE/100 g, respectively. Consequently, their antioxidant capacity was also affected (from 59.2 to 42.5%). The HPLC analysis carried out by Afaf (2015) and Ahmed and Ali (2013) on raw and steamed cauliflowers showed a significant loss in phenolic acids and flavonoid content. In the same line, significant losses in glucoiberin, progoitrin, sinigrin, glucoraphanin, gluconapin, glucoiberverin, glucobrassicin and gluconasturtiin were reporded by Hwang (2019) during steaming of fresh cauliflowers. The author justified the decrease in glucosinolates by the cooking time and elution of part of the bioactives from the food. Steaming was also shown to reduce the total glucosinolate content in five cultivars of cauliflower ('Aviso' (white), 'Dania' (white), 'Grafitti' (purple), 'Emeraude' (green) and 'Celio' (romanesco)) by 18–22%, which is lower than that 30–52% with blanching and 46–61% with boiling (Volden et al. 2009). The level of aliphatic glucosinolate decreased by 11–35% and the decrease in indole glucosinolate was 16–39%. The indole glucosinolates in cauliflower significantly decomposed during steaming, but those found in cabbage were almost not affected (reduced by 2%) (Rungapamestry et al. 2006). The difference in thermal decomposition properties of glucosinolates in different varieties can be attributed to different types of glucosinolates or to the cell environment (Sarvan et al. 2014). The second point can be seen from the findings of Hanschen et al. (2012) where the same glucosinolate showed different thermal stability behavior under different pH values in the environment. According to the data of Rungapamestry et al. (2006), it can be seen that the total glucosinolate content first increases and then decreases during steaming. This may be related to the inactivation process of myrosinase. A model of myrosinase inactivation is a rapid initial inactivation period, followed by a slow decay, and finally plateaus (Minchinton et al. 1982). Total glucosinolates were affected with reductions of 19% in steamed red cabbage (Volden et al. 2008). Similar observations were made by Wu et al. (2019) with some glucosinolates during steaming of raw broccoli. At the same time, they also showed that the concentration of kaempferol (Km) 3-*O*-caffeoyldiglucoside-7-*O*-glucoside, Km 3-*O*-sinapolylferuloyltriglucoside-7-*O*-diglucoside and Km 3-*O*-sinapolylferuloyltriglucoside-7-*O*-glucoside increased or remained unchanged during steaming, which was justified by the good ability of this cooking method in retaining bioactives compared to boiling. A related study was carried out by

López-García et al. (2018) on quintonil (*Amaranthus hybridus*) leaves harvested in spring and fall seasons and a significant increase in total phenolic (TPC) and flavonoid (TFC) contents was recorded after steaming the vegetable for 10 min in a stainless-steel steamer. Before processing the TPC and TFC content of samples harvested in spring and fall were 145 and 583 mg GAE/kg; and 78.6 and 349 mg QE (quercetin equivalent)/kg, respectively. After steaming, the values changed to 1480 and 1706 mg GAE/kg, and 489 and 797 mg QE/kg, respectively. However, concentration in vitamin C significantly decreased (21% for steaming in spring and 27.9% for fall). Similar data on the change in total phenolic and flavonoid contents were obtained by Saikia and Mahanta (2013) after steaming of some vegetables, i.e. pea, cabbage, carrot, and beetroot. The authors explained the increase in total phenolic and flavonoid contents by the fact that cooking breaks and releases bound phenolic acids and flavonoids from cell constituents. They also mentioned that the disruption releases oxidative enzymes such as polyphenol oxidase that can destroy the phenolic antioxidants present in fruits and vegetables as reported by Chism and Haard (1996), and that steaming seems to deactivate those enzymes, thereby preserving the phenolic antioxidants present in the food. Concerning vitamin C, its loss was explained by its thermal instability. As reported by Gregory (1996), a decrease in vitamin C content is attributed to a chemical breakdown involving ascorbic acid's oxidation into dehydroascorbic which is broken down into 2,3-diketogulonic acid and finally polymerization leading to inactive molecules. According to the data of Warthesen et al. (1984), the retention rate of vitamin C during steaming was relatively high. Except for the highest loss rate of 60% in spinach after steaming for 4 min, the retention rate of vitamin C in other vegetables including broccoli, carrots, cauliflower, peas, zucchini and green beans was 68–90%.

Pellegrini et al. (2010) reported a decrease in broccoli and cauliflowers polyphenols of about 33.1% and 45.5%, respectively. In addition, polyphenols in *Gymnema lactiferum*, *Sesbania grandiflora* and *Olax zeylanica*, which are consumed as leafy vegetables, showed retentions of 59.0, 60.1 and 36.2%, respectively, while the polyphenol content of *Cassia auriculata* and *Centella asiatica* almost doubled during steaming. Only *Passiflora edulis* showed a relatively low polyphenol retention rate of 11.5% (Gunathilake et al. 2018). Another example of the increase in polyphenol content was described by Turkmen et al. (2005) who demonstrated that the polyphenol content of green beans increased by 30%, and the content of broccoli increased by 18%. When processing and cooking cruciferous vegetables, the total flavonoid retention rate of steamed cauliflower was reported to be 85%, while that of boiled cauliflower was 26.1% (Hwang 2019). Steamed artichokes also showed higher phenol content (increased by 34% compared to raw). From the above point of view, steaming can be considered as the best cooking method for vegetables with high antioxidant capacity and can even significantly increase the level of antioxidant activity (Murador et al. 2017).

Boiling is one of the most commonly used methods used in the preparation of foods. Here, the food is totally immersed in water and cooking is performed in boiling water or other water-based liquids such as stock or milk at similar temperature for a specific period depending of the food product. During this process, water

soluble vitamins and polar bioactive compounds are affected. Azizah et al. (2009) and Afaf (2015) investigated the effect of boiling for 2, 4 and 6 min on the total phenolic content of pumpkin and cauliflower and indicated a significant loss in phenolics (18.3–53.8%). Podsędek et al. (2008) showed that the longer the boiling time, the greater the loss of phenols. When the boiling time was shortened from 20 min to 10 min, the retention rate of phenols in cooked cabbage was increased by 3.8–6.7%. After 5 min cooking of conventional cauliflower, the total phenol retention rate was 30% (Vallejo et al. 2003; Zhang and Hamauzu 2004). In the research conducted by Turkmen et al. (2005), 94% of phenols were retained in boiled broccoli. In addition, reducing the amount of water by 50% was reported to reduce the loss of phenolics in cooked foods (Palermo et al. 2013; Podsędek et al. 2008). Lee et al. (2019) investigated the influence of boiling on functional compound contents of Shiitake mushrooms. They found that boiling reduced the contents of gamma aminobutyric acid (GABA), ergothioneine, gallic acid and catechin with retention of 7.41, 26.3, 32.7 and 45.4% respectively. The authors explained the loss by the disruption of the plant tissues and the release from the matrix after heating (Faller and Fialho 2009). Hwang (2019) observed a significant decrease in glucoiberin (retention: 60.0%), progoitrin (59.8%), sinigrin (59.8%), glucoraphanin (85.7%), glucobrassicin (56.1%), gluconapin (59.9%), gluconasturtiin (97.0%) and glucoiberverin (59.9%) content during steaming of cauliflower for 2 min.

Glucosinolate content was significantly reduced when foods such as vegetables were washed before being cooked or blanched. Generally, discarding boiling water significantly reduces the glucosinate content (by 40–60%), same with long cooking times (Sing and Thornalley 2007). Similar results were obtained for broccoli, described by Wu et al. (2019).

pH value, temperature and heat treatment time are factors that affect vitamin loss (Warthesen et al. 1984). Dang (2000) showed that the retentions of folate after boiling of chickpeas and peas were 52.3 and 45%, respectively. The report of Lee et al. (2018) showed that the vitamin C content of spinach and broccoli were reduced by 34.8 and 44.6% respectively, after boiling.

3.2.4 Summary of Frying, Cooking, and Baking on Retention of Polar Bioactives

The data explored showed that frying, cooking and baking are processing techniques involving high temperatures that significantly influence the retention of polar bioactives present in foods.

All frying processes (deep-fat frying, shallow, stir and pan frying) have a considerable influence on the retention of polar bioactives present in foods. Their influence is generally justified by factors such as thermal degradation of labile molecules; the hydrolysis of some bioactives into others; the leaching of bioactives into the frying oil (deep-fat frying) and the interaction between some of the bioactives and other

molecules such as free radicals in view to limit oxidation reactions taking place during the process. The retention of phenolic compounds in general as well as that of ascorbic acid was significantly reduced during frying. A contradictory observation was made with caffeic acid which retention increased during frying due to the hydrolysis of chlorogenic acids. The reduction in retention was more severe with ascorbic acid compared to phenolic compounds in general. Amongst the frying techniques, shallow, stir and pan frying were better than deep-fat frying in retaining bioactives. This can be attributed to the difference in the amount of oil used and the nature of the process. It is however important to note that the length of the frying process as well as the nature of the food being fried can lead to significant variations in retention time of particular bioactives.

As far as processes with water or steam as heat transfer medium are concerned, steaming and boiling were found to differently affect the retention of polar bioactives. The decrease in retention of polar bioactives was significantly higher with boiling than with steaming. This is because polar molecules amongst which active principles, e.g. some phenolic compounds, vitamin C and glucosinolates, are broken down due to high temperature and others are leached-out into the water during boiling. This is facilitated by the affinity between the substrate (polar bioactives) and the solvent (water). The decrease in polar active principles during steaming is attributed to their thermal breakdown. It was also noticed that during steaming, the concentration of some polar bioactives such as some glucosinolates and phenolic compounds can increase instead. This was justified by the effect of heat that breaks and releases bioactive molecules bound on cell constituents. Compared to frying, even though boiling is performed at lower temperature, it seems to be more efficient in retention of polar bioactives. Steaming seems to be the best technique amongst them for the retention of polar active principles in foods. The variations in retention observed can be attributed to the nature and type of food.

Concerning baking, its effect on polar bioactives varied, too. As previously mentioned, nature and type of the food as well as processing conditions can justify the recorded variations. In the same line, baking reduces the retention of folic acid and anthraquinone glycoside derivatives for more than 50%. Since these molecules have low thermal stability, they easily decompose under high processing temperatures. The report from the literature show that the retention is better during baking compared to boiling.

3.3 Retention of Non-polar Food Bioactives

This part of the chapter is focused on the non-polar or fat-soluble bioactives. PUFA can be considered as a class of fatty acids with more than one double bond in the carbon chain. Especially, the long chain PUFA with three or more double bonds where the first double bond can be found at the third carbon atom (*n*-3 or ω-3) are of interest in nutrition. They are considered to improve brain function and to reduce the risk of cardiovascular diseases, diabetes, cancer, Alzheimer's disease and

dementia (Shahidi and Ambigaipalan 2018). Within this section, especially the stability of three members of this class will be discussed. The first one is α-linoleic acid (ALA) with 18 carbon atoms and three double bounds. Typical sources of ALA are edible plant oils, such as canola oil or linseed oil. The other two PUFAs with high relevance in nutrition and health are the eicosapentaenoic acid (EPA) with 20 carbon atoms and 5 double bounds and the docosahexaenoic acid (DHA) with 22 carbon atoms and 6 double bonds. The main source of these long chain PUFAs is fish, fish oil or algae oil. All these PUFAs are normally bound either in triacylglycerides (TAG) together with other fatty acids or in phospholipids depending on the origin of oil (Schneedorferová et al. 2015). Due to the high number of unsaturation, these fatty acids are very susceptible to oxidation and further degradation, if they are subjected to higher temperatures. Availability of oxygen during heating contributes to a faster deterioration of these substances. Additionally, light and presence of metal ions can further accelerate the degradation of these fatty acids (Choe and Min 2006).

Phytosterols are organic compounds from plants consisting of a steroid skeleton with a polar hydroxyl group and with or without a double bound in the ring structure. The different sterols can be distinguished in the aliphatic side chain at one of the rings. Often the hydroxyl group is esterified with a fatty acid usually palmitic acid or oleic acid. Examples for phytosterols are stigmasterol, camposterol and β-sitosterol, e.g. Normén et al. (2002). Phytosterols are relevant for lowering blood cholesterol level (Bartnikowska 2009). During heating in presence of oxygen, they can be oxidized losing their health effects in human nutrition (Hovenkamp et al. 2008).

Carotenoids are another class of plant-based non-polar bioactives consisting of long-chain hydrocarbons. They can be divided into two classes: carotenes (without oxygen, non-polar, e.g. β-carotene) and xanthophylls (containing oxygen, polar, e.g. lutein). Especially β-carotene as a precursor for vitamin A is of health relevance. However, other carotenoids, like lutein are potential non-polar antioxidants, as well (Jørgensen and Skibsted 1993). They are susceptible to oxygen (discoloration), but also to temperature at very high levels, e.g. heat bleaching during deodorization of palm oil (Rossi et al. 2001).

Finally yet importantly, the vitamin E family has been included in this review because of its various bioactivities in humans. The family covers both the tocopherols with the α-tocopherol possessing the highest vitamin E activity, but also the class of tocotrienols. Due to their excellent antioxidant activities, tocopherols play an important role in preventing oxidation in food also during heating for preparation. This might also result in a degradation of these compounds losing their bioactivity as discussed below (Piironen et al. 1987; Schneider 2005).

Overall, lower retention levels of these non-polar bioactives after heating processes might originate both from thermal degradation due to the influence of higher temperatures and due to oxidation, if the bioactives in the food matrix are exposed to oxygen. Another aspect, which could be relevant for the final retention, is the leaching effect by the surrounding oil in the case of frying. Because the non-polar

bioactives are soluble in lipids, parts of them can migrate into the frying oil and lower the overall retention in the food.

Therefore, the three different food preparation processes have their own unique particularities with respect to retention of such compounds.

3.3.1 Influence of Frying on Retention of Non-polar Bioactives

With respect to non-polar bioactives, frying could either result in their leaching into the frying medium or, in the case of products with a low initial fat content, to an addition of non-polar bioactives to the product during frying, because the frying oil or fat itself could be a source of bioactives as is explained below.

Because of the similarity between stir frying, pan frying and shallow frying in comparison to deep-fat frying, only two classes of frying (deep-fat and pan frying) are discussed with respect their influence on non-polar bioactives.

The latter one is of special interest for **PUFA**. Because of their high degree of unsaturation, PUFA are very susceptible to oxidation (Brühl 2014). Therefore, fat and oils used for long-term deep-fat frying are rather low in PUFAs due to their low stability at high temperatures. Otherwise, food cooked in the fryer can contain PUFA, e.g. fish, which retention is of interest in this context. Al-Saghir et al. (2004) reported only slight modification in fatty acid patterns of salmon fish during pan-frying. Especially, no changes in *n*-3 PUFAs were measured after 6 min of pan-frying. In contrast, Flaskerud et al. (2017) detected a reduction in PUFAs during pan frying of rainbow trout in different oils with an influence of frying oil on degree of reduction. Frying in corn oil and canola oil did not alter the PUFA content, whereas a decrease in PUFA was detected after frying in palm oil and high oleic sunflower oil indicating that exchange of fatty acids between product and frying oil distinctly influenced PUFA retention in the product. Candela et al. (1998) investigated the influence of deep-fat frying of high-fat fish in sunflower oil. They found a rather high decrease in *n*-3 PUFA from 24.0 to 6.6 g/100 g fat in raw sardines (27.5% retention), for example. Similar reductions were reported for the oil from mackerel and salmon. They also measured a distinct shift in the ratio of *n*-6 to *n*-3 PUFAs resulting from exchange of fatty acids with the sunflower oil used in the fryer. Similar results with respect the *n*-6 to *n*-3 ratio shift due to a combination of fat exchange and deterioration of the *n*-3 PUFAs during deep-fat frying were mentioned by Sanchez-Muniz et al. (1992). Choo et al. (2007) reported a decrease in ALA during heating of flaxseed oil for 6 min at 150 °C with losses of about 10%.

Multari et al. (2019) investigated the changes in frying oils during long deep-fat frying for 60 min. They found only a small (approx. 1.5%) but significant decrease in PUFA content of hemp oil, but no significant decrease of PUFA content in lupin and oat oils. Additionally, Al-Khusaibi et al. (2012) found only 'minimal reductions' in *n*-3 and *n*-6 PUFAs during repeated frying of potato chips in a blend of palm olein and canola oil. If only canola oil is used for deep-fat frying of French fries, the decrease in PUFA correlates with frying time and temperature (Aladedunye

and Przybylski 2009). After a period of 7 days, only about 50% of the initial contents of PUFA were found. Aniolowska et al. (2015) measured the loss of 20% for PUFA from rapeseed oil after 8 h of deep-fat frying compared to 7% loss in a commercial blend with rapeseed oil containing also high oleic sunflower oil and palm oil.

Vacuum frying as a deep-fat frying process under reduced pressure seemed to have a special influence on PUFA contents in fish patties (Albertos et al. 2016). It enables a lower temperature due to the reduced water boiling point resulting in higher retention rates of PUFA compared to traditional frying processes. The authors measured losses of 53% and 59% during vacuum and traditional frying for 10 min, respectively.

With respect to stability of **tocopherols** during deep-fat frying, Rossi et al. (2007) stated that it depends on the fatty acid composition of the oil, especially PUFA content, and the composition of the tocopherol fractions. After using palm oil for frying of French fries at 175 °C for 18 h, about 27% of the α-tocopherol could be found whereas tocotrienols were completely deteriorated. However, the authors measured a very high retention of more than 80% after 12 h of frying in the case of sunflower oil.

Reductions of α-tocopherol between 4.9 and 99.8% were measured by Crosa et al. (2014) during traditional and vacuum frying in refined high oleic sunflower oil for 10 days depending on type of frying and addition of antioxidants. In the similar way, Albertos et al. (2016) measured a higher retention of tocopherols during vacuum frying compared to traditional frying of fish. They mentioned an increase in tocopherol content of the fish after frying in both process originating from the surrounding frying oil. However, tocopherol content in the fish subjected to vacuum frying (10 min) was nearly two times higher than that after traditional frying after the same frying time.

Aladedunye and Przybylski (2009) demonstrated that tocopherol loss is highly dependent on temperature. After 7 days of frying at 185 °C about 31% of the tocopherols were still in the oil, whereas the entire amount was lost after frying at 215 °C for the same time. A better retention of tocopherols in blends compared to the single oils was reported by Al-Khusaibi et al. (2012) for frying of potato chips. The kinetics of tocopherol losses in commercial oils used for deep-fat frying were recently investigated by Liu et al. (2019). They confirmed a linear reduction rate with frying time depending on the type of oil and, therefore, its fatty acid composition. Considering pan frying, instead of deep-fat frying, of salmon for 6 min at 180 °C, almost stable tocopherol levels were found by Al-Saghir et al. (2004). Retention was between 88 und 93% for α-tocopherol.

The main sources of **phytosterols** in frying processes are related to the frying oil, mostly plant-based edible oils. Whereas several data about phytosterol contents in fresh refined and non-refined oils can be found in the literature, only few studies on their stability during frying are available. Winkler and Warner (2008) investigated the loss of this class of bioactives in soybean and high-oleic sunflower oil heated at frying conditions (180 °C, 8 and 12 h). All initial antioxidants were removed from the oils before addition of phytosterols. They found retention levels for the individual phytosterols between 87 and 93% in soybean oil and 80–87% in sunflower

oil, respectively. Recently, Kasprzak et al. (2020) investigated the loss of phytosterols in refined rapeseed oils. After heating at 180 °C for 8 h, they found a decrease in total phytosterol content from 797.3 to 665 mg/100 g corresponding to a retention of about 87%, which is close to the previous results. Phytosterol retentions of 69%, 74% and 84% were measured in sunflower oil, olive oil and palm oil, respectively, after using them for pan frying of French fries at 175 °C for 6 min (Chiou et al. 2009). Orozco-Solano et al. (2011) investigated the phytosterol concentrations in different vegetable oils during 20 heating cycles (from ambient temperature to 180 °C with 5 min holding time). They found significantly different retention rates between only 10% and more than 100% depending on oil and individual phytosterols. They also detected an increase of some phytosterols after the first five cycles, which they attributed to a hydrolysis of conjugated forms.

Considering the food being fried in the oil, e.g. French fries, an enrichment of phytosterols in the final food originating from the oil used for drying can be observed due to the oil absorption (Salta et al. 2006; Chiou et al. 2009).

Compared to phytosterols, both the oil and the food being fried can be considered as a source of **carotenoids** in the frying process. Only about 25% of the initial contents measured before frying were detected for lutein after a simulated frying process at 180 °C for 3 h (Blasi et al. 2018). Another study determined the degradation kinetics of total carotenoids in crude palm oil, refined canola oil and a blend of both during their use for frying of French fries for at temperatures between 170 and 190 °C for 20 h (Mba et al. 2017). They determined order of reaction (n = 1.5), reaction rates and activation energies for carotenoid degradation in each oil which enabled calculation of retention rates for selected frying times, temperatures and oils. Additionally, Schroeder et al. (2006) calculated a retention rate of 48% for carotenoids in red palm oil after 60 frying cycles (3 h in total) of French fries at 165 °C.

As mentioned above, product subjected to frying can also represent a source for carotenoids. Kidmose et al. (2006) reported retention rates between 73 and 93% for β-carotene in pepper, sweet potato and tomato after stir frying for 3–12 min. Interestingly, they observed low amounts of extraction of carotenoids by the frying oil used. Whereas, Lee et al. (2003) described a distinct increase of carotenoids in soybean oil during frequent frying of carrot enriched dough slices fried at 160 °C and a total heating time of 20 h. The carotenoid content in the fried products remains nearly constant during the frying experiments. The changes in the carotenoid contents in carrot chips themselves during stir frying in different oils and at temperatures between 165 to 185 °C was investigated by Sulaeman et al. (2001). They detected significant influence of temperature but not of the oil type on carotenoid degradation. Retention rates between 68 and 95% for total carotenoids were measured after frying times of 3–5 min of each chip depending on frying temperature. Similar to other non-polar bioactives, a positive effect of vacuum frying (lower oil temperature) on retention of carotenoids was found by Da Silva and Moreira (2008). They fried different fruits and vegetables in a traditional fryer (165 °C) as well as in one with lower pressure (125 °C). The final carotenoid contents were higher by

18%, 19% and 51% for green beans, mango chips and sweet potato chips, respectively, compared to those after traditional frying.

3.3.2 *Influence of Baking on Retention of Non-polar Bioactives*

The source of the non-polar bioactives during baking of food is limited to the food subjected to the baking process.

Sources for **PUFA**, which are relevant for the discussion of the retention during baking, can be found in fish as a supplier of long chain *n-3* PUFA as well as in bread and other baking goods enriched with PUFA. Several authors investigated the PUFA retention during baking of fish. Weber et al. (2008), Sengör et al. (2013), and Flaskerud et al. (2017) measured no alteration in fatty acid profiles during baking of catfish, rainbow trout, and salmon. This was confirmed by Neff et al. (2014) by claiming that baking has little effect on *n*-3 fatty acids in four different freshwater fish. Controversially, decreases in PUFA during baking of pike, carp, cod and herring were detected by Schneedorferová et al. (2015). They found retentions between slightly more than 100% and less than 30% after baking (200 °C for 15 min) depending on species. Higher losses were measured in the marine fish.

One possibility for enrichment of traditional baking products with PUFA is the addition of linseed high in α-linolenic acid (ALA). Simbalista et al. (2012) studied the retention in linseed-enriched bread. They found similar fatty acid profiles before and after baking (dough and crumb) with no significant decrease in ALA. Comparable stabilities were reported by Chen et al. (1994) for linseed-enriched muffins after baking at 178 °C for 2 h, both for the application as a whole seed or as a coarsely ground powder. These results indicate that linseed contains a sufficient amount of antioxidants to stabilize the ALA during baking. In another approach, Henna Lu and Norziah (2011) fortified bread with a microencapsulated *n*-3 PUFA powder based on fish oil. They observed losses up to 20% in EPA and DHA after baking for 30 min at 180 °C, which could be attributed to an insufficient amount of available antioxidants to protect these fatty acids.

Compared to the data on stability of PUFA during baking, much less data are available on the stability of **tocopherols** in the baking process. Similar to PUFA, several researchers investigated their behavior in enriched breads. Tocopherol retention rates between 70 and 90% were reported by Alvarez-Jubete et al. (2009) for gluten-free breads based on amaranth, quinoa and buckwheat. Palm oil as an ingredient to obtain higher levels of tocopherols and tocotrienols in bread was investigated by Buddrick et al. (2015). They found lower retentions (about 60%) in rye bread compared to more than 80% in wheat bread. The individual influence of baking on the retention of the tocopherol fractions was studied by Piironen et al. (1987). They also observed differences between wheat and rye bread with retentions of 75 and 88% for α- and β-tocopherol, respectively, in wheat bread and only 50% of

α-tocopherol in rye bread. Even an increase in tocopherol content in tomatoes baked at 160 °C for 20 min was found by Hwang et al. (2012). They addressed this effect to a breakdown of cell walls enhancing the release of these substances from the matrix.

With respect to the fate of **phytosterols** during baking, Rumiyati et al. (2015) did not observe a significant reduction of these substances after baking of muffins at 190 °C for 25 min.

The retention of **carotenoids** in tomatoes during baking was investigated by Hwang et al. (2012), too. Similar to tocopherols, they measured an increase after baking due to an enhanced release from the matrix as it was explained. Especially the retention of β-carotene during baking of buns enriched either with carrot or drumstick leaves or with a synthetic form of β-carotene was investigated by Thatte et al. (2011). They reported retentions between 62 and 72% for these products whereas only 35% in buns enriched with the synthetic substance. Baking of sweet potato at temperatures between 150 and 200 °C resulted in an increase of β-carotene at the beginning of the baking process and a decrease at longer baking, e.g. 30 min at 200 °C, as it was reported by Huang et al. (2012). Different results with respect to the so-called 'true' retention of carotenoids were reported by Shin et al. (2016) for yellow-fleshed and white-fleshed sweet potato after baking at 200 °C for 20 min followed by 140 °C for 10 min. They found retentions of about 75% and 110% for the yellow-fleshed and the white-fleshed crops, respectively. Retentions above 100% were also addressed to improved liberation resulting from the baking process. An average retention rate of only 12% for carotenoids was measured by Kotíková et al. (2016) in yellow-, purple- and red-fleshed potatoes (22 samples in total) after a very long baking of a quarter of each potato tuber (180 °C, 45 min). If the local temperature was lower than 100 °C, e.g. in the crumb during baking of bread, the retention of carotenoids was much higher. Hidalgo et al. (2010) measured about 97% retention in the crumb compared to only 71% in the crust.

3.3.3 Influence of Cooking on Retention of Non-polar Bioactives

As already discussed in Sect. 3.2.3, boiling and steaming can be considered as the main relevant cooking methods where water, either as liquid or as vapor, acts as a heat transfer medium.

Because this part is related to non-polar bioactives, their leaching into the surrounding polar water does not really contribute to changes in retention of these bioactives. Immersion in water during cooking prevent food from access to oxygen, which distinctly reduces oxidative degradation of susceptible bioactives such as PUFA. Differently to frying but similarly to baking, the food being cooked is the only source of the non-polar bioactives. Therefore, only this source has to be considered for determination of retention.

Degradation of **PUFA** during cooking can be mostly attributed to effects of high temperature. Since fish are a good source of PUFA, especially the EPA and DHA, several authors investigated the fate of PUFA during boiling of fish as often used in home cooking and restaurants. Asghari et al. (2013) investigated the effect of boiling (15 min) on rainbow trout fillets and found no significant changes in PUFA content after boiling. Similar findings were reported by Sengör et al. (2013) for steaming of salmon (40 min at 100 °C). PUFA retention rates of 97 to 99% in oil seeds during boiling (30 min) were reported by Kiczorowska et al. (2019). Two groups investigated the retention of PUFA in hard-boiled eggs (12 min) enriched in these fatty acids by a special hen's diet. Botsoglou et al. (2012) found a slight but significant decrease with retention rates between 92 and 96%. In a similar approach, but with different hen's diets, Cortinas et al. (2003) measured a PUFA retention of about 98% after hard-boiling of eggs for about 30 min.

With respect to **tocopherols**, the same authors described retention rates between 86 and more than 100% for α-tocopherol in hard-boiled eggs of hens fed with tocopherol supplementation. The lowest retention rates were detected for eggs with a higher content of long chain PUFA, which can be attributed to a higher consumption of antioxidant capacity in these eggs during heating to protect the PUFA. Retention rates of 99, 100 and 96% were found for total tocopherol contents after cooking of camelina seed, sunflower seed and flax seed, respectively (Kiczorowska et al. 2019). Also in this case, the lowest retentions can be attributed to a higher content of the ALA in flax seed compared to the other ones. An increase in the apparent concentrations of α- and γ-tocopherol after cooking of broccoli for up to 10 min was reported by Hwang and Kim (2013). They explained the effect by an improved extractability of the substances after cooking. Similar results were found by Lee et al. (2018) studying cooking of vegetables using different, but adapted cooking times (between 5 and 20 min). Retention was calculated as the apparent 'true' retention where the masses of the food before and after cooking were considered (Murphy et al. 1975). For vegetables cooked only 5 min, e.g. spinach, chard, or broccoli, Lee et al. (2018) also found true retentions much higher than 100%. Whereas the longer cooking times, 20 min for potatoes or 12 min for carrots, resulted in a distinct loss of tocopherols with retentions of about 70% or 47%, respectively.

Normén et al. (2002) investigated the retention of **phytosterols** in several cereals and cereal-based products during cooking and they observed no significant differences before and after cooking related to dry matter. Changes in total content could be attributed to water uptake. On the other hand, an increase of free phytosterols related to dry matter was found by Kaloustian et al. (2008) in plant products cooked for 30 min. In this case, the increase was explained by a hydrolysis during cooking process releasing the free phytosterols.

Retention of **carotenoids** during cooking of sweet potato for 45 min was recently studied by Kourouma et al. (2019). Interestingly, they observed relatively low retention rates of only 55% and 71% after boiling and steaming, respectively, for β-carotene as the main carotenoid in this plant. Parallel to tocopherols, Lee et al. (2018) also investigated the retention of β-carotene in the vegetables. They

measured a loss of this substance due to cooking, e.g. for broccoli, perilla leaf and carrots with retentions between 47 and 90%, as well as an increase in the content as for chard and spinach. The retentions higher than 100% after cooking were attributed to a disruption of the carotenoid-protein-complex in the plant cells. An increase in the apparent carotenoid concentration after cooking of broccoli was found by Hwang and Kim (2013), too. It was also attributed to a higher extractability of these substances. Such an effect was confirmed by the results of Shin et al. (2016) for different pulses. On the other hand, Nunn et al. (2006) reported retentions between only 70 and 88% for β-carotene and lutein in different vegetables cooked for about 7 min.

3.3.4 Summary of Frying, Baking, and Cooking on Retention of Non-polar Bioactives

Starting with the frying process, e.g. deep-fat frying and stir or pan frying, losses of up to 50% can be expected considering EPA and DHA in fish. Leaching into frying oil as well as thermal deterioration contribute to the low retentions. Controversially, retention of ALA as another bioactive PUFA is less affected by frying resulting in relatively high retentions of 90% and more. Highly connected to PUFA is the retention of tocopherols during frying, because they act as antioxidants protecting higher amounts of PUFA in food being fried. Therefore, higher PUFA contents result in a lower retention of tocopherols and vice versa. Additionally, higher availability of other antioxidants can contribute to a higher retention of tocopherols. Furthermore, pan frying is better than deep-fat frying in tocopherol retention because of the shorter usage of the oil. Keeping this in mind, the absolute retentions of tocopherols during frying spread within a wide range making it impossible to deliver generalizable values. Phytosterol stability during frying seems to be better than that of tocopherols with retention values higher than 50%. One study even found an increase in phytosterol content after short frying times. Carotenoids are reported to be even more stable compared to phytosterols. For this class of bioactives, the leaching into the surrounding oil during frying is the most relevant reason for losses.

Effects of baking can be compared to those of cooking (see below) especially in the inner parts of the food, e.g. breadcrumb, where the temperature remains below 100 °C. Therefore, ALA in enriched bread is stable in the breadcrumb. Whereas, retention of PUFA during baking of fish depends on type of fish with lower retentions in marine fish compared to fresh water fish. Baking also reduces the amounts of tocopherols with retentions of about 60%. Similar to cooking, tocopherol retentions strongly depend on PUFA content. Contents of phytosterols and carotenoids seem to increase during baking indicating changes in their extractability. Only in potatoes baked for a relatively long time, retention of carotenoids can become very low demonstrating a considerable loss of these substances attributable to heat damage.

Boiling or steaming as cooking processes generally have lower effects on non-polar bioactives compared to frying because of the lower temperature level and the polar heating medium. Retentions between 90 and 100% are reported for PUFA contents after these processes. Additionally, tocopherols does not show significant changes. Sometimes, even an increase after cooking is possible due to hydrolyses of oligomers or better release from food matrix. Similar effects regarding a retention above 100% after cooking can be observed for phytosterols and carotenoids. In the case of the latter one, the enhanced extractability seems to indicate even a better bioavailability of these substances after cooking. However, some of the results found in different studies are very inconsistent. For example, both an increase as well a decrease in carotenoid content after cooking of broccoli could be found in the literature signifying the need for further investigations.

3.4 Summary

The objective of this review was to investigate the impact of frying, dry-heat cooking (baking) and moist-heat cooking processes (boiling and steaming) on bioactive retention. Data available in the literature show that food processing plays a significant role on bioactive retention. High temperature processing methods which improve the safety of the food have both beneficial and detrimental effect on food bioactives. When such processes are applied with care, it is possible to improve the availability of active principles in foods through structural breakdown of parent compounds into other beneficial molecules, or release those that are covalently linked to the cell wall. Under certain conditions, processing will lead to the leaching or thermal decomposition of bioactives rendering them unavailable. Research should be centered toward the optimization of thermal methods that have the potential of retaining, releasing or at best transforming bioactive compounds present in foods into more bioavailable forms.

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Chapter 4
Influence of Chilling, Freezing and Thawing on Food Bioactives

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4.1 Chilling

Refrigeration is applied to foods to limit the various detrimental changes that occur during storage. These changes can be classified as microbiological, physiological, biochemical, and physical changes and result in deterioration of product quality. Chilling is defined as the process of reducing product temperature down to 0–8 °C (James and James 2014). Although the chilling process is applied to inhibit microbial growth, preserve quality and extend shelf life, it has been indicated that most food products are sensitive to low temperature and chilling results in changes in nutritional quality depending on processing conditions. A brief summary of the findings of some recent studies focusing on investigation of the effects of chilling process on bioactive compounds is presented in Table 4.1.

Fratianni et al. (2017) investigated the changes in various quality parameters of three different sweet basil (*Ocimum basilicum* L.) cultivars (*Italico a foglia larga, Cammeo,* and *Italiano classico*) during the postharvest storage at chilling (4 °C) or non-chilling (12 °C) temperatures. For this purpose, basil leaves were packed in macro-perforated polyethylene bags and stored for 9 days and visual quality, and chemical parameters were monitored. The authors reported that fresh basil leaves showed good antioxidant activity, with the sample concentration necessary to scavenge 50% of the DPPH radical activity ranging between 0.8 mg and 1.0 mg. However, storage at 4 °C or 12 °C was found to have a significant effect on antioxidant activity of basil. It was indicated that basil leaves stored at 4 °C showed chilling injury symptoms and complete loss of radical scavenging activity. A similar trend was also observed in in total polyphenols content, which was reported to be 2.5 mg

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Table 4.1 Findings of some recent studies investigating the effects of chilling process and several treatments on bioactive components

Product	Chilling temperature (°C)	Treatment(s) applied	Effects observed	Reference
'Kinnow' mandarin	5	Carboxymethyl cellulose coating	Higher ascorbic acid content, antioxidant enzyme activity, total phenolics content and radical scavenging activity in coated samples.	Ali et al. (2021)
Lemon	8	Methyl jasmonate and salicylic acid	Higher ascorbic acid content, antioxidant enzyme activity, total phenolics content, individual phenolics and antioxidant activity in treated samples.	Serna-Escolano et al. (2021)
Blood orange	3	Γ-aminobutyric acid, methyl jasmonate or methyl salicylate	Delayed decrease of total antioxidant activity and ascorbic acid content during cold storage in treated samples. Higher total phenolic content, total anthocyanin content and the major individual anthocyanins in treated samples. Methyl jasmonate treatment was found to be the most effective.	Habibi et al. (2020)
Blood orange	5	24-Epibrassinolide	Higher total phenolics, hydrophilic total antioxidant activity, and total anthocyanins in treated samples.	Habibi et al. (2021)
Pomegranate	4	Arginine	Higher ascorbic acid content, antioxidant enzyme activity, DPPH scavenging capacity, total phenols and anthocyanins accumulation in treated samples.	Babalar et al. (2018)
Pomegranate	2	Malic acid, oxalic acid and chitosan coating	Higher anthocyanin, ascorbic acid, total phenolic content, and antioxidant activity in treated samples.	Ehteshami et al. (2020)

(continued)

Table 4.1 (continued)

Product	Chilling temperature (°C)	Treatment(s) applied	Effects observed	Reference
Apricot	1	Methyl jasmonate and salicylic acid	Higher total antioxidant capacity, total soluble phenolic compounds and carotenoids content, phenylalanine ammonia lyase, peroxidase and superoxide dismutase activities in methyl jasmonate and salicylic acid treated fruits, increased catalase activity in salicylic acid treated and control samples.	Ezzat et al. (2020)
Apricot	2	Methyl salicylate	Relatively higher total phenolic content, total flavonoid content and antioxidant capacities in treated samples.	Fan et al. (2021)
Plum	2	Oxalic acid	Higher phenolics, anthocyanins, carotenoids and antioxidant enzyme activity in treated samples.	Martinez-Espla et al. (2019)
Kiwifruit	2 and 4	Ozone	Different effects observed in polyphenols, flavonoids, ascorbic acid, and carotenoids depending on temperature.	Goffi et al. (2019)
Sweet cherry	5	Ozone	Combination of precooling and ozone treatment helped retain the anthocyanin content.	Liu et al. (2021)
Hawthorn fruit	1	Glycine betaine	Higher antioxidant enzyme activity, phenols, flavonoids anthocyanins and ascorbic acid accumulation and DPPH scavenging capacity in treated samples.	Razavi et al. (2018)
Eggplant	4	Low-temperature conditioning and methyl jasmonate	Treatment was found to inhibit the decrease of chlorophyll and total phenolics and result in enhanced antioxidant enzyme activity.	Shi et al. (2019)
Bell pepper	5	Hot water treatment at 53 °C for 1–3 min	The lowest ascorbic acid loss was observed at 1 min treatment. Phenolic composition was found to be correlated with antioxidant activity.	Lopez-Velazquez et al. (2020)
Zucchini	4	Near-saturated relative humidity	Higher antioxidant enzyme activity and radical scavenging capacity in treated samples.	Zuo et al. (2021)

GAE/g in the fresh leaves and exhibited 20–78% loss at the end of storage period. On the other hand; storage at 12 °C was reported to preserve total polyphenols content and antioxidant activity of basil. Polyphenol profile of sweet basil leaves indicated that rosmarinic acid was the dominant polyphenol in all cultivars studied. Storage at 4 °C for was reported to result in 30–85% loss of rosmarinic acid whereas storage at 12 °C resulted in 6–47% loss. Varying amounts of losses were also reported for gallic, chlorogenic and caffeic acids depending on the storage temperature. On the other hand, apigenin, which was reported to be the only flavonoid initially present in fresh basil was found to increase after storage, for both 4 °C and 12 °C. It was indicated that chilling stress led to loss of membrane structure and oxidative stress and scavenging of newly formed radical oxygen species could result in decrease of phenolic compounds.

In a similar study, Vithana et al. (2018) investigated the effect of storage temperature and duration on bioactive compounds of mango (*Mangifera indica* L.). Mangoes were stored at 5 and 13 °C for 12–24 days and concentrations of lupeol, mangiferin, phenolic acids, ascorbic acid, carotenoids, total phenols and antioxidant capacity were monitored. Concentrations of lupeol, chlorogenic and caffeic acids were reported to be significantly higher in mangoes stored at 5 °C. On the other hand, concentrations of mangiferin, gallic, chlorogenic, vanillic, ferulic, and caffeic acids, total phenolic content, total carotenoids, and DPPH radical scavenging activity were found to be significantly higher in mangoes stored at 13 °C. Storage duration was found to have varying effects which mainly depended on the type of the bioactive constituent investigated. The authors proposed that storing mangoes at 5 °C for 12 days could be beneficial for preserving the concentration of lupeol, which was indicated to be an important anticarcinogenic compound in mangoes. Contrary to the observations of Fratianni et al. (2017) for sweet basil, Vithana et al. (2018) reported that antioxidant capacity in the mango pulp was significantly higher for fruits stored at 5 °C compared to 13 °C. Increase in the concentrations of lupeol, mangiferin and phenolic acids observed in the study were attributed to enhanced activity of phenylalanine ammonia lyase enzyme which activates biosynthesis of terpenes and phenols. Not only the pulp, but also the peel of the mango fruit stored at 5 or 13 °C was indicated to be a good source of health-promoting bioactive compounds.

Zaro et al. (2014) indicated that maturity stage of the fruit at harvest and storage conditions affect postharvest quality and bioactive compounds in fruits. The authors harvested purple eggplants at varying maturity stages (12–23 days after fruit set, indicated as stages I–V) and stored at 0 or 10 °C in order to determine the combined effects of fruit development level and storage temperature on chilling injury symptoms and bioactives of eggplants. It was reported that delphinidin-3-rutinoside was the main anthocyanin in purple eggplants at all maturity stages. The concentration of anthocyanins in peel was reported to reach the maximum level at stage II (day 15) and remain unchanged afterwards. Additionally, samples harvested at early development stages were reported to contain significantly higher amounts of carotenoids,

ascorbic acid and phenolic compounds in the pulp compared to samples harvested at late stages. Hydroxicinnamic acids were reported to represent 75–80% of total phenolic compounds whereas flavonoids represented the remaining 20%. Both hydroxicinnamic acids and flavonoids were indicated to decrease during fruit development. Similar to the trend observed for phenolic compounds, antioxidant capacity was indicated to decrease through development. Fruits harvested at early development stages were reported to maintain quality attributes better when stored at 0 °C. On the other hand, samples harvested at late stages storage at 10 °C was indicated to result in higher postharvest quality maintenance.

Abidi et al. (2015) investigated the effect of antioxidant compounds and sugars on susceptibility of peach (*Prunus persica* (L.) Batsch) to chilling injury. The authors studied 130 cultivars over 3 years for their phenotypic diversity in antioxidant compounds. Although all genotypes were reported to be grown under the same environmental conditions; a wide variation was observed in ascorbic acid, total phenolics and flavonoids content and relative antioxidant capacity among the genotypes studied. The variation observed in bioactive compounds and antioxidant capacity was attributed to the differences in genotype. The authors selected six genotypes with high total sugar and bioactive content and antioxidant capacity. Selected samples were stored at 5 °C and 95% relative humidity for 2–4 weeks where chilling injury symptoms were monitored. The selected genotypes were reported to show relatively low or intermediate chilling injury symptoms. Principal component analysis indicated that genotypes with relatively higher bioactive and sugar content, antioxidant capacity and fruit weight showed relatively lower flesh bleeding, which was monitored as a chilling injury symptom. It was concluded that the correlations observed between antioxidant compounds and chilling injury symptoms could be utilized for selecting the most suitable cultivars in terms of high bioactive content and good postharvest quality.

In a recent study, Fabroni et al. (2020) investigated the effect of cold storage on various components of blood and common oranges (*Citrus sinensis* L.). For this purpose, the authors stored three blood orange varieties ('Tarocco TDV', 'Tarocco Gallo', and 'Moro') and a common orange variety ('Washington navel') at 6 °C for 60 days and monitored the changes in various volatile and non-volatile components related with fruit flavor and quality. In case of bioactives, ascorbic acid content was reported to remain almost unchanged for all varieties during the entire storage period. On the other hand, total anthocyanins were reported to increase significantly in blood oranges which was attributed to activation of enzymes involved in phenylpropanoid metabolism and accumulation of anthocyanin pigments during exposure to low temperature.

Several pre- and post-harvest treatments have been applied to fruits and vegetables to overcome the development of chilling injury, maintain product quality during storage and preserve the bioactive compounds. These treatments can be listed as temperature dependent treatments such as precooling and intermittent warming, controlled or modified atmosphere storage, application of ethylene and ethylene

inhibitors, edible coatings, treatment with glycine betaine, methyl jasmonate and salicylic acid, and ultraviolet radiation (Rodrigues et al., 2020).

In a recent study, Ehteshami et al. (2020) applied malic acid and oxalic acid treatments to pomegranate fruits (*Punica granatum* L.) followed by chitosan coating. Organic acids were selected for their ability to induce tolerance for chilling injury in some fruits according to previous reports. Pomegranate fruits were dipped in oxalic acid (5 and 10 mM) and malic acid (50 and 100 mM) solutions for 10 min, air-dried and immersed in chitosan solution (1.5% w/v). Dried fruits were stored at 2 °C for 4 months. Chilling injury symptoms and bioactive content of fruits were monitored during storage. The authors reported that ascorbic acid content of fruits decreased during cold storage. Fruits treated with 5 mM oxalic acid + chitosan coating and 50 mM malic acid + chitosan coating were observed to contain the highest amount of ascorbic acid, total anthocyanins, total phenolic content and DPPH radical scavenging activity at the end of storage period. Reduced ascorbic acid oxidation due to chitosan application and antioxidant properties of organic acids were suggested to be effective factors for higher ascorbic acid content observed in treated samples compared to control. Higher anthocyanin retention in organic acid-treated samples was attributed to enhanced anthocyanin biosynthesis and decreased polyphenol oxidase activity. Moreover, higher total phenolic content observed in treated samples was suggested to be arising from the antioxidative properties of organic acids and reduced fruit tissue respiration due to chitosan. Finally, higher antioxidant activity in terms of DPPH radical scavenging capacity observed in treated samples was attributed to higher ascorbic acid, total anthocyanin and total phenolic contents. The authors concluded that 5 mM oxalic acid and 50 mM malic acid treatments combined with chitosan coating could be effectively used for maintaining fruit quality and bioactive properties during cold storage of pomegranates.

Ali et al. (2021) also investigated the effect of edible coating application on chilling injury symptoms and various quality attributes of ‘Kinnow’ mandarin (*Citrus nobilis* L. x *Citrus deliciosa* T.) fruits. The authors treated the fruits with 1% carboxymethyl cellulose (CMC) coating before cold storage at 5 °C for 30 days. It was reported that the CMC-coated fruits showed significantly higher antioxidative enzyme activities including ascorbate peroxidase, peroxidase, superoxide dismutase and catalase activity compared to uncoated fruits which was attributed to the protective effect of CMC coating which suppressed senescence and conserved the antioxidant enzyme activities. CMC-coated fruits were also reported to show significantly higher ascorbic acid content due to action of coating as a barrier against oxygen diffusion suppressing oxidative degradation of ascorbic acid. Although total phenolics content and 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity were observed to decrease in all samples, the decrease in antioxidative properties was reported to be significantly higher in uncoated fruits compared to CMC-coated fruits. At the end of the 30 days storage period, total phenolics content and radical scavenging activity of CMC-coated fruits were reported to be ~1.5-folds higher.

This finding was based on the fact that coating application kept the fruit surface intact and acted as a protective barrier against oxidation of phenolics and other antioxidant compounds. The authors concluded that edible coating application was found to result in higher antioxidative enzyme activity, phenolics content and radical scavenging activity, reduced oxidative damage of the fruits, which helped suppressing the development of chilling injury symptoms in mandarins.

In another recent study, Serna-Escolano et al. (2021) investigated the effects of methyl jasmonate and salicylic acid treatments on quality aspects of lemon (*Citrus limon*) fruit. For this purpose, fruits were treated with 0.1 mM methyl jasmonate and 0.5 mM salicylic acid solutions before cold storage at 8 °C for 35 days. It was indicated that the activity of the antioxidant enzymes catalase, peroxidase and ascorbate peroxidase was increased significantly by methyl jasmonate and salicylic acid treatments. The authors also measured the expression of the gene codifying for ascorbate peroxidase and it was found to be 1.5 and 1.7-fold higher in methyl jasmonate and salicylic acid treated fruits, respectively. Total antioxidant activity measured by the 2,2′-azinobis-(3-ethylbenzothiazoline-6-sulfonate) (ABTS)-peroxidase system, total phenolic content and the major individual phenolics (hesperidin and eriocitrin) and ascorbic acid content were found to be significantly higher in methyl jasmonate and salicylic acid-treated fruits compared to control samples. Increase in total phenolic content due to methyl jasmonate and salicylic acid treatments was attributed to increased phenylalanine ammonia lyase activity, which is indicated to be a key enzyme in phenolic biosynthesis pathway. Methyl jasmonate and salicylic acid treatments were found to be effective in controlling the senescence of lemon fruit during cold storage and resulted in higher antioxidant activity due to enhanced antioxidant enzyme activity and increase in phenolics and ascorbic acid.

Habibi et al. (2020) investigated the effects of γ-aminobutyric acid (GABA), methyl jasmonate (MeJA) or methyl salicylate (MeSA) treatments on bioactive compounds and quality attributes of blood oranges (*Citrus sinensis* L. Osbeck) during cold storage. Blood oranges were stored at 3 °C for 150 days followed by 2 days storage at 20 °C after the postharvest treatments. The authors reported that total antioxidant activity and ascorbic acid decreased during cold storage; however, the decrease was delayed in the treated fruits. Total phenolic content, total anthocyanin content, cyanidin 3-glucoside and cyanidin 3-(6″-malonylglucoside) were found to be higher in the fruits treated with GABA, MeJA and MeSA compared to the control fruits which was attributed to enhanced phenylalanine ammonialyase activity. It was concluded that MeSA treatment was found to be the most suitable treatment for maintaining fruit quality and anthocyanin content.

Hosseinifarahi et al. (2020) evaluated the effect of salicylic acid and *Aloe vera* gel on various quality characteristics of strawberry (*Fragaria* × *ananassa* Duch.) fruits. *Aloe vera* application was studied due to its previously reported antimicrobial properties and protective effects against quality loss in fruits whereas salicylic acid was selected based on its role in inhibition of ethylene biosynthesis and enhancing antioxidant enzyme activities. Strawberry samples were immersed in *Aloe vera* gel

(100%), salicylic acid solutions (1–2 mM), and a combination of *Aloe vera* gel (100%) and salicylic acid solutions (1–2 mM), dried and stored at 4 °C for 15 days. Ascorbic acid content of strawberries was reported to decrease in all samples during the storage period. Samples treated with a combination of *Aloe vera* gel and salicylic acid were observed to show the highest retention of ascorbic acid which was attributed to the barrier properties of the gel application against atmospheric oxygen and the ability of salicylic acid to decrease the destruction of ascorbic acid. Since single uses of *Aloe vera* gel and salicylic acid were not found to show a protective effect against ascorbic acid degradation, it was proposed that simultaneous use of the gel and salicylic acid had a synergistic effect on retention of ascorbic acid during storage. Similar to the trend observed in ascorbic acid, total phenolic content of strawberries was reported to decrease during storage and the samples treated with a combination of *Aloe vera* gel and salicylic acid showed the highest total phenolic content. This finding was explained by the protective effect of the gel coating reducing the oxidation of phenolics by limiting oxygen availability and enhanced activity of phenylalanine ammonia-lyase by salicylic acid resulting in biosynthesis of phenolics. Total anthocyanins content of all samples was also reported to decrease during storage. At the end of the storage period, samples treated with 2 mM salicylic acid and a combination of *Aloe vera* gel and 2 mM salicylic acid were observed to show the highest anthocyanin retention. Antioxidant activity of strawberry fruits in terms of DPPH radical scavenging capacity was reported to increase until the fifth day; but then decreased until the end of 15 days-storage period for all samples. Reduction in antioxidant activity was attributed to the generation of free radicals during cold storage. Simultaneous application of *Aloe vera* gel and salicylic acid was reported to enhance the antioxidant activity compared to control samples. It was indicated that higher ascorbic acid content and total phenolic content observed in the treated samples were effective in the higher antioxidant activity. The authors suggested that combined treatment of *Aloe vera* gel and salicylic acid could be utilized for maintaining quality parameters and bioactive compounds in strawberry fruits during cold storage.

Koyuncu et al. (2019) treated pomegranate (*Punica granatum* L.) fruits with salicylic acid, oxalic acid and putrescine and monitored various bioactive compounds and quality attributes during storage at 6 °C for 6 months under controlled atmosphere conditions (5% O_2 + 15% CO_2). Ascorbic acid content of all samples was reported to decrease during cold storage. Applied treatments were not found to be effective in retention of ascorbic acid in pomegranate fruits during cold storage. On the other hand, treated samples were indicated to have higher total phenolic content and antioxidant activity in terms of ferric reducing antioxidant power compared to control samples. Putrescine treatment was found to be the most effective treatment in maintaining bioactive compounds and antioxidant activity. It was proposed that salicylic acid, oxalic acid and putrescine treatments can be potentially used to delay quality losses, retain bioactive compounds and antioxidant activity during cold storage of pomegranate fruits.

Babalar et al. (2018) followed a different approach and applied a combination of preharvest arginine spray and postharvest arginine immersion to pomegranate fruits.

Samples were stored at 4 °C for 120 days and effects of arginine treatment on chilling injury symptoms, antioxidant enzymes, radical scavenging activity, ascorbic acid, total phenolics, and anthocyanins content were investigated. Arginine application was selected based on the ability of the amino acid to enhance the tolerance of agricultural products to chilling stress due to its role in biosynthesis of various signaling molecules. Preharvest spray and postharvest immersion of arginine were applied at varying concentrations of 0–2 mM. It was reported that activity of antioxidant enzymes catalase, superoxide dismutase, and ascorbate peroxidase decreased during cold storage. However, samples treated with arginine were reported to show significantly higher antioxidant enzyme activity compared to control samples which was attributed to partial minimization of membrane unsaturated fatty acids peroxidation by arginine treatment enhancing antioxidant activity. Similarly, ascorbic acid content of pomegranate fruits was reported to decrease during the storage period, with arginine-treated samples showing significantly higher ascorbic acid content compared to control samples. Higher ascorbic acid content in arginine-treated samples was attributed to enhanced glutathione reductase/ ascorbate peroxidase system activity and/or lower ascorbic acid oxidase activity. Moreover, samples treated with arginine were reported to show significantly higher total phenolic content, anthocyanins accumulation and radical scavenging activity during storage. Higher accumulation of phenolics and anthocyanins in arginine-treated pomegranate fruits was attributed to enhanced phenylalanine ammonia-lyase activity and decreased polyphenol oxidase activity. It was concluded that higher radical scavenging capacity observed in arginine-treated samples was due to enhanced antioxidant system activity and accumulation of bioactives which were found to be effective in enhancing not only chilling tolerance but also nutraceutical properties of pomegranate fruits.

Gao et al. (2016) investigated the effect of 24-epibrassinolide application on chilling injury symptoms and phenolic and proline metabolisms in peach (*Prunus persica* Batsch) fruits. Peach samples were immersed in 15 mM 24-epibrassinolide solution, which was selected due to the regulatory role of brassinosteroids in plant growth and developing resistance to environmental stress factors. Various chilling injury symptoms, total phenolic content, proline content and antioxidant enzyme activity were monitored during storage at 1 °C for 28 days. It was reported that 24-epibrassinolide application resulted in phenolic accumulation and scavenging of reactive oxygen species in such a way that total phenolic content was ~50% higher in treated samples compared to control during the whole storage period. Treated samples were observed to show significantly higher activities for enzymes associated with synthesis and metabolism of phenolic compounds, including shikimate dehydrogenase, phenylalanine ammonia lyase, cinnamate-4-hydroxy-lase and 4-coumarate: coenzyme A ligase. On the other hand, polyphenol oxidase and peroxidase activities were observed to be reduced in treated samples, resulting in higher total phenolic content compared to control. Treated samples were also reported to contain higher amounts of proline, which is indicated to have the ability to act as a potent antioxidant. The authors concluded that 24-epibrassinolide

treatment could be used as an alternative strategy for maintaining the postharvest quality of peach fruit due to its ability to regulate phenolic and proline metabolisms.

Goffi et al. (2019) applied postharvest ozone treatment to kiwifruit (*Actinidia chinensis* 'Soreli'). Samples were treated by ozone gas in continuous flow at 300 ppb and stored at 2 and 4 °C for 60 days. Effects of ozone treatment and storage temperature on various physicochemical and quality attributes of kiwifruits were investigated. Ozone is used as an antimicrobial agent in postharvest applications; however, it is also indicated to possess several disadvantages including targeting ethylene biosynthesis and cell walls turnover in some fruits. In this sense, the main goal of the authors was to determine the most efficient postharvest treatment conditions to maintain kiwifruit quality during storage. Microbial growth was reported to be delayed under gaseous ozone storage at 2 °C. Ascorbic acid content of fruits was indicated to decrease during storage and the authors observed no significant difference between the postharvest treatments or storage temperature. Decrease in ascorbic acid during cold storage was attributed to several possible factors including radical scavenging mechanism activated against decomposed ozone radicals, activation of ascorbate oxidase and the onset of senescence due to loss of cellular integrity. Total carotenoids content was reported to show a non-linear behavior which was based on the stimulation of the defense mechanisms due to ozone treatment. Total polyphenol content was indicated to decrease during storage at both 2 and 4 °C due to changes induced in polyphenol oxidase activity leading to degradation of phenolic compounds. Moreover, both ozone treatment and storage time were found to be effective in such a way that total polyphenol content of ozone-treated samples was found to be significantly smaller than that of untreated samples under storage at 4 °C at the 15. and 60. days. On the other hand, total flavonoids content was reported to be affected by both ozone treatment and storage temperature while DPPH radical scavenging activity was found to be affected by ozone treatment, storage temperature and their interaction. It was suggested that ozone treatment could be used to stimulate antioxidant activity of kiwifruits during storage at 2 °C. Combination of ozone treatment and storage at 2 °C was proposed to be an efficient postharvest treatment of kiwifruit since it delayed microbial growth and helped maintain the bioactive content of the fruit.

Vega-Alvarez et al. (2020) investigated the mechanism involved in chilling injury tolerance in mango fruit (*Mangifera indica* L., 'Keitt') induced by hot water treatment application. For this purpose, mature green mangoes were treated with hot water at ~46 °C for 75–90 min, stored at 5 °C for 20 days and then ripened at 21 °C for 7 days. It was reported that hot water-treated fruits showed a lower incidence of chilling injury symptoms. Hot water treatment was reported to show varying effects on fruit metabolites in such a way that it resulted in an increase in some metabolites including galloylquinic acids, gallic acid esters, and quercetin 3-O-rhamnoside. On the other hand, mangiferin, malic acid, and palmitic acid were reported to decrease in hot water-treated samples. Total phenolics content and antioxidant activity were determined to be higher in water-treated fruits compared to control. The authors indicated that higher total phenolics content and radical scavenging capacity was associated with increased levels of metabolites such as galloylquinic acids and

gallic acid esters in hot water-treated fruits. Chilling injury tolerance developed due to hot water treatment was attributed to accumulation of flavonoids and phenolic compounds, enhanced antioxidant activity and decreased oxidative stress. It was concluded that the mechanism behind chilling injury tolerance developed in mangoes treated with hot water could be explained by increased level of bioactive compounds and enhanced antioxidant activity, as well as increased level of osmolytes.

Although most of the studies focus on effect of chilling on food bioactives in fruits, there are recent reports on effect of chilling on various other agricultural commodities as well. For example, Zuo et al. (2021) investigated the effect of relative humidity on chilling injury symptoms and antioxidant system of zucchini (*Cucurbita pepo* L.) fruit. For this purpose, zucchini samples were stored at 4 °C and two different relative humidity conditions referred to as: "near-saturated relative humidity" (96–100%) and "normal relative humidity" (72–76%) for 15 days. Near-saturated relative humidity storage conditions were selected based on previous reports which indicated that high relative humidity storage could be effective in reducing water loss, decreasing chilling injury symptoms and maintaining postharvest quality in various fruits. The authors reported that near-saturated relative humidity storage enhanced the activity of antioxidant enzymes superoxide dismutase, catalase, and ascorbate peroxidase, which in turn resulted in higher DPPH radical scavenging activity compared to control. It was indicated that high relative humidity storage delayed the accumulation of free radicals. The authors concluded that near-saturated relative humidity could be effective in reducing chilling injury symptoms and maintaining postharvest quality of zucchini fruits by enhancing antioxidant enzyme activities and energy status.

4.2 Freezing

Freezing is a food preservation method in which chemical reactions and microbial spoilage are hindered, and the deterioration of physical properties of the food material such as color, texture or flavor are significantly reduced by conversion of liquid water into ice at low temperatures (Wu et al. 2017). Rapid freezing generates uniform and small ice crystals, causing less damage to cells in contrast to slow freezing thus providing a higher quality frozen food material (Cheng et al. 2017).

Freezing process takes place in three stages, namely, pre-cooling, phase change and sub cooling. Firstly, the food material is cooled to the freezing point temperature from an initial temperature at pre-cooling stage, then at the freezing point, water crystallizes into ice hence the phase change occurs and finally the material cools to a sub-freezing point storage temperature (Dalvi-Isfahan et al. 2017).

In direct freezing system, convection heat transfer is provided by transferring heat from one place to another with the movement of liquid (such as brine or cryogenic refrigerant) or gaseous (such as air). In indirect contact freezing systems, the material to be frozen needs to be in touch with the metal surface, which is directly

cooled by a refrigerant or cooled secondarily by another medium (conduction heat transfer) (Gökoğlu and Yerlikaya 2015).

In the food industry, plate contact, immersion, air blast, fluidized-bed, and cryogenic freezing are common methods. The selection criteria of a freezing method are;

- Type of the product (shape, size, uniformity)
- Desired product quality
- Easy cleaning ability
- Rate of freezing
- Exclusion of oxygen during freezing
- The availability of the product to be packed (Gökoğlu and Yerlikaya 2015; Rahman and Velez-Ruiz 2007)

4.2.1 Indirect Contact Systems

The product and refrigerant are separated by a barrier throughout the freezing process. Indirect contact freezing system is illustrated schematically in Fig. 4.1 (Singh and Heldman 2013).

4.2.1.1 Plate Freezers

In the plate freezer method, as illustrated in Fig. 4.2, the product is sandwiched between metal plates and pressure is usually applied for good contact. Plate freezers are only suitable for regular shaped materials or blocks (Rahman and Velez-Ruiz 2007).

Plate freezers are in batch mode or continuous mode. The batch plate freezers can be in horizontal or vertical arrangements containing many layers of plates. The feeding of the freezing system starts with the bottom layer in horizontal systems, whereas loading starts from the upper layer in vertical systems. The frozen product is released on the other side of the freezer (Gökoğlu and Yerlikaya 2015).

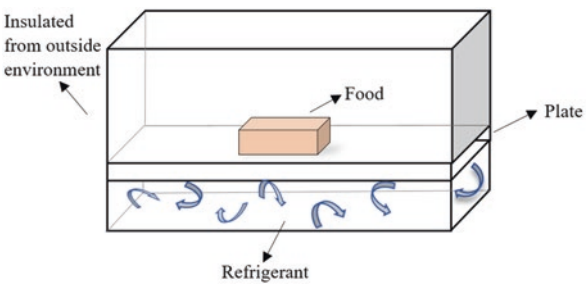

Fig. 4.1 Schematic diagram of an indirect-contact freezing system (Adapted from Singh and Heldman 2013)

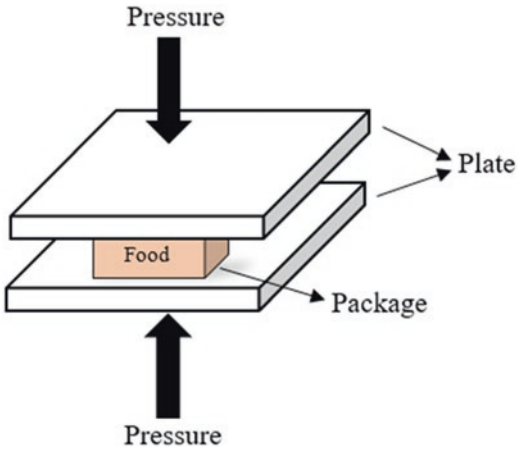

Fig. 4.2 Schematic illustration of a plate freezing system (Adapted from Singh and Heldman 2013)

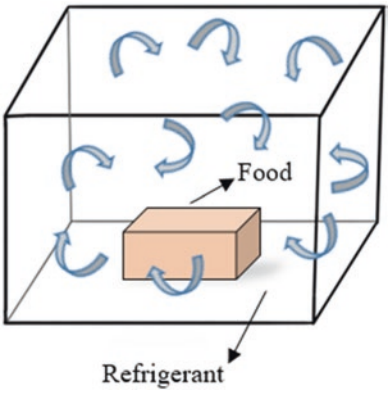

Fig. 4.3 Schematic diagram of a direct-contact freezing system (Adapted from Singh and Heldman 2013)

4.2.1.2 Freezers for Liquid Foods

Indirect heat exchangers are used for freezing of liquid foods, the most common type is a scraped-surface system. The basic system is a scraped-surface heat exchanger using refrigerant during phase change as the cooling medium. The rotor acts as a mixing device, and the scraper blades enhance heat transfer at the heat-exchange surface (Singh and Heldman 2013).

4.2.2 Direct Contact Systems

Direct contact freezing system is illustrated schematically in Fig. 4.3. The refrigerants used in these systems may be low-temperature air at high speeds or liquid refrigerants with phase change while in contact with the product surface (Singh and Heldman 2013).

4.2.2.1 Cabinet Freezing

In cabinet freezing method, cold air is circulated in a cabinet where product is placed in a tray. A cabinet freezer with air velocity at least 5 m/s generates high heat-transfer rates.

4.2.2.2 Air Blast Freezing

The temperature of food is reduced with cold air flowing at a relatively high speed in air blast freezing method. The air is cooled by the evaporator of the refrigeration equipment and circulated by fans over the product. Air velocities between 2.5 and 5 m/s give the most economical freezing. Lower air velocities result in slow product freezing, and higher velocities increase unit-freezing costs considerably and cause freezer-burn and dehydration of unpacked food material. Air blast freezing method can be further classified into tunnel freezing, belt freezing, and fluidized bed freezing, depending on how air interacts with the product (Rahman and Velez-Ruiz 2007; Gökoğlu and Yerlikaya 2015).

4.2.2.3 Immersion/Cryogenic Freezing

In cryogenic freezing, food is exposed to an atmosphere below −60 °C through direct contact with liquid nitrogen or liquid carbon dioxide or their vapor. A cryogen is the refrigerant that absorbs heat during phase transition. Cryogenic freezants have boiling points around −50 °C/−60 °C and a high latent heat of vaporization. Solid food material loaded on a conveyor is carried through a liquid nitrogen bath in cryogenic freezers. High heat transfer rates are achieved as liquid nitrogen is used rather than gaseous nitrogen in tunnel freezers. Around 500–800 Wm^{-2} $°C^{-1}$ heat transfer rates can be reached, however higher quantity of nitrogen is used in this method as 3–4 kg per kg of product compared to methods using gaseous nitrogen. Individual quick frozen (IQF) products, for instance dried cheese and hard-boiled eggs are produced by using immersion freezing. The product can be exposed to a cryogenic medium in three ways: (a) the cryogenic liquid is directly sprayed on the product, (b) the cryogenic liquid is vaporized and blown over the food, or (c) the product is immersed in cryogenic liquid (Rahman and Velez-Ruiz 2007; Gökoğlu and Yerlikaya 2015; Coulomb 2019).

4.3 Thawing

Thawing is a crucial part of freezing applications. Numerous food processing operations begin with thawing of frozen raw materials, therefore it is important to minimize adverse effects of thawing such as textural changes, microbial growth and drip

losses (Uyar et al. 2015). Conventional thawing methods include water thawing, air thawing and contact thawing. Novel methods such as radio frequency, microwave, ultrasonic and vacuum thawing can enhance process outcomes in terms of improved quality, shorter operation time and reduced oxidative reactions (Wang et al. 2020).

4.3.1 Air Thawing

Heat transfer takes place through convection by still air or forced movement of air typically with 6–10 m/s speeds. Lipid oxidation and surface drying may occur during slow thawing rates of air thawing (Backi 2018).

4.3.2 Water Thawing

Frozen food material is immersed into a vessel containing cold water for thawing. Heat transfer occurs by convection through surrounding water. Convection can be forced by stirring or by using running water (Leung et al. 2007). Continuous and batch processing can be applied in water thawing method and thawing rate is particularly higher compared to air thawing, surface drying kept at minimum and is used broadly in the industry accompanying immersion tanks or continuous thawers with conveyor belts (Backi 2018).

4.3.3 Contact Thawing

In contact thawing method, the food material is not in contact with the thawing medium, rather, a thawing medium with high specific heat capacity flows through plate walls, and the food material is in contact with metal plates (Backi et al. 2016).

4.3.4 Emerging Thawing Methods

Compared with common thawing methods referred above, some efficient and novel thawing methods available involving radio frequency, microwave, vacuum, high pressure, and ultrasonic thawing have been applied for frozen foods with the advance of technology (Wang et al. 2020).

Radio frequency (RF) thawing is an innovative dielectric technique that generates heat energy within food products due to ionic displacement, leading to direct conversion of electric energy to heat, a phenomenon often referred to as volumetric heating. The dielectric properties, shape, and size of the foods, and the location and

distance between RF parallel plate electrodes are important factors RF-treated foods (Choi et al. 2017).

High-pressure thawing can preserve food quality and reduce the necessary thawing time. During high pressure thawing, the drip loss of beef was too small to detect and there were no negative effects on colour, penetration force or cooking loss of thawed beef. Limitations on the application of high-pressure thawing are mainly high cost, and pressure-induced protein denaturation and meat discoloration (Li and Sun 2002).

Microwave thawing requires shorter thawing time and smaller space for processing, and reduces drip loss, microbial problems and chemical deterioration. Although the microwave thawing rate is faster, the thawing is not uniform. Localised overheating has limited the application of microwave thawing in food systems. In this case, food products take risk of excess water loss and chemical deterioration (Li and Sun 2002; Cai et al. 2018).

Ultrasonics thawing can speed up thawing process and shorten thawing time. There are two reasons for that, one is that the ultrasonics absorption of frozen part of food is much higher than that of thawed part, especially at the confluence of thawing region and unthawed region. The other is that the vibration energy carried by ultrasonics is absorbed by food materials and converted into the heat energy. The biggest deficiency of ultrasonics thawing is longer durations compared to microwave thawing (Cai et al. 2018).

4.4 Influence of Freezing and Thawing on Food Bioactives

Food constituents are affected and altered by processing methods applied for preservation or conservation of the subjected food material. Capacity of flavonoids, polyphenols or other bioactive compounds a food material retains depend notably on the type and intensity of processing methods. Freezing, frozen storage and thawing have serious impacts on the structural and textural quality of foods, therefore the amount of bioactive compounds would also be affected by the type of freezing method, the duration of frozen storage and the thawing method applied. Various researchers studied referred preservation method and significant influences on the bioactive content of foods are reviewed.

Flavonoid contents of frozen strawberry were evaluated in a study and the effects of 3, 6, and 9 months of storage are presented in Table 4.2. Approximately 35% higher quercetin levels were observed in strawberries stored at −20 °C for 9 months in contrast to fresh samples stored at 22 °C or 5 °C. The possible mechanism behind this finding is quercetin becoming more extractable during frozen storage which might be due to the degradation of cell structure of the food material. In red raspberry and black currant, quercetin levels were preserved during frozen storage, however myricetin content was reduced for black current and became unobservable after 9 months (Häkkinen et al. 2000).

Table 4.2 Quercetin and myricetin contents (milligrams per kilogram) in fresh berries kept at 22 °C or 5 °C for 24 h and in frozen berries kept at −20 °C for 3, 6, and 9 Months (Häkkinen et al. 2000)

	Fresh		Frozen		
	22 °C	5 °C	3 months	6 months	9 months
Strawberry					
Quercetin	4.2 ± 0.1	5.2 ± 0.3	6.9 ± 0.6	7.9 ± 0.5	8.0 ± 1.0
Red raspberry					
Quercetin	na	9.5 ± 1.2	8.3 ± 1.0	10.9 ± 1.8	9.7 ± 0.9
Black currant					
Quercetin	52.2 ± 1.0	52.9 ± 5.0	43.8 ± 11.2	43.5 ± 7.8	48.7 ± 3.1
Myricetin	85.8 ± 12.3	104.1 ± 10.0	70.8 ± 8.2	72.7 ± 1.5	nd
Bilberry					
Quercetin	na	41.2 ± 3.5	29.0 ± 2.7	30.8 ± 2.2	25.3 ± 1.7
Myricetin	na	nd	21.0 ± 2.1	19.0 ± 1.1	nd
Lingonberry					
Quercetin	na	169.0 ± 4.7	146.2 ± 56.6	137.3 ± 18.3	101.2 ± 7.5

Mean ± standard deviation of triplicate assays. na = not analyzed. nd = not detectable

Effects of individual quick freezing and storage of 6 months at −20 °C on anthocyanin concentration were evaluated for black raspberries and a slight increase was observed, which may be caused by the enhanced extraction of these components by tissue softening. Antioxidant capacity - ORAC values were constant after 3 months of storage and increased by 18% after 6 months apparently due to increased anthocyanin content (Hager et al. 2008).

In an extensive study conducted on blackberries, anthocyanin content of fresh fruits was compared to that of individually quick-frozen fruits with liquid nitrogen and slow frozen fruits (at −20) both stored 7 months at −20 °C. Hydroxycinnamic acids were found to be stable after frozen storage of IQF blackberries, besides slow freezing resulted in higher contents compared to fresh and IQF samples. The extraction of flavonols also increased after frozen storage due to cell damage caused especially by slow-freezing (Veberic et al. 2014).

De Ancos et al. (2000) studied the effects of quick freezing and subsequent frozen storage on the anthocyanin contents of four different cultivars of raspberry fruit, where two of them were early cultivars (cvs. Heritage and Autumn Bliss) and two were late cultivars (cvs. Rubi and Zeva). In early cultivars, frozen storage of 360 days showed increased values of anthocyanin contents by 41% and 31%, respectively for Heritage and Autumn Bliss. On the other hand, late cultivars Rubi and Zeva, lost their anthocyanin content by 19% and 9%, respectively, during frozen storage. Biochemical activity and composition differences of early and late cultivars were the reason for the contrasting effects of frozen storage on the anthocyanin content. The increase in the total anthocyanin content was due to the better extraction of these compounds as a result of cellular disruption caused by freezing, but the loss of these compounds for late cultivars was probably caused by the presence of

more reactive anthocyanins or the higher release of degradative oxidoreductase enzymes due to freezing.

In the case of frozen mango slices where four different cultivars (cvs. Lippens, Smith, Palmer and Davis-Haden) of fruits were stored at −18 °C for 120 days, ascorbic acid levels reduced by 57–73% due to freezing. It was suggested that drip losses of mango slices during thawing was minimal, thus ascorbic acid reduction was not induced by the thawing process. β-carotene contents were also lower after frozen storage except for Davis-Haden cultivar, which may be caused by cis-trans isomerization, epoxidation and oxidation reactions as suggested (Antonia Marín et al. 1992).

Ten fruits rich in phytochemicals, namely sour cherries, cherries, strawberries, red currants, raspberries, hawthorn, cornelian cherries, red grapes and white grapes, were frozen at −20 °C and analyzed after 1, 2, 3, 6 and 12 months of frozen storage in terms of total phenol, flavonoid and anthocyanin contents. Total phenol content of red currants, strawberries and raspberries increased by 27, 19 and 10% respectively, after 12 months of storage, while only hawthorn had a significant loss. Most of the fruits showed a significant increase in total flavonoid content after 1 year of frozen storage except strawberries, raspberries and hawthorn. Additionally, red grapes displayed a 91% increment after 3 months of storage. Further to this, total anthocyanin contents of cherries, red grapes and cornelian cherries showed significant increase by 77, 58 and 17%, respectively, while hawthorn and strawberries lost considerable amounts of their capacity (Šamec and Piljac-Žegarac 2015).

Allaith et al. (2012) studied the effects of thermal treatments and frozen storage on the antioxidant activity of two Bahraini date cultivars (cvs. Khalas and Khunaizi), given in Table 4.3. One month of frozen storage at −20 °C was notable on increasing the phenolic content where 168% and 143% increase for Khalas and Khunaizi cultivars were reported, respectively. Besides, total flavonoids showed a significantly increased content for both cultivars after storage. Researchers also measured the ferric reducing ability and diphenylpicrylhydrazyl (DPPH) quenching capacity of dates after frozen storage, where FRAP values increased by 87.6% in Khalas dates but reduced by 24.3% in Khunaizi dates. DPPH radical inhibition capacity of fresh and frozen Khalas dates were similar, however frozen Khunaizi dates showed a substantial increase in antiradical capacity compared to fresh samples.

Pilar Cano et al. (1993) studied the effects of frozen storage on four Spanish kiwi cultivars (Monty, Bruno, Abbot and Hayward), by freezing fruit slices of 6–8 mm in

Table 4.3 Effect of freezing on antioxidant activity of date cultivars (Allaith et al. 2012)

Cultivar	Treatment	FRAP value (mmol/100 g) ± SD	Weight (mg) resulted in 50% DPPH inhibition ± SD
Khales	Fresh	2.69 ± 0.08	121.76 ± 0.31
	Frozen (−20 °C)	5.06 ± 0.11	123.58 ± 1.43
Khunaizi	Fresh	3.48 ± 0.05	13.03 ± 4.14
	Frozen (−20 °C)	2.63 ± 0.00	0.67 ± 0.14

diameter in an air-blast freezer at −40 °C, and storing at −18 °C for 360 days. Controlled thawing was conducted at room temperature for 3 h. Ascorbic acid content was highest for Bruno cultivar compared to others, for frozen and raw fruit, however frozen storage for 12 months caused 37% reduction for this cultivar, while other cultivars faced 10–25% reduction depending on storage time and cultivar. Authors discussed that the decrease of ascorbic acid content could be due to drip loss occurring while thawing and the high liability of this molecule to processing conditions in unblanched fruits.

Novel technologies are being studied to overcome the adverse effects of various food preservation methods. Freezing process causes textural and structural damage on the food material and consequently loss of firmness after thawing. One novel approach proposed by Kong et al. (2017) to reduce the adverse effects of freezing on foods is the use of antifreeze peptides (AFPs). These molecules limit the size growth of ice crystals during freezing process by binding to crystal surface, thus improving final textural quality of the freeze-thawed food material. Researchers used three AFPs which were synthesized analogue to their natural counterparts produced by species of fish and insects. Cherry samples were kept immersed in AFP solutions for 7 days at 2 °C and subsequently slow-frozen and stored at −20 °C for 31 days. Samples treated with AFPs exhibited significantly lower drip loss compared to the untreated samples analyzed after thawing. Approximately 50% loss in the phenolic content was also observed for untreated samples after frozen storage, while the AFP treated samples showed insignificant decrease compared to fresh fruit. Correspondingly, AFP treatment reduced total anthocyanin loss compared to untreated fruits where around 55% of anthocyanin content was lost after 1 month of storage. Reduction of textural disruptions and drip loss caused by slow freezing can hinder the loss of water-soluble components such as anthocyanins and pehnols, therefore the use of AFPs suggests aid to preserve nutritional content of frozen foods.

Pretreatments are used before various food processing operations to scale down severe conditions and to reduce unfavorable impacts of these conditions. Osmotic dehydration is principally used as a pretreatment to remove part of water from fruits or vegetables, by immersing the food material into a hypertonic solution for a specific duration of time. Li et al. (2017) studied the effects of osmotic pretreatment followed by cryogenic freezing on the quality characteristics and the freezing rate of tomatoes. Samples were immersed in a 55% (w/w) maltodextrin solution for 24 h at 35 °C, subsequently pretreated tomato cubes frozen in liquid nitrogen until centers reached −40 °C. Water loss and solid gain of samples during osmotic dehydration was recorded as 15.74 g water/g initial dry mass and 0.22 g solid/g initial dry mass, respectively. Pretreated samples showed a better final quality in regards of drip loss and water activity, also the mean freezing durations were significantly lower for pretreated samples, as, 3.13 min for treated and 4.48 min for untreated samples. Considering the bioactives; lycopene content of pretreated tomatoes was 1157.78 μg/g of dry mass, while that of untreated samples was 1122.46 μg/g of dry mass, as a result treated samples had better chromatic values and more vivid color as lycopene is attributed to favorable red color of tomatoes. Authors suggest that the

enzymatic activity, particularly peroxidase activity, may be reduced by osmotic pretreatment leading to improved preservation of lycopene and other labile molecules.

Another novel method suggested by Rubinsky et al. (2005) to reduce the adverse effects of frozen storage on biological materials, is isochoric freezing. In this method, food material is stored at a subzero temperature without ice formation taking place by keeping the material submerged in an isotonic solution inside a constant volume chamber. As a result, textural and structural damage is reduced and nutritional quality of food material is proposed to be retained. Bilbao-Sainz et al. (2021) studied the effects of isochoric freezing on the physical and chemical quality of grape tomatoes by storing samples at −2.5 °C in an isochoric chamber for 4 weeks and comparing the treatment with storage at −2.5 °C at atmospheric pressure (isobaric), cold storage at 10 °C and 85% RH, and IQF treatment followed by storage at −20 °C. Tomatoes stored at isochoric conditions preserved ascorbic acid capacity during 4 weeks, while isobaric stored samples had a slight decrease in 2 weeks but lost 90% of its ascorbic acid content after 4 weeks and IQF samples lost 17% content after 2 weeks. Total soluble phenolic content retained by isochoric samples showing an insignificant decrease by 12% compared to fresh samples (6.2 ± 0.7 mg mg gallic acid equivalent/g dry base), however isobaric, IQF and cold stored samples all lost considerable amounts being 18, 40 and 52%, respectively, after 4 weeks. Similarly, isochoric stored samples retained antioxidant activity, while all other treatments suffered activity losses in the ascending order of cold stored, IQF and isobaric stored.

Process parameters are highly important on the final quality of frozen foods. Determination of the freezing temperature and duration influence the eventual texture quality and bioactive composition of a freeze-thawed food material. Response surface methodology (RSM) is used to determine the optimal process parameters for various operations including freezing. In a study conducted to determine the ideal freezing parameters for pumpkins, researchers used RSM to analyze impacts of temperatures between −10 to −20 °C and durations between 5–9 h. Total phenolic content and flavonoid content for samples frozen at various temperature and durations were found as 8.31–15.84 mg gallic acid equivalent/g and, 5.85–11.28 mg quercetin equivalent/g, respectively. The highest level of total phenolic and flavonoid contents were achieved by freezing at −20 °C for 9 h, where 54.4% increase for phenolics and 60.4% increase for flavonoids compared to fresh pumpkin were reported (Kristianto et al. 2021).

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Chapter 5
Influence of Drying on Food Bioactives

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5.1 Introduction

The concept of functional food appeared at the end of the twentieth century, when the idea of a proper diet that provides sufficient nutrients to meet organic needs, i.e., carbohydrates, proteins, fats, vitamins, minerals, and essential fatty acids, all in adequate proportions. This scheme evolved into the wider concept of an optimal diet, which considers the potential of food to improve health and well-being in addition to its essential nutritive function. From this approach, bioactive compounds could be defined as substances other than nutrients, present in small amounts in food, with the capacity of acting on physiological mechanisms, lowering the risk of diseases and resulting in health benefits. There is a broader approach that considers as bioactive compounds those that, beyond having or not a nutritional function, exert a beneficial effect on health, as would be the case of vitamins. Since bioactives are found mostly in plant-based foods, often called phytochemicals. However, this term, associated to the fruits, vegetables and grains we consume, excludes a considerable range of bioactive compounds that can be provided by a varied diet, as essential fatty acids from fish, lutein from eggs, probiotics from milk, conjugated linoleic acid and peptides from meat and milk, antioxidant compounds from fungi and algae. A "consensus" encompassing all the precedent ideas was presented by Biesalski et al. (2009): "Bioactive compounds are essential and nonessential compounds (e.g., vitamins or polyphenols) that occur in nature, are part of the food chain, and can be shown to have an effect on human health".

Phytochemicals, in the restricted sense of bioactive compounds from plant origin, are secondary metabolites of plants and four main groups can be differentiated

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according to their chemical properties: sulphur compounds, nitrogenous compounds, terpenes (phytosterols and carotenoids) and phenolic compounds (Martínez-Navarrete et al. 2008; Liu 2013). Sulphur substances are typically related to the organoleptic properties of the cabbage family and also of vegetables of the *Allium* genus, as onion, garlic and leek (Poojary et al. 2017). Many of them, as ajoene and allicin, are bacteriostatic agents besides having anticarcinogenic and anti-inflammatory activities (Oommen et al. 2004; Dirsch et al. 1998). Nitrogenous compounds are mainly alkaloids, which usually present very high bioactivity even in low concentrations. Many of them have clinical uses, such as morphine, atropine and codeine, while others can be part of the diet, as caffeine or theobromine from cocoa. Some are toxins, such as the glycoalkaloid solanine from potatoes (Omayio et al. 2016). Even so, they all fit the definition of Biesalski et al. (2009) because they have a demonstrated effect on physiology, although this effect is not necessarily beneficial. Finally, terpenic and phenolic substances are the most representative bioactive compounds from vegetable origin for being widely distributed in plant-based foods. These compounds have a main function as antioxidants (Shahidi and Ambigaipalan 2018) and were the most studied bioactives over the last three decades (Cacace and Mazza 2007).

In this chapter, we will focus on carotenoids and phenolic compounds as affected by drying processes, and will also revise the case of some vitamins found in vegetables (tocols and ascorbic acid), as they have the double function of essential nutrients and bioactive compounds with antioxidant activity, and are considerably affected by drying processes (Dewanto et al. 2002).

The selection of a drying method is not simple and cannot be aimed only to protect the major bioactives in the food. Both the organoleptic and nutritional characteristics must be considered to obtain a high-quality product. The decision will also depend on the product to be dried, the production stipulated and the existing facilities, among other issues not related to the bioactives retention. So, as one thinks on the effects of drying on food bioactives, we should compare the conventional or current methods (mostly convective, heated air drying) with alternative or innovative techniques which often involve different heat and mass transfer mechanisms (Orsat and Raghavan 2007). Besides, the diversity of food matrices must be considered, as well as other factors that can act and/or interact on bioactives retention, such as processing time, temperature, water content, or oxygen partial pressure. Beyond this already complex picture, comparison of experimental results implies to pay due attention to the extraction method used to determine the quantity of bioactive compounds before and after drying, to consider the extractability from the food matrix (which may change along the process) and the polarity of the solvents used (e. g., total antioxidant activity may be measured in hydrophilic or lipophilic extracts). Moreover, besides the decrease, increase, or constancy of the phytochemicals content, a processed food may experience a decrease, increase, or no change in functionality (Wang 2007). The growing interest in this idea, related to the concept of bioavailability and bioaccessibility is reflected in assays *in vitro* and *in vivo* developed to complete the study of functional foods (Parada and Aguilera 2007).

Dried products and, in general, processed foods, are considered not as good as the fresh or natural food, because the consumers associate the drying process with a decrease in nutritional quality. However, this is not always true and researchers have the responsibility of clarifying the subject, by reviewing and carefully choosing the methodologies for experimental work. The present review has the purpose of finding general tendencies to describe how the more ubiquitous food bioactives (carotenoids, phenolics, ascorbic acid and tocols) are affected by drying processes, identifying the more relevant factors acting on each group of bioactive compounds. The revision of the literature in this field could help to promote more detailed research, resulting in optimized drying processes and better quality dried products.

5.2 Main Bioactives as Affected by Drying Processes

Numerous experimental studies have been developed with the aim of evaluating the nutritional retention during drying of different food matrices. Table 5.1 shows some works focused on widely consumed foods containing bioactive compounds.

5.2.1 Carotenoids

Carotenoids are the most widespread and important group of pigments in nature, owing to their colour and antioxidant activity, being the last property due to the unique structure an extended system of conjugated double bonds. Their singlet oxygen quenching properties and their ability to trap peroxyl radicals are the main mechanism responsible for the beneficial effects on health. Their basic structure is a symmetrical tetraterpene skeleton, formed by tail-to-tail condensation of two C20 units. In many carotenoids the end-groups are modified into five- or six-membered rings giving monocyclic or dicyclic compounds. They easily undergo oxidation in the presence of air and light. Based on their composition, carotenoids are divided into two classes: carotenes, which are hydrocarbons, such as lycopene (acyclic) and β-, α- and λ-carotene (bicyclic) and xanthophylls, containing an oxygenated functional group, as lutein and criptoxantin.

5.2.1.1 β-Carotene

β-carotene is the most abundant compound of the so-called carotenoids family and also the most important regarding pro-vitamin A activity, representing around 50% of the active vitamin assembled in the human body. However, this molecule is also considerably unstable and degrades easily with light and heat. Therefore, it constitutes an important quality parameter to be measured during the drying process (Lin

Table 5.1 Studies on nutritional retention during drying for selected foods

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Carrot	– Pre-treatments: (i) Blanching in hot water: 70 °C –20 min; 100 °C - 3 min; 80 °C –20 min. (ii) Glycerol, $CaCl_2$, Na metabisulfite, L-cysteine HCl, N-acetyl-L-cysteine – Hot air drying: 50, 60 y 70 °C	Ascorbic acid, carotenoids content	– Titration – Spectrophotometer	– L-cysteine HCl preserved highest content of ascorbic acid plus a thermal blanching at high temperature and short time. – Ascorbic acid was mainly affected by longer drying times – The oxidation of carotenoids could be retarded by Na metabisulphite but not cysteine – Carotenoids were sensitive to drying temperature more than to drying time.	Mohamed and Hussein (1994)
Potato	– Hot air drying: 40, 50 y 60 °C – Relative humidity: 4.5 and 30% – Initial moisture content: 2.3 and 4.6 kg water/kg dry matter	Ascorbic acid	Numerical simulation for evaluating the ascorbic acid degradation	– Nutrient loss increased with increase in drying air temperature and relative humidity, and decrease in initial moisture content	Rovedo and Viollaz (1998)
Carrot	– Vacuum microwave drying: 0.3–0.45 kW, pressure 100 mmHg. – Hot air dying: 70 °C – Freeze-drying: 1.6 mm Hg, chamber temperature, 20 °C – Blanching process: 90 °C – 7 min	Vitamin C α-carotene β-carotene	– Microfuorometric method – HPLC	– A substantial loss of vitamin C and α-carotene occurred during the blanching process. – Retention of vitamin C was 38% with air drying and 79% with microwave-vacuum. – Total losses of carotenoids during drying were of 19.2% for the hot air samples and 3.2% for the vacuum-microwave samples. – No loss was detected in freeze-dried samples for vitamin or carotenoids content.	Lin et al. (1998)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Paprika, carrot and potatoes	– Steam blanching: 100 °C – 3 min – Hot air/N_2: 60 °C, air/gas velocity 1.0 m/s, relative humidity 10%	Vitamin C Carotenoids	HPLC	– The use of inert gas improved the retention of both nutrients in all vegetables. – The blanching treatment contributed to improve carotenoids retention.	Ramesh et al. (1999)
Tomato	– Hot air drying: 95 °C, 6–10 h – Vacuum drying: 55 °C, 4–8 h – Combined method: *Osmotic treatment*: 65°Brix, 25 °C, 4 h + *vacuum drying*: 55 °C, 4–8 h.	Lycopene	HPLC	– The drying methods considered presented a high retention of lycopene, above 96%. – The observed loss of color was attributed to the change from *trans* lycopene to *cis* isomer. – The osmotic-vacuum treatment had less effect on isomerization than vacuum-drying and conventional air-drying.	Shi et al. (1999)
Tomato pulp	– Thermal treatment 88 °C: 2, 15 and 30 min	Lycopene Ascorbic acid Total phenolics Flavonoid content Antioxidant capacity	HPLC Titration Folin Spectrophotometer Tosc	– Thermal processing considerably increase the content of available lycopene from 54 to 162% as time increases from 2 to 30 min. – The loss of vitamin C was between 10 to 29% as a function of processing time. – Thermal processing have not an effect on total phenolics and flavonoids content. – Antioxidant capacity increases in 28, 34 and 62% at process times of 2, 15 and 30 min, respectively.	Dewanto et al. (2002)

(continued)

Table 5.1 (continued)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Rosehip	– Hot air drying, 50–80 °C, air velocity 1.67 m/s – Mixtures with different: O_2-CO_2 ratios – Whole fruit and fruit cut into pieces	Vitamin C	Spectrophotometer	– Higher temperatures decreases retention – Cut fruit increases loss of vitamin C. – Degradation is reduced by using inert gas.	Erenturk et al. (2005)
Carrot	– Low-pressure superheated steam drying (LPSSD): 60–80 °C, 7 kPa – Hot air drying (AD): 60–80 °C, air velocity 0.8 m/s – Vacuum-drying (VD): 60–80 °C	β-carotene	HPLC	– As temperature increases the degradation on β -carotene was higher. – LPSSD leads to a high retention of this nutrient, around 74%. – AD and VD produce the greatest losses in this nutrient, 56 and 50% respectively.	Suvarnakuta et al. (2005)
Tomato pulp	– Spray drying. (i) Inlet air temperatures: 110–140 °C (ii) Air flow rates: 17.5, 19.3, 21 and 22.8 m3/h (iii) Atomizing agent flow: 500–800 l/h	Lycopene	Spectrophotometer	– Lycopene loss ranged between 8.1% and 21% as the inlet air temperature increases. – A decreased compressed air flow rate resulted in decreased lycopene loss. – Lycopene loss also increased with increased drying air flow rate – The decrease in lycopene content during spray drying could be attributed to a progressive conversion from trans to cis form.	Goula and Adamopoulos (2005)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Tomato pulp	– Tomato pulp was concentrated at 90 °C to moisture contents of 95, 85, 65, 55, 50, 45, and 35% (*w.b.*) – Heating tomato pulp: 50–90 °C	Lycopene	Spectrophotometer	– Lycopene content in tomato pulp with different moisture contents decreases during heating for all temperatures. – The lycopene degradation depends on product moisture content, in addition to temperature. – The lowest loss of this bioactive compound is reached with initial moisture contents between 50 at 55% (w.b.)	Goula et al. (2006)
Paprika	– Convective drying: 25–100 °C – Storage process: 4–6 °C, 3 months. – Two varieties of the vegetable	Carotenoids	HPLC	– The total carotenoid content decreased in both varieties as temperature increased. – 16 carotenoids were identified: 5 unesterified, 7 mono-esters and 4 di-esters. – Carotenoids showed diverse thermal stability being β-carotene the most stable	Daood et al. (2006)
Apricot	Hot air drying, 50–80 °C, air velocity 1.0 m/s – Sun drying: max 38 °C – min 29 °C – Pre-treatment: sulphuring	β-carotene	HPLC	– Sulphuring did not affect β-carotene content. – Retention of this bioactive compound was higher as temperature increases – To avoid undesirable colour changes, the most appropriate temperature was 70 °C, leading to nutrient retention above 60%.	Karabulut et al. (2007)

(continued)

Table 5.1 (continued)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Tomato	– Hot air drying: 40, 60 and 120 °C, 1.5 m/s – Six cultivars from varieties RCG and YCG.	Lycopene Total phenolics and flavonoids	HPLC Folin Colorimetric	– Lycopene contents were affected by tomato cultivars, drying temperatures, and time. – For both cultivars, total phenols and flavonoids decrease for longer times. This drop is more noticeable at high temperatures. – Lycopene content on RCG cultivar drop greatly after 75 min in all temperatures. In contrast, lycopene increase in the YCG cultivars was independent of time for all temperatures except 120 °C.	Chang and Liu (2007)
Strawberries	– Convective drying: 70 °C; air velocity 1 m/s – Microwave-vacuum: 240 W, 360 W, 480 W; 4–6 kPa – Vacuum drying: 50 °C, 100 Pa – Freeze-drying: 65 Pa, shelf temperature 30 °C	Ascorbic acid Polyphenols Antioxidant capacity	HPLC DPPH FRAP ABTS	– Convective and vacuum drying methods promote nutrient losses between 40 and 60%. – Convective drying increases the antioxidant capacity probably due to Maillard products. – Freeze-drying (FD) retain a 98% of nutrients. – Microwave-vacuum with 240 W and 6 kPa retain higher levels of polyphenols and vitamin C, with the advantage of reducing processing time compared to FD.	Wojdylo et al. (2009)
Kiwifruit	– Hot air drying, 35–65 °C, – Air velocities 0.3, 0.6 and 0.9 m/s – Relative humidity 40, 55, 70 and 85%.	Vitamin C	Titration	– Increasing the air temperature and decreasing the relative humidity lowers retention of vitamin C.	Kaya et al. (2010)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Carrots	– Hot air drying, 50–80 °C, air velocity 2.0 m/s – Slices thickness: 3, 6 and 9 mm	β-carotene	Spectrophotometer	– β-carotene retention was higher as sample thickness increased. – The β-carotene content decrease from 79 to 71% as temperature rises from 50 to 80 °C	Goula and Adamopoulos (2010)
Quinoa	– Convective drying: 40–80 °C; air velocity 2.0 m/s	Total phenolic Vitamin E Antioxidant capacity	Folin HPLC DPPH	– A clear reduction in total phenolic content was found for increasing temperature. – An important increase of Vitamin E, above 100%, was observed, at 70 and 80 °C. – The antioxidant capacity presented similar values as temperature increased from 40 to 80 °C.	Miranda et al. (2010)
Latvian cranberries	– Convective drying, 50 °C, air velocity 1.2 m/s – Microwave-vacuum: 640 W, 70–56 mm Hg – Pre-treatments: *(i) perforation and halving* *(ii) steam-blanching, 94 °C*	Vitamin C	HPLC	– Pre-treatment method influenced vitamin C decrease during drying. – The steam-blanching method contributed to a higher retention – Vacuum-microwave drying method had better performance as regards Vitamin C retention	Dorofejeva et al. (2011)

(continued)

Table 5.1 (continued)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Apricot	– Hot air drying, 60–100 °C, air velocity 0.2 m/s – Two varieties	β-carotene Antioxidant capacity	HPLC ORAC	– β-carotene contents of both varieties increased between 45 to 59% with temperature at 80 and 100 °C, respectively. – The antioxidant capacity increased considerably, above 90%, at 100 °C. – The samples dried at 60 °C (longer drying times) presented a considerable reduction in vitamin C and hydrophilic ORAC compounds.	Ihns et al. (2011)
Starfruit, Mango, Papaya, Muskmelon and Watermelon	– Freeze-drying	Total phenolic Ascorbic acid β-carotene Antioxidan capacity	Folin HPLC DPPH FRAP	– A higher retention, above 95%, on ascorbic acid and β-carotene was reported in all fruits. – Total phenols content decreased slightly in some fruits after the process. – A small increase in antioxidant capacity was found in the freeze-dried samples. – Phenolics are the dominant compounds contributing towards the antioxidant activity of the fruits tested.	Shofian et al. (2011)
Tomato fruits and juice	– Heating at 95 °C by 3 min (juice) – Air drying: 45 °C, 38 h (fruits)	Lycopene Carotenoids	Spectrophotometer	– A positive effect was obtained for lycopene content in both thermal treatments. – Tomato juice presented an increase in lycopene in order to 51% while the dried fruit has a value of 78% in lycopene. – A significant increased content of carotenoids after heat treatment of the fruit at using temperatures of 45 and 95 °C.	Mendelová et al. (2013)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Apricot	– Hot air convective: 60 and 70 °C, air velocity 2.3 m/s – Microwave oven: nominal power output of 2 kW, at a frequency of 2.4 GHz	Carotenoids	HPLC	– Seven carotenoids were identified: antheraxanthin, lutein, zeaxanthin, β-cryptoxanthin, 13-cis-β-carotene, all-trans-β-carotene and 9-cis-β-carotene. – The microwave drying caused a decrease up to 50% in all carotenoid compounds compared to the convective method which presented a reduction between 30 to 50%. – Antheraxanthin was the most sensible carotenoid to drying conditions in both methods and could be used as a process marker for the evaluation of thermal damage.	Fratianni et al. (2013)
Button Mushroom	– Freeze-drying (FD): 108 Pa, Shelf Temperature 40 °C – Freeze-drying + convective drying (FD + CD): 40 °C – Freeze-drying + vacuum drying (FD + VD): 90 kPa – Freeze-drying + microwave-vacuum drying (FD + MVD): 60 W/g power density, 90 kPa	Ascorbic acid	Kit	– Compared with the value obtained from FD samples FD + VD presented the higher nutrient retention, up to 85% followed by the FD + CD with 73%. – FD + MVD, at the declared power density, lead to a marked decrease of 40% in vitamin C content	Pei et al. (2014)

(continued)

Table 5.1 (continued)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Sour Cherries	– Convective drying: 50, 60 and 70 °C; air velocity 1 m/s – Microwave-vacuum: 240 W, 360 W, 480 W; 4–6 kPa – Freeze-drying: 65 Pa, shelf temperature 26 °C	Polyphenol compounds Antioxidant capacity	UPLC ABTS FRAP	– The Freeze-drying presented a retention in polyphenol compounds of up to 90%. – Convective and Microwave-vacuum produce large losses in these compounds being the air treatment the most severe. – In order to reduce energy consumption and process times, the optimum drying conditions were established for the conventional drying at temperature at 50 °C and for the microwave drying at 120 W, as declared by the authors.	Wojdylo et al. (2014)
African eggplant	– Convective drying: 50–70 °C, air velocity 2 m/s. – Vacuum-drying: 50–70 °C, Pressure 60 mbar – Freeze-drying: 0.055 mbar – Solar drying – Five varieties of eggplant	β-carotene Total phenols Lycopene	Spectrophotometer HPLC	– The highest retention rate was observed in freeze-dried samples. Overall, 36.3–95.1% (total phenolics) and 31.4–99.2% (β-carotene) were retained during FD. – Convective, vacuum and solar drying presented a loss in β-carotene among 46 and 79% while the total phenols was affected between 20 to 37%. – Lycopene was only detected in one variety and with low concentration.	Mbondo et al. (2018)

Food	Drying method and conditions	Studied bioactives	Method of quantification	Observations	Reference
Hazelnuts	– Convective drying: 38, 43 and 59 °C; air velocity 1.0 m/s – Relative humidity: 40 and 60% – Three Hazelnuts varieties	Vitamin E Total phenolic Antioxidant capacity	HPLC	– The degradation of vitamin E was low ($\leq$ 3%) for all the drying conditions and cultivars. – The total phenolic and antioxidant capacity were negatively affected by the increase in temperature. – Low temperature and relative humidity promoted a higher bioactive retention.	Wang et al. (2018)
Orange puree	Freeze-drying – Freezing rate: slow – fast – Shelf temperature: 30 °C, 40 °C, 50 °C – Absolute pressure: 5 Pa – 100 Pa	Ascorbic acid Total polyphenolic β-carotene	HPLC DPPH FRAP	– Vitamin C was preserved in almost 98% at higher temperaturess 40 and 50 °C and at the lower pressure (5 Pa). – To maintain a high retention of β-carotene ($\approx$ 65%), the shelf temperature of 30 or 40 °C at lower pressures (5 Pa) presented the better performance. – Total phenols was not affected by pressure and had a weak dependence with temperature in the 30 to 50 °C experimental range – Freezing rate did not present a significant effect on the variables studied.	Silva-Espinoza et al. (2020)

et al. 1998; Suvarnakuta et al. 2005; Karabulut et al. 2007; Goula and Adamopoulos 2010).

Some publications as those of Karabulut et al. (2007) and Ihns et al. (2011) have evaluated the effect of convective drying carried out in a wide range of air temperatures, 50–100 °C, on the β-carotene content in apricots. Both authors reported losses up to 60% at low temperatures around 50 °C while, at high temperatures, above 70 °C, losses were reduced to 35%. β-carotene is degraded by a free radical oxidation mechanism and the extent of degradation depends on process time, heating temperature, and presence of oxygen. At low temperatures, a longer drying time is required therefore the oxidation in this molecule takes place to a greater extent. A similar behaviour was reported by Goula and Adamopoulos (2010) in carrots, showing this compound as relatively heat-stable at higher temperatures. Furthermore, this effect is maximized in carrots by the presence of α-tocopherol, which develops a function as a natural antioxidant against the oxygen. However, similar studies carried out by Suvarnakuta et al. (2005) in carrot found a loss in this bioactive compound in the range of 50 to 55% with vacuum (VD) and hot air drying (AD), respectively. They proposed a low-pressure superheated steam method as a novel process, in which the presence of oxygen is minimal. In this alternative method, their results showed a retention of 76% in the β-carotene content. Other drying technologies such as the vacuum-microwaves (VM) combination were also studied. Lin et al. (1998) compared the effects of the application of this VM method with those obtained by the convective drying (CD) in carrots. The results achieved showed that the application of the combined method led to an increase in the nutrient retention to 97%, compared to 81% obtained by the CD.

In recent years the freeze-drying (FD) method has been gaining ground in food drying with the purpose of improving the nutritional and organoleptic characteristics of foods (Silva-Espinoza et al. 2020). In this context, Mbondo et al. (2018) compared the effect of four drying methods as convective drying (CD), vacuum-drying (VD), freeze-drying (FD) and sun-drying (SD) on the retention of β-carotene for African eggplant cultivars. The highest retention rate, of 76%, was observed in freeze-dried samples with. Regarding the other methods, retention was in the range of 57 to 22%, being CD the most detrimental treatment; these results are in agreement with those reported earlier by other authors such as Wojdylo et al. (2009) and Pei et al. (2014) who studied the effects of FD on the retention of various bioactive compounds in different food matrices.

5.2.1.2 Lycopene

Lycopene is one of over 600 carotenoids found in nature, being the main pigment found in tomatoes, representing about 83% (w/w) of total pigments, and is responsible for the characteristic deep-red colour of ripe tomato fruits and tomato products. This vegetable and its derivatives are the major source of lycopene and, therefore, are considered to be an important contributor of carotenoids to the human diet (Dewanto et al. 2002; Chang and Liu 2007). Lycopene is also found in rosehip,

watermelon, guava and pink grapefruit, and it exists in nature in the all-*trans* form. In the conventional processing of tomatoes, a conversion from the *trans* to the *cis* form can take place. These changes are mainly attributed to the heat stress imposed by thermal processes. From a nutritional point of view, this isomerization is desirable since *cis*-lycopene is more bioavailable for humans (Moran et al. 2015; Cooperstone et al. 2016). However, authors as Shi et al. (2008) and Mendelová et al. (2013) have suggested that the *cis*-isomer is thermodynamically more unstable and susceptible to oxidation by light. In processed tomato products, reisomerization can occur during storage to the more stable all-*trans* form. For that reason, strategies to protect and stabilize *cis*-lycopene in processed products must be developed. In fact, exposure to oxygen and light, heat treatment, state and composition of the food matrices are factors that were shown to have marked effect on isomerization and oxidation (Shi et al. 1999; Sablani 2006; Mendelová et al. 2013).

The effect of different heat treatments on the content of lycopene in tomato has been studied by several authors as Chang and Liu (2007) who evaluated the content of this bioactive in some cultivars of tomato during hot air drying carried out between 40 and 120 °C and have determined an increase of lycopene concentration under all drying conditions. This was attributed to damage to the cellular structure, which makes lycopene more accessible to the extraction treatment. Similar results were found by Dewanto et al. (2002) and Mendelová et al. (2013) who evaluated the effect of heating treatment (88–95 °C) on tomato products, as pulp and juice, respectively. The authors suggested that other processes as cutting or grinding can also improve lycopene extraction up to 80%, by breaking down cell walls and weakening the bonding forces between lycopene and tissue fibres. However, an opposing trend was reported by Goula and Adamapoulos (2005) and Goula et al. (2006) in thermal processing of tomato pulp by spray drying (110–140 °C) and heating (50–90 °C), respectively. These authors found a decrease in lycopene content, in the range of 8–21%, as temperature rises. A possible explanation for this was the oxidative degradation of lycopene into a colourless form that may occur during drying. Regarding the lycopene isomerization, a detailed study was carried out by Shi et al. (1999) during tomato drying by different methods such as convective drying, vacuum and a combined methodology by osmotic pre-treatment and vacuum-drying (OVD). In this work, the authors have found a moderate extent of conversion of 16% from *trans* to *cis* form, which takes place in all treatments. The latter is important because, in tomatoes, and as mentioned above, the major content corresponds to *trans* lycopene, which is the most stable isomer. Then, a substantial degree of isomerization to the *cis* form can promote oxidation in this molecule. On the other hand, osmotic treatment was beneficial for preserving the lycopene, whose content remained stable with a retention of 98% of its initial value. The authors proposed that the sugar solution remaining on the surface of samples prevents the oxygen from penetrating and oxidizing lycopene. On the other hand, while lycopene is preserved, the osmotic treatment poses the problem of leaching water soluble vitamins and minerals towards the solution. With respect to the other drying methods, hot-air and vacuum drying, both decreases slightly the lycopene content, with a retention of 96%.

A recent study developed by Tan et al. (2021) who compared the effects of freeze-drying (FD) and convective drying on lycopene content of three cultivars of tomatoes, found striking results. Their work showed that the FD samples in all cases presented a significant decrease in the lycopene content in contrast with the oven drying tomatoes. The authors attributed the results to a possible oxidation, taking place due to the longer process time, even at lower oxygen concentration. However, this is an aspect that still needs to be investigated in depth, considering other factors as light or perhaps that the structure is preserved during freeze-drying thus lowering the extraction efficiency. An interesting remark was reported by Bilek et al. (2019) who investigated the effect of freezing rate on lycopene content in cherry tomato. These authors suggested that the mechanical damage caused by the ice-formation during freezing may disintegrate the membranes, facilitating their oxidative degradation during subsequent treatments. Finally, another aspect that requires more detailed research is lycopene isomerization, to identify the factors affecting the equilibrium between isomeric forms (Shi et al. 2007).

5.2.1.3 Xanthophylls

Xanthophylls are chemical compounds similar to carotenes, with the difference that, in addition to containing carbon and hydrogen, they have one or more oxygen atoms within the molecule. However, as well as the carotenes, xanthophylls have impressive colours in the range of red, orange and yellow. These substances are generally part of a balanced diet since they are commonly consumed by humans in fruits and vegetables, such as apple, apricot, mandarin, mango, papaya, red and chilli pepper, potato or squash (Demming-Adams 1990; Daood et al. 2006; Bunea et al. 2014). In fruits and vegetables, the xanthophylls are present either in a free, unesterified form, or as fatty acid esters, having both the same antioxidant capacity but being the esterified xanthophylls more efficiently absorbed into the human body (Mínguez-Mosquera et al. 1993: Maiani et al. 2008; Bunea et al. 2014).

Fratianni et al. (2013) studied the degradation of carotenoids in apricot during microwave drying and convective hot-air drying (60–70 °C). Within this large family of compounds, the authors identified and evaluated the retention of some members of the xanthophylls, as antheraxanthin, lutein, zeaxanthin, and β-cryptoxanthin. They found that the microwave method caused a higher, up to 50%, decrease in all carotenoid compounds if compared to the convective method, for which the reduction was between 30 to 50%. Under both thermal treatments, antheraxanthin was the most sensitive to drying conditions, so that it might be used as a process marker for the evaluation of thermal damage in apricot. A similar analysis was carried out by Daood et al. (2006) in two varieties of red pepper, considering a wider range of temperatures, between 25 and 100 °C. Under such conditions the total carotenoid content decreased in both varieties as a result of temperature rise, although some molecules showed different thermal stability with zeaxanthin and cryptoxanthin being observed as the most stable compounds. In turn, Mínguez-Mosquera et al. (1993) studied in detail the effect of thermal processing on xanthophylls content for

some varieties of paprika and suggested that the main factors affecting degradation are heat-induced degradation and accelerated oxidation in presence of molecular oxygen, while the effect of the possible isomerization in other structures was considered negligible.

5.2.2 Phenolic Compounds

Phenolics are compounds presenting one or more aromatic rings with one or more hydroxyl groups, and are generally categorized as phenolic acids, flavonoids, stilbenes, coumarins, tannins, isoflavones and lignans. The flavonoids are further classified as flavonols, flavones, flavanols (catechins), flavanones, and anthocyanins. These compounds, generally named as polyphenols, are usually part of a complex structure or are present in bound forms. They are found in many fruits including cherries, apples, strawberries and mangoes. Polyphenols can act as antioxidants due to their property of radical scavengers or chain breakers, depending on their chemical structure (McSweeney and Seetharaman 2013). There are many health benefits derived from eating foods that contain these bioactive compounds: numerous polyphenol-rich vegetables and fruits are effective in protecting against some diseases as colon cancer and cardiovascular diseases (CVD), or in decreasing the risk of neurodegenerative disorders (Vanzour et al. 2010; Ramírez et al. 2015).

As phenolic compounds are the most ubiquitous and studied bioactives, many works can be found in the literature about their retention after different drying methods. Authors as Del Caro et al. (2004) have investigated the effect of temperature during the hot air drying of prunes and observed that polyphenols content was higher in samples dried at temperatures above 70 °C, than in those treated at 60 °C. These authors suggested that the lower contents may be due to higher residual activity of polyphenol oxidase (PPO). In fact, it is possible that PPO activity be still high at 55 °C and decrease as the temperature approaches 70 °C. A similar result was reported by Gallegos-Infante et al. (2009) who found a reduction of up to 70% on flavonols and total phenols in nopal samples dehydrated at 45 °C. In addition, they concluded that an increase in the air flow rate, from 3 to 5 m/s, increases the loss by approximately 1.3 times. In this case, the effect of time (lower drying time achieved by the higher air flow rate) would not be adequate to explain the lower retention. Possibly, another factors related with the higher airflow (and thus oxygen) should considered. On the other hand, the increase of phenols in samples dehydrated at high temperatures was the object of study by other authors such as Kerkhofs et al. (2005) and Chang et al. (2006) in different tomato cultivars. In both publications, the authors proposed that phenolic compounds (usually stored in the cell vacuoles), are released after the breakdown of the cellular structure, induced by hot air drying. The disruption of cell walls will also release the enzymes that can degrade polyphenols, though the high temperature is able to inactivate these enzymes or slow their activity down. Regarding anthocyanins, a short-time, high-temperature process was recommended for higher retention. For instance, in red fruit juices

heated for 12 min at 100 °C, anthocyanin losses appear to be negligible (Wilska-Jeszka 2007). However, this concept is difficult to apply to a usually long process as drying.

Other method with good ability to preserve this type of compounds is vacuum freeze-drying. Michalczyk et al. (2009), De Torres et al. (2010) and Valadez-Carmona et al. (2017) compared the effect of air-drying and freeze-drying on the content of polyphenols in different foods. These authors concluded that freeze-drying is the most convenient technique to preserve these bioactives, since low temperatures and the practical absence of oxygen allow them to remain unchanged. These advantages attracted the attention of the food and pharmaceutical industry to encapsulate polyphenols and incorporate them in functional foods for human diet. However, despite the benefits offered by freeze drying, the long process times (24–48 h) usually pose restrictions when considering energy consumption. This is another feature that must still be studied in order to optimize process conditions (Fang and Bhandari 2010; Ramírez et al. 2015). In this sense, Mejia-Meza et al. (2008) applied a combined method in blueberries, consisting in a hot air pre-drying from 87.2 to 40% moisture content (99 °C for 45 min), followed by freezing and ending with microwave-vacuum drying of the frozen sample (71 °C for 60 min). Hence, these authors combined a high-temperature, process with some advantages of freeze drying (vacuum conditions and low sample temperature) in a relatively short time. Results were promising, being the retention of anthocyanins and total polyphenols higher than that of conventional hot air drying, and comparable with that of freeze drying for aglycone forms.

5.2.3 Ascorbic Acid (Vitamin C)

Ascorbic acid is an essential substance found mainly in fruits and vegetables. It has four isomers, but only the L-ascorbic acid has physiological activity as vitamin C. The product of ascorbic acid oxidation, dehydroascorbic acid, still has vitamin C activity although it is less stable. This oxidation can be reversed by other antioxidants in the matrix, but further degradation of dehydroascorbic acid is irreversible and leads to the total loss of bioactivity. This nutrient not only prevents diseases like scurvy but is also regarded as the most important water-soluble antioxidant in humans, working as a free-radical scavenger, along with vitamin E. In aqueous solutions or in foods, its stability is related to the storage conditions, processing parameters and to the composition of the matrix. The vitamin C can be easily degraded, depending on several variables such as pH, temperature, light, and presence of enzymes, oxygen, and metallic catalysers. Thus, numerous studies on food processes utilise vitamin C as a quality indicator (Lin et al. 1998; Ramesh et al. 1999; Erenturk et al. 2005; Santos and Silva 2008; Pei et al. 2014; Silva-Espinoza et al. 2020; Demarchi and Giner 2020).

Researches carried out on convective hot air drying agree that the presence of oxygen and high temperatures considerably affect the loss of vitamin C and, even to

a higher extent, for cut or grinded products as more surface area is exposed to the drying medium (Mohamed and Hussein 1994; Erenturk et al. 2005; Wojdylo et al. 2009). However, Rovedo and Viollaz (1998) and Kaya et al. (2010) have suggested that this deteriorative effect may be hindered with increasing relative humidity of the drying air. These authors found positive effects on the retention of this nutrient with a relative humidity in air above 30% which, in heated air drying, is a high value. Meanwhile, Dorofejeva et al. (2011) compared the effect of convective drying (CD) and microwave-vacuum (MV) method on the content of vitamin C in some berries. The results showed that the MV affected the vitamin content to a lesser extent in comparison with the large losses originated by the hot air drying, even when the latter was carried out at a moderate temperature of 50 °C. In this sense, the authors concluded that the time of exposure to oxygen is one of the most important variables that impact degradation of this compound, since in MV drying the process time was 82 min while the CD required 55 h to reach the same final moisture content. Other works, such as those of Lin et al. (1998) and Wojdylo et al. (2009), extended their analysis by comparing three drying methodologies as microwave-vacuum (MV), convective drying (CD) and freeze-drying (FD) and their effect on vegetables and fruits such as carrots and strawberries, respectively. Both authors agree that longer process times associated with the CD contributes to a severe loss of this compound. Regarding the other methods, the residual ascorbic acid in FD was 98% of the initial value while the MV drying retention was in the range of 79–87%. A possible explanation for these losses is that during the vacuum step a minimal amount of oxygen can result in a certain degree of oxidation of ascorbic acid. However, other authors such as Pei et al. (2014) who studied the effect of microwave-vacuum drying on retention of vitamin C in button mushrooms, proposed another explanation for the losses that occur with this technique: the instantaneous high temperature produced by the microwaves in a few seconds is the main cause for the destruction of the thermally sensitive nutrients such as vitamin C. Even so, losses are lower than for CD.

In general, freeze-drying has been the subject of numerous studies with favourable results in comparison with other methodologies, showing a better performance for the retention of bioactive components or other nutritive substances (Lin et al. 1998; Wojdylo et al. 2009; Pei et al. 2014). On this topic, Silva-Espinoza et al. (2020) carried out a more detailed study to evaluate the impact of FD conditions on some nutrients, such as vitamin C for orange pulp puree. The authors concluded that the retention of ascorbic acid was mainly determined by the shelf temperature and pressure. Temperatures of 40 or 50 °C led to samples with higher ascorbic acid retention than those freeze dried at 30 °C. In this last condition, the prolonged process time and exposure to a minimum amount of oxygen may have been the main degradation factors. Regarding the pressure of the drying chamber, the authors observed that retention was higher than 98% for process pressures of 5 Pa compared to those obtained at 10 Pa, considering in both cases a freeze-drying time of 25 h. This means that, in order to preserve the nutrients in the best possible way, the presence of oxygen should be minimized. On the basis of the reasons exposed above,

ascorbic acid experiences an important extent of degradation during long processes, even at low temperatures and low oxygen partial pressure.

5.2.4 Vitamin E

Both tocopherols and tocotrienols are generally known as vitamin E and are present in almost all naturally occurring plant oils, although tocotrienols are specific for some oils from palm or amaranth (Stołyhwo 2007). The highest concentration of tocopherols is found in the germ of seeds. Alpha-tocopherol is the most active form of vitamin E in humans, acting as a powerful antioxidant, protecting cell membrane lipids from destruction by free radicals, as well as lipids in LDLs from oxidation (Miranda et al. 2010; Wang et al. 2018). Plants in their growing phase usually contain α-tocopherol. The content of this molecule varies among the species, variety, stage of maturity, season, processing technologies and storage time (André et al. 2010). The α-tocopherols as well as the rest of the members of the tocopherols family, are unstable in the presence of oxygen, ultraviolet light, metal ions, and can be adversely affected by drying treatments (Kanner et al. 1979; André et al. 2010; Wang et al. 2018).

The influence of air drying at moderate temperatures (between 38 and 49 °C) and relative humidity between 40 and 60% on bioactive compounds of hazelnut were investigated by Wang et al. (2018). These authors reported a minimal degradation on the vitamin E (α-tocopherol), with retention values above 97%. Besides, the results showed a protective effect of this compound on the fatty acids contained in hazelnut against oxidation during heat treatments. On the other hand, a similar study was carried out by Miranda et al. (2010) who evaluated the effect of temperature during convective drying of quinoa seeds. The authors found an important increase in vitamin E, above 100%, as temperature increases from 40 to 80 °C. In this context, the authors suggested that a considerable amount of vitamin E linked to proteins or phospholipids could be released by the heat-induced structure breakdown, depending on the food matrix. Thus, a higher content of this molecule can be obtained compared to the non-processed food. This increasing availability of bioactive substances due to heat treatments has been reported by other authors in tomatoes and tomato products (Goula and Adamopoulos 2005; Goula et al. 2006; Mendelová et al. 2013). Vinson et al. (2005) studied the content of different bioactive molecules and antioxidant properties in various tropical fruits in fresh and dehydrated states. They concluded that, although thermal treatments may decrease the content of total phenols, they still have a positive effect by increasing the bioavailability of vitamin E and the total antioxidant capacity. On these grounds, the lipophilic characteristics of tocopherols and their strategic location in the cell structure seem to protect them from degradative reactions during drying processes. While heat application is supposed to increase extractability with no impact on vitamin E activity (because this effect is assumed as positive), the increase in extractability may occur along with some degradation reactions. On the other hand, the

vitamin E content informed for fresh samples is usually lower than for processed products (on a dry mass basis), indicating that extraction procedures are unable to recover the total amount of vitamin E from fresh samples. More research is needed to throw light on such issues.

5.3 Principal Factors Affecting Bioactives During Drying

5.3.1 Temperature

From a kinetic perspective, activation energy (E_a) is a useful and widely used parameter to evaluate the effect of temperature on the rate of different reactions. This parameter can be seen as the energy barrier that molecules need to overcome in order to be able to react (Sablani 2006), being indicative of the degree of dependence of kinetics of the phenomenon with the temperature. Several authors have applied mathematical models for a better comprehension of the process (see Table 5.2). For a given drying method, E_a values both for the drying kinetics and the bioactive degradation kinetics can be estimated and compared. In the literature, some authors reported E_a values for the degradation kinetics of different bioactive compounds during drying: Goula and Adamopoulos (2010), 32.1 kJ/mol for β-carotene in hot air-dried carrot; Niamnuy et al. (2012), 26.6 kJ/mol for isoflavones in infrared-dried soybeans; Demarchi et al. (2018), 49.8 kJ/mol for ascorbic acid in vacuum-dried rose hip leathers; and Jha and Sit (2020), 42.5 kJ/mol for polyphenols and 12.4 kJ/mol for flavonoids, both in hot air-dried chebula fruit. Higher activation energy would imply more sensitivity of the phenomenon analysed to a temperature change. Therefore, lower E_a values would suggest a greater stability against temperature.

The rate of degradation reactions commonly increases with higher temperatures and it can be said that, generally, thermal processes bring about a decrease in carotenoids, polyphenols, ascorbic acid, and tocopherols contents. However, lower temperatures not always lead to better results. When the E_a for drying is relatively higher than the E_a for bioactives degradation, it would mean that a moderate increase in the process temperature would allow a significant reduction in processing time with a low impact on bioactives. Following this idea, high-temperature, short-time processing leads to higher bioactive retention when hot air drying is applied, because the high oxygen concentrations involved cause the rapid degradation of bioactives. In the case of vacuum drying, as the effect of oxygen is hindered, the effect of temperature may become important or may be compensated by the duration of the process However, the increase of temperature that a food can withstand is always limited (Demarchi et al. 2018).

On other hand, a positive impact is attributed to temperature, when a higher content of some carotenoids and polyphenols are found in processed foods, explained by cellular structure damage leading to increased bioavailability or extractability

Table 5.2 Mathematical models for degradation kinetics of some bioactives in dried foods

Bioactive	Food	Drying method	Mathematical model	Reference
Lycopene	Tomato pulp	Heating concentration, spray drying	$C_t = C_0 \ \exp(-k\,t)$ $k = a\exp(bX)\exp\left(-\frac{c}{T}\right)$ *a, b, c:* model constants	Goula et al. (2006)
β-carotene	Carrot slices	Hot air drying	$-\frac{dC}{dt} = kC$	Goula and Adamopoulos (2010)
	Carrot cylinders	Hot air drying	$k = k_0 \exp\left(\frac{-E_a}{RT}\right)\left(A_1 + A_2X + A_3X^3\right)$ A_1, A_2, A_3: model constants	Timoumi et al. (2019)
	Carrot cubes	Low-pressure superheated steam drying (LPSSD), vacuum drying, hot air drying.	$\frac{C_t}{C_i} = a + b\frac{X_t}{X_i} + c\frac{T_p}{T_i}$ *a, b, c:* model constants	Suvarnakuta et al. (2005)
Carotenoids and ascorbic acid	Red pepper	Cross flow hot air drying	$\frac{dC}{dt} = -kC$ $k = k_0 \exp\left(\frac{-E_a}{RT}\right);\ k_0 = K_1 X^{K_2};$ $\frac{E_a}{R} = K_3 + K_4X$ K_1, K_2, K_3, K_4: model constants	Di Scala and Crapiste (2008)
Ascorbic acid	Sweet potato	Hot air drying	$C_t = C_0 \ \exp(-k\,t)$ $k = k_0 \exp\left(\frac{-E_a}{RT}\right)$	Orikasa et al. (2010)
	Rose hip leather	Vacuum drying	$-\frac{dQ}{dt} = k_0 \exp\left(\frac{-E_a}{RT}\right)Q^{(n_1+n_2T_b)}$ $k_0 = k_1 + k_2X$ n_1, n_2, k_1, k_2: model constants	Demarchi et al. (2018)

(continued)

Table 5.2 (continued)

Bioactive	Food	Drying method	Mathematical model	Reference
Phenolic content	Germinated soybean	Microwave drying	$C_t = C_0 \exp(-k\,t)$ $k = k_0 \exp\left(\frac{-E_a m}{P}\right)$ *m:* dry mass, g; *P:* microwave potency, W	Lien (2018).
Phenolic content and anthocyanins	Pomegranate arils	Vacuum drying	$C_t = C_0 \exp(\pm k\,t)$ $k = \frac{k_B}{h} T \exp\left(\frac{\Delta H^* - T\Delta S^*}{RT}\right)$ ΔH*: enthalpy of activation, kJ/mol ΔS*: entropy of activation, kJ/mol K k_B: Boltzmann constant = 1.381×10^{-23} J/K h: Planck constant = 6.626×10^{-34} J.s	Karaaslan et al. (2014)
Phenolic and flavonoid content	Terminalia chebula fruit	Hot air drying	$C_t = C_0 \exp(\pm k\,t)$ $k = k_0 \exp\left(\frac{-E_a}{RT}\right)$	Jha and Sit (2020)
	Brussels sprouts	Microwave drying		Nakilcioglu-Tas and Otles (2018)
Isoflavones	Soybean	Infrared drying	$-\frac{dC}{dt} = kC$ $k = k_0 \exp\left(\frac{-E_a}{RT}\right)$	Niamnuy et al. (2012)

General Nomenclature. *C:* bioactive concentration (dry basis); E_a*:* activation energy (kJ/mol); *X:* moisture content (dry basis); *T:* product temperature (K); T_b*:* tray temperatures (K); *R:* universal gas constant = 8.314 kJ/mol K; *t:* time (min or seconds); k_0*:* pre-exponential factor. **Subscripts.** *i:* initial; *t:* drying time; *p*: product

(Wang 2007). However, more specific research is needed to determine whether such advantage is really convenient or at the same time causes detrimental effects on other quality parameters.

After reviewing the available data, it can be said that during drying of complex food matrices, the effect of temperature on bioactives can be direct, increasing the rate of degradative reactions; indirect, by inhibition of enzyme activity or formation of new compounds having antioxidant activity, as occurs during the development of the Maillard reaction; or can be interacting though time with the effect of oxygen (lower temperature, longer drying time, higher oxidation extent). So, the real effect

of temperature may have to be evaluated on pure components, under strictly controlled conditions, to avoid interactions with other factors. Although such controlled experiments would not represent the state of affairs during drying, it may throw light on the pure mechanisms and thus provide basic knowledge necessary to build a more complex theory to explain coupled, interactive, "real" phenomena occurring on the food matrix at changing temperatures, moisture contents and time. Such theories would lead to a more accurate understanding and simulation of the real process, and therefore may be useful to the food industry.

5.3.2 Oxygen

Most of bioactive compounds have antioxidant properties, implying that antioxidants easily react with oxygen to prevent the oxidation of other cellular constituents. For this reason, the oxygen partial pressure during drying is an important factor affecting bioactives retention. Such effect can be observed when comparing hot air and vacuum drying at the same temperature, being the latter the most convenient. When vacuum is applied, not only the oxygen concentration in the drying chamber is lowered, but also the drying time, because boiling of water usually takes place. Again, we should consider interacting factors when comparing drying methods, or design experiments to avoid them. The high vacuum levels used for freeze-drying, along with the very low temperature of the sample are responsible for the benefits of this method over others. However, as the time required is significantly higher and the sample is usually pulped or cut in small pieces, some factors not commonly considered as light could be detrimental Sample preparation for freeze-drying also requires time and manipulation, during which atmospheric oxygen may exert his effect.

The variation of air velocity in convective drying, for a certain air temperature, is another situation in which the effect of oxygen on bioactives is manifested. Since drying rates are increased by high air velocities in high moisture foods, the process times are expected to be shorter and the retention, higher. However, the higher flow of air in contact with the sample appears to be decisive, leading to lower bioactive retention. Here, air temperature affects drying rates more than airflow, though as far as oxidation is concerned, the situation may be opposite and the bioactive compound of interest may more affected by temperature or oxygen, being case-specific. More experimental work is needed to confirm these tendencies in particular food systems.

5.3.3 Light

It is known that carotenes and polyphenols, especially flavonoids, are very sensitive to light. They undergo photo-oxidation, i.e., they interact with molecular oxygen in a reaction catalysed by the energy provided by light. Moreover, the incidence of intense light from the visible or the UV spectrum can directly induce the cleavage of carotene structure, leading to the loss of colour and bioactivity.

Photodegradation of bioactives has been studied in detail, describing how the exposure to light affects isolated compounds or model systems containing them (Zhongwei et al. 2014; Faia Fernandes et al. 2016; Marques da Silva et al. 2017; Djediat et al. 2020). However, the effect of light is not commonly considered in the context of food drying. If the design of drying equipment is analysed, a subject not always found in the literature, drying cabinets are observed to be totally constructed in solid materials that do not allow light to pass through, or may have glass or acrylic windows. Besides the laboratory conditions may also influence the process by natural or artificial lighting. This should be considered during freeze-drying at laboratory scale, when the sample is usually pulped or cut, with a high exposed surface over a large period of time, in a drying chamber consisting of shelves with a transparent cover thus, not protected from light incidence.

5.3.4 Food Sample

Beyond external factors related to the drying conditions, the physicochemical characteristics of the food are relevant for bioactives retention. Regarding the chemical composition, the presence of enzymes that mediate oxidation reactions would have a negative impact; on the other hand, protecting compounds may be favourable, e.g. tocopherols protecting β-carotene from oxidation or polyphenols recovering the ascorbic acid from its oxidized form. It is important to determine food composition and also consider the changes it may experience during the drying process.

Concerning macro and micro structure, the presentation form is important for samples, the fruit or vegetable may be whole, cut, pulped, foamed, or emulsified. Aspects as the integrity of cellular structure, the exposed surface area, or the interaction between compounds will depend on the sample presentation form.

Finally, we could observe “dynamic factors” that change along drying, as the water activity (a_w) of the sample. Depending on the food composition, a_w may decrease slowly or rapidly, leading to a less or more stable sample during the process, respectively.

5.4 Concluding Remarks

On the grounds of the precedent description, researchers should pay attention to interacting factors during drying and design experiments to isolate the effect of such factors; data to be compared should be carefully chosen, and extraction procedures for bioactives quantification have to be rigorously observed, considering the history of the food matrix along the entire process (e.g., when freeze-dried samples are rehydrated for extraction, enzymatic activity might be recovered and oxidation would occur to some extent). Not only the final bioactive retention values should be analysed but also the degradation kinetics, taking advantage of mathematical models to help in the understanding of interacting factors (e.g., a final bioactive content may be achieved by different drying conditions because the effect of interest is concealed by drying time).

In the practical area and although the theoretical aspects can be known, the selection of the best drying method for a food is not a simple endeavour, because food products cannot be seen just as bioactive delivery systems. Beyond the nutritive aspect, organoleptic characteristics and presentation forms are required in order to judge quality. There are dried products developed to be rehydrated before consumption or incorporated to different food preparations, while others are ready-to-eat snack products. Any one of them must present specific properties that are achieved by certain drying techniques. The challenge is to find "targeted solutions" applying the drying methods in the least aggressive way possible for bioactives. The application of experimental measurements and models based on coupled differential equations, or even neural networks is becoming a necessity in order to take into account the diverse interacting variables and the relative influence of each one, acting simultaneously with all the others, in order to allow decision support systems for drying equipment and process design, on the basis of an index formed by a weighted diversity of organoleptic, nutritional and economic parameters.

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Chapter 6
Influence of Canning on Food Bioactives

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Abbreviations

DHA	Docosahexaenoic acid
EPA	Eicosapentaenoic acid
fw	Fresh weight
GAE	Gallic acid equivalent
UHT	Ultra-high temperature
USDA	United States Department of Agriculture

6.1 Introduction

In recent years, a decrease in the consumption of fruits and vegetables including legumes and seeds was observed, owing to the necessary time for domestic preparation, processing and cooking. Thus, nutritious vegetables such as beans that require a long cooking time have been replaced by other foods in the diet (Pedrosa et al.

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2015). Industrially processed plant and animal-based products are safe alternatives that meet this demand for convenient and nutritious ready-to-eat foods. Heat-treated products can offer convenience and at the same time safe, nutrient rich foods to the consumer. Canned fish, for instance, is a traditional healthy, convenient and tasty food (Ferraro et al. 2013). Canned beans are another example of healthy canned food, as it provides 8% of the daily protein intake, 5% of the daily carbohydrates intake and 10% of the daily dietary fibre intake, exhibiting adequate nutritive profile according to the United States Department of Agriculture (USDA) dietary recommendations (Pedrosa et al. 2015).

Canned foods are traditionally defined as thermally processed, shelf-stable products in sealed containers, for which different modes of heating and packaging can be applied (Deak et al. 2012). Thermal processing is the most commonly used method of preservation by the food industry, as it can prevent the growth of bacteria, fungi, and other microorganisms. Canning is considered the safest process of food preservation, assuring products a very long shelf life. The canning industry successfully produces billions of cans, jars, packets, and pouches annually (Deak et al. 2012).

The preservation, and sometimes the enhancement, of the nutritional quality of the products subjected to heat treatment is of great interest to the food industry. With this aim, studies on the improvement and optimisation of the processing parameters for the thermal processing of food products have been carried out. In this work, some of these studies are discussed in detail. In general, the ultimate goal of the most recent investigations on canning is the optimisation of the microbial safety and quality of thermally processed foods without compromising their nutritional, functional and sensory characteristics. Moreover, it is of great interest to minimize the destructive influence of heat on valuable food components, such as vitamins and phytochemicals, that can present beneficial bioactive effects for human health.

6.1.1 Canning Methods: Plant-Based Products

The first step in the canning process of fruits and vegetables consists in selecting and cleaning the plant material, usually by washing in potable running water. Another option is dry cleaning, which can be done by employing mechanical shakers, slowly rotating cylinders, or a rotating soft brush (Homayouni et al. 2015). Subsequently, the fruits and legumes are peeled, pitted, sliced/cut into cubes, depending on the material. The pre-processed plant-based products are then added to canning containers, which can be empty cylindrical metal cans (Arampath and Dekker 2020), sometimes made with enamel-coated bodies (Hong et al. 2004), or glass flasks (Oliveira et al. 2012).

The filling medium used in the canning of fruits is usually sugar syrup, while legumes and seeds receive the addition of brine. Sugar content of the canning syrup varies greatly among studies reported in the literature, as well as the syrup temperature, which sometimes is heated before being transferred to the containers.

6.1.1.1 Fruits

A summary of the manufacture method for canned peaches is presented in Fig. 6.1, and different methods and processing parameters for the canning of fruits are discussed below.

In the thermal processing of peach, a 30 °Brix sucrose syrup prepared with filtered potable water is added to metal cans filled with fruit pieces (Hong et al. 2004), while a 16 °Brix hot syrup (70 °C) is added to apricot halves before the cans are sealed (Le Bourvellec et al. 2018). In the processing of cherries, Nr 303 cans with dark fruit enamel are filled with pitted fruits and a 19 °Brix hot syrup (80 °C) is added (Chaovanalikit and Wrolstad 2004). Similarly, in the canning of blueberries, a 40 °Brix boiling syrup prepared by adding Sweetose 4300 corn syrup to boiling water is added to cans to the brim (Brownmiller et al. 2008). Furthermore, in a recent study on the canning process of mango and pineapple (Arampath and Dekker 2020), a 15 °Brix sugar syrup was applied. The empty cans were filled with fruit pieces and the sugar syrup, leaving 10% headspace, while uniform weight and filling height were kept across all cans.

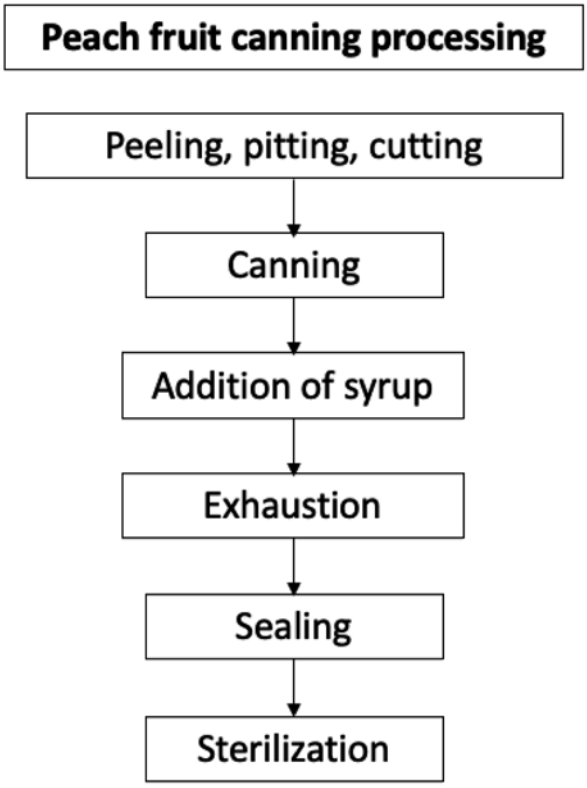

Fig. 6.1 Block diagram for the manufacture of canned peach

6.1.1.2 Vegetables

The canning process of vegetables differs slightly from the processing of fruits. Some of these methods are discussed below, and a summary of the canning method for beans is presented in Fig. 6.2.

Before being added to cans, beans are soaked in decalcified water (12–18 h) followed by blanching (70 °C for 9 min) (Pedrosa et al. 2015). The cans are then filled with brine composed of water, salt and ascorbic acid (Pedrosa et al. 2015). Another method for the canning of beans starts by submerging the seeds in water at room temperature for 1 h, and then soaking them in water at 90 °C for 5 min (Rocha-Guzman et al. 2013). Cans are then filled with beans and soaking water, leaving 1 cm of headspace (Rocha-Guzman et al. 2013).

In the processing of tomato sauce, dried salted tomatoes are previously blanched in boiling water for 8 min and then drained (Cosmai et al. 2013). All the other sauce ingredients, such as sunflower oil/extra virgin olive oil, vinegar, herbs and seasonings are gradually added, mixed and blended in a mechanical cutter until a homogeneous product is achieved. The resulting product is then filled, with the same oil as

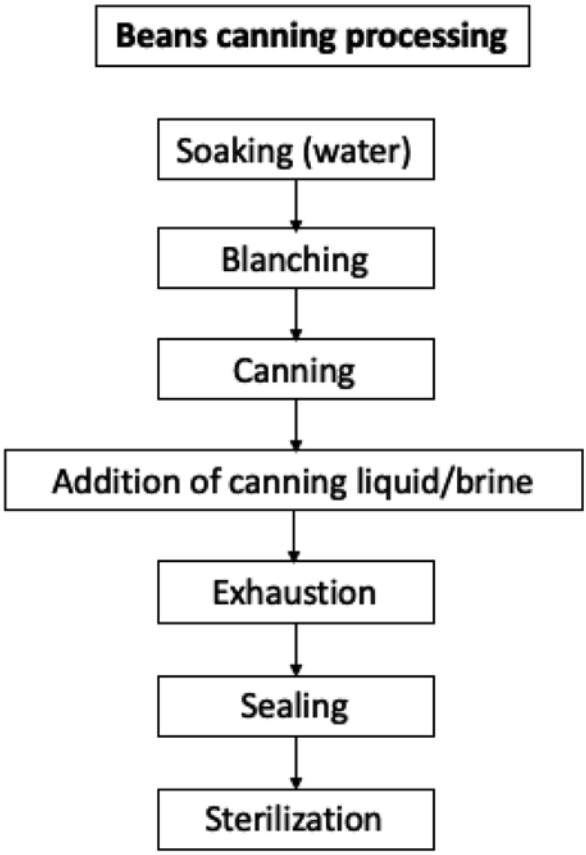

Fig. 6.2 Block diagram for the manufacture of canned beans

used in the ingredient formulation, into glass vessels which are hermetically sealed with metal caps (Cosmai et al. 2013).

Stevanato et al. (2020), in its turn, studied the thermal processing of peach palm heart, which are the edible portion of peach palm stems. The plant material was sanitized with 0.1 ml/L of chlorine solution before being cut/sliced and packed in 250 mL glass jars, which had been previously sterilized in water at 100 °C for 30 min. Finally, the heart palm samples received the addition of brine (1.2% w/w NaCl and 0.5% w/w citric acid solution, pH <4.2) (Stevanato et al. 2020).

After receiving the plant material and the filling liquid (either syrup, brine or sometimes just water) the canning containers (cans, jars) are usually exhausted. The exhaustion process can be carried out by introducing the filled containers directly to an exhauster (Rocha-Guzman et al. 2013), in a hot water bath (at 80 °C for 20 min) (Arampath and Dekker 2020), in a steam box (at 87.8–93.3 °C for 4 min) (Brownmiller et al. 2008) or a steam bath (for 2 min and 30 s) (Chaovanalikit and Wrolstad 2004).

Subsequently, the cans/jars/flasks are immediately sealed to create vacuum in the headspace. Traditional canning implies the heat processing of products in hermetically sealed containers (Deak et al. 2012). This step is necessary to prevent the product's recontamination with microorganisms. Sealing can be done by manually adding lids/caps to the containers or by employing equipment such as a steam-flow can seamer (Chaovanalikit and Wrolstad 2004).

Sealed containers are then submitted to thermal treatment, for which parameters can vary depending on the product. Various levels of, and strategies for, heating are used during processing, and the final heat treatment is often pasteurization or sterilization (Deak et al. 2012). For instance, in a study by Hong et al. (2004), peach processing conditions were designed to ensure commercial sterility of the product at 104 °C for 10 min. In another study by Oliveira et al. (2012), peach cubes were pasteurised by being heated in a water-bath at 90 °C. The temperature at the centre of the cubes was monitored with a thermometer, reaching 84.5 °C after 15 min and remaining that temperature for 5 min thereafter (Oliveira et al. 2012). In the processing of apricots, cans were sealed and then heated at 95 °C (Le Bourvellec et al. 2018). The temperature of the syrup in the can was monitored by thermocouples, reaching 95 °C after 14 min and remaining at this temperature for 2 min thereafter (Le Bourvellec et al. 2018). Regarding the thermal processing of berries, cans filled with cherries were immersed in a water-bath and heated at 100 °C for 12 min (Chaovanalikit and Wrolstad 2004). Similarly, blueberries were immersed in boiling water for 15 min (Brownmiller et al. 2008).

In the processing of peach palm heart, filled jars were treated at 100 °C for 30 min (central portion) or 25 min (basal and apical portions), following the method industrially applied in Brazil. Beans were cooked at 116 °C for 42 min by Pedrosa et al. (2015), while Rocha-Guzman et al. (2013) employed a vertical retort at 15 lbs of pressure for the same product.

More recently, mango and pineapple were thermally treated using pressurized steam (1.5–3.0 bar) in a pilot scale rotary-type retort (Arampath and Dekker 2020). The rotary-type retort delivers efficient internal heat transfer that combines

continuous rotating and mixing action, facilitating the canning process. These authors applied different sets of temperature–time combinations for their fruit canning experiments, comprising 115,120, 125, and 130 °C (retort temperature) and 10, 20, 30, and 40 min thermal treatments (Arampath and Dekker 2020).

Finally, after being submitted to thermal processing, the containers are then cooled to room temperature, which can be done by placing the cans/jars in water-bath (usually 25 °C) or by being sprayed with cold water (Chaovanalikit and Wrolstad 2004; Le Bourvellec et al. 2018). The cans/jars can then be stored at room temperature and in a place away from direct sunlight.

6.1.2 Canning Methods: Animal-Origin Products

6.1.2.1 Fish

Canned fish is a traditional product widely consumed worldwide, as canning is one of the most used methods for fish preservation, accounting for 14.4% of total fish market (Ferraro et al. 2013; Prego et al. 2021). Sardines were first canned in France back in 1834, while salmon canning is said to have started in 1877 in northern Japan (Jarvis 1988). Nowadays, the main canned fish species are tuna, sardine, sardine-type and mackerel (Ferraro et al. 2013).

Briefly, the canning fish processing comprises the reception of fish (either refrigerated or frozen), brining, removal of unwanted parts, (steam) drying, canning, sterilization and packing (Ferraro et al. 2013). A summary of the manufacture of canned fish is presented in Fig. 6.3.

Seet and Brown (1983) described a method for the production of canned tuna. First, the raw fish is received, precooked and placed in a 2 °C cold room overnight. Three pieces of solid meat are then packed in each metal can. Salt is then placed on top of the meat, and distilled, deionized water is next added, leaving a headspace of about 10 mm. The cans are then treated in a steam retort at two processing temperatures, at either 115 °C for 120 min or 121 °C for 95 min. The cans are finally allowed to cool and stored.

6.1.2.2 Meat

In a recent study, Rather et al. (2020) described the canning process of a traditional Indian cuisine product based on lamb meat. Lamb meat balls (goshtaba) were prepared and transferred to tin cans, followed by the addition of gravy (yakhni) at 80 ± 5 °C to each can. A headspace of 10% was left in the cans, that were sealed by single seaming operation and exhausted using steam in an exhaust box at 100 °C for 10 min. After exhausting, cans were double seamed. The cans were then thermally treated in a retort at 121 °C and 15 psi pressure for about 35 min. Finally, cans were cooled by spraying potable cold water and stored at ambient conditions (20–30 °C).

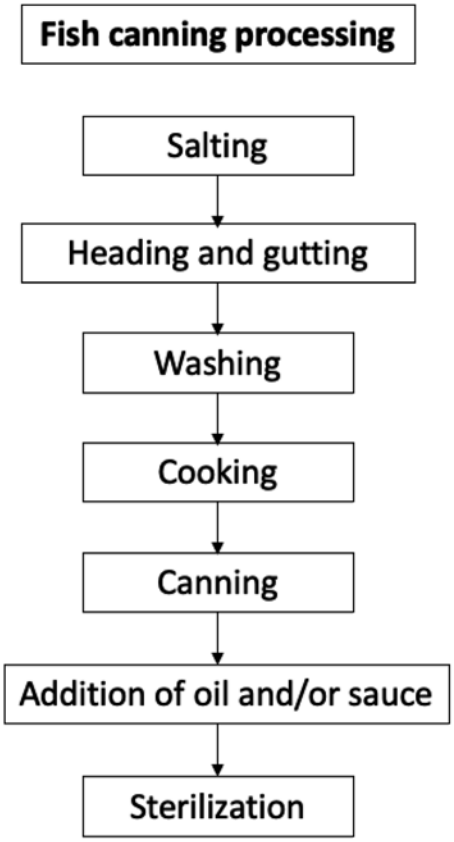

Fig. 6.3 Block diagram for the manufacture of canned fish

6.1.2.3 Dairy

Fresh dairy products have a limited shelf-life, due to their high-water content. Hence, thermal processing of dairy products is traditionally carried out to improve their microbiological stability. For instance, in the production of canned evaporated milk, the water content is reduced by evaporation, and the final concentrated product is then sterilised. This processing improves the product's shelf-life by preventing microbiological growth (Oliveira et al. 2009).

Processing of evaporated milk starts with the reception of raw milk followed by preliminary treatments, such as clarification, fat separation or milk standardisation. The milk is then pre-heated (115–128 °C, 1–6 min) to inactivate enzymes and to destroy microorganisms and spores. Pre-heating also contributes for the heat stability of the product, facilitating the sterilisation process. Milk is then concentrated by vacuum evaporation at (45–70 °C). Next, the product is homogenised (usually at 65 °C) and sterilised. Sterilisation can be done by either the in-bottle method or by Ultra High Temperature (UHT). The in-bottle sterilisation is performed on packed milk either in batches (autoclaves) or continuously. The combinations of

time-temperature for this method are usually 100–120 °C for 15–20 min or 140 °C for 3 s. Another alternative is the UHT treatment (15 s at 140 °C), which is more effective in the destruction of spores than the in-bottle sterilisation. After sterilisation, the product is cooled down and can be stored (Oliveira et al. 2009).

6.2 Impact of Canning on Food Components

Table 6.1 presents a compilation of studies found in the literature on the impacts of canning on food components, which are discussed in detail in the next sections.

6.2.1 Impact of Canning on the Nutritional Value

6.2.1.1 Carbohydrates

Carbohydrates are macronutrients that usually remain stable during thermal processing. In the canning of sardines, thermal processing had no effect on the carbohydrate content (Tarley et al. 2004), and the same was observed for canned tomato (Abdullahi et al. 2016).

In canned beans, thermal processing showed some effects on starch content. Canned common beans presented a 80% higher amylose content in comparison with raw beans, while the amylopectin content was reduced in 27% after thermal treatment (Pedrosa et al. 2015).

Resistant starch is known to be beneficial to the human health by protecting against colon carcinogenesis and facilitating cholesterol and glucose metabolism, as it becomes a substrate for the colonic flora to produce short-chain fatty acids. In the same study, the resistant starch content of beans was reduced by the canning process (a reduction of more than 54%) (Pedrosa et al. 2015). This reduction in the resistant starch content after thermal treatment has also been observed for other legumes (barlotto bean, chickpea, faba bean and white kidney bean) (Güzel and Sayar 2012).

6.2.1.2 Dietary Fibre

Dietary fibres can exert beneficial effects for human health, such as the regulation of intestinal transit and the prevention of diabetes, cardiovascular diseases and obesity (Stevanato et al. 2020).

In the processing of peach palm heart, canning slightly decreased the content of most fibres, also leading to changes in mechanical properties of the canned product (Stevanato et al. 2020). Canning also significantly reduced the soluble and insoluble dietary fibre content of common beans (Kutoš et al. 2003). These authors also observed that changes in the dietary fibre content due to thermal processing can vary

Table 6.1 Compilation of studies found in the literature on the impacts of canning on food components

Canned food product	Canning method summary	Studied compounds	Main findings	Reference
Peach	Peach fruits were sliced, pitted, and peeled with 2% lye, rinsed, and packed into cans. Cans were filled with 30 °Brix syrup prior to pulling a vacuum on the pack and sealing the lids to the cans. Processing conditions were designed to ensure commercial sterility at 220 °F for 10 min.	Procyanidins	Thermal processing resulted in an 11% reduction in procyanidin monomers, a 9% reduction in dimers, a 12% reduction in trimers, a 6% reduction in tetramers, and a 5% reduction in pentamers. After 3 months of storage, levels of monomers had decreased by 10%, dimers by 16%, trimers by 45%, and tetramers by 80%; oligomers larger than tetramers were not observed. A similar trend was observed in the canning syrup. The migration of procyanidins into the canning syrup can account for the losses observed during the thermal processing step.	Hong et al. (2004)
Peach	Peach fruits were peeled, pitted, and the flesh cut into cubes. Fruit pieces were placed in glass flasks, covered with aluminum foil, and heated in a water-bath at 90 °C (20 min). The flasks were then capped and cooled to room temperature in a water bath during 30 min. Flasks with pasteurized fruit pieces were stored in the dark for 90 days at room temperature.	Carotenoids and phenolic compounds	Significant reductions in total carotenoids were observed immediately after pasteurization but total antioxidant activity and the concentration of total phenolics were unaffected. Pasteurization induced significant reductions in the concentration of protocatechuic acid, zeaxanthin and β-cryptoxanthin. After 90 days of storage, there was a significant reduction in antioxidant activity, total phenolics, and total carotenoids.	Oliveira et al. (2012)

(continued)

Table 6.1 (continued)

Canned food product	Canning method summary	Studied compounds	Main findings	Reference
Apricot	Apricot fruits were cleaned, cut and pitted. Apricot halves were put into each can, to which hot syrup (70 °C) was added (16 °Brix, sucrose). Cans were sealed and then heated at 95 °C. The temperature reached 95 °C after 14 min and remained at this temperature for 2 min thereafter. Finally, cans were sprayed with cold water and stored.	Polyphenols and carotenoids	Procyanidins were retained in apricot tissue over thermal processing. Hydroxycinnamic acids, flavan-3-ol monomers, flavanols and anthocyanins leached in the syrup. No significant reductions of total carotenoids were observed after processing. Flavonol concentrations and cis-β-carotene isomer were significantly increased after processing. After 2 months of storage, among polyphenols only hydroxycinnamic acids, flavan-3-ol monomers and anthocyanins were reduced, along with the total carotenoid content.	Le Bourvellec et al. (2018)
Cherry	Stemmed Bing cherries were washed and pitted. Cans with dark fruit enamel were filled with pitted cherries and a 19 °Brix sucrose syrup (at 80 °C). The cans were exhausted on a steam bath for 2 min and 30 s and sealed with a can seamer. The canned cherries were cooled by placing in 25 °C water and then stored.	Anthocyanins and total phenolics	Canning resulted in approximately 50% transfer of anthocyanins and total phenolics from the fruits into the syrup. Heat processing did not result in a loss of total anthocyanins, total phenolics, and antioxidant activity when the values for syrup and cherries were combined.	Chaovanalikit and Wrolstad (2004)
Blueberries	Frozen blueberries were added to cans. Boiling syrup (40 °brix) or water were added to the cans to the brim and cans were exhausted for 4 min in a steam box (87.8–93.3 °C). The cans were then sealed, immersed in boiling water for 15 min, and stored at 25 °C.	Procyanidins	Processing blueberries resulted in significant losses of total procyanidins, with 65 and 78% being retained in berries canned in syrup and canned in water, respectively. Procyanidins were further degraded during 6 months of storage, with 22% retained in berries canned in syrup and 32% in berries canned in water.	Brownmiller et al. 2009

Blueberries	Frozen blueberries were added to cans. Boiling syrup (40 °Brix) or water were added to the cans to the brim and cans were exhausted for 4 min in a steam box (87.8–93.3 °C). The cans were then sealed, immersed in boiling water for 15 min, and stored at 25 °C.	Anthocyanins	Processing of berries canned in syrup or canned in water resulted in total monomeric anthocyanin losses of 28% and 34%, respectively, compared to the original levels found in fresh berries. Levels of monomeric anthocyanins declined during storage of 1, 3 and 6 months, with losses of 35%, 60%, and 71% observed in berries canned in syrup, respectively, and losses of 48%, 52%, and 62% for berries canned in water, respectively.	Brownmiller et al. (2008)
Mango and pinneaple	Mango and pineapple fruits were washed in potable running water, peeled, and cut into cubes. Empty cans were filled with fruit pieces and sugar syrup, leaving 10% headspace. Filled cans were exhausted in a hot water bath at 80 ± 1 °C for 20 min and immediately sealed. The sealed cans were thermally treated using pressurized steam (1.5–3.0 bar) in a pilot scale rotary-type retort temperature–time combinations for the canning experiments were 115,120, 125, and 130 °C (retort temperature) and 10, 20, 30, and 40 min thermal treatments. Cans were then cooled immediately using running water, air-dried and stored.	Vitamin C, β-carotene, polyphenols and flavonoid content	Health-promoting phytochemicals in canned products were present in substantially lower concentrations than in fresh mango and pineapple. The vitamin C content was reduced in canned mango and pineapple pieces during thermal treatment, and leaching of vitamin C to the canning syrup was also detected. At the end of the heat treatment at 115 °C for 40 min, the retention of vitamin C was 27% in mango pieces and 31% in pineapple pieces.	Arampath and Dekker (2020)

(continued)

Table 6.1 (continued)

Canned food product	Canning method summary	Studied compounds	Main findings	Reference
Beans	Bean samples were soaked in decalcified water (for 12–18 h), blanched (70 °C for 9 min) and canned. Salt and ascorbic acid were added to the canning liquid. The canned product was cooked (116 °C for 42 min), cooled and stored.	Proximate composition, micronutrients and phenolic compounds	Industrial canning significantly increased the protein (>7%) and dietary fibre (>5%) contents of both beans varieties (Curruquilla and Almonga beans). However, the minerals, total a-galactosides and inositol phosphates contents were reduced (>25%) in both canned seeds. The trypsin inhibitors content was almost abolished by canning, and no lectins were found in either of the canned samples. Canned Curruquilla showed a decrease (38%) of their antioxidant activity.	Pedrosa et al. (2015)
Beans	Not available (in this study, raw beans were compared to commercial canned beans).	Dietary fibre	Canning decreased the soluble and insoluble dietary fibre content of brown beans of the pinto variety.	Kutoš et al. (2003)
Beans	Common beans were washed, then submerged in water at room temperature for 1 h, and then soaked in water at 90 °C for 5 min. Cans were filled with common beans and soaking water, leaving 1 cm of headspace. Cans were introduced to an exhauster and then sealed. Cans were introduced in a vertical retort at 15 lbs. of pressure, then cooled and stored.	Phenolic compounds	Canned beans presented bioactive phenolics such as catechin, kaempferol and ferulic acid, as well as anticancer phytochemicals such as quercetin, protocatechuic acid, myricetin, naringenin and their derivatives, and procyanidins. Moreover, it was detected the presence of hydroxybenzoic and hydroxycinnamic acids, flavonols, and monomeric flavan-3-ols. Polyphenols in canning beans displayed chemoprotective potential as they activate mechanisms involved in apoptosis pathways. Therefore, a diet including canned beans might represent health benefits and cancer preventive effects.	Moreno-Jiménez et al. (2019)

Beans	Common beans were washed, then submerged in water at room temperature for 1 h, and then soaked in water at 90 °C for 5 min. Cans were filled with common beans and soaking water, leaving 1 cm of headspace. Cans were introduced to an exhauster and then sealed. Cans were introduced in a vertical retort at 15 lbs. of pressure, then cooled and stored.	Total phenolic content and antioxidant activity	The canning process reduced the polyphenolic content and the antioxidant activity of Mexican common bean cultivars. The best cultivar for canning process was Bayo Victoria. This cultivar showed higher values for integrity, total phenolic content and antioxidant capacity measured by the 2,2-diphenyl-1-picryl-hydrazyl-hydrate (DPPH) method after the canning process.	Rocha-Guzman et al. (2013)
Peach palm heart	Peach palm heart was sanitized (with 0.1 ml/L of chlorine solution), diced/cut in pieces and packed in glass jars. The product received the addition of brine (1.2% w/w NaCl and 0.5% w/w citric acid solution, pH < 4.2). The jars were exhausted, sealed, and treated at 100 °C for 30 min (central portion) or 25 min (basal and apical portions), according to industrial processing guidelines. Canned products were allowed to cool and stored at 25 °C and away from direct sunlight.	Phenolic compounds and carotenoids	Canning decreased the content of total phenolics (38%–56%), carotenoids (17%–27%), and antioxidant activity (16%–35%). After 120 days of storage, it was observed a further decrease in the contents of phenolics (7%–33%), carotenoids (22%–33%), and antioxidant activity (30%–76%), in relation to the initial time.	Stevanato et al. (2020)
Tomato sauce	The product (a paste composed of dried tomatoes, mushroons and seasonings) was filled into transparent glass vessels which were hermetically sealed with metal caps, then submitted to thermal stabilization (95 °C for 60 min; 85 °C in the core), using a Sterile disk jumbo spd Technosoft apparatus and then quickly cooled to room temperature.	Volatile compounds and carotenoids	The thermal degradation of carotenoids and fresh tomato-derived compounds, along with lipid oxidation and Maillard reaction, caused an increase in volatile compounds in canned tomato sauce. The terpenic compounds showed significant decreases after the thermal stabilization process treatment, due to their degradation and oxidation favored by high temperatures. The results highlighted the influence of the thermal stabilization process on the evolution of volatile composition of tomato-based products.	Cosmai et al. (2013)

(continued)

Table 6.1 (continued)

Canned food product	Canning method summary	Studied compounds	Main findings	Reference
Tomato paste	Not available (commercial canned tomato of different company products were obtained from Tarauni market in Kano State, Nigeria).	Proximate composition, minerals and vitamins	Canned tomato showed a lower moisture and fat contents, and a higher carbohydrate, protein, crude fibre and ash contents than fresh tomatoes. Sodium, potassium and calcium concentrations were significantly higher in canned tomato, while iron was found to be significantly higher in fresh tomato. Vitamin A content was higher in fresh tomato, while the vitamin C content was higher in canned tomato.	Abdullahi et al. (2016)
Tuna	The fish were dressed, washed, and precooked in a steam chest at atmospheric pressure (at 100 °C) for 3 h. After precooking the fish were placed in a 2 °C cold room overnight, and then packed into cans. Salt was placed on top of the meat, and distilled, deionized water was next added, leaving a headspace of about 10 mm. Cans were treated in a steam retort at two processing temperatures, 115 °C and 121”C, for 120 and 95 min respectively. The cans were allowed to cool and stored.	Water-soluble vitamins and minerals	The levels of water-soluble vitamins and minerals were reduced in canned tuna in comparison with raw fish. Thiamin retention for the canned tuna was about 5%, while niacin and riboflavin ranged from 71–73% and 49–50% respectively. The values for Cu, Fe, K, and Ca were significantly lower in canned tuna.	Seet and Brown (1983)

Lamb meat product	Indian lamb meat balls (goshtaba) were prepared and transferred to tin cans, followed by the addition of gravy (yakhni) at 80 ± 5 °C to each can. Cans were sealed and exhausted. Thermal treatment was carried out in a retort at 121 °C and 15 psi pressure for about 35 min. Cans were cooled by spraying potable cold water and stored at ambient conditions (20–30 °C).	Fatty acid profile	The fatty acid composition (saturated, monounsaturated, polyunsaturated and trans) during processing and storage showed non-significant difference in a lamb meat product. The cholesterol content decreased significantly after canning in all products. During storage all products exhibited significant decrease in cholesterol up to 6 months of storage, and non-significant variation thereafter.	Rather et al. (2020)
Sardines	Not available (commercial canned sardines were purchased from different markets in Parana State—Brazil).	Proximate composition and fatty acids	Protein contents of canned sardines were equivalent to the values found for sardines in natura. The highest levels of total lipids were found for sardines canned in soybean oil, as well as the highest levels of essential C18:2n-6 and C18:3n-3 fatty acids. The EPA (C20:5n-3) and DHA (C22:6n-3) concentrations presented the highest levels in sardines canned in tomato sauce.	Tarley et al. (2004)

depending on the type of beans, the processing method and its duration. In contrast, Pedrosa et al. (2015) found an increase in the total dietary fibre content of common beans after thermal treatment.

6.2.1.3 Protein

The protein content of food can be affected by thermal processing, and the results vary among different canned products.

The canning process caused a slight increase in the total protein content (more than 7%) of beans from the Spanish common varieties Curruquilla and Almonga (Pedrosa et al. 2015). This increase may be attributed to the loss of soluble solids during processing, which would increase the relative protein concentration.

On the other hand, in a study of four Mexican bean varieties (Black, Pinto Saltillo, Pinto Durango and Bayo), the canning processing effects on the protein content of beans varied (Rocha-Guzman et al. 2013). For the Black and Pinto Durango varieties, there was not a significant difference in the protein content of raw and canned beans. For the variety Pinto Saltillo, there was a slight increase in the protein content after canning, while for the Bayo bean variety the canning process reduced the protein content in almost 50%.

In canned sardines, heat-processing did not affect the total protein content, as the values were equivalent to the ones found for fish *in natura*, ranging from 19.8 to 24.4%, both for sardines canned in oil and in tomato sauce (Tarley et al. 2004). Similarly, the canning process of tuna did not affect its amino acid composition and the protein in vitro digestibility (Seet and Brown 1983). These results indicate that the manufacturing process does not modify the protein value of the fish.

Finally, considering the canning of dairy products, it is known that milk-proteins can be adversely affected and denatured by heat-processing, particularly whey proteins (Mehta 1980).

6.2.1.4 Fat

The fat content and fatty acids composition can be also influenced by heat processing, and the effects can vary depending on the product. Pedrosa et al. (2015) found that heat processing significantly reduced the total fat content of common beans (more than 16%). The same reduction in fat content after heat processing was observed by Abdullahi et al. (2016) for canned tomato.

On the other hand, in canned lamb meat, thermal processing and storage did not cause any significant changes in the fatty acids profile of lipids in the product (Rather et al. 2020). The stability of fat fraction in this meat product was associated with the protective effect of the canning process, which functions as oxygen and light transfer barrier that might have hindered the oxidation process.

In a study conducted with commercial Brazilian canned sardines, it was found that the choice of the liquid used as can filling can also influence the fatty acid

composition of the product (Tarley et al. 2004). In this study, the authors compared the fatty acids composition in different commercial brands of whole sardines canned in soybean oil and in tomato sauce. The results showed that sardines canned in soybean oil had the highest levels of the essential fatty acids C18:2n-6 and C18:3n-3, while sardines canned in tomato sauce presented the highest concentrations of eicosapentaenoic (EPA, C20:5n-3) and docosahexaenoic (DHA, C22:6n-3) acids (Tarley et al. 2004). Numerous health benefits have been attributed to fish oils, particularly EPA and DHA, hence the importance of optimising their content retention in canned fish products.

6.2.2 Impact of Canning on the Physicochemical Composition

6.2.2.1 Soluble Sugars

Changes in the soluble sugar content of canned products can happen due to leaching of compounds and/or equilibration of the concentrations between product and canning liquid.

In common beans, the canning process caused a significant reduction in the sucrose content (>57%) of the product (Pedrosa et al. 2015). Ciceritol was the free sugar most greatly reduced (by approximately 64% in the variety Almonga and 82% in the variety Curruquilla) (Pedrosa et al. 2015). These results can be explained by the leaching of these water-soluble compounds into the canning liquid, that is usually discarded before the consumption of canned beans.

In apricots, a significant increase of sucrose concentration was observed after industrial canning (Le Bourvellec et al. 2018). This increase might be due to an equilibration of the fruit and syrup sucrose concentration consecutive to a softening of the fruit tissue. During storage of canned apricots, a slight decrease in the sucrose content was observed, while glucose and fructose concentrations decreased significantly. This could be due to a syrup sucrose hydrolysis during storage due to low syrup pH (below 4.0) (Le Bourvellec et al. 2018).

6.2.2.2 Vitamin C

Canned fruits and vegetables can present losses of the vitamin C content compared to fresh products, which can occur by non-enzymatic reactions, oxidation during peeling, cutting, and can exhausting (Arampath and Dekker 2020). Variation in the decomposition mechanism of ascorbic acid can be affected by intrinsic characteristics of the product such as its maturity, variety, pH, and dissolved oxygen level (Arampath and Dekker 2020).

After thermal treatment, the vitamin C content in canned mango and pineapple pieces was reduced (Arampath and Dekker 2020). Higher temperatures resulted in higher rates of ascorbic acid decomposition, as the retention of vitamin C in mango

pieces ranged from 4% (130 °C) to 27% (115 °C) after 40 min of thermal treatment. In canned pineapple, the retention rate ranged from 6% (130 °C) to 31% (115 °C) under the same conditions. In the same study, the authors detected leaching of vitamin C into the filling sugar syrup. The vitamin C content measured in the sugar syrup of canned mango was 2.7 mg/100 g (at 115 °C), 3 mg/100 g (at 120 °C), and 2 mg/100 g (at 125 °C), and it was undetectable at 130 °C after 10 min of thermal treatment. Similarly, in the sugar syrup of pineapple cans, the ascorbic acid content measured was 2.8 mg/100 g (at 115 °C), 1.6 mg/100 g (at 120 °C), and 1.2 mg/100 g (at 125 °C), and it was undetectable at 130 °C following a 10 min treatment (Arampath and Dekker 2020). In contrast, Abdullahi et al. (2016) found an increase in the vitamin C of canned tomatoes.

A strategy to slower the degradation rate of ascorbic acid is to reduce the product's contact with oxygen, which can be achieved by assuring optimal conditions during the exhausting step. According to the results found by Arampath and Dekker (2020), a slower degradation reaction rate of vitamin C could be achieved even at high temperatures under anaerobic conditions inside the cans during exhaustion.

6.2.2.3 Vitamin B

In canned tuna, it was observed a decrease in the content of water-soluble vitamins (Seet and Brown 1983). Thiamin retention was of 5%, while niacin and riboflavin ranged from 71–73% and 49–50%, respectively.

6.2.2.4 Vitamin A

In canned tomato, it was observed a reduction of around 50% in the vitamin A content after thermal processing (Abdullahi et al. 2016). In the same study, the authors found a reduction in the total fat content of tomatoes after canning. This might explain the reduction in the vitamin A content for being a fat-soluble vitamin.

6.2.2.5 Citric Acid and Malic Acids

Canning significantly reduced the concentration of citric and malic acids in apricots (Le Bourvellec et al. 2018). The industrial heat-treatment induces the softening of the apricot flesh causing loss of compounds from fruit to syrup. Furthermore, after 2 months of storage, the concentration of malic acid decreased significantly in the canned product. On the other hand, the citric acid concentration remained stable during storage. This can be explained by the distribution of these compounds in the fruit tissues. Malic acid is usually located in the pulp, which facilitates the leaching of this compound to the syrup. Citric acid, in turn, has a subepidermal distribution,

which aids in the stabilisation of this compound in the fruit during storage (Le Bourvellec et al. 2018).

6.2.2.6 Carotenoids

Thermal processing can affect carotenoids, which are heat-sensitive compounds that may be degraded, isomerized, or oxidized after cooking processes (Petropoulos et al. 2019).

In a study by Arampath and Dekker (2020), β-carotene content was degraded with increasing temperatures of thermal treatment in canned mango and pineapple. The retention percentage of β-carotene in mango pieces decreased from 56% (at 115 °C) to 14% (at 130 °C), following retorting for 40 min. For canned pineapple, the retention of β-carotene decreased from 47% to 20% for the same combination of time-temperature. This reduction is likely to be due to the leaching of β-carotene from pieces to sugar syrup during the canning processing. After 10 min of thermal treatment (at 115 °C), a β-carotene concentration of 1.25 μg/g fw was detected in the syrup of canned mango, and of 0.91 μg/g fw in the syrup of canned pineapple processed in the same conditions (Arampath and Dekker 2020).

The same was observed in canned peach, as significant reductions in total carotenoids were detected immediately after pasteurization, particularly in zeaxanthin and β-cryptoxanthin (Oliveira et al. 2012). In this study, the content of all carotenoids decreased significantly during storage, with the exception of zeaxanthin, that had its content increased over shelf-life.

Degradation of carotenoids by oxidation reactions was also reported in the canning of tomato sauce (Cosmai et al. 2013). Thermal degradation of carotenoids, along with the degradation of other fresh tomato-derived compounds, caused an increase in volatile compounds in the final product (Cosmai et al. 2013).

In contrast, in the canning processing of apricots, processing increased extraction efficiency leading to an increase in the concentration of carotenoids, that showed to be more stable than phenolic compounds upon thermal treatments (Le Bourvellec et al. 2018). The stability of carotenoids during thermal processing could be attributed to their crystalline form, as crystalline carotenoids tend to be more stable than dissolved carotenoids. In apricot, β-carotene appears in a crystalline form within the chromoplast, and partially solubilized in lipid droplets. During pasteurisation, the crystalline form remains stable, while the β-carotene solubilized in lipid droplets isomerizes, resulting in a stabilization of trans-β-carotene and an increase in cis-β-carotene. In the study by Le Bourvellec et al. (2018), the crystalline form remained stable during storage, as evidenced by trans-β-carotene contents after 2 months of storage. In turn, the cis-isomer forms were reduced, degraded by oxidation (Le Bourvellec et al. 2018). Nevertheless, the total content of carotenoids decreased during storage.

6.2.2.7 Minerals

A reduction in the mineral content of canned products has been reported, and this could be explained by the leaching of minerals into the soaking and the canning liquids. The canning process caused a significant decrease in the contents of the minerals P (28%), Mg (41%), Ca (17%), Fe (70%) and Zn (30%) of Spanish common beans of the variety Almonga (Pedrosa et al. 2015). In contrast, the same authors found a much less expressive decrease in the Fe content of beans from the variety Curruquilla (13%), and even an insignificant increase in the Zn content (Pedrosa et al. 2015). Catechin and protocatechuic acids were only detected in the Curruquilla samples, which might explain the greater iron retention in this variety: molecules bearing catechol groups can form complexes with iron which have extremely large stability constants, being released in less extent to the canning liquid during thermal treatment (Pedrosa et al. 2015).

However, despite the quantitative large mineral losses, canned beans remain a good source of minerals (Pedrosa et al. 2015). It is also important to notice that for all products that receive the addition of salt into the canning liquid (brine), an increase in the final Na content is expected. In canned tuna, Seet and Brown (1983) also reported a significant decrease in the values of the minerals Cu, Fe, K, and Ca, with copper and calcium showing the lowest retention. In contrast, in canned tomato, sodium, potassium, and calcium concentrations were significantly higher, while iron was found to be significantly higher in fresh tomato (Abdullahi et al. 2016).

6.2.3 Impact of Canning on Phenolic Compounds

6.2.3.1 Polyphenols and Total Phenolic Content

For some products such as fruits, thermal processing can increase the final content of phenolic compounds in comparison with the fresh food matrix (Arampath and Dekker 2020). This increase could be attributed to the release of hydrolysable polyphenols from the cellular matrices at high temperatures, as fruits in general present a phenolic profile composed of soluble-free and bound forms (Sun et al. 2002). Therefore, the canning process could increase the total polyphenol content due to the transformation of bound phenolics into soluble, detectable forms with potential antioxidant capacity.

In the experiments performed by Arampath and Dekker (2020), higher concentrations of total polyphenol content was found in mango and pineapple pieces, as well as in the filling sugar syrup, after thermal treatment. A total polyphenol content of 29.4 mg GAE/100 g fw was found in fresh mango. To assess the impact of thermal treatment parameters on the polyphenol content of the product, the authors tested the following temperatures for 40 min: 115 °C, 120 °C, 125 °C and finally

130 °C. The total polyphenol content in fresh mango at first was slightly reduced, and gradually increased with higher temperatures. The final retention rates for the studied temperatures were as follows, respectively: 77%, 87%, 98%, and 118%. Similar results were found for canned pineapple, that retained the following percentages of total phenolic content in pineapple pieces for the same thermal treatments: 79%, 92%, 134, and 152%, respectively.

It is also important to notice that with the progression of heat treatment, leaching of hydrolysable polyphenols from pieces to the syrup can occur. This results in an increase of the total polyphenol content in the sugar syrup of canned fruits after thermal treatment. (Arampath and Dekker 2020). This leaching effect was observed in the canning of cherries, for which 50% of anthocyanins and polyphenolics were redistributed to the syrup (Chaovanalikit and Wrolstad 2004). In this study, the total phenolic content remained stable when values for fruits and syrup were combined, which indicates that the compounds were leached to the syrup however not degraded by heat-processing.

In the canning processing of peach, the concentration of total phenolic compounds and the total antioxidant activity after pasteurisation remained unaffected (Oliveira et al. 2012). Nevertheless, after storage for 90 days, there was a significant decrease in total phenolics (expressed as gallic acid equivalents) and in the antioxidant activity (expressed as ascorbic acid equivalents) of pasteurised peach.

In contrast, the industrial canning process reduced the total phenolic content of beans. Common Spanish beans presented a reduction of in 9–11% in their total phenolic content (Pedrosa et al. 2015). The same significant reduction was observed particularly in the flavonol content after canning, as well as in tartaric esters (Pedrosa et al. 2015). In the same study, catechin glucoside was the main component of the phenolic fraction of raw beans; however, this compound was not detected after thermal processing, indicating its degradation (Pedrosa et al. 2015). These losses can be attributed to the heat exposure during thermal processing and also to the leaching of some of the phenolic compounds into the canning liquid, due to the softening of the cell walls of beans during cooking. Although the canning process promotes decreases in the content of phenolic compounds, the authors of this study pointed out that the remaining concentrations of phenolic compounds in the canned beans were still relevant to exert beneficial activity to the human health, as the final products also presented antioxidant activity (Pedrosa et al. 2015).

These results are in agreement with Rocha-Guzman et al. (2013), who also reported a reduction of the total phenolic content of beans after canning. These authors studied four varieties of Mexican common beans, comparing the effects of open pan cooking and canning process (sterilisation) on the total phenolic content of beans. In general, the impacts of open pan cooking on the phenolic composition were greater than of canning, which could be explained by the shorter exposure time of these compounds to heat required by this method (Rocha-Guzman et al. 2013).

Following the same canning process described by Rocha-Guzman et al. (2013), Moreno-Jiménez et al. (2019) reported the detection of the following phenolic compounds in two bean varieties (Bayo Victoria and Negro 8025) after being submitted to canning processing: hydroxybenzoic and hydroxycinnamic acids, flavonols,

monomeric flavan-3-ols, catechins, kaempferol, ferulic acid, quercetin, protocatechuic acid, myricetin, naringenin and their derivatives, and procyanidins (Moreno-Jiménez et al. 2019). These results confirm the presence of potentially bioactive phenolic compounds in canned beans, despite of the reduction in their content that might happen due to heating.

In peach palm heart, canning decreased the total phenolic content (38%–56%) and the values for antioxidant activity (16%–35%) (Stevanato et al. 2020). Furthermore, the storage time of 120 days caused a further decrease in the content of phenolic compounds (7%–33%) and in the antioxidant activity (30%–76%).

Pasteurization of fresh apricots also caused a significant loss of total phenolic compounds, from 13% to 47% (Le Bourvellec et al. 2018). The storage time (2 months), however, did not provide a significant loss of total phenolic compounds. This was explained as the main type of compounds found in apricot were procyanidins, which remained stable within apricot tissues during storage. In the same study, the authors found that the following compounds leached in the canning syrup: hydroxycinnamic acids, flavan-3-ol monomers and flavonols (Le Bourvellec et al. 2018). These results are in agreement with Wani et al. (2018), who found that canned apricot pulp retained most of its nutritional and antioxidant properties during processing and storage up to 12 months. Moreover, Jiménez et al. (2008) also found no changes in the different antioxidant activity assays from 1 to 150 days of storage of canned apricots.

Flavonoids are a class of polyphenolic secondary metabolites found in plants, and thus commonly consumed in diets. The concentration of flavonoids can be reduced substantially during the pre-processing of fresh fruits (preparation for canning), the exhausting step and the thermal treatment. Arampath and Dekker (2020) reported a 50% reduction (compared with the content found in fresh mango and pineapple) in the content of the following compounds, after thermal treatment for 10 min: catechin, tannic acid, chlorogenic acid, epicatechin, and gallic acid compounds.

Interestingly, in the same study the retention rates of flavonoids increased with higher temperatures. The canned fruit pieces presented a catechin retention percentage of 9% at 115 °C, while at 130 °C the retention rate increased to 20%. The same was observed for chlorogenic acid (the retention rate of was 19% and 34% in canned mango pieces following treatment at 115 °C and 130 °C for 40 min, respectively), epicatechin (the retention rate of was 17% and 44% following treatment at 115 °C and 130 °C for 40 min, respectively) and gallic acid (the retention rate of was 11% and 27% following treatment at 115 °C and 130 °C for 40 min, respectively). Finally, in this study, the authors identified the following flavonoid compounds in the sugar syrup, indicating their leaching: catechin, chlorogenic acid, epicatechin, gallic acid and hydroxymethylfurfural (Arampath and Dekker 2020).

6.2.3.2 Procyanidins

Procyanidins are compounds members of the class of flavonoids that can also be affected by heat processing. A few studies reported losses in the procyanidin content of fruits during canning. In peach, thermal processing resulted in a 11% reduction

in procyanidin monomers, a 9% reduction in dimers, a 12% reduction in trimers, a 6% reduction in tetramers, and a 5% reduction in pentamers (Hong et al. 2004). During the storage of canned peaches, a further reduction in the procyanidin content was observed, and by 3 months of storage oligomers larger than tetramers were no longer detected (Hong et al. 2004). Similar results were reported for canned blueberries (Brownmiller et al. 2009), that showed a retention of 65% of total procyanidins when canned in syrup and of 78% when canned in water. These contents were further decreased during 6 months of storage, with 22% retained in berries canned in syrup and 32% in berries canned in water (Cindi Brownmiller et al. 2009). Procyanidins are water-soluble compounds, hence this decrease in concentration can be explained by the migration of procyanidins into the canning liquid, accounting for the losses observed during the thermal processing step.

In contrast, in the canning of apricots, procyanidins were stable during storage, being retained in the tissue upon processing (Le Bourvellec et al. 2018). Procyanidins can form complexes with food macromolecules, such as proteins and carbohydrates, being adsorbed onto cell walls by physicochemical interactions. This can explain the retention of procyanidins of high molecular weight within apricot tissues (Le Bourvellec et al. 2018).

6.2.3.3 Anthocyanins

Anthocyanins are compounds that also belong to the class of flavonoids and can present health benefits such as the prevention of cardiovascular disease (Di Gioia et al. 2020). These compounds are known to be sensitive to heat and can easily be converted to the colourless chalcone during heating (Petropoulos et al. 2019). Hence, the importance of studying the effects of canning on the content of anthocyanins in food products.

Fresh berries are naturally rich sources of anthocyanins. Thermal processing of blueberries resulted in losses of the total monomeric anthocyanin content (a loss of 28% in blueberries canned in syrup and 34% in berries canned in water), compared to the original levels found in fresh blueberries (Brownmiller et al. 2008). Nevertheless, in the canning of pitted Bing cherries, samples showed an apparent slight increase in total anthocyanin content when the values for syrup and cherries were combined (Chaovanalikit and Wrolstad 2004). In this study, approximately 50% of anthocyanins were transferred from the fruits into the syrup. Hence, heat processing of cherries did not result in a loss of total anthocyanins, as well as for total phenolics and antioxidant activity. This might be due to increased extraction efficiency in the softened fruits. The same leaching effect of anthocyanins to the syrup was observed in canned apricots (Le Bourvellec et al. 2018). In the canning of common Spanish beans, the reduction of the anthocyanin content was not significant ($p < 0.05$) (Pedrosa et al. 2015).

6.3 Conclusion

In general, the content of bioactive compounds after canning processing and storage is a net result of a combined increase in phytonutrient extractability, and a loss by degradation and leaching of compounds into the canning liquid (usually sugar syrup, brine or just water). Despite of the losses, canning processing in general can preserve a good part of the nutritional and physicochemical potential of fruits, vegetables and animal-products, which is of interest to the consumer.

For instance, canned fruits such as apricots show a rich nutritional profile and beneficial physiological effects, maintaining a high level of scavenging radical ability during long periods of storage, that can help in the prevention of lifestyle-related diseases and promote health. Industrial canning of beans is also an adequate treatment for obtaining a nutritious and healthy product since they retain bioactive components in amounts suitable for prebiotic effects, to enhance glycemic control or to reduce cancer risk of the consumer. Moreover, the canning process can improve food quality by delaying the onset of lipid oxidation and lengthening product's storage time.

Therefore, canned food can be a safe and healthy alternative to consumers who prefer to spend limited time on food preparation, as these convenient ready-to-eat products can retain, and sometimes even increase, the nutritional value and bioactive composition of raw foods.

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Chapter 7
Influence of Juice Processing on Food Bioactives

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7.1 Introduction

Fruit and vegetable juices are important sources of bioactive compounds, including phenolics (flavonoids, tannins, quinones, others), carotenoids, vitamins and minerals (Quan et al. 2020; Silva et al. 2020). Nowadays, juices have gained a great deal of attention from consumers due to bioactive compounds' health-promoting characteristics such as the preventative impact on hypertension, cardiovascular diseases, cancers and diabetes (Akyıldız et al. 2020; Quan et al. 2020). Several studies indicate that different types of juices have positive health effects such as reducing insulin resistance and blood pressure (Zhang et al. 2021a), anti-inflammatory effect (da Silva Haas et al. 2019) and preventive effect on bladder cancer (Mortada et al. 2020). However, bioactive components are sensitive to oxygen, light, pH and especially temperature. Due to reasonable price and efficiency, thermal pasteurization is still the most common preservation technique. However, nowadays, researchers focus on the new techniques on juice processing to obtain optimal sensorial and nutraceutical quality, food safety and diminish degradation of bioactive compounds (Guo et al. 2014). Recent studies show that bioactive compound content increases with nonthermal technologies in fruit juices (Nadeem et al. 2018; El Kantar et al. 2018; Baykuş et al. 2020; Koley et al. 2020).

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Bioactive compounds should first be released from fruit, absorbed by the gastrointestinal tract and eventually available for metabolism, so it is necessary to determine the stability and antioxidant potential of phenolic compounds affected by gastrointestinal digestion. (Quan et al. 2020). Through gastrointestinal digestion, processing and storage conditions play a vital role in releasing, converting, and absorbing health-related compounds (Linhares et al. 2020). Besides, in fruit juices, content and bioavailability of bioactive compounds can be affected by fruit type, juice matrix and processing conditions (Quan et al. 2020). Several researchers have investigated the effects of high hydrostatic pressure (HHP), pulsed electric field (PEF), ultraviolet light (UV), ultrasound (US), ohmic heating, cold plasma, high-pressure homogenization (HPH) on fruit juices bioactive compounds. Although nonthermal technologies are the main topics of recent studies based on the retention of bioactive compounds, research on clarification, fermentation and filtration processes is also available in the literature.

The present chapter reviews the studies investigating the effects of several juice processing steps on bioactive compounds' content and bioaccessibility. Thus, reviewed findings and key outcomes will guide researchers working on this issue and further investigations.

7.2 Polyphenols

7.2.1 Contents

Phenolic contents are significantly affected, particularly in juice processing, by various processes. Nowadays, researchers have mostly studied the impact of juice processing on phenolic contents. The effects of processes on phenolic compounds can change mainly depending on processing type and processing conditions. As well, the raw material that is subjected to the juice processing is another important factor.

After thermal pasteurization, a reduction in total phenolic content (TPC) has been reported for several juices such as a blend of carrot, carob, ginger, grape, lemon (Baykuş et al. 2020), carrot (Zhang et al. 2016) and orange (Velázquez-Estrada et al. 2013) juice. In contrast, in few studies, it was reported that pasteurization resulted with an increase in the phenolic content of pomegranate (Herceg et al. 2016; Guo et al. 2014), orange (Agcam et al. 2014) and mulberry (Yu et al. 2014) juices.

In a study, application of pectolytic enzymes for clarification of pomegranate juice increased the polyphenol content from 1601 mg/mL to 2972–3360 mg/mL based on the concentration. The small difference of polyphenol content of 10 and 12.5 mg/L enzyme-treated samples could be explained with protein-polyphenol complex due to higher protein content of samples treated with pectolytic enzymes with a concentration of 12.5 mg/L, which showed lower total polyphenol content

(Rinaldi et al. 2013). Additionally, Markkinen et al. (2019) reported that the amount of flavonol aglycones and the total amount of anthocyanin increased with the enzyme treatment in black chokeberry and sea buckthorn juices. These results showed that the enzyme treatment enhances the release of flavonols bound to cell wall matrices and polysaccharides.

Erkan-Koç et al. (2015) studied the effects of several proteins (albumin, casein and gelatin) and polysaccharide-based (chitosan and xanthan gum) clarification agents on bioactive compounds in pomegranate juice. Total phenolics (7.2–17.2%), hydrolyzable tannins (16.7–59.5%) and anthocyanin content (11.7–23.7%) were significantly reduced with the use of protein-based agents compared to natural sedimentation. However, a similar impact of natural sedimentation was observed with polysaccharide-based agents: chitosan and xanthan gum. The highest reduction in phenolic content with protein-based agents such as albumin, casein and gelatin resulted from the difference between isoelectric point and the juice samples' pH. Similar to this study, impacts of clarification agents on phenolic compounds were shown for apples (Oszmiański and Wojdyło, 2007; Taştan and Baysal, 2017) and chokeberry (Lachowicz et al. 2018) juices. In addition to this study, Taştan and Baysal (2017) found the phenolic content of clarified samples with chitosan and sample which traditionally clarified as the same. However, there was a significant difference in phenolic contents among juices stored under different temperatures and storage periods. Farahmand et al. (2017) showed that filtration significantly decreased the total phenolic contents. However, pasteurization had no significant impact. Besides, total flavonoid content (TFC) decreased by 8%, 16% and 18% during pasteurization, enzymatic treatment and filtration, respectively.

In addition to these processes, fermentation may affect the phenolic components of juices. Wu et al. (2020) found that gallic acid, epicatechin and phlorizin contents were increased by 87% 38% and 17% respectively, while ferulic acid and ellagic acid contents were decreased as a result of fermentation with lactic acid bacteria in apple juice. Markkinen et al. (2019) observed that anthocyanin content decreased with chokeberry's fermentation (Markkinen et al. 2019). Similarly, fermentation with *Lactobacillus plantarum* in bog bilberry juices caused a reduction in anthocyanins, phenolic acids and flavonols contents. Epicatechin and epigallocatechin contents decreased after fermentation. *L. plantarum* metabolism, oxidation and anthocyanin interaction might cause these reductions (Wei et al. 2018).

In another study, clarified pomegranate extract was treated with UV-C light as an alternative to heat treatment (90 °C, 2 min). The reduction of total monomeric anthocyanin content was higher compared to the heat-treated sample. The losses in monomeric anthocyanin content were 3.89% and 8.4% after UV-C treatment of 37.41 J/mL and 62.35 J/mL doses, respectively. Individual anthocyanin pigments decreased in the range of 8.1–16.3% after 62.35 J/mL of UV-C treatment, but the losses were between 15.4 and 28.3% after heat treatment. Pelargonidin 3-glucoside pigments showed different stabilities during UV-C and heat treatments. Additionally, there were no significant differences in total phenol content (Pala and Toklucu, 2011). Similarly, Islam et al. (2016) reported that UV irradiation did not cause any significant change in the total phenol content of apple juice. On the other hand,

epicatechin content decreased significantly depending on the UV dose, while catechin content increased remarkably. Additionally, phloridzin, which is the most abundant polyphenolic compound in apple juice, was found to be sensitive to UV-C treatment. Ultraviolet-light emitting diodes (UV-LEDs) treatment resulted in TPC increase in a blend of carrot, carob, ginger, grape and lemon juice. This increase could be attributed to the breakdown of polyphenols (complex phenolic components) into smaller phenolic components (Baykuş et al. 2020). However, Xiang et al. (2020) observed the reduction of TPC in apple juice by 3.03%, 5.40%, 8.97%, and 10.45% after exposure UVC-LEDs at 200, 400, 800, and 1200 mJ/cm^2, respectively. These reductions might have resulted from the degradation of polyphenolic compounds after UVC-LED irradiation.

Suárez-Jacobo et al. (2011) reported that different ultra-high pressure homogenization (UHPH) treatments did not result in significant differences in total polyphenol content of apple juices. However, chlorogenic acid content, which is the dominant constituent of hydrocinnamic acid, was significantly different in raw, pasteurized, and UHPH-treated juices according to their polyphenol profiles. The highest content of chlorogenic acid and 4-caffeinoylquinic acid were observed in pasteurized juices. Additionally, juices that were treated with UHPH at 300 MPa had higher chlorogenic acid content than those treated with 100 MPa. On the other hand, there were no significant changes in other polyphenols. Similar to these studies, Yu et al. (2014) observed that during UHPH treatments, anthocyanins, phenolic acids (gallic, caffeic, protocatechuic and *p*-coumaric acids), and quercetin aglycone contents were lower than the thermally pasteurized mulberry juices. However, high-pressure processed açaí juice showed higher anthocyanin content than thermally treated, but it was found to be lower than the control sample (da Silveira et al. 2019). Besides, Velázquez-Estrada et al. (2013) observed that orange juice's pasteurization process reduced polyphenol content compared to the raw and UHPH treated juices.

Moreover, flavonone contents of juices were found to be higher after the application of UHPH at either 200 or 300 MPa. It was pointed out that flavonoid improvement may occur due to decreasing the cloud fraction of juice and improving flavonoids' extractability. UHPH treatment can also be combined with other non-thermal methods. Varela-Santos et al. (2012) reported that the application of HHP to pomegranate juices at 350 and 550 MPa increased the total polyphenol content. The increase in total phenolics was explained with HHP treatment because it increased the extractability of some antioxidant compounds. Sauceda-Gálvez et al. (2012) studied the combined effect of UHPH and short-wave ultraviolet radiation in apple juice. Juices treated with only 200 MPa did not show any difference in polyphenol content. The polyphenol content of samples treated with 300 MPa UHPH and 14.3 J/ml of UV-C was reduced. When the UV-C dosage was increased, the polyphenol content was also increased. However, when juices were treated with 21.5 and 28.7 J/mL UV-C and 300 MPa UHPH, polyphenol content increased compared to the untreated sample.

It was also reported that the total phenolic content of supercritical CO_2 (SC-CO_2) treated pomegranate juice increased, while that of thermally treated juice decreased. However, there was only a slight change observed with high hydrostatic pressure

treatment. The amount of ellagic acid, which is the main phenolic compound found in pomegranate juice, did not change after these treatments. However, after 28 days of storage, only non-treated and heat-treated juices preserved ellagic acid content. HHP treatment caused a reduction from 53.6 to 30.8 μg/mL, while SC-CO_2 treatment caused a drastic reduction from 42.8 to 19.1 μg/mL (Bertolini et al. 2020). In some studies, researchers have studied the phenolic compounds of grape juice subjected to SC-CO_2 treatments. Del Pozo-Insfran et al. (2006) reported that thermal pasteurization negatively affected the anthocyanin and phenolic contents by reducing 16% and 26%, respectively. However, dense phase-CO_2 treatment did not change the anthocyanin and phenolic acid contents. In another study, Amaral et al. (2018) found that high-temperature short time and SC-CO_2 did not affect the whey-grape's total phenolic and total anthocyanin capacity juice beverages. There was only a slight reduction in (7.8%) total anthocyanin content after heat treatment when compared with control.

Another technology that is investigated in the case of its effects on bioactive compounds is PEF. It was reported that the PEF process did not change the total phenolic and anthocyanin content when compared with unprocessed pomegranate juices. However, after thermal treatment, significant increases were observed in phenolic content which were attributed to an increase in ellagitannins. However, after 12 weeks of storage, anthocyanin concentration slightly decreased, while phenolic concentration was not affected (Guo et al. 2014). Furthermore, increase of phenolic contents was reported for citrus juices (El Kantar et al. 2018) and apple (Carbonell-Capella et al. 2016) juices treated with pulsed electric fields. The increase of phenolic compounds might be explained by the electroporation of cells by PEF treatment (El Kantar et al. 2018). In addition to this, phenolic compounds such as gallic acid, vanillic acid and chlorogenic acid concentrations were reported to increase except syringic acid and neoeriocitrin after PEF treatment in orange juice (Agcam et al. 2014).

Some studies investigated the impact of the pomegranate juice processing line on phenolic compounds. Akyıldız et al. (2020) observed the lowest total monomeric anthocyanin content at the clarification stage, while the highest was at the pressing stage. The anthocyanin level was found to be four times higher after the concentration step because of the removal of excess water. Anthocyanin compounds were observed to decrease with the enzymation step, and this increase was also triggered by pasteurization, membrane filtration and clarifying with agents. Moreover, the highest gallic acid, ellagic acid and *p*-coumaric acid levels were observed at the pressing stage, but after filtration, they decreased by 45%, 58% and 57%, respectively. On the other hand, caffeic acid, ferulic acid and rutin levels were not affected by the pomegranate juice processing steps.

In another study, high-intensity ultrasound was applied to strawberry juice for 0 min (control), 4, 8, 12 and 16 min. However, the peak position of -OH compounds was observed in juice treated for 12 min. The decrease after 12 min could be explained by the highest extraction rate of phenolic compounds at 12 min treatment. The highest total flavonoid content was obtained in a 12 min treated sample with an 80% increase compared with the control sample. Additionally, catechin, gallic acid

and ellagic content in strawberry juice were increased with ultrasound treatment. Nevertheless, catechin content reduced by 41.5% compared with 12 min treated juice (Wang et al. 2019a). Another study by Wang et al. (2019b) investigated kiwifruit juice treated with US using the same conditions. However, the highest total phenolics and flavonoid content were found with 16 min treated juice, which increased by 108.65% and 105.56%, respectively, compared with control. Moreover, US treatment increased the catechin and gallic acid content, while ferulic acid content was not significantly changed, derived from a minor amount in kiwifruit juices. Cheng et al. (2020) evaluated the conventional thermal pasteurization (TP: 90 °C and 30 s), high-pressure processing (HPP:600 MPa, 4 °C and 300 s), US (50 °C, 750 W and 36 min) and microwave processing (MW:800 W, 80 °C and 70 s) on Mandarin (*Citrus unshiu*) juice. The retention of phenolic content in juice samples was 51.29%, 73.25%, 66.62% and 71.29% for TP, HPP, US and MW, respectively. TP resulted in the most significant reduction in total phenolic contents. Besides, the retention of phenolic compounds with the US could be explained by the generation of strong shear force, destruction of cell walls and release of polyphenols from the cell. Similar results were reported for Cape gooseberry juice (Ordóñez-Santos et al. 2017) and carrot-grape juice (Nadeem et al. 2018) with US (Table 7.1). Nadeem et al. (2018) explained that higher phenolic contents were observed after the US, and this increase was explained by the release of phenolic compounds from the cell wall due to cavitation in colloidal particles. After US and MW treatments, concentration of phenolic compounds (gallic acid, protocatechuic acid, caffeic acid, ferulic acid) increased except for *p*-hydroxybenzoic acid and erucic acid. However, flavonoids (narirutin, hesperidin, neohesperidin, nobiletin) except naringin decreased after all of the mandarin juices' treatments (Cheng et al. 2020). Similarly, microwave-treated blackberry juice showed higher total polyphenol, and monomeric anthocyanin contents compared to control (Pérez-Grijalva et al. 2018) and Martins et al. (2020) observed similar results on orange-juice milk beverages.

In addition to the nonthermal methods, dielectric barrier discharge plasma was applied to different juices. Farias et al. (2020) observed a 20% and 54% increase in total phenolic content at 50 and 600 kHz applications, respectively, on apple juices. However, it should be considered that overexposure to plasma treatment may result in the oxidation of phenolics. In another study, phenolic content was increased with the plasma processing on pomegranate juice. Also, researchers found that when processing time and gas flow rate increased, phenolic content increased. Ellagic acid content was observed three times higher than the untreated juices. Action mechanisms of plasma have been explained with the improvement of hydrolysis and depolymerization of ellagitannins with covalent bonds' breakage and inducement chemical reactions (Herceg et al. 2016). Recently, increase of phenolic content with the phased plasma has been reported on different juices including beetroot (Dzimitrowicz et al. 2020); camu-camu (de Castro et al. 2020) and chokeberry (Gan et al. 2021). However, de Castro et al. (2020) found that total monomeric anthocyanin content reduced after cold plasma treatment, and the higher frequency also resulted in lower total monomeric anthocyanin content.

Table 7.1 Impact of juice processing on polyphenols

Juice	Compounds	Treatment	Conditions	Key outcomes	References
Açaí	Anthocyanin Non-anthocyanin phenolics	HPP Thermal Pasteurization	450–600 MPa, 5 min, 20 °C 85 °C, 1 min	• Anthocyanin content was higher in HPP juices than thermally treated. • The highest concentration of non-anthocyanin phenolic compounds was observed in 500 MPA treated samples	da Silveira et al. (2019)
Apple	Polyphenol Chlorogenic acid 4-Caffeinoylquinic acid	UHPH Pasteurization	300 MPa–500 MPa, 20 °C 90 °C, 4 min	• There were no significant differences between UHPH and PA juices in terms of chlorogenic acid and 4-caffeinoylquinic acid contents, however significant differences were observed in raw juices. • Significant differences were observed in total polyphenol content for 300, 400, and 500 MPa UHPH treated juices.	Suárez-Jacobo et al. (2011)
Apple	Chlorogenic acid Phloridzin Total phenolic content	UV-C	240 mJ/cm^2	• Chlorogenic acid and phloridzin were observed to decrease by 19.3% and 50%, respectively. However, total polyphenol content didn't change significantly.	Islam et al. (2016)
Apple	Phenolics	Dielectric barrier discharge plasma	20 V, 15 min	• The highest increase in total phenolic content have been found 34% at 600 Hz.	Farias et al. (2020)
Beetroot	Phenolic compounds	Cold atmospheric pressure plasma	4.5 mL/min, 45 mA	• Depending on the power supplies as cathode, anode and electrode in cold plasma treatment, phenolic compounds have been increased by 15%,19% and 36%, respectively compared to untreated juices.	Dzimitrowicz et al. (2020)

(continued)

Table 7.1 (continued)

Juice	Compounds	Treatment	Conditions	Key outcomes	References
Blackberry	Total phenolic content Monomeric anthocyanin	MW	60 s	• Higher polyphenol content was observed in microwaved juices compared to the control sample. • There was a significant increase in total monomeric anthocyanin with microwave treatment.	Pérez-Grijalva et al. (2018)
Blend of carrot, carob, ginger, grape and lemon juice	Total phenolic content	UV-LED	280–365 nm, 60–100 min	• When compared with heat-treated samples, total phenolic contents were considerably increased (1.75 fold)	Baykuş et al. (2020)
Blueberry	Total phenolic content Anthocyanin	*Lactobacillus plantarum* fermentation	37 °C, 24 h, 3 g/100 mL, w/v	• After fermentation process, total phenolic and anthocyanin content have been increased by 43.32% and 15.38%.	Zhang et al. (2021b)
Carrot	Phenolic acid Flavonoids	HPP	600 MPa, 5 min	• Significant changes were found in phenolic acid profile. • The concentration of several flavonoids cirsimaritin, didymin, kaempferol 3-*O*-(6-acetyl- galactoside) 7-*O*-rhamnoside and prodelphinidin dimer was significantly increased after HPP treatment. • The higher flavonoid content was observed in multi-pass HPP treatment.	Szczepańska et al. (2020)
Carrot-grape	Total phenolic content Total flavonoid content	US	20 kHz, 5 s, 2–6 min	• The highest total phenolic and flavonoid content was found with 6 min ultrasound treated juices, while control samples were the lowest.	Nadeem et al. (2018)

Juice	Compounds	Treatment	Conditions	Key outcomes	References
Chokeberry	Total anthocyanin content Total anthocyanin	Clarification with several agents	0.1–1.0 g/L, 1–16 h	• The highest anthocyanin losses were found with Guar gum (GG) and Locust bean gum (LBG) as clarification agents. • The content of flavan-3-ols was significantly affected from doses and time. • However, the content of phenolic compounds was slightly affected by reaction time.	Lachowicz et al. (2018)
Grape	Total phenolic and flavanol/flavonol contents Anthocyanin profile	Maceration	0–3 doses of enzyme and 50–60 °C	• Among the different conditions, total phenolic content was found to be similar. • Catechin concentration was observed to be higher without the addition of enzymes. • Total quantified flavonols decreased with the addition of 1.5 mL/kg enzyme addition.	dos Santos Lima et al. (2015)
Grape	Total anthocyanins	Dense phase CO_2	34.5 MPa, 6.25 min, 30 °C	• Thermal heat treatment (75 °C, 15 s) had no significant effects on total anthocyanin and phenolic content, whereas dense phase processing reduced total anthocyanin and phenolic content by 16 and 26%, respectively.	Del Pozo-Insfran et al. (2006)
Lemon, Orange, Pomelo	Polyphenols	PEF	40 kV, 70 μs	• The release of polyphenols from the inner surface of orange, pomelo, and lemon juices was increased by 39, 66, and 135%, respectively.	El Kantar et al. (2018)
Mandarin	Phenolic content	HPP and US	600 MPa, 4 °C,300 s and 50 °C, 750 W, 36 min	• The most significant reduction of phenolic content was found under US (73.25%) retention, while HPP treatment (71.29%). • The minimum retention was observed with pasteurization process (51.39%).	Cheng et al. (2020)

(continued)

Table 7.1 (continued)

Juice	Compounds	Treatment	Conditions	Key outcomes	References
Mulberry	Phenolic acid	UHPH	200 MPa, 1 and 3 pass	• While one passes of UHPH reduced all five phenolic acids by 10–35%, three passes reduced them by 30–40%.	Yu et al. (2014)
Muscadine Grape	Total phenolic content	Clarification	3000 × *g*, 3 min	• The total phenolic content was observed to be significantly higher in the juice centrifuged after thermal treatment compared to the value before thermal treatment.	Martino et al. (2013)
Orange	Total phenolic content, syringic acid, neoeriocitrin, gallic acid and protocatechuic acid ethyl ester (PAEE)	PEF and heat Pasteurization	25.26 kV/cm, 1206 μs and 90 °C, 10 s	• Among the different treatments, there were significant changes detected in total phenolic contents. • The concentration of phenolic compounds was increased after PEF and HP treatment, except syringic acid and neoeriocitrin. • Gallic acid and PAEE contents were not significantly different in juices treated by PEF or HP.	Agcam et al. (2014)
Pomegranate	Anthocyanin Total phenolic content	UV-C	12.47–62. 45 J/mL, 1–5 pass	• The total monomeric anthocyanin content did not significantly change after UV-C treatment. • UV-C treatment reduced individual anthocyanin pigments to 8.1% and 16.3%, while thermal treatment reduced them to 15.4–28.3%. • Total phenolic content was not significantly change after different UV dose.	Pala and Toklucu (2011)
Pomegranate	Total anthocyanin	Hollow-fiber ultra-filtration	0–2.5 bar, 900 mL/min	• The anthocyanin concentration was reduced to 164% with filtration.	Cassano et al. (2011)

Juice	Compounds	Treatment	Conditions	Key outcomes	References
Pomegranate	Total phenolic content Ellagic acid	SC-CO_2 Heat treatment HHP	12.7 MPa, 45 °C, 40 min 90 °C, 1 min and 600 MPa, 3 min	• The SC-CO_2 caused a 22% increase in TPC, while thermal treatment resulted in 15% decrease. • HHP treated juices did not show any significant difference in TPC when compared with the control sample. • At the end of 28 days of storage ellagic acid content was only preserved in thermal-treated and non-treated samples.	Bertolini et al. (2020)
Pomegranate	Ellagic acid Protocatechuic acid, caffeic acid and punicalagin 2	Cold atmospheric plasma jet	25 kHz, 2.5 kW, 3–7 min, 0.75–1.25 dm^3/min	• While protocatechuic acid, caffeic acid and punicalagin 2 were decreased, ellagic acid content was increased.	Herceg et al. (2016)
Pomegranate	Total phenolic Total anthocyanin	US	30 kHz,60 min	• The total phenolic content was not significantly changed after US treatment. • Total anthocyanin content was reduced to 22.6%.	Aliasghari Aghdam et al. (2015)
Pomegranate	Phenolics, punicalagins	PEF Pasteurization	100 L/h, 60 kV 88 °C, 30 s	• During 12 weeks of storage, anthocyanin content was slightly decreased, while total phenolic content was not altered in PEF samples. • Punicalagins concentration was increased after thermal treatment.	Guo et al. (2014)
Strawberry	Flavonoid Phenolic compounds	High intensity ultrasound	20 kHz, 400 W, 4–16 min	• The highest total polyphenol and flavonoid content was observed in 12 min treated samples. • Ferulic acid content was not changed.	Wang et al. (2019a)
Tomato	Total phenolic content	UV	2.16 J/m^2, 30 min	• Total phenolic content was significantly increased.	Bhat (2016)

(continued)

Table 7.1 (continued)

Juice	Compounds	Treatment	Conditions	Key outcomes	References
Whey Grape	Phenolic compounds Anthocyanins	SC-CO_2 Pasteurization	16 MPa	• Total phenolic and anthocyanin content was not affected by treatments. • Total anthocyanin level was decreased with pasteurization.	Amaral et al. (2018)

HHP: High Hydrostatic Pressure, *HPP*: High-Pressure Process, *MW*: Microwave, *PEF*: Pulsed Electric Field, *SC-CO_2*: Supercritical CO_2, *US*: Ultrasound, *UV*: Ultraviolet

7.2.2 *Bioaccessibility*

Few studies have reported the effect of juice processing on improving the bioaccessibility of phenolic compounds such as HPH in pomelo (Quan et al. 2020) and orange (Stinco et al. 2020) juices, the US in exotic fruit juices (Buniowska et al. 2017) and PEF for fruit juice beverage (Rodríguez-Roque et al. 2015).

The total phenolic bioaccessibility (TPB) was found to be between 71% and 86% in high-pressure homogenized carrot, apple, and peach mixed juices. Polyphenols are generally found in vacuoles and different intracellular organelles of plant cells. After cell disruption, polyphenolics are bound to pectins and proteins due to diffusion out of vacuoles (Wellala et al. 2019). In a recent study, high-pressure homogenization resulted in a 1.43 fold increase in total flavonoid bioaccessible content in mandarin juices compared to the untreated juice (Sentandreu et al. 2020). Stinco et al. (2020) observed that pasteurization and HPH treatment at 150 MPa did not significantly affect the bioaccessibility of flavonoids in orange juice. On the contrary, the bioaccessibility of individual phenolic compounds was reduced by the process of HPH in pomelo juices, while there was a minor change in kiwi juices (Quan et al. 2020).

Linhares et al. (2020) observed 2-fold higher (132%) anthocyanin bioaccessibility with thermal sterilization (UHT) in açai juice. However, the combination of US and UV-pulse light treatment decreased bioaccessibility to 40% compared to control. Thermal processing is reported to result in higher available phenolic concentration due to modification of cellular walls. On the other hand, TPB was lower than the control with thermal treatment. Wang et al. (2020a) reported higher bioaccessibility of phenolic compounds in ultraviolet-assisted ultrasonic pre-treated probiotic mango juice (UFJ) compared to the fresh juice (FJ). The bioaccessibility of phenols in UFJ changed from 9.71% to 22.66% and 19.23% at day 0 and day 30, respectively, while FJ reduced to 10.58% and 4.8%. A similar result was found by de Sousa Carvalho et al. (2020), who reported that after high energy ultrasound processing bioaccessibility of total monomeric anthocyanins was improved in açai and buriti juices. Researchers reported that depending on the increase in viscosity after the ultrasound process, bioactive compounds may become less accessible to react with digestive enzymes. Moreover, this situation results in higher bioavailability of bioactive compounds.

PEF and high voltage electrical discharge (HVED) treatments were reported to improve phenolic compounds and anthocyanin bioaccessibility in stevia-sweetened mango and papaya juices, while ultrasound treatment only improved phenolic content (Buniowska et al. 2017). In HVED treatment, increasing energy input from 32 kJ/kg to 256 kJ/kg resulted in a significant reduction in total phenolic bioaccessibility. However, increasing the energy input to 256 J/kg in PEF and ultrasound treatment, phenolic compounds' bioaccessibility was increased. These technologies might promote the release of phytochemicals from the matrix with no formation of electrolysis products. Besides, higher bioaccessibility was observed for anthocyanins in 32 kJ/kg of HVED treated juices (Table 7.2). Rodríguez-Roque et al. (2015)

Table 7.2 Effect of juice processing on bioaccessibility of polyphenols

Juice	Compounds	Treatment	Conditions	Model	Key outcomes	References
Apple	Total phenolic content	HPH Thermal Treatment	250 MPa, 10 min 80 °C and 90 °C, 30 min	Gastrointestinal digestion	• Total phenolic content was decreased between 21.9–37.3%. However, 90 °C thermally treated samples did not significantly change during intestinal digestion. • During gastric digestion, a small change was observed on HPH treated samples.	He et al. (2016)
Exotic fruit juice- stevia mixture	Total phenolic content Anthocyanins	HVED and PEF US	32 kJ/kg and 256 kJ/kg, 25 kV/cm 400 W, 24 kHz	Three-stage in vitro digestion model	• Bioaccessibility of TPC was significantly increased without the formation of electrolysis products. • Higher energy input reduced anthocyanin bioaccessibility on HVED, while ultrasound and PEF resulted with an increase.	Buniowska et al. (2017)
Grape	Phenolic compounds	HPH Thermal Treatment	250 MPa, 10 min 80 °C and 90 °C, 30 min	Gastrointestinal digestion	• Caffeoyl-tartaric acid bioaccessibility increased with HPH treatment, but epicatechin, proanthocyanidin, and protocatechuic- glucoside content did not change. • Food matrix affected the phenolic compounds' bioaccessibility. • Bioaccessibility of caffeoyl-tartaric acid and proanthocyanidin improved by 85.5–87% and 1.2–1.5 fold, respectively.	He et al. (2016)
Mandarin	Flavonoids	Pasteurization HPP	65 °C and 85 °C, 15 s 150 MPa	In vitro digestion	• Pasteurization resulted in better flavonoid bioaccessibility, besides at 65 °C two fold increase was observed. • HPP treatment resulted in a 1.43 fold increase. However, it was not statistically significant.	Sentandreu et al. (2020)

Juice	Compounds	Treatment	Conditions	Model	Key outcomes	References
Mixed juice	Total phenolic content	HPH	140 mPa, 2 L/h	In vitro digestion	• Total phenolic bioaccessibility was affected by the juice ratio. • Lipid in the food matrix did not affect the polyphenol bioaccessibility.	Wellala et al. (2019)
Orange	Flavonoids	HPH Pasteurization	150 MPa 92 °C, 30s	In vitro digestion	• Flavonoid bioaccessibility was not significantly affected by pasteurization treatments. • Vicenin-2 bioaccessibility only significantly changed with HPH treatment.	Stinco et al. (2020)
Orange	Phenolic compounds	HPH Thermal Treatment	250 MPa, 10 min 80 °C and 90 °C, 30 min	Gastrointestinal digestion	• Naringin, naringenin-trisaccharide and luteolin-rutinoside bioaccessibility were improved with thermal treatment and HPP. • Quercetin bioaccessibility was increased in all of the treatments except 90 °C thermal treatment.	He et al. (2016)
Pomelo	Phenolic compounds	HPH Thermal treatment	250 MPa 80 °C, 30 min and 90 °C, 30 s	In vitro simulated gastrointestinal digestion	• Thermal treatment at 80 °C and soy milk addition decreased bioaccessibilities of phenolic compounds. • Naringenin-rutinoside, isorhamnetin-rutinoside, and feruloyl-glucoside bioaccessibilities decreased between 8.0% to 9.0%, naringenin-rutinoside-glucoside, proanthocyanidin, and proanthocyanidin-glucoside bioaccessibilities were not significantly changed.	Quan et al. (2020)
Probiotic mango	Total phenolic content	US-UV pretreatment	10 min, 600 W and 254 nm, 20 kHz	In vitro-simulated gastrointestinal digestion	• The bioaccessibility of fresh juice decreased day by day on fresh juice. However, treated samples' bioaccessibility decreased after 15 days of storage.	Wang et al. (2020a)

(continued)

Table 7.2 (continued)

Juice	Compounds	Treatment	Conditions	Model	Key outcomes	References
Water, milk or soymilk-fruit juice beverage	Phenolic compounds	Thermal treatment HIPEF High Pressure	90 °C, 60 s 35 kV/cm, 200 Hz 400 MPa	In vitro gastrointestinal digestion	• The process did not alter the several phenolic compounds. • The lowest bioaccessibility was found on thermal-treated samples. • Bioaccessibility of juice was affected by the food matrix. • Bioaccessibility of juices was decreased with the combination of milk or soy milk. • After thermal application, several phenolic compounds bioaccessibility were increased, such as caffeic and p-coumaric acids	Rodríguez-Roque et al. (2015)

HHP High Hydrostatic Pressure, *HIPEF* High-Intensity Pulsed Electrical Field, *HPH* High-Pressure Homogenization, *HPP* High-Pressure Process, *HVED* High Voltage Electrical Discharges, *PEF* Pulsed Electric Field, *US* Ultrasound, *UV*: Ultraviolet

examined fruit juice beverages based on water, milk and soymilk treated with high-intensity pulsed electric fields (HIPEF), HPP, and thermal treatment. Improvement up to 38% in bioaccessibility was detected for various phenolic compounds such as caffeic and *p*-coumaric acids, hesperidin and rutin in water fruit juice beverages. However, the lowest bioaccessibility of phenolic compounds was observed in thermally-treated beverages. Thermo-labile phenols are lost or polymerized due to high temperatures. Additionally, in untreated and thermally treated beverages, rutin was not bioaccessible, but for HIPEF and HPP treated drinks, it was found to be 7.2% and 8.4% bioaccessible, respectively. It has also been reported that phenolic compound bioaccessibility is affected by the food matrix. For example, the highest bioaccessibility was found in water-fruit juice beverages followed by soymilk-fruit juice beverages. In another study, He et al. (2016) found that HPH decreased phenolic bioaccessibility more than thermal treatment on apple juice. HPH was reported to reduce the individual phenolic bioaccessibility by 22.6–26.3%. However, by thermal treatment at 80 °C, chlorogenic acid bioaccessibility was reduced by 12%, whereas bioaccessibility of phloridzine, EGCG and hesperidin were not significantly affected. On the other hand, in grape juice, HPHP increased the bioaccessibility of caffeoyl-tartaric acid. Additionally, it was reported that caffeoyl-tartaric acid and proanthocyanidin bioaccessibility were improved by 85.5–87% and 1.2–1.5-fold, respectively. Similar to the study of Rodríguez-Roque et al. (2015), grape juice's phenolic bioaccessibility was reduced with soy milk, skimmed and whole milk with 31.6–83.9%, 9.8–52.5% and 16.7–34.3% respectively.

7.3 Carotenoids

7.3.1 Contents

Various juice processing techniques may have different effects on the nutritional contents. Moreover, the stability of carotenoids was found to vary in response to different processing applications. On the other hand, effects of novel processing techniques on carotenoid content were also investigated in recent studies. Abliz et al. (2021) investigated the dynamic high-pressure microfluidization treatment on the carotenoid content of sea buckthorn juice processing. They reported that α-carotene and β-carotene could be protected by dynamic high-pressure microfluidization treatment in sea buckthorn juice. However, the total carotenoids content was decreased in the samples. Similarly, Koley et al. (2020) reported that high-pressure microfluidization processing resulted in an increase in carrot juice carotenoid content. The concentrations of β-carotene and lutein significantly increased with an increasing number of pass (Koley et al. 2020). In another study, the concentration of individual and total carotenoids was differentially affected depending on the orange cultivar and HPP treatment (De Ancos et al. 2020). The researchers reported that the application of 200 MPa and 400 MPa increased the concentration of phytoene (40% and 97%) and phytofluene (9- and 12-fold) in Navel-orange juice compared to untreated freshly-prepared juice (Table 7.3).

Table 7.3 Impact of juice processing on carotenoids

Juice	Compounds	Treatment	Key outcomes	References
Carrot	Total carotenoids Lycopene Lutein	Blanching (100 °C for 4 min) Sonication (frequency 20 KHz and amplitude level 70%, at 15 °C for 2 min)	• The total carotenoids, lycopene and lutein in blanched samples significantly increased. However, this increase was higher in blanched and sonicated samples.	Jabbar et al. (2014)
	β-carotene lutein	High-pressure microfluidization (34.47 MPa, 68.95 MPa and 103.42 MPa and with three different passes)	• The microfluidization processing significantly improved the carotenoids contents in carrot juice. • The concentrations of β-carotene and lutein significantly increased with the increasing number of pass.	Koley et al. (2020)
Chili	Capsorubin, Capsanthin, Zeaxanthin, β-Cryptoxanthin, β-Carotene,	Ultrasonication (40 kHz for 30 min, 240–480 W) Microwave (1 min at 300, 400, 500, 600, and 700 W) Heat (30 min at 60, 70, 80, 90, and 100 °C) Light treatments (2000 lux, 10 h)	• Low-power ultrasonic and microwave treatments increased the carotenoid and capsaicinoid contents but decreased at high-power treatments. • Ultraviolet light significantly diminished the contents of carotenoids and capsaicinoids. • The stability of carotenoids and capsaicinoids were significantly improved in an oil-based system	Zhang et al. (2020)
Sea buckthorn	α-carotene β-carotene	High-pressure microfluidization (50, 75, 100, 125, 150 MPa and different passes (1, 2 and 3)).	• α-carotene and β-carotene were preserved by dynamic high-pressure microfluidization treatment in sea buckthorn juice. • The total carotenoids content was decreased in the samples.	Abliz et al. (2021)
Mandarin	Total carotenoid content	Conventional thermal pasteurization (90 °C and 30 s), High-pressure processing (600 MPa, 4 °C and 300 s), Ultrasound processing (50 °C, 750 W and 36 min) Microwave processing (MW: 800 W, 80 °C and 70 s).	• Ultrasound, microwave processing and high-pressure processing preserved total carotenoid content better than conventional thermal pasteurization. • The total carotenoid content was the highest in the ultrasound processed juice with 102.45% retention.	Cheng et al. (2020)
Mango	16 different carotenoid Compounds including 11 xanthophylls and 5 carotenes	Ultrasound combined with ultraviolet treatment (10 min 600 W)	• The total carotenes significantly increased from 49.04% to 95.15%, whereas xanthophylls decreased from 50.96% to 4.85%.	Wang et al. (2020b)

Orange	Phytoene Phytofluene Lycopene β-carotene β-Cryptoxanthin Zeaxanthin Antheraxanthin All-E-Violaxanthin 9-Z-Violaxanthin	High-pressure processing (200 MPa/25 °C/1 min and/or 400 MPa/40 °C/1 min)	• The concentration of individual and total carotenoids was differentially affected depending on the orange cultivar and high-pressure processing treatment.	De Ancos et al. (2020)
Orange	Lutein Zeaxanthin β-Cryptoxanthin α-carotene β-carotene	Ultra high-pressure homogenization processing (two inlet temperatures:10 and 20 °C and processed at 100, 200 and 300 MPa)	• The level of depletion of carotenoids significantly depended on the pressure applied during processing. • In general, at higher pressures (and consequently, higher temperatures) a higher depletion in carotenoid content was observed	Velázquez-Estrada et al. (2013)
Sea buckthorn	α-carotene β-carotene	High-pressure microfluidization (50, 75, 100, 125, 150 MPa and different passes (1, 2 and 3)).	• α-Carotene and β-carotene were preserved by dynamic high-pressure microfluidization treatment in sea buckthorn juice. • The total carotenoids content was decreased in the samples.	Abliz et al. (2021)
Tomato	β-cryptoxanthin lycopene zeaxanthin β-carotene α-carotene	Thermal processing (70, 80 and 90 °C)	• The thermal processing significantly decreased the concentration of the carotenes. • The degree of thermoresistance in the phytonutrients followed the order of β-cryptoxanthin>lycopene>β-carotene>α-carotene	Ordóñez-Santos and Martínez-Girón (2020)

Nevertheless, high-pressure processing treatments decreased 16% total carotenoid content, mainly lycopene in Cara Cara-orange juice (De Ancos et al. 2020). On the other hand, UHPH processing at higher pressures (and consequently, higher temperatures) resulted in a higher depletion in carotenoid content of orange juice (Velázquez-Estrada et al. 2013). More recently, US, MW and HPP preserved total carotenoid content better with retention rates of 102.45%, 89.34%, 80.32%, and 80.32%, respectively, compared to the conventional thermal pasteurization (retention rate: 59.884%) of mandarin juices. The total carotenoid content was the highest in the ultrasound processed juice due to ultrasound cavitation and mechanical effects (Cheng et al. 2020). Similarly, after ultrasound combined with ultraviolet treatment, the total carotenes significantly increased from 49.04% to 95.15%, whereas xanthophylls decreased from 50.96% to 4.85%, only all-trans-zeaxanthin remained unchanged in mango juice (Wang et al. 2020b). Zhang et al. (2020) investigated carotenoids' stability in chili juice in various food matrices (oil and water) and under various processing conditions, including ultrasonication, microwave, heat, and light treatments. They reported that low-power ultrasonic and microwave treatments increased the carotenoid and capsaicinoid contents in chili juice but decreased at high-power treatments. On the other hand, ultraviolet light significantly diminished the contents of carotenoids and capsaicinoids in chili juice (Zhang et al. 2020). Moreover, the same researchers observed that carotenoids and capsaicinoids' stability could be significantly improved in an oil-based system (Zhang et al. 2020).

It is known that thermal processing significantly decreases the concentration of carotenes. The thermoresistance of bioactive compounds and thus the degree that they are affected during thermal treatments may be different. For example, the degree of thermoresistance of phytonutrients in tomato juice was observed to follow the order of β-cryptoxanthin > lycopene > β-carotene > α-carotene (Ordóñez-Santos & Martínez-Girón, 2020). In another study, α-carotene, β-carotene, β-cryptoxanthin, zeaxanthin, and lutein decreased by 44%, 38%, 32%, 23%, and 21% after thermal pasteurization of orange juice, respectively (Velázquez-Estrada et al. 2013).

On the other hand, processing steps also affect the carotenoid contents. However, the effect of processing steps on the carotenoid contents is limited. Ma et al. (2015) investigated the effects of different peeling methods (hand peeling, hot water peeling, lye peeling, composite phosphate peeling), blanching treatment factors (blanching time, blanching temperature, the amount of ascorbic acid added), enzyme liquefaction treatments (pectinase treatment, cellulase treatment, pectinase before cellulose, cellulose before pectinase, pectinase and cellulose mixed treatment) and different sterilization temperatures (85, 100, and 121 °C) on the contents of α-carotene, *β*-carotene and lutein in carrot juice. They reported that composite phosphate peeling, blanching temperature of 86 °C for 10 min and an addition of 0.25% of ascorbic acid, single enzyme treatment or mixed enzyme treatment showed the best effect among the methods used. However, excessive blanching temperatures and times, using pectinase and cellulase at the same time and high-temperature sterilization enhanced the loss of carotenoids. More recently, Servent et al. (2020) reported that the concentration step caused an increase in the carotenoid content by

up to 19-fold, and the diafiltration step allowed the carotenoid purity to be multiplied by 5. In another study, after homogenization, carotenoids of carrot juice were significantly decreased from 61.87 to 58.76 mg/kg, respectively, resulting from the oxidation or degradation of carotenoids (Liao et al. 2007).

7.3.2 Bioaccessibility

It is more important to determine carotenoids' bioaccessibility in juice, which is mainly affected by different processing approaches rather than their concentration (Barba et al. 2017). Carotenoid bioaccessibility is indicated to be very low due to the crystalline nature of the carotenoids and their entrapment within chromoplasts. In this sense, the bioavailability of carotenoids can be improved using specially designed oil-in-water emulsions. For example, tomato juice including excipient emulsion was reported to significantly enhance the lycopene bioaccessibility (12.5%), leading to more mixed micelles to solubilize the carotenoids. The lowest lycopene bioaccessibility was observed in the raw tomato juice without any excipient emulsion (7.5%) (Salvia-Trujillo & McClements, 2016). Zhong et al. (2019) investigated the novel food processing techniques, i.e., on carotenoid bioaccessibility and Caco-2 cell uptake from tomato and kale-based juices compared with conventional thermally treated and raw (non-processed) juices. PEF increased lycopene bioaccessibility by 150% in tomato juice, whereas β-carotene bioaccessibility decreased by 44% compared to raw juice (non-processed). This decline could be explained by the incomplete inactivation of lipoxygenase after PEF treatment, whereas β-carotene was protected in raw juice by lipoxygenase remaining sequestered (Zhong et al. 2019). Thermal treatment and pulsed electric field degraded β-carotene and lutein in kale juices (Zhong et al. 2019). In another study, the digesta from high-pressure processing and pressure-assisted thermal processing (700 MPa for 5 min at 30 and 100 °C, respectively) samples had significantly higher levels of all-trans-β-carotene compared to the digesta from raw tomato juice and thermally processed juice (0.1 MPa, 100 °C for 5 min) (Gupta et al. 2011). Besides, lycopene's bioaccessibility was limited regardless of the processing method (Gupta et al. 2011) (Table 7.4). Jayathunge et al. (2017) investigated the thermal and non-thermal processing on lycopene in vitro bioaccessibility of tomato juice. They reported that blanching followed by HIPEF showed a significant release of trans-(4.01 μg/g) and cis-(5.04 μg/g) lycopene, achieving 15.6% total lycopene bioaccessibility. More recently, homogenization in 150 MPa sample had a positive impact on bioaccessibility (~80%) and carotenoid bioaccessible content (from 3- to five-fold) of carotenoids lutein, zeaxanthin, β-cryptoxanthin, α-carotene, β-carotene, lycopene, phytoene and phytofluene compared to fresh juice (Stinco et al. 2020). Bioaccessibility of epoxycarotenoids had a five-fold increase by high-pressure homogenization (Stinco et al. 2020). In another study, treatment of different thawing conditions (microwave, room and refrigeration temperature) on the carotenoids bioaccessibility in ultra-frozen orange juices were investigated. Microwave thawing

Table 7.4 Effect of juice processing on bioaccessibility of carotenoid

Juice	Compounds	Treatment	Model	Key outcomes	References
Kale	β-carotene, Lutein chlorophyll a + b	Pulsed electric field (35 kV/cm, 500 Hz) Ohmic heating, High-pressure processing	In vitro Caco-2 cell	• Thermal treatment and pulsed electric field degraded β-carotene and lutein.	Zhong et al. (2019)
Orange	Total carotenoid content Violaxanthin ester Luteoxanthin ester	Thermal pasteurization, Pulsed electric fields, High-pressure processing, Subsequent ultrasonication	In vitro	• The *in vitro* solubilization and the micellarization efficiency was greatly enhanced by ultrasound, the latter by approximately 85.3–159.5%.	Etzbach et al. (2020)
Orange	Carotenoids and epoxycarotenoids	High-pressure homogenization (150 MPa) and pasteurization (92 °C for 30 s and 85 °C for 15 s)	In vitro	• High-pressure homogenization had a positive effect on the bioaccessible content (from 3- to 5-fold) of carotenoids lutein, zeaxanthin, β-cryptoxanthin, α-carotene, β-carotene, lycopene, phytoene and phytofluene compared to fresh juice. • Bioaccessibility of epoxycarotenoids had a 5-fold increase by high-pressure homogenization.	Stinco et al. (2020)
Ultrafrozen orange juices	Lutein Zeaxanthin β-cryptoxanthin α-carotene β-carotene	Different thawing conditions (microwave, room and refrigeration temperature)	In vitro	• Microwave thawing treatments of these juices showed the highest values for the % of relative bioaccessibility of lutein, zeaxanthin, β-cryptoxanthin, α-carotene, β-carotene compared to the other thawing conditions and fresh orange juices	Stinco et al. (2013)
Tomato	Lycopene	Thermal treatment (90 °C, 10 min) with nanoemulsion	In vitro	• Lycopene bioaccessibility increased with the addition of nanoemulsions in tomato juice (12.5%) • The lowest lycopene bioaccessibility was observed in the raw tomato juice without any excipient emulsion (7.5%)	Salvia-Trujillo and McClements (2016)
Tomato	Lycopene, β-carotene, Lutein phytoene phytofluene	Pulsed electric field (35 kV/cm, 250 Hz) Ohmic heating, High-pressure processing	In vitro Caco-2 cell	• Pulsed electric field increased lycopene bioaccessibility by 150%, whereas reduced β-carotene bioaccessibility by 44% compared to raw juice (non-processed). • All processing treatments enhanced lutein uptake. • Pulsed electric field and ohmic heating enhanced total lycopene and lutein delivery from tomato juice to Caco-2 cells.	Zhong et al. (2019)

Tomato		Moderate intensity pulsed electric field (4 µs, 1 kV/cm, 0.1 Hz) Blanching (90 °C/ 2 min) Ultrasonic (7 min 20% amplitude) high intensity pulsed electric field (1500 µs, 35 kV/cm)	In vitro	• Pulsed electric field treatment increased the lycopene bioaccessibility. • Blanching and thermal processing decreased lycopene released during digestion. • The blanching treatment followed by high intensity pulsed electric field showed a significant release of trans-(4.01 µg/g) and cis-(5.04 µg/g) lycopene, achieving 15.6% total lycopene bioaccessibility.	Jayathunge et al. (2017)
Tomato	Lycopene β-Carotene	Thermal processing (93 °C/60 s) High pressure (500–700 MPa/30 °C/0–10 min) Pressure assisted thermal processing (500–700 MPa/100 °C/10 min)	In vitro	• The digesta from high-pressure processing and pressure-assisted thermal processing samples had significantly higher levels of all-trans-β-carotene than the digesta from raw juice and thermally processed • The bioaccessibility of lycopene was limited regardless of the processing method used	Gupta et al. (2011)

treatments of the juices showed the highest values for the % of relative bioaccessibility of lutein, zeaxanthin, β-cryptoxanthin, α-carotene, β-carotene when compared to the other thawing conditions and fresh orange juices (Stinco et al. 2013). In general, nonthermal processing technologies have an important role in vitro digestion process since they enable carotenoids to be released from plant tissues. These innovative technologies could play an important role in producing food products with high levels of bioaccessible carotenoids.

7.4 Vitamins

7.4.1 Contents

Vitamins are sensitive nutritional compounds to several factors such as oxygen, light, thermal processes etc. In the case of vitamins, fruits and vegetables are rich in vitamin C, so literature studies are commonly conducted based on this vitamin. Several studies have indicated that vitamin C is destroyed mainly due to the presence of oxygen during processing because of oxidation reactions. Moreover, heat treatments are other causes of vitamin losses. In this case, thermal processes, which are widely applied to extend the shelf life of food products, can cause irreversible changes and significant losses in terms of heat-sensitive components (Plaza et al. 2006). Compared with heat treatments, higher vitamin C content can be achieved by applying nonthermal technologies such as HPP and HIPEF (Rodríguez-Roque et al. 2015). In red fruit juice processing, it was reported that thermal applications at atmospheric pressure conditions had a disintegrating effect on bioactive components. However, combination of high pressure (HP) application with temperature had no significant effect on these components (Verbeyst et al. 2012).

Other than conventional processes, application of HP was reported for some fruit juices to enhance the vitamin content. While traditional thermal treatments did not significantly affect total carotenoid or vitamin A content, HP treatment of freshly squeezed orange juice led to an increased vitamin A value (38.74%). However, HP application did not cause any significant change in total vitamin C content (Sánchez-Moreno et al. 2005). The increase in the release of vitamin C (11.4–43.6%) of comminuted orange after applying HHP treatments was reported by Escobedo-Avellaneda et al. (2015). It was stated that this might be due to the increased release of vitamin C as a result of cellular disruption after HPP application. Hence, it becomes more easily accessible and measurable (Escobedo-Avellaneda et al. 2015).

In contrast, several studies reported decreased vitamin C content after thermal and nonthermal applications. One research was conducted with pasteurization, high pressure and pulsed electric fields application by Sánchez-Moreno et al. (2005). Even though the decrease in vitamin C content was lower than 9%, treatment with higher temperatures tended to show a higher decrease in vitamin C content (Sánchez-Moreno et al. 2005). The reduction in vitamin C content of juices was also shown by the study of De Ancos et al. (2020). They applied HPP at 200 MPa and 400 MPa

to whole oranges and orange juices, respectively. Although the vitamin C content in the juice of treated oranges showed <5% reduction, application of 400 MPa after juicing of untreated oranges caused a significant reduction in Navel orange juice. The high reduction level may result from the mechanical juicing method and/or the pressure-induced enzyme activation of food enzymes. In a study that investigated the effect of refrigeration on vitamin C content of HP, PEF, and low pasteurization (LPT) treated orange juices, it was reported that after application of HP, PEF and LPT, orange juices showed a quite low decrease in ascorbic acid and total vitamin C content (˂8%) (Plaza et al. 2006). Higher vitamin degradation levels reported in the literature were mostly based on application conditions. Although the correlation was not reported with the process parameters, processing factors may affect vitamin loss levels. Higher ascorbic acid losses were reported after 400 MPa applications (84%) compared with the application of 550 MPa (18%) to muscadine grape juice (Del Pozo-Insfran et al. 2007). Losses of vitamins were reported for different non-thermal processes as well as for high-pressure applications. In fruit juice (mango and papaya)-stevia mixture, after application of different technologies such as HVED, PEF and US, ascorbic acid was found to be significantly lower than the control (Buniowska et al. 2017). Another study investigating the vitamin C content of fruit juice-based beverages after processing was carried out by Rodríguez-Roque et al. (2015). In this study, it was concluded that vitamin C concentration was significantly influenced by high-intensity pulsed electric fields, high-pressure processing and thermal treatment, but not by the food matrix (Rodríguez-Roque et al. 2015). In another study conducted with milk- and soy-based fruit beverages, the ascorbic acid content in HPP applied whole milk−fruit beverage was not significantly different from the control. In all tested beverages, thermal treatment caused a significant reduction in ascorbic acid content (Cilla et al. 2012).

Hydrodynamic cavitation is another process that influences vitamin C in juice processing. A study investigating the effect of nonthermal hydrodynamic cavitation on the stability of bioactive compounds in orange juice stated that vitamin C in untreated orange juice was significantly higher than the hydrodynamic cavitation treated orange juices. While the degradation of vitamin C was significantly lower compared to the thermal processes, it was found to be correlated with the generation of hydroxyl radicals at the bubble's surface. Degradation of vitamin C may occur due to the reaction of vitamin C with hydroxyl radicals (Katariya et al. 2020). There are several studies based on the effect of US on the vitamin C content of fruit juices. One of them is carried out by Ordóñez-Santos (2017). They observed a significant decrease in Cape gooseberry juice's ascorbic acid content after both US (25.45%–78.81%) and heat pasteurization (24.40%) treatments compared to untreated samples. It was also proved by Santhirasegaram et al. (2013) for Chokanan mango juice. Similarly, they stated that ascorbic acid content was significantly decreased by sonication and thermal treatment compared to control. While the highest degradation in ascorbic acid was reported after HP treatment at 65%, the lower degradation was reported after sonication at 13% (Santhirasegaram et al. 2013). The effect of different juice processing steps on vitamins are summarized in Table 7.5.

Table 7.5 Impact of juice processing on vitamins

Juice	Compounds	Treatment	Treatment conditions	Key outcomes	References
Cashew apple juice	Vitamin C	Indirect cold plasma	80 kHz, 30 kPa, 10, 30 and 50 mL/min	• While vitamin C content was increased after 5 and 10 min treatment (10.4% and 10.8% ↑, respectively), decrease was detected after 15 min treatment (4.5% ↓) (At 10 mL/min). • After 30 mL/min treatment, a slight decrease was observed, lower retention was reported after 50 mL/min treatment.	Rodríguez et al. (2017)
Clear apple	Ascorbic acid Total vitamin C (ascorbic plus dehydroascorbic Acid)	Pasteurization UHPH	90 °C for 4 min 100, 200 and 300 MPa	• UHPH and thermal treatment had no significant effect on ascorbic acid content. • While UHPH had no significant effect on the total vitamin C content of samples, pasteurization significantly reduced vitamin C (88% ↓).	Suárez-Jacobo et al. (2011)
Exotic fruit juice- stevia mixture	Ascorbic acid	HVED and PEF US	32 kJ/kg and 256 kJ/kg, 25 kV/cm 400 W and 24 kHz	• HVED, PEF and USN treatments significantly lowered the ascorbic acid content. • Retention of ascorbic acid was in the range of 17–91% when compared with untreated fresh fruit juice-stevia mixture	Buniowska et al. (2017)
Goat whey orange juice beverage	Ascorbic acid	Microfiltration Conventional heat treatment	0.005 m^2, 0.2 μm, 2 kgf/cm^2 (20 °C, 30 °C, 40 °C and 50 °C) 63 °C/ 30 min	• Microfiltered beverages (using up to 40 °C) had significantly higher ascorbic acid content than conventional pasteurization and microfiltration at 50 °C.	Vieira et al. (2020)
Melon	Vitamin C	Microfiltration Concentration	0.24 m^2, 0.2 μm,7 m/s 7.5 L, 0.20 m/s, 12 h	• Vitamin C losses were noted in permeate (7% ↓) and retentate (30% ↓) during microfiltration processing. • No significant vitamin C loss was observed between the initial clarified melon juice (P) and the concentrate (C).	Vaillant et al. (2005)

Orange	L-ascorbic acid Total vitamin C Vitamin A value (in terms of retinol activity equivalents)	HP PEF LPT HPT HPT + F F	400 MPa/40 °C/1 min 35 kV cm^{-1} /750 μs 70 °C/30 s 90 °C/1 min 90 °C/1 min and −38 °C/15 min −38 °C/15 min	• While HP, PEF, HPT, and HPT + F treatments significantly affected L-ascorbic acid content (7.79% ↓), LPT and F treatments did not exert any change. • PEF, HPT, HPT + F, and F caused a decrease (8.24% ↓) in vitamin C content, whereas HP and LPT did not exert any change. • HP treated orange juice showed the highest vitamin A value within both freshly squeezed and HP, PEF, LPT, HPT, HPT + F and F applied juices.	Sánchez-Moreno et al. (2005)
Orange	Vitamin C	HC Pasteurization	3–15 bar, 8–25 min 90 °C, 30 s	• Vitamin C retention was in the range of 79.66%, and 99.09% in HC treated orange juices. • Pasteurization significantly affected the vitamin C content. • Vitamin C retention of HC treated orange juices was 1.2 times higher when compared to heat-treated orange juice.	Katariya et al. (2020)
Orange	L-ascorbic acid Total vitamin C	LPT HP PEF	70 °C/30 s 400 MPa/40 °C/1 min 35 kV/cm, 750 μs	• Ascorbic acid content was significantly influenced by HP (4.74% ↓) and PEF (7.70% ↓). • While HP and LPT had no significant effect, PEF application significantly reduced the total vitamin C content (6.78% ↓). • At the end of refrigerated storage (40 days), vitamin C losses were observed in HP (14% ↓) and LPT (18% ↓) juices, whereas during storage HP juices maintained better both ascorbic acid and vitamin C content.	Plaza et al. (2006)

(continued)

Table 7.5 (continued)

Juice	Compounds	Treatment	Treatment conditions	Key outcomes	References
Orange	Vitamin A activity (in terms of retinol activity equivalents)	Pasteurization HPH	92 °C 30 s and 85 °C 15 s 150 MPa	• While there were no significant differences in RAE activity of pasteurized and fresh juice, homogenization caused a 1.35-fold significant reduction (26% ↓).	Stinco et al. (2020)
Orange (Navel and Cara cara)	Vitamin C (ascorbic acid + dehydroascorbic acid)	HPP	200 MPa/25 °C/1 min (HPP-200) 400 MPa/40 °C/1 min (HPP-400) 400 MPa/40 °C/1 min after 200 MPa/25 °C/1 min (HPP-200-400)	• Except for HPP-400 in Navel orange juice (30% ↓), HPP did not significantly affect vitamin C content. • Vitamin retention after HPP was about 90% except in HPP-400 N-juice.	De Ancos et al. (2020)
Ortanique	Vitamin A activity (in terms of retinol activity equivalents)	Pasteurization HPH	92 °C 30 s, 85 °C 15 s and 65 °C 15 s 150 MPa	• Thermal treatments at 92 °C and 85 °C caused a significant reduction of retinol activity equivalents. • The highest reduction in the retinol activity equivalents was observed in the HPH-treated sample.	Sentandreu et al. (2020)
Pineapple	Vitamin C	Microfiltration	0.011 m^2, 0.2 μm, 1.2 m/s	• Microfiltration was stated as an effective method for retaining vitamin C in pineapple juice. • In the first month of storage, vitamin C content was sharply decreased possibly due to the degradation of L-ascorbic acid.	Laorko et al. (2013)

Water-fruit juice beverage, milk-fruit juice beverage, soymilk-fruit juice beverage	Vitamin C	HIPEF HPP TT	5 kV/cm, 4-μs, 200 Hz 1800 μs 900 MPa, 100 °C (max.) 90 °C for 60 s	• Vitamin C content was significantly reduced by HIPEF (8–15 ↓). • By HP, vitamin C content was reduced only in soybean-fruit juice (10.5% ↓) • By TT, the highest vitamin C losses occurred (up to 31% ↓).	Rodríguez-Roque et al. (2015)

UHPH Ultra-high pressure homogenization, *HVED* High voltage electrical discharges, *PEF* Pulsed electric fields, *US* Ultrasound, *HP* High pressure, *LPT* Low pasteurization treatment, *HPT* High-pasteurization treatment, *HPT + F* High-pasteurization plus freezing treatment, *F* Freezing, *HC* Hydrodynamic cavitation, *HPH* High-pressure homogenization, *HPP* High pressure processing, *HIPEF* High-intensity pulsed electric fields, *TT* Thermal treatment

Not only thermal processes but also several other processing steps may affect the vitamin content. One of those processing steps is enzyme treatment. Vitamin C retention after different enzymatic treatment conditions of *Spondias tuberosa* (umbu) pulp was studied by Gouvêa et al. (2017). After processing for 2 h with enzymes, the vitamin C contents were reduced. Under optimum process conditions, vitamin C content was not significantly affected (Gouvêa et al. 2017). Pectinase treatment of litchi pulp decreased ascorbic acid content from 17.6 mg/100 g to 11.8 mg/100 g in litchi juice due to ascorbic acid oxidation during processing (Vijayanand et al. 2010). In a study conducted by Nur'Aliaa et al. (2011), red pitaya pulp was subjected to enzymatic treatment. Untreated pulp had higher vitamin C content compared to enzyme-treated sample.

Other than above mentioned processes, filtration and concentration processes may also affect the vitamin content. In this sense, melon juice was subjected to clarification by crossflow microfiltration and then concentration by osmotic evaporation. Although the concentration process did not affect the vitamin C content, the losses by clarification were correlated with the oxygen exposure. It was also stated that this situation could be drastically eliminated by degassing before microfiltration (Vaillant et al. 2005). In another study, the effect of clarification and the concentration for acerola juice were also studied by Matta et al. (2004). While concentration by reverse osmosis increased the vitamin C content by 4.2 times, vitamin C content was maintained after clarification by microfiltration (Matta et al. 2004). In tomato juice, osmotic distillation, membrane distillation, and their combination were investigated as an alternative to traditional thermal evaporation. While all of the concentration processes caused a total vitamin C decrease (46.3–61.2%), concentrated samples by membrane systems had higher vitamin C contents when compared with thermal evaporation (Bahçeci et al. 2015).

7.4.2 Bioaccessibility

In recent years, studies on bioactive compounds in foods are increasing based on the consumer's consciousness about these bioactive compounds' health advantages. However, these health effects may appear depending on their intake level and their bioaccessible and bioavailable contents (Santos et al. 2019). It is recommended to use innovative technologies in obtaining food products that contain bioactive components with improved bioaccessibility. The use of thermal technologies in fruit and vegetable products can destroy some food ingredients. It also has some implications for the accessibility of bioactive ingredients. In some cases, as thermal processes damage the cell wall, bioaccessibility may increase due to the breakdown of components (Barba et al. 2017).

In a study by Buniowska et al. (2017), the effect of nonthermal processing on bioactive compounds' bioaccessibility was examined. One of the bioactive components examined in this context was ascorbic acid. During gastrointestinal digestion, ascorbic acid content was decreased in fruit juice-stevia mixture treated with HVED,

PEF and US. Moreover, after the intestinal phase, no ascorbic acid was detected in both dialyzed and undialyzed fractions. It was reported that this may be due to the temperature lability of this vitamin during simulated gastrointestinal digestion (Buniowska et al. 2017). For α-tocopherol, it was reported that high-pressure processing provides higher α-tocopherol bioaccessibility compared to thermal-treated samples, and it may be related to modification of the location of tocopherols in the food, and thus they become more bioaccessible (Cilla et al. 2012).

Several factors may affect the bioaccessibility of bioactive compounds. In particular, ascorbic acid's bioaccessibility was found to be significantly affected by both the food matrix and the applied treatment (Cilla et al. 2012). From this respect, the enhanced bioaccessibility after processing was reported in many studies. Cilla et al. (2020) indicated that the bioaccessibility of vitamin C was 1.2 times higher in HPP-treated juice compared with untreated juice. Indeed, HPP treatment at 400 MPa was recommended for mandarin juices to increase bioactive compounds' bioaccessibility (Cilla et al. 2020).

In a study in which theoretical vitamin A activity was expressed in retinol activity equivalents (RAE), HPP application on orange juice led to increased bioaccessibility compared to fresh juices (Stinco et al. 2020). Similarly, in terms of RAE, while pasteurization did not significantly affect the bioaccessibility in mandarin juices, the RAE level was enhanced after in vitro digestion of high pressure homogenized juices in comparison with fresh juice (Sentandreu et al. 2020).

While much more studies are conducted with a variety of nutrients in foods, studies on vitamin bioaccessibility are limited (Uğur et al. 2020). Findings of studies investigating the effects of juice processes on bioaccessibility of vitamins are summarized in Table 7.6.

7.5 Others (Minerals, Fatty Acids, Amino Acids)

The production process of juices from several sources, including fruits and vegetables, also affects minerals, fatty acids and amino acids. As an alternative to conventional thermal treatments that may cause losses in juices' content and quality, nonthermal technologies have become popular. However, although nonthermal methods can eliminate the adverse effect of heat application, several parameters influence the juices' overall quality. In the literature, many techniques have been discussed up to date to understand their impacts on minerals, fatty acids and amino acids existing in juice composition.

Aadil et al. (2015) studied the effect of sonication on quality parameters of grapefruit juice, which are rich in several minerals, including magnesium (Mg), calcium (Ca), potassium (K), sodium (Na) and zinc (Zn). Their results showed the positive change of mineral element content in reference to control prepared by the same method of samples exposed to ultrasound. They also commented that the positive impact of sonication could arise from cell destruction, which results in releasing of minerals inside cells (Table 7.7). In another study, effect of sonication treatment

Table 7.6 Effect of juice processing on bioaccessibility of vitamins

Juice	Compounds	Treatment	Treatment conditions	Model	Key outcomes	References
Clemenules mandarin	Ascorbic acid Total vitamin C	HPP	400 MPa/40 °C/1 min	In vitro gastrointestinal digestion	• While bioaccessibility of ascorbic acid was increased from 64.35% to 81.58%, bioaccessibility of total vitamin C was increased from 76.09% to 92.07% untreated to HPP-mandarin juices.	Cilla et al. (2020)
Exotic fruit juice-stevia mixture	Ascorbic acid	HVED and PEF USN	32 kJ/kg and 256 kJ/kg, 25 kV/cm 400 W and 24 kHz	In vitro digestion model	• Ascorbic acid values were significantly affected by applied treatment, the energy input level and the digestion phase. • While a decrease in ascorbic acid content was observed throughout gastrointestinal digestion, ascorbic acid was not detected following intestinal digestion.	Buniowska et al. (2017)
Orange	Vitamin A activity (in terms of retinol activity equivalents)	Pasteurization HPH	92 °C/30 s and 85 °C/15 s 150 MPa	Simulated in vitro digestion	• Thermal treatments had no significant effect on the RAE, but HPP enhanced RAE's bioaccessibility (4.4-fold increase).	Stinco et al. (2020)
Ortanique	Vitamin A activity (in terms of retinol activity equivalents)	Pasteurization HPH	92 °C 30 s, 85 °C 15 s and 65 °C 15 s 150 MPa	Simulated static in vitro digestion	• While pasteurization had no significant effect on the bioavailability of RAE, homogenization enhanced the RAE in the micellar fraction (about 4 -fold).	Sentandreu et al. (2020)

Water-fruit juice beverage, Milk-fruit juice beverage, Soymilk-fruit juice beverage	Vitamin C	HIPEF HPP TT	5 kV/cm, 4-μs, 200 Hz 1800 μs 900 MPa, 100 °C (max.) 90 °C for 60 s	In vitro gastrointestinal digestion	• Except for HPP treated MB, HIPEF and HPP did not significantly affect the bioaccessibility of vitamin C. • Except for MB, TT had a significant effect of WB (16.5% ↓) and SB (11.6% ↓) on bioaccessibility of vitamin C. • Both the food matrix and the process affected the bioaccessibility of vitamin C.	Rodríguez-Roque et al. (2015)
Whole milk−fruit beverages, Skimmed milk−fruit beverages, Soy milk−fruit Beverages	Ascorbic acid	HPP TT	400 MPa/40 °C/5 min 90 °C for 30 s	In vitro simulated gastrointestinal digestion	• Except for JS, HPP caused a significant decrease in ascorbic acid content. • Thermal treatment caused a significant increase in the bioaccessibility of ascorbic acid.	Cilla et al. (2012)

PEF Pulsed electric fields, *HVED* High voltage electrical discharges, *USN* Ultrasound, *HPP* High-pressure processing, *TT* Thermal treatment, *HIPEF* High-intensity pulsed electric fields, *HPH* High-pressure homogenization

Table 7.7 Impact of juice processing on minerals, fatty acids and amino acids

Juice	Compounds	Treatment	Treatment conditions	Key outcomes	References
Apple	Na, K, Ca, P, Mg, Cu, Zn	US	30, 60 min 25 kHz, 20°	• Treatment caused a significant increase in Na, K and ca and a significant decrease in P, Mg and Cu content. • The change in their content was observed to increase with increasing treatment time. • The increase in Zn content from 1.3 mg/L to 1.35–1.4 mg/L was not evaluated to be significant.	Abid et al. (2014)
Broccoli	Fe, K, Mg, Ca, Cu, Mn, Na, P, S, Zn, Se	HIPEF Thermal	15–35 kV/cm, 100 kHz, 500–2000 µs 90 °C, 60 s	• Based on the treatment parameters (time, electric field strength, polarity), HIPEF showed the ability to increase significantly and preserve the mineral content. The highest increase was seen for Fe (214.2%), Mn (117.7%) and Zn (148.4%). • Except Mg (105.6%), Cu (123.7%), P (101.3%) and S (103.2%), other mineral contents either slightly or significantly decreased with thermal treatment.	Sánchez-Vega et al. (2020)
	Free amino acids			• The highest significant increase was observed for His (159.3%) and Lys (157.8%) based on changing treatment parameters among eighteen free amino acids. • All of the amino acid contents showed no change or significant decrease by thermal treatment.	
Fruit juice-soymilk beverage	Cu, Mn, Zn, Fe, Ca, Mg	HIPEF Thermal	35 kV/cm, 200 kHz, 800–1400 µs 90 °C, 60 s	• There was no major change on most of the investigated mineral contents based on untreated samples. • A significant increase in Fe and Zn contents was observed after longer HIPEF treatment.	Morales-De La Peña et al. (2011)
	Fatty acid profile			• Lower total fatty acid content than untreated but higher content than thermal-treated samples were determined. • There was a notable decline by increasing treatment time for some saturated and unsaturated fatty acids.	
Grapefruit	K, Na, Ca, Mg, Zn	US	30, 60, 90 min 28 kHz, 20 °C	• Treatment significantly increased mineral content except for Mg that decreased from 45.85 mg/L to 45.29–45.46 mg/L. • There was no change between 60 and 90 min treatments, but it was observed that increasing duration from 30 to 60 min caused the increase mostly.	Aadil et al. (2015)

Hami melon	Amino acids	HHP	400–500 MPa, 45 °C, 10 min	• Alongside the increasing content of amino acids present in untreated samples, absent amino acids in untreated sample composition, isoleucine and lysine were detected after 400 MPa treatment.	Pei et al. (2020)
	Palmitic acid, palmoleic acid, oleic acid, linoleic acid, α-linolenic acid			• A notable decline at 500 MPa was noted for the composition of all indicated fatty acids. • There was no significant difference in palmitoleic and oleic acids in samples subjected to 400 MPa and untreated.	
Orange	Total amino acid content	Fermentation Fermentation-pasteurization	Fermentation: 15 days Pasteurization: 85 °C, 30 s	• The highest and significant increase (from 8194 mg/L to 13,092 mg/L) was observed on the 9th day of fermentation. • Processes enhanced the nutritional value of the product in terms of amino acids, but pasteurization limited the increase ratio.	Cerrillo et al. (2015)
Spinach	7 non-essential amino acids 10 essential amino acids	Single and combination of US and PEF	US: 21 min, 40 kHz PEF: 9 kV/cm, 1 kHz 30 °C	• Except for glutamic acid, other non-essential amino acids' content significantly increased or remained unchanged after some treatments. • There was no definite result for essential amino acids. • In terms of total amino acids, each treatment resulted in a significant increase.	Manzoor et al. (2020)
	Zn, Fe, Mn, Ca, K			• The combined treatment positively and significantly impacted all examined minerals by a relative change of 6.30–63.82%. • The relative change by single treatment has no difference for only K, but others' content decreased or remained unchanged.	
Strawberry	Total amino acid	HIPEF	35 kV/cm, 100 kHz	• There was a significant decrease from 464.5 mg/L to 418.7 mg/L.	Odriozola-Serrano et al. (2013)
Tomato	Total amino acid	HIPEF Thermal	35 kV/cm, 100 kHz 90 °C, 60 s	• HIPEF resulted in the highest content than thermally treated samples.	Odriozola-Serrano et al. (2013)

HHP High Hydrostatic Pressure, *HIPEF* High-Intensity Pulsed Electric Field, *PEF* Pulsed Electric Field, *US* Ultrasound

on quality parameters and mineral content of apple juice was investigated. While a decrease was observed in the content of some minerals, some others were observed to increase (Abid et al. 2014). In accordance with the study of Aadil et al. (2015), they emphasized the potential of sonication to damage cell integrity as a reason for the increase. Besides, the adverse effect of longer exposure time on mineral contents was mentioned by these studies.

Alongside individual treatment, several methods may be conducted in combination. Manzoor et al. (2020) worked on the combined treatment of US and PEF as an alternative to pasteurization of spinach juice. In terms of the nutritional properties of the product, the content of free amino acids and minerals (Zn, Ca, K, iron (Fe) and manganese (Mn)) were determined. Although their contents decreased in the spinach juice treated with a single method, especially by PEF treatment, usage of the combination of US-PEF was suggested to increase the content of both free amino acids and minerals.

Amino acids are important not only for human nutrition but also for sensory characteristics, including taste, aroma and colour (Hounhouigan et al. 2014), although they exist in low amounts in juice composition (Buedo et al. 2000). In this sense, tomato juice, including a notable amount of free glutamic acid, was exposed to ultra-high hydrostatic pressure (UHP) to minimize thermal damage. Contrary to heat-treated samples, the amount of free glutamic acid in UHP-treated tomato juice was preserved mostly (Porretta et al. 1995). Similarly, Odriozola-Serrano et al. (2013) studied the free amino acid profile in tomato juice and strawberry juice after HIPEF application. As expected, processing impacted the quantity of amino acids, but a notable decline was observed in thermal-treated samples. While HIPEF slightly reduced the total amino acid in tomato juice, the same treatment resulted in a significant increase for strawberry juice. It has also been mentioned that contents should be controlled not only just after treatment but also during storage. Morales-De La Peña et al. (2011) focused on the fatty acid and mineral profiles of fruit juice-soy milk subjected to either HIPEF or thermal processes with the same point of view. During storage, non-thermal treated products exhibited higher fatty acid and stable mineral contents. The adverse effect of thermal treatment was indicated by this study. In another study, HIPEF was also compared with thermal treatment for broccoli juice in mineral and free amino acid concentration. Their contents were mostly influenced depending on the treatment conditions of HIPEF. On the other side, the contents remain at a remarkable level concerning samples exposed to heat (Sánchez-Vega et al. 2020).

Linhares et al. (2020) showed that while nonthermal treatments including the US, ultraviolet (UV)-pulsed light and low-pressure plasma increased the betaine, thermal treatments of high-temperature short time (90 °C, 6 s) and ultra-high temperature (138 °C, 6 s) increased fatty acids of açai juice. The other nonthermal treatment, high hydrostatic pressure, was evaluated for Hami melon juice by Pei et al. (2020). They found that the content of amino acids presents in Hami melon juice showed an upward trend, and adverse effects on fatty acids were observed after treatment.

Even though nonthermal processes have attracted attention for the preservation of juices, other production steps have a non-negligible impact on composition. Cerrillo et al. (2015) examined the effect of fermentation and pasteurization on orange juice amino acids. After fermentation, the amino acid profile showed no change, but the total amino acid content increased. On the other hand, while the amino acid profile was detected to be similar to with control, the concentration of 20 amino acids decreased. This result might be correlated with the degradation due to heat implications. Pineapple is a type of tropical fruit which is used to produce fruit juice. The reduction in mineral content of pineapple juice was shown by Akinyele et al. (1990) due to thermal treatment, and changes in the amino acid profile during production were mentioned by Hounhouigan et al. (2014).

The quality characteristic of juices is also critical for other products produced from juices such as wines. For this purpose, the fatty acid and amino acid composition of grape juice after either thermal or PEF treatment were evaluated, and no remarkable changes were observed (Garde-Cerdán et al. 2007).

The consumption and bioaccessibility of minerals, fatty acids and amino acids are notable concerns for consumers. Even though juices are not commonly considered as a major source of these bio-compounds, supplying a certain amount has importance for people. However, only a few studies are available about these compounds' bioavailability and bioaccessibility after juice processing. Da Silva Haas et al. (2019) showed that available minerals in grape juice sediments were less bioaccessible. It was also observed that the existence of bio-compounds was different in grape juices prepared from different varieties. Therefore, studies in this respect should be diversified and improved.

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Chapter 8
Influence of Extrusion on Food Bioactives

Sibel Yağci and Aylin Altan

8.1 Introduction

Extrusion technology plays an important role in the food industry as an efficient production process. In extrusion, ingredients go through cylinder-formed barrel with the help of one or two rotating screws, and then forced through the orifice at which the material expanded through. Extrusion technology has been developing rapidly; it has been grown from simple handling devices into more complex units in the last years. An important advantage of extrusion process is the possibility to produce different products from several food groups by changing various ingredients and processing conditions. Different types of unit operations such as mixing, melting, heating/cooling, shearing, cooking, puffing, final shaping and drying can be combined in a single energy efficient and rapid continuous process. Extrusion cooking process is frequently used for cereal-based product processing. Nowadays, extrusion has been gaining more and more interest in the food industry for its potential to be used as a reactor for flavor generation, formation of functional bioactives and encapsulation. Therefore, this emerging technology can produce various types of products with different shape, texture and taste.

In recent years, manufacture of health-giving extruded foods is rising, as consumers are ever focusing on the consumption of nourishing foods enriched with functional ingredients such as dietary fiber and phenolic compounds, possessing bioactivities. However, use of specifically bioactive compounds in the extruded formulations brings up some challenges due to the extreme conditions of both

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temperature and shear employed during this process. Many researchers have studied the effects on bioactive compounds including phenolics, anthocyanins, flavonoids, and lycopene of various ingredients used for extrusion technology. Bioavailability of bioactive compounds is improved by extrusion process by destruction of protein bonded complexes. Understanding the influence of extrusion operation conditions on bioactive compounds is substantial in keeping the health beneficial properties in processed food products. Therefore, the results of many studies suggest that the optimization of the operation conditions is mandatory. Extruder is a reactor of high temperature short time (HTST) process. This technology is also used for microencapsulation of sensitive bioactive components within a stable polymer. It is easy to scale up of extrusion-based microencapsulation process with avoiding the usage of organic solvents. Development of new chemicals and methods, which are applicable to microencapsulation by extrusion process, will surely lead to novel processes/products.

This chapter would be of importance to processors and researchers as they wonder the role of extrusion technology for production of nutritious food products through its effect on bioactive compounds of raw materials and ingredients. The aim of this chapter is also to provide a detailed outline about the potential use of extrusion process in microencapsulation technology.

8.2 Food Extrusion Process

Extrusion processes raw ingredients by pushing it through high temperature and/or pressure region and then through a restricted opening to give a final shape. The extrusion process is made continuous by substituting the piston in the cylinder with a helical screw in which, material is transported forward by the rotating screw. As the material approaches the die, there is usually increase in temperature and pressure, which is sufficient to force extrudate through the die (Rokey 2000). Food extrusion has been known since the late eighteenth century. Single screw type extruders used for chopping or mincing soft food by forcing them thorough die plates have been the first screw extruders used in the food industry. In the 1930's, a single screw extruder was firstly used for production of pasta, for mixing and shaping the resulting dough into macaroni in one continues operation. The principle of these extruders remains the same with recent developments focusing on increased capacity and improved control. During the late 1940's, directly expanded corn curls were produced using high shear extruders. Further refinement of the food extrusion process and extension of its applications occurred during the 1970's. Examples include use of extruder as a mixer and pasteurizer in the processing of soft-moist pet foods and co-extruded products containing more than one component (Harper 1989). The use of twin-screw extruders for food processing started in the 1970's. These machines have improved conveying and mixing capabilities and interchangeable screw profiles (Rokey 2000).

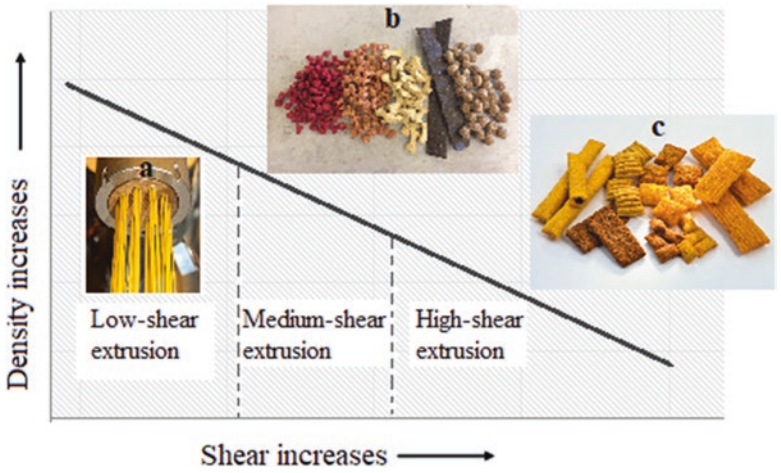

Fig. 8.1 Classification of extruded products according to shear forces applied (**a**) pasta, (**b**) pet food, (**c**) co-extruded snacks (adapted from Rokey 2000)

A food extruder can be considered as a continues chemical reactor, that processes food mixtures at high temperatures, high pressures under shear forces and, in most cases, at relatively low water contents and residence times (Cheftel 1986). This process is controlled by many operational variables such moisture content during operation, screw speed, screw profile, temperature of barrel, feed rate and die configuration (Thymi et al. 2005). Shear forces applied during extrusion mostly depend on the screw geometry and screw speed. There are different types of screw elements (eg. mixing blades, reverse screws) that increase the shear force during extrusion process. As the screw moves through the die exit, the ingredients warmed up and change their physical and chemical properties (Yağcı et al. 2014). According to shear force applied during extrusion process, there are three types, at which different products are obtained, as shown in Fig. 8.1 (Rokey 2000):

- Low-shear extrusion: This process is widely used to produce pasta type products with higher density. Both of extrusion temperature and screw speed are lower in low-shear extrusion. This process is known as cold forming extrusion.
- Medium-shear extrusion: Moderately high shear forces is applied during processing of ingredients with lower moisture content (e.g. pet foods and texturized vegetable protein).
- High-shear extrusion: The ingredients are extruded at temperatures over than 100 °C and under very high-pressure extrusion conditions. The products (e.g. snacks, breakfast cereals) have high expansion ratios in this case.

Extrusion cooking technology has many advantages over the classical food processing methods. Some of the advantages and limitations are summarized in Table 8.1 (adapted from the studies of Riaz 2000; Guy 2001; Yağcı et al. 2014).

Table 8.1 Advantages and limitations of extrusion process

Functions	Advantages	Limitations
Versatility	Wide range of food products with various shapes, textures, colors and appearances, can be manufactured by changing the operational settings of extruder and/or the ingredients. Low-cost raw materials can be handled easily.	Usually cereal-based ingredients with powder form can be processed. Versatility depends on the type of the extruder; twin screw extruders are more versatile.
Productivity	Extruder has high productivity, easy to scale up to high capacity and is automated. The product quality is maximum due to perceiving of inappropriate quality product immediately.	High initial investment costs and automated machines need preventative maintenance costs, and the cost of training employees.
Product Characteristics	It is readily feasible to produce appealing products in terms of visual characteristics with respect to other production methods. The process take place at a very short time, so products relatively higher nutritional quality over the traditional process because of less destruction of heat sensitive ingredients. Extrusion also provides inactivation of microbes, deterioration enzymes and several anti-nutritional factors.	In high-shear extrusion, nutritional loss may occur due non-enzymatic browning and caramelization reactions. Color loss may take place especially during extrusion of heat sensitive ingredients due to expansion on excessive heat.
New Foods	An important advantage of extrusion process is the possibility to produce products with puffed, crispy structure from the raw ingredients, which cannot be puffed by another manufacturing process. Different raw ingredients like animal and vegetable proteins, various starch sources, and fatty seeds can be formulated to produce a wide variety of new and unique snack food products. Extrusion process can be used as a chemical reactor for production of food additives, flavors and for encapsulation. Nowadays, the extruded flours can be used as novel natural ingredients such as hydrocolloids, fat replacers and for developing gluten-free, phenolic-rich, low-glycemic and functional foods.	Studies assessing extruders as a reactor for new chemistries and new material formulation methods are still scarce.
Environmentally Friendly	Extrusion produces little or no waste streams. This is important because new environmental regulations are stringent and costly.	
Energy Efficiency	Extrusion combined different types of unit operations and provides savings in labor cost, floor space cost and energy cost whilst increasing productivity. Operation under relatively low moisture contents reduces the quantity of heat required for cooking and re-drying the product after cooking, so reduces operational energy costs.	

8.2.1 *Types of Food Extruder*

Any food extrusion system is composed of series of different parts like feeder, screw, barrel, die and cutting blade connected to each other in a one-unit operation. Feeder is used for feeding of raw materials to the extruder barrel at a controlled feed rate (Yağcı et al. 2014). Liquid ingredients are usually fed by peristaltic pumps. Solids are fed through the screw feeders, particularly in powder form (Yacu 2012). There are two types of feeders used: volumetric and gravimetric feeders, the latter is well-controlled and preferred to avoid clogging through the extruder barrel. Extruder barrel can be cooled/or heated by circulating of water, steam and air through the jacketed barrel segments (Choton et al. 2020). Screw can be single or twin, at which different screw components can be assembled on. The function of screw components changes; for example, conveying screws are used for transporting feed material into the barrel and kneading screws are used to compress the melt (Fig. 8.2) (Burtea, 2001). The extruder die presents two main functions: giving shape to the final product and promoting resistance to material flow within the extruder, resulting in an increase in internal pressure (Choton et al. 2020). Rapidly moving knife placed at the exit of the die and is used to cut the certain pieces of

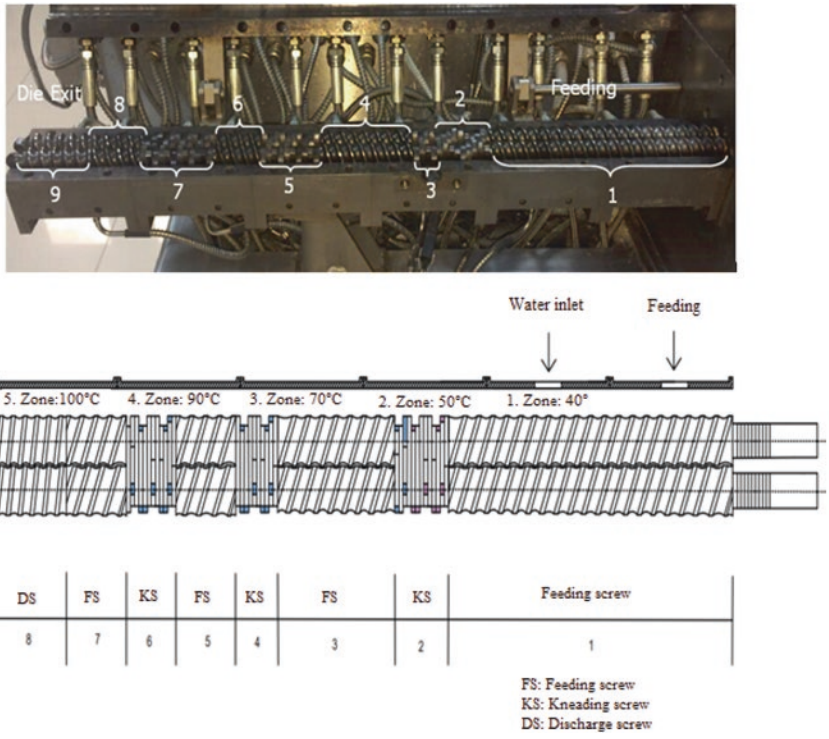

Fig. 8.2 Typical screw configuration for the expanded snack

Table 8.2 Comparison of single and twin-screw food extruders

	Extruder Type	
Features	Single screw	Twin-screw
Equipment	• Single screw: poor mixing ability, low degree of heat transfer. • Transport in the barrel: frictional drag flow in the solid conveying zone and viscous drag flow in the melt-conveying zone (Yacu 2012).	• Co- or counter- rotating two screws: well mixing ability, high degree of heat transfer. Intermeshing (engagement of two screws) and non-intermeshing screws • The transport in the barrel: sweeping created by rotation of two screws (Riaz 2001). • Various alternatives for screw profile and geometry
Process control	• Difficult, single purpose, easy cleaning.	• Better process control, versatile, easy cleaning, and rapid product change (Rokey 2000; Riaz 2001).
Application	• Limited number of raw materials can be processed. • Pasta, confectionery, precooked flours, encapsulation of bioactive compounds and flavors	• Wide variety of formulations can be processed. • Counter-rotating extruders: used for relatively non-viscous materials (gum and jelly) (Riaz 2001). • Intermeshing co-rotating extruders: high degree of heat transfer, used for expanded snack products (Ainsworth and Ibanoglu 2006). • Ready to eat breakfast cereals, co-extruded products, texturized vegetable protein, pet food, encapsulation of bioactive compounds.

extrudate. There are two different types of extruders in commercial food production: single-screw and twin-screw extruders. Table 8.2 summarized the well-known features of single and twin-screw extruders comparatively.

8.2.2 Extrusion Process Parameters

Process control in the extrusion process must be carried out carefully to control the final product quality. The extruded product properties mainly depend on the several parameters such as ingredients in the formulation, moisture content of the feed, feed rate, length to diameter ratio, screw geometry and speed, die design, temperature of the barrel zones and die temperature. These variables are independent process variables which can be controlled freely during extrusion process. As a result of the extrusion, dependent variables like die temperature & pressure, viscosity and residence time vary. Some of the important process variables are explained as below:

Raw material characteristics: There are two important factors affecting the final product quality through extrusion process: composition of the feed and particle size.

Particle size is important from the point of starch modification through the extruder barrel. Finer particles give products with softer textures and smaller cell structures, while coarse particles let extrudate become crunchier in texture and expanded structure (Harper 1989).

Extruded foods are produced using a wide and diverse range of raw materials. Combinations of different ingredients like cereals, grains and starches, tubers, oil seeds, legumes and meat and protein sources, are possible in extrusion process (Choton et al. 2020). Wheat and maize flours are the most used raw materials, followed by other ingredients are rice flour, potato, rye, barley, oats, sorghum, cassava, tapioca, buckwheat, pea flour and other related materials (Guy 2001). Nowadays fruits and vegetable-based formulations become popular due to their bioactive properties. Starch is the major carbohydrate source in extruded feed give expanded structure to the extrudate through the modification (Bahattacharya and Hanna 1987). During the extrusion process, starch is partially hydrated and subjected to increasing shear, while it is mechanically conveyed and heated; and finally gelatinized and/dextrinized through extrusion process. The protein content may be derived from cereals, legumes, or animal proteins. Starch composes continuous phase, while protein forms dispersed phase in the extruder melt. Protein acts as a 'filler' in cereal extrudates, increases sites for cross-linking and changes the flow behavior of melt and textural characteristics (Ainsworth and Ibanoglu 2006). Lipids are generally used to improve eating quality of extruded products. Lipids act as lubricants between the particulate matter and the screws of the extruder. They are mixed with other materials and are rapidly dispersed into small droplets and are trapped in the continuous phase (Guy 2001). Lipid content over 5–6% impair extruder performance. Fiber is used as a bulking agent and can increase the nutritional quality of the product. Fiber usage is often limited by its effect on product texture by reducing expansion. Beet pulp, fruit, pea, and soy fibers may be added as fiber source to starch-based formulations at levels ranging from 5 to 10% (Huber 2001).

Moisture in the feed function as a plasticizer in the extrusion process, is usually adjusted to the range of 10–40%. Above 10% moisture level, polymers begin to move and slide across each other, so the solid feed changes its structure from glassy state to viscous melty state (Guy 2001). This is of particular interest for many roles of water plays: as a diluent or lubricant reducing viscosity; as a heat sink restricting temperature variations; as a reactant, particularly in gelatinization; and as a direct influence on puffing, product moisture, and structure through its effects on elasticity and plasticity (Miller 1990).

Operational parameters: The most important operational parameter is the temperature, which affects rheological properties of the extruded melts (Moraru and Kokini 2003). The control of temperature has a profound effect on the condition of the dough just behind the die and on the final expansion of the product. Vapor pressure of the moisture at the die depends on the temperature; the higher the vapor pressure, the greater the extrudate expansion. (Harper 1986). Increasing screw speed led to increase of shear in extruder barrel and decrease of melt viscosity, so it eventually causes the product to be expanded (Kokini et al. 1992). Feed rates are

normally kept low enough that the extruder operates under starve-fed (partially filled screw) conditions. Controlling feed rate in a starve-fed extruder will also affect product texture. Lowering of feed rate will reduce the fill of extruder. With only a partially filled screw, pressure flow (caused by a pressure difference across the open channel) can be proportionally more significant, causing greater circulation in the channel and potentially more mechanical damage to food molecules (Harper 1986). Die pressure, measured at die exit, has been commonly used in determining performance of an extruder.

8.2.3 Advantages of Extrusion Technology from the Point of Nutritional Concern

Todays, consumers demanding more functional and healthier foods, this is also valid for extruded food products. So, increasing and maintaining the nutritional quality of extruded foods has a vital importance. Nutritional relevance is particularly important when extrusion used particularly to develop nutritionally balanced or enriched foods. The major changes take place during extrusion cooking are binding, cleavage, loss of native conformation, recombination of fragments, and thermal degradation (Camire 2000). Among the macromolecules, proteins are the most influenced component. The extrusion process affects proteins particularly by denaturation. Extrusion decreases protein solubility, reduces availability of amino acids through Maillard reactions (Moscicki et al. 2013) and improves protein digestibility by destruction of trypsin inhibitors, haemagglutinins, tannins and phytates (Singh et al. 2007; Steel et al. 2012; Camire 2000; Rathod and Annapure 2017). Extrusion depolymerizes starch, and increases its digestibility by way of gelatinization, inactivation of endogenous α-amylase inhibitor, disruption of cellular structure (Cheftel 1986). Extrusion also produces resistant starch like amylose-lipid complex, acidic environment also favors resistant starch formation during extrusion (Camire 2012). Oxidation of starch-lipid complexes and lipid components like DHA and EPA fatty acids are fairly limited during extrusion (Camire 2001; Camire 2012). Extrusion solubilizes fibers by cleavage of non-covalent and covalent bonds between fibers and other molecules (Steel et al. 2012). The effects of extrusion on the minor components are more severe. Water soluble vitamins such as vitamin C (Moscicki et al. 2013) and thiamine; fat soluble vitamins such as vitamin A, E and their related compounds (carotenoids, and tocopherols) are destructed during thermal treatments at 100 °C and above (Camire 2000). Riboflavin, niacin, pyridoxine, folic acid, vitamin D and vitamin K appear to be comparatively stable during extrusion (Asp and Bjorck 1989, Camire 2000). Extrusion destructs inhibitory factors such as condensed tannins and phytates, so improves mineral absorption (Steel et al. 2012). The effects of extrusion on bioactive components of food ingredients will be explained in detail in Sect. 8.4.

8.3 Bioactives Found in Common Ingredients of Extrusion

Extrusion processing is useful tool for handling of unconventional ingredients and food processing wastes, they can be supplemented into food formulations effectively (Choton et al. 2020). Nowadays, scientific and industrial studies are mostly focusing on fortification of extruded products with health-beneficial compounds, by incorporation of the under-utilized nutrient-dense ingredient such as scarce cereals, pseudo cereals, legumes, plant materials and several fruit and vegetable by-products. Specifically, enrichment is usually made to increase the contents of bioactives in extrudates. One of the main reasons for the seeking the novel ingredients for extruded type foods is the reduction of crop production, increasing world population and hence, the rise of the crop prices all over the world. Hence, today's scientists and governments show much more interest into the use of cereal-composite flours and whole grain flours to produce novel type of cereal based extruded products (Saleh et al. 2019).

Cereal flours are among the most important ingredients of extrusion process. Although refined flours were preferred at first, recently whole grain flours are commonly used because of their health-beneficial compositions. Whole grains are composed of starchy endosperm, germ, and bran parts. Their health benefits come from synergistic effects of micronutrients and phytochemicals presents in bran and germ; for example, phenolic acids, sterols, tocols, tannins, anthocyanins, dietary fiber (Saleh et al. 2019). Table 8.3 summarizes some important bioactives present in common cereal grains used in extrusion process. Phenolics, carotenoids, vitamin E compounds, lignans, β-glucan, and inulin are the major phytochemicals found in whole grains. Among the phytochemicals, ferulic acid and diferulates are more specific to grains, they are limited in fruits and vegetables (Liu 2007). Compounds, that have one or more aromatic rings attached to hydroxyl group, are defined as phenolics. Phenolics are generally categorized as phenolic acids, flavonoids, stilbenes, coumarins, and tannins (Liu 2004). The concentrations of phenolic compounds in whole grains are influenced by grain types, varieties, and the part of the grain sampled. Cereal phenolics are primarily located in the grain outer layers (Beta 2003; Dykes and Rooney 2007). Phenolic acids, most common components of phenolic compounds, have hydroxybenzoic acid and hydroxycinnamic acid derivatives (Liu 2007). Hydroxybenzoates are commonly represented by gallic, p-hydroxybenzoic, protocatechuic, vanillic, and syringic acids. The most common hydroxycinnamates are p-coumaric, caffeic, and ferulic acids (Jaganath and Crozier 2010). Phenolic acids reported in cereals occur in both free phenolics (mainly located in the pericarp) and bound phenolics (esterified to cell wall). Bound phenolics are bound to sugars, organic acids, and lipids through ester links (Calinoiu and Vodnar 2018). The major phenolic acids are ferulic and p-coumaric acids, requiring acid or base hydrolysis for extraction (Dykes and Rooney 2007). Particularly the bioavailability of the ferulic acid is low in cereals, because of strong binding to indigestible cell wall. Flavonoids are a class of compounds consisting of two aromatic rings and one heterocyclic C3 structure (C6–C3–C6). They can be composed of flavonols,

Table 8.3 Classification of dietary bioactive compounds present in frequently used grain flours (re-constructed from the studies of Beta 2003; Hemery et al. 2007; Liu 2007; Dykes and Rooney, 2007; Calinoiu and Vodnar 2018; Zhu 2019; Xiong et al. 2019)

Grain type	Class	Compound(s)
Wheat	Phenolic acids	p-hydroxybenzoic, protocatechuic, salicyclic, vanillic, syringic, ferulic, caffeic, cinnamic
	Flavanoids	Anthocyanins: cyanidin 3-galactoside, cyanidin 3-glucoside, cyanidin 3-rutinoside, delphinidin-3-glucoside, delphinidin-3-rutinoside, peonidin 3-glucoside, petunidin 3-glucoside, petunidin 3-rutinoside Flavones: apigenin glcyosides, tricin
	Tocopherols	β-Tocotrienol
	Condensed tannins	Prodelphinidin and some procyanidin units, propelargonidin units, procyanidin B-3, prodelphinidin B-3
	Nonstarch polysaccharides	Arabinoxylans in bran, lignans, β-glucan
Rice	Phenolic acids	Protocatechuic, p-hydroxybenzoic, vanillic, syringic, ferulic, caffeic, p-coumaric, sinapic
	Flavanoids	Anthocyanins: cyanidin 3-rutinoside, peonidin 3-glucoside
	Tocopherols	α-Tocopherol, α-Tocotrienol
	Condensed tannins	B-type proanthocyanins, catechin (most abundant), dimer, trimer, tetramer, and pentamer
Maize	Phenolic acids	Protocatechuic, p-hydroxybenzoic, vanillic, syringic, ferulic, caffeic, p-coumaric
	Flavanoids	Anthocyanins: cyanidin 3-glucoside, pelargonidin 3-glucoside, peonidin 3-glucoside Flavanones: kaempferol, quercetin Flavanols: leucocyanidin
	Tocopherols	ɣ-tocopherol
	Nonstarch polysaccharides	Arabinoxylans in bran
Barley	Phenolic acids	p-hydroxybenzoic, vanillic, protocatechuic, caffeic, o-coumaric, p-coumaric, m-coumaric ferulic, chlorogenic, salicylic, syringic, sinapic
	Flavanoids	Anthocyanins: cyanidin, cyanidin 3-glucoside, delphinidin, pelargonidin, pelargonidin glycosides, petunidin 3-glucoside, petunidin 3-rutinoside, prodelphinidin B-3, procyanidin Flavanones: chyrsoeriol Flavanols: catechin, leucocyanidin, leucodelphinidin, procyanidin B-3, prodelphinidin B-3, Catechin
	Tocopherols	α-Tocotrienol
	Condensed tannins	Catechin, gallocatechin, prodelphinidin and procyanidin units, such as procyanidin C-2, prodelphinidin B-3, procyanidin B-3, prodelphinidin C-2, prodelphinidin dimer I, prodelphinidindimer II
	Nonstarch polysaccharides	Arabinoxylans, lignans, β-glucan

(continued)

Table 8.3 (continued)

Grain type	Class	Compound(s)
Sorghum	Phenolic acids	Gallic, p-hydroxybenzoic, vanillic, protocatechuic, caffeic, p-coumaric, ferulic, chlorogenic, gallic, gentisic, syringic, cinnamic, sinapic
	Flavanoids	Anthocyanins: apigenidin, apigenidin 5-glucoside, luteolinidin, luteolinidin 5-glucoside, 5-methoxyapigenidin, 7-methoxyapigenidin, 7-methoxyapigenidin 5-glucoside, 5-methoxyluteolinidin, 5-methoxyluteolinidin 7-glucoside, 7-methoxyluteolinidin Flavones: apigenin, luteolin Flavanones: eriodictyol, eriodictyol 5-glucoside, naringenin, kaempferol 3-rutinoside Flavanols: taxifolin, taxifoln 7-glucoside, apiforol, luteoforol, catechin, procyanidin B-1
	Condensed tannins	Heteropolyflavan-3-ols (tetramers, pentamers, hexamers, heptamers, octamers, nonamers), glucosylated heteropolyflavans
Millet	Phenolic acids	Gallic, p-hydroxybenzoic, gentisic, vanillic, syringic, ferulic, caffeic, p-coumaric, cinnamic, sinapic
	Flavanoids	Flavones: apigenin, apigenin glycosides, glucosylorientin, glucosylvitexin, luteolin, tricin, vitexin
Oat	Phenolic acids	Protocatechuic, p-hydroxybenzoic, vanillic, syringic, ferulic, caffeic, p-coumaric, sinapic
	Flavanoids	Flavones: isovitexin, apigenin, luteolin, tricin, vitexin
	Tocopherols	α-Tocotrienol
	Hemicellulose	Lignans, β-glucan
Rye	Phenolic acids	Protocatechuic, p-hydroxybenzoic, vanillic, syringic, ferulic, caffeic, p-coumaric, sinapic
	Flavanoids	Anthocyanins: cyanidin 3-glucoside, peonidin 3-glucoside
		Lignans

anthocyanins, isoflavones, flavanones, and flavones according to heterocyclic structures (Dykes and Rooney 2007). Colored cereals contain more phenolic compounds, which is mostly flavonoid content (X. Yu et al. 2020). Pericarp of pigmented varieties of cereals such as barley, maize, wheat, rice, and rye contain most of the flavonoids (Nayak et al. 2015). Sorghum varieties have been reported to contain the highest levels of flavanones among food plants (Xiong et al. 2019) as seen in Table 8.3. The anthocyanins are another major flavanoids present in colored cereals, such as colored barley, maize, rice, and wheat. Proanthocyanidins are commonly known as condensed tannins, which have free radical scavenging activity, anticarcinogen andcardiopreventive effects, antimicrobial and antiviral activities (Zhu 2019). Antioxidant activity of these compounds are more than the monomeric phenolic compounds. Polymerization of flavan-3-ols (catechin, epicatechin, epigallocatechin) produce chemical structure of proanthocyanidins. The condensed tannins, which contribute to the astringency in food, are usually found in pigmented sorghum, finger millet and barley (Dykes and Rooney 2007).

Carotenoids provide pigmentation essential for photosynthesis, reproduction, and protection in plants. They act as antioxidants in lipid environments of many biological systems through their ability to react with free radicals. Carotenoids commonly found in whole grains are lutein, zeaxanthin, β-cryptoxanthin, β-carotene, and α-carotene (Liu 2007). Grains also contain tocotrienols, tocopherols, and oryzanols. Vitamin E compounds are present in the germ fraction of grains (Liu 2007). Panfili et al. (2003) reported 75 mg/kg of tocopherol content in wheat and barley, 33–43 mg/kg of b-tocotrienol content in wheat, 45 mg/kg of d-tocopherol in corn and 56–40 mg/kg of a-tocotrienol in oat and barley, respectively. Major components of cereal cell walls are arabinoxylans and β-glucans; otherwise, the minor components are cellulose and lignin. Arabinoxylans are important constituents of hemicelluloses in the endosperm and outer layers of cereal grains, including corn, wheat, rye, barley, oat and rice (Wang et al. 2020). Most hydroxycinnamic acids, such as caffeic, p-coumaric and ferulic, exist in a covalently bound manner and are esterified to arabinose residues in the arabinoxylans; this contributes to polymer cross-linking (Hemery et al. 2007). Hydrolysis of arabinoxylans by xylanase to produce feruloylated oligosaccharides; and then hydrolysis by ferulic acid esterase to produce free ferulic acid are the main biochemical reactions. Feruloylated oligosaccharides and free ferulic acid have higher antioxidant activity compared to feruloylated polysaccharides (Baublis et al. 2000). The digestive processes of lignans present in grains could be effective in modifying whole grain phenolics to increase their bioavailability and antioxidant activity. Lignans are a class of dietary phytoestrogen compounds that are predominant in flaxseed, however, they are also found in barley, oat, rye, triticale, and wheat (Dykes and Rooney 2007). The common plant lignans are secoisolariciresinol, matairesinol, lariciresinol, pinoresinol, and syringaresinol. When consumed, plant lignans such as secoisolariciresinol and matairesinol are converted to the mammalian lignans, enterodiol and enterolactone, by intestinal microflora in humans (Liu 2007). These compounds are reported to reduce the risk of hormone dependent cancers, colon cancer, and cardiovascular diseases, and show strong antioxidant activities (Dykes and Rooney 2007). β-glucan, is a type of soluble fiber, is found in mostly in barley and oats. β-glucan has proven to be effective in lowering plasma cholesterol, improving lipid metabolism, and reducing glycemic index. β-glucan consumption of about 3 g/day lowers blood cholesterol levels according to U.S. Food and Drug Administration. It has also been allowed for whole grain barley/oats and barley−/oat-containing products to carry a claim that they reduce the risk of coronary heart disease (Hu et al. 2015). Phytic acid (myoinositol 1,2,3,4,5,6-hexakis dihydrogen phosphate) is found in most cereals and legumes at approximately concentrations of 1–3%. This compound is generally considered as an antinutrient, as it can bind minerals and decrease their bioavailability (Hemery et al. 2007). On the converse, it has ability to inactivate oxidative reactions by binding to transition metals, so prevent cell damage. However, the efficiency of inhibition is limited, because it unable to chelate additional prooxidative metals in the phosphatidylcholine liposome model or *in vivo* (Baublis et al. 2000).

In more recent years, enrichment of extrudates has been made by utilization of novel protein sources such as legumes and oilseeds. Among the legumes, chickpea, lentil, dry beans, mung beans, soybeans, and peas are important sources of protein. Blending of whole grain flours with Moreno legumes and seeds brings balanced composition of the essential amino acids (Shah et al. 2019) and higher amounts of bioactive compounds and dietary fiber content with respect refined cereal flours (Saleh et al. 2019). Legumes are an excellent source of protein, peptides and phytochemicals. Legumes contain several non-nutritive and bioactive compounds such as enzyme inhibitors, lectins, phytosterols, phenolic compounds, and saponins (Moreno-Valdespino et al. 2020). Various legume types including mung bean, adzuki bean, kidney beans, lupin bean, soybeans, chickpea, red and green lentils, cowpea, pea and their germinated forms have been reported to be rich in phytochemicals such as phenolics, saponins, proanthocyanidins, flavonoids. Legumes are also a good source of carotenoids and tocopherols (Moreno-Valdespino et al. 2020). Many of these compounds have been reported to be able to reduce the growth of different types of cancer cells and to lower cholesterol levels and also the risk of developing obesity and type-2 diabetes. Among phenolic compounds, flavonoids have been extensively studied in legumes; particularly isoflavones stand out for lowering of cardiovascular disease risk. Both highly polymerized polyphenols, which are tannins, and low molecular weight phenolics (phenolic acids, flavonoids) have been found to be present (0.01–4.0 g/100 g of dry weight) in legumes (Carbonaro 2007). Legume seed proteins have been demonstrated to exert cholesterol-reducing properties, thus representing powerful bioactive components. Many studies showed that legumes contain bioactive peptides, which have antioxidant, antihypertensive and hypocholesterolemic activities (Moreno-Valdespino et al. 2020). Bioactive peptides can be defined as specific protein fragments that have a positive impact on body functions; and may ultimately influence health namely, the cardiovascular, digestive, immune and nervous systems (Korhonen and Pihlanto 2006). Legume storage proteins consist of two major subgroups: vicilins and legumins. They are an excellent source of bioactive peptides, which can be produced by enzymatic hydrolysis reactions to yield a protein hydrolysate with various biological potential (Moreno-Valdespino et al. 2020).

Fruits, vegetables, and their by-products are known to be rich in bioactive compounds such as phenolic acids, flavonoids, anthocyanins, carotenoids, and vitamins (Saleh et al. 2019). Nayak et al. (2015) reviewed previous studies in literature on the major phytochemicals and antioxidant compounds in fruits and vegetables and their changes with respect to different processing operations. They noted that concentration of total phenolic compounds in different fruits and vegetables changed from 15 to 500 mg gallic acid eq/100 g for fresh fruits and from 10 to 140 mg mg gallic acid eq/100 g for fresh vegetables, respectively. Anthocyanin content of the colored fruits and vegetables is notably higher than the others, making them suitable for use as natural food colorants. Fruits contain high moisture and sugar; this limits their usage in extrusion. But powders and concentrated forms of fruits can be supplemented into product formulations such as breakfast cereals (Camire et al. 2007). Moreover, fruits and vegetables pomaces, source of dietary fiber and other

functional compounds including flavonoids, anthocyanins, and carotenoids (Altan and Maskan 2011), could be utilized. Thermal processes damage phenolic substances and reduce antioxidant activity. It was stated that the process of baking and cooking reduced the total phenolics in fruit and vegetable-based formulations, but just the opposite effect could be obtained for the antioxidant activity. The main reason for these types of unpredictable changes is formation of new compounds through thermal treatment by specified chemical reactions. At high temperatures of 90–120 °C, chemical oxidation of phenolic antioxidants to quinones and their polymers occurs in addition to caramelization, strecker degradation and hydrolysis of esters and glucosides of antioxidants. The increase or retention of antioxidant activities in processed foods are related with production of new compounds with potential antioxidant capacity. Adversely, high shear forces inside the extruder barrel affect the functionality of phytochemicals in the extruded products. Most of the researchers observed that reduction in the antioxidant activity and anthocyanin contents because of reduction in the natural phenolic antioxidants (Nayak et al. 2015).

8.4 Effect of Extrusion Cooking on Bioactive Compounds in Different Food Products

Consumers increasingly demand nutritious and healthy foods that have additional health promoting functions. This demand drives the food industry to develop processing technologies to improve the nutritional properties of foods with additional health benefits. The processing of whole grains, legumes or fruits and vegetables with different technologies has gained importance due to bioactive compounds of these foods which provide health benefits. Extrusion cooking is one of the versatile technologies that allows to process any formulations based on these raw materials into a different variety of products at high productivity. The growing demand has also triggered the research efforts for the development of new products using different raw materials containing bioactive compounds by extrusion technology to ensure maximal nutritional and functional properties as well as improving the overall quality of a product. Processing of foods has an impact on not only physical and sensory properties of final product but also on nutritional properties negatively or positively. Recent studies have focused on the effect of extrusion processing on the content of bioactive compounds, their functional properties and potential health benefits. Table 8.4 shows a summary of recent studies investigating the effect of extrusion processing conditions on the bioactive compounds in extruded products.

Hirth et al. (2014) investigated the effect of extrusion processing parameters on the retention of anthocyanins in extruded foods produced from native maize starch and bilberry extract. They reported that total anthocyanin loss increased from 18% to 36% when the barrel temperature was increased from 100 °C to 160 °C. On the other hand, high moisture content resulted in a higher retention of total anthocyanin content of extruded foods compared to the low moisture content. At low moisture

Table 8.4 A summary of recent studies investigating the effect of extrusion processing on bioactive compounds

Raw materials	Product	Process conditions	Bioactive compounds	References
Native maize starch (98%), bilberry extract (2%)	Extruded product	Twin-screw extrusion BT:100, 130, 160 °C SS:180, 300, 540, 720 rpm FR:10, 17, 16, 24, 30 kg/h MC:17, 18.6, 22, 22.5, 28.4%	Total anthocyanins (↓) Total phenolics (—)	Hirth et al. (2014)
Native corn starch (97.5%), red cactus pear powder (2.5%)	Extruded product	Twin-screw extrusion BT:80, 100, 120, 140 °C SS:225, 275, 325 rpm MC:18%	Total polyphenol (↓) Betaxanthins (↓) Betacyanins (↓) Betanin (↓) Isobetanin (↑)	Ruiz-Gutiérrez et al. (2015)
Oat flour (60.2%), potato starch (25.8%), apple pomace (14%)	Extruded product	Single-screw extrusion BT:80, 100, 120 °C DT:104, 115, 140, 165, 175 °C SS:180 rpm MC:21, 23, 26, 29, 30%	Total phenolics (↓) phenolic acids (chlorogenic acid Caffeic acid, *p*-coumaric acid, ferulic acid, rutin and phloridzin)	Leyva-Corral et al. (2016)
Rice or corn grits, freeze-dried cactus fruit (*Opuntia ficus-indica*) powder (0, 2, 6, 10%)	Extruded product	Single-screw extrusion BT:100, 140, 160 °C SS:160, 250 rpm MC:16%	Flavonols (—)	Moussa-Ayoub et al. (2015)
Rice flour (70%), horse gram flour (30%)	Snack-type products	Twin-screw extrusion DT:140, 180 °C SS:150 rpm FR:14 kg/h MC:14, 18%	Total polyphenols Total flavonoid contents (↓) Trypsin inhibitor (↓)	Gat and Ananthanarayan (2015)
Milled fractions of black rice (bran (B), polished rice (PR) and brown rice (BR))	Extruded black rice	Twin-screw extrusion BT:60, 100, 120 °C DT: 120 °C SS:200 rpm FR:25 kg/h MC:12–17%	Total phenolics (B↑, PR↓, BR↓) Total anthocyanins (B↑, PR↓, BR↓) Phenolic acids (gallic, chlorogenic, vanillic, caffeic, syringic, *p*-coumaric and ferulic acids)	Ti et al. (2015)

(continued)

Table 8.4 (continued)

Raw materials	Product	Process conditions	Bioactive compounds	References
Brown rice, wheat, and oat	Extruded cereals	Single-screw extrusion BT:50, 65, 85, 120 °C DT: 95 °C SS:26.6 rpm MC:30%	Total bound (↑) and free (↓) phenolic acids Phenolic acids (*p*-hydroxybenzoic, chlorogenic, vanillic, caffeic, syringic, *p*-coumaric, ferulic and sinapic acids)	Zeng et al. (2016)
Whole sorghum grains	Extruded grains	Twin-screw extrusion BT:30, 60, 90, 100, 100, 120, 120, 150, 150 °C SS:600 rpm FR:9 kg/h Final MC:12%	Total 3-deoxyanthocyanidins (↓) Total flavanones (↓) Proanthocyanidins (monomers, dimers (↑))	Cardoso et al. (2015)
Whole grain sorghum flour and roasted coffee powder (0, 10, 15, 20%)	Fiber-rich extruded products	Single-screw extrusion BT:60, 120, 140 °C SS:180 rpm FR:4.0 ± 0.9 kg/h MC:16, 20%	Total phenolics (↓) Phenolic acids (gallic, vanillic, chlorogenic, caffeic, syringic, *p*-coumaric, ferulic and sinapic, o-coumaric acids)	Chávez et al. (2017)
Lentil flour (100–80%), orange peel powder (0–20%)	Extruded product	Twin-screw extruder BT: 85, 105, 125, 100, 120, 140, 115, 135, 155 °C DT:130, 150, 170 °C SS:150, 200, 250 rpm FR:20.4 kg/h MC:14–22%	Total polyphenols (↓) Total flavonoids (↓) Total tannin (↓) contents	Rathod and Annapure (2017)
Jatropha cake	Protein hydrolysate	Single-screw extrusion DT:50–160 °C SS:50–240 rpm MC:28%	AOXC (↑) ACEI activity (↑) Trypsin inhibitor (↓) Lectin (n.d.) Phytic acid (↑)	Valdez-Flores et al. (2016)
Pea (20, 40%), rice (50, 55, 60, 70, 75, 80%), carob (0, 5, 10%), salt (0.5, 0.75, 1%), calcium carbonate (0.5, 0.75, 1%)	Gluten-free snack or breakfast cereal	Twin-screw extrusion BT:125 °C SS:900–950 rpm FR:25 kg/h	Inositol phosphates (IP3, IP4(↑), IP5(↑), IP6(↓)) α-Galactosides (↑) Trypsin (↓) and chymotrypsin (n.d.) inhibitors Lectin (↓) Anthocyanins (↓) Flavonols (↑) Total phenols (↑)	Arribas et al. (2018)

(continued)

Table 8.4 (continued)

Raw materials	Product	Process conditions	Bioactive compounds	References
Lentil flour, yeast (0, 4, 8, 12, 16%)	Snack-type products	Twin-screw extrusion BT:160 °C DT:140, 160 °C SS:500 rpm FR:20 kg/h	Total (↑), soluble (↑) and insoluble dietary fibers Phenolic compounds (↓) Total tocopherols (↓)	Ciudad-Mulero et al. (2018)
Broken rice, lyophilized açaí pulp (0, 5, 10, 15, 20%)	Pregelatinized flour	Single-screw extrusion BT:41, 61, 84 °C MC:12.5%	Total phenolics (↓) Monomeric anthocyanins (↓)	Oliveira et al. (2019)
Broken chickpea flour (50%), yoghurt (25%), potato starch (14%), yeast (1%), tomato powder (4%), spice (1%), locust bean gum (0–5%)	Snack-type products	Twin-screw extrusion BT:40, 50, 70, 90, 100 °C DT:130–150 °C SS:300–400 rpm FR:2.5 kg/h MC:17%	Phytic acid (↓) Condensed tannin (↓) Trypsin inhibitor (↓) Total, soluble and insoluble dietary fibers	Yağcı et al. (2020)
Red sorghum bran	Extruded product	Single-screw extrusion BT:60, 80, 110 °C DT:140–180 °C SS:100 rpm MC:25–35%	Total phenolics (↑) free total phenolics (↑) Bound total phenolics	Ortiz-Cruz et al. (2020)

BT Barrel temperatures, *DT* Die temperature, *SS* Screw speed, *FR* Feed rate, *MC* Feed moisture content, *nd* Not detected

content, the shear stress induced in extrusion cooking increases and therefore more dissipative heating results in greater destruction of anthocyanins. The results suggested that the increased feed rates allow a better retention of anthocyanins due to the low residence time even at the higher screw speed. The color of extrudates remained unchanged after storage of 40 months at room temperature in a sealed glass tube. The anthocyanin content in the extruded sample was preserved up to 82% of its initial value after storage. Ruiz-Gutiérrez et al. (2015) determined changes in the bioactive compounds of encapsulated red cactus pear powder during extrusion cooking process. The study showed that extrusion process caused a significant reduction in total polyphenol content and antioxidant activity compared to the raw mixture. The decrease in phenolic compounds may be due to the decomposition of phenolic compounds or alterations in their molecular structure resulting from the high extrusion temperature (Altan et al. 2009). Antioxidant activity depends on the level of bioactive compounds in extruded products and the

composition of these bioactive compounds (Brennan et al. 2011). The results of the study showed that the retention of betaxanthins in extrudates ranged from 46–63.5%, however it was in the range of 33–51% for betacyanins after extrusion. The loss of betalains pigments was affected not only by temperature and screw speed but also by the interaction effect of screw speed and temperature. The retention of betanin and isobetanin, which belong to the red-purple color betacyanins of red cactus pear, was determined to further investigate the reduction in betacyanins during extrusion. Ruiz-Gutiérrez et al. (2015) reported higher retention of betanin at lower temperatures, but an increase in isobetanin content under all extrusion conditions. It was indicated that structural changes in betanin occur at high temperatures during extrusion, leading to isomerization and decarboxylation of betanin, which produces its C15-stereisomer corresponding to isobetanin.

In the study of Leyva-Corral et al. (2016), mixtures of oat flour, apple pomace and potato starch were extruded using a single screw extruder. The effect of feed moisture content and extrusion temperature on the bioactive compounds in extruded products were determined. The phenolic compounds identified in the raw mixture and extruded products were chlorogenic acid, caffeic acid, *p*-coumaric acid, ferulic acid, rutin and phloridzin. The content of phenolic compounds other than ferulic acid tended to increase up to the extrusion temperature of about 140 °C or 150 °C, but further temperature increase resulted in a decrease of these phenolic compounds. These results were explained by the physical transformations and ruptures that occurred in the apple pomace due to the shear stress and temperatures during extrusion cooking process. The changes in the cell wall structure after extrusion can make phenolic compounds more accessible, resulting in an increase in phenolic contents. The reduction of rutin was attributed to the transformation of rutin into quercetin by loss of the sugar fragment. The results showed that intermediate extrusion temperature (140 °C) and moisture contents of 25–26% were the most appropriate conditions to maintain the amount of phenolic compounds detected in extruded products at a high level. It was reported that the antioxidant activity of extruded products did not change after extrusion.

Whole grains are good sources of phytochemicals such as carotenoids, phenolics and vitamin E. They have gained importance due to the increasing awareness of consumers about health. The level of phytochemicals in whole grains after processing such as extrusion cooking has been explored in recent studies. Ti et al. (2015) investigated the phytochemical profile and antioxidant activities of milled fractions (bran, polished rice and brown rice) of black rice before and after extrusion. They observed that the free phenolic content of rice bran increased by 17% but the bound phenolic content reduced by 15% after extrusion. In the case of the polished and brown rice, there was a significant reduction in free (53.4%, 25.0%) and bound phenolic content (79.5%, 24.5%) of rice samples. Phenolic compounds detected in the milled rice fractions were gallic, protocatechuic, chlorogenic, caffeic, syringic, *p*-coumaric and ferulic acids. Major phenolic acids found in the black rice bran were chlorogenic and ferulic acids. After extrusion, bound and total ferulic acid content increased by an average of 14%, but free ferulic acid content decreased by 4.5%. The amount of the free chlorogenic acid increased by 27%. The modification

of cell wall structure of rice bran during extrusion may cause the release of bound phenolics and breakdown the conjugated phenolics into free phenolics. Among the phenolic compounds of polished rice fractions, vanillic, *p*-coumaric and ferulic acids were found mainly in bound form. These phenolic compounds increased in the extruded polished rice, but there was a decrease in their free form after extrusion. High reduction in chlorogenic acid (75%) was observed among the free phenolic acids of extruded brown rice. The decrease in phenolics might be due to decarboxylation occurring in free phenolic acids during extrusion, which may promote polymerization of phenolics and tannins, leading to reduced extractability and antioxidant activity (Repo-Carrasco-Valencia et al. 2009; Brennan et al. 2011). Cyanidin 3-glucoside (Cy-3-G), cyaniding 3-rutinoside (Cy-3-R), and peonidin 3-glucoside (Pe-3-G) were identified in extruded milled fraction of rice. The total anthocyanin content of the black rice bran increased after extrusion, while a significant loss was observed for the polished and brown rice. The effect of improved extrusion cooking treatment in which gelatinization occurs at low temperature and high pressure on the phenolics of brown rice, wheat, and oat was studied by Zeng et al. (2016). The improvement in extrusion cooking process has been achieved by the modification of traditional single screw extruder with long screw, long residence time, high die pressure, low temperature and low screw speed. Extrusion cooking led to an increase of the bound phenolics in brown rice and oat by 4.47% and 15.60% but a significant loss of the free phenolics in brown rice (76.18%), wheat (38.63%) and oat (27.16%). The major phenolic acids found in raw and extruded cereals were determined as *p*-hydroxybenzoic, chlorogenic, vanillic, caffeic, syringic, *p*-coumaric, ferulic and sinapic acids. The decrease in free phenolic acids after extrusion has been attributed to the decomposition and increased polymerization of phenolic acids caused by the high temperature. Among cereals, extruded oat samples had the most significant increase in bound phenolic acids content (49.1%) after extrusion process. The results demonstrated that the impact of extrusion cooking process on phenolic acids was different depending on the cereals due to the difference in the cereal matrix and the sensitivity of free and bound phenolic acids during the extrusion process. The extrusion of whole grain sorghum has been attracted interest due to the bioactive compounds of sorghum. Cardoso et al. (2015) evaluated the effect of conventional dry heating in oven and extrusion cooking on the phenolic content of three sorghum genotypes. The whole grains were heated at 121 °C for 25 min in a conventional oven. The whole sorghum flour was extruded using a co-rotating twin-screw extruder. The retention of 3-deoxyanthocyanidins content in the genotypes after dry heating in a conventional oven were higher than about 83%, which indicates thermal stability of 3-deoxyanthocyanidins. It has been reported that the chemical stability provided by the absence of a hydroxyl group at position C-3 may contribute to the higher stability of 3-deoxyanthocyanidins. The average retention of the 3-deoxyanthocyanidins content in extruded sorghum genotypes was reported as 16.4%. After extrusion cooking process, apigenin and luteolin were not detected in sorghum genotypes but the retention of sorghum flavones varied between 28.8% and 68.3% in dry heated samples. On the other hand, the content of proanthocyanidin monomers and dimers increased three-fold after extrusion. Extrusion

cooking caused a significant reduction in antioxidant capacity of sorghum genotypes and this reduction was correlated with decreased 3-deoxyanthocyanidins content. In a study of Anunciação et al. (2017), the content of bioactive compounds in breakfast cereals produced from whole sorghum and wheat was investigated. The study demonstrated that the 3-deoxyanthocyanidins were present in the sorghum breakfast cereal, but not in the wheat breakfast cereal. Flavones and flavanones were not detected in either breakfast cereals. Total vitamin E content in whole wheat breakfast cereal was higher than in sorghum breakfast cereal. A higher γ-tocopherol content (59.5%) followed by α-tocopherol (28.9%) were found in the total vitamin E content of sorghum breakfast cereals. In whole grain wheat breakfast cereals, tocotrienols especially β-tocotrienol (80.4% of total vitamin E), were detected to be higher than tocopherols. Both tocopherols and tocotrienols are known as naturally occurring antioxidants present in cereals (Brennan et al. 2011). The whole sorghum breakfast cereals had higher antioxidant activity than whole wheat breakfast cereals. Chávez et al. (2017) studied the effect of extrusion on bioactive compounds and antioxidant capacity in extruded products formulated with roasted coffee powder and whole grain sorghum flour. They reported a significant reduction in total phenolic content in extruded sorghum, but the addition of coffee powder resulted in a significant increase in total phenolic content by 48% and 40% depending on the genotype of whole grain sorghum after extrusion. The study showed that the amount of chlorogenic and caffeic acids in extruded products increased by the addition of coffee powder in blend with sorghum flour. The free phenolic acids content after extrusion decreased, but the extrusion cooking process led to increase in bound phenolic acids in some cases. A significant positive correlation between total phenolic content and antioxidant capacity was reported.

The reduction in the antinutritional factors such as phytate or inositol hexaphosphate, trypsin inhibitors, chymotrypsin inhibitors and lectins has been reported after extrusion cooking of gluten-free legume-based formulations (Morales et al. 2015; Arribas et al. 2018; Yağcı et al. 2020). Phytate can form insoluble complexes with minerals, which reduces the absorption of minerals (Nikmaram et al. 2017). However, other inositol phosphates (IP) which are less phosphorylated phytate forms IP4, IP3, IP2 and IP have been reported to have an essential role in human health (Morales et al. 2015). The extrusion processing had a more pronounced effect in reducing the inositol hexaphosphate (IP6) content, but a significant increase in IP4 and IP5 was observed for both lentil and pea based extruded snack foods (Morales et al. 2015; Arribas et al. 2018). Morales et al. (2015) reported that the total inactivation of lectin in extruded lentil formulations and the reduction in trypsin inhibitor activity ranged from 93.2 to 97.61% depending on the different lentil formulations of extruded samples. Contradictory results were reported by Arribas et al. (2018), who determined that extrusion was effective in destroying trypsin and chymotrypsin inhibitor activities of pea-based extrudates. After extrusion processing, an increase in the total α-galactosides content has been reported for extrudate samples containing lentil and pea flour. The presence of α-galactosides in the extrudates has been considered to add value to the extrudates due to the prebiotic activity of α-galactosides (Morales et al. 2015; Arribas et al. 2018). Arribas et al. (2018)

reported that the phenolic groups of pea-based extrudates were affected differently by the extrusion cooking process. The content of anthocyanins decreased (4–50%) but flavonol content increased about threefold after extrusion. The total phenolic content of extrudates containing carob flour and 40% pea flour increased with the extrusion cooking process. This increase has been attributed to the phenols bound to the cell wall, which could be extracted more after extrusion process. Ciudad-Mulero et al. (2018) evaluated phytochemical composition and antioxidant activity of extruded snacks developed from lentil flour and nutritional yeast. They obtained a significant increase in total dietary fiber content of snack samples without yeast after extrusion at 140 °C and 160 °C. Extruded lentil samples containing different percentages of yeast had higher total dietary fiber compared to the raw sample. This increase was attributed to the increase of soluble dietary fiber. The modification in physicochemical properties of dietary fiber that occurs during extrusion processing due to high temperature and shear conditions can cause a redistribution of fractions of insoluble fiber to soluble fibers (Vasanthan et al. 2002; Ciudad-Mulero et al. 2018). Catechin hexoside was determined to be the most abundant phenolic compound in extruded lentil snacks. The total phenolic content and the content of individual phenolic compound were shown to decrease after extrusion treatment. Extrusion cooking caused a reduction of 81.5–92.0% in the total tocopherol content of lentil extrudates. The antioxidant activity of lentil extrudates decreased compared to the corresponding raw samples.

Recently, enrichment of extruded cereal snacks or cereal flour with freeze-dried vegetable and fruit has been reported (Gumul et al. 2018; Oliveira et al. 2019). Gumul et al. (2018) investigated the use of freeze-dried potatoes of red (Magenta Love) and purple (Blue Star) varieties in maize extrudates to increase the antioxidant capacity of extruded cereal snacks. The total phenolic content in extrudates enriched with red potatoes at 25% level that was 5.3 times higher than that of control extrudates. The highest content of chlorogenic acid in the extrudates was obtained with the addition of 25% dried red potatoes. It has been reported that there was a positive correlation between both antioxidant activity and antiradical activities and total phenolic content. In another study, Oliveira et al. (2019) studied the effect of extrusion on the bioactive compounds of pregelatinized flours prepared from broken rice grains and lyophilized açaí pulp. They observed significant losses in total phenolic and monomeric anthocyanins contents after extrusion of rice flour and lyophilized açaí pulp, even if the processing conditions were at mild extrusion conditions.

Extrusion cooking process has been used to improve bioactivities of *Jatropha* protein hydrolysate (Valdez-Flores et al. 2016). The effects of extrusion temperature and screw speed were found to be significant on the antioxidant capacity of protein hydrolysates, while antihypertensive activity was only affected by the extrusion temperature. The results of this study showed that the extrusion conditions under high temperature (160 °C) and screw speed (240 rpm) produced protein hydrolysates with high antioxidant capacity which might be due to the release of high amount of small peptides with high antioxidant capacity. In the case of antihypertensive activity, extrusion of protein hydrolysates at high temperature (105 °C)

exhibited high hydrolysis degree and antihypertensive activity. Valdez-Flores et al. (2016) concluded that high temperatures and short processing time in extrusion cooking process result in changes in protein making more available sites for the cleavage of peptide bonds and thus generating protein hydrolysates with different levels of hydrolysis degree.

8.5 Extruded Functional Foods Produced by Microencapsulation

Extrusion processing variables play a key role in changing physicochemical and nutritional properties of the extruded products. The use of whole grain and composite flour, legumes or different raw materials containing bioactive compounds as a functional ingredient has been increasingly explored to increase the content of bioactive components in extruded products. However, several studies have shown that the effect of extrusion processing on reducing the content of bioactive compounds in extruded products. Encapsulation has been used as a strategy that could protect the functional ingredients during extrusion process (Favaro-Trindade et al. 2020; Shrestha et al. 2012).

In the study of Favaro-Trindade et al. (2020), the proanthocyanidin-rich cinnamon extract (PRCE) was prepared from cinnamon barks. The extract was spray-dried and encapsulated using gelatin and gum arabic by the complex coacervation technique. The spray-dried and encapsulated PRCE particles mixed with cornmeal were extruded at 110 °C and 130 °C with shear rates of 500/s and 1000/s, respectively. The retention of total carotenoids in cornmeal extruded products ranged from 12.42% to 52.11% after extrusion. A higher retention of carotenoids was obtained at a lower extrusion temperature. The combination of high temperature (130 °C) and lower shear rate (500/s) resulted in a decreased retention in total phenolics of extruded samples containing spray-dried and encapsulated PRCE. The retention varied from 18% to 26% for samples containing spray-dried PRCE. The phenolics in samples containing spray-dried PRCE were more sensitive to the extrusion processing than those in extruded products containing encapsulated PRCE, where the retention changed from 39% to 78%. The use of lower extrusion temperature resulted in higher retention of proanthocyanidins in extrudate samples. The retention in encapsulated PRCE was around 3 times higher than the spray-dried PRCE. The results of this study demonstrated that encapsulation was effective in protecting phenolics from cinnamon extract against the extrusion conditions. Shrestha et al. (2012) investigated the microencapsulation of 5-methyltetrahydrofolic acid (5-MTHF) and evaluated the stability of the encapsulated vitamin under extrusion conditions. A combination of pectin and sodium alginate was used for encapsulation of 5-MTHF by spray drying. The encapsulated 5-MTHF powder mixed with maize starch was extruded at a screw speed of 200 rpm and different barrel temperature profiles. Starch extrudates containing encapsulated powder had a high 5-MTHF retention ranging from 84.8% to 94.5%, while the retention in extrudates

with unencapsulated 5-MTHF powder varied from 65.3% to 83.2% after extrusion. The retention of 5-MTHF in extrudates fortified with unencapsulated powder decreased with the increase in extrusion temperature from 100 °C to 150 °C. It was shown that the microencapsulation provides better protection to 5-MTHF compared to unencapsulated powder especially at higher extrusion temperatures. Few studies have been performed so far to evaluate the stability of encapsulated bioactive compounds during extrusion processing. Extrusion technology described as hot-melt extrusion itself has also been used to encapsulate bioactive compounds in recent studies (Tackenberg et al. 2015; Khor et al. 2017; Chang et al. 2019).

Microencapsulation can be defined as the process in which active materials are surrounded by a coating wall to form small capsules. Microcapsules consist of two components which are a core and a shell material. The core material contains internal phase or fill of the active ingredient while the shell or wall material surrounds the core material to protect the active ingredient (Bakry et al. 2015). The wall material used in hot-melt extrusion can be starch, maltodextrin, modified starches, sugars, cellulose ethers (hydroxypropyl cellulose or hydroxypropyl methyl cellulose), proteins, and/or gums (Zuidam and Heinrich 2010). Encapsulation of active ingredients by extrusion is similar to the process used to make expanded cereal snack products. In the feeding zone, the screw is designed to create a low pressure that provides homogenization of the feeding. At this section, the wall material is introduced with plasticizer such as water into extruder. The pressure is gradually increased depending on the screw design in the subsequent zone(s). The melt is obtained and further homogenized in this section. The active ingredients may be added in the mixing/dispersing zone of the extruder. The minimum residence time of the active ingredients will avoid exposure to the relatively high temperatures required to plasticize the wall material such as starch. The molten material is ejected under high pressure through a die head. The shape of the final product depending on the die geometry can be sheet, rope or thread. A rotating knife can be used after the die head to cut the extrudates into smaller pieces. The granular form of extrudates can alternatively be obtained by grinding using grinders or mills (Zuidam and Shimoni 2010). Hot-melt extrusion can be an advantageous process for encapsulation applications due to a high throughput continuous process and many unit operations such as melting, mixing, and cooling performed simultaneously. There is no need for a post-extrusion drying process because melt can be obtained at low water content levels (Castro et al. 2016). The enhanced stability of essential oils with less surface oil compared to the spray drying has been reported for the microencapsulated oils by extrusion (Gouin 2004). However, hot-melt extrusion is more expensive, which is almost double the cost, compared to spray drying (Bakry et al. 2015). The particle size formed by extrusion typically ranges from 500 to 1000 μm, which limits their use in some food applications where mouthfeel is a critical factor (Gouin 2004).

Orange terpenes were encapsulated in a mixture of maltodextrin and sucrose at different mixing ratios using extrusion process (Tackenberg et al. 2015). The authors used a decreasing temperature profile in the extruder barrel. The temperature used in the conveying zone ranged from 110 °C to 165 °C, while the mixing zone temperature changed from 115 °C to 110 °C followed by the rest zone temperatures of

90–80 °C and 35–20 °C. The die temperature was between 40 °C and 80 °C. A water feed rate was adjusted between 4.0% and 5.7%. The amount of orange terpenes was around 6.0% or 7.9%. Product temperature measured after the die exit varied between 64 °C and 80 °C. The extruded products obtained had amorphous and partly crystalline matrices. The orange terpenes were encapsulated in the range of 2.1–4.1%, corresponding to an orange terpenes retention of 34.5–67.3% in extrudates. Khor et al. (2017) investigated microencapsulation of quercetin by hot-melt extrusion for taste-masking. The wall materials, carnauba wax, shellac or zein were plasticized with 20 w/w% water and mixed with different ratios of quercetin (20–50:80–50). The mixture was extruded without using a die at 80 °C or 90 °C and screw speed of 100 rpm. The taste masking efficiency was evaluated by dissolution of the microencapsulated powders in simulated enzyme-free saliva. In vitro digestion of the microencapsulated powders was also determined in simulated enzyme-free gastric and intestinal fluids. The theoretical quercetin content in carnauba wax and shellac-microencapsulated powders was found to be closer to the theoretical value compared to that of zein-microencapsulated powders, especially at lower quercetin content. The more melting of carnauba wax and shellac probably resulted in a better mixing of quercetin with carnauba wax and shellac than zein. Among the three wall materials, the lowest dissolution of quercetin in simulated digestion conditions was observed for zein. This was due to its high hydrophobicity and non-erosion in the simulated enzyme-free digestion solutions. This study demonstrated that microencapsulation by hot-melt extrusion can be used as an effective method to mask the bitter taste of polyphenol compounds.

In another study, Chang et al. (2019) studied the encapsulation of ascorbic acid in maltodextrin, maltodextrin-gum arabic and maltodextrin-trehalose using a holt-melt extrusion technology. The ratio of maltodextrin in the formulation was 80.5%, and its ratio in the mixture of maltodextrin-gum arabic and maltodextrin-trehalose were 70.5% and 78.5%, respectively. The oil-water emulsion was prepared and mixed with 16% concentration of acetic acid and the rest of solid materials in the formulation. The barrel zone temperatures were 85 °C, 105 °C, 120 °C and 105 °C, respectively. The melt temperature measured in the extruder ranged from 108.73 °C to 111.92 °C. The low residence time of material (<3 min) was reported for all samples. The high ascorbic acid yield of over 97% indicated that ascorbic acid could be encapsulated by hot-melt extrusion with minimal loss. The milled extrudates having particle size in the range of 20–40 mesh showed a fast dissolution rate. The incorporation of trehalose lowered the glass transition temperature, resulting in a more efficient extrusion process.

8.6 Conclusions and Future Perspectives

There is an increasing interest in alternative raw materials rich in bioactives to produce food products due to the increasing consumer's demands for nutritious and healthy foods. Extrusion is a versatile technology that can be used in various applications such as production of a wide variety of foods from different raw materials,

oil extraction and encapsulation. In recent years, studies have focused on the extrusion of whole grains, legumes, fruit by-products and fruit extracts or powders due to their bioactive compounds to obtain functional extruded products. Several studies have shown a significant reduction in total phenolics content of extruded products after extrusion cooking. However, the increase in free/bound phenolic acids content has been reported in some cases due to the modification of structure and increased release of these compounds. Although the losses in total anthocyanins occurred during extrusion, an increase in levels of monomeric and dimeric proanthocyanidins in extruded products has been reported. Recent studies have shown that extrusion cooking causes the reduction or partial inactivation of antinutrient factors in legumes, but also increases bioactive compounds such as inositol phosphates and α-galactosides. The changes in these bioactive compounds have shown to depend on the extrusion processing conditions, nature of raw materials and other active ingredients used. Therefore, by optimizing processing variables such as temperature, screw speed and moisture content, the losses of bioactive compounds can be minimized, thus enhancing the bioactivity of extruded products. Recently, encapsulation of active ingredients has been proposed to protect bioactive compounds during extrusion cooking. The flexibility of the extrusion process allows to design a wide variety of food products from different raw materials rich in fiber and bioactive compounds. On the other hand, the bioaccessibility and bioavailability of bioactive compounds retained after extrusion in extruded products should be investigated by *in vivo* or *in situ* animal models. Extrusion technology also offers a unique opportunity for encapsulation of active ingredients by combining several steps in one step without the need for a post-treatment after extrusion. Microencapsulation by extrusion can be used to design new delivery systems which allow for not only protection of the active compounds but also their controlled release.

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Chapter 9
Influence of Fermentation and Germination on Food Bioactives

Fereshteh Ansari and Hadi Pourjafar

9.1 Introduction

Food processing is a very crucial issue in the present century. The population is growing fast and the world society is facing increased demand for adequate and healthy foods. Climate change, food waste, and urbanization are other factors that are thought to increase the need for improved techniques for processing foods. The availability of water and cultivable soil has been decreased in many regions due to the changing climate which causes a drop in agricultural productions (Xie et al. 2021a). Annually a great section of the produced foods is wasted through reasons such as poor storage facilities, poor infrastructure and transportation, and inadequate market facilities (Schanes et al. 2018). On the other hand, urbanization decrease the labor force of agriculture and the expanded urban areas are continually disappearing the adjacent land resources. Urbanization will also increase food consumption and the need for upgraded foods (Xie et al. 2021a).

Fermentation and germination are two of the food processing techniques which are applied in order to improve the availability of agricultural products throughout the year, consumer's acceptability, and retaining the nutritional value of the foods. These techniques have been traditionally applied in many countries. Some of the

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used techniques are special for a location and others are practiced universally. For instance, fermentation and malting are applied in developing countries of Africa and South America, while nixtamalization is mostly practiced in Mexico (Nkhata et al. 2018).

During the fermentation process, the food undergoes controlled microbial growth and fermentation. Microorganisms such as yeasts and bacteria are responsible for this anaerobic process which breaks down food compounds (especially sugars) into acids and alcohols (Moodley and Kana 2019). The food "ogi," produced by acid fermentation of sorghum, millet, or maize, is widely consumed in West Africa and "chicha" and "masa" are common South American countries' fermented foods made from fermented maize (Nkhata et al. 2018). Cultured milk and yogurt, wine, beer, cider, tempeh, miso, kimchi, sauerkraut, and fermented sausage are common examples of fermented foods (Marco et al. 2021). Germination is a different process in which edible seeds such as cereals and legumes are decontaminated, soaked in the water, and then germinated (sprouted). The sprouted seeds are then used for many kinds of foods such as flours, desserts, and beverages (Samofalova and Safronova 2017).

During both of the techniques, the ingredients of the substance products undergo alterations which can have a broad effect on the structure, and content of the product as well as the concentration and bioavailability of bioactive. Despite all the positive effects of fermentation and malting on the elongation of shelf life, sensory properties, transferability of the products, reducing anti-nutritional factors, and increasing nutritional availability some negative points are also reported. Due to the widespread use of fermentation and germination across the world which constitute a large section of the population diet, we provide this chapter to thoroughly review the various effects of the processing techniques on the essential nutrients of the produced foods.

9.2 Energy Content of Food

Fermentation is expected to affect the energy content of food. Fermentation occurs in anaerobic environments and starts with glycolysis which breaks down glucose into two pyruvate molecules and creates 2 ATP and two NADH. Fermentation permits glucose to be uninterruptedly broken down to create ATP by reason of the recycling of NADH to NAD+ (McFeeters 1988). In fact, throughout lactic acid/alcoholic fermentation, a great quantity of the sugars is metabolized, nevertheless, the energy content produced to either lactic acid or ethanol is only 2 mol ATP/mol hexose (Fig. 9.1). This amount of energy is comparable to the production of 38 mol ATP/mol of hexose once sugar is totally oxidized (Fig. 9.2). Therefore, almost 95% of the energy existing in the sugars remains subsequently the fermentation process (McFeeters 1988; Jayanegara et al. 2011).

The phase of seed germination is the preliminary point of the lifecycle, and it needs energy for its metabolic demands. Immediately after seed germination

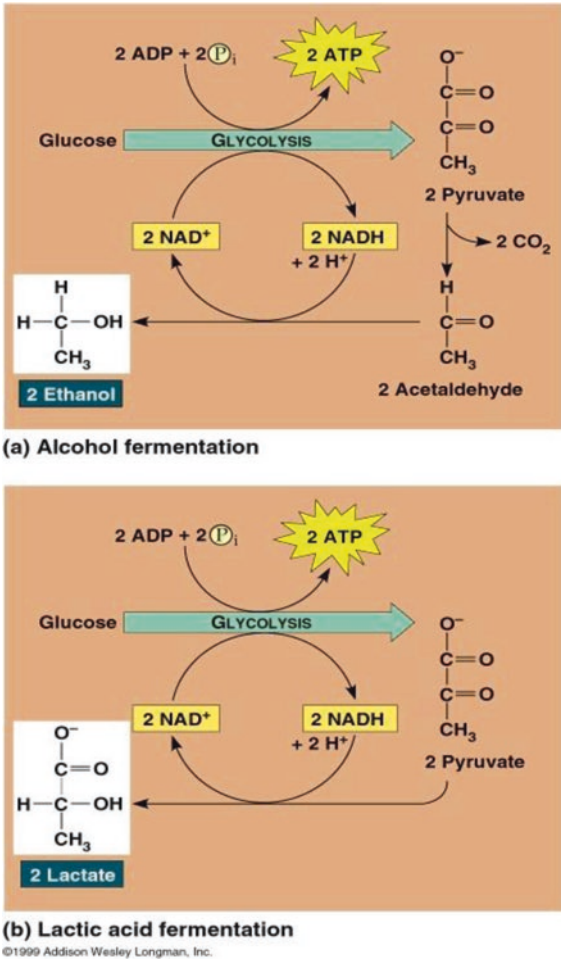

Fig. 9.1 Lactic acid and alcoholic fermentation (Source: https://www.createwebquest.com/tuckerr/rachel-tucker-webquest-lactic-acid-vs-fermentation)

commences, a sharp rise in CO_2 release and O_2 uptake occurs, and the ATP amount starts to rise swiftly (Steinbrecher and Leubner-Metzger 2017; Benamar et al. 2008). Evaluates of the Arabidopsis transcriptome throughout seed germination have proposed that fermentation, glycolysis, the tricarboxylic acid (TCA) cycle, and the oxidized pentose phosphate pathway (OPPP) are stimulated throughout the germination process (Weitbrecht et al. 2011). In fact, ATP is formed at the initial phase of seed germination and offers the energy for metabolism. It is thought that Perl's pathway is the source of ATP, nonetheless, this has not yet been approved (Qu et al. 2020; Zhang et al. 2015a). Recently, Qu et al. (2020) provided investigational information display that Perl's pathway (Fig. 9.3) takes part in providing energy throughout the initial phases of poplar seed germination.

According to Qu et al. (2020), the alters in respiration rate throughout poplar seed germination are revealed in Fig. 9.4a. The respiration rate commenced to rise

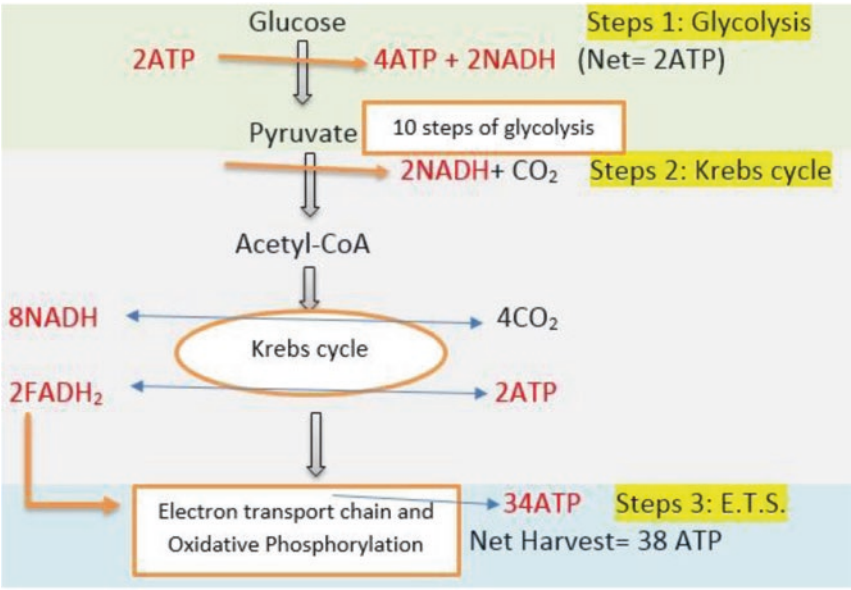

Fig. 9.2 Total oxidation of sugar in three steps (Source: https://www.microbialfacts.com/steps-of-cellular-respiration/)

at 0.5 h, and reached 1.387 μmol mol^{-1} at 0.75 h, 2.131 μmol mol^{-1} at 6 h and 2.536 μmol mol^{-1} at 24 h of germination**.** These consequences display that ATP synthesis started to recover at 0.75 h of seed germination. Some ATP was found at the initial phase of poplar seed germination, nevertheless, the ATP amount rose considerably at 0.75 h and continued to rise subsequently 6 h (Fig. 9.4b), consistent with the alters in the respiration rate.

9.3 Description of Important Nutritional Changes

9.3.1 Carbohydrates

9.3.1.1 Fermentation

The main carbohydrate in cereals (as the major foods in most countries) is starch which offers the highest level of calories. Fermentation stimulates starch-hydrolyzing enzymes for instance maltase and α-amylase which break starch into maltodextrins and simple sugars respectively, and finally, due to the continuous activity of these enzymes, glucose levels increase. The same thing happens in the process of microbial fermentation in different foods, and within 24 h, the amount of carbohydrates present decreases, and the amount of glucose increases (Nkhata et al. 2018). Once

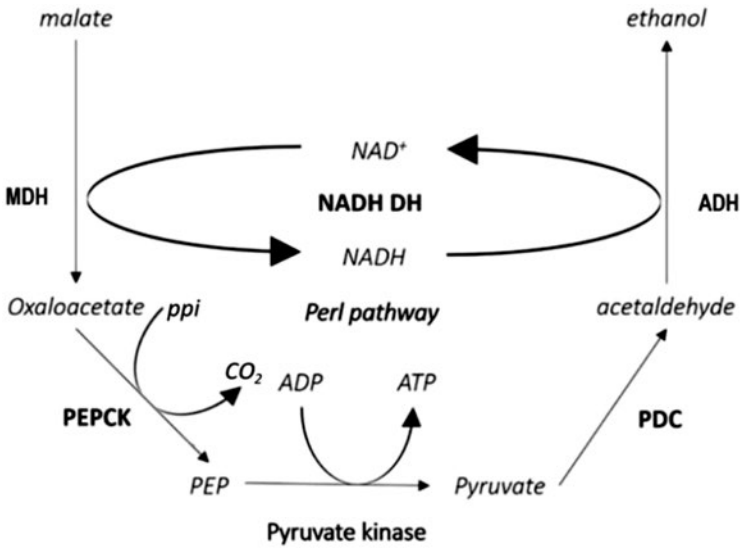

Fig. 9.3 Schematic illustration of Perl's pathway. *ADH* Alcohol dehydrogenase, *MDH* Malate dehydrogenase, *NADH-DH* Nicotinamide adenine dinucleotide dehydrogenase, *PDC* Pyruvate decarboxylase, *PEPCK* Phosphoenolpyruvate carboxylase, *PEP* Phosphoenolpyruvate (Source: Qu et al. (2020))

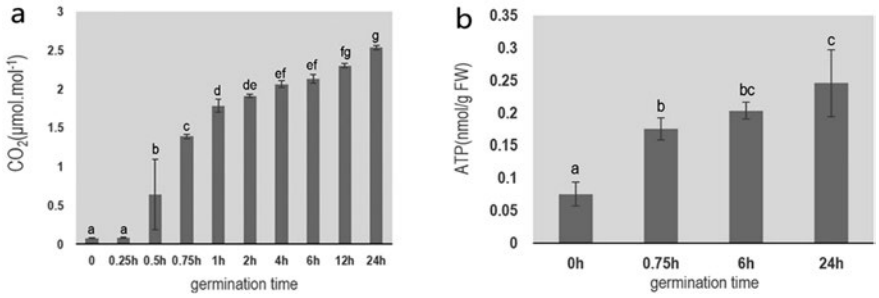

Fig. 9.4 Alters in ATP amount and CO_2 release rate throughout the initial phase of poplar seed germination. (**a**) CO_2 release rate throughout poplar seed germination. (**b**) Alters in ATP amount throughout poplar seed germination (Source: Qu et al. (2020))

both fructose and glucose are existing through the fermentation process, microorganisms choose glucose to fructose as a source of energy subsequently the amount of fructose keeps on constant (Nkhata et al. 2018; Diether and Willing 2019). Furthermore, the starch amount decreases with succeeding enhance in CO_2 and ethanol creation during fermentation time. Additionally, pH reduces considerably which stimulates the phytase enzyme (Alexander et al. 2019).

In dairy products, where lactose is the major sugar present, fermenting microorganisms convert lactose into lactic acid, causing a significant drop in pH. In fact,

this process occurs in two forms, Homofermentative and heterofermentative. In the first form, due to the fermentation of lactose by microorganisms such as *Lactococcus lactis*, only lactic acid is produced, which in addition to lowering the pH in the product causes milk clotting and is involved in the production of fermented dairy products such as yogurt and cheese (Homayouni et al. 2018; Abdolhosseinzadeh et al. 2018; Homayouni et al. 2020). It also creates a lactic and sour taste in the product which is desirable to a certain extent. In heterofermentative form, due to the fermentation of lactose by microorganisms such as *Lactococcus diacetylactis*, in addition to lactic acid, other compounds such as acetaldehyde (yogurt flavor index), diacetyl (butter flavor index), CO_2, and other compounds are produced. They are effective in creating flavor, aroma, and gas in different types of dairy products (Thrune et al. 2009; Walstra et al. 2005; Pourjafar et al. 2020).

Also, fermentation is broadly practiced in the meat industry as a technique of making and preserving meat products e.g. fermented sausages. In meat products, fermentation process has beneficial effects on the final product, including the creation of a favorable taste and odor, and antimicrobial properties. Most of these useful products are related to the fermentation of sugars (mainly glucose) used in the formulation of meat products and the production of substances such as lactic acid and ethanol and lowering the pH (Homayouni et al. 2020; Pourjafar et al. 2020; Yilmaz and Velioglu 2009; Laranjo et al. 2019).

In fermented beverage, for example, Kombucha, chemical analysis of kombucha have shown the existence of several organic acids, such as lactic, acetic, glucuronic, gluconic, citric, malic, malonic, tartaric, succinic, oxalic, usnic, pyruvic; as well sugars, for instance, fructose, sucrose, and glucose (Jayabalan et al. 2014; Ansari et al. 2017). Some microorganisms (yeasts and bacteria) are involved in such metabolic productions that use substrates by diverse ways. For example, yeasts hydrolyze sucrose into glucose and fructose via invertase and yield ethanol by glycolysis, with a predilection for fructose (as a substrate). Also, acetic acid bacteria use ethanol to yield acetic acid and glucose to yield gluconic acid, and actually, the pH rate of beverage declines due to the creation of organic acids throughout the fermentation process (Jayabalan et al. 2014; Ansari et al. 2017; Villarreal-Soto et al. 2018; Coelho et al. 2020; Villarreal-Soto et al. 2019; Ansari et al. 2019).

9.3.1.2 Germination

The effect of germination on carbohydrates mainly depends on the activity of hydrolytic and amylolytic enzymes, which leads to a decrease in the amount of starch and an increase in the amount of simple sugars thus improving digestibility (Nkhata et al. 2018; Oghbaei and Prakash 2016). In fact, germination facilitates the enzymatic breakdown of complex carbohydrates such as starch into simple sugars including glucose (through stimulation of endogenous enzymes e.g. α-amylase), which can easily be used to provide the energy needed for grain growth (Nkhata et al. 2018; Zhang et al. 2015b). Ghavidel and Prakash (2007) reported that the

digestibility of starch of cowpea, green gram, chickpea, and lentil increased by 53%–82% subsequently germination for 24 h.

Tian et al. (2010) showed that germination in oat seeds for 24–144 h decreased starch amount from 60% to 21%, whereas simple sugars enhanced from 5% to 28%. In another study, germination of kidney, soybean, mung beans, and peanuts disclosed an increase in whole sugars by 14%, 19%, 22%, and 26%, respectively (Megat et al. 2016). Fructose and glucose levels are normally small in the raw cereals, nevertheless, during the germination process, these simple sugars surge considerably such that their levels surpass that of sucrose stimulation of invertase which hydrolyzes sucrose into fructose and glucose throughout germination (Traoré et al. 2004).

Germination may perhaps have an actual role in improving the fiber amount in food products (Nkhata et al. 2018; Rumiyati et al. 2012; Jan et al. 2016). It has been shown that the germination of peas and lupin could increase fiber amount by 100% and 456% on a dry basis, respectively (Rumiyati et al. 2012). In fact, decreased dry matter due to enzymatic hydrolysis of starch and microbial breakdown of cellular macromolecules such as carbohydrates, fats, and proteins may explain the increase in fiber. It can also consequence from the rise in the cellular construction of the plants as they germinated. The crude fiber comprising of hemicelluloses, cellulose, and lignin enhances considerably throughout germination progression as the plant cells create diverse cellular elements (Nkhata et al. 2018; Laxmi et al. 2015).

Increasing the amount of fiber in the food is desirable as increasing the amount of dietary fiber actually reduces the release of glucose, which is especially useful for people with diabetes (Yu et al. 2014). Since digestive enzymes are unable to break down dietary fiber, these intact compounds reach the colon and are broken down thereby bacteria, eventually producing short-chain fatty acids such as butyric acid, propionic acid, and acetic acid which are useful for the body (Nkhata et al. 2018; Byrne et al. 2015). For example, butyrate and acetate play an important role in weight loss in obese individuals by stimulating lipolysis in adipocytes (Rumberger et al. 2014).

9.3.2 Proteins

9.3.2.1 Fermentation

Fermentation is applied to a wide variety of foods such as dairy products, legumes, cereals, and meat products, which are significant protein resources. Deviations in the nutritional rate of proteins as an outcome of the fermentation process are predominantly significant for legumes and cereals (Nkhata et al. 2018; McFeeters 1988). The main aim for fermentation of protein-rich foods is to adjust the texture or flavor properties of the starting-food components. These alterations mostly are produced via a fermentation process that is restricted both in the time and extent to which fermenter organisms are permitted to grow. Consequently, great variations in

entire protein content would not typically be anticipated (McFeeters 1988; Tangyu et al. 2019; Norouzi et al. 2019).

Albeit alterations in the extent of protein by fermentation process seem to be slight or nonexistent, significant studies has been done to explore changes in the nutritive value of the protein, and the consequences of these investigations recommend that protein quality can be upgraded via a fermentation process in some cases (Nkhata et al. 2018; Diether and Willing 2019; Tangyu et al. 2019; Rollán et al. 2019; Rui et al. 2019; Xie et al. 2021b). On the contrary, some investigations exhibited no considerable alteration in protein quality (Tahir et al. 2018; Lilis 2019). Hence, if the fermentation process is to be employed for the purpose of improving protein quality, processing parameters (temperature, time, relative humidity, and so on), components, and fermentative microorganisms that can provide improvement require to be well-defined for each instance.

The nutritional quality of proteins depends mainly upon the quantity and availability of the restrictive essential amino acid in a diet (Nkhata et al. 2018; McFeeters 1988). There is the opportunity that throughout fermentation process the entire quantity of any specific amino acid can enhance or decline that the availability can alter considerably. For most products, especially fermented foods, the restrictive amino acids are lysine or sulfur amino acids (Nkhata et al. 2018; McFeeters 1988; Lilis 2019).

Osman (2011) showed that fermentation of pearl millet (for 24 h) enhanced protein amount due to loss of carbohydrates. In this study, after fermentation, the amount of some amino acids such as arginine, lysine, and glycine were decreased (Osman 2011). However, in a similar study by Penka and Petrov (2020) the amount of methionine was enhanced (Penka and Petrov 2020).

Even though enhanced protein content can somewhat be related to loss of dry matter throughout the fermentation process, microbial fermentation is recognized to rise and concentrate lysine amount in grains (Nkhata et al. 2018). This surge can somewhat be due to the breakdown of complex proteins via fermenting microorganisms, yielding amino acids and peptides (Pranoto et al. 2013). Nevertheless, it is shown that fermenting microorganisms use amino acid which can reduce the protein content and quality of various fermented food products (Nkhata et al. 2018; Pranoto et al. 2013).

Studies have shown that the phenomenon of fermentation increases the digestibility of plant proteins (Pranoto et al. 2013; Alka et al. 2012; Kumitch et al. 2020; Çabuk et al. 2018; Ranjan et al. 2019; Ogodo et al. 2019). In fact, plant proteins are less digestible compared to animal proteins, which makes the digestive system unable to digest these substances, and eventually, most of the intact plant proteins are excreted in the feces. Henceforth, amplified protein digestibility can diminish the amount of undigested proteins which may be the reason for food allergies because of low absorption in the gastrointestinal tract (Manuyakorn and Tanpowpong 2019; Untersmayr and Jensen-Jarolim 2008).

The most important role of fermentation in the digestibility of plant proteins is the partial breakdown of the complex storage proteins into further soluble forms (Jannathulla et al. 2019; Jannathulla et al. 2018). Subsequently, the efficiency of the

fermentation process depends on the stimulation of the phytase enzyme, it is not astonishing that fermenting heated or baked grains does not decrease phytic acid considerably as heating or baking abolish phytase enzyme (Egli et al. 2003). In a study, the impact of natural fermentation and *L. plantarum* on protein digestibility of sorghum flours was investigated, and the results showed that protein digestibility was enhanced up to 47% and 92% respectively (Pranoto et al. 2013). In fact, this surge was associated with enhanced proteolytic enzymes of *L. plantarum* that can not only destroy tannins that compound with proteins but also destroy complex proteins, releasing further amino acids and peptides. Inappropriately, fermenting microorganisms can use proteins and amino acids throughout the fermentation process resulting in reduced amount of proteins and amino acids in fermented foods (Nkhata et al. 2018; Pranoto et al. 2013).

9.3.2.2 Germination

So far, several studies have been performed on the effect of germination on protein content of cereals and legumes (Gulewicz et al. 2008; Ghumman et al. 2016; Eun-Ji et al. 2017; Ramirez-Peralta et al. 2012). Some studies have shown an increase in protein during germination, and this increase in protein depends on the type of grain or legume (Laxmi et al. 2015; Otutu et al. 2014). In contrast, studies have reported a decrease in protein during germination (Verma and Rao 2006), although in some cases, along with a decrease in total protein, an increase in some amino acids, such as tryptophan, methionine, and lysine have been reported (Bhathal and Kaur 2015). Therefore, said it is indicated that the effect of germination on the protein content of the product is a complex issue and several factors can affect it.

In the germination process, an increase in protein content can occur following a decrease in dry weight, such as a decrease in the composition of some compounds such as fats and carbohydrates during respiration, while some amino acids are also synthesized and increased (Nkhata et al. 2018; Jan et al. 2016). Furthermore, protein content reduction throughout the germination process has been related to their degradation via protease enzymes (Nkhata et al. 2018). Consequently, the actual protein amount can be determined via the net outcome of breakdown and synthesis. Generally, it gives the impression that the net protein the synthesis overshadows breakdown because of the serious necessity for synthesis of nucleic acids mandatory for evolution, which can impact a net rise in proteins (Nkhata et al. 2018; Moongngarm and Saetung 2010).

Studies have also shown that the germination process increases the digestibility of proteins in products such as cowpea and chickpea by up to 18%, which is nutritionally valuable (Nkhata et al. 2018; Ghavidel and Prakash 2007). Also, Mbithi-Mwikya et al. (2000) showed that germination enhances the digestibility of proteins in finger millet by 64%, that the increase was due to proteolysis and limited solubilization that arises with germination of the seeds, as proved via enhanced free amino acids and water-soluble proteins in the sprouted products (Mbithi-Mwikya et al. 2000). Contradictory consequences are representing a reduction in protein amount

in white sorghum possibly due to the variances in germination parameters such as time, temperature, culture, and steeping (Nkhata et al. 2018; Ogbonna et al. 2012).

9.3.3 Vitamins

9.3.3.1 Fermentation

Fermentation process affects the vitamin content of foods via different mechanisms (see Fig. 9.5). B vitamins are among the most important vitamins that undergo changes in the fermentation process in different types of fermented foods.

Thiamin (vitamin B1) is among the water-soluble B vitamins, and naturally existing in some foods, added to some food products, and can also be consumed as a dietary supplement. Aliya and Geervani (1981) showed increases of vitamin B1 from 30 to 150% in Ambali and Dhokla fermentations. In another study, the amount of Thiamin enhanced in soy idli with either a natural fermentation or fermentation of sterilized batters inoculated with *S. faecalis* or Lactobacilli (Rajalakshmi and Vanaja 1967). In a similar study, the amount of Thiamin reduced when sterilized batters were inoculated with either *A. cloacae*, *L. plantarum*, or a mixture of these microorganisms (Kiin-Kabari et al. 2018). In general, it can be concluded that the decrease or increase of vitamin B1, as well as its amount during the fermentation process, depends on several factors such as time, temperature, type of food, and type and number of fermenting microorganisms (McFeeters 1988; Xing and Edwards 2019; Labuschagne and Divol 2021).

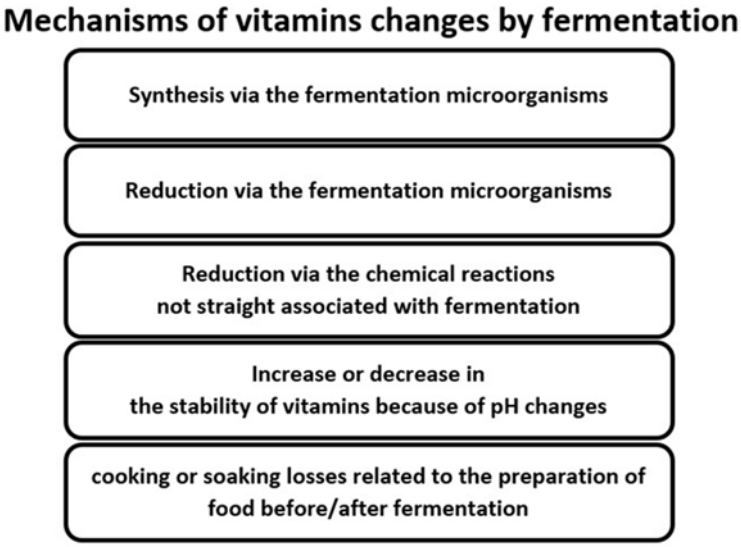

Fig. 9.5 Summary of important mechanisms of changes in vitamins via fermentation process

Riboflavin (vitamin B2) belongs to the water-soluble B vitamins group, and found in various foods and is also employed as a dietary supplement. It is essential for the body and especially cellular respiration demands this vitamin. Riboflavin variations have been studied mainly in legume and cereal fermentations (McFeeters 1988; Witten and Aulrich 2018). The fermentation process, depending on the type of food and its fermentation conditions, may reduce the Riboflavin amount in the product, for example in the preparation of Tofu (Fernando and Murphy 1990); or there may be no significant changes in the amount of riboflavin in the product during fermentation, for example, such as in the fermentation of milk by lactic acid bacteria (Alm 1982a); or fermentation may increase the amount of this vitamin, which is mostly the case, for instance, in the preparation of products such as tempeh and miso from chickpea, horsebean and soybean (Robinson 1977), and the fermentation of cowpeas (Zamora and Fields 1979), melon seed, and African oil bead seed (Achinewhu and Ryley 1986).

Niacin (vitamin B3) is a B vitamin that is synthesized and employed by the body to turn food into energy. Several studies have shown that the fermentation process causes a significant increase in Niacin content in fermented products, for example, a five-fold increase in this vitamin has been reported in soy tempeh (Robinson 1977; Van Veen and Steinkraus 1970; Xiang et al. 2019; Jamuna et al. 2020). Surges in Niacin have also been reported in natural khaman and idli fermentation too (Rajalakshmi and Vanaja 1967; Ramakrishnan et al. 1976). Also, Niacin changes have been measured in batters fermented with some lactobacilli, *S. faecalis*, and *A. aerogenes*. The amount of Niacin enhanced considerably overhead the sterilized control in each case. *L. fermenti* strain mainly caused enhances which were comparable to the 40% rise detected in a natural fermentation (Ramakrishnan et al. 1976). On the other hand, some studies indicated no significant changes in the amount of this vitamin during fermentation. For example, Alm (1982b) observed virtually no alteration in the amount of Niacin in fermented milk samples prepared with the common lactic acid bacteria. In rare studies, a decrease in Niacin levels during fermentation has also been reported. For example, Zamora and Fields (1979) showed a considerable reduction of Niacin throughout the fermentation of chickpeas and cowpeas.

Pantothenic Acid (Vitamin B5) is among the water-soluble B vitamins, and it is synthesized from the pantoic acid and amino acid β-alanine (Kelly 2011). Studies have shown that the amount of this vitamin does not change during the fermentation process of milk, except during the fermentation and production of yogurt, which is reduced by a maximum of 30% (Alm 1982b). During the ripening of the cheese, usually during the first 2 months of storage, the amount of this vitamin decreases, but after that, increases over time (Lodianov et al. 2021; Nilson et al. 1965). The changes in the amount of this vitamin, similar to the previous vitamins described, also depend on the type of product and the fermentation conditions. Van Veen and Steinkraus (1970) detected a 28% reduction in pantothenic acid throughout the fermentation of tempeh, nonetheless, some studies found significant rises of this vitamin in tempeh (Robinson 1977; Murata et al. 1967).

Pyridoxine (Vitamin B6) is a form of B vitamin, and it can be originating in certain foodstuffs for instance vegetables, cereals, legumes, meat, and eggs (Brown and Beier 2017). Murata et al. (1967) detected rises in Pyridoxine contents of 4.4- and 14-fold in two batches of soybean tempeh. In another study, rises in pyridoxine content were also observed in miso and tempeh prepared from soybeans, chickpeas, and horsepeas (Robinson 1977). Ekıncı (2005) surveyed the impact of fermentation on the water-soluble vitamin content of tarhana, a traditional Turkish cereal food. The results of this study showed no significant changes in pyridoxine content. Also, Alm (1982b) observed insignificant alters in the amount of pyridoxine in the fermented milk. In another study, no change was observed in the pyridoxine content of the cottage cheese (Reif et al. 1976).

Biotin (Vitamin B7) is another water-soluble B vitamin. It is involved in a wide variety of metabolic procedures, mainly associated with the use of carbohydrates, amino acids, and fats. It's also recognized as vitamin H (Zempleni et al. 2009). The changes in the amount this vitamin during fermentation in different products and conditions are varying and accompanied by a decrease or increase. In Alm (1982b) study, changes in the amount of Biotin were detected in fermented milk, and the biotin content reduced by less than 20%. In another study, throughout Cheddar cheese maturing, the amount of Biotin enhanced through the initial 2 months by 60%, nevertheless then reduced such that later 6 months the amount was less than the primary amount (Nilson et al. 1965).

Folic Acid (Vitamin B9): Folate also recognized as folacin, also belongs to B vitamins. Manufactured folic acid, which is changed into folate via the body, is employed as a dietary supplement and in food enrichment as it is more unchangeable throughout processing and storage (Donnelly 2001). Ekıncı (2005) studied the impacts of fermentation (30 °C for 4 days) on the content of folic acid in tarhana, a traditional Turkish food. The fermentation resulted in a considerable increase in folic acid content of the samples. It has been reported a 59% rise of folate in fermented steamed idli in comparison with the nonfermented starting material (McFeeters 1988). Some studies have reported a three to ten-fold increase in folic acid during cottage cheese fermentation. However, these studies showed that after the second month of cheese ripening, the amount of folic acid decreases to its original amount (Homayouni et al. 2018; Nilson et al. 1965). It seems that folic acid can be produced via some microorganisms/starters employed in the fermentation of various dairy products.

Cobalamin (Vitamin B12) is one of the water-soluble B vitamins involved in the metabolism of all human cells (Wolffenbuttel et al. 2019). Bao et al. (2019) studied the impact of *L. reuteri* on Cobalamin content in furu (fermented bean curd) fermentation. Results of this study disclosed that the amount of cobalamin in furu inoculated with *L. reuteri* was slowly enhanced up to 141.7 ng/g (wet weight), which was higher than the control group (36.0 ng/g). In another study, Van Veen and Steinkraus (1970) showed a surge of over 30-fold from 0.15 to 5 μg/kg in the Cobalamin content of tempeh in comparison with the starting soybeans. Some studies determined that Cobalamin was created via contaminating bacteria typically existing in the traditional process (Liem et al. 1977). It has been shown that the

fermentation does not at all times result in enhanced vitamin contents since microorganisms also need these nutrients for their growth. Several microorganisms are recognized for creating vitamins, for example, Propionibacteria for producing Cobalamin (Walther and Schmid 2017). Ro et al. (1979) enhanced the amount of Cobalamin (to 102 ng/100 g from 47 ng/100 g in the control at 1 Week of fermentation at 4 °C) in kimchi via adding *Propionibacterium freudenreichii* subsp. shermanii to the natural fermentation. Also, beef extract (0.05%)/soy flour (0.5%) added (as protein sources) to the inoculated kimchi, more enhanced the vitamin activity to 203 and 197 ng/100 g, respectively, at 1 Week (see Fig. 9.6). Conversely, Alm (1982b) showed the reduction of up to 50% in yogurt and other fermented dairy foods inoculated with lactic acid bacteria. Generally, it has been disclosed that the fermentation parameters, such as time and temperature of incubation, and kind of growth culture are other factors affecting the Cobalamin content of a fermented product (Walther and Schmid 2017).

Ascorbic acid (Ascorbate/Vitamin C) is a water-soluble vitamin found in many foods and is also consumed as a dietary supplement. Ascorbic acid is an important nutrient involved in the overhaul of tissue and the enzymatic creation of specific neurotransmitters. It is employed to avert and cure scurvy (Arrigoni and De Tullio 2002). This vitamin is stabilized via acid circumstances and the exclusion of O_2 (Aguilar et al. 2017; Huelin et al. 1971). Different variations of ascorbic acid have been reported in various fermented products depending on the type of product and the fermentation conditions. A variety of 1–35 mg ascorbic acid/100 g of sauerkraut was detected in canned sauerkraut (Pederson et al. 1956). Martinez-Villaluenga et al. (2009) studied the effect of fermentation conditions on the amount of ascorbic acid in white cabbage (*Brassica oleracea var. capitata* cv. Taler) cultivated in diverse seasons. The amount of ascorbic acid detected was higher in products cultivated in the summer season and the fermentation process led to considerable decreases. Significant increases in the amount of ascorbic acid were detected in both miso and tempeh prepared from soybeans, chickpeas, and horsebeans (Robinson 1977).

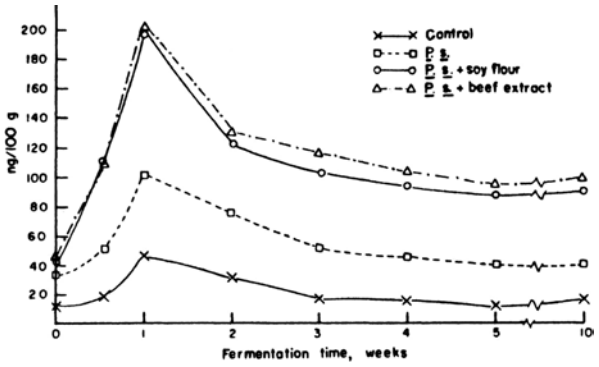

Fig. 9.6 The amount of Cobalamin in control and *Propionibacterium freudenreichii* Shermanii inoculated kimchi, with or without beef extract/soy flour (fermented at 4 °C). (Source: Ro et al. (1979))

9.3.3.2 Germination

In cereals and legumes, during the germination process, the amount of some vitamins, including B vitamins such as riboflavin, niacin, pyridoxine, as well as vitamins such as vitamins E and C, increase due to their synthesis by fresh sprouts (Kim et al. 2012; Žilić et al. 2015; Liu et al. 2017; Siles et al. 2018). Nevertheless, studies have shown that some water-soluble vitamins, such as thiamine, decrease during germination (Moongngarm and Saetung 2010). Various studies have shown that the amount of ascorbic acid in some cereals and legumes, including mung beans, wheat, and chickpea increases significantly during the germination process. This increase is due to enzymatic hydrolysis of starch by diastases and amylases and the production of large amounts of glucose, which is used in the biosynthesis of this vitamin (Laxmi et al. 2015; Desai et al. 2010; Lu and Guo 2020). Gan et al. (2016) exhibited that the amount of ascorbic acid of the green and black mung bean sprouts enhanced from 13.5 to 24.0 and 10.3 to 21.3 folds in comparison with their own raw seeds later germination process for 1–5 days.

In another study, Walker et al. (2002) detected a reduction in the amount of folate in kilned malt compared to green malt, demonstrating temperature sensitivity of the folates. They showed that the germination of cereals and legumes can be an easy process for enhancing the uptake of folates. Shohag et al. (2012) showed a considerable rise in folate content of mung bean and soybean sprouts in comparison with raw seeds by 78%–326% and 65%–274%, respectively, after germination.

It has been shown that vitamins B2, B5, and B6 rise throughout the germination of barley, whereas vitamins B1 and B3 change very slightly (Hübner and Arendt 2013). Carotenoids can be metabolized to vitamin A or retinol. The source of vitamin A is mainly from animal foods, however, about a quarter of the daily requirement can be provided from plant sources (Lindgren et al. 2003; Matusova et al. 2005). Cereals, especially corn, are among the major plant sources of vitamin A (Fardet et al. 2008). However, the germination process can also cause a significant increase in this vitamin in cereal grains, especially in grains that have low levels of carotenoids (precursors of vitamin A) (Matusova et al. 2005; Luo et al. 2020).

9.3.4 Lipids

9.3.4.1 Fermentation

According to the results of different studies, fermentation process can affect the lipid composition of products. De Reu et al. (1994) studied the changes in lipids of soya beans throughout the fermentation of Tempe. In this study, soya beans were fermented via pure cultures of *Rhizopus oryzae* and *Rhizopus oligosporus*. With *R. oligosporus*, the content of glycerides reduced from 22.3 to 11.5% (w/w, dry matter) after 69 h fermentation at 37 °C. In the last product, only 4.3% (w/w, dry matter) of free fatty acids were detected, henceforth the variance of 6.5% (w/w, dry

matter) of fatty acids was lost. Lordan et al. (2019) investigated the impact of the fermentation process on the antithrombotic characteristics of polar lipids in ovine milk over the making of yogurts. The results showed that the amount of Monounsaturated fatty acids (MUFAs) enhanced in the fatty acids of the polar lipids, nevertheless, there was a decrease in Polyunsaturated fatty acids (PUFAs) as milk was fermented to yogurt. All yogurt polar lipids displayed strong antithrombotic impacts with IC_{50} values ranging from 45 to 77 μg. In a study, Li et al. (2019) showed that a co-fermentation of *Rhodococcus* strains with considerably enhanced lignin degradation and/or lipid biosynthesis capacities was recognized, which allowed concurrent change of lignin, glucose, and its derivatives into lipids. Tesnière (2019) reviewed the importance and role of lipids in wine fermentation. This study showed that lipids not only act as a major food source for yeasts and play a vital role in stress management but also play a key role in the production of volatile yeast metabolites. Therefore, these compounds due to the breakdown of lipids during fermentation play an important role in the constancy of fermentation and the development of desirable organoleptic properties in the final product.

9.3.4.2 Germination

Numerous studies have been performed on changes in lipids during germination process in cereals and legumes. Traoré et al. (2004) showed that the lipid content of cereals somewhat enhances throughout the steeping phase of malting nevertheless later drops throughout the germination stage by the way of lipids are employed for respiration procedure. Poxleitner et al. (2006) showed that throughout seed germination, caleosin has an important role in the degradation of stored lipid in oil bodies. The outcomes showed that communication of oil bodies with vacuoles is one mechanism that helps degradation of storage lipid. In different studies, the lipid changes during germination are contradictory. In a study, Kim et al. (2012) detected a rise in oleic acid, linoleic acid, and crude lipids in germinated rice, even though some studies did not find deviations in the amount of lipids during germination of rice grains (Moongngarm and Saetung 2010). Also, some investigations showed that germination decreases the amount of lipids due to hydrolysis and consumption of lipids as a sources of dynamism for biochemical reactions throughout the germination process (Jan et al. 2016; Moongngarm and Saetung 2010; Chinma et al. 2009).

9.3.5 Minerals

9.3.5.1 Fermentation

The minerals in the structure of plants are bound to complex and indigestible compounds such as phytate and cell wall polysaccharides, so they have low bioavailability and are not absorbed by the human body. Fermentation process is one of the

most important processes that release minerals from complex structures and increases their availability and absorption (Nkhata et al. 2018; Pranoto et al. 2013). Some studies showed that fermentation process enhanced the amount of calcium, magnesium, zinc, and iron in some fermented products which were related to the reduction in phytates content (Nkhata et al. 2018; Pranoto et al. 2013). Nevertheless, the rise in the amount of minerals can be due to the loss of dry matter throughout the fermentation process as microorganisms break down protein and carbohydrates (Nkhata et al. 2018; Day and Morawicki 2016). Various mechanisms are involved in increasing the bioavailability of minerals during the fermentation process (Fig. 9.7).

According to Fig. 9.7, In the first place, fermentation process reduces the amount of phytic acid that is attached to the minerals, consequently, large amounts of minerals are released and become available (Nkhata et al. 2018; Lopez et al. 1983). Nevertheless, this effect is offset by the release of tannins throughout the fermentation process, particularly in some grains that are high in tannins such as sorghum (Osman 2011). In fact, tannins attach minerals and decrease their availability depending on the period of the fermentation process. It has been shown that the extended fermentation diminish the tannin content due to the action of bacterial/yeast phenyl oxidase (Nkhata et al. 2018; Emmambux and Taylor 2003). However, conversion of tannins to phenols happening throughout fermentation increases the amount of phenol that interacts with minerals resulting in prevention of mineral availability (Nkhata et al. 2018).

In the second place, the fermentation process releases the complex matrix that embeds minerals. In other words, it makes the matrix loose via degrading starch and phytate respectively in cooperation with α-amylase and phytase. Therefore, the impact of fermentation is related to food composition and other food ingredients for instance dietary fibers may delay the availability of minerals (Nkhata et al. 2018).

In the third place, decrease in pH throughout the fermentation process enhances iron absorption due to alteration from ferrous iron (low absorbable) to ferric iron, which is easily absorbed. Furthermore, fermentation process offers high pH for the enzymatic abolition of phytate. Also, when the fermentation process is performed by grinding, the availability of minerals increases, which is due to the fact that grinding increases the surface area of the grain and breaks down the cellular

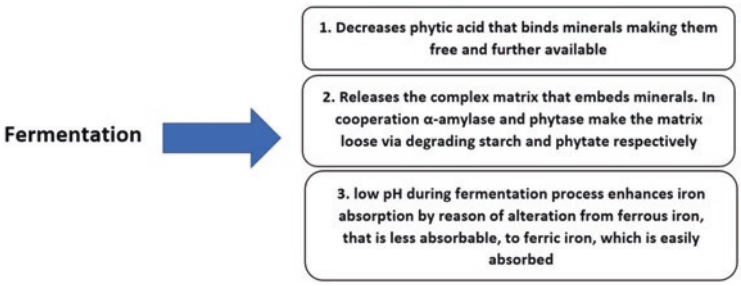

Fig. 9.7 Various mechanisms involved in enhancing the bioavailability of minerals throughout the fermentation

structure which leads to releasing the enzyme phytase, which affects phytate and breaks it down (Nkhata et al. 2018; Reale et al. 2007).

9.3.5.2 Germination

One of the most important anti-nutritional compounds in cereals is phytic acid, which plays a key role in binding minerals and making them inaccessible, and this is a major nutritional defect for cereals (Liang et al. 2008). Germination, similar to fermentation, plays an important role in the release of minerals from phytic acid. With increasing germination time, the amount of phytic acid decreases due to the activation of the enzyme phytase, which breaks down phytic acid into myoinositol and phosphoric acid, eventually, minerals are released from the structure (Liang et al. 2008; Kumar and Anand 2021). The variance in mineral bioavailability from diverse legumes and cereals after the germination process for the analogous era can be associated with differences in phytase activation, the amount of phytate, amount of binding of minerals inside the milieu, or collaboration of these items. Some studies have shown that the germination of various seeds such as foxtail, sorghum, and chickpea considerably enhance the amount of calcium, sodium, magnesium, potassium, and phosphorus, however, diminishes the amount of iron and calcium (Laxmi et al. 2015; Ogbonna et al. 2012; Desai et al. 2010). Legumes are rich in factors such as phytic acid, tannins, protease inhibitors, α-amylase, lecithin, and polyphenolic compounds, all of which cause poor absorption and digestion of minerals and other micronutrients (Yasmin et al. 2008). Legumes also contain a large amount of the enzyme phytase, which is activated during germination and breaks down phytate, releasing minerals and making them available (Nkhata et al. 2018; Sandberg 2002; Zhang et al. 2020).

9.3.6 Phytochemicals

9.3.6.1 Fermentation

Phytochemicals are significant botanical minor metabolic ingredients formed in phenylpropanoid synthesis and shikimate paths throughout the growth of plants. During plant growth, L-phenylalanine, influenced by the catalyzation of phenylalanine ammonia-lyase/PAL and alters into cinnamic acid. At that time, various phenolic elements, for instance caffeic acid, and ferulic are produced. They can be converted into flavonoids, tannins, lignins, and other ingredients (Nkhata et al. 2018). Various studies have shown that the key role of these phytonutrients in human health is via their antioxidant characteristics, decrease in the creation of pro-inflammatory cytokines and immunosuppressive cells, and reduction in cholesterol (Nkhata et al. 2018; Zhang et al. 2015b; Lesinski et al. 2015). Studies have shown that the fermentation process has an important effect on phytochemicals, in some

cases these effects are positive and sometimes they are negative. For example, fermentation has been shown to significantly reduce carotenoids in corn (Li et al. 2007; Ortiz et al. 2017) and also to increase beta-carotene absorption (Phorbee et al. 2013). The release and increase of some phytochemicals during the fermentation process can cause these compounds to react with carbohydrates, proteins, and minerals and make them inaccessible (Nkhata et al. 2018; Duodu et al. 2003). In some cases, microbial fermentation, such as lactic acid bacteria fermentation, can lead to the consumption of phytochemicals and eventually reduce them (Hubert et al. 2008). Hubert et al. (2008) observed a reduction in tocopherols, phytosterols, and glycosylated soyasaponins once soybean was fermented by lactic acid bacteria. It has been shown that fermentation can decrease isoflavones considerably as a result of hydrolysis of glucosides into aglycone (Manach et al. 2004). Kuo et al. (2006), and Dueñas et al. (2005) showed the role of *B. subtilis* and *L. plantarum* to have β-glucosidase that can break glycosidic bonds between sugars and phytochemicals leading to the release of phytochemicals.

9.3.6.2 Germination

Studies have shown that germination process can increase compounds such as flavonoids, phenolics and tannins (Zhang et al. 2015b; Zhou et al. 2019; Zilic et al. 2015). It has also been shown that increased phytochemicals during the germination of cereals and legumes increases their antioxidant capacity (Nkhata et al. 2018; Ge et al. 2021). Phytochemicals can undesirably impact the bioavailability of various nutrients, as well. Furthermore, extended drenching and fermentation can aid decrease the amount of these phytochemicals over leaching (Ogbonna et al. 2012). The activity of microorganisms throughout fermentation decreases the amount of tannin as does the activity of the acyl hydrolase (Ojha et al. 2018). As a result of the decrease in the amount of phytic acid and tannin in germinated cereals and legumes, minerals become further available, thus enhancing the nutritional quality of the foodstuff (Nkhata et al. 2018; Ojha et al. 2018).

9.4 Glycemic Index

Some studies have shown that the fermentation process increases the glycemic index (GI) (Ihediohanma 2011; Ihekoronye and Ngoddy 1985), whereas some studies indicate a reduction (Mlotha et al. 2016; Demirkesen-Bicak et al. 2021). The low GI of fermented foods is related to the production of short-chain organic acids such as acetic acid, propionic acid, and lactic acid during the fermentation period (Östman et al. 2005). Various studies have shown that the consumption of lactic acid-rich fermented foods reduces postprandial blood glucose due to reduced starch hydrolysis in the early part of the small intestine, and it is thought that the presence of lactic acid may decrease the activity of starch hydrolyzing enzymes significantly (Östman

et al. 2005). Acetic acid and propionic acid also reduce the activity of gastric enzymes and reduce the rate of gastric emptying (Darwiche et al. 2001). Some studies have shown that the rise of GI is possibly due to the ease of ingestion and absorption of glucose as a result of degradation of fibers in cereals and legumes via microorganisms throughout the fermentation period (Nkhata et al. 2018). It has been reported that maltose is produced throughout the fermentation of starch which is further transformed to D-glucose once hydrolyzed in an aqueous solution in the order starch–dextrin–maltose–glucose (Nkhata et al. 2018). Extension of fermentation time can release further glucose and then surge postprandial glycemic retort. However, the impact of fermentation is mostly related to organic acids formed, starch degradation, and distraction of fiber. (See Fig. 9.8).

Generally, sprouted and fermented foodstuffs have revealed hypoglycemic impacts on the body, via dropping content of soluble carbohydrates, an enhancing amount of dietetic fiber, and resistant starch. de Oliveira Lopes et al. (2019) studied the impacts of sprouted and fermented quinoa on GI of diet and chemical factors of the blood of rats fed a high carbohydrate diet. The results of this study showed that the sprouting and fermentation of quinoa can decrease the GI of foods with simple carbohydrates. Furthermore, food ingestion, blood lipid and glucose ranges, and gathering of epididymal adipose tissue were diminished in rats fed with quinoa. These consequences can be due to the nutritional composition of the complemented diets, in addition to the chemical alterations promoted via processing quinoa. Świeca et al. (2013) surveyed digestibility, starch content, glycemic index and

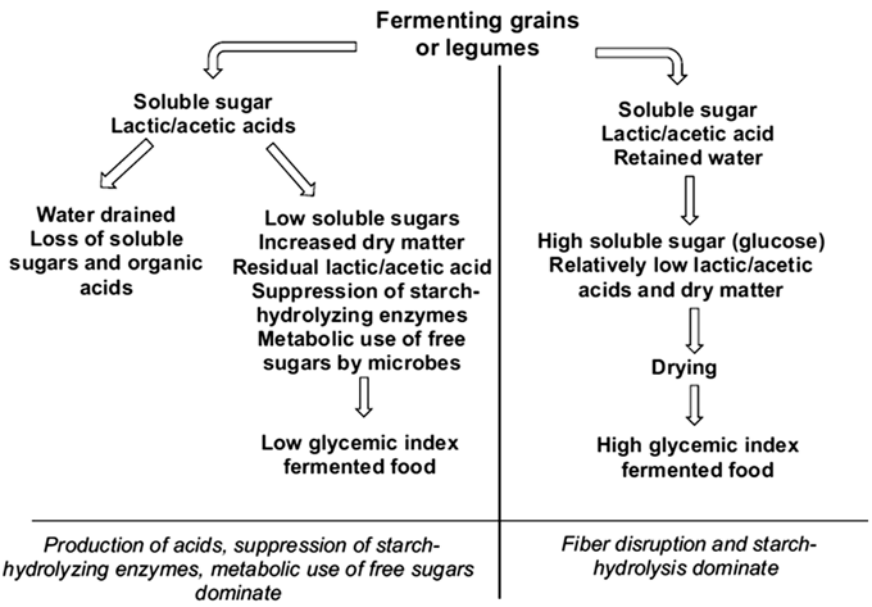

Fig. 9.8 Diagram showing how fermentation results in diverse glycemic index of fermented food products (Source: Nkhata et al. (2018))

antidiabetic impact of lentil sprouts attained via diverse germination methods. They indicated that germination raised starch bioavailability and decreased the activity of α-amylase inhibitors. In fact, the activity of α-amylase inhibitors and the amount of resistant starch affected starch bioavailability. Elicitation permitted varying starch content and GI values during the germination period.

9.5 Antioxidant Activity

Oxidation is a vital mechanism for living organisms which in turn leads to the production of free radicals and other reactive oxygen species. The compounds have lethal cellular effects by interfering with the normal cellular functions and signaling pathways, and destructing membrane lipids, cellular proteins, DNA, and enzymes. The protective mechanism against free radicals involves enzymes such as superoxide dismutase, catalase, and peroxidase. Oxidation is also an undesired mechanism in food ingredients and can cause chemical spoilage, off-flavors and negative effects on nutritional and organoleptic characteristics of the foods accompanied by increased safety concerns (Antolovich et al. 2002). Accordingly compounds with antioxidant effects are gaining special attention owing to their health benefits and their impact on extending the shelf life of food products. Fermentation and germination can both improve the phenolic content and antioxidant activity of the final product.

9.5.1 Fermentation

Oluwafemi et al. have scrutinized the effects of fermentation on the antioxidant activity of the whole grain cereals in a recent review (Adebo and Gabriela Medina-Meza 2020). The main effect of fermentation on antioxidant activity of the product has been attributed to the release of bound phenolic compounds (PCs) through activities of hydrolytic enzymes and modulating contents of cereals during fermentation; the increased extractability of PCs with antioxidant activities facilitated cleavage of the bonds between PCs and other elements, products of protein hydrolysis through proteolytic actions through fermentation which leads to the compounds with positive interactions with PCs anti-oxidant activity (Adebo and Gabriela Medina-Meza 2020).

Potential mechanisms of antioxidant activities of phenolic compounds involve detoxification via phase II conjugation reactions, modifying some cellular signaling processes, donating an electron/transfer hydrogen atom to free radicals, and activating endogenous antioxidant mechanisms. Even though the contents of phenolic compounds of grains are usually increased after fermentation, there are some reports on the decreases during the process. For instance, it has been shown that the total phenolic content (TPC) of fermented Working Group (WG)-sorghum is lower than

that of the whole grain. It should also be noticed that some microbial strains act on the PCs and may metabolize or degrade the PCs while others do not (Adebo and Gabriela Medina-Meza 2020; Hur et al. 2014).

Other products of fermentation with antioxidant activity may similarly decrease after fermentation due to modifications that influence the extractability of compounds such as tannins, phenols, and proteins. It is also worth mentioning that increasing PCs after fermentation does not always equivalent to an increase in antioxidant activity of the product (Adebo and Gabriela Medina-Meza 2020). The total phenolic content does not include other antioxidant compounds. On the other hand, there are interactions among antioxidants which is difficult to measure. *In vivo* studies on the fermented cereals suggest improved antioxidant activity by protecting livers of rats from oxidative stress and lowering oxidative states of rats fed with fermented breads. In conclusion, fermentation is accompanied by increased antioxidants such as PCs, peptides, and oligosaccharides, and positively influenced bioavailability, and bio-accessibility of the anti-oxidants (Adebo and Gabriela Medina-Meza 2020; Juan and Chou 2010; Liu et al. 2020).

9.5.2 Germination

Several components with antioxidant activity such as phenolic compounds, sterols, vitamins, and phytic acid can be enriched both in the bran or the germ. Germination has different effects on each of them. Furthermore, some minerals and trace elements that act as co-factors to some enzymes indirectly affect the antioxidant mechanisms (Sandoval-Sicairos et al. 2020).

The amounts of phenolic compounds may increase or decrease during the process of malting. There are also some reports that in the earlier stages of malting the content of phenolic acids decreases and then increases during the kilning process. Yang and Ooraikul (2001) demonstrated that ferulic and vanillic acids moderately decrease or remain constant in the early stages of germination and then in the later stages they significantly increase. The antioxidant activity of the malted cereals alters accordingly. However, as we explained in the previous section, presence of PCs does not necessarily reflect the antioxidant activity of the whole grain. A decrease in phenolic compounds accompanied by an increase in antioxidant activity has also been detected (Yang and Ooraikul 2001). It was explained with the formation of Millard products and degradation of phenolic acids (Hübner and Arendt 2013; Yang and Ooraikul 2001).

Different factors influence the phenolic content of the produced germ. The variety used and harvest years are of great importance. In some varieties phenolic compounds decrease after germination, however, tannins decrease in most varieties. The leaching of water-soluble compounds during steeping, formation of insoluble complexes of phenolic compounds and proteins, and breakdown of tannins have been proposed as probable explanations. Tannins may form insoluble complexes with proteins and complex minerals, so they are not desirable compounds in nutrition. It

is a positive aspect of germination that they are broken down during the process and the products also have antioxidant properties (Hübner and Arendt 2013).

Sterols generally increase after germination. It has been demonstrated that sterol content after germination is 20% higher in oat and 20-fold greater in maize. Even though in germinating rye only small changes were detected (Hübner and Arendt 2013). Vitamins E and C are part of the natural defense mechanism against free radicals and both of them increase after germination, as a result, the nutritional and antioxidant properties are improved in malted grains (Hübner and Arendt 2013). Further information on this subject has been provided in Sect. 9.3.3.

Phytic acid is a natural antioxidant constituting 1–5% of most cereals. It is typically known for its antinutritive effect which inhibits the availability of divalent cations such as Fe^{2+}, Ca^{2+}, Mg^{2+}, and Zn^{+}. Phytic acids are not very soluble and are of very limited digestibility. The amount of phytic acid is generally reduced after germination (Singh et al. 2015). Nonetheless, this improves the bioavailability of essential minerals, and the total antioxidant capacity of the grain decreases.

9.6 Microbiological Properties

9.6.1 Fermentation

The microbial composition of the bran including molds, yeasts, coliforms, and other bacteria alters during the process of fermentation. Generally, these changes favor the growth of beneficial microorganisms and reduce the count of pathogenic ones. It would be of great importance for food preservation. It has been shown that as fermentation time increases mold count and coliform count decrease. The yeast and lactobacillus count increase after fermentation which have health benefits for the consumers (Buta and Emire 2015). According to the result of Buta and Emire (2015) on the quality protein maize (QPM) and soybean blends, mold count decreased from 2.1×10^4 to 4×10^2 and the coliform count decreased from 4.3×10^2 to 3.1×10^2 after 24 h natural fermentation. Meanwhile, the yeast count increased from Nill to 2.9×10^2. In a similar study, the population of LAB raised markedly during fermentation. High temperature and long fermentation time favored the growth of bacteria. High temperature also increased the diversity of LAB, as at the highest temperature (35 °C) eight species or genotypes were identified (Katina et al. 2007). The growth of LAB and augmented production of organic acids and other antimicrobial substances inhibited the growth of aerobic heterophilic bacteria and the viable count decreased after fermentation (Katina et al. 2007).

9.6.2 Germination

The process of germination includes three main steps: steeping, germination, and kilning. Each of the steps has its special impact on the microbial population. Regarding the influence of microbial activity on the quality of the final product, it is very important to control microbial activity during germination. Generally, the growth of microorganisms deteriorates the quality of malt. They can compete for oxygen with embryos and produce some signaling hormones such as indole-3-acetate which may have different effects on the growth of the grains. After kilning, malt is hygroscopic and rich in soluble nutrients which is a favorite environment for the microorganisms and may cause microbial spoilage (Bokulich and Bamforth 2013). Growth of molds such as *Fusarium* head blight and production of mycotoxins could also be hazardous for the consumers (Jin et al. 2021).

At the steeping stage, availability of nutrients, moisture, warmth, and aeration lead to increased microbial count. Following cycles of steeping and aeration also promote bacterial growth, however, kilning reduces viable microbe count. After this stage, it is crucial to maintain low-moisture conditions to avoid microbial spoilage (Bokulich and Bamforth 2013).

Some suggested actions to control microbial count during germination include: changing the steep fluid between air rests, decreasing dissolved nutrients and restoration of suspended biomass, controlling the steep temperature, and microbial inoculation of steep liquor to control the growth of damaging microbiota during germination. For instance, it has been shown that *Geotrichum candidum* and *Lactobacillus plantarum* are able to diminish the *Fusarium* growth on malt (Bokulich and Bamforth 2013).

9.7 Functional Properties

The term functional food originated from Japan in the early 1990s and refers to foods with physiologically active ingredients and health-promoting properties. Functional foods have health benefits beyond basic nutrition and their consumption as part of a diet can increase the health status of the consumers by improving immunological defense, physical, mental, and general health conditions, preventing diseases, and slowing the aging process. The presence of probiotics, prebiotics, and bioactive compounds, antioxidant activity, and reduced anti-nutritional compounds are characteristics of a cereal-based functional food (Schwan and Ramos 2019; Xiong et al. 2020).

9.7.1 Fermentation

Cereals and milk have great potential as substrates for functional foods. During the fermentation process, using an adequate starter culture and preparing a suitable environment for the growth of beneficial bacteria and producing materials with functional properties is very important. Further research is still needed on developing fermented products with desirable organoleptic characteristics (Schwan and Ramos 2019).

9.7.2 Germination

Malting is a process that has the potential to improve the nutritional and functional properties of grains. As discussed in this section, malting can reduce some anti-nutrients such as phytic acid, and it is accompanied by accelerating the production of vitamins, antioxidants, and other beneficial nutrients. Malting is a low-cost process without any sophisticated and expensive equipment which makes it an attractive choice for the production of functional foods (Singh and Sharma 2017). However, similar to the fermentation process, more research is needed to find the best approaches of malting to produce functional foods with acceptable organoleptic characteristics.

9.8 Conclusion

The results of current research and available evidence show that fermented and germinated foods are rich in many micronutrients compared to conventional foods. This is due to the fact that during the fermentation and germination process, on the one hand, the activation of degrading enzymes of anti-nutritional factors occurs, and on the other hand, the production and release of useful micronutrients from complex structures occurs, and then bioavailability, digestibility, and absorption rate of nutrients are increased. Also, following the release and increase of many nutritional factors such as vitamin C, vitamin E, B vitamins, and minerals, health beneficial effects, including antioxidant properties increase. Therefore, fermented and germinated foods are recommended to promote health and prevent many diseases, especially in developing countries. On the other hand, during the fermentation and germination processes, major complex nutrients such as proteins, fats, and especially carbohydrates in the raw food are broken down and simpler and more accessible compounds are formed. For example, in the case of carbohydrates, after the breakdown of compounds such as starch, the amount of glucose increases, which also acts as an energy source and with the continuation of the fermentation process, produces abundant organic acids and reduces the glycemic index, and these foods

are recommended for diabetics. In this regard and proper guidance of fermentation and germination processes, several factors are involved, including time, temperature, pH, etc., which can be achieved with accurate and purposeful control. It can be concluded that fermented and germinated foods, especially when produced under controlled conditions, can be categorized in the functional food class and contain health-promoting micronutrients.

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Chapter 10
Influence of the Supercritical Fluid Extraction (SFE) on Food Bioactives

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Abbreviations

d	Vessel diameter
DOE	Design of experiment
F	Feed mass
FA	Fatty acid
H	Vessel height
LCA	Life cycle assessment
Q_{CO2}	CO_2 volumetric flow
S	Solvent mass
$scCO_2$	Supercritical CO_2
SCF	Supercritical fluid
SFE	Supercritical fluid extraction
T_{RES}	Residence time

10.1 Supercritical Extraction of Food Bioactives

Bioactives are metabolites synthesized by plants, where one of their main objectives is to contribute to the self-defense mechanism. Examples of food bioactives are pigments, carotenoids, omega -3 fatty acids, polyphenols, terpenes, lipids, vitamins, peptides, proteoglycans, and polysaccharides (Puri 2017). These specialty chemicals are typically found in low concentrations compared to macronutrients.

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Therefore, they can be used as nutraceuticals in foods to improve the nutritional value or treat diseases.

The introduction of new non-conventional methods for the extraction of food bioactives has been a topic of interest for the last decades; this is necessary for improving traditional extraction. Conventional extraction shows excessive use of solvents, usually compromising yield, separation, toxicity, and environmental effects. As a result, new emerging technologies aim to replace conventional extraction in the coming years. The latter is a great challenge since yield and selectivity play a key role in selecting the most appropriate extraction method. In addition, aspects such as scale-up and cost are crucial when moving into an industrial scale. Therefore, these considerations are essential when selecting an extraction method in the laboratory during the early stages of process development.

For several years, supercritical fluid extraction (SFE) has been employed to extract food bioactives. As a result, it is one of the most common processes employing supercritical fluids (SCF). Recently, there has been an interest in new technologies employing SCF, not only extraction (Asiri 2020). However, this chapter will focus only on SFE and its most significant control variables to successfully remove food bioactives.

10.1.1 Effect of Supercritical Fluids Properties in the Extraction of Bioactives

A SCF is a fluid that has reached a thermodynamic state above its critical temperature, pressure, and density (Smith et al. 2013b). A characteristic of this state is that the fluid is highly compressible, gaining liquid and gas characteristics. Therefore, the first approach to understanding the SCF concept is analyzing a fluid, its phase behavior, and its thermodynamic transitions caused by temperature and pressure. Carbon dioxide (CO_2) is a typical fluid employed in SFE. Its critical temperature and pressure (31 °C and 7.38 MPa) and its transitions through different phases towards the supercritical state are good examples. Figure 10.1 highlights some thermodynamic changes of CO_2 when it reaches a supercritical state.

When CO_2 is under the supercritical temperature and pressure, it is possible to find it as gas, liquid, solid, or a combination of these phases. One example is the vapor-liquid equilibrium in Fig. 10.1a. As shown in Fig. 10.1a, the vapor-liquid state characterizes by a boundary (green semicontinuous line) between the two phases. Suppose temperature and pressure continue to rise. In that case, a subcritical (near critical) state is reached (Fig. 10.1b), where the boundary between the vapor and the liquid phase is more difficult to detect (thin green semicontinuous line). Finally, a critical state is reached if we continue applying pressure and temperature (Fig. 10.1c). In that case, the layer boundary disappears, giving rise to a unique thermodynamic state that is not considered either liquid or gas. Instead, a new homogeneous supercritical phase takes place. More interestingly, being a

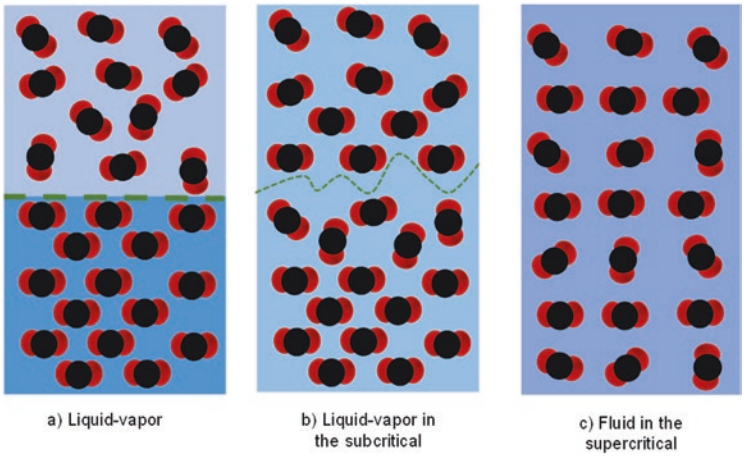

Fig. 10.1 Different CO_2 phases transitions

supercritical homogeneous phase means that it is not liquid or gas, so properties obtained are a combination of both.

In the case of the retention of food bioactives, this combination of properties is beneficial for extraction. A more efficient mass and heat transfer are possible during extraction (Smith et al. 2013b). SCFs show higher diffusion coefficients, higher compressibility factors, low viscosity, low dielectric constant, and low solubility parameters compared to liquids. While making the analogy to gases, SCFs offer high thermal conductivity and high heat capacity. SCFs show a viscosity similar to gases, densities resembling liquids, and diffusivities ranging from those to liquid and gas (Vardanega et al. 2019).

10.1.2 SFE Differences from Other Extraction Techniques

Compared to other conventional and non-conventional extraction techniques, SFE shows exciting features. Some of these features are well reported in the literature (Smith et al. 2013b; Vardanega et al. 2019): high yield and selectivity, production of few or no waste, non or few solvents employed, extraction of bioactives with higher shelf life, no solvent residues in products or by-products, employment of environmental solvents such as $scCO_2$. Moreover, SFE aids in preserving food matrices and their bioactives by the inactivation of microbial and enzymatic activity. As a result, better preservation of sensory characteristics is possible. SFE allows retaining various value-added bioactives from the same matrix through the biorefinery concept, opening opportunities for a more sustainable and economically feasible extraction process (Vardanega et al. 2019).

A unique characteristic of SCFs from other extraction methods is that by adjusting variables such as pressure, temperature, and co-solvent, it is possible to fine-tune the system solubility, making SFE a high-performance solvent (Smith et al. 2013a). In most extraction technologies (conventional or non-conventional), solubility is a direct consequence of the solvent molecular composition. Therefore, solubility improvements include optimizing solvent selection (like dissolve likes), increasing temperature, or developing a solvent mixture. On the contrary, the solubility of a SCF results from its critical pressure, temperature, density, or co-solvent composition. Hence, the subcritical and supercritical states allow exploring different solubilities with the same solvent. As a result, it is possible to attain the optimum solubility for a specific bioactive. Simultaneously, the high pressures provide pretreatment for enhancing solvent diffusion into the matrix. Hence, SCF changes in density produces considerable effects in parameters such as dielectric constant, solubility, and partition coefficient.

Optimizing the solubility in SFE for different food bioactives relies greatly on exploring the effect of pressure and temperature. All this while selecting an appropriate experimental design (Sharif et al. 2014). These two variables will directly affect the affinity of the bioactives for the SCF. A co-solvent, used in small proportions, is usually employed when extracting bioactives with polar behavior and long molecular structures. Hence, through experimentation, SFE can define the most appropriate conditions for removing the food bioactive and exploring different extraction pathways according to the purity requirements. SFE can be employed not only for extracting value-added bioactives from different matrices. One example is when the matrix is a high-value product, and an undesired compound must be removed. The decaffeination of ground coffee is an example where SFE removes caffeine (undesired) from the matrix (ground coffee). As a result, SFE can increase the matrix value or remove added-value bioactives. Another aspect to highlight during SFE is the separation of the bioactives from the SCF. Separation commonly occurs when there is a transition from the supercritical phase to the gas phase. During this separation, there is an abrupt change in the thermodynamic state. Consequently, the extract obtained has a typically high degree of purity with no need for intensive downstream separation, making the process attractive from an economic perspective.

10.1.3 Process Steps and Control in the Removal of Food Bioactives

Developing an efficient SFE of food bioactives demands a robust knowledge of the different steps and equipment involved in the process. Moreover, it is paramount to understand how variables such as temperature and pressure influence the SCF and co-solvent thermodynamic state, affecting the system's solubility. Other essential variables during SFE are SCF flux, dynamic and static extraction time. A typical

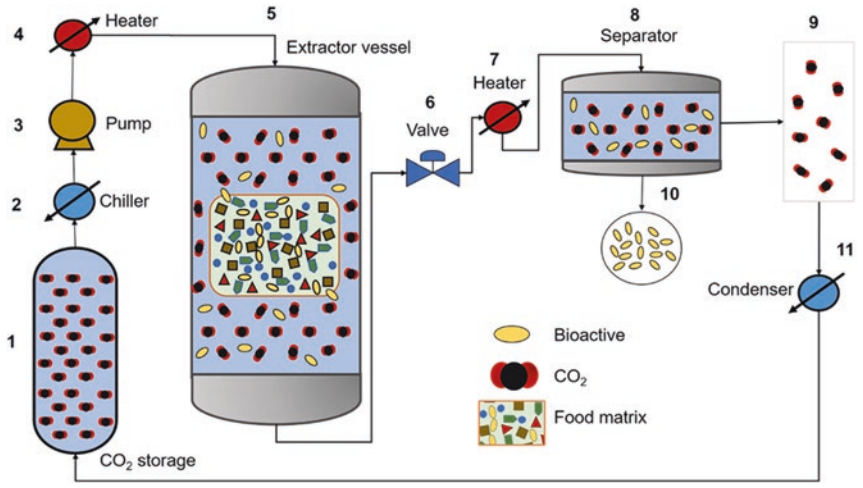

Fig. 10.2 Representation of the SFE process

SFE process consists of different components: solvent storage and supply, extraction chamber, separation, and solvent recycling (Smith et al. 2013b). Figure 10.2 displays a representation of the SFE system employing $scCO_2$.

Storage and supply are the initial steps, and they are found before the extraction vessel. The solvent storage tank is where the SCF is stored (Fig. 10.2-1), generally CO_2. For the extraction of food bioactives, these tanks require high purity standards such as food-grade specification (99%). Another important aspect is that these tanks come with an inner tube to collect CO_2 from the bottom of the tank; this is important for delivering liquid CO_2 into the following stages. After the storage tank, a chiller (Fig. 10.2-2) removes the necessary heat, keeping the CO_2 at a low temperature and ensuring that the liquid phase prevails previously entering the pump. After the chiller is the CO_2 pump (Fig. 10.2-3), this step plays a vital role since higher pressures will lead to higher energy demands and process costs. Pumps are critical since they deliver and keep the necessary flux and pressure during extraction and separation. Before the CO_2 reaches the extraction vessel, a heat exchanger (Fig. 10.2-4) adjusts the SCF to a target temperature.

After the $scCO_2$ reaches the temperature selected, it enters the extraction vessel (Fig. 10.2-5). The batch operation mode is typical on SFE, where two units can operate alternatively, and the extraction process can be continuous. While one vessel is in extraction mode, the other is under cleaning and preparation. The food matrix containing the bioactive is introduced inside the extraction vessel, and after being closed and sealed, the CO_2 starts the pressurization. After sealing, the pump will pressurize the vessel by adjusting the flux of the CO_2 delivered into the vessel. Once the desired critical conditions are reached, a static extraction takes place. Static extraction consists of allowing the supercritical fluid to diffuse into the matrix. During static extraction, solvent diffusion and bioactive solvation (with other similar compounds) occur.

Dynamic extraction occurs after static extraction. It consists of opening the control valve (Fig. 10.2-6) before the separation stage. At this point, the SCF moves to the separation valve, generating a dynamic SFE and making the pump compensate for keeping the system's pressure. In the dynamic extraction, soluble bioactives are extracted by the SCF from the matrix and carried into the separation step (Fig. 10.2-6). Previously to the separation stage, a heat exchanger (Fig. 10.2-5) is usually employed to increase the SCF's temperature. The SCF reaches atmospheric conditions at the separation step, generating different phases. When only $scCO_2$ is used as an SCF, the bioactives precipitate in the separator (Fig. 10.2-10) while the CO_2 displaces into the vapor phase (Fig. 10.2-9). If the SCF consists of CO_2 plus a co-solvent, the co-solvent condenses (usually as a liquid) with the bioactives. When the CO_2 reaches the separator, a solvent recycling step can be introduced, consisting mainly of a condenser (Fig. 10.2-11) and a filter. After condensation and filtration, the CO_2 returns to the storage tank.

10.2 Understanding the Thermodynamics of SCFs

Understanding the thermodynamic principles controlling SCF enables developing fine tunning solvation properties, benefiting the extraction of food additives. For SFE, the effect of pressure and temperature plays a critical role in variables such as density. Slight modifications of pressure and temperature lead to significant changes in density. Moreover, alterations in density make substantial changes in the solubility of the SCF for a specific bioactive. Hence, this section highlights the strong connection between temperature, pressure, and density.

10.2.1 Effect of Pressure and Temperature

Figure 10.3 displays the pressure-temperature (P-T) phase diagrams for CO_2 and water; common fluids employed in the extraction (subcritical and supercritical) of food bioactives. These charts help predict the predominant phases in a pure component as temperature and pressure changes occur. Moreover, they are necessary for predicting the properties of the substances (Smith et al. 2013a).

These charts show the different phases when CO_2 and water are under various pressures and temperatures. At the bottom left of each diagram, we find the solid state; this phase typically occurs at low temperatures and different pressures. The green curves in this section delimit a phase transition called sublimation, where there is an equilibrium between the solid and the gas phase. Water and CO_2 triple points are located at low temperatures and pressures (yellow circles). A characteristic of the triple point is the coexistence of three phases: solid, liquid, and gas.

When the pure substance finds in the blue lines, both solid and liquid phases coexist. The blue lines define the solid-liquid equilibrium, starting from the critical

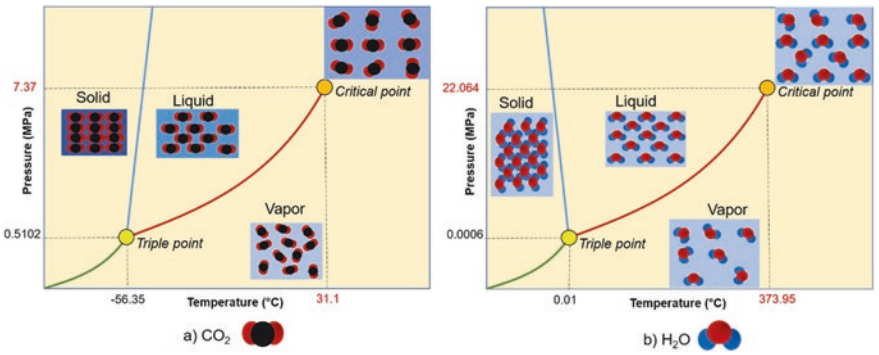

Fig. 10.3 Pressure-temperature (P-T) phase diagrams for CO_2 and water (Adapted from Smith et al. 2013a)

point and extending at different pressures but showing no variation with the temperature. For the CO_2 chart, the solid-liquid equilibrium (blue lines) slants to the right, whereas water slants slightly to the left. The slanting to the right in the water solid-liquid equilibrium attributes the crystalline (regular hexagonal) structure in the solid-state (Smith et al. 2013a).

By moving to the right inside the diagrams (increasing temperature), molecules increase their motion, generating a vapor (under the red curve) and a liquid (above the red curve) phase. The red curve dividing the liquid and the vapor phase represents the equilibrium where both liquid and vapor coexist. An important aspect to highlight is that as temperature and pressure increase, the vapor-liquid equilibrium curve reaches a state where the liquid or the vapor phase no longer exists (orange circle). Substances above these transition states form a homogeneous non-condensable single-phase resembling a gas (Smith et al. 2013a). This homogenous phase is known as a supercritical state, and as we mentioned earlier, it does not show a transition phase, displaying the properties of both liquids and vapors.

There are significant differences between the CO_2 and water phase diagrams. Molecular structure and intermolecular interactions are responsible for such a difference in behavior. For instance, critical water temperature (373.946 °C) and critical pressure (22.064 MPa) are considerably higher than those of CO_2 (31.1 °C, 7.37 MPa). The explanation of why water has a higher critical temperature and pressure attributes to hydrogen bond formation, which gives stronger molecular attractions in the liquid phase (Smith et al. 2013a). Another important aspect is that compared to CO_2 and other solvents, water can melt when the pressure is increased; solid-liquid equilibrium (blue line) slants to the right.

In terms of extraction, solubility plays a crucial role in developing an efficient SFE of food bioactives. Many industrial issues, such as environmental and economic, are easier to address if there are an acceptable yield and selectivity during extraction. Appropriate solvent solubility is vital to obtain adequate yields and selectivity. Optimizing the solubility for a desired bioactive in the SCF is the first step in developing a feasible SFE. From this perspective, pressure and temperature

variations in the SCF can significantly affect the system's solubility for a specific bioactive.

Therefore, by analyzing CO_2 and water P-T diagrams, it is possible to detect favorable specific solubility conditions for food bioactives. For instance, small changes in temperature and pressure near the critical region (subcritical region) significantly change the system's solubility. Above the critical region and the subcritical region, considerable solubility changes occur. The effect of pressure and temperature on solubility strongly relates to a drastic change of density. Hence, the impact of pressure and temperature on density demands further analysis.

10.2.2 Effect of Pressure, Temperature, and Density

When working with SCFs, the selection of pressure and temperature strongly affects density behavior. Hence, pressure-density (P-ρ) phase diagrams help understand the effect of pressure and temperature isotherms on density. Figures 10.4 and 10.5 display de (P-ρ) phase diagrams of CO_2 and water. The red lines represent different isotherms, while the blue dashed curve delimits saturation and the vapor-liquid state. The green semicontinuous lines delimit critical parameters (T, P, density), all meeting at the critical point (yellow circle). An interesting aspect to highlight is that critical water density is lower (0.322 g/cm^3) than CO_2 (0.467 g/cm^3).

As we can see in the diagrams, SCF pressure is plotted against density, and the temperature is represented as isotherms. One aspect to highlight from this plot is the

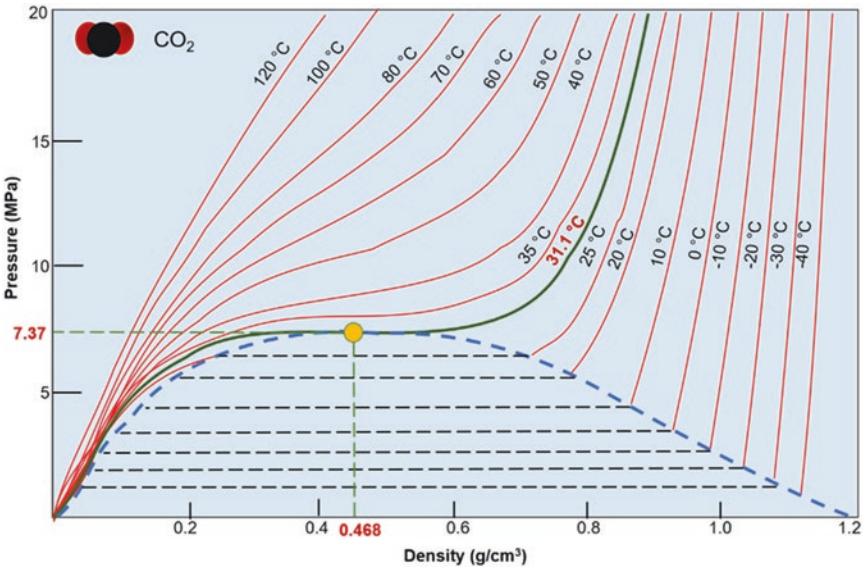

Fig. 10.4 Pressure-density (P-ρ) phase diagrams of CO_2 (Adapted from Smith et al. 2013b)

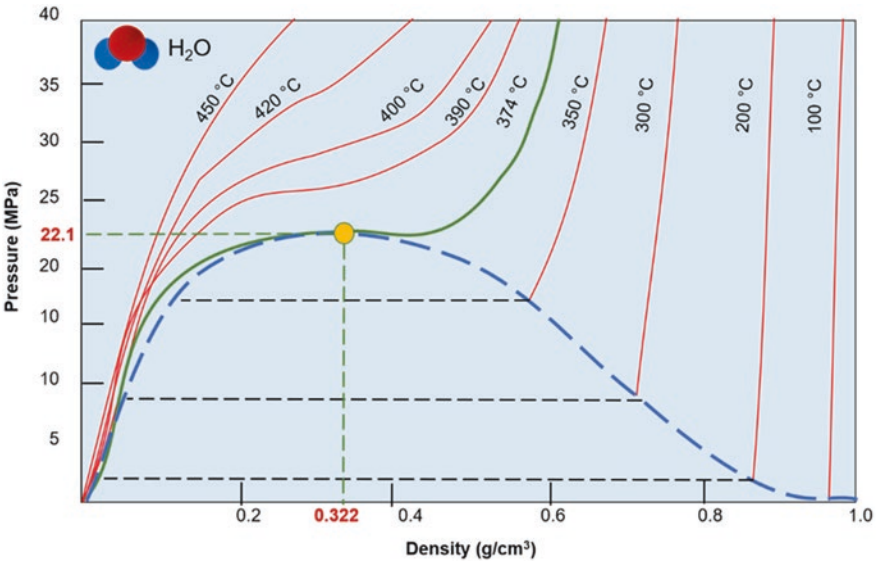

Fig. 10.5 Pressure-density (P-ρ) phase diagrams of water (Adapted from Smith et al. 2013b)

considerable density variations generated by the pressure and temperature, leading to solubility variations. For the case of CO_2, huge density variations (no linear behavior) emerge at temperatures and pressures under critical point to pressure up to the 15 MPa and temperatures up to 80 °C. For water, considerable density variations appear similarly in the subcritical and critical regions and extend to the 40 MPa and the 420 °C, respectively.

Considerable density changes occur when the temperature is higher or equal to the critical point. Increasing temperature leads to reduced density, whereas higher pressures increase density. As a result, the plots make evident the density variations by modifying pressure and temperature. In SFE, density variations allow fine-tuning the solubility of the SCF towards a specific bioactive. Changes in density also will bring changes in other key properties such as viscosity. Usually, a low viscosity in synergy with an intermediate diffusivity and a null surface tension present in the $scCO_2$ gives fast penetration into the matrix (Vardanega et al. 2019).

Another way to understand the interaction between pressure, temperature, and density is by looking into a 3D (P-T-ρ) phase diagram. 3D phase diagrams combine the previous charts, displaying pressure, temperature, and density in one chart. For example, Figs. 10.6 and 10.7 show the 3D diagrams for CO_2 and water, respectively.

The (P-T-ρ) 3D plots make it possible to visualize all the different thermodynamic states due to the three variables' influence. One of these benefits is that they provide a more realistic view of the relationship between the variables (P-T-ρ) and the different transitions between the thermodynamic states. The light green lines represent the temperature, whereas the blue ones represent the pressure. A red line represents the critical temperature, and a purple line represents the critical pressure.

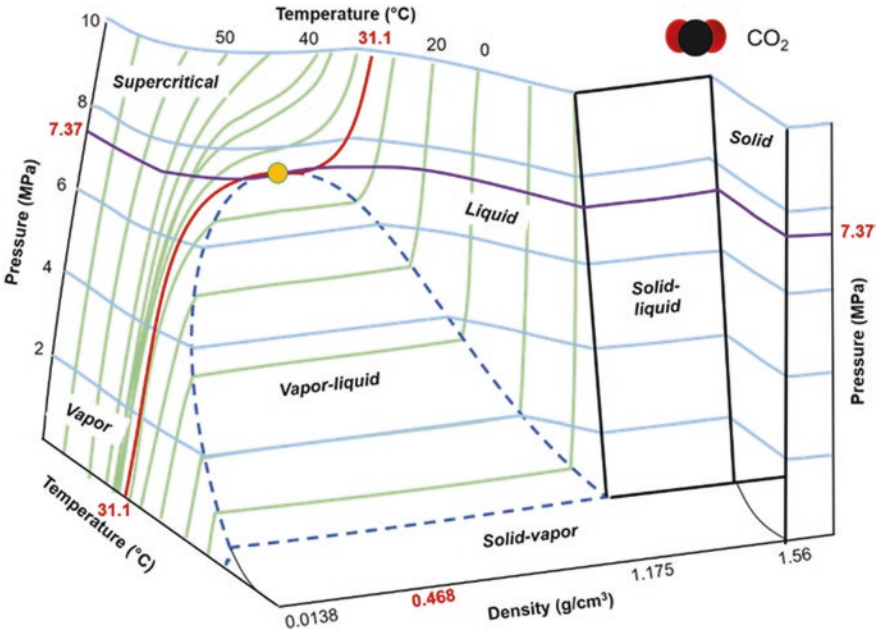

Fig. 10.6 Pressure-Temperature-density (P-T-ρ) phase diagrams of CO_2 (Adapted from Smith et al. 2013b)

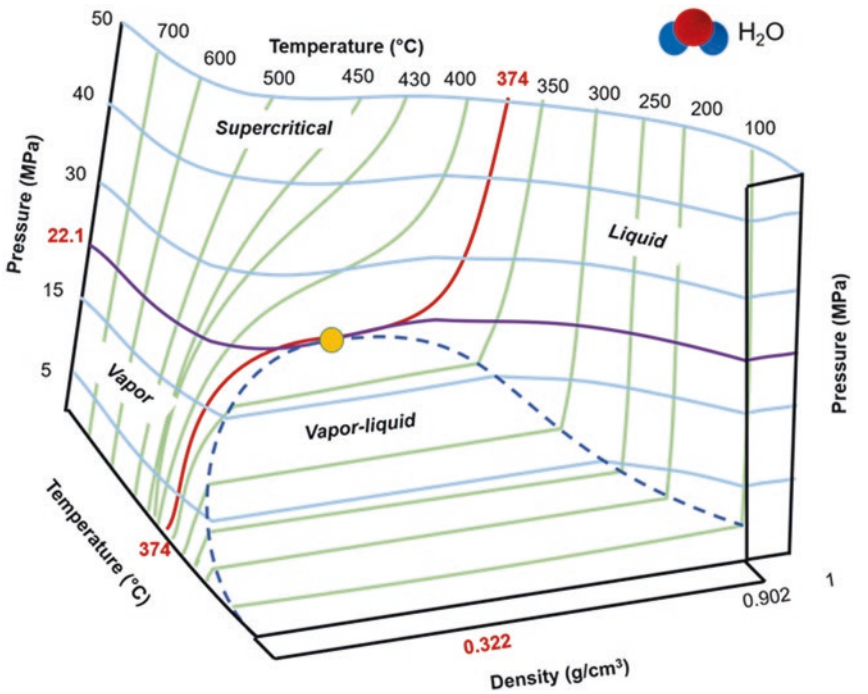

Fig. 10.7 Pressure-Temperature-density (P-T-ρ) phase diagrams of water. (Adapted from Smith et al. 2013b)

Each component's critical point is found at the center of each 3D graph (yellow circle), where the critical temperature, pressure, and density merge. As can be seen, the supercritical region locates above the temperature and pressure lines. CO_2 and water P-T-ρ plots look similar, where the main differences are the high temperatures, high pressures, and low densities found in water. The liquid-vapor phase in water typically extends more compared to CO_2. The hydrogen bond interactions among water molecules during their liquid state give water higher supercritical temperatures and pressures than other pure substances (Smith et al. 2013a). It is also responsible for the extended liquid-vapor equilibrium region extend in the 3D chart.

Consequently, the subcritical and supercritical regions favor changing density and solvent solvation capacity. This flexibility can represent a considerable advantage, where success depends on finding the appropriate solubility for a specific solute, making understanding isotherms and isobars vital for SCF behavior during extraction.

10.2.3 Developing a Thermodynamic Path for the SFE of Food Bioactives

Moving a fluid into the desired thermodynamic state (supercritical o subcritical) will depend on its initial state, path selection (3D chart), and final state. Some thermodynamic paths may be isothermic (constant temperature), isobaric (constant pressure), or isochoric (constant density). Moreover, the path must consider the fluid initial state and trajectory through a 3D plot during extraction and its return to the original state during separation (Smith et al. 2013a). The latter is critical for developing an efficient path easy to scale from the lab to an industrial scale. Aspects such as higher pressures make SFE costly to implement and should be considered. Hence, developing a path with optimum pressure and temperature will lead to a more efficient and economically feasible SFE process.

For instance, when removing water from delicate solid samples, the liquid-vapor phase must be avoided since developing a path through the vapor-liquid phase can generate abrupt changes in the volume. Avoiding the liquid-vapor phase (region under the blue semicontinuous curve) by increasing the system's pressure will lead to a gradual change in density or volume, especially for removing water in sensible food bioactives (Smith et al. 2013a).

10.2.4 Solubility Influence in the SFE of Food Bioactives

Similar to other extraction methods, SFE efficiency will be given by the solubility equilibrium of the food bioactive in the SCF. As we know, pressure and temperature considerably influence the SCF's solubility during the SFE. Hence, the ideal scenario for an efficient SFE will rely on optimizing temperature and pressure (among

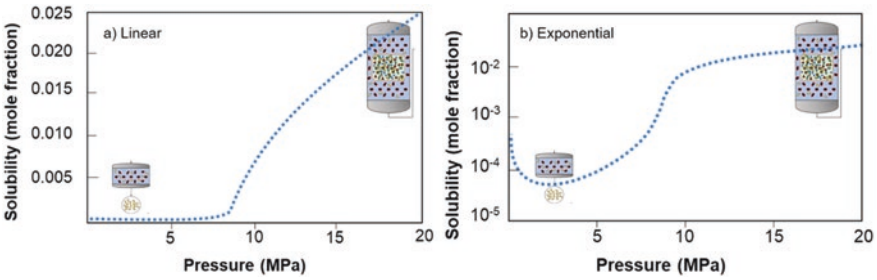

Fig. 10.8 An organic solid's Linear and logarithmic solubility profiles at different pressure and constant temperature ($T > T_C$) (Adapted from Smith et al. 2013b)

other variables) to improve the SCF solubility towards the bioactive of interest. For instance, if edible oil is extracted with $scCO_2$, an appropriate selection of pressure and temperature might lead to optimum solubility for maximizing yield. Figure 10.8 shows an example of the effect of pressure and constant temperature in bioactive solubility profiles.

Figure 10.8 displays two different solubility profiles: linear and logarithmic. For the first case (Fig. 10.8a), solubility increases linearly, showing solubilities in molar faction from 0.000 to 0.025. The second solubility profile (Fig. 10.8a) shows logarithmic behavior with solubilities from 10^{-4} to 10^{-2}. Both solubility profiles are at a constant temperature, where the effect of pressure on the system's solubility is evident. From this case, pressures from 10 to 20 MPa increase the solubility considerably; hence, it would be expected to take a high-pressure thermodynamic path. Simultaneously, the separation of the bioactives occurs when the fluid has low solubility for the bioactive. Another critical aspect is that solubility conditions can be considerably different during extraction and separation. As can be seen, high pressure (supercritical) is necessary during extraction, and lower pressures such as atmospheric enable the separation of the food bioactive from the SCF.

As shown in Fig. 10.9, the variation of pressure and temperature leads to significant differences in density and solubility, allowing the extraction of different food bioactives. Bioactives such as phenolics and terpenoids are successfully extracted under the supercritical point (subcritical). In contrast, bioactives such as polyphenols (highly polar) demand the highest pressures and temperatures to fine-tune solubility. For phospholipids and carotenoids, these long molecules also demand high pressures and temperatures for their extraction. Other bioactives such as triglycerides, diterpenes, and sterols are removed at low critical temperature and pressure. As discussed in other sections, bioactives molecular weight and moieties influencing polarity determine the most suitable SFE conditions. High pressures and temperatures can make extraction more efficient when molecules are long and with a considerable number of polar moieties such as polyphenols.

However, SFE efficiency should not be only measured by bioactive yield but also by assessing undesired by-products extracted (Smith et al. 2013b). An enhanced solubility for the desired bioactive can also extract similar compounds from the

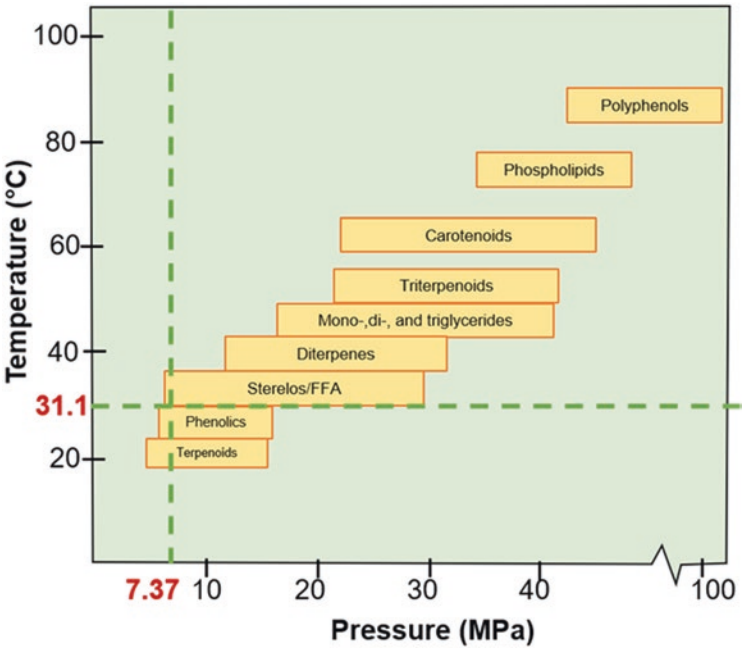

Fig. 10.9 Optimum CO_2 SFE conditions for important food bioactives (Adapted from Attard et al. 2018)

food matrix, leading to expensive separation methods such as chromatography. Therefore, implementing robust SFE experimentation during the early stages of process development is critical to identifying a balance between the optimum solubility necessary by the SCF during the extraction and future downstream implications. A robust analysis of the effects of pressure, temperature, and density on path selection might lead to optimum conditions (Attard et al. 2018). These conditions must allow acceptable mass transfer coefficients during the extraction of bioactives.

10.2.5 Cosolvent Effect Bioactives Retention

For the case of insoluble food bioactives, the use of a co-solvent is necessary. There are different co-solvents used in the SFE of food bioactives. From all these, ethanol is the most typical. Co-solvent concentrations between 5 and 10% vol. are typically employed. Introducing a co-solvent allows modifying the SFE system's solubility, increasing its polarity and the affinity towards polar bioactives, such as polyphenols or bioactives showing polar moieties in their molecular structure.

A co-solvent is a substance that dissolves in the SCF, enhancing the system's solubility for the target bioactive (Smith et al. 2013b). Co-solvents are customarily employed to increase the polarity of non-polar SCF. One example is CO_2, showing

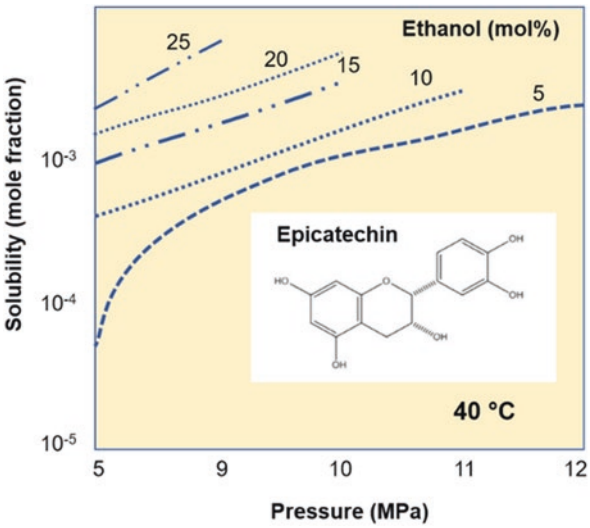

Fig. 10.10 Effect of co-solvent (ethanol) and pressure in epicatechin solubility at 40 °C. (Adapted from Smith et al. 2013b)

limited solvation capacity for polar bioactives. The term "polar" is sometimes difficult to interpret since it is usually associated with molecules such as alcohol and water. Nevertheless, bioactives usually are complex molecular structures with different moieties affecting their polarity. For example, it is common to find food bioactives with molecular structures showing both non-polar and polar segments on their structure. Thus, creating the necessity to be carefully assessed to understand the bioactive's overall polarity. One example is the SFE extraction of bioactives considered non-polar such as fatty acids (FA) and terpenoids. These compounds usually are better extracted using ethanol as a co-solvent since they contain polar moieties on their structure, affecting the overall polarity. Another example is the SFE of epicatechin using ethanol as a co-solvent (Fig. 10.10), where extraction efficiency is affected by the percentage of ethanol employed.

Extractions with more co-solvent (25% mol) increase epicatechin's solubility at lower pressures. On the other hand, when small percentages of co-solvent are employed, higher pressures aid to compensate and increase the solubility of epicatechin in the fluid. Thus, variations in ethanol percentage result in different solubility behaviors. Besides, the increment of pressure also aids by increasing the solubility of epicatechin. The decision of the most appropriate percentage of co-solvent employed during extraction will depend on several factors. For instance, employing high percentages of co-solvent might be a good strategy when working at low pressures. Simultaneously, some consequence will be the need for a more intensive solvent separation stage for bioactive isolation. On the other hand, when working with a low amount of co-solvent, it might need higher pressures to increase the solubility, demanding less solvent and less downstream operations.

For other cases such as the SFE of polyphenols, polar moieties in the polyphenolic structure are more abundant; hence, using a co-solvent might be necessary. Typical organic co-solvents from the alcohols are ethanol, methanol, and propanol;

from the ketones, acetone, and butanone; from the aldehydes, acetaldehyde; and the ester group, ethyl acetate (Smith et al. 2013b). A new SCF system forms when a co-solvent incorporates into the SCF extraction system. Cosolvent can strongly affect parameters such as the supercritical temperature and pressure of the new system. For instance, when alcohols such as ethanol are used as a co-solvent with $scCO_2$, the supercritical parameters (temperature, pressure, and density) change according to the mixture's composition. Co-solvents usually show higher supercritical temperatures than pure CO_2. As a result, when a SCF mixture is formed (SCF and co-solvent), a considerable increase in the mixture's supercritical temperature occurs. One can claim to extract in a supercritical region when the reality is that the system moved into the subcritical region because of the co-solvent addition. When $scCO_2$ is employed alone, the supercritical state can be easily reached. The addition of co-solvent yields a new solubility with new critical properties. As a result, the system's polarity increases, allowing the extraction of bioactives poorly soluble in $scCO_2$.

Hence, the variation in selected co-solvent, concentration, temperature, and pressure leads to many possible combinations for the optimization of SFE. This is a unique and exciting aspect of the subcritical and supercritical states, where customed solubility systems can be developed, leading to the possibility of always finding the optimum extraction conditions for a food bioactive.

The employment of a co-solvent also brings changes into the SFE, especially during bioactive separation. Contrary to direct bioactive precipitation, a liquid phase containing the co-solvent and bioactive exists from the separation step. Simultaneously the CO_2 exits as a gas phase (Fig. 10.11). For instance, extra separation steps might be required to isolate the food bioactive. Examples of these separation operations can be solvent evaporation, distillation, sublimation, and crystallization. As a result, the liquid phase containing the bioactive and the solvent will demand a more intensive separation to remove the co-solvent from the bioactive. Nevertheless, typically low co-solvent volumes (5–10%) are employed during SFE, obtaining concentrated bioactive liquid phases, making downstream separation less intensive than diluted phases usually obtained in other extraction methods.

From a laboratory perspective, low concentration systems where high solvent volumes are employed (high solvent ratio) might not seem to be an issue. Nevertheless, low concentration solutions commonly make the extraction challenging to scale up. This is because low concentration phases require energy/mass intensive operations to isolate the bioactive. A liquid phase (co-solvent + bioactive) produced from a SFE is typically a highly concentrated solution, making it easy to separate. Recovery and recycling are essential aspects that need full consideration when employing a co-solvent. They become a critical issue, especially if an industrial SFE is foreseeing. Usually, all these aspects are driven by food bioactive market value, by-products, co-solvent purity required, and co-solvent ease to handle at the industrial level. A robust SFE implementation will explore all these scenarios during the early stages of process development.

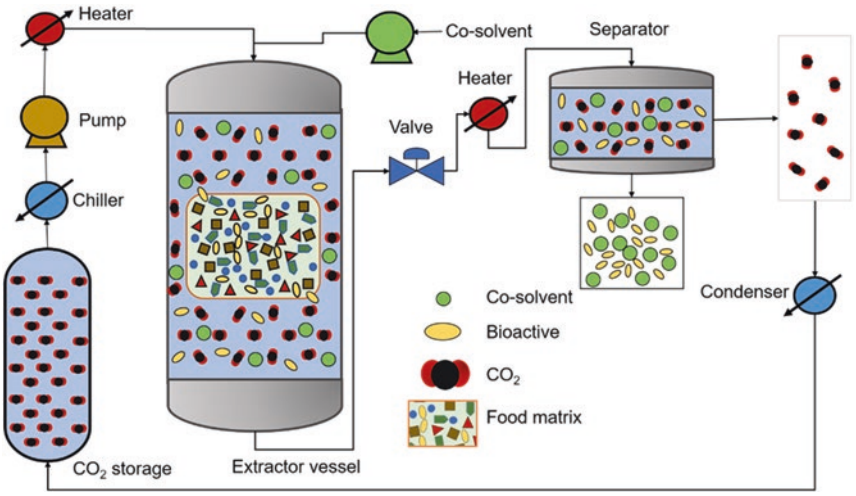

Fig. 10.11 SFE extraction system with the use of a co-solvent

10.2.6 Coextraction for the Retention of Bioactives in SFE

Coextraction consists of developing a previous extraction before the matrix is in contact with the SCF. An example of co-extraction is the SFE of caffeine from ground coffee. Caffeine (weakly polar) is soluble in $scCO_2$; however, it shows limitations because the caffeine in ground coffee is bound to chlorogenic acids. One example is 5-caffeoylquinic acid. Chlorogenic acids contained polar moieties, making them polar in some segments of their molecular structure, showing a high water affinity. Thus, when chlorogenic acids are in contact with water (moisturized), their structures become loose, promoting the release of free weekly polar caffeine and allowing its extraction by $scCO_2$ (Smith et al. 2013b).

Coextraction is also an excellent approach to increase yield during the SFE of food bioactives. However, as with co-solvents, it also demands introducing an additional solvent for enhancing bioactives solubility.

Understanding the difference between using a co-solvent and a coextract is essential. For instance, a co-solvent will mix with the SCF to create a homogenous (SCF + co-solvent) phase. This homogeneous phase diffuses into the food matrix and extracts bioactives of similar solubility. After the static (diffusion) extraction, the SFC and co-solvent phase leaves the food matrix by desorption (dynamic extraction) with the extracted bioactives. In the case of co-extraction, a high molecular weight solvent such as a complexing agent, fatty acid, or surfactant is in contact with the food matrix. Hence, a new heterogeneous phase between the food matrix and the solvent forms. This new heterogeneous phase leads to the co-extraction of bioactives with solubility similar to the co-extract. After that, the heterogeneous phase (matrix + coextract) is in contact with the SCF. The SCF extracts the coextract with the bioactive previously extracted. As a result, the co-extract works as an intermediary to make the extraction possible.

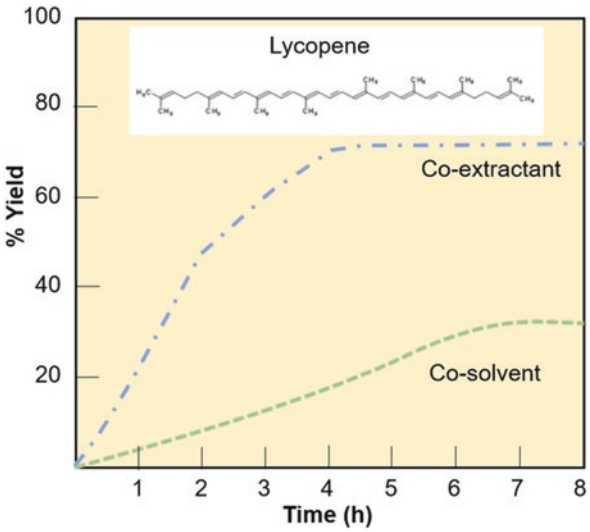

Fig. 10.12 Effect of hazelnut oil as co-solvent and co-extract in the SFE of lycopene. (Adapted from Ciurlia et al. (2009) and Smith et al. (2013b))

An example of SFE and co-extraction is the extraction of lycopene from tomatoes (Ciurlia et al. 2009). This long chain carotenoid has a high molecular weight, and its extraction with pure $scCO_2$ shows low yields. Therefore, an alternative strategy consists of employing a co-solvent or co-extract. For this case, using a co-extract is more effective (72% yield) than using a co-solvent (Fig. 10.12).

Figure 10.12 shows lycopene yield percentage against time. The blue dashed line represents the extraction yield when hazelnut oil is employed as a co-extractant. The green dashed line represents the same process when the oil is used as a co-solvent. Hazelnut oil is shown to be an effective lycopene coextract; its composition consists of triacylglycerols of similar structure and functional groups (carbonyl, ester, and alkene) similar to those of lycopene (Smith et al. 2013b). Moreover, viscosity modifications during the co-extraction improve the mass transfer of the bioactive inside the food matrix. An essential aspect of using a co-extract is that it is recommended that it should be part of the final product or formulation to avoid further separation.

10.3 Food Matrix Pretreatment in the SFE of Food Bioactives

Any solid-liquid extraction process consists of the solvent's diffusion through several barriers inside the matrix and the solvation of bioactives. Some examples of matrices for extracting food bioactives are parts of plants such as stigmas, roots, seeds, rhizomes, leaves, fruits, flowers, bark, and buds (Vardanega et al. 2019). Other bioactives food matrices are algae, yeasts, mushrooms, and animal tissue. The nature of the food matrix: its composition, the bioactives location inside the matrix,

and how the bioactive is bound to the matrix will dictate the most efficient pretreatment.

Different pretreatment methods are available (Marathe et al. 2017). They usually are classified as physical, mechanical, chemical, and biological (enzymatic). The selection of an appropriate pretreatment method demands an in-depth analysis of the matrix structure. Moreover, the selection must be ruled under minimum resource usage and enable an economically feasible process. Essential aspects such as waste generation from employing additional chemicals and solvents, potential implications of pretreatments on upstream and downstream operations need a complete evaluation. Regularly increasing yield and selectivity for the target bioactive while keeping the simplicity of the process are good indicators.

10.3.1 Physical Pretreatments

Some physical pretreatments typically employed in SFE of bioactives are maceration, particle size reduction, high-pressure, extrusion, or flaking. Maceration, extrusion, and high-pressure help disrupt the cell wall, exposing bioactives for future extraction. For the case of particle size reduction, previous evaluation is necessary to define the optimum particle size. Small particle sizes benefit the extraction process by providing a greater surface area at the moment of extraction. However, tiny particles can generate agglomeration leading to channeling, affecting the diffusion (Ordoñez-Quintana et al. 2020). The latter is critical when working with a food matrix. Food solids tend to become cohesive when the particles are reduced into considerably small sizes, leading to compaction and channeling. Ultrasound can also be considered a physical pretreatment since the cavitation emitted from a probe hits into the matrix, releasing bioactives and making them more available for the solvent. Something similar happens with microwave-assisted extraction, where microwaves contact the food matrix, aiding in releasing bioactives. Examples of these hybrid methods are ultrasound-assisted supercritical fluid extraction (UASFE) and microwave-assisted supercritical fluid extraction (MASFE).

10.3.2 Chemical Pre-treatments

Chemical pretreatment improves the solvent's diffusion, especially when bioactives are bound to other chemical species. Examples of this kind of pretreatment are acid or alkaline hydrolysis. These pretreatments are ideal for breaking strong lignocellulosic networks or other large hydrocarbons structures, retaining the bioactives of the food matrix. One example is the alkaline hydrolysis that can take place in cereals, such as forage oats, for the efficient SFE of bound polyphenols (Escobedo-Flores et al. 2018). Another example is dimethyl sulfoxide (DMSO) for releasing carotenoids (Monks et al. 2012). However, chemical pretreatment is not

recommended in all cases since it can be aggressive for some matrices. Preliminary experimentation is necessary to ensure that the acid or alkali does not react or degrade the bioactives in the matrix.

10.3.3 Enzymatic Pretreatment

Biological pretreatment can also release key bioactives from the food matrix by employing enzymes. The most common enzymes employed in extracting bioactives are pectinases, hemicelluloses, and cellulases. They usually come from bacteria, fungi, animal organs, or extracts from vegetables and fruits (Marathe et al. 2017). Usually, these pretreatments are less aggressive than chemical pretreatments. Therefore, they do not consume high volumes of solvents or generate problematic waste. One example is the enzymes used to release β-glucans from oats or cause cell rupture in algae, known for having a solid wall cell structure. Examples of specific enzymes used to release bioactives are available in the literature (Marathe et al. 2017). As a result, during the last years, SFE, including enzymatic pretreatments known as enzymatic assisted supercritical fluid extraction (EASFE), has been considered as a sustainable alternative. Examples of EASFE are releasing bioactives such as bound polyphenols (Mushtaq et al. 2015) and oleoresins (Dutta and Bhattacharjee 2015).

10.3.4 SFE as an Intensified Operation in the Retention of Bioactives

Ideally, there should be a synergy between the pretreatment and the SFE for enhanced bioactives removal. For the case of SFE, it will be expected that the SCF penetrates the matrix in a reasonable time (diffusion), showing considerable solvation for the bioactive. Usually, bioactives are trapped by chemical structures and cellular membranes inside the matrix (Smith et al. 2013b). Therefore, appropriate matrix pretreatment aids in making bioactives available for the SCF. However, pretreatment and extraction are two different phenomena that are sometimes not well understood; they often occur almost simultaneously. Figure 10.13 displays a representation of the two processes taking place during extraction.

As shown in Fig. 10.13, pretreatment helps to increase the surface area for solvent diffusion. Pretreatment ruptures the matrix for the release of bioactives. An important aspect to highlight is that pretreatment does not involve the solvation process, consisting of the solvent's interactions with the dissolved bioactives. Solvation is the process when the solvent (usually a liquid or SCF) is in contact with the previously pretreated matrix, and by molecular interactions (hydrogen bonding, Van der Waals forces), solvates the bioactives of similar solubility (likes dissolve likes). Hence, the pretreated matrix must interact with a solvent (organic, water,

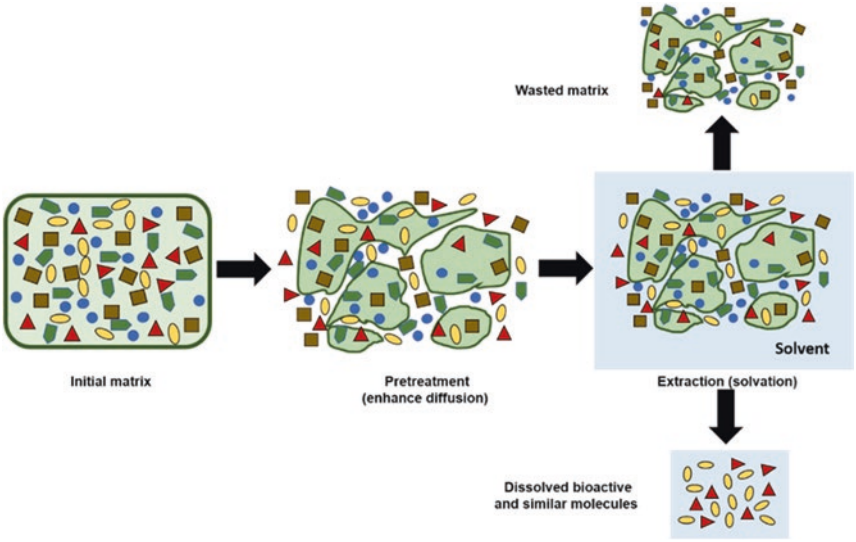

Fig. 10.13 Representation of a conventional pretreatment and extraction

SCF) to fulfill extraction. Almost all extraction methods, conventional or non-conventional, demand the use of a solvent for their solvation process, making solvent selection a critical task. Solvent's characteristics such as solubility, toxicity, life cycle, ease to handle, solid-solvent ratio, and yield considerably influence extraction economy and sustainability.

SFE can be implemented without employing a conventional solvent while taking advantage of high pressures as pretreatment. The latter is possible if the extraction relies only on $scCO_2$, where no co-solvent or coextract is employed. Thus, SFE can be considered an intensified operation (Fig. 10.14), where CO_2 high pressures provide pretreatment by penetrating the matrix and enhancing diffusion. At the same time, it is directly taking part in the extraction of the bioactive (solvation). As previously discussed, this approach will be limited to low polarity/small molecules. For other cases, a co-solvent or coextract will be necessary. Nevertheless, if a co-solvent is employed, high concentrations of bioactive can be obtained, and in the case of co-extraction, the coextract is typically part of the formulation.

As a result, it is critical to assess quantitively by experimentation to what extent a bioactive yield is given by the pretreatment (diffusion and release of bioactives) or by the solvent (solvation), which are two different processes. Nevertheless, the synergy of both processes leads to higher bioactives retention. For some cases, the correct selection of a SCF, co-solvent, or coextract will be sufficient for having an efficient SFE, requiring simple pretreatments, such as particle size reduction or maceration. However, for other cases, implementing more complex pretreatments is necessary, such as in matrices where access for the bioactives is difficult. Thus, it is critical to identify the mechanism of how pretreatments release bioactives, improving diffusion and contributing to improved solvation during SFE.

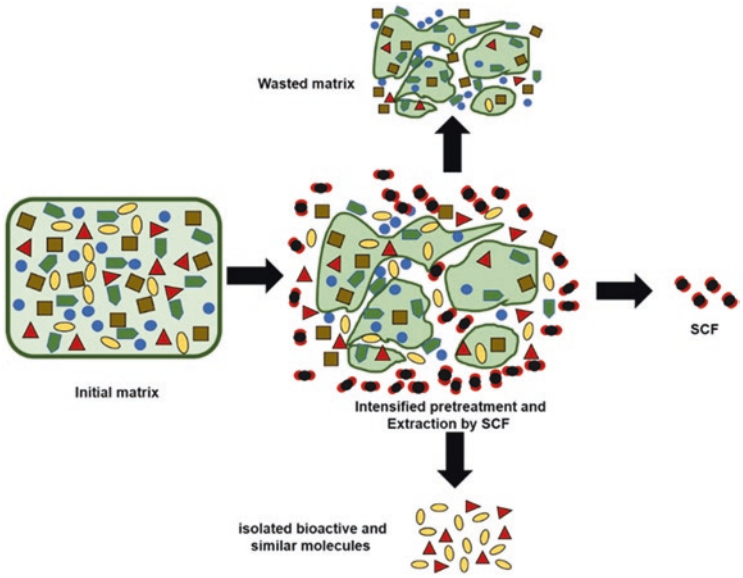

Fig. 10.14 Representation of an intensified SFE

10.4 Retention of Specific Food Bioactives by SFE

10.4.1 Glucosinolates and Derivatives (Isothiocyanates)

Glucosinolates (GLs) hydrolysis by-products have received particular attention for their anticancer and antibacterial properties. GLs are a family of secondary metabolites generally found in vegetables such as broccoli, cabbage, kale, brussels sprouts, and cauliflower (Angelino and Jeffery 2014), with concentrations between 0.1 and 2.5 g/kg. GLs are not biologically active until in contact with the enzyme myrosinase. Myrosinase hydrolyses GLs into glucose and aglycones of low stability, following a rapid conversion into isothiocyanates (ITC) and indoles (valuable bioactives).

When the vegetable is in its original (not processed) form, the GLs and the myrosinase are present in different locations of the food matrix. Hydrolysis can not take place until an external pretreatment (physical and chemical) process puts them into contact. Therefore, the recovery of GLs and ITCs from a vegetable matrix is critical for formulating nutraceuticals and functional foods, mainly when considerable quantities of vegetable waste generates along the food chains.

Conventional methods present limitations for the extraction of these compounds; a high volume of solvent and high temperatures are typically employed. One example is the losses (>60%) of GLs reported when extraction temperatures are higher than 50 °C in conventional extraction methods (Mohn et al. 2007). Another research focused on myrosinase's inactivation at 70 °C and 800 MPa (Okunade et al. 2015).

SFE of ITCs such as allyl isothiocyanate (AIT) from wasabi was reported (Li et al. 2010), where SFE employed higher pressures and low temperatures. The yield substantially increases by employing ethanol as a co-solvent. SFE fractionation was implemented to retain valuable bioactives such as GLs and polyphenols from rocket salad (Solana et al. 2014), showing a temperature of 75 °C, a pressure of 30 MPa, and water as co-solvent increased the yield.

GL can also be considered an undesired bioactive, depending on the purpose of the final food product. For instance, canola meal, the waste product of oil extraction from the canola seed, is investigated as a potential protein source (Sun et al. 2008). Furthermore, sensory characteristics improve during meal processing by removing GLs and polyphenols that impart a bitter taste. As a result, the SFE extraction of GLs from the food matrix has been successful, producing a protein-rich meal with a better chemical and functional composition than commercial meals.

As a result, for specific types of matrices (vegetables), precautions against thermal and enzymatic degradation are important; they might reduce phytochemicals such as GLs. Furthermore, it demonstrates that high temperatures can cause adverse effects such as cell lysis, inactivation of myrosinase, and loss of cofactors (Nugrahedi et al. 2015). Hence, using SFE at a lower temperature, higher pressure, and employing co-solvent might aid in the more efficient removal of bioactives.

As a result, an important area for SFE is removing bioactives such as GLs from waste vegetable sources used in food fortification. The fortification of foods rich in GLs can be precursors of important bioactives such as ITCs iberin and sulforaphane (Zinoviadou and Galanakis 2017). On the other hand, SFE also can help remove specific GLs responsible for providing bitterness, toxicity, and increasing consumer acceptance.

10.4.2 Lipids and Carotenoids

SFE is a viable alternative for the extraction of carotenoids, where high pressure and the appropriate co-solvent in the subcritical or supercritical region might show synergy. Carotenoids are an important group of food bioactives extracted from various source matrices. Some examples are grains, fruits, herbs, and yeasts. For example, lycopene is an essential bioactive extracted from apricot, watermelon, tomatoes, apricot, guava, and papaya. On the other hand, β-carotene finds in carrots, sweet potatoes, leafy vegetables, and olive oil.

For the case of lipids and carotenoids, pretreatments are essential and need an assessment before SFE. Assessment of the matrix cell wall composition and location of the bioactives inside the matrix can help develop a cell disruptor strategy (Klimek-Ochab et al. 2011). The cell wall blocks lipids' release; hence, cell disruption becomes essential for efficient extraction. Pretreatment such as high-pressure before conventional solvent extraction has shown reducing extraction time and temperature while obtaining intact lipids (Cho et al. 2012), leading to the possibility of implementing SFE operational pressure for cell disruption. Hence, accessibility to

the lipids in different compartments of the matrix is possible by higher pressures commonly employed for adjusting the polarity in the system in the extraction of lipids.

Optimum pressure and temperature for the extraction of carotenoids finds to be 30 MPa and 50 °C from dry biomass from a marine cyanobacterium *Synechococcus* sp. (Macías-Sánchez et al. 2007). Also, a pressure of 40 MPa and temperature of 60 °C were optimum for extracting 50% of the total β-carotene from a freeze-dried powder of the marine microalga *S. almeriensis* (Macías-Sánchez et al. 2010). Other studies show how temperature, pressure, and co-solvent influence the SFE of carotenoids from different algae (Macías-Sánchez et al. 2009), where high pressure and temperature seem to benefit the extraction. However, in the case of the co-solvent effect, it is not always observed to be beneficial. Chemical and physical pretreatments also can be employed for the release of carotenoids. For example, chemical pretreatment with DMSO (dimethyl sulfoxide) for cell disruption and SFE was proven to extract carotenoids (Monks et al. 2012). However, the process demanded an additional extraction with organic solvents. Ultrasound was employed as a pretreatment for the extraction of carotenoids. The cavitation effect over the matrix's cell wall efficiently released carotenoids from different matrix compartments (Singh et al. 2013). Furthermore, implementing pretreatments, such as maceration and enzymatic lysis, resulted in higher extraction yields than mechanical and chemical cell disruption methods (Michelon et al. 2012). As a result, implementing pretreatments such as ultrasound and enzymatic might help disrupt complex cell walls commonly found on yeasts and algae.

10.4.3 Essential Oils

SFE is an attractive technique for the separation of essential oils. The employment of low SFE temperature avoids degradation issues. Moreover, SFE allows adjusting the system's solubility towards an improved selectivity of a rich oil profile with no organic contaminants (Priyanka and Khanam 2018), all this while having an easy separation and avoiding the use of co-solvents. Typically pressures between 9 and 12 MPa and temperatures between 35 and 50 °C are effective in the SFE of essential oils (Fornari et al. 2012).

10.4.4 Phenolics

Nowadays, due to their multiple uses and benefits, phenolics are considered essential bioactives. The term phenolics include many compounds considering simple to very complex structures. An essential aspect is that they have phenol groups on their structures, from single phenolic groups to highly polymerized compounds. Like other bioactives, phenolics can be sensitive to temperature, oxygen, and light

(Yaqoob et al. 2020). SFE of phenolics with pure $scCO_2$ from different matrices can take place, where temperatures between 25 and 120 °C and pressures of 8 to 65.6 MPa are employed (Tyśkiewicz et al. 2018). More recent reviews (Yaqoob et al. 2020) have gathered different conditions in extracting phenolics from matrices such as fruits, vegetables, species, flowers, showing optimum temperatures from 35 to 300 °C and pressures from 3 to 57 MPa. Variations in temperature and pressure for optimum SFE from different matrices are common. It is a consequence of the phenolic's molecular structure, how phenolics are bound, and their location inside the matrix. Other aspects, such as pretreatments, can also influence optimum temperature and pressure during the SFE. Adding a co-solvent in the extraction of phenolics by SFE results in a considerable increase in extraction yield. Such efficiency is attributed to co-solvent's effect on molecular interactions such as dipole-dipole interaction and dipole-induced dipole, leading to density increments in the SCF (Ekart et al. 1993). Moreover, the use of ethanol and isopropyl alcohol (IPA) allows tailoring for specific phenolic selectivity (Yaqoob et al. 2020).

10.5 Influence of Variables on SFE of Food Bioactives

10.5.1 Summary of Important Variables

Variables such as density, pressure, temperature, co-solvent, and pretreatment directly affect solubility. Table 10.1 provides a summary of the effects of these variables.

Each SFE process has its challenges, demanding an in-depth analysis of the thermodynamics and the transfer phenomena controlling the extraction (Vardanega et al. 2019). Differences in matrix composition and pretreatments can demand different SFE conditions. Aspects such as matrix origin, species, production site, growing stress, and environmental factors during growth can considerably change the composition. Since diffusion and solvation are important variables, it is critical to understand which aspects affect them. For instance, diffusion is directly affected by pretreatment and SCF pressure; if there is an appropriate selection, the solvent's access into the matrix is improved. For solvation, the key variables are temperature, pressure, and co-solvent, directly affecting the solvation of bioactives during the SFE.

10.5.2 Keeping Flavors in the Food Matrix

When SFE is employed to remove an undesired compound, the removal of desired flavors can also occur. As discussed previously, solubility fine tunning occurs by adjusting variables such as temperature, pressure, and co-solvent. A food matrix can be a complex structure where thousands of different molecules are found. Moreover,

Table 10.1 Important variables and their effect on the SFE of food bioactives

Variable	Effects on bioactive removal by SFE
Density	The solubility of the system is a direct consequence of the SCF density. As a result, modifications on the pressure results in changes in density and bioactive solvation capacity (da Silva et al. 2016)
Pressure and temperature	Small changes in pressure and temperature can bring considerable density changes (Bhusnure et al. 2015). The control of pressure allows for a selective extraction, while an increase in temperature at constant pressure increases the polarity, benefiting the extraction of polar bioactives. In addition, solubility improves by increasing SCF density, resulting from increasing pressure. Keeping the temperature constant (isothermal process) and increasing pressure increases density and solvation power (Pereira and Meireles 2010) Keeping pressure constant (isobaric process) and increasing temperature will reduce SCF density and solvent power (Pereira and Meireles 2010)
SCF flow rate	High flow rates usually give shorter extraction times and increase capacity. The latter results from an enhanced convective and diffusive extraction rate, increasing SCF velocity over the solid matrix (Pires et al. 2019)
Matrix pretreatment	Pretreatments typically increase the surface area available for the SCF, releasing bond bioactives, increasing the mass transfer and the extraction rate.
Matrix characteristics	Aspects such as the porosity of the solid matrix directly affect the heat and mass transfer during extraction (de los Angeles Rodriguez Salazar et al. 2019)
Matrix moisture	Moisture on the food matrix and the bioactive molecular weight can also affect the system's polarity in SFE (Attard et al. 2018) On the one hand, moisture in the solid matrix can negatively affect the SFE of non-polar bioactives such as lipids, where drying before extraction is appropriate (de los Angeles Rodriguez Salazar et al. 2019). On the other hand, moisture can be employed as a coextract when it shows solubility for the bioactive, and there is no solubility between the SCF and the bioactive

many of these molecules show a similar chemical structure and similar solubility. Therefore, it is possible to expect the removal of other similar compounds. Hence, it is crucial to identify these similar species and define if they do not compromise the critical attributes of the food matrix or extract. The retainment of flavors in the food matrix can be essential for sensory characteristics. One example is the decaffeination of ground coffee. As mentioned in the previous section, a successful SFE in the industry is the removal of caffeine. Specific considerations concerning flavor are necessary to avoid removing these valuable compounds from coffee. One way of doing this is by injecting a mixture of these flavors with the sCO_2 and reaching their saturation. As a result, these essential compounds will not be extracted from the coffee matrix; hence, caffeine keeps its sensory attributes.

10.5.3 SFE Fractionation for the Removal of Different Bioactives

Usually, an extraction matrix is a complex heterogeneous structure with hundreds of bioactives of variable molecular structures located in different sections. Each of these matrix sections can have additional barriers, making its access difficult. When

SFE is carried at the correct pressure, temperature, co-solvent, and pretreatment, various added value bioactives become available for extraction. A real constraint is that all these bioactives have different optimum extraction conditions, making extracting all the bioactives in a single SFE unit impossible. As a result, a sequence of SFEs known as fractionation can be implemented as a strategy for the recovery of a wide range of bioactives of similar structures.

One example is the fractionated SFE of non-polar compounds such as essential oils and lipids from a food matrix. The process takes place by introducing different supercritical pressure at different separation steps. For example, the first fractionation or separation step occurs typically at a lower pressure ($scCO_2$, 10 MPa), extracting oily volatiles. Then, a second fractionation step is carried out at higher pressures (30 MPa), where the target bioactives are lipids and triglycerides.

10.6 A Holistic Perspective for SFE: Process, Economics, Scale-Up, LCA, and Future

10.6.1 SFE Scale-Up

Nowadays, SFE is applied on an industrial scale. The critical variables controlling SFE and its effect when scaling up have been analyzed for the last years. Among these analysis outcomes, we find that SFE has its challenges and must be carefully evaluated (Stoica et al. 2015). For example, transport phenomena expressed in mass transfer coefficients are critical. Therefore, criteria selection during SFE scaling up must keep or enhance this coefficient. Some essential reported criteria (York et al. 2004) for SFE scale-up are displayed in Table 10.2.

Criteria such as residence time (T_{RES}), SCF volumetric flow (Q_{CO2}), solvent mass (S), feed mass (F), vessel height (H), vessel diameter (d), and Reynolds numbers are critical to the successful scale-up of SFE. In addition, during experimental trials, a

Table 10.2 Important considerations and SFE scale-up criteria

Critical scaling factor	Scale-up criteria
Extraction limited by internal diffusion	Keep or increase the solvent's time with the solid known as residence time (T_{RES})
Extraction limited by internal diffusion	Keep the ratio Q_{CO2}/F to conserve T_{RES}
Extraction limited by solubility	Keep S/F ratio, solvent mass over feed mass
Extraction is limited by solubility and diffusion	Keep both Q_{CO2}/F and S/F
Extractor size (geometry and dimensions)	$(H/d)_{extractor\ 1} = (H/d)_{extractor\ 2}$
Dynamic criteria for diffusion and solubility	$(Q_{CO2})(d/F)$
Constant pressure and temperature	Keep Reynolds number

Table 10.3 Scale-up criteria employed in the SFE of food bioactives

Bioactive	Scale-up criteria employed	References
Oil (Peach Almonds)	Q_{CO2}/F	Mezzomo et al. (2009)
Oleoresins (Marigold flowers)	Q_{CO2}, d, H, Schmidt number	López-Padilla et al. (2017)
Phenolics (Mango leaves)	(Q_{CO2}) (D/F)	Fernández-Ponce et al. (2016)
Triterpene acids (*Eucalyptus globulus deciduous* bark)	(Q_{CO2}/F)	de Melo et al. (2014)
Fatty acids (grape (*Vitis vinifera L.)* seed)	(S/F)	Prado et al. (2012)
Ginger Oil (*Zingiber officinale var. Amarum)*	(S/F)	Salea et al. (2017)

scaling factor must be established. The scaling factor is considered a key variable and significant for bioactive yield. Usually, these variables have a significant impact on mass diffusion and solubility. Moreover, these variables can also be grouped mathematically. For instance, the relation S/F has been employed successfully as a scaling criterion in the SFE of food bioactives (Table 10.3). Other vital criteria employed in food bioactives SFE scale-up are the Q_{CO2}/F ratio, the diameter, and the vessel's height. The Schmidt number is also employed for keeping mass diffusion at the time of scaling.

As discussed above, diffusion and solvation are critical processes that, if optimized, typically will lead to high yield and selectivity. As a consequence, downstream operations will be easy to implement during scale-up. An extraction path selection and SFE equipment design usually will rely on understanding the different trajectories of an SCF in a thermodynamic phase diagram. Moreover, extraction vessels are critical for SFE; their design and manufacturing demand unique operation specifications under supercritical pressures (Martínez and Vance 2007). The vessel's design is critical for avoiding leaks and depressurization during extraction.

10.6.2 Retention of Food Bioactives by SFE Vs. Environmental Impacts

All processing comes with an environmental burden, and SFE is not the exception. Hence, it is necessary to understand the environmental implications when developing a strategy for retaining food bioactives in SFE. Nowadays, a holistic analysis is compulsory, where environmental impacts are detected from processing (micro-scale) and food system (macro-scale) perspectives. These make process developers understand the significant impacts during different steps of the food system: production, processing, retail, and disposal, and more importantly, the impact of that processing, in this case, SFE, adds to the food system.

One way to understand a food system's environmental impacts is by developing a life cycle analysis (LCA). This methodology relies on raw materials and energy balance inventories. At the same time, LCA quantifies substances that impact the environment from the beginning of a process (cradle) to their disposal (grave).

Several LCA studies have been taken place for the last 20 years with essential conclusions. One of these conclusions is that most environmental burdens in a food system are generated during the primary food production stages (Roy et al. 2009). The burdens are strongly related to fertilizers, water, and land use to produce crops. Another important conclusion is that animal food sources and livestock production considerably contribute to global warming gases and high consumption of resources such as water and land.

Food processing also contributes to environmental impacts. Commonly energy-intensive processes are linked to the generation of greenhouse gases (GHGs). Since SFE demands supercritical pressures, it demands high energy consumption in the pumping stages. The latter has been demonstrated on the LCA of the decaffeination of ground coffee by SFE (De Marco et al. 2018). The primary stages of coffee production present higher burdens, such as fertilizers and diesel consumption.

10.6.3 Towards a Sustainable SFE

Energy consumption is an issue that needs to drag the attention of SFE developers in the early stages of process development. Aspects such as optimizing process variables, heat integration, green energy adoption, and biorefinery approach are vital for developing a sustainable SFE process.

The optimization of the SFE to reduce energy consumption can be the first approach towards a more energy-efficient process. Its implementation does not demand considerable investment since it relies on laboratory experimentation to optimize food bioactive extraction. An important aspect is to pay attention to those variables that influence energy consumption. Higher pressures and long contact times strongly influence energy consumption in SFE. Hence, when developing an SFE thermodynamic path during the early stages, emphasis should be given to selecting a path where bioactive is successfully extracted while keeping pressure and extraction time to a minimum. Processing strategies such as pretreatment implementation can play a critical role in relaxing and minimizing operating variables in the SFE. The selection of food matrices with a high concentration of bioactives also helps bust the economy and reduce the process's energy expenditure. For instance, food matrices such as algae, yeasts, mushrooms, and some vegetables show considerably higher concentrations and variety of bioactives, making them attractive as raw materials.

Heat integration relies on taking advantage of the cold and hot streams produced in a process. For the case of SFE, the removal of heat to keep the SCF cold before it enters the pump is an example. Another opportunity for heat integration is during the compression and expansion of the SCF; one releases heat while the other absorbs

it from the environment. Heat integration's main goal consists of using cold and heat waste streams generated during SFE in operations inside the process demanding heat exchange. This analysis demands a critical economic evaluation since it typically demands high investment for process modification, and the economic return is generally seen in the long term.

Adopting renewable technologies such as solar panels, eolic, and biogas to produce energy is a potential alternative. The selection of the most appropriate technology will depend on several aspects, such as the technology available, the location of the SFE extraction plant, and the political and environmental context. Typically, the successful implementation of green energies involves a supporting policy. This alternative will demand a high initial investment, producing short and long-term benefits.

Another alternative is to develop an SFE based on a biorefinery concept where the food matrix goes through several upstream and downstream operations, removing added value bioactives. An example would be employing matrices rich in bioactives such as algae, yeast, and mushrooms. These matrices are rich in phenolic compounds, polysaccharides, terpenes and terpenoids, carotenoids, phenols, and peptides proteins. Other specific cases will be the SFE of bioactives such as carotenoids and polyunsaturated fatty acids (PUFAs). Algae is an optimum source of carotenoids and PUFAs since they have higher concentrations of these food bioactive and do not compromise excessive resources. In addition, they are resilient to different environments (Singh et al. 2011), making the SFE process more economically feasible. Marine yeast, fungi, bacteria, and marine algae are also rich sources of PUFAs. A typical example of these bioactives is omega-3 fatty acids (FA) (Gupta et al. 2017).

Implementing the correct pretreatment, selecting appropriate SFE conditions, and fractionation makes it possible to isolate different bioactives in a biorefinery. Since SFE can be particularly useful for isolating bioactives, the SFE biorefinery concept is a promising technology (Attard et al. 2018; Herrero and Ibañez 2018). Moreover, the economic benefits overcome other implications such as energy consumption. Thus, if the SFE biorefinery can extract bioactives from heterogeneous waste, considerable benefits to the economy and the environment can be obtained.

10.7 Conclusion

SFE has been around for some decades. Nevertheless, it has plenty of advantages for the extraction of food bioactives. Among these unique advantages is the capacity of fine-tuning solubility for the efficient extraction of polar, non-polar, complex, and non-complex molecular structures. It also allows processing intensification by developing pretreatment and solvation processes simultaneously with no conventional solvents or using co-solvent in small quantities. For the following years, more research will be necessary for the SFE of bioactives since each bioactive presents its challenges for optimization. Moreover, new multidisciplinary studies will bring more opportunities to increase the scale-up of this technology into sustainable businesses such as the biorefinery.

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Chapter 11
Influence of Modified Atmosphere Packaging on Food Bioactives

Gozde Oguz Korkut and Gurbuz Gunes

11.1 Introduction

Modified atmosphere packaging (MAP) involves changing the gaseous atmosphere in headspace of packages. MAP is used to extend the shelf life of food products through preserving their sensory properties and nutritional values. The most common gases used in MAP are CO_2, O_2 and N_2. For many food products, two or three different gas combinations are used according to product requirements. Bioactive components are generally found in small amounts in foods, but they have positive effects on important health problems such as cancer, cardiovascular and chronic diseases. Bioactive compounds play a protective role in human health by acting through an assortment of mechanisms (Dauchet et al. 2006). They enhance the activity of enzymes associated with detoxification of carcinogens and other harmful foreign substances; forestall cancer-causing nitrosamines; inhibit blood clotting; lower blood cholesterol level and platelet aggregation; control hypertension and diabetes. These compounds also contribute positively to human health by protecting polyunsaturated fatty acids, cell membranes and DNA from oxidative damage; inhibiting or diminishing cell proliferation; ensuring protection against heart disease and cataracts; blocking or suppressing cancerous changes as an antioxidant (Dauchet et al. 2006; Van Duyn and Pivonka 2000). Many studies have been carried out to determine how MAP affects the bioactive compounds in a wide variety of food products during storage.

This chapter ensures an overview of the basic principles of MAP and focuses on the effects of MAP on bioactive compounds in different food groups.

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11.2 Principles of MAP

MAP involves replacement of a package headspace with a gas mixture different than air in order to protect quality of products and to extend their shelf life. The desired atmosphere can be created in two different ways, active or passive modification. Active modification involves displacement of air in a package with a specific gas mixture by direct gas flushing or vacuum followed by gas flushing before sealing (Bodbodak and Moshfeghifar 2016; Shayanfar 2014). Passive modification can be created naturally because of the product's respiration and diffusion of gases from the packaging material after the products are hermetically sealed in a package (Opara et al. 2019; Shayanfar 2014). Vacuum packaging can also be considered as MAP and involves removal of the headspace gas before sealing so that the amount of oxygen in the package would be <1%. Reducing the O_2 concentration prevents the growth of aerobic organisms and reduces oxidative rancidity (Galić et al. 2009; Smith and Simpson 1996). Controlled atmosphere (CA) storage is based on maintaining a gaseous environment in a food storage rooms/chamber through continuous monitoring and regulation throughout the storage period (Blakistone and Blakistone 1998). Although CA is not a packaging process, different gaseous environments are used in large storage rooms to preserve quality of especially fresh fruits and vegetables.

The MAP is designed based on various factors such as product factors (weight, density, respiration and transpiration rate, ripeness stage, initial microbial load), extrinsic factors (gas concentration, storage temperature, relative humidity, storage duration), packaging factors (volume, thickness of the film, film surface area for gas flux, gas permeability), perforation factors (number of perforations, radius of perforations, permeability of packaging materials) (Arvanitoyannis 2012; Fonseca et al. 2002; Mahajan et al. 2008; Opara et al. 2019).

Optimum modified atmosphere is the equilibrium gas composition in the package headspace that has the most beneficial effects on product quality. It must be determined for each product before designing MAP. For non-respiring products (those other than fresh produce products) a packaging material with a high gas barrier property is required. The package headspace is actively modified with the desired gas mixture sealing. For fresh produce products MAP design is more complicated due to respiratory activity of the product. Mathematical models are utilized to successfully design MAP for respiring products. Gas concentrations in the package headspace do not change over time at steady state, thereby creating an equilibrium modified atmosphere (EMA). MAP is designed based on the following design equations representing the steady state conditions:

$$\frac{P_{O2}A_f}{x}P_{\text{atm}}\left[y_{O2,\text{out}}-y_{O2,\text{eq}}\right]=R_{O2,\text{eq}}w \tag{11.1}$$

$$\frac{P_{CO2}A_f}{x}P_{\text{atm}}\left[y_{CO2,\text{eq}}-y_{O2,\text{out}}\right]=R_{O2,\text{eq}}w \tag{11.2}$$

where $y_{O2,eq}$ *and* $y_{CO2,eq}$ *are the* O_2 *and* CO_2 *partial pressures, respectively, in the headspace at equilibrium;* $y_{O2,out}$ *and* $y_{CO2,out}$ *are the* O_2 *and* CO_2 *partial pressures, respectively, outside the package;* $R_{O2,eq}$ *and* $R_{CO2,eq}$ *are the* O_2 *consumption rate and* CO_2 *production rate, respectively, in the equilibrium gaseous atmosphere;* A_f*, surface area of the package;* P_{O2} *and* P_{CO2}*, permeability coefficient of package for* O_2 *and* CO_2*, respectively; x, thickness of packaging film;* P_{atm}*, atmospheric pressure; W, weight of product in the package.*

11.3 Gases Used in MAP

The main gases used in MAP are CO_2, O_2 and N_2. Besides, other gases such as Ar, N_2O, He, Xe and CO are also used in MAP applications. The effects of these gases on food quality are briefly summarized in the following sections.

11.3.1 Oxygen

Oxygen has both positive and negative effects on quality of food. O_2 partial pressures <1–2 kPa cause growth of anaerobic microorganisms such as *Clostridium perfringens* and *Clostridium botulinum*. At the same time, excessive reduction of the O_2 concentration causes anaerobic respiration, which leads to the production of ethanol and acetaldehyde, resulting in off-flavors and off-odors in fresh produce product (Bodbodak and Moshfeghifar 2016). In addition, low O_2 delays oxidative reactions in foods such as meat products and bakery products, or the browning reaction that occurs on the surfaces of fresh-cut fruits and vegetables. Oxygen is needed to maintain aerobic respiratory metabolism of fruits and vegetables and to preserve red meat color (Bodbodak and Moshfeghifar 2016; Parry 2012; Blakistone and Blakistone 1998).

11.3.2 Carbon Dioxide

Carbon dioxide has a strong inhibitory effect against the growth of aerobic microorganisms such as *Pseudomonas* species. However, the presence of CO_2 increases growth of lactic acid bacteria (Parry 2012). Dissolved CO_2 produces carbonic acid, which causes a pH reduction and acidifying effect, and thus, can suppress growth of microorganisms. CO_2 can also prolong shelf life by slowing the respiration rate of some fresh products (Bodbodak and Moshfeghifar 2016; Gunes and Kirkin 2012). It is known that CO_2 partial pressures >1–2 kPa decrease the susceptibility of fresh produce to ethylene: inhibiting its undesirable effects on product quality (Bodbodak and Moshfeghifar 2016). CO_2 is also used in hard cheeses, bakery products and fatty

fish in high concentrations to control microbial growth and extend shelf life. On the other hand, excessive absorption of CO_2 in foods with high moisture and high fat such as meat products can lead to package collapse. It may also cause taints in cream-based dairy products, which are very sensitive to high carbon dioxide concentrations (Parry 2012).

11.3.3 Nitrogen

Nitrogen is an inert gas and generally used as filling gas in MAP. Therefore, it contributes delaying oxidation, preventing rancidity and delaying growth of aerobic microorganism indirectly through replacing headspace O_2 in packages. It also prevents package collapse since it has low solubility in oil and water phases (Gunes and Kirkin 2012; Parry 2012).

11.3.4 Carbon Monoxide

Carbon monoxide is known to inhibit a wide variety of bacteria, molds and yeasts at partial pressures <1 kPa (Bodbodak and Moshfeghifar 2016; Blakistone and Blakistone 1998). When used with O_2 at 2–5 kPa, CO delays the oxidative browning of fresh products (Bodbodak and Moshfeghifar 2016). It stabilizes the bright red color in fresh meat through the formation of carboxyglobin (Blakistone and Blakistone 1998). CO is also used in MAP applications to prevent oxidation caused by high O_2 or irradiation (Gunes and Kirkin 2012; Kusmider et al. 2002). However, because its high toxicity and explosiveness creates safety hazards for employees in manufacturing facilities, its commercial use is very limited (Blakistone and Blakistone 1998).

11.3.5 Sulphur Dioxide

Sulfur dioxide (SO_2) is a highly chemically reactive in aqueous solution. SO_2 shows antimicrobial properties in its unbound non-ionized molecular form and inhibits bacteria by causing the formation of sulfide compounds in acidic conditions (pH < 4) (Bodbodak and Moshfeghifar 2016). SO_2 is used to control microbial growth in grapes, dried fruits, juices, wines, shrimp, pickles and some sausages and to prevent oxidative browning (Blakistone and Blakistone 1998). SO_2 is mostly used to reduce the spoilage of fungi during the storage and transportation of table grapes (Bodbodak and Moshfeghifar 2016). However, some restrictions have been imposed on its use due to the negative effects of SO_2 on product quality, human health and the environment. The European Food Safety Authority (EFSA) Panel on Food Additives and

Nutrient Sources has stated the maximum acceptable daily intake (ADI) of 0.7 mg SO_2/kg of body weight (EFSA Panel on Food additives and Nutrient Sources added to Food (ANS) 2016). The US Food and Drug Administration (FDA) has determined the maximum amount of SO_2 residue in fruit that is considered safe to be 10 μL/L (Lou et al. 2017). The International Organization of the Vine and Wine (OIV) recommends 150 mg/L total SO_2 for red wines. The European Union permits the total use SO_2 to 160 mg/L for red wines and 210 mg/L for white and rose wines. Australia has limited total SO_2 use to 350 mg/L for all wines (Guerrero and Cantos-Villar 2015).

11.3.6 Other Gases

Noble gases such as helium (He), argon (Ar), xenon (Xe) and neon (Ne), and nitrous oxide (N_2O) are also used in MAP applications. It has been stated that the use of noble gas reduces the growth of degrading microorganisms and has inhibitory effects on oxidative reactions and color changes (Bodbodak and Moshfeghifar 2016; Gunes and Kirkin 2012).

Argon is an inert gas like N_2 for the product. Ar has a higher density and is much more soluble in both water and oils than N_2. Therefore, it provides the use of less gas volume and better control against oxidation of flavor and color components of foods by showing a higher efficiency in displacement and removal of O_2 (Spencer 2005; Spencer and Humphreys 2002; Lide 2001). Ar is shown to have antimicrobial effects, thus its use together with CO_2 would require smaller concentration of CO_2 and thus reduce undesirable side effects of CO_2 on packaged products (Spencer 2005). In addition, Ar is effective in controlling enzymatic browning of fresh produce (Spencer 2005; Spencer and Humphreys 2002). The use of Ar in MAP was more effective in enhancing the total phenolic content of fresh-cut watercress, maintaining the 2,2 diphenyl-1-picrylhydrazyl (DPPH) scavenging activity and b-carotene blanching inhibition capacity than N_2, air, or vacuum packaging (Pinela et al. 2016).

N_2O has a high solubility in water and effects on various physiological processes such as inhibiting activity of cytochrome oxidase C and reducing respiration rate of fruits (de Siqueira Oliveira et al. 2020; Silveira et al. 2014; Sowa and Towill 1991), prolonging lag phase before ethylene increase and reducing ripening, and delaying color change in fruits such as tomatoes, avocados and bananas (de Siqueira Oliveira et al. 2020; Silveira et al. 2014; Leshem and Wills 1998; Gouble et al. 1995). Silveira et al. (2014) stated that the antioxidant activity of watercress increased with packaging in He and N_2O atmospheres at the end of the storage period compared to Ar, N_2 and air. However, no significant differences were found among gases in terms of color parameters, polyphenol contents and sensory parameters. Compared to N_2O and N_2, the use of Ar in MAP showed a better inhibitory effect on bacterial spoilage and hypoxanthine development during refrigerated storage and allowed to enhance the shelf life of sardine fillets by up to 12 days (Pinheiro et al. 2019).

11.4 Effect of MAP on Food Bioactives

Bioactive components are defined as compounds that are naturally occurring in foods, or can have and/or formulate physiological and biochemical functions when consumed by people or during food processing (Park 2009). The main bioactive compounds include prebiotics, probiotics, amino acids, peptides, proteins, omega-3, structured lipids, phytochemicals, vitamins and minerals; all of which can be obtained from various food groups such as fruits-vegetables, cereals, legumes, dairy products, and meat products (Fernandes et al. 2019).

The content of bioactive compounds found in foods is affected by various factors including environmental circumstances and storage conditions. Optimum storage conditions under CA and MAP are investigated to protect the content of these components. Effects of MAP on food bioactive compounds have been investigated in many studies, which are discussed in this section.

11.4.1 Effect of MAP on Fruit and Vegetable Bioactives

It is known that daily consumption of more fruits and vegetables have a significant impact on consumers' health by reducing the risk of cancer, Alzheimer's disease, cardiovascular disease including high blood cholesterol, hypertension, obesity, and diabetes (Liu et al. 2000). Fruits and vegetables are essential food groups rich in various bioactive compounds such as phytochemicals, dietary fibers, vitamins, and minerals (Liu 2004, 2013). Among the bioactive substances found in fruits and vegetables, the largest groups of compounds are phytochemicals, including carotenoids, phenolics, alkaloids, nitrogen-containing compounds and organosulfur compounds. These compounds are organic substances responsible for the colors, aromas, flavors and odors of foods.

Carotenoids are pigment groups liable for the yellow, orange, or red color of numerous fruits and vegetables. Carotenoids have provitamin A activity and demonstrate anticancer and antimutagenic properties. This group includes α-carotene (present in carrots, pumpkin, and red and yellow peppers), β-carotene (found in carrots, spinach, and sweet potatoes), β-cryptoxanthin (present in Citrus fruits), lycopene (found in tomato and tomato products), γ-carotene, lutein, zeaxanthin, capsanthin compouns. The important sources are red/yellow fruits and red/yellow/dark green leafy vegetables (Shashirekha et al. 2015).

Phenolic compounds, also called polyphenols, are one of the classes of secondary metabolite in plants. Polyphenols have antioxidant, antimutagen and anticarcinogenic activity (Yang 2011). Phenolic compounds are classified in 5 main groups as phenolic acids, flavonoids, stilbenes, coumarins, and tannins (Liu 2004). Broccoli, spinach, yellow onion, red pepper, carrot, mushroom, cabbage, potato, lettuce, Brussels sprouts, eggplant, asparagus, celery and cucumber are also rich vegetables, while fruits rich in phenolic compounds are berries, apple, red grape, pomegranate,

nectarine, mango, kiwifruit, pineapple, banana, peach, plum, lemon, orange, pear, grapefruit, cantaloupe and watermelon (Liu 2004, 2013). Flavonoids are one of the most comprehensive groups of phenolic compounds. Flavonoids can be subdivided into six subclasses such as flavonols, flavones, flavanols, flavanones, anthocyanidins and isoflavonoids (Liu 2013). Another component group, phenolic acids, is classified into two groups as hydroxybenzoic acid such as gallic, *p*-hydroxybenzoic, protocatechuic, vanillic and syringic acids, and hydroxycinnamic acid including include p-coumaric, caffeic, ferulic, and sinapic acids (Liu 2004).

Glucosinolates are sulfur and nitrogen-containing secondary metabolites that are found exclusively in *Brassicaceae (Cruciferae)* family including broccoli, cauliflower, Brussels sprouts, cabbage, collards, kale, turnip greens and mustard (Gil et al. 2020; Cartea and Velasco 2008). Myrosinase or thioglycoside glucohydrolase enzyme hydrolyzes glucosinolates to form biologically active compounds such as isothiocyanates, nitriles, thiocyanates, indoles, epithionitriles, oxazolidin-2-thiones and epithioalkanes (Cartea and Velasco 2008).

Vitamin C and Vitamin E (tocopherols and tocotrienols) found in fruits and vegetables, are important in human nutrition because of their antioxidant properties (Yalcin and Çapar 2017). Fruits and vegetables with abundant vitamin E are broccoli, leafy vegetables, olives, nuts, peanuts, avocados and almonds (Vincente et al. 2014). Vitamin C is found in a plentiful in tropical fruit species and leafy vegetables such as spinach, broccoli, cabbage rosehip, jujube, guava, persimmon, strawberry, kiwifruit, tomatoes, potatoes, cantaloupe and citrus fruits (Vincente et al. 2014; Yalcin and Çapar 2017).

To preserve the quality of fresh whole or fresh-cut fruits and vegetables, they should be packaged in a modified atmosphere to increase the shelf life. Reduced respiration rate is associated with extended shelf life during postharvest period. Low O_2 and high CO_2 as compared to air are often used to lower respiration rate of fresh produce. Optimum gas concentrations are different for each product. For fresh-cut products, higher CO_2 and lower O_2 concentrations can be applied as compared to their whole forms to get maximum benefits (Gunes and Kirkin 2012). With successful MAP applications, enzymatic browning, tissue softening, respiration rate, ethylene production rate, and microbial growth can be decreased on fresh-cut surfaces (Gunes et al. 2001).

Exposure of fresh produce products to MAP may also affect bioactive compounds as a consequence of physiological response to MAP. Some studies to determine the influence of MAP on fruits and vegetables bioactives are summarized in the Table 11.1 and discussed in the following paragraphs.

Figs were packaged under MAP (~4.3 kPa CO_2 + ~10.7 kPa O_2) for 28 days at 2 °C (Valero et al. 2013). Unpackaged samples were used as controls. It was found that total antioxidant activity and total polyphenols increased under MAP condition compared to unpackaged figs.

Fresh fenugreek leaves were packaged in polypropylene (PP) film (MAP1: perforated (one perforation) (8.3% O_2 + 10% CO_2), MAP2: perforated (two perforation) (8.8% O_2 + 10.2% CO_2), MAP3: non-perforated (2.8% O_2 + 11.9% CO_2) and MAP4: control samples were kept in packages under ambient conditions) and stored

Table 11.1 Effects of MAP on the bioactive compounds of fresh fruits and vegetables

Fruit/Vegetable	MAP conditions	Results	References
Fig (*Ficus carica* L.)	~4.3 kPa CO_2 + ~10.7 kPa O_2 Storage for 28 days at 2 °C	Total antioxidant activity and total polyphenols increased under MAP conditions compared to control	Valero et al. (2013)
Fenugreek leaves (*Trigonella foenum-graecum*)	MAP1:8.3% O_2 + 10% CO_2 MAP2:8.8% O_2 + 10.2% CO_2 MAP3:2.8% O_2 + 11.9% CO_2 Storage at 15 °C and 75% RH for up to 8 days	Antioxidant activity, total phenolics and β-carotene contents were higher in MAP3	Sidhu et al. (2016)
Green chillies (*Capsicum annuum* L.)	MAP1: 5% O_2 + 3% CO_2 MAP2: 2.1% O_2 + 1.9% CO_2 MAP3: 0.49% O_2 + 2.6% CO_2 MAP4: 12% O_2 + 0.5% CO_2 MAP5: control (unpackaged) Storage at 8 °C, 85–95% RH for 28 days	Total chlorophyll, antioxidant activity, capsaicin and ascorbic acid content were higher in MAP1	Chitravathi et al. (2015)
Broccoli (*Brassica oleraceae* L. var. italica cv. *Parthenon*)	MAP (5% CO_2 + 10% O_2) Control (unpackaged) Storage at 5 °C for 12 days	The contents of vitamin C, total phenolics, intact glucosinolate, carotenoid pigments and AA values in MAP samples was higher than control	Fernández-León et al. (2013)
Mixed mini broccoli (cv. *Milady*) and mini cauliflower (cv. *Clarke*)	MAP1: 8% O_2 + 14% CO_2 MAP2: 1% O_2 + 21% CO_2 Storage at 8 °C for 7 days	MAP2 for mini cauliflower and MAP1 for mini broccoli were best for preserving the aliphatic and indole glucosinolates	Schreiner et al. (2006)
Fresh-cut cauliflowers (var. *Star 4405*)	MAP1: 0.38% O_2 + 28.53% CO_2 MAP2: 19.4% O_2 + 1.62% CO_2 MAP3: 6.06% O_2 + 11.43% CO_2 MAP4: 16.87% O_2 + 5.87% CO_2 Storage at 5 °C, 85% RH for up to 12 days	MAP3 was best to preserve ascorbic acid content and maintain total phenols, flavonoid and glucosinolates concentrations	Mashabela et al. (2019)
Cauliflowers (B. oleracea var. *botrytis* cv. *Freemont*)	Controlled-atmosphere: 3% O_2+ 5% CO_2 Ambient air: 20.5% O_2 + 0.03% CO_2	No differences in glucosinolate profiles of cauliflowers between the two treatments	Hodges et al. (2006)

(continued)

Table 11.1 (continued)

Fruit/Vegetable	MAP conditions	Results	References
Chinese cabbage (*Brasicca rapa* L. ssp. *Chinensis*)	MAP1: 0.3% O_2 + 25% CO_2 MAP2: 18% O_2 + 3% CO_2 MAP3: 2% O_2 + 7% CO_2 MAP4: 5% O_2 + 15% CO_2 MAP5: 20.9% O_2 + 0.03% CO_2	MAP3 maintained the chlorophyll a, chlorophyll b, carotenoid and total phenolic contents and provided the highest AA and antioxidant activity at the end of the storage period	Mampholo et al. (2013)
Japanese plum cv. 'Blackamber' (*Prunus salicina* L.)	CA1: 1% O_2 + 3% CO_2 CA2: 2.5% O_2 + 3% CO_2 MAP: ~10% O_2 + 3.8% CO_2 Air Storage for 5 and 8 weeks at 0–1 °C plus 6 days at 21 ± 1 °C after each storage interval	During the first 5 weeks, the total phenolic content was maintained in the CA samples while it decreased in the samples stored in air and MAP remarkably During subsequent storage, the total phenolic concentration decreased regardless of the storage atmosphere At the end of storage period, CA treatments were more effective than MAP and air Antioxidant activity of samples in CA was higher than others during storage, while CA2 was higher than CA1	Singh and Singh (2013)
'Beynari' Pomegranates (*Punica granatum* L.)	MAP1: 17.60 kPa O_2 + 4.40 kPa CO_2 (Xtend® film) MAP2: 12 kPa O_2 + 5 kPa CO_2 (ZOEPAC) Storage at 6 ± 0.5 °C and 90 ± 5%RH for 120 days	At the end of the storage; the contents of total phenolic, ascorbic acid reduced and the total anthocyanin increased in all treatments Ascorbic acid, total phenolic and total anthocyanin contents were higher in the control(air) samples compared to the MAP treatments Malic and tartaric acid content were higher in the control than MAP samples, however, citric acid and oxalic acid contents were higher in MAP2	Selcuk and Erkan (2016)
Fresh-cut 'Piel de Sapo' melon (*Cucumis melo* L.)	MAP1: 2.5 kPa O_2 + 7 kPa CO_2, MAP2: 10 kPa O_2 + 7 kPa CO_2, MAP3: 21 kPa O_2, MAP4: 30 kPa O_2, MAP5: 70 kPa O_2 Storage at 4 °C for 14 days	MAP1 maintained vitamin C content and resulted in higher total phenolic content than others Induced an increased antioxidant capacity under MAP1 and MAP2	Oms-Oliu et al. (2008)

(continued)

Table 11.1 (continued)

Fruit/Vegetable	MAP conditions	Results	References
Cranberries ('Stevens' and 'Pilgrim' cultivars)	CA storage at combination of O_2 (2, 21, 70%) and CO_2 (0, 15, 30%), storage at 3 °C for 2 months	No effects of CA on total phenolics. Antioxidant activity decreased by 30% CO_2 in the presence of 21% O_2. 'Stevens' had higher antioxidant activity than 'Pilgrim'	Gunes et al. (2002)

at 15 °C and 75% RH for up to 8 days (Sidhu et al. 2016). The chlorophyll content, the total phenols content and β-carotene were best retained in MAP3 with lower O_2 levels compared to other packages at the end of storage. The antioxidant activity (DPPH) was also higher in MAP3. On the other hand, flavonoid content was highest in MAP1, while it was lowest in MAP3.

Green chilies were packaged under different MAP conditions MAP1: 5% O_2 + 3% CO_2; MAP2: 2.1% O_2 + 1.9% CO_2; MAP3: 12% O_2 + 0.5% CO_2, MAP4: 0.49% O_2 + 2.6% CO_2) and stored for 28 days at 8 °C and 85–95% RH (Chitravathi et al. 2015). Unpackaged samples were used as control. The ascorbic acid content of all the samples reduced gradually during storage but this reduction was significantly higher in peppers packaged under MAP3 and control samples, because they had higher O_2 and lower CO_2 concentration and had a faster rate of ripening. During storage, total phenolic content enhanced, whereas total flavonoids declined progressively in the control and the MAP specimens. The combination of higher O_2 and lower CO_2 concentration resulted in a further increase in the total phenolic content and in a further decrease of total flavonoids. While total antioxidant activity increased in early days of storage, it decreased in later stages due to senescence. After 28 days, it was observed that antioxidant activity was lower in higher O_2 and lower CO_2 concentrations. MAP1 provided better preservation of its antioxidant activity due to the delay in the progression of senescence. It was observed that capsaicin content was higher under MAP1 compared to other MAP conditions due to delay in the process of senescence.

Broccoli was packaged in an active MAP (5% CO_2 + 10% O_2) and stored at 5 °C for 12 days (Fernández-León et al. 2013). Unpackaged samples were used as controls. During the storage period, the carotenoid (β-carotene and lutein) contents decreased in both samples, but the reduction was lower in MAP as compared to air-packages. At the end of storage, the contents of vitamin C (ascorbic and dehydroascorbic acid) in MAP was approximately 1.5 fold higher as compared to air-samples. The MAP resulted in lower reductions in total phenolics and intact glucosinolate contents during storage as compared to the control. The loss of total phenolic content and glucosinolate content in MAP samples was approximately 20% (from 86.75 to 69.41 mg chlorogenic acid equivalent/100 g FW) and 23% (from 96.55 to 74.34 mg sinigrin equivalent/100 g fresh weight), respectively, whereas these values were 48% (from 86.75 to 45.12 mg chlorogenic acid equivalent/100 g FW) and 57% (from 96.55 to 41.73 mg sinigrin equivalent/100 g fresh weight) in the control samples. Moreover, ascorbic acid reduction at the end of the

storage period was lower in the MAP samples (24.76% less than the fresh sample) than the control samples (43.36% less with regard to the fresh sample).

Mixed mini broccoli and mini cauliflower were packaged in two different MA conditions (MAP1: 8% O_2 + 14% CO_2 and MAP2: 1% O_2 + 21% CO_2) and stored for 7 days at 8 °C (Schreiner et al. 2006). It was reported that the aliphatic and indole glucosinolates of mini broccoli were best preserved by MAP1 at the end of seventh day. In contrast to broccoli, MAP2 provided the best retention of the glucosinolates of the mini cauliflower (Schreiner et al. 2006). However, in order to store mixed broccoli and cauliflower florets in the same package for up to 7 days at 8 °C, MAP2 was recommended since it also maintained external appearance and prevented formation of off-odors (Schreiner et al. 2007).

Fresh-cut cauliflowers were packaged in four different films with different equilibrium MA before stored at 5 °C and 85% RH for 12 days; MAP1: 0.38% O_2 + 28.53% CO_2 with polypropylene (PP) film, MAP2: 19.4% O_2 + 1.62% CO_2 with Peakfresh® film (PF; an ethylene absorber film), MAP3: 6.06% O_2 + 11.43% CO_2 with NatureFlex film patched over 20% of a BOPP (20NF; a cellulose-based polymeric film), (and MAP4: 16.87% O_2 + 5.87% CO_2) with NatureFlex film patched over 40% of a BOPP (40NF) (and (Mashabela et al. 2019). MAP1 and MAP 2 resulted in the highest total phenol, flavonoid (quercetin), antioxidant activity (the ferric reducing antioxidant power (FRAP) and 2,2′-Azino-bis(3-Ethylbenzothiazoline-6-Sulfonic Acid) (ABTS+) assays) on day 4 and day 8, respectively. Thus, the phenolic contents and antioxidant activities were stimulated by low O_2 and high CO_2 in the first 4 days but this effect was faded towards the eighth day. On day 8, indole glucosinolates such as glucobrassicin, 4-methoxy glucobrassicin and 1-methoxy glucobrassicin concentrations in fresh-cut cauliflower were in the following order: MAP1 > MAP3 > MAP4 > MAP2.The concentration of sinigrin reduced during storage in the presence of higher CO_2 atmospheres in MAP1 compared to the other packages. The researchers suggested that cauliflowers would be packaged under MAP3 as it preserved the color and ascorbic acid content as well as moderately maintaining total phenols, flavonoid and glucosinolates concentrations for fresh-cut cauliflower.

Cauliflower heads were stored in ambient air (~20.5% O_2 + 0.03% CO_2) and a CA (3% O_2 + 5% CO_2) at 0 °C for up to 56 days in another work (Hodges et al. 2006). There was no difference in glucosinolate profiles of cauliflowers between treatments during storage. Gluconapine and gluobracycin increased with both treatments, while the levels of sinigrin, progoitrin, epiprogoitrin, 4-OH-gluobrassicin, 4-MeOH-gluobrassicin were unchanged during storage.

Chinese cabbage were packaged under different MA with different BOPP films: MAP1 (0.3% O_2 + 25% CO_2), MAP2 (18% O_2 + 3% CO_2), MAP3 (2% O_2 + 7% CO_2), MAP4 (5% O_2 + 15% CO_2), MAP5 (20.9% O_2 + 0.031% CO_2; with two 2-mm holes at the bottom (macro-perforation)) and stored at 10 °C, 80% RH, for up to 10 days (Mampholo et al. 2013). The unpacked leaves were used as control. High oxygen in control, MAP2 and MAP5 caused a further decrease in the amount of chlorophyll and carotenoids by increasing the enzymatic degradation activity on the pigments. The loss of AA content was significantly higher in the control, MAP2 and

MAP5 during storage time due to higher O_2 concentration. During post-harvest senescence, it has been reported that the total phenolic content increased because of wilting, dehydration and fragmentation of leaf tissue in the leaves of MAP5 and in control samples. The total phenolic content was lower in MAP1 and MAP4 packages with higher CO_2 concentration. MAP3 demonstrated the highest radical scavenging activity, maintained the chlorophyll a and b, carotenoid and total phenolic contents, and provided the highest AA and antioxidant activity as well as minimizing post-harvest spoilage and maintaining overall quality at the end of the storage period.

'Blackamber' Japanese plums were stored in air, CA1 (1% O_2 + 3% CO_2), CA2 (2.5% O_2 + 3% CO_2) and MAP (~10% O_2 + 3.8% CO_2) for 5 and 8 weeks at 0–1 °C plus 6 days at 21 ± 1 °C after each storage interval (Singh and Singh 2013). Total phenolic content was maintained in the CA samples while it decreased in the samples stored in air and the MAP remarkably during the first 5 weeks. As the storage time increased to 8 weeks, a greater decrease in the total phenolic was observed in the fruit regardless of the storage atmosphere. At the end of the storage period, no difference between total phenolics of samples in CA1 and CA2 was observed, and the samples in the CA had higher total phenolics than those in the MAP and air. In addition, DPPH radical scavenging activity of the samples in CA2 was higher than that of samples in CA2 which resulted in higher antioxidant activity than the MAP and air.

Pomegranates were packaged with two different commercial packaging materials (MAP1: 17.6 kPa O_2 + 4.4 kPa CO_2; MAP2: 12 kPa O_2 + 5 kPa CO_2) at 6 °C and 90% RH for 120 days (Selcuk and Erkan 2016). Unpackaged fruits stored in plastic boxes were used as control. While the contents of total phenolic, ascorbic acid and organic acids (except for tartaric acid) reduced, the total anthocyanin contents increased in all treatments at the end of the storage. Ascorbic acid, total phenolic and total anthocyanin contents were higher in the control samples compared to the MAP treatments at the end of the storage. Effect of MAP applications on ascorbic acid, total phenolic and total anthocyanin content was not statistically different from each other. The contents of malic and tartaric acid were higher in the control than both MAP, however, citric acid and oxalic acid contents were higher in MAP2.

Fresh-cut melon were stored under different MAP conditions (MAP1: 2.5 kPa O_2 + 7 kPa CO_2, MAP2:10 kPa O_2 + 7 kPa CO_2, MAP3: 21 kPa O_2, MAP4: 30 kPa O_2, and MAP5: 70 kPa O_2) during 14 days at 4 °C (Oms-Oliu et al. 2008). MAP1 containing the low O_2 maintained vitamin C content and resulted in higher total phenolic content than others. It was found that the MAP1 and MAP2 with low O_2 induced an increased antioxidant capacity during storage compared to the other treatments.

Cranberries (Stevens and Pilgrim cultivars) were stored at wide ranges of CA including super atmospheric O_2 (70%) and elevated CO_2 (30%) at 3 °C (Gunes et al. 2002). Total phenolic contents of the fruits were not affected by the CA while total flavonoid content was decreased by 15% CO_2 as compared to 0 and 30% CO_2. Antioxidant activity of the berries were lower at CA of 21% O_2 + 30% CO_2 as compared to air. However, this CA was the best in terms of prevention of decay and shelf-life extension.

11.4.2 Effect of MAP on Meat Bioactives

Although meat products are thought to have negative effects on health due to their high cholesterol, saturated fatty acids and sodium content, they are important food group because of their positive effects against some chronic diseases such as obesity, diabetes, alcoholism, neurodegenerative diseases (Alzheimer, Parkinson) and cancer (Kulczyński et al. 2019; de Castro Cardoso Pereira and dos Reis Baltazar Vicente 2013). Meat and meat products are rich in minerals such as iron, iodine, magnesium, zinc, calcium, selenium, manganese and vitamins A, C, D, E and B (B_2, B_3, B_6 and B_{12}) (Arihara and Ohata 2008; Mulvihill 2004; Pogorzelska-Nowicka et al. 2018). Meat fat consists of saturated fatty acids (SFA), polyunsaturated fatty acids (PUFA) and monounsaturated fatty acids (MUFA) (Jiménez-Colmenero 2007). Other components such as conjugated linoleic acid, L-carnitine, taurine, coenzyme Q_{10} (ubiquinone), creatine and creatine phosphate, putrescine, spermidine, spermine, tocopherols, carotenoids, ascorbic acid, glutathione, lipoic acid, uric acid, ancerin and l-carnosine are also meat-based bioactive compounds. These compounds play an important role in preventing cells from harmful effects, wound healing, metabolic diseases and stress-related diseases by showing anti-carcinogenic, anti-arteriolerotic, antioxidative and immunomodulatory activities, reducing oxidation reaction, increasing the activity of antioxidant enzymes (Arihara and Ohata 2008; Kulczyński et al. 2019).

The MAP conditions vary for different types of meat and meat products. Microbial growth and oxidation (off-flavor, off-odor and discoloration) are the primary cause of quality loss in fresh red meat. High O_2 is required to protect the bright red color of beef and prevent growth of microorganisms on its surface. Despite these positive properties, high O_2 can cause oxidation of meat fats. The most common gas mixture used in the packaging of fresh red meat is 80% O_2 + 20% CO_2 and 25–90% O_2 + 15–80% CO_2 (McMillin 2008; Gunes and Kirkin 2012). Although vacuum packaging is another packaging method used for fresh meat, the product is subjected to mechanical strain and it causes more drip (Robertson 2016; Gunes and Kirkin 2012). In addition, MAP with low or no O_2 can also be used. In this case, high CO_2 (up to 100%) balanced with N_2 is added into the packaging (Gunes and Kirkin 2012). Vacuum packaging is also an application used to prolong the shelf life of poultry products. Oxygen-free or low-O_2 application with high CO_2 (≥20%) provides better protection compared to vacuum packaging (Gunes and Kirkin 2012). It is recommended that fresh chicken meat is packaged in 25–70% CO_2 balanced with N_2 (Saucier et al. 2000). It has been reported that high CO_2 (≥40%) in anaerobic MAP prevents microbial spoilage and thus increases the shelf life of fish and seafood (Gunes and Kirkin 2012; Yesudhason et al. 2009; Sivertsvik et al. 2002). Seasoned ready-to-cook meatballs, marinated ready-to-cook products, fermented products such as sausages, hotdogs, salami, pastrami etc. can be packaged in anaerobic MAP containing elevated CO_2 (20–35%) or in high CO_2 with low O_2 (up to 5%) (Gunes and Kirkin 2012; Parry 2012).

Table 11.2 Effects of MAP on bioactive compounds of meat and meat products

Meat products	MAP conditions	Results	References
Fresh chicken carcasses	MAP1: 70% CO_2 + 30% N_2 MAP2: 30% CO_2 + 70% N_2 Storage at 2, 4, 7 and 9 °C up to 24 days	FFA levels of the samples stored at 2 °C were similar in both MAP until the 17th day, FFA increased at a faster rate in MAP2 afterwards The inhibitory effect of MAP on FFA was found to be more evident in MAP1 at low temperatures (2 and 4 °C)	Sawaya et al. (1995)
Fresh chicken breast	MAP: 30% CO_2 + 70% N_2 air Storage at 4 °C for up to 17 days	Tyramine, putrescine and cadaverine levels increased, whereas spermine and spermidine levels decreased during storage in all samples Higher spermine, putrescine and cadaverine levels in control samples and higher spermidine and tyramine levels in MAP were observed	Balamatsia et al. (2006)
Precooked chicken	MAP: 30% CO_2 + 70% N_2 Air Storage at 4 °C for up to 23 days	Tyramine, putrescine and cadaverine levels increased with storage time in both treatments Higher putrescine and tyramine levels in control and higher cadaverine and spermine in MAP was found	Patsias et al. (2006)
Pork loin	Air Vacuum MAP: 70% O_2 + 30% CO_2 Storage at 4 °C up to 20 days	Cholesterol levels increased at the end of storage in all treatments The highest increase in MAP followed by air and vacuum	Cayuela et al. (2004)

The primary objective of the use of MAP in meat product is to inhibit organoleptic quality loss and shelf-life extension. MAP can also have influence on bioactive components of meat products, but limited information on this exist in literature. Some studies to determine the influence of modified atmosphere packaging on meat bioactives are summarized in the Table 11.2 and discussed in the following paragraphs.

Fresh chicken carcasses were packaged under two different MAP (MAP1:70% CO_2 + 30% N_2; MAP2: 30% CO_2 + 70% N_2) and stored at 2, 4, 7 and 9 °C up to 24 days (Sawaya et al. 1995). While the free fatty acids (FFA) levels of the samples stored at 2 °C were similar in both MAP until the 17th day, it increased at a faster rate in MAP2 afterwards. The inhibitory effect of MAP on the production of FFA was found to be more evident in MAP1 at low temperatures (2 and 4 °C).

Fresh chicken breast was packaged under air and MAP condition (30% CO_2 + 70% N_2) and stored at 4 °C for up to 17 days (Balamatsia et al. 2006). During the storage period, putrescine and cadaverine levels increased linearly, while spermine and spermidine decreased in both air and MAP conditions. Putrescine, cadaverine and spermine levels were higher in air than MAP at the end of storage. On the

other hand, samples in MAP had higher tyramine and spermidine levels as compared to the ones in air.

Precooked chicken meat was packaged under air and MAP (30% CO_2 + 70% N_2) and stored at 4 °C for up to 23 days (Patsias et al. 2006). During the 23 days of storage, putrescine, cadaverine and tyramine levels increase in both air and MAP conditions. Putrescine and tyramine were higher the air-samples, while cadaverine and spermine were higher in the MAP-samples at the end of the storage period.

Thus, in these two similar studies discussed above, it was observed that the levels of putrescine and cadaverine increased during storage, and putrescine levels were higher in the samples stored in air. However, the level of cadaverine was higher in precooked chickens at the end of the storage period. In fresh chicken samples, both spermine and spermidine values decreased irrespective of packaging conditions. But, in precooked chicken samples, spermidine level increased in samples packaged with MAP. While the main polyamine formed in fresh chicken samples was putrescine and cadaverine, it was emphasized that spermine and spermidine concentrations were higher in precooked samples.

Pork loin were packaged under air, vacuum and MA (70% O_2 + 30% CO_2) and stored at 4 °C while being exposed to fluorescent light up to 20 days (Cayuela et al. 2004). Cholesterol level increased in all samples during storage, and this increase was higher (84%) in the MA followed by air (18%) and vacuum (13%).

11.4.3 Effect of MAP on Dairy Bioactives

Milk is an important food source for the nutrition of children and adults as well as newborns (Park 2009). Milk contains almost 5% lactose, 3.2% protein, 4% fat and 0.7% mineral salts (Séverin and Wenshui 2005; Shah 2000). Milk- based major bioactive compounds are lipids (triacylglycerides (TAG), saturated fatty acids (caproic acid, caprylic acid, capric acid, butyric acid, acetic acid, arachidic acid), unsaturated fatty acids (oleic acid, linoleic acid, linolenic acid), conjugated linoleic acid (CLA)), bioactive proteins (caseins (α, β, and κ), whey proteins (β-lactoglobulin, α-lactalbumin), Glycomacro-peptide), bioactive peptides (casomorphins, casokinins, casoxins, casoplatelins, α-Lactorphin, β-Lactorphin, lactoferroxins, immunopeptides, caseinophosphopeptides, phosphopeptides) and vitamins (Park 2009; Shah 2000; Park and Nam 2015; Fontecha et al. 2011). In addition to major bioactive compounds, milk and other dairy products contain minor bioactive compounds such as minor whey proteins (lactoferrin, lactoperoxidase, lysozyme, and immunoglobulins (A, M and G), proteose-peptones, serum albumin, osteopontin), minor lipids (gangliosides, glycolipids, glycosphingolipids, and cerebrosides), hormones, cytokines, oligosaccharides and nucleotides (Séverin and Wenshui 2005; Park 2009).

The bioactivity of milk components is classified into four main groups; (1) gastrointestinal development, activity and function; (2) infant development; (3) immunological development and function; and (4) microbial activity, including antibiotic and probiotic effects (Park and Nam 2015; Gobbetti 2007). These components in

milk have very important biological functions such as antimicrobial, antihypertensive, anticytotoxic, antithrombotic, antioxidative, opioid, anti-appetizing, immunomodulatory, mineral-binding and growth promoting activities (Park and Nam 2015). Dairy products derived from milk would also have these bioactive compounds and functionalities. Bioactive peptides resulting from hydrolysis of milk proteins by proteolytic enzymes of the starter bacteria are present in significant amounts in processed dairy products such as different types of cheese, yoghurt, sour milk, fermented milk such as kefir (Hafeez et al. 2014). Examples of bioactive components in yogurt are prebiotics such as β-glucans, inulin and fiber; bioactive lipid like conjugated linoleic acid; vitamins such as D, B_{12}, thiamine, riboflavin, niacin, folate and minerals such as calcium, phosphorus, magnesium, zinc (Ibeagha-Awemu et al. 2009). In many studies, it has been reported that yogurt is beneficial for human health as a bioactive by showing antioxidant and antidiabetic (Ejtahed et al. 2012), antihypertensive, antimicrobial and antithrombotic properties (FitzGerald and Murray 2006). Among dairy products, cheese has been an important part of the daily diet (Akuzawa et al. 2009). Kefir is particularly rich in calcium and magnesium, vitamin B_1, B_2, B_5, B_7, B_9, B_{12} and K. Although mare's milk is rich in vitamins A, C, E, B_1, B_2, B_7 and B_{12}, kumiss, a fermented mare's milk product, is richer in B_1, B_2 and B_{12} since lactic acid bacteria and yeasts stimulate their biosynthesis in koumiss (Lv and Wang 2009). It has been stated that kefir has bioactive properties thanks to its antimicrobial activity (Chifiriuc et al. 2011), anti-allergic and anti-inflammatory (Lee et al. 2007), anticarcinogenic and antimutagenic activity (Guzel-Seydim et al. 2006, 2011). MAP is used for dairy products for quality maintenance and shelf-life extension. A combination of elevated CO_2 and N_2 is often used with no low or no O_2 (0–5%) (Gunes and Kirkin 2012; Arvanitoyannis and Tziatzios 2012). Generally, CO_2 levels of 100, 90, 80, 70, 50, 40, and 20% balanced with N_2 are used in MAP of dairy products (Arvanitoyannis and Tziatzios 2012). However, 100%CO_2 usage is generally not recommended due to the possibility of package collapse (Arvanitoyannis and Tziatzios 2012). It has been reported that high CO_2 levels inhibit growth of yeasts and molds and administration of anaerobic MAP containing 70% or 30%CO_2 results in a longer shelf life of cheese compared to vacuum packages (Papaioannou et al. 2007; Gunes and Kirkin 2012). These gas combinations can also be applied to other dairy products such as yogurt, fermented milk drinks (Hotchkiss et al. 2006).

MAP applied to dairy products for shelf-life extension would also affect the bioactive compounds in the product. But, the majority of published research on MAP is on the microbiological properties and general quality parameters of dairy products, information on the effects of MAP on bioactives of dairy product is limited. Most of the modified atmosphere studies in dairy products were carried out in cheeses. Some studies on determining the influence of modified atmosphere packaging on milk bioactives are summarized in the Table 11.3 and discussed in the following paragraphs.

Cheddar cheese shreds were packaged under two different MA conditions (MAP1: 100% CO_2 or MAP2: 100% N_2) and stored at 4 °C under fluorescent light and in dark during 6 weeks (Colchin et al. 2001). It was determined that samples in

Table 11.3 Effects of MAP on the bioactive compounds of cheese

Dairy products	MAP conditions	Results	References
Cheddar cheese shreds	MAP1: 100% CO_2 MAP2: 100% N_2 Storage at 4 °C for 6 weeks	Higher total FFA in MAP1	Colchin et al. (2001)
Cameros cheeses	Vacuum, Air, MAP1: 20% CO_2 + 80% N_2 MAP2: 40% CO_2 + 60% N_2 MAP3: 50% CO_2 + 50% N_2 MAP4: 100% CO_2 + 0% N_2 Storage at 4 °C for 28 days	MAP samples had the lower FFA than air packaged samples FFA content of the samples in MA packages were not different	Gonzalez-Fandos et al. (2000)
Whey cheese 'Myzithra Kalathaki'	Vacuum, Air, MAP1: 20% CO_2 + 80% N_2 MAP2: 40% CO_2 + 60% N_2 MAP3: 60% CO_2 + 40% N_2 Storage at 4 °C for 45 days	MAP samples had the lower FFA than air-packaged samples Samples in MAP3 had the lowest degree of lipolysis and proteolysis	Dermiki et al. (2008)
Graviera Agraphon cheese	MAP: 50% CO_2 + 50% N_2 Storage at 4 °C for up to 60 days	During the first 30 days; increased total saturated fatty acids and decreased total monounsaturated, total polyunsaturated fatty acids and CLA During the last 30 days; the amount of all fatty acids remained stable	Fletouris et al. (2015)
Graviera Agraphon cheese	MAP: 50% CO_2 + 50% N_2 Air Storage at 4 °C and 10 °C for up to 85 days	Protein and fat contents in both treatments were not changed	Solomakos et al. (2019)
Kashar cheese	Air MAP1: 20% CO_2 + 80% N_2 MAP2: 40% CO_2 + 60% N_2 MAP3: 100% CO_2 + 0% N_2 Storage at 4 °C for up to 120 days	Caproic acid, capric acid, lauric acid and SFA levels of samples packaged in MAP2 were lower than others The highest medium-chain fatty acid in MAP1	Temiz (2010)

(continued)

Table 11.3 (continued)

Dairy products	MAP conditions	Results	References
Semihard cheese	MAP: 30% CO_2 + 70% N_2 Vacuum Storage at 4 °C for 90 days	The peptide profiles of cheeses were affected by packaging conditions Higher α_{S1}-casein f (24–32), α_{S1}-casein f (25–32) and β-casein (44–52) in vacuum Higher α_{S1}-casein f (1–16), β-casein f (46–52-32) and β-casein (74–82) in MAP	Sánchez-Rivera et al. (2013)

MAP1 had lower amount of alcohol and esters and higher levels of aldehydes and free fatty acids as compared to the ones in MAP2. Acetic acid and butanoic acid concentrations of samples in MAP1 (both light and dark conditions) were higher compared to the ones in MAP2 (both light and dark conditions). Octanoic acid and hexanoic acid were not detected in MAP2-samples, while both components were found at 0.003 μg/g level in MAP1.

Cameros cheeses were packaged under different MA conditions (vacuum, air (control), MAP1: 20% CO_2 + 80% N_2, MAP2: 40% CO_2 + 60% N_2, MAP3: 50% CO_2 + 50% N_2, MAP4: 100% CO_2 + 0% N_2) and stored at 4 °C for 28 days (Gonzalez-Fandos et al. 2000). At the end of the storage period, the highest FFA content was observed in the air-packaged, while MA packaged samples had the lowest FFA. There were no significant differences among the FFA content of the samples in MA packages.

Whey cheese 'Myzithra Kalathaki' was packaged in five different conditions (vacuum, MAP1: 20% CO_2 + 80% N_2, MAP2: 40% CO_2 + 60% N_2, MAP3: 60% CO_2 + 40% N_2 and air as control) before storage at 4 °C for 45 days (Dermiki et al. 2008). The results on effects of MA on FFA levels were similar to the ones obtained by Gonzalez-Fandos et al. (2000). A lower degree of proteolysis expressed as free aminoacids (α-lactalbumin, β-lactoglobulin and albumin) present in the sample was noted in the MA-packaged samples as compared to others. At the end of the 45th day, samples in MAP3 had the lowest degree of lipolysis and proteolysis. Thus, CO_2 seems to inhibit proteolysis and lipolysis in cheese during storage.

Graviera Agraphon cheese was packaged in MA (50% CO_2 + 50% N_2) and stored at 4 °C for up to 60 days (Fletouris et al. 2015). Until the 30th day of storage, total saturated fatty acids (SFA) increased, whereas total monounsaturated and total polyunsaturated fatty acids (PUFA) containing CLA decreased. It was determined that the amount of all fatty acids remained stable during subsequent storage periods. While the SFA concentration was the highest, PUFA concentration was the lowest among the fatty acids throughout storage. In another work, MAP (50% N2 + 50% CO2) did not affect protein and fat contents of Graviera Agraphon cheese during storage at 4 and 10 °C for 85 days (Solomakos et al. 2019).

Kashar cheese was packaged under four different MA conditions (air, and MAP1: 20% CO_2 + 80% N_2, MAP2: 40% CO_2 + 60% N_2, MAP3: 100% CO_2 + 0% N_2)

before being stored at 4 °C for 120 days (Temiz 2010). Caproic acid (C_6), caprylic acid (C_8), capric acid (C_{10}), lauric acid (C_{12}), myristic acid, linoleic acid, medium-chain fatty acid (MCFA) and SFA were greatly affected from packaging conditions. The C_6, C_{10}, C_{12} and SFA levels of samples packaged in MAP2 were lower as compared to samples in other gas mixtures. The highest MCFA was noted on the samples in MAP1.

A semi-hard cheese was packaged under MA (30% CO_2 + 70% N_2) and vacuum, and stored at 4 °C for up to 90 days (Sánchez-Rivera et al. 2013). It was observed that the peptide profiles of cheeses were different under both packaging conditions. After 90 days of storage, amounts of peptides such as α_{S1}-casein f (24–32) α_{S1}-casein f (25–32) and β-casein (44–52) were higher in vacuum packaged samples than those in MA-packaged ones. On the other hand, the amounts of α_{S1}-casein f (1–16), β-casein f (46–52-32) and β-casein (74–82) peptides were higher in the MA packaged cheeses. These peptides identified in cheese were known to have different bioactivity. For instance; the α_{S1}-casein f (25–32) peptide has moderate antihypertensive properties due to its moderate angiotensin-converting enzyme (ACE) inhibitory activity (Contreras et al. 2009). The peptide α_{S1}-casein f (24–32) has been shown to demonstrate both antimicrobial activity (Rizzello et al. 2005) and ACE-inhibitory activity (Ong et al. 2007). ACE inhibition can affect diverse regulatory systems such as regulation of blood pressure, immune defense and nervous system activity as well as its antihypertensive effect (Ong et al. 2007; Meisel 1998).

11.4.4 Effect of MAP on Cereal Bioactives

Economically important cereals such as wheat, corn, rice, oats, barley, rye, sorghum and millet have bioactive compounds such as phenolics, carotenoids, vitamin E, lignans, β-glucan, inulin, resistant starch, sterols, phytates, and minerals like Se, Cu, Zn and Mn (Zielinski et al. 2001; Bartłomiej et al. 2012; Donkor et al. 2012). Although the concentrations of these ingredients are small in cereal and cereal-based products such as bread, cakes, biscuits, pasta and dough, they are important in human nutrition because of their health benefits against free radical scavenging, minimizing peroxide formation, metal ion chelation, reducing the risk of diabetes, cardiovascular diseases, breast cancer and prostate cancer (Smuda et al. 2018; Broekaert et al. 2011; Zhou and Yu 2004).

MAP is commonly used in preservation of various cereal products. CO_2 is the most common gas used together with N_2 in MAP of bakery products to control quality degradations and extend shelf life. Although the most commonly used gas combination in cereal-based products is 60% CO_2 + 40% N_2, in some cases higher CO_2 levels in the anaerobic atmosphere are also used (Galić et al. 2009; Gunes and Kirkin 2012). MAP can also have consequences on bioactive compounds in the products, but limited information on this exists in the literature. Some studies dealing with the influence of MAP on cereal bioactives are summarized in Table 11.4 and discussed in the following paragraphs.

Table 11.4 Effects of MAP on the bioactive compounds of cereal products

Cereal products	MAP/CA conditions	Results	References
Wheat (Cappelli (*T. durum*) and Verna (*T. aestivum*))	CA: 98.5 ± 0.5% N_2 (v/v) Air (control) Storage for 18 months	CA reduced the loss of vitamin E in both cultivars during storage	Moncini et al. (2020)
Brown rice	Air, Vacuum, MAP1: 90% N_2 + 10% O_2 MAP2: 70% N_2 + 30% O_2 MAP3: 100% N_2 Storage at 37 °C for 180 days	Total phytosterol, flavonoid and phenolic content decreased during storage After storage of 180 days; total phytosterol contents were higher in MAP1, MAP3, and vacuum than air, while it was lower in MAP2 than air MAP1 significantly inhibited the degradation of phenolic compounds, followed by vacuum, MAP3, air and MAP2, respectively The decrease of total flavonoid contents in packaging of MAP1 and MAP3 was lower than those of other packaging	Huang et al. (2020)
Black and Red rice	CA1: 100% N_2, CA2: 5% O_2 + 95% N_2, CA3: 10% O_2 + 90% N_2 Storage for 4 months	During the storage, the total anthocyanin contents of both rice varieties were not changed by the CA treatments and were quite stable Total phenolic contents decreased during the storage and minimum losses of phenolic contents were observed in the rice samples from CA1 storage after 4-month storage Polyphenols were not different between both black and red rice stored at CA2 and CA3 After 4 months of storage; CA1 resulted in the highest antioxidant activity and minimum losses of free and soluble conjugated phenolic contents of both samples	Nyein et al. (2010)

Wheats (Verna and Cappelli) were stored under CA (98.5 ± 0.5% N_2) and traditional storage (control; a silo with the lid partially open) for up to 18 months (Moncini et al. 2020). The antiradical activities and polyphenol contents of both cultivars of wheat samples were not affected by storage conditions. The CA reduced loss of vitamin E in both cultivars during storage. The Cappelli wheat had higher α-tocopherol content compared to the Verna.

Brown rice was packaged in five different conditions (air, vacuum, MAP1: 90% N_2 + 10% O_2; MAP2: 70% N_2 + 30% O_2; MAP3: 100% N_2) and stored at 37 °C for 180 days (Huang et al. 2020). Total phytosterol, flavonoid and total phenolic contents of the samples decreased during storage. At the end of the storage period, the

total phytosterol contents of the samples in vacuum, MAP1 and MAP3 were 26%, 40% and 9% higher than air, respectively, while it was 33% lower in MAP2 as compared to air. It was reported that phytosterol content showed the highest rate of degradation in packages with high O_2 content. MAP1 significantly inhibited the degradation of phenolic compounds, followed by vacuum, MAP3, air and MAP2, respectively. At the end of the storage period, the degradation of phenolic compounds was prevented by 74% (air), 83% (vacuum), 84% (MAP1), 72% (MAP2) and 75% (MAP3). The decrease of total flavonoid contents in packaging of MAP1 and MAP3 was lower than those of other packaging. Total flavonoids deterioration was prevented by 32% (air), 34% (vacuum), 46% (MAP1), 32% (MAP2) and 45% (MAP3). The reduction of total flavonoids content in the packaging of MAP1 and MAP3 because of higher content of N_2. At the end of storage, the DPPH scavenging activity and FRAP of MAP1 packaging were higher than those of the control by 85% and 137%, respectively.

Black and red rice were stored under varying CA (CA1: 100% N_2, CA2: 5% O_2 + 95% N_2, CA3: 10% O_2 + 90% N_2) for 4 months (Nyein et al. 2010). The total anthocyanin contents of both rice varieties were not changed by the CA treatments and were quite stable during the storage. Total phenolic contents decreased during the storage, while soluble conjugated phenolics decreased throughout the first 2 months, and by stages enhanced subsequently. Polyphenols contents were not different between both black and red rice stored at CA2 and CA3. Storage at CA1 resulted in the highest soluble conjugated phenolic and total phenolic content in black rice, while it resulted it the highest free phenolic and lowest total phenolic and bound phenolic content in red rice. Radical scavenging activity was affected by storage time in both rice varieties, but not influenced by O_2 concentration. Antioxidant activities decreased slightly during the first month of storage but significantly increased afterwards. The highest antioxidant activities were observed in both samples stored at CA1.

11.5 Concluding Remarks

The content of bioactive compounds is affected by storage time, temperature, relative humidity (RH), product features and the gas composition in the environment (O_2, CO_2 etc.). MAP is applied to food products primarily to extend their shelf-life through inhibiting organoleptic quality degradations. MAP has also effects on bioactive components of the foods usually in a positive manner. Most of the studies evaluating the effect of MAP application on bioactive compounds were carried out in fresh fruits and vegetables. MAP generally resulted in increased levels of bioactive compounds in fresh produce through activation of secondary metabolisms. Studies in other food groups such as meat, dairy, and cereal products generally focus on microbial spoilage and general quality deterioration. For these product groups, more studies are required to determine the effects of MAP on their bioactive compounds.

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Part III
Influence of Novel Thermal Processes on Food Bioactive Compounds

Chapter 12
Influence of Microwave Heating on Food Bioactives

Maria Paciulli, Massimiliano Rinaldi, Antonella Cavazza, Frank Younce, and Emma Chiavaro

12.1 Introducing Microwave-Assisted Processing of Food: Fundamentals and Principles of the Technology

Microwaves are electromagnetic waves within a frequency band from 300 MHz to 300 GHz. In the electromagnetic spectrum they are inserted between the radio frequency range at lower frequencies as well as infrared and visible light at higher frequencies. Thus, microwaves belong to the non-ionizing radiations. The frequencies used for domestic and commercial heating are selected from the Industrial Scientific Medical (ISM) bands to avoid interference with other uses such as communications and radar. Commonly used frequencies for food heating are 896 MHz and 915 MHz for food manufacturing, and 2450 MHz which is used for both manufacturing and domestic home ovens (Venkatesh and Raghavan 2004). These waves can be reflected off, transmitted through, or absorbed into food materials. The electromagnetic waves consist of alternating magnetic and electric fields in planes perpendicular to the wave's direction of travel. Since food materials are not affected by a magnetic field the electric field provides the heating effect (Stuchly and Stuchly 1983). A material's ability to store electrical energy is known as the dielectric constant e' which is associated with transmission of the waves into the food. While the material's ability to convert the energy to heat is known as the dielectric loss factor e'' which is associated with absorption of the waves energy into the food. These

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electrical material properties are analogous to the behavior of the viscoelastic properties of the storage modulus g' and the loss modulus g'' in rheology.

At microwave frequencies an electric field can excite molecular motion in food components in the form of dipole rotation (dipole heating) and oscillatory movement of ions (ionic heating). Together these forms of heating are related to the food material's dielectric loss factors of dipole heating ε_d'' and ionic heating ε_σ'' respectively in the following equation (Eq. 12.1) (Ryynänen 1995):

$$\varepsilon'' = \varepsilon_d'' + \varepsilon_\sigma'' = \varepsilon_d'' + \frac{\sigma}{2\pi f \varepsilon_0} \tag{12.1}$$

where ε'' is the dielectric loss factor, ε_d'' is dipole heating, ε_σ'' is ionic heating, σ is electrical conductivity (S/m), f is frequency (Hz) and ε_o is the permittivity in free space (8.854×10^{-12} F/m).Unless a material is microwave transparent, the microwave energy is dissipated as the waves penetrate deeper into the food and are converted into heat along their path. And the instantaneous conversion of the absorbed microwave energy into heat is given by (Eq. 12.2):

$$Q = 2\pi\varepsilon_0\varepsilon'' f E^2 \tag{12.2}$$

where Q is the instantaneous heating (W/m^3) and E is the electric field (V/m).A commonly used measure of this is the penetration depth of waves entering perpendicularly into a plane surface of a semi-infinite body, where the power is reduced to $1/e$ (36.79%), can be predicted from the dielectric properties (Tang 2015) (Eq. 12.3).

$$P_\mathrm{d} = \frac{\lambda_0}{2\pi\sqrt{2\varepsilon'\left[\sqrt{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2}-1\right]}} \tag{12.3}$$

where P_d is penetration depth (m), λ_0 is wavelength in free space (m), ε' is the dielectric constant and ε'' is the dielectric loss factor.Microwave heating is a radiative process. A typical microwave heating system consists of several major components. The first is microwave source which traditionally is an oscillator tube called magnetron that converts electric energy into microwave energy. Alternatively more recently, with the advent of high power and frequency transistors, microwave energy may be obtained from a solid-state generator (Rao 2015). This energy maybe radiated directly into the second component, that is a closed chamber (resonant cavity) containing the product to be heated or via a waveguide that channels the microwaves from the magnetron to the chamber. The microwave power conveyed to the chamber may be measured with a directional coupler (a specially tuned antennae sensor) whose output signal can be used for generator power control and monitoring. The applicator cavity (oven chamber) is typically constructed of highly reflective metal walls to guide the waves in a particular pattern for single mode heating or

it is designed to reflect the waves back and forth as in multimode heating to maximizing the efficiency and uniformity of heating. In addition, there is usually a rotary wave deflector ("mode stirrer") near the entrance of a multimode cavity that helps distribute the radiation. Moreover, the product to be heated may be placed on a turntable to promote a more uniform distribution of heating in the multimode chamber (Rao 2015).

12.1.1 Effect of Product Formulation on Microwave Heating

Microwaves can be used to heat any dielectric material with a significant dielectric loss factor, as are most food products. The dielectric loss properties of foods are affected by their dipole molecules and free ions (e.g. moisture content and salt respectively). When microwaves impact a dielectric material, part of the energy is transmitted, part is reflected, and part is absorbed by the material where it is dissipated as heat. When an oscillating electrical field is applied to a material containing polar dielectric molecules, the dipoles attempt to align themselves with the polarity of the field. Due to the high frequency of the electric field, this realignment occurs millions of times per second and causes internal friction of molecules resulting in the conversion of the electromagnetic energy into heat and increasing the temperature of the food (Chandrasekaran et al. 2013). Due to the dipolar nature of water, the presence of moisture in a food product has a strong effect on dielectric heating. Dissociative ions in foods also produce heat, through ionic conduction; where the ions oscillate toward and away from the polarity of the electric field in the food creating friction which generates heat that conducts through the food. In liquid foods, this heat is also transferred through convection.

The heat generated is directly proportional to the frequency, the loss factor and the square of the field strength. The power 'lost' into the material (i.e. dissipated as heat) is related to the efficiency with which the electromagnetic radiation is converted to heat. A large loss factor indicates the food will readily heat with microwaves. The penetration depth is also important. It is inversely proportional to frequency. Deeper penetration is obtained at lower frequencies while higher frequencies result in more surface heating. The microwave frequency, dielectric constant and dielectric loss properties and their associated penetration depth into food are among the main factors which affect microwave heating and heat distribution (Raghavan et al. 2005).

The heat generated in a material is a function of the dielectric properties of the material. It is important to remember that dielectric properties of the food are dependent on temperature and microwave frequency, but also on the density, structure, composition, and moisture content of the food. Since water is the predominant component of biological materials, its content directly influences heating. At 2.45 GHz frequency, the electric field swings the orientation of water molecules 10^9 times every second creating an intense heat that can quickly increase of 10 °C per second. However, there are contributions from other factors: heating is accelerated by ionic

effect (mostly salt content) and the specific heat of the composite material. Specific heat is an important property in the thermal behavior of a food subjected to microwaves. Products with low specific heat may heat very rapidly, and even faster than water of the same weight. Thus, oil heats faster than water due to its much lower specific heat. Hence for oily materials, the influence of specific heat becomes the determining factor in microwave heating owing to low specific heat of oils which are often less than half that of water (Meda et al. 2017).

To predict product heating rates, penetration depths, and heating patterns the dielectric properties of food are measured and the effects of food composition, temperature are studied (Stuchly and Stuchly 1983). To minimize natural biological variation in food properties Guan et al. (2003) used reconstituted mashed potatoes as a uniform test bed. By varying moisture, salt and oil content of the mash; the effects of temperature and microwave frequency on the dielectric properties could be determined and used in electromagnetic and thermal coupled computer models. Moreover, with the addition of thermal chemical markers such test beds have been used as model foods to study and verify the uniformity of heating patterns in microwave applicator configurations (Kim and Taub 1993; Ramaswamy et al. 1996; Pandit et al. 2006). Wang et al. (2008) investigated the variation of dielectric properties in salmon fillets according to physical location on the fish and temperature for commonly used radio frequency (RF) and microwave frequencies. Coronel et al. (2008) measured the dielectric properties in dairy, puddings, and avocado products over a range of processing temperatures for use in designing microwave in tube continuous systems for pumpable foods. Tables of dielectric properties for a variety of foods at various temperatures and frequencies can be found in Nelson and Datta (2001), Stuchly and Stuchly (1983), and Tang (2015).

12.1.2 Advantages and Pitfalls of Microwave Processing

Advantages: Today's consumers have shown a strong preference for fresh and minimally processed products. Conventional thermal processes for microbial and enzyme inactivation for product safety and shelf life extension compromise the sensory and nutritional quality of the food (Tang 2015). The process heating time using a steam or water cook retort for pasteurization or commercial sterility can be significant, particularly for viscous and solid foods in large containers where heat is transferred primarily by conduction. This can result in an excessively long thermal exposure to the outer layers of a food while waiting for the cold spot in the core to reach the required microbial lethality.

Microwaves can deliver heat below the product surface in a volumetric manner, which can significantly reduce the heating time for thermal treatment of food products and thereby reduce the thermal degradation associated with quality losses (Datta and Hu 1992). Thus, microwave technology offers a higher retention of fresh-like attributes without the wait for conductive heat penetration. It also

provides the possibility of directed heating in multicomponent packages, potential energy savings and the reduced need for large fossil fueled boilers.

Controlled microwave cooking can be useful for foods that are heat sensitive and benefit from a shorter heating process such as pasta, fish, cheese formulations. Extreme cases for heating are tomatoes and beans. Beans by their nature require relatively long cook times to soften so a meaningful quality improvement may not be obtained by rapid microwave heating. Additionally, heat-sensitive products such as tomatoes which soften very quickly may not receive any discernable benefit from rapid heating. Between these extremes lies a wide range of conventional canned products whose quality can be significantly improved with shortened cooking times. Additionally, there are opportunities for new products that were not possible or practical due to their heat sensitivity to the long conventional retort heating times. Another benefit of microwave volumetric heating is a greater freedom to use thicker, more viscous sauces that have thermally conductive heating characteristics. Products such as canned macaroni and cheese and soups are generally formulated thinner than home-cooked to promote convective heating and/or in-package product stirring in a conventional agitating retort to minimize thermal damage. Another corresponding product development opportunity afforded by rapid volumetric microwave heating is the inclusion of delicate food components or utilizing flexible packages that could be damaged by the vigorous mechanical action in an agitating retort.

Pitfalls: Microwave inactivation of microbes is predominately from the ionic and dipole heating effects (Datta and Hu 1992) which in theory could be applied at very high heating rates. However, delivery of microwave energy has its challenges such as applying a microwave field which results in a predictable and consistent thermal distribution. The uniformity of treatment is dependent on a microwave source with consistent controlled power and frequency, the design of the microwave applicator, the geometry of the food product (and its package), and on the dielectric properties of the food itself which change with temperature (Ryynanen and Ohlsson 1996). Without consistent thermal treatment it is not practical to validate a rapid microwave heating process for food safety while maintaining product quality.

Multi-mode microwave heating of food is problematical for sterilization and pasteurization applications because of the unpredictable uniformity of the hot and cold spots within the food (Tang 2015). This can be compounded by thermal runaway where the warmed portion may preferentially heated due to the increase in the dielectric loss factor with temperature as is commonly seen in reheating frozen foods in domestic ovens. Attempts to improve multimode heating uniformity have focused on power control, applicator design, mode stirring, inclusion of additional microwave sources or applicator ports, package movement (turntable) and immersing the food package within pressure vessels of temperature-controlled air or liquid.

Single mode heating delivers the waves in a predictable pattern which can be applied to food in a consistent manner (Bows 2000). However, even with a carefully designed applicator, objects can still have multiple hot and cold spots depending on the foods dielectric properties which change with temperature, product and package geometry, and the uniformity of the electromagnetic field applied. Since the field pattern is consistent, the nonuniformity can be mitigated to achieve process

validation with slow heating rates, rest periods between applicators for thermal conduction and immersion in temperature-controlled fluids for control of edge and corner heating. Even so, the challenge remains for full implementation of rapid high-power heating rates with temperature uniformity.

Industrial microwave food processing systems commonly use high power microwave generators of 50 kW or more based on magnetrons traditionally supplied with high voltage direct current power supplies. Industrial magnetrons are relatively expensive, have limited life of about 2000–8000 h and slowly decay in performance over time. Recent use of high voltage switching power supplies has reduced the size of these generators by eliminating the need for the massive 3 phase transformers used in linear DC power supplies. Other advances in solid state technology have replaced the magnetron and its high voltage power supplies with an antenna (launcher) fed by combined banks of high-power transistors which parallels as similar technology transition by the radio and television broadcast industry. Although the efficiency of the early solid-state generators was significantly less than magnetron generators, the recent transition to GaN transistors has significantly improved overall efficiency of solid state generators to approach that of magnetron technology with a significant longer service life (Atuonwu and Tassou 2019; Trew 2002). Additionally, multiple solid-state generators can be readily applied to systems with multiple applicators which reduce the need for the splitters, tuners, circulators and other waveguide components associated with using a single high power magnetron based generator.

This situation will improve with the precise control capabilities of the solid state microwave sources and internet of things smart ovens where the product identity can be coupled to databases describing the product to be heated (type of food, geometry, dielectric and thermal properties) and the capability and performance of the oven/applicator (Yam 2000). For example, a product specific power/time control schedule for heating a retail entree based on product ID (from skew number, QR code, package RFID, etc.) could be downloaded from a food processor and domestic oven manufacture's collaborative cloud database and performed by the oven for the optimal desired cook. Alternatively, the food could be downloaded from the processors database to a smart oven where the optimal cooking process would be calculated using artificial intelligence. Proper product formulation and measurement of dielectric properties will play a large role in enabling this technology for new business opportunities. A similar, but more controlled situation, is also possible for industrial pasteurization and sterilization processes using solid state technology where the applicator characteristics can be optimized for specific product with a high degree of control or with the flexibility to produce a range of products. Such capabilities would be very useful for the small processor, with short runs of a variety of entrees, that desires flexibility and quick changeovers their manufacturing line(s).

12.2 Microwave Blanching

Blanching is an important step in the industrial processing of fruits and vegetables. It consists of a thermal process that is traditionally performed by immersing food materials in hot water, steam or boiling solutions containing acids or salts. Blanching is carried out before freezing, frying, drying, and canning. The main purpose of this process is to inactivate the enzymes that may cause changes in color, flavor, texture and nutritional properties. Additional benefits of blanching are the cleansing of the product, the decreasing of the initial microbial load, exhausting gas from the plant tissue, and the preheating before processing. Unfortunately, this operation has also some inconvenient effects such as losses in product quality (texture and turgor), leaching and degradation of nutritive components, such as sugars, minerals, and vitamins. Blanching process should assure enzyme inactivation while minimizing negative effects (Xiao et al. 2017). The use of microwaves for food processing has increased through the last decades, even if microwave blanching of fruits and vegetables is still limited. Some of the advantages compared with conventional heating methods include speed of operation, energy savings, easy to install and clean-up, precise process controls and faster start-up and shut-down times. No additional water is required in microwave blanching, leading to reduction or elimination of wastewater, and also to lower leaching of vitamins and other soluble nutrients (Xiao et al. 2017).

In this regard, Ramesh et al. (2002) compared the effect of the conventional water and microwave (2450 MHz) blanching at 95 ± 2 °C on vitamin C and carotenoids retention in spinach, carrot, and bell peppers. Vitamin C retention resulted in 68.85%, 84.72% and 82.62% retention in microwave blanched (MWB) spinach, bell pepper and carrots, respectively. Carotenoid retention in MWB carrots was 56.88%. The difference in the vitamin retention between vegetables treated with different methods showed significantly higher values for the microwave blanched in comparison to the water blanched ones. The authors related these results with the avoidance of leaching losses during processing and internal heat generation by microwaves. Severini et al. (2016) studied the effect of three blanching methods (microwave, boiling water, and steaming) on some functional properties of broccoli. The effectiveness of each blanching process, assessed by testing the peroxidase inactivation, was reported to be more rapid in microwaves and steam treatments (50 and 60 s, respectively) than in boiling water treatment (120 s). The increase of treatment time caused a vitamin C decrease in samples blanched by boiling water and steam; this trend was not observed in microwaved samples. The phenols content did not significantly vary depending both on type and time of treatment. Nguyen et al. (2019) explored the effects of microwave blanching conditions on the quality of green asparagus (*Asparagus officinalis L.*) butt segments, a rich source of fiber and antioxidants, often discarded during processing. The experiments were designed varying blanching time (2, 4, 6, and 8 min) and microwave power output (150, 300, 450, and 600 W). Phenolics, and free-radical scavenging activity retention were evaluated. The results showed that longer blanching time or higher microwave

power was associated with reduced quality of green asparagus butt segment. Besides, the appropriate parameters for microwave blanching of the green asparagus butt segment was found at 300 W for 4 min.

Remaining in the field of food by-products, Dibanda et al. (2020) studied the effect of microwave-blanching (720 W for 1, 3 and 5 min) on antioxidant activity, phenolic content and composition of mango, apple, orange, and banana peelings. Generally, increased microwave blanching was accompanied by a significant increase of total phenolics content. Increased antioxidant activities at 3 and 5 min of blanching varied with the type of fruit peelings. Epicatechin, ferulic acid, caffeic acid, rosmarinic acid, p-coumaric acid and gallic acid were identified in fruit peelings. Generally, microwave blanching for 3 or 5 min was found to preserve antioxidant activities.

Xanthakis and co-workers (Xanthakis et al. 2018) studied the effect of microwave assisted and conventional water blanching on ascorbic acid oxidase (AAO) inactivation and vitamin C retention on mango pieces. They studied two different blanching scenarios: high temperature short time (HTST) and low temperature long time (LTLT). For water blanching (WB) the samples were submerged in water bath at 90 °C for 5 min and at 70 °C for 12 min for HTST and LTLT, respectively. For microwave blanching (MWB), the process conditions were: for HTST, 5 min at power of 120 W–14.39 W/cm^3; for LTLT, 12 min at power of 100 W–11.99 W/cm^3. Regarding the enzymatic inactivation, WB-LTLT showed >65% AAO remaining activity, which resulted to be statistically higher than that obtained with WB-HTST and both MW blanching methods (~40%). MWB led to higher retention of total vitamin C in both LTLT (90.3 ± 4.1%) and HTST (91.5 ± 4.4%) treatments, in comparison to WB that resulted in 76.4 ± 2.8% and 84.7 ± 2.9% retention, for LTLT and HTST, respectively. The authors also observed a further inactivation of the thermostable fraction of AAO and degradation of total vitamin C after frozen storage for 130 days at −18.63 ± 0.48 °C. Minimal differences among the treatments were observed in terms of Vitamin C degradation, while AAO inactivation was reported to be higher for WB-HTST and MWB-LTLT compared to the other two conditions.

The comparison among WB and MWB was also explored by Wang et al. (2017) on red bell peppers, in terms of red pigments, ascorbic acid and antioxidant capacity retention. Water blanching was performed at 80 °C for 1 and 2.5 min or 90 °C for 1 min whereas MWB was performed at 650, 750 and 900 W for 100 s. WB treated peppers obtained the least retention of red pigments and ascorbic acid, with retentions of 62.66% and 27.29% respectively, compared to control samples. MWB better retained the bioactive compounds; no differences were observed with the control sample when low intensities were applied, slight ascorbic acid loss was observed with increasing MWB intensity. Regarding the antioxidant capacity, WB led to a reduction in comparison to the control sample, however MWB enhanced the antioxidant activity of the peppers, proportionally to the applied intensity. This result is closely associated with the high retention of bioactive compounds and also to the fact that thermal blanching can destroy the cell walls of vegetables and facilitate the antioxidants release (e.g., phenolic compounds). Blanching of red bell peppers was also studied by Jeevitha et al. (2013), using infrared (IR) and microwave (MW)

radiations in comparison to conventional water and steam blanching. Processing conditions were standardized on the basis of the degree of enzyme inactivation (POD and PPO). Water and steam blanching were observed to require lower processing time (1.0 and 1.5 min, respectively) compared to IR (6.0 min) and MW blanching (3.0 min). However, microwave-blanched samples retained higher amounts of ascorbic acid (94.7%). Dry blanching with IR (15C) and MW (17.5 W/g) resulted in higher retention of β-carotene (103.2 and 118.6%, respectively) compared to water (60.3%) and steam blanching (88.3%). In another similar study involving blanching of peppers using microwaves, it was found that phenolic compounds were reduced from 9.6 to 7.6 mg/g and antioxidant activity was enhanced from 29 to 42 M Trolox/g with thermal microwave blanching. Changes in the content of phenolic compounds were confirmed using HPLC and the emergence of other phenol derivatives with enhanced antioxidant activity was detected in blanched samples (Dorantes-Alvarez et al. 2011).

12.3 Microwave and Microwave-Assisted Drying

Conventional air drying is one of the most frequently used operations for food dehydration. Significant color, structural and nutritional changes occur during air drying. Microwave drying (MD) is an alternative drying method, recently used in food industry and has been proven to reduce the drying time with improvements in the final quality (Krokida and Maroulis 1999). There are significant differences in the mechanisms of microwave and conventional drying processes; the temperature and moisture gradients are in the same direction in case of microwave heating unlike conventional heating, wherein significant moisture loss from the material against temperature gradient is pronounced (Pradeep et al. 2013).

Leusink et al. (2010) studied antioxidant retention of cranberries subjected to vacuum microwave drying and reported that drying cranberry fruit using this method resulted in a greater retention of anthocyanins (cyanidin-3-galactoside, cyanidin-3-arabinoside, peonidin-3-galactoside and peonidin-3-arabinoside) compared to air drying at 55 °C.

Si et al. (2016) applied microwave-assisted IR drying to raspberry and found that the combined technique preserved the quality of raspberry powders among the thermal drying techniques with better results compared to single drying methods. Therefore, combined drying techniques with relatively lower temperature and/or shorter time could be recommended for use in the drying industry.

Nawirska-Olszańska et al. (2017) studied the effects of microwave-vacuum drying on bioactive compounds of golden berry (*Physalis peruviana* L.) compared to traditional convective drying. Authors reported efficient results for microwave drying at 480 W and 4–10 kPa.

Saha et al. (2019) carried out a detailed study (from 180 to 900 W) on corncob microwave drying in order to experimentally obtain the parameters to be used in mathematical model. MD allowed also a significant increase of availability of

phenolic compounds due to higher product temperature, moisture removal intensity, and internal pressure that are responsible for the greater breakdown of polymerized complexes in MD samples, and the effect was reported to be more pronounced at higher MW powers. In addition to the enhanced accessibility of polyphenols, the highly emaciated structure observed in MD samples greatly enhanced the extractability of polyphenols. Finally, the highest antioxidant capacity was observed at 300 W.

Zielinska and Zielinska (2019) reported that microwave-vacuum drying significantly increased total phenolic (TP) content of cranberries, suggesting that microwave-vacuum drying preserves polyphenols more effectively than convective drying. Moreover, the increase in the TP content of cranberries subjected to microwave was attributed to the release of phenolic compounds bound to cellular structures. On the contrary, total flavonoid (TF) content of cranberries resulted deeply influenced by microwave power (Zielinska and Zielinska 2019): particularly, an increase in microwave power above 300 W led to a substantial decrease in TF content (from 8 to 96%). Authors concluded that microwave-vacuum drying of berries at low microwave power (100–300 W) produced fruits with the highest content of bioactive compounds as well as the highest levels of antioxidant activity and suggested this technique as an equivalent alternative of freeze drying. This consideration is in agreement with Saha et al. (2019).

Shi et al. (2019) reported similar results with solid-state fermented okara even if for this kind of product antioxidant capacity of MD samples was significantly lower compared to freeze drying while interestingly isoflavone content improved with microwave treatment and presented significantly higher values than freeze drying. Authors hypothesized that microwaves stimulate the conversion of isoflavone glycoside structure to an isoflavone aglycone structure without reaching excessive temperatures that will degrade isoflavone aglycone.

Confirm of previous observations was reported by Liu et al. (2019) on hawthorn slices: microwave at atmospheric pressure and at high power (480–800 W) resulted in lower total phenolic content and antioxidant capacity compared to hot air drying confirming the positive and necessary benefit of vacuum as well as that low power must be used.

More recently, Chen et al. (2020) successfully applied MD to saffron (*Crocus sativus* L.) with very promising results for this technology due to very short drying times. Particularly, authors determined retention of crocins, water-soluble carotenoids (cis and trans glucosyl esters of crocetin), after MD similar to freeze drying that is reported as the best drying method for preserving compounds of interest. Thus, the authors concluded that due to the high costs and energy consumption of freeze drying, MD may be the best method for preserving crocins in saffron.

Finally, Zielinska et al. (2018) proposed utilization of microwave as a pretreatment before osmotic dehydration to promote dehydration and decrease impregnation. However, for better preserving bioactive compounds, the authors suggested to use low power as in cranberries microwaved at 800 W, FRAP values were approximately 75% lower than those noted in the freeze-dried sample. On the contrary, however, cranberries subjected to microwave-vacuum pretreatment at 100 W were

characterized by a high content of phenolic compounds, high levels of antioxidant activity and high concentrations of anthocyanins and flavonoids, which contributed to the attractive color of the final product.

12.4 Microwave Thawing and Tempering of Foods

Stinco et al. (2013) compared three different thawing methods on ultrafrozen orange juice: room temperature, refrigeration temperature and microwave defrosting at maximum power (800 W) for 20 s. The authors reported a significant decrease in the provitamin A and carotenoids levels, as well as in the antioxidant activity relative to the fresh samples due to microwave thawing but interestingly, the bioaccessibility of bioactive carotenoids increased by the microwave thawing.

Similarly, Villarreal-García et al. (2015) studied broccoli microwave thawing procedure in comparison to boiling water thawing: also, in this case carotenoids decreased after microwaving but hydrosoluble compounds such as phenolic compounds, vitamin C, and glucosinolates were less affected by microwaving showed a moderate or no effect.

More recently, Xu et al. (2021) applied microwave thawing on frozen red radish and reported that microwave system had the highest thawing efficiency; however, thawed samples had the highest drip loss and the lowest retention of firmness and vitamin C value compared to air and ultrasound-assisted ones.

Finally, microwaves are very effective in improving tempering procedure with the most important application on meat blocks (James and James 2002). In addition, tempering operation could be enhanced by microwaves in combination with other technologies such as infrared heating as proposed by Seyhun et al. (2009) on potato puree. During tempering process, only few effects on bioactive molecules are expected and thus no data are available for comparison between microwave and traditional technologies.

12.5 Food Stabilization with Microwave

Present microwave food preservation technologies include industrial scale systems for products that are pre-sealed in packages before thermal treatment and pumpable slurries which are subsequently aseptically packaged after thermal treatment. Parallel improvements in packaging technology (Regier 2014) include microwave transparent materials with ranges of oxygen and water vapor transmission rates (OTR and WVTR) can provide retail products with a practical shelf life, the use of susceptors in for focusing the microwave field to minimize cold spots or provide desired browning (e.g. pizza crust) and controlled venting of steam during cooking. These packaging innovations also provide consumer convenience and improved quality when reheating an entrée in a domestic microwave or combination oven.

The heating rate from microwave power in an industrial system is limited by the uniformity of heating of the product and package in the applicator (Datta and Hu 1992). High microwave power levels can amplify the temperature differences between the hot and cold spots within the package due to product geometry and preferential heating from dielectric properties that change with temperature (thermal runaway). Preheating the package, operating at a moderate power level and use of successive multiple applicators with rest periods allows time for conductive heat transfer from any hot spots to colder portions of the food or to a water immersed surface of the package while still providing a faster overall heating rate to the coldest spot than conventional surface heating. Similarly, in tube stirring it can be used to reduce nonuniformity for viscous slurries heated in microwave tube applicators. For each type of system, the commercial development of solid-state microwave generators with precise control of frequency and power is expected to lead to further improvements in uniformity which will allow for even faster heating rates and lower preheat temperatures.

In general, the commercial process systems for microwave pasteurization and sterilization follow a sequence of preheating the product to a uniform temperature, microwave heating (sometimes accompanied with thermal surface heating), holding at a specific temperature for thermal treatment (lethality or enzyme inactivation) and cooling cycles which is similar to conventional heating technologies. The advantageous reduction in the overall process time comes from the rapid microwave heating. The shortened heating period for the cold spot necessitates a holding time for lethality which is analogous to that provided by the high temperature short time (HTST) pasteurization "holding tube" commonly used for low viscosity fluid foods such as milk or juice. The preheating step is required for establishing a uniform product entrance temperature and can be used to minimize any nonuniformity in microwave energy distribution (applicator) and conversion to heat in the product (dielectric loss factor) by reducing the required temperature rise in the heating step. The preheating, heating, holding and cooling steps may be applied in a batch or continuous process (Ahmed & Ramaswamy, 2004).

Early attempts at microwave thermal processing exposed the food in a multimode heating application chamber similar to a large domestic box oven fed with a 2450 or 896 or 915 MHz source. Due to the semi random nature of multimode heating, the heating patterns in food in a basic simple box applicator are inconsistent. Attempts to mitigate the nonuniformity of multimode heating have included combinations of optimizing the applicator geometry, adding more microwave sources (ports) in various polarities, placing multiple microwave emitters (magnetrons) directly in the application cavity, using mechanical mode stirring devices in the cavity to more evenly disperse the field, rotating or moving the food package, providing the cavity with temperature controlled circulated air or immersion of the food package in water, the use of successive application cavities and pulsed heating and holding cycles (Lee et al. 2002) with varying degrees of success.

More recently the research has shifted towards single mode heating which produces a consistent predictable heating pattern in the food. Although the single mode heating pattern may not be fully uniform, the application can be modified and

controlled by utilizing many of the techniques mentioned for multimode heating to produce a repeatable thermal profile that can meet the requirements of a thermal process validation. Advances in dielectric property measurement (Uan et al. 2004), thermal and electromagnetic coupled finite element and finite difference time domain models, fiber optic temperature sensing, shielded data loggers, thermal chemical markers in model foods (Prakash et al. 1997) along with traditional inoculated package studies have created a framework for designing, commissioning and validating single mode heating processes for commercial use.

12.5.1 Microwave Pasteurization

Pasteurization provides microbial safety and extends the shelf life of chilled foods. The growing demand for convenient high quality minimally processed meals has resulted in the growth of food distribution services featuring online ordering and overnight delivery of fully or partially cooked entrées or measured portions of raw ingredients as home cook kits. Chilled and frozen foods shipped with gel packs can be vulnerable to temperature abuse and logistical shipping delays to residences. Once received, the microbial risk from a compromised shipment with high microbial load may not be readily apparent to the consumer who, despite the instructions and warnings on the manufacture's label, may or may not thoroughly cook the food prior to consumption. Application of a pasteurization step for microbial reduction in the manufacturing process helps reduce this risk. Microwave pasteurization offers shortened thermal processing times which when properly applied can meet guidelines (Food and Drug Administration 2020) while retaining the quality attributes associated with high value fresh like meals (Tang et al., 2018).

The Microwave Assisted Pasteurization System (MAPS) developed at Washington State University (WSU) utilizes single mode applicators for prepackaged foods heated from two sides while immersed in circulating hot water at or above the desired processing temperature (Tang 2015). Treatment at 915 MHz was selected to provide a longer wavelength for greater penetration depth into the food. This continuous system is a lower temperature version of the WSU Microwave Assisted Thermal Sterilizations system (MATS) with zones of preheating, microwave heating, holding for the required lethality, and cooling. However, since the selected range of pasteurization temperatures are below boiling, little or no overpressure is required to protect the package integrity, the entire process may be performed in an open bath/tunnel without a pressure vessel. The patented single mode heating in water geometry provides a repeatable consistent heating pattern which has been validated by a combination of electromagnetic and thermal computer models, chemical makers, miniature temperature loggers measuring the heat penetration of the cold spot and inoculated packaging studies for a variety of products (Wang et al. 2018).

Researchers at North Carolina State University developed an in-tube continuous-flow microwave heating system which can be used with aseptic filling technology to

produce pasteurized and sterilized products (Coronel et al. 2005). The system consists of specially designed applicators that focus the microwave energy into a polytetrafluoroethylene (PTFE) tube. Uniformity of the heat treatment can be enhanced by including mixers between applicators (Kumar et al. 2008). Commercialized systems are available to processing a variety of pumpable foods including vegetable purees (e.g. sweet potato), milk and juices from Industrial Microwave Systems of Garner, North Carolina USA and MicroThermics Raleigh North Carolina USA.

Another commercialized innovation for pasteurization is steam-vent package systems known as MICVAC (Regier 2014; Haamer and MicVac 2005). Microwave energy is used to heat the food and the moisture in the package with moisture to create a controlled steam environment for products such as loose vegetables with exposed surfaces. A specially designed valve on the package allows for elevated steam pressure inside without compromising the package integrity. This results in rapid heating and uniform steam temperature exposure to the product surfaces inside the package. Upon cooling the valve closes securely to prevent post process contamination.

12.5.2 Microwave Sterilization

Microwave sterilization systems were developed to improve the quality of traditional shelf stable canned products. Early attempts at microwave sterilization in the 1970s consisted of food conveyed through plastic tunnels which passed through a microwave cavity (Tang 2015). Since the food was surrounded with air, which was pressurized to prevent package bursting, the product quality suffered from edge heating. Another system patented by Lennart Stenstrom of Alfa Laval in 1974 was the Multitherm microwave sterilization process which included 2450 MHz microwave heating of packages successfully reduced the edge heating by immersing in hot water, however but the system was not widely commercialized (Brody 2012).

The Officine Meccaniche Attrezzature per Ceramiche (OMAC) systems of Italy produced over 200 microwave pasteurization and sterilization systems distributed worldwide with production rates of two tons of food per hour (Stanley and Petersen 2017). The OMAC system consists of 2450 MHz multimode microwave heating zones followed by holding and cooling zones. Packages of food progress through the zones in batches while surrounded with compressed air which is temperature controlled for the respective zone (Tang 2015). Although OMAC has ceased manufacturing these systems, some of them are still producing food products at companies such as Tops Foods of Olen Belgium.

The Microwave Assisted Thermal Sterilizations System (MATS) developed at Washington State University (WSU), which is a pressurized version of the MAPS described earlier, also utilizes single mode 915 MHz microwave applicators for package heating from two sides with immersion in circulating hot water at or above the desired processing temperature with added overpressure for commercial

sterilization (Tang et al. 2006). The combination heating provides conventional convection heating of the outside surface of the packages with overpressure hot water while simultaneously applying microwave energy to the top and bottom of a package for faster heating of the package core which can reduce the overall cook time for the desired lethality (heating and holding) by more than 50% when compared to typical standard steam or water cook retort process for low acid canned foods in a similar volume container (Tang et al. 2008).

The WSU team submitted the first ever FDA accepted filing for a microwave sterilization process in the United States for product in a 295 mL (10 oz) tray in October of 2009. This was followed by second accepted FDA filing for salmon fillets in a sauce packed in a pouch in 2011 and a letter of nonobjection from the USDA FSIS for a chicken and dumpling product. These processes were validated by electromagnetic and thermal computer models of the applicator coupled with the thermal and dielectric properties of the food, packaging and surrounding water, the use of thermal chemical markers to measure heating uniformity (Lau et al. 2003), measurement of heating temperature profiles with miniature data loggers, and inoculated pack studies (Guan et al. 2003). The MATS and MAPS systems are commercialized through 915 Labs of Denver, Colorado USA in partnership with Tata Industries of India.

A similar system known as Coaxially induced Microwave Pasteurization and Sterilization (CiMPAS) manufactured by Meyer Burger GMbH (Hohenstein-Ernstthl, Germany), passes food packages on a conveyor through a 915 MHz coaxial antenna array immersed in water in an overpressure vessel. Soni et al. (2020) utilized CiMPAS to study the use of tiny spore pouches embedded in mashed potatoes in a tray to evaluate sterilization effectiveness. The CiMPAS was used in a batch format with packages passing back and forth through the coaxial microwave array in overpressure temperature-controlled water at 65 °C and 121 °C to evaluate the systems effectiveness for pasteurization and sterilization respectively. A chemical marker method (Bornhorst et al. 2017; Martins et al. 2000) was used for determining the heating uniformity and cold spots within individual trays and among trays processed on a rack simultaneously.

The effects of microwave pasteurization or sterilization on bioactive substances and antioxidant activity is the object of several studies (Guo et al. 2017). Applying microwave sterilization to aronia juice, Piasek et al. (2011) found that the loss of total anthocyanins ranged from 39.7 to 59.1%, which was lower than those (66.1–99.8%) treated by thermal processing at 100 °C. In another work, microwave heating (90 °C, 10 s) was found to be less destructive compared to conventional heating (90 °C for 15 min) for strawberry purée (Marszałek et al. 2015). Microwave treated samples showed lower losses of total content of polyphenols (5.7%), total content of anthocyanins (19.2%) and vitamin C (3.4%), than those treated conventionally (14.0%, 60.2% and 61.7%, respectively) (Marszałek et al. 2015). In both these studies the phenomenon was justified with the lower microwave exposure time (7 s for aronia, 10 s for strawberry purée) than thermal processing time (1–5 h for aronia, 15 min for strawberry purée) (Marszałek et al. 2015; Piasek et al. 2011). In addition, Lu et al. (2011) found that the losses of ascorbic acid content and

lycopene content of grape tomato after microwave heating were less than 6.83% and 13.52%, respectively. Based on these results, it is possible to asses that microwave sterilization has low effects on bioactive substances and antioxidant activity of foods.

12.6 Microwave Cooking

Cooking is among the major applications of microwave. Cooking can cause partial or total loss of valuable nutrients depending on the process parameters. Research has demonstrated that many food vitamins are thermolabile and leach out during thermal processing. Over the years, microwave processing has demonstrated the advantage over traditional processing to reduce nutrient losses. However, microwave cooking process presents controversial results in the literature due to the different conditions that are employed (time, power, and added water) (Guo et al. 2017).

In this regard, studying broccoli, Zhang and Hamauzu (2004) pointed out that both microwaving (600 W) and conventional boiling, applied for up to 300 s, affected at the same way the antioxidant composition (phenols, ascorbic acid, carotenoids) and *in vitro* activity. On the other hand, Turkmen et al. (2005) established that after microwaving (1000 W; 1.5 min) and conventional boiling (5 min), the total antioxidant activity remained unchanged in broccoli, despite the total phenolic content was even found to be enhanced for the microwaved samples. Another study (Song et al. 2007) has found that the glucosinolate content of some Brassica vegetables, including broccoli, was maintained after steaming (5–20 min), microwave (900 W, 30–180 s), and stir-fry (3, 5 min) cooking processes, but was affected by the boiling (5–30 min) process due to the glucosinolates leaching into the cooking water.

Akdas and Bakkalbasi (2017) reported that microwave treatment without water was suitable for cooking kale as the retention of ascorbic acid, total carotenoids, and total chlorophylls of kale was 89.4%, 99.8%, 44.7%, respectively. Tian et al. (2016) observed losses of total phenolics (negligible), total anthocyanin (14.01%) and chlorogenic acid (20.01%) after microwaving purple-fleshed potatoes without water were much lower than stir-frying, baking, air-frying and frying. Xu et al. (2014) reported that there was negligible change in total phenolic content and no significant loss of vitamin C of red cabbage (adding 10 mL water to 300 g sample) after microwave heating, whereas obvious changes were observed on losses after stir-frying and boiling. The retention of bioactive components of vegetables processed by microwave is higher than those processed by other cooking methods because of shorter heating time, and without the pretreatment of soaking (Tian et al. 2016; Xu et al. 2014). Therefore, microwave cooking should be performed without water or with a small amount of water for retaining antioxidant activity and bioactive components. However, when massive water is added to foods during microwave heating, the retention of nutrients drops greatly. Dolinsky et al. (2016) found that microwave cooking with water was not recommended to cook selected vegetables owing to significant reduction in the contents of polyphenols (soluble polyphenol and hydrolysable polyphenol) of kale (reduction of 23.4%), tomato (reduction of 21.9%), and

green beans (reduction of 22.9%), while steaming with little osmotic exchange was more suitable for maintaining higher levels of their polyphenol concentrations. This observation was similar to that reported by de Lima et al. (2017), who showed that cassava (adding 500 g water to 500 g sample) treated by steaming retained more phenolic compounds (retention of 236.1%) and antioxidant activity (retention of 308.6%) compared to microwave treatment (retention of 164.4% of phenolic and 273.4% of antioxidant activity). For bioactive substances of vegetables, microwave cooking increased the retention rate to varying degrees because water may cause softening and rupture of the lignocellulosic structure, enabling those soluble bioactive substances to be released from the food matrix, while increasing the loss owing to leaching and thermal liability. Barba et al., (2008) investigated the change in phenolic constituents during microwave baking of cv. Agria potatoes at various microwave power levels (300–1000 W; 95–420″). The baking time was found to increase with a decrease in the power level and at the same time a decrease in water losses was observed due to a slow heating rate. By reducing water loss, the thermal damages of nutritional components were avoided due to its high thermal capacity. It was found that the phenolic compound was retained at a good level when the potato samples were cooked at 500 W. Moreover, Liu et al. (2019) studied the effect of steaming, high-pressure cooking, and microwave cooking on thiamine, riboflavin, phytic acid (PA), and mineral contents (Mg, Ca, Mn, Zn, and Fe) of different cultivars rice. Cooking decreased the vitamin B and PA contents, and high-pressure cooking exerted more remarkable effects than those of steaming and microwave cooking. Moreover, cooking improved the bioaccessibility of Mg, Fe, and Ca, but decreased those of Zn and Mn.

In case of animal-based products, Nishioka et al. (2011) studied the loss of vitamin B12 in fish (round herring) meats during various cooking treatments. The B12 content was significantly decreased up to 59, 47, 41, 43 and 59% during cooking by grilling (7.5 min), boiling (5 min), steaming (9 min), frying (4 min) microwaving (1 min, 500 W), but not at all during vacuum-packed pouch cooking. The effects of microwave heating on the retention of selected nutritional components in animal muscle was studied by Uherova et al. (1993). The degree of retention of the thermolabile vitamin B6 and thiamine after thermal treatment of pork and chicken meat was determined in a conventional and in two microwave ovens. In conventionally roasted samples 48–96% of thiamine was retained, whereas microwave-treated samples showed retention as high as 85.6–94.2% and 88–96% for the two microwave ovens, respectively. After conventional roasting, meat samples retained only 21.6–48.5% of vitamin B6. Microwave treatment, on the other hand, retained 59.9–80.9% and 64.2–86.8% of vitamin B6, for the two ovens respectively. The measured values of vitamin retention demonstrate clearly that heating of muscle tissue with microwaves is less destructive to heat-sensitive vitamins compared to conventional roasting.

12.7 Microwave-Assisted Extraction of Bioactive Compounds

Nowadays the extraction of active molecules from natural products is a quite common procedure linked to the important strategy to replace the use of synthetic compounds in many products. Natural extracts are appreciated by consumers, and are gaining a strong success, in the fields of coloring agents, antioxidants, and antimicrobial compounds used to preserve food products, cosmetics, and detergents, from degradation or alteration (Delgado et al., 2019). Similarly, the development of innovative systems of active packaging take advantage of the use of natural bioactive molecules for proposing a wide variety of devices made by biodegradable ingredients. Furthermore, interesting progresses concerning food ingredients or supplements, and active principles used in the field of nutraceuticals, herbal, and functional foods, are in progress.

An interesting source of such compounds is constituted by agroindustrial byproducts, lately being object of many studies and researches. Despite a very low economic value, they contain high amounts of useful molecules that can be re-introduced in the productive cycle following the guidelines developed in the context of the circular economy (Zuluaga et al. 2020; Elik et al. 2020; Littardi et al. 2021). Moreover, often, the non-edible parts of fruits and vegetables may contain even a higher amount of bioactive compounds than edible portions (Lourenço et al. 2019).

Since phytochemicals are embedded in a complex matrix, the quality of a natural extracts strongly depends on the technology that is applied to separate the active molecules from interfering substances. Besides, in most cases, it is mandatory to take into account the necessity of preserving the active compounds from deterioration, since many compounds are sensitive to thermal treatment, light exposure, and oxygen contact that can lead to oxidation and degradation. Therefore, for example in the case of phenolics, their final yield strongly depends on the process to which the matrix is submitted (Chan et al. 2009).

In the late years, there is a strong attention to green and environment-friendly systems, and a growing interest to reduce the use of organic solvents and energy consumption. For this reason, conventional extraction techniques are often being replaced with new technological processes such as supercritical fluid extraction, ultrasound-assisted extraction, and microwave-assisted extraction (Lourenço et al. 2019). All of them permit to reduce the impact on the environment thanks to a limited amount of solvent employed, and to a reduced time needed.

In particular, the use of microwaves in the extraction procedure can be considered a quite modern and simple technique that combines green and economic features: it can in fact requires a low cost equipment, permitting to achieve good results with reduced operating time. Besides, it limits energy consumption and amount of solvent needed. The reason for MAE efficiency is founded on the enhancement of the effects of the solvent placed in contact with the solid matrix, since the technology applied allows an increase in the diffusion of soluble compounds to the solvent. Complete reviews underlining the progresses achieved with this technique, and

showing a very high number of applications in different fields have been written in the late years (Mirzadeh et al. 2020; Kala et al. 2016; Bagade and Patil 2019; Sadeghi et al. 2017).

12.7.1 Basic Principles

The mechanism of extraction of MAE is based on the use of a source of energy that is rapidly transmitted to polar molecules leading to ionic conduction, dipole rotation and rapid diffusion, and determining a quick heating (Ibrahim and Zaini 2017). Moisture represents the target for microwaves, that act exploiting the presence of small traces of water occurring even in dried plant. The energy transmitted to water generates high temperature determining its evaporation. The primary consequence is a considerable increase of the pressure on the surrounding tissues and the cell walls, ensuring a homogeneous and efficient heating and heat diffusion through the matrix. Therefore, a swelling of cells membranes and cytoplasm occurs, promoting the release of the molecules of interest to the liquid medium (Ameer et al. 2017; Catena et al. 2020).

MAE has been demonstrated to be very efficient when polar solvents such as water, methanol, and ethanol are used. The use of water is able to accelerate the extraction process, with a significant reduction of organic solvent consumption thanks to the increase of the solubility of natural compounds such as in the case of polyphenols, and an action on cell wall components that enhance its permeability (Spigno and Faveri 2009). On the other hand, advantage of the use of MAE is limited when volatile media are used (Wang and Weller 2006).

Jain et al. (2009) focused their attention on the importance of some intrinsic solvent properties that affect the final behavior: for example, the solvent dielectric constant affects the ability to absorb microwave energy, the dielectric loss has a consistent impact on the efficiency of converting microwave energy into heat, and the dissipation factor is relevant for the duration effect.

Non-polar solvents, such as hexane and petroleum ether are not effective since they cannot interact with microwaves, and therefore are not able to result in any thermal effect (Alfaro et al. 2003). For this reason, when apolar solvents are needed, the addition of small amounts of a polar component, such as ethanol, can be performed. For example, 5% of ethanol in hexane has been shown to promote solvent heating and high efficacy in the extraction of castor oil from seeds with higher yield in a shorter time compared to that of Soxhlet extraction (Ibrahim and Zaini 2017).

An interesting option in MAE is the possibility of using sealed vessels placed in a controlled temperature and pressure conditions, which allow the solvent to reach temperature values above the boiling point. Under these conditions, extraction occurs with even higher efficiency (Ballard et al. 2010; Kaufmann and Christen 2002).

12.7.2 Parameters Affecting Extraction Yield and Condition Optimization

In all applications concerning the extraction of compounds from a matrix, independently from the selected technology, it is necessary to set up the optimum conditions to maximize the extraction yield. A clear knowledge of the parameters involved, and their relative interactions are not easy to achieve, and requires a deep investigation.

In fact, as it is well known that many parameters influence the efficiency of the extraction of active compounds from plant material (Bagade and Patil 2019). It is important to take into account, besides the chemical nature, also the part of plant where the substances of interest are located (cell walls, or vacuoles, etc.), and the eventuality that components are bound to plant material that can act in several manners affecting solubility and diffusibility of the molecules. Furthermore, the size of the raw material particles, the storage conditions, the potential presence of interfering substance, and the possible occurrence of biochemical or chemical reactions can be of significant importance (Suwal and Marciniak 2018).

In the setup of experimental conditions, the main factors that can be adjusted when performing a MAE extraction are the following, as reviewed by Ameer et al. (2017):

Microwave power: the intensity of the energy directed towards the material is expected to lead to an increase in the final yield. However, it has been observed that a too high energy value can affect the stability of the extracted components, probably since the high temperature value reached can degrade thermosensitive molecules.

Extraction time: longer exposure should have a positive effect on yield, hoeverut in the case of MAE it has to be taken into account that can also rise temperature at too high levels; therefore, to avoid reaching critical values, it is generally suggested to repeat several brief cycles of irradiation instead of prolonging a single treatment.

Plant matrix condition: the properties of the material considered are very important: a high moisture favors the extraction, and a pretreatment of the matrix with the solvent before the extraction step, has been reported to increase the final efficiency.

Type of vessels: a system based on the use of closed vessel can be applied to limited amount of samples, although it shows advantages for the extraction of volatile substances, and requires less amount of solvent. On the other hand, the high pressure achieved inside the container poses some safety risks and requires a cooling step after the treatment. The open-vessel system gives the possibility to add or remove solvents and/or reagents during the process, and allows the processing of large amount of samples; however, it has been reported to achieve less performance concerning precision.

***Choice of the solvent*:** an accurate selection of the proper solvent is very important for achieving an optimal extraction in MAE. The choice obviously depends on the specific chemico-physical features and the solubility of the target compounds. However, the interaction between solvent and plant matrix, and the behavior of the solvent submitted to microwaves have to be taken into account. The use of combination of water and an organic solvent seems to be the best solution to increase

extraction efficiency, compared to the use of a single organic solvent. For example, hexane can be in many cases a good extraction solvent but is not a good microwave absorber. On the other hand, ethanol is a good microwave absorber, therefore, the addition of small amount of it to hexane improves the resulting dielectric behavior. However, excessive amount of ethanol can lead to a too aggressive heating.

Several examples from literature underlined the advantage of MAE over traditional technologies, describing better results in terms of yield and time compared to Soxhlet extraction, maceration, and reflux extraction (Martino et al. 2006; Pan et al. 2003; Rafiee et al. 2011). Use of MAE has been reported to reduce the required time with respect to traditional systems. For example, Pan et al. (2003) reported that, in a process of only 4 min of radiation, higher results for polyphenols and caffeine extraction from green tea leaves were obtained respect to ultrasound-assisted extraction, heating reflux extraction, and extraction at room temperature. Similarly, Rafiee et al. (2011) compared results on the extraction of phenolic compounds from olive leaves by maceration and MAE, and found that the highest yield was achieved after 15 min of MAE, and was significantly superior to that at 24 h in maceration method.

However, in relation to innovative technologies, MAE was sometimes found to be less efficient, as example when compared to ultrasound-assisted extraction (Catena et al. 2020). Nevertheless, a definite general comparison cannot be achieved by considering data from single experiment sets, since results reported are generally limited to the specific case studied, and referred to a selected product.

Besides, for a punctual comparison between MAE and other extraction technology, the conditions selected for each method should be previously optimized. However, from the high number of parameters listed above, it does not appear to be easy to select the optimum conditions to achieve a maximum yield: a comprehensive study for the description of the multiple causes affecting the final effect should be performed. In fact, in addition to the number of variables involved, it is important to take into account that some of them are linked. Indeed, between many of them there are significant interactions, and these cannot be considered individually by following a "one variable at a time" approach.

The only strategy to follow in order to select the best combination of conditions, is to propose a multivariate approach, as clearly explained by the tutorial published by Leardi (2009). Such an optimization cannot be based on single experiments and requires a construction of an experimental design planned with a scientific approach. This methodology also allows to study a wide range of variability of the considered parameters, reducing the numbers of experiments. It also permits to gain a deeper knowledge, leading to an increase of the quality of information. Some examples can be found in literature for MAE (Zuluaga et al. 2020; Elik et al. 2020; Singh et al. 2011; Li et al. 2017) and other extraction technologies (Turrini et al. 2018), and can be of great help to enhance the knowledge about the comprehensive effect of the involved variables, constituting complex systems.

12.8 Conclusions and Future Perspectives

Microwaves find several uses in the food sector, presenting many advantages over traditional methods, regarding the retention of bioactive compounds. Volumetric heating, lower processing time and the lack of contact with the heating medium (hot water or steam) reduce the bioactive losses, due to thermal effects or leaching. However, in literature are reported experiments which investigate the behavior of one or two variables, generally time and power, without considering the global effects exerted by the interaction of several parameters. These data are not sufficient to draw proper conclusions. On this basis, we can suggest that the only way to evaluate the effect of the different variables on the final result is the application of a chemometric approach. This can also be of great help to investigate the possible differences in the pattern of extracted/retained compounds with respect to other methods, and in targeting the extraction/retention of a specific compound, as can be required for future industrial applications, as recently suggested (Kala et al. 2016).

Less information is available regarding the comparison of microwaves with other novel technologies. These comparisons may lead to a better understanding of the mechanisms involved and investigating some interesting matters such as the effect of the energy intensity on the integrity of bioactive substances. A rigorous scientific approach is therefore needed to find a right equilibrium between the recorded effects to reach a real optimization.

Another interesting prospect is also to study the combination of microwaves with different technologies, with the aim of implementing the results and promoting the industrial scale-up, as recently pointed out by Ekezie et al. (2017).

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Chapter 13
Influence of Ohmic Heating on Food Bioactives

Anne Kathrin Baier and Cornelia Rauh

13.1 Ohmic Heating: A Promising Alternative to Conventional Heating

Heating belongs to the most important and most common process steps in food manufacturing. It serves the extension of shelf-life by inactivation of undesired microorganisms and enzymes, the induction of textural and structural alterations via tissue softening, starch gelatinization or protein denaturation as well as the preparation for and efficiency enhancement of subsequent processing steps. Besides these desired effects, there may also be adverse effects. Sensorial quality may be decreased by the loss of aroma and flavor or nutritionally detrimental effects may occur via degradation and loss of nutrients (Varghese et al. 2014). Especially bioactive ingredients such as vitamins or polyphenols often possess high temperature sensitivity and thus are strongly affected by intense thermal processing. Conventional heating is realized via external heat generation and subsequent heat transfer to the product by conduction, convection or radiation (de Alwis and Fryer 1992; Goullieux and Pain 2005; Varghese et al. 2014). Conduction of heat is often a slow process leading to inhomogeneous temperature distributions in the product. This results in overprocessing in some parts of the product connected to higher quality losses (de Alwis and Fryer 1992). Thus, technologies are required that meet both the requirements for food safety or structure changes as well as sufficient product quality. Besides non-thermal technologies such as high isostatic pressure or pulsed electric fields, alternative heating methods are of interest achieving rapid and uniform heating of the product (Varghese et al. 2014).

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One of these methods is ohmic heating where an electric current is passed through a material with electrical resistance with the purpose of heating it (Vicente et al. 2006). Similar to other volumetric heating methods, e.g. microwave or inductive heating, heat is generated inside the food and therefore the dependency on thermal conduction and convection is low (Goullieux and Pain 2005). This leads to fewer temperature gradients in the product. The name ohmic heating is derived from the relation between current, voltage, and resistance known as Ohm's law (Icier 2012):

$$R = V / I \tag{13.1}$$

where R is the electrical resistance in ohms, V is the voltage applied in volts and I is the current in amps (Fellows 2009).In literature, the technology is also referred to as Joule heating, electric resistance heating, direct electric resistance heating, electro heating, and electro conductive heating (Bengston et al. 2006; Varghese et al. 2014; Chen 2015). The food material takes up the position of the resistance in the electric circuit (Icier 2012). Most foods contain sufficient water and ions from salts and acids to be electrical conductive and function thus as a resistance in the process. This enables an electric current to pass through it when a voltage gradient is applied (Halden et al. 1990; Icier 2012). The resistance of food material to the electrical current causes heating by converting electric energy into heat energy (Sastry 1992; Sastry and Barach 2000). In contrast to metals, in which a current is transported via movement of electrons, current flow in foods occurs due to the movement of ions. Depending on their net charge ions move within the electric field towards the electrode with opposite charge. Collision and friction between ions and other molecules during these movements lead to energy dissipation in form of heat. The amount of heat is directly related to the current flow caused by the voltage gradients in the food and the electrical conductivity of the food material (Sastry and Li 1996). Therefore, ohmic heating can be regarded as a technique for internal thermal energy generation and not as a method for thermal energy transfer (Knirsch et al. 2010).

This direct heating provides some advantages compared to conventional indirect heating methods. One requirement in food preservation is that each part of the food has been subjected to sufficient treatment intensity to reach the desired level of sterility. In conventional thermal processing, there is usually a marked time lag between the outer and inner parts of the material reaching critical temperature and experiencing sufficient thermal preservation. In consequence, a huge part of the food needs to be over-processed to guarantee complete pasteurization or sterilization and consider the whole product as safe (de Alwis and Fryer 1992). One main indication for good process design of conventional heating is therefore a narrow temperature and residence time distribution (Goullieux and Pain 2005). Uniform internal heat generation may avoid over-processing due to the absence of marked temperature gradients in the product leading to a more homogeneous treatment and a better preservation of temperature sensitive nutrients, color and aroma (Wang and Sastry 2002; Castro et al. 2004a, b; Icier and Ilicali 2005; Leizerson and Shimoni 2005a, b; Vikram et al. 2005). The absence of hot surfaces required for conventional heat transfer reduces

the risk of local overheating and food burning onto the equipment (Ayadi et al. 2005; Bengston et al. 2006; Fellows 2009; Sakr and Liu 2014). Beside fewer effects on product quality, this also brings reductions in costs for clean-up and maintenance of the equipment (Reznick 1996; Varghese et al. 2014). Target temperatures can be reached very fast with ohmic heating (Sastry 2005; Bengston et al. 2006; Fellows 2009) reducing the contribution of heating time on the thermal load of the product, which also helps to preserve nutritional and sensorial quality. Due to the homogeneity of heating, there is no need for intensive mixing, which protects shear-sensitive materials (Icier 2012). The energy conversion efficiency of ohmic heating is very high (Bengston et al. 2006; Icier 2012). Around 90% of the energy is converted to heat in the food (Fellows 2009), which might save energy and costs to the processors (Varghese et al. 2014) and can be considered as a green technology. The capital costs for the equipment are relatively low compared to other direct heating methods (Bengston et al. 2006; Varghese et al. 2014).

The technology is also suitable to heat mixtures of liquid and particles that are difficult to process with conventional methods (Varghese et al. 2014). If the two phases possess an identical electrical resistance, they can be heated homogeneously (de Alwis and Fryer 1992; Icier 2012) avoiding cold spots in the solid phase and reducing the need for overcrossing the liquid. Using ohmic heating, it is thus possible to use High Temperature Short Time (HTST) and Ultra-high Temperature (UHT) techniques on particulate food materials (Imai et al. 1995). These process designs are considered to be more gently for some heat sensitive compounds compared to longer heating at lower temperatures. In case of a high electrical conductivity of the solid phase it is even possible to obtain higher temperatures in the particles than in the liquid phase, which is impossible to achieve with conventional indirect heating (Chen 2015). Particles with diameters up to 2.5 cm may be treated (Bengston et al. 2006; Chen 2015). As the food should have sufficient fluidity to be pumped and treated continuously, particle concentration is limited to approximately 60% (Bengston et al. 2006; Fellows 2009; Chen 2015). In conventional aseptic processing the particle size is limited to 15 mm maximum and 30–40% particle concentration (Varghese et al. 2014). Ohmic heating is also feasible to evenly process high viscous foods (Fellows 2009; Sakr and Liu 2014) or to pasteurize protein-rich materials such as egg white and whey without inducing their thermal coagulation (Icier 2010; Icier and Bozkurt 2011).

13.2 Applications, Equipment and Process Parameters

First patents for the use of direct resistance heating were already filed in the nineteenth century and first industrial use was reported in the 1930s for milk pasteurization (Goullieux and Pain 2005; Bengston et al. 2006; Chen 2015). Due to insufficient process control and deficiencies in inert electrode materials, commercial usage was limited in the following decades. In the 1980s, research on ohmic heating reawakened with the demand for adequate sterilization techniques for foods containing

large particles (Bengston et al. 2006; Chen 2015). Since the FDA approval to process stable low acid food in 1993 commercial usage of ohmic heating took place in Europe, the USA and Japan along with improvements in process control and electrode materials (Goullieux and Pain 2005).

Ohmic heating can be applied in different areas of food processing including pasteurization and sterilization, blanching, cooking, evaporation and dehydration, thawing, extraction and fermentation (Goullieux and Pain 2005; Bengston et al. 2006; Icier 2012). Applications of ohmic heating are more determined by the type and characteristics of the food material, rather than the processing area. In food preservation, ohmic heating provides the aforementioned advantages in regard to heating rate and homogeneity, feasibility for certain product characteristics and preservation of food quality. Inactivation kinetics show similar results for ohmic and conventional heating in terms of D, z and activation energy value (Sastry and Barach 2000). Some authors also report additional benefits due to a higher inactivation rate of microorganism and a potential decrease in processing time (Pereira et al. 2007; Sun et al. 2008; Baysal and Icier 2010; Somavat et al. 2012a, b, 2013). Ohmic heating systems are suited to be adapted to aseptic food-processing lines (Kim et al. 1996) and are considered a useful addition for the hurdle concept (Sastry 2008). The technology may also bring advantages in the blanching of fruits and vegetables. Due to the ability to homogeneously treat larger pieces, there is no need for previous dicing. This reduces leaching of solutes during blanching due to the lower surface to volume ratio (Mizrahi 1996). Ohmic heating can as well be applied to improve thawing of foods with large sizes where conventional thawing is very time consuming. As the electrical conductivity markedly increases with phase transition and temperature, the temperature distribution of the food has to be controlled well to avoid over-processing in some parts of the product (Varghese et al. 2014). Enhanced mass transfer due to higher diffusion rates at elevated temperature and cell disintegration by thermal and electric effects can be used to improve the recovery of food compounds and the removal of water (Bengston et al. 2006). Wang and Chu (2003) found higher evaporation rates and improved results in quality analysis using ohmic heating during vacuum evaporation of orange juice. Ohmic heating is commercially used in the United States to process liquid egg and in the United Kingdom and Japan for processing of whole fruits (Bengston et al. 2006). Laboratory trials include applications on fruits and vegetables, juices, stews, meats, seafood, pasta and soups (Bengston et al. 2006). Ohmic heating is even investigated in terms of its suitability for heating and sterilizing food and waste during long term space missions (Somavat et al. 2012a, b).

The basic elements of an ohmic heating system are an AC power source to deliver electrical energy to the system, a variable power supply to adjust the desired voltage, a treatment cell as well as measurement units to control voltage, current and temperature (Bengston et al. 2006; Icier 2012). An illustration of a pilot scale system is given in Fig. 13.1. Ohmic heaters can be designed for discontinuous or continuous processing. The typical batch treatment cell consists of a cuboid or cylindrical box with two plate electrodes at opposite sides (Chen 2015). The

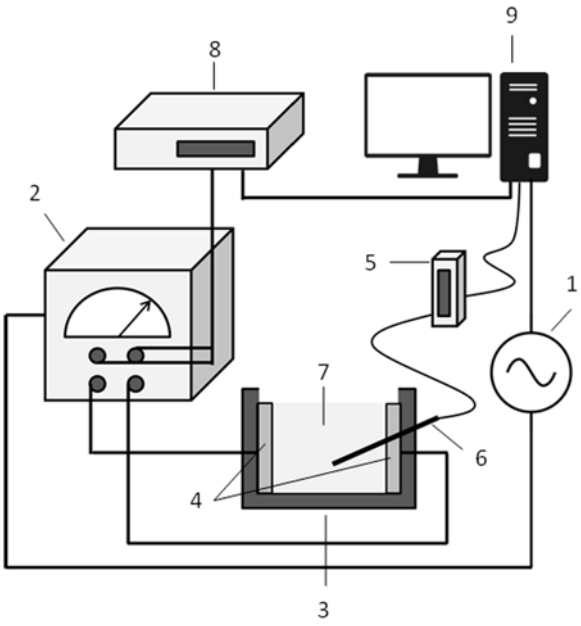

Fig. 13.1 Schematic illustration of a pilot plant scale ohmic heating system, redrawn according to Salari and Jafari (2020): (1) AC power supply, (2) adjustable voltage transformer, (3) treatment cell, (4) electrodes, (5) thermocouple with (6) sensor, (7) sample, (8) data logger, (9) computer

electrodes are generally separated by a tube or plate space that is electrically insulated (Goullieux and Pain 2005).

The total resistance in ohmic heater determines the current flow in the product. If the resistance is too high, the current will be too low, even when maximum voltage is reached. If the resistance is too low, the maximum limiting current is reached at a low voltage and the heating power will be too low as well (Fellows 2009). The total resistance of the heater can be calculated using the following equation:

$$R = \left(Rs^{*}L\right) / A \tag{13.2}$$

where R is the total resistance of the heater in ohms, R_s is the specific resistance of the product in ohms/m, L is the distance between the electrodes in m and A is the area of the electrodes in m^2 (Fellows 2009). As the specific resistance of a food is given by its composition and recipe, the geometry of the treatment cell has to be adapted to the food properties. Every material has a critical current density which exceedance may cause arcing in the heater. The current density is given by

$$I_d = I / A \tag{13.3}$$

where I_d is the current density in amps/m^2, I is the current in amps and A is the area of the electrodes in m^2. When the critical current density of a food material is known, the electrode area can be calculated accordingly (Fellows 2009). Industrial ohmic heating units are usually run continuously and are connected to other unit operations such as pumps, holding tubes etc. (Bengston et al. 2006). There are many

different designs of commercial ohmic heating units available depending on the manufacturer (Vicente et al. 2005; Icier 2012; Varghese et al. 2014). Important equipment parameters to consider when constructing an ohmic heating system are the electrode configuration, the electrode distance, heater geometry, the frequency of the alternating current, current density, applied voltage, product velocity and velocity profile (Bengston et al. 2006). Two possible electrode configurations exist for continuous processing – transverse and collinear configuration (Goullieux and Pain 2005; Varghese et al. 2014). In transverse mode, the food is transported parallel to the electrodes and the current flow and electric field are perpendicular to the product flow (Goullieux and Pain 2005). The construction is rather simple with plane or coaxial electrodes (Varghese et al. 2014). Problems may occur due to leakage currents through the product and inhomogeneities in the current density close to the electrode edges (Varghese et al. 2014). This may lead to local overheating of the food and erosion of the electrode material. Further advanced heating systems and units used for particulate foods thus follow a collinear configuration. In collinear configuration the product food flows from one electrode to the other and parallel to the current flow and the electric field (Goullieux and Pain 2005). Electrode housings and space tuber are alternated in the treatment cell. Due to the high voltages present, tighter security precautions need to be implemented (Varghese et al. 2014).

It can further be distinguished in in-line and cross-field systems depending on the positons of the electrodes along the product flow path (Varghese et al. 2014). Often several ohmic heater columns are combined in one unit (Icier 2012). This can increase the product throughput without increasing the voltage necessary to generate the requested electric field strength. Control systems are usually connected to the equipment to monitor process parameters including temperature, flow rate and specific heat of the product (Icier 2012). Based on these data the treatment intensity can be adjusted to achieve the desired heating profile. Detailed descriptions of available designs of commercial ohmic heaters can be found in Varghese et al. (2014) and Chen (2015).

Besides equipment design and process parameters used, characteristics of the food material markedly influence the ohmic heating process. Critical factors are the electrical conductivity of each food component, viscosity, density, pH-value, where there exist, the size and concentration of particles, thermal conductivity and the specific heat capacities of each component (Fellows 2009; Chen 2015).

The electrical conductivity is the main parameter affecting heating rate in an ohmic heating process. It is the inverse of the specific electrical resistance of a material (Fellows 2009) and can be measured by the quantity of electricity transferred across a unit area, per unit potential gradient and per unit time (Goullieux and Pain 2005). Assuming negligible heat losses, the temperature rise during a continuous ohmic heating process can be calculated using the following equation:

$$\Delta T = V^{2*} \sigma a^* A / \left(L^* m^* c_P \right) \tag{13.4}$$

where ΔT is the temperature rise in K, σ_a is the average product conductivity throughout temperature rise in S/m, A is the cross sectional area in m^2, L is the distance between electrodes in m, m is the mass flow in kg/s and c_P is the specific heat capacity of the product in J/kg/K (Fellows 2009).The electrical conductivity differs for different foods (Sarang et al. 2008; Chen 2015) and varies to a much greater extent than their thermal conductivities (Fellows 2009). The conductivity of a complex material is typically a sum contribution of individual ions, molar equivalent concentrations of individual ions and molar equivalent conductivity (Goullieux and Pain 2005). Ions from salts and acids and moisture increase the electrical conductivity, lipids and alcohol decrease it (Bengston et al. 2006). Analogue to the overall resistance of a heater, an optimum area can be found for the specific electrical conductivity of a product. This limits the range of foods feasible for the application of ohmic heating technology (de Alwis and Fryer 1992).

The electrical conductivity of foods is as well dependent on temperature. Usually, the conductivity increases linearly with temperature due to the enhanced ionic mobility at higher temperatures (Palaniappan and Sastry 1991; Reznick 1996; Wang and Sastry 1997a, b; Goullieux and Pain 2005). Non-linear, sigmoidal temperature-dependencies were determined at lower field strengths below 60 V/cm. Conductivity is furthermore a function of the food structure (Halden et al. 1990). Phase transitions such as starch gelatinization, protein denaturation or melting of lipids may alter the conductivity; inter alia due to the decrease in water availability (Halden et al. 1990; Wang and Sastry 1997a, b).

Increased heating rates were observed for plant tissue after reaching a critical temperature range (Goullieux and Pain 2005). The cell membranes in plant tissue are electrical insulators and current flow is usually restricted to the intercellular fluid. High temperatures may affect the cell integrity via denaturation of membrane proteins and breakdown of pectic cell wall components leading to higher ion mobility and a higher electrical conductivity (Halden et al. 1990; Palaniappan and Sastry 1991). Cyclic ohmic heating trials (Wang and Sastry 1997a, b) and previous cell disintegration using other techniques (Imai et al. 1995) confirmed the increased conductivity after cellular breakdown. The orientation of vascular bundles in the electric field and the shape of parenchyma cells can also influence conductivity and may lead to different heating rates for the same food material (Wang et al. 2001).

For multiphase systems, the electrical conductivities of all phases have to be considered. This makes prediction and controlling of heating characteristics very complex (Fellows 2009; Chen et al. 2010; Chen 2015). Beside differences in the conductivity, differences in density and moisture content may also affect the heating of the different phases (Fellows 2009). Thus particles may heat faster than the surrounding liquid even when their conductivity is lower (Fellows 2009). Thermal diffusivity and surface heat transfer coefficient between carrier fluid and particles were found to have weak effects on the process temperature (Chen et al. 2010). Frequency and waveform of the alternating current are additional factors influencing electrical conductivity and thus heating rate (Lima et al. 1999a,b; Bengston et al. 2006). In some cases it might be reasonable to increase the conductivity of the particle via salt

infusion to increase heating rate and minimize overall processing time (Goullieux and Pain 2005). Salt infusion at different salt concentratons and soaking times was used to increase conductivity and heating rate of potato tissue (Wang and Sastry 1993). Due to the slow salt diffusion from the particle surface inwards suitability of salt infusion is determined by particle sizes. Obviously, a balance between process optimization and sensorial and nutritional requirements has to be achieved (Palaniappan and Sastry 1991).

Besides all benefits, there are some limitations and open questions that have to be mentioned. As mentioned before, the presence of parts with very high or low conductivity remain a problem. Non-food materials such as metals, wood or plastic possess such extreme conductivities, but they are removed from food before processing and therefore constitute no actual issue (de Alwis and Fryer 1992). Presence of fat globules in the food may create an actual problem as the current may bypass them, when conductivity of the surrounding material is high enough. This may lead to less heat treatment and insufficient microbial inactivation in these product parts (Icier 2012). Thermal conductivities of solids vary by three orders of magnitude, whereas electrical conductivity may vary by many orders of magnitude from effectively zero to infinity. Therefore, non-conductive inclusions have a much higher impact in ohmic heating compared to conventional treatments (de Alwis and Fryer 1992).

Although there is no theoretical limit for the size of particles in ohmic heating, there are some practical limitations. While heating can be performed very fast with direct methods, cooling of the product occurs via thermal conduction (de Alwis and Fryer 1992). Although very low temperatures at the heat transfer surface are considered as a minor problem in regard to food quality compared to very high temperatures, slow cooling rates may lead to over-processing in the particle center. Furthermore, particles need to be pumpable and fit through the aseptic packing system (de Alwis and Fryer 1992).

Due to the direct contact between electrodes and food material, undesired reactions such as corrosion or electrolysis may occur (Goullieux and Pain 2005; Sastry 2005) affecting food quality and safety. Extent of the reactions is affected by frequency and density of the current, temperature, electrode material and aggressiveness of the product composition (Goullieux and Pain 2005). Approaches to prevent electrolysis include application of higher frequencies up to 100 Hz, usage of titanium electrodes coated with platinum, limitations in the current density and addition of electrolyte layers between product and electrodes (Goullieux and Pain 2005). Nevertheless, control of electrode reactions is a very important aspect that needs to be considered during ohmic heating.

In addition, there are some non-technological issues affecting ohmic heating application. Although the process costs of ohmic heating are comparable to conventional ones, investment costs for the systems may deter companies from switching their processing lines (Icier 2012), especially in case of small and medium enterprises. In addition, ohmic heating systems need well-trained personnel and availability of adequate safety and quality-assurance protocols. Furthermore, consumer constraints to electrically processed products limit industrial use of the technology (Icier 2012).

13.3 Improved Retention of Bioactives During Ohmic Heating

As mentioned before, the usage of ohmic heating provides interesting advantages regarding temperature homogeneity and heating rate. Faster heating to process temperature and thus lower overall thermal load are considered to bring as well benefits in terms of temperature sensitive valuable food components. Several research groups addressed this issue in their publications.

Vikram et al. (2005) compared ohmic, microwave, infrared and conventional heating in regard to the degradation of nutrients in orange juice. Samples were treated with different process temperatures and holding times. In the tested temperature range of 50–90 °C ohmic heating showed the best values for the retention of vitamin C. Ohmic heating led to the fastest heating of the samples whereby overall processing time was shorter compared to conventional and infrared heating. Heating up with microwaves was rather uncontrolled due to lack of adequate temperature control and was thus of limited comparability.

Achir et al. (2016) determined the carotenoid profiles of grapefruit and blood orange juices treated with ohmic or conventional heating. Pasteurization values of both technologies were matched and amounted 50 and 150 min at a reference temperature of 70 °C using a z-value of 10 °C. Contents of lycopene and β-carotene were not affected by both heating techniques. Degradation of xanthophylls was much more pronounced for both heating techniques. The presence of oxygen in the xanthophyll structure leads to a higher heat sensitivity compared to carotenes (Salari and Jafari 2020). Losses in epoxyxanthophylls and hydroxyxanthophylls amounted up to 70% and 40%, respectively, for conventional heating, whereas reduction up to 30% and 20% were observed for ohmic heating (Achir et al. 2016). This can be traced back to the differences in temperature profiles. A temperature of 95 °C was reached after 48 s with ohmic heating whereas it took 24 min to obtain a sample temperature of 80 °C in an oil bath.

Abedelmaksoud et al. (2018) applied surface response methodology to compare the effects of ohmic and conventional heating on quality parameters of apple juice. Ohmic heating was performed in a batch cell with titanium electrodes applying voltage of 30, 35 and 40 V/cm and a sinusoidal current of 60 Hz until temperatures of 60, 70 and 80 °C were obtained. Conventional heating was performed in a shaker water bath at 90 °C. Holding time for all samples was 60 s. Ascorbic acid and carotenoid contents in ohmic heated samples were higher than those of conventionally treated ones. The improved retention of these bioactive components can again be traced back to faster heating and a lower total thermal load. Only slight differences were found for polyphenol content, total soluble solids, titratable acidity, and viscosity of the juices. Cloud value strongly increased for both heat treatments. Only slight color changes compared to the untreated sample occurred during ohmic heating that still fulfil sensorial product requirements.

Farahnaky et al. (2018) compared ohmic heating of high and low intensity with microwave and conventional cooking. Kohlrabi, turnip, potato and radish were cut in tubes and treated at a frequency of 50 Hz and voltages of 4.3 and 7.4 V/cm.

Ohmic heating showed the highest rate in softening the vegetable tissue and thus possessed the shortest cooking time. The degradation of vitamin C after cooking was lower in kohlrabi, potato and radish after ohmic heating. Ohmic heating was also superior in preservation of total phenols and flavonoids as well as leaching of iron for all vegetables tested which can be explained by the shorter overall processing time.

A further advantage of ohmic heating is the opportunity to increase shelf-life of foods containing solids without the need to accept marked overprocessing of the liquid phase. Wattanayon et al. (2021) simulated vitamin C degradation in beverages with particles by adding alginate particles to orange juice. Ohmic heating of the sample in a conductive packaging was compared to conventional treatment. A pasteurization value of 5D was required for the inactivation of *E. coli* in the alginate particles. The total heating time to reach the pasteurization value amounted 2.35 min for ohmic and 6.03 min for conventional processing. Vitamin C content of the ohmic heating sample was similar to that of the untreated control while conventional treatment reduced the vitamin C content by more than 13%. This confirms the high potential of ohmic heating as a preservation method for solid liquid mixtures.

Furthermore, enhanced microbial inactivation due to the application of an electric field is discussed by several authors (Sastry and Barach 2000; Goullieux and Pain 2005; Knirsch et al. 2010). Lower D values were reported for *Streptococcus thermophilus, Escherichia coli, Bacillus licheniformis* and total aerobes (Pereira et al. 2007; Sun et al. 2008), which were traced back to pore formation in the cell membranes within the electric field. This is confirmed by results of Yoon et al. (2002) who reported a greater amount of intracellular material in ohmic heated samples compared to conventionally heated samples with similar time-temperature-history indicating electroporation of microbial cells. In addition, inactivation may be caused by local hot spots or formation of toxic substances such as free chlorine or hydrogenperoxide (Palaniappan et al. 1990). Significant lower D values were as well found for spore forming organisms, including *Geobacillus stearothermophilus*, *Bacillus coagulans* and *Alicyclobacillus acidoterrestris* (Baysal and Icier 2010; Somavat et al. 2012a, b; Somavat et al. 2013). This was attributed to effects on spores' proteins and dipicolinic acid molecules (Somavat et al. 2012a, b). Cho et al. (1999) compared single versus double stage heating for ohmic and conventional inactivation of *Bacillus subtilis* spores. A higher lethality and a greater tyndallization effect were observed for ohmic heating.

An improved inactivation was as well reported for several enzymes. Ohmic blanching of artichoke heads at 24 V/cm and 80 °C led to higher enzyme inactivation rates compared to hot water blanching at 100 °C. Total inactivation times for peroxidase and polyphenoloxidase were 360 s and 480 s for ohmic and conventional treatment, respectively (Guida et al. 2013). Ohmic blanching of pea puree also resulted in a faster inactivation of peroxidase compared to conventional processing in a boiling water bath when voltage gradients of 30 V/cm or higher were used (Icier et al. 2006). Saxena et al. (2016) compared inactivation of polyphenoloxidase in sugarcane juice at 80 °C with and without application of an electric field. Applying a voltage gradient of 32 V/cm and keeping a process temperature of 80 °C for 1 min

led to a reduction of enzyme activity to 10.07%. Conventional heating to 80 °C and maintenance of the temperature for 10 min achieved a residual activity of 6.47%. Higher inactivation rates of polyphenoloxidase were as well found by (Makroo et al. 2017) in watermelon juice comparing ohmic heating at 24 V/cm and 50 Hz to 90 °C and a water bath treatment at the same temperature. Castro et al. (2004a, b) investigated enzyme inactivation applying ohmic and conventional heating with matched temperature profiles. Inactivation of alkaline phosphatase, pectinase, and β-galactosidase was not affected by the electric field. Enhanced inactivation due to ohmic heating was determined for lipoxygenase and polyphenoloxidase.

The enhanced inactivation of microorganisms and enzymes may serve a further reduction in processing time and thus help as well to maintain valuable food components. The degradation of many heat sensitive compounds such as ascorbic acid, thiamine, riboflavin or anthocyanins follows first order kinetics (Van den Broeck et al. 1998; Vikram et al. 2005; Mercali et al. 2013, 2015; Kadakal et al. 2018). Thus a reduction in processing time is directly related to a marked benefit for the retention of food quality.

Demirdoven and Baysal (2014) compared the quality of orange juices after ohmic heating at 42 V/cm to 69 °C, 44 V/cm to 70 °C and conventional heating to 95 °C with a subsequent holding time of 60 s. Thermal treatments led to inactivations of pectin methylesterase of 96%, 95.5% and 88.3%, respectively. A slightly better retention of ascorbic acid was determined for the juice processed with the ohmic system. This was explained by the high temperature sensitivity of ascorbic acid and the lower temperatures required for enzyme inactivation using ohmic heating.

The individual temperature sensitivity of bioactives markedly influences their retention after ohmic heating. High degradation rates were especially reported for heat sensitive ascorbic acids while better retention or even higher concentrations were observed for other compounds.

Hashemi et al. (2019) compared ohmic, microwave and conventional heating for their potential to pasteurize cantaloupe juice. Ohmic heating and microwave heating were performed at 100 and 200 V and 400 and 800 W for 110 s, respectively. Conventional treatments were conducted in a hot water bath. All heating methods led to a reduction in microbial load as well as contents of vitamin C, β-carotene and phenolics. Effects increased with increasing voltage, microwave power and temperature in the conventional sample. Direct heating led to a faster inactivation of microorganisms and a higher degradation of vitamin C, but to a better preservation of β-carotene and phenolics compared to water bath heating.

Yildiz et al. (2010) applied ohmic heating to puree of blanched spinach leaves and compared the results to heating in a water bath. Lab scale equipment was used at 50 Hz and applied voltages varied between 10 and 40 V/cm. Depending on the voltage gradient heating to temperatures of 60–90 °C occurred up to four times faster than in a water bath. Slightly higher contents of β-carotene could be found in samples treated with ohmic heating compared to the untreated spinach puree. A holding time of 600 s at temperatures of 60–80 °C increases this effect. Conventional treatment led to slightly decreased contents of β-carotene indicating that ohmic

heating provides better retention of carotenoids. There were no significant changes in chlorophyll content for both heating methods.

Ramnath et al. (2018) investigated the effect of electric field strength, type and concentration of lye salt on bioactive compounds in tomato puree. A laboratory unit with stainless steel electrodes was used and electric field strength varied from 928 to 1214 V/cm. Temperature development was influenced by the type and concentration of salt used. Higher temperatures were obtained with sodium chloride compared to potassium hydroxide and sodium hydroxide, respectively. Higher concentrations of vitamin A were determined in the puree when higher temperatures were reached due to use of higher electric field strength, salt type or concentration. On the contrary, the vitamin C content decreased with increasing process temperature, which was explained by the higher temperature sensitivity of vitamin C compared to vitamin A.

Similar results were obtained by Somavat (2011) who explained minor changes in carotenoid content of tomato juice after ohmic heating to temperatures between 95 and 110 °C with the high heat stability of these compounds. The content of total phenols was as well not markedly affected in this study, which was considered as a potential advantage of the ohmic technology compared to conventional heating.

Ohmic heating of rice bran was performed in lab-scale with titanium electrodes at electric field strengths of 75, 150, 225 V/cm and a frequency of 50 Hz adjusting different moisture levels (Loypimai et al. 2009). Temperature profiles were recorded with a data logger and final product temperatures after 10 min of treatment varied between 60 and 124 °C. Higher moisture contents and higher electric field strength led to higher heating rates. Higher contents of total phenolics, α-tocopherol and γ-oryzanol could be detected with highest values for 40% of moisture or 30% moisture and electric field strengths of 150 and 225 V/cm. In regard to antioxidant activity, best results were obtained with a treatment of 30% moisture and 150 V/cm electric field strength.

Rinaldi et al. (2020) pretreated peach cubes in syrup with ohmic heating and investigated the quality impact of subsequent processing with ohmic heating, high pressure and conventional pasteurization. Treatment intensities of ohmic and conventional heating were equal to a pasteurization of 100 s at 98 °C; high pressure treatment was performed at 600 MPa for 3 min. The content of previously added ascorbic acid in the samples was reduced by 6%, 16% and 22% for high pressure, ohmic and conventional heating, respectively. The total phenol content was not affected by high pressure, but increased by about 50% for both thermal treatments. This was traced back to observed tissue disintegration and connected release of phenolic compounds.

Ohmic blanching of artichoke heads at 24 V/cm at 80 °C also led to an increase in total polyphenol content of 29% compared to fresh samples, whereas hot water blanching decreased the content by 27% (Guida et al. 2013). The authors mention different potential reasons for the increased phenol detection. High temperatures could lead to a release of bound phenols from cellular tissue and chemical complexes and thus to their improved interaction in the assay. Inactivation of food endogenous enzymes during thermal treatment may impede oxidation and

complexation of phenolics. Higher inactivation rates obtained with ohmic heating might lead to a better retention of monomeric phenols. Furthermore, release of cellular substances due to electroporation of cell membranes may lead to higher phenol contents in the samples after ohmic processing.

Bioactive proteins are formed during food processing in dependence of the process conditions applied. Costa et al. (2018) processed sweet whey with ohmic heating of 60 Hz and voltage gradients from 2 to 9 V/cm. A higher number of bioactive peptides were found compared to a conventional treatment at 75 °C for 15 s. The positive effect was more pronounced at lower electric field strengths. Ferreira et al. (2019) investigated the impact of different voltage gradients and current frequencies on bioactive peptide formation in whey beverages. Lower voltage gradients and frequencies resulted in higher antioxidant activities compared to higher treatment intensities. Ohmic heating led to lower anthocyanin contents and higher formation of bioactive peptides in relation to a conventional processing.

13.4 The Potential of Ohmic Heating for the Extraction of Bioactive Compounds

Several authors reported positive effects of ohmic heating on the extraction efficiency of plant compounds. Raw materials investigated include fruits, vegetables, herbs, oilseeds and algae. A detailed overview on the impact of moderate electric fields on extraction of food compounds is given by Gavahian et al. (2018). Improved mass transfer during ohmic heating is traced back to a combination of thermal and non-thermal effects. High temperatures induced by electrical energy dissipation may affect solubility and diffusion characteristics of compounds to be extracted. Reaching critical temperature ranges causes a thermobreak of the cell membrane via protein denaturation and leads to degradation of the cell wall by β-elimination and solubilization of pectins. This enables diffusion of intracellular components into the extraction medium. In addition, there were also non-thermal effects on cell tissue reported for processes involving application of an electric field.

Imai et al. (1995) applied electric fields of 40 V/cm for treatment times of 10, 30 and 50 s. to Japanese white radish. Temperature increase after 50 s was only 2.1 K so that the overall process temperature remained beneath 20 °C. Therefore, thermal effects on plant tissue could be neglected. Electrical impedance of the radish decreased with increasing treatment time indicating cell permeabilization and improved mobility of ions. This finding was confirmed by nuclear magnetic resonance imaging analysis of the plant tissue. Lebovka et al. (2005) as well reported a strong increase in cell disintegration index of potato and apple tissue after applying electric fields. A voltage gradient of 40 V/cm for approximately 100 s and a temperature range of 20–50 °C were used in this experiment. Pootao and Kanjanapongkul (2016) investigated changes in oil palm tissue on a cellular level. Light microscopic images revealed cell disordering and disruption of the cell wall after ohmic heating

at 60 °C whereas the sample without a voltage gradient appeared intact. Diffusion of beet juice from beet cubes was enhanced by ohmic heating compared to conventional heating in the temperature range from 42 to 58 °C (Lima et al. 2001). At a temperature of 72 °C, only small diffusion differences between the both technologies were detected. Probably, the temperature for thermal cell disintegration in beet tissue was reached enabling diffusion as well in the control samples. This supports the hypothesis that tissue alterations at lower temperature can be traced back to non-thermal electric effects.

It is assumed that electroporation is the main mechanism of these non-thermal effects on tissue integrity and mass transfer. Cell electroporation implies the formation of pores in the cell membrane due to application of an electric field (Knirsch et al. 2010). In intact plant tissue, the phospholipid bilayers of the cell membranes function as a barrier for ion movement in the electric field. Accumulation of ions at the cell membrane may lead to exceedance of the critical membrane potential followed by pore formation in the phospholipid bilayer. This effect may either be reversible or irreversible. Electro-osmosis, the movement of charged molecules in a liquid in dependence of the direction of the electric field, has shown to positively affect mass transfer steps (Bazhal and Vorobiev 2000) and might contribute to improved extraction using ohmic heating as well.

Process parameters applied markedly influence the improvement of mass transfer and as well the dominant mechanism affecting cellular tissue. Thermal or non-thermal effects or a combination of both may occur during ohmic heating depending on the process temperature applied. At temperatures below the denaturation temperature of the cell membrane non-thermal effects occur whereas at temperatures markedly exceeding this temperature area thermal effects dominate (Gavahian et al. 2018).

Lima et al. (2001) observed an ohmic-enhanced diffusion of beet dye. This effect increased with increasing electric field strength. Similar results were obtained by Schreier et al. (1993) who determined increased betanin diffusion from beetroot with increasing electric field strength. Extraction yields of palm oil and sesame oil were affected by the voltage gradient applied during ohmic heating as well (Kumari et al. 2016; Pootao and Kanjanapongkul 2016). This can be explained by the higher attraction forces between ions appearing at higher field strength which lead to a higher heating rate and a pronounced electroporation effect. Onwuka and Ejikeme (2005) investigated the effect of voltage type and electrode material on yield and quality of fruit juices. Using alternating current at a treatment intensity of 110 V, juice yield of orange and tomato pulp increased with increasing treatment time. A much smaller impact on juice yield was obtained for treatments with direct current of 9 V. This can be traced back to much higher temperatures obtained at 110 V AC leading to disintegration of the tissue. Higher voltages and alternating electric fields both lead to a more intense movement of ions in the fruit pulp. Decreased juice extraction was observed after ohmic heating for pawpaw pulp. There was no marked influence of electrode material on temperature profile and juice yield.

Frequency of the applied voltage significantly affected drying rate of yam and juice yield of apples (Lima and Sastry 1999). Sinus waves of 60 Hz were compared

to sawtooth waves of 4 Hz. Improvements in mass transfer were markedly greater using 4 Hz for both processes. Extraction yields from fresh mint leaves were much higher for a frequency of 50 Hz than for 500 and 5000 Hz (Sensoy and Sastry 2004). Low frequencies allow ions to accumulate at the cell wall and build up sufficient charge for electroporation. At high frequencies the rapid alteration of the electric field direction does not allow sufficient charge build-up (Icier 2012).

Benefits of ohmic-assisted extraction are the synergistic effects of temperature and electric field on cell tissue, lower temperatures needed for cell permeabilization compared to conventional heating and simpler requirements regarding the equipment when compared to other electric treatments such as pulsed electric fields or high voltage electrical discharges (Vorobiev and Lebovka 2010). The non-thermal effects on cell disintegration are of special interest for the processing of foods containing high amounts of heat sensitive components. Some research activities were done on the improved extraction of bioactives during or after ohmic heating.

El Darra et al. (2013) investigated the effect of pulsed ohmic heating on the extraction of polyphenols from grape pomace using a batch cell with stainless steel electrodes. Approximately 20 to 50 series with 300 pulses of field strengths up to 800 V/cm were applied. Pulse duration was about 100 μs. Cell disintegration index increased with temperature and electric field strength. Using a voltage gradient of 100 V/cm, even with a temperature of 60 °C a cell permeabilization index below 0.4 was determined, while with electric fields strengths of 300 and 400 V/cm values above 0.6 were reached at temperatures around 40 °C. This indicates a high impact of non-thermal cell rupture at high electric field strength. Calculating the energy consumption needed to achieve a certain degree of cell permeabilization, higher electric field strength showed to be more beneficial. Polyphenol extraction in water and 30% ethanol was improved by ohmic heating at 400 and 800 V/cm.

For wheat bran extraction, ohmic heating with electric field strengths of 14, 20 and 44 V/cm led to a faster heating up to a process temperature of 80 °C than conventional heating (Al-Hilphy et al. 2015). Despite the shorter processing time, slightly higher phenol contents and antioxidants activities were measured in the extracts.

Pereira et al. (2016) investigated the influence of voltage gradient, temperature and extraction time using as well a Box-Behnken design. Temperature profiles at voltages of 0, 15 and 30 V/cm during heating up to 90 °C were matched to figure out the impact of the electric field. Extraction of total phenols and anthocyanins from colored potatoes was affected by all three parameters. Temperature and time as well as their interactions were the main factors affecting the extraction of solutes. The yield of total phenols also increased with increasing voltage gradient whereas for anthocyanins an optimum yield at an electric field strength of 15 V/cm was observed. Combining high field strength and temperatures above 70 °C a decreased anthocyanin content which could be a result of degradation into the constituent phenolic acids.

Fraccola et al. (2016) extracted pigments from the microalgae *Chlorella vulgaris* using electric fields of 25 kHz and 50 V/cm. The temperature increased from 22 to 45 °C. Compared to a conventional extraction process without heating, the

concentration of extracted pigments, carotenoids, chlorophyll a and b, was 15 times higher when applying ohmic heating.

Aamir and Jittanit (2017) compared extraction efficiency of ohmic heating for unpolar compounds using a water-hexane mixture with conventional hexane extraction supported by conductive heating. Both extractions were performed with increasing hexane to gac aril powder ratios at a temperature of 50 °C. Ohmic heating led to a higher extraction efficiency of gac aril oil and markedly higher contents of lycopene and β-carotene in the oil. Scanning electron micrographs revealed a rupture of cell walls in the ohmic heated samples whereas conventionally heated and untreated cells were intact and closely packed.

Bhat et al. (2017) investigated the effect of ohmic pretreatment on the phenol content of subsequently recovered gourd juice. Cubes of bottle gourd were blanched using ohmic and conventional water-bath heating and the juice extracted afterwards was analyzed on total phenols and color. An ohmic heating cell with stainless steel electrodes was used at 220 V and a frequency of 50 Hz leading to an electric field strength of approximately 30 V/cm. All thermal treatments increased the extraction of phenols into the juice. Higher total phenolic contents were detected in samples pretreated with ohmic heating to temperatures of 70 °C and above while no marked differences could be observed between temperatures of 60 to 90 °C for conventional treatments and ohmic heating of 60 °C. Optimum ohmic heating conditions in this study were 80 °C and 4 min, while prolonged heating and higher temperatures reduced the benefit of the treatment. This was traced back to an overlap of positive effects of temperature on polyphenol extraction and thermal degradation effects. LC-MS analysis revealed an increased content of gallic acid and theaflavin 3-gallate in the ohmic heating samples. Color changes were temperature dependent and did not differ markedly for ohmic heating and water bath.

Coelho et al. (2017) performed experiments on the impact of process parameters on the ohmic assisted extraction from by-products of tomato processing. A Box-Behnken design with three levels was used and temperatures of 40, 55, and 70 °C, extraction times of 0, 15 and 30 min and ethanol in water concentrations of 0, 35 and 70% were applied. Extraction yields of phenolics and carotenoids as well as the antioxidant activity of the extracts increased with increasing temperature, extraction time and ethanol concentration.

Ferreira-Santos et al. (2019) compared extraction efficiency of phenols from pine bark for ohmic heating and thermal treatment in a water bath. Polyphenol contents were markedly higher after ohmic heating when 50% ethanol was used for extraction. A slight increase in the extraction yield could be observed increasing the electrical conductivity of the medium. Extraction yields were much lower using water as an extraction medium, but still a slight improvement by ohmic heating could be observed. Results for antioxidant activity measurements were in accordance with the higher polyphenol contents. The composition of phenolic compounds was influenced by the extraction method as well as by the medium conductivity.

Moongngarm et al. (2019) studied the effect of an ohmic pretreatment on the hexane extraction of oil from rice bran and its phytochemical composition. Higher

extraction yields of tocopherols, γ-oryzanol and total phenols were obtained for all ohmic heating intensities tested. Analysis of antioxidant activities resulted in positive or negative impact of ohmic heating depending on the assay used.

Mannozzi et al. (2019) compared the potential of ohmic heating as a pretreatment to juice recovery with that of pulsed electric fields. Various temperature-process combinations were investigated and the influence on juice quality determined. Conclusions on bioactive components in apple and carrot juice were drawn by means of the antioxidant activity. Results showed best results for treatments where temperatures of 80 °C were reached. This was explained by the inactivation of peroxidase and polyphenoloxidase that prevented reaction of valuable plant compounds. Additional benefits of pulsed electric fields and ohmic heating were considered the facilitated release of plant metabolites as well as the reduction in thermal load due to rapid heating.

Extraction of phenols from grape pruning residue was carried out at low and high electric field strength, 496 and 840 V/cm respectively, and compared to conventional heating and extraction at ambient temperature (Jesus et al. 2020). Increasing the temperature to 80 °C led to a higher phenol extraction independent from the heating method used. Ohmic heating provided a better antioxidant activity of the extracts, especially when the high electric field strength was used. The extracts differed as well in their polyphenolic composition.

The results presented highlight the great potential of ohmic heating to improve the mass transfer of bioactives. Improved extraction of phenols and carotenoids from plant materials may enhance recovery efficiency by increasing product yield or by reducing processing times and energy required. Higher contents of these plant metabolites in fruit and vegetable juices and sauces may contribute to a higher product quality in regard to sensorial and nutritional perspective,

13.5 Adverse Effects of Ohmic Heating on Bioactive Compounds

While many positive effects of ohmic heating on the retention and extractability can be found in literature, some research revealed negative impacts of electroheating on food material compared to conventional processing. This can be traced back to the electrodes in direct contact with the food system and reactions occurring during application of electric fields on their surfaces.

Electrolysis of water may occur at the electrodes leading to formation of hydrogen and oxygen at cathode and anode, respectively (Assiry et al. 2003):

$$\text{Cathode}: 2\text{H}^+(\text{aq}) + 2\text{e}^- \rightarrow \text{H}_2(\text{g})$$

$$\text{Anode}: 2\text{H}_2\text{O}(\text{l}) \rightarrow \text{O}_2(\text{g}) + 4\text{H}^+(\text{aq}) + 4\text{e}^-$$

The formation of oxygen may enhance oxidation reactions in the food. Arcing during the treatment may even lead to formation of singlet oxygen strongly increasing reaction rate (Assiry et al. 2003). The intensity of electrolysis is directly affected by the height of the voltage gradient. As usually alternating current is used and thus the direction of the electric field is periodically changing, both reactions may occur on each side of the treatment cell. Oxygen may interact with the food compounds until it ascends from the sample or reacts with the hydrogen formed. The frequency used is thus strongly affecting the electrode hydrolysis.

Corrosion of the electrode material may occur via direct oxidation or electrochemical formation of corroding species (Assiry et al. 2003). The generated metal ions may further oxidize, undergo secondary reactions or complex formation with food constituents or function as a catalyzer for their reaction among each other. The corrosion affinity of electrode materials markedly differs. Samaranayake and Sastry (2005) performed experiments with different electrode materials and identified titanium and platinized titanium as less prone to corrosion compared to graphite and stainless steel, who exhibit much higher corrosion rates at the tested pH values of 3.5, 5 and 6.5. Lima et al. (1999a, b) determined rust formation and bubble formation at stainless steel electrodes, while no such observations were made using titanium electrodes.

Due to the differences in material costs, stainless steel is often used in practice. Treatment parameters thus have to be optimized in regard to preservation of food quality and safety. Several research groups investigated the impact of ohmic heating on the degradation of bioactive compounds.

Onwuka and Ejikeme (2005) applied ohmic heating as a pre-treatment for the recovery of orange and tomato juice. Voltage type and electrode material were varied. Copper/copper and copper/aluminum electrodes were used to generate direct current of 9 V or alternating current of 110 V. All treatments led to a degradation of vitamin C which was more pronounced applying higher voltages and temperatures. Even in samples treated with direct current that reached a maximum temperature of 30 °C vitamin C degradation was observed. Considering the low process temperatures, this was traced back to electrochemical reactions of the electrode material. This effect was more pronounced for copper/copper electrodes than for copper/aluminum electrodes. Visual observations also indicated stronger electrolysis at the copper/copper electrodes.

On the contrary, Leizerson and Shimoni (2005a, b) did not find significant differences in the vitamin C content of orange juice treated in a continuous ohmic heater or a plate heat exchanger. In this experiment an electroheater with graphite electrodes and a frequency of 50 Hz were used. Differences between effects of ohmic heating temperatures of 90, 120 and 150 °C and varied product flow rates were as well not significant, probably due to the low overall treatment time.

Mercali et al. (2012) compared the degradation of ascorbic acid in acerola pulp during ohmic and conventional heating. An ohmic heating setup with a batch Pyrex glass cell and titanium electrodes was used. The applied voltages amounted 120–200 V and the current frequency was 60 Hz. Ohmic and conventional samples were heated to 85 °C and kept at this temperature for 3 min. Lower voltage gradients

applied influenced ascorbic acid in a same magnitude as conventional heating. Higher electric field strengths led to an increased degradation of this bioactive compound. The same experimental setup was used to evaluate the impact of ohmic heating on anthocyanins in acerola pulp (Mercali et al. 2013). There were no significant differences between the degradation rates of ohmic and conventional heating in the studied temperature range of 75–90 °C.

Sarkis et al. (2013) investigated the impact of voltage gradient and solid content on anthocyanin degradation in blueberry pulp. Both parameters were positively correlated to the degradation rate. At lower voltage gradients similar or higher anthocyanin retention as for conventional treatments were obtained. Higher voltage gradients led to an increased degradation of this plant metabolite.

Saberian et al. (2015) compared ohmic heating and conventional heating for the pasteurization of aloe vera gel juice. An ohmic heating unit with stainless steel electrodes and a frequency of 60 Hz was used. Samples were heated up top 90 °C and the temperature was kept for 1 min. Conventional heating was performed in a 90 °C water bath for a holding time of 1 min. Markedly lower contents in vitamin C were found for ohmic heated samples than in conventionally treated and raw juices. This was traced back to synergistic effects of temperature, oxygen and metal ions. The contents of total phenols were vice versa and the highest content was found after ohmic heating.

Athmaselvi et al. (2017) investigated the effect of electrode material on ascorbic acid content of tropical fruit pulp. Pulp from guava, sapota and papaya was treated in a batch system with either stainless steel or titanium electrodes. Voltages of 10 and 23.33 V/cm were applied at a frequency of 50 Hz. Target temperatures amounted 70, 80, 90 and 100 °C and were kept for a holding time of 5 min. Ascorbic acid concentration in all samples decreased with increasing voltage, temperature and holding time. Usage of titanium electrodes led to a better ascorbic acid retention. This was traced back to a higher heating rate using the electrode material.

Sabanci et al. (2019) concentrated pomegranate juice via conventional and ohmic heating assisted vacuum evaporation. Titanium electrodes and voltage gradients of 7.5, 10 and 12.5 V/cm were used. Samples concentrated using ohmic heating gave lower values for their anthocyanin and total phenolic contents as well as in their antioxidant activity compared to the conventional samples. This was traced back to electrochemical reactions at the electrode surface. The use of more electrochemically inert electrode materials is suggested by the authors. The same equipment was used for the concentration of sour cherry juice (Sabanci and Icier 2019). Higher retention of total phenols and anthocyanins compared to conventional evaporation was observed. Electric fields of 10, 12 and 14 V/cm were applied and the positive effect on retention of bioactive compounds increased with the voltage gradient. This can be traced back to shorter processing times needed. Antioxidant activities in a similar range were determined for both concentration techniques.

Detrimental effects of ohmic heating were mainly observed for ascorbic acid/vitamin C and anthocyanins. Ascorbic acid degradation can occur via an oxidative or an anaerobic pathway (Assiry et al. 2003). In conventional food processing ascorbic acid losses are primarily a consequence of chemical oxidation. Chemical

degradation via the anaerobic pathway is less important in food processing as usually a sufficient amount of oxygen is present. Release of oxygen by electrolysis might directly affect degradation rate by the higher (local) concentration of a reaction substrate. Presence of metal ions, especially Fe^{3+} and Cu^{2+} may accelerate the reaction by several orders of magnitude (Assiry et al. 2003). Therefore, liberation of metal ions due to corrosion reactions may lead to enhanced ascorbic acid degradation. These reinforcing factors in the electric field are considered as a third electrochemical degradation pathway (Assiry et al. 2003). Similar explanations can be found regarding the degradation of anthocyanins although the reaction pathways are less well understood. Degradation of anthocyanins is mainly caused by oxidation and is thus strongly affected by the presence of oxygen (Patras et al. 2010). Sinela et al. (2017) reported reaction path ways of metal-catalyzed oxidation and a molecule scission that are both influenced by the nature and concentration of metals present. In contrast, a stabilization of anthocyanins due to complexations with metal ions is as well reported (Liu et al. 2018).

These findings highlight the importance to separately regard individual impact factors of ohmic heating for different food systems. Some systematic research work is performed comparing ohmic and conventional heating with matched temperature profile to intensively study the influence of electric field characteristics. Results are summarized in Table 13.1.

Lima et al. (1999a, b) did not find significant differences in ascorbic acid degradation in orange juices for ohmic and conventional heating with same thermal history. Although gas production and dissolution appeared at stainless steel electrodes, there was no difference comparing this electrode material with specially coated titanium in regard to ascorbic acid concentration.

Assiry et al. (2003) studied degradation kinetics of ascorbic acid during ohmic heating with thermal holding time. Ohmic heating with uncoated stainless-steel electrodes and a frequency of 60 Hz and intensities of 0, 100, 150 and 300 W were used in a temperature range from 40 to 80 °C. Buffer solutions with pH 3.5 and different NaCl contents of 0.25, 0.5 and 1% served as a model for acidic food systems and ascorbic acid was added to the buffer after reaching a constant desired temperature to exclude the impact of heating rate. No significant differences between ohmic and conventional heating were found for ascorbic acid degradation, except for a power of 150 W, a salt concentration of 1% and a temperature of 40 °C. At the highest power and salt content, citrate complexation and a significant loss of buffering capacity were noted, resulting in an increased pH value.

Castro et al. (2004a, b) compared ohmic heating and conventional heating with matched temperature profiles in regard to ascorbic acid retention in strawberry pulp. The pulp was heated to temperatures up to 100 °C using an ohmic system with titanium electrodes and electric field strengths of 25–100 V/cm. Conventional treatments at the same temperatures were performed in industrial scale with a scraped-surface heat exchanger. Ascorbic acid degradation followed first order kinetics for both conventional and ohmic heating treatments. The electric field did not affect the rate of ascorbic acid degradation.

Table 13.1 Effects of ohmic and conventional heating with same temperature profile

Bioactive compound	Food system	Electrode material	Frequency	Treatment intensity	Treatment temperature	Maximum treatment time	Degradation[a]	Author
Ascorbic acid	Buffer pH 3.5, 0.25–1% NaCl	Stainless steel	60 Hz	100–300 W	40–80 °C	60 min	o	Assiry et al. (2003)
	Buffer pH 3.5, 1% NaCl	Stainless steel	60 Hz	150 W	40 °C	60 min	↑	Assiry et al. (2003)
	Orange juice	Stainless steel	n.d.	18.2 V/cm	65, 75, 80 and 90 °c	5.5 min	o	Lima et al. (1999a, b)
		Coated titanium	n.d.	18.2 V/cm	65, 75, 80 and 90 °C	5.5 min	o	Lima et al. (1999a, b)
		Stainless steel	50 Hz	10–33 V/cm	80 °C	450 s	o	Tumpanuvatr and Jittanit (2012)
	Pineapple juice	Stainless steel	50 Hz	10–33 V/cm	80 °C	480 s	o	Tumpanuvatr and Jittanit (2012)
	Orange pulp	n.d.	80 Hz	30 V/cm	70–100 °C	30 min	o	Stojceska et al. (2019)
	Strawberry pulp	Titanium	50 Hz	25–100 V/cm	up to 100 °C	250 s	o	Castro et al. (2004a, b)
	Acerola pulp	Titanium	100 Hz–100 KHz	4–5.2 V/cm	85 °C	120 min	o	Mercali et al. (2014)
		Titanium	10 Hz	5–5.2 V/cm	85 °C	120 min	↑	Mercali et al. (2014)
		Titanium	60 Hz	4.3–5.5 V/cm	80, 85, 90 and 95 °C	60 min	o	Jaeschke et al. (2016)
	Liquid infant formula	Titanium	25 Hz	15 W	140 °C	150 s	↓	Roux et al. (2016)
Anthocyanins	Jaboticaba	Titanium	60 Hz	3.5–4.5 V/cm	60–90 °C	20 min	o	Mercali et al. (2015)
	Blackberry pulp	Platinum	60 Hz	4.1 V/cm	70–90 °C	90 min	o	Sarkis et al. (2019)

(continued)

Table 13.1 (continued)

Bioactive compound	Food system	Electrode material	Frequency	Treatment intensity	Treatment temperature	Maximum treatment time	Degradation[a]	Author
		Platinum	60 Hz	22.3 V/cm	80 °C	90 min	↑	Sarkis et al. (2019)
Phenols	Pomegranate juice	Stainless steel	50 Hz	10–40 V/cm	90 °C	12 min	o	Yildiz et al. (2009)
	Watermelon juice	Stainless steel	50 Hz	24 V/cm	90 °C	1 min	o	Makroo et al. (2017)
Carotenoids	Acerola Pulp	Titanium	60 Hz	4.3–5.5 V/cm	80, 85, 90 and 95 °C	60 min	o	Jaeschke et al. (2016)
Lycopene	Watermelon juice	Stainless steel	50 Hz	25 V/cm	91 °C	1 min	o	Makroo et al. (2017)

[a]Impact of ohmic heating on degradation in comparison to conventional heating: ↑increased degradation, ↓ decreased degradation, o no difference

Yildiz et al. (2009) investigated the ohmic heating potential for preservation of pomegranate juice. The same thermal history for the conventional and ohmic heating was applied to figure out the impact of the electric field on juice quality. Therefore the electric field was varied between 10 and 40 V/cm at a frequency of 50 Hz to achieve desired heating rate. Statistical analyses revealed no significant difference in the phenol content of juices treated with ohmic heating and conventional heating in a water bath in a holding time of 12 min.

Similar degradation of vitamin C in orange and pineapple juice was detected by Tumpanuvatr and Jittanit (2012) for ohmic heating with stainless steel electrodes when performed with the same heating rate as conventional processing.

Mercali et al. (2014) performed trials on ascorbic acid concentration and color changes in acerola pulp. A batch unit with titanium electrodes was used. The effect of current frequency and process time at constant temperature was evaluated and compared to thermal treatment in a water bath. The effect of heating-up on quality changes was subtracted to eliminate the effect of different heating rates. Ascorbic acid degradation was measured for all samples during thermal treatment.

After 120 min at 85 °C values were reduced by 13–17% for ohmic heating and by 14% for the conventional treatment. The highest degradations were found in samples ohmically treated with 10 Hz, indicating that electrochemical reactions were stronger at the low frequency. Color changes were in accordance with these results.

The same research group investigated anthocyanin degradation in jaboticaba juice applying ohmic and conventional heating with same temperature profiles (Mercali et al. 2015). Ohmic processing conditions were a voltage of 25 V, an electrode distance between 5.5 and 7 cm, a frequency of 60 Hz and a treatment time of 20 min. Rate constants were in a similar range for ohmic and conventional heating in the considered temperature range of 60–90 °C.

Jaeschke et al. (2016) studied non-thermal effects of ohmic heating on quality of acerola pulp. Ascorbic acid and carotenoid degradation were similar for ohmic and conventional heated and probably prevented by limited oxygen availability. In this study titanium electrodes and a frequency of 60 Hz at 30 V were used. Temperatures and treatment time for both heating methods were 80, 85, 90 and 95 °C and 60 min, respectively.

Higher inactivation rates of polyphenoloxidase in watermelon juice were obtained by ohmic heating at 24 V/cm and 50 Hz to 90 °C compared to water bath treatment at the same temperature (Makroo et al. 2017). Similar polyphenol degradation occurred in both technologies and only slight changes in the lycopene content were found.

The effects of ohmic heating and steam injection on quality of liquid infant formula were compared using the same conditions of pre-heating and holding (Roux et al. 2016). Electric fields were applied in a continuous coaxial chamber with titanium electrodes. Currents of 25 kHz and a power of 15 kW were used to achieve heating up to 140 °C. Slightly better preservation of vitamin C and color was observed after ohmic heating leading to the conclusion that ohmic heating is suited as an equivalent sterilization technique.

No significant differences in the vitamin C content of grapefruit and orange pulp during to conventional and ohmic heating assisted drying were reported by Stojceska et al. (2019). A voltage gradient of 30 V/cm, a frequency of 80 Hz and temperatures of 70 and 100 °C were used in this study.

Sarkis et al. (2019) also applied electric fields only during temperature holding time to eliminate the impact of sample heating up. Treatment intensities of 60 Hz and 4.1 V/cm showed no differences in anthocyanin degradation in blackberry pulp compared to conventional treatments with the same temperature profile. Higher voltage gradients of 22.3 V/cm led to an increased degradation at a temperature of 80 °C.

These findings show that additional degradation of bioactives due to the electric field can be prevented choosing an adequate process design with electrochemically inert electrode materials and process parameters. Low frequencies and high voltage gradients should be avoided when processing foods containing components sensitive to oxidation or metal catalyzed reactions to reduce electrode reactions to a minimum. Regarding the summarized literature data in Table 13.1, a safe process window in regard to field strength and frequency can hardly be identified. The data suggests that especially the application of long treatment duration of 60 min or more leads to additional degradation by the electric field. It is therefore required to spend more research activity on the influence of ohmic heating parameters on electrode reaction in dependence of the food matrix processed.

13.6 Conclusions and Future Perspective

Preservation and recovery of bioactives to obtain high nutritional quality for foods is an important issue for the food industry. Ohmic heating offers great potential to deliver a valuable contribution to both areas – gentle thermal processing of foods and enhanced extraction of food components. Improved homogeneity and increased rates of heating as well as additional inactivation effects on microorganisms and enzymes may lead to a decrease of required processing time and overall thermal load preventing heat sensitive compounds from thermal degradation. Combination of thermal and non-thermal effects on tissue integrity and molecule diffusion might lead to higher extraction yield and decreased extraction times or solvent usage. Nevertheless, there are still several knowledge gaps that need to be filled.

Adverse effects on product quality due to the electric field have been mainly investigated for ascorbic acid/vitamin C. There is lack of research data on the influence of electrolysis and corrosion on the degradation of other vitamins, secondary metabolites, essential fatty acids, bioactive peptides or minerals (Salari and Jafari 2020). Literature findings on adequate levels for electric field strength and process intensity were not consistent and safe process windows for individual food systems need to be identified. Especially, when non-thermal effects of the electric field are desired to improve mass transfer, high voltages and low frequencies have shown to

provide better results. These parameter recommendations are in contrast to those made to avoid electrode reactions and specific attention should be paid to a potential overlap of higher extractability and degradation of bioactive compounds. Further research is as well needed to rank the potential of ohmic heating in comparison to other cell disintegration techniques in regard to extraction yield, energy efficiency and product quality. Knowledge is as well required in regard to bioactive retention for a wider range of valuable compounds and products. Especially in terms of particle-rich foods and products with inhomogeneous conductivity distribution, effects on bioactive compounds are insufficiently documented.

Conductivity differences are also an issue in regard to food safety. The DFG Senate Commission on Food Safety (German Research Foundation, DFG) identified research needs for ohmic heating in different areas to guarantee product safety (SKLM 2015). Uniformity of heating and avoidance of cold spots during preservation due to different conductivities of food elements shall be assured and need a more detailed knowledge on material properties and possibilities to influence them. A more detailed investigation of microbial inactivation kinetics in the individual fractions of a heterogeneous product is also essential as well a distinction between thermal and non-thermal effects in the electric field. A more detailed analysis of potential chemical changes, especially due to electrochemical reactions, is also recommended. This includes possible effects on product allergenicity. A satisfying clarification of these safety issues will lay the foundations for maximizing the retention of bioactive substances as it sets the framework within a nutritional optimization is feasible.

To promote industrial uptake of the ohmic technology, factors related to upscaling have to be studied more intensively. Beside the higher throughputs and the requirement for continuous processing systems, higher inhomogeneities regarding the raw material properties may occur due to regional and seasonal variation. This is especially important in terms of electrical conductivity as a crucial factor for thermal and non-thermal process effects. Respective guidelines for different process targets and food systems need to be published. Process equipment with treatment chambers and electrode designs suitable for different food matrices must be available together with a reliable and easy to handle process control systems. Due to the higher energy conversion efficiency, ohmic heating can be regarded as a sustainable way of heat processing of foods. Energy footprints of developed process designs shall be considered as well during process optimization to connect healthy foods with sustainable processing.

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Part IV
Influence of Novel Non-thermal Processes on Food Bioactive Compounds

Chapter 14
Effects of Irradiation on Food Bioactives

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14.1 Introduction

Food irradiation is a promising technology that uses ionizing radiation for food processing. Although the history of using food irradiation for several benefits has more than 100 years, only after the 1960's the concept of commercial radiation sources became available. Irradiation is a non-thermal, clean and eco-friendly process, not involving the use of chemicals or generating chemical residues. It has been considered a safe and effective technology by the World Health Organization (WHO), the Food and Agriculture Organization (FAO) and the International Atomic

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Energy Agency (IAEA). Furthermore, it is firmly established that the food does not become radioactive, can be treated in its final packaging, which avoids re-contamination of the products and makes it available for immediate distribution to food suppliers.

In this chapter, after presenting general aspects about irradiation processes, we will discuss the effects of this technology on the food bioactives, as well as the potential applications of these recovered compounds in food, cosmetic and pharmaceutical industries thus promoting the circular economy for sustainable development.

14.2 Irradiation Processes

The three forms of ionizing radiation authorized to be used in food irradiation applications are gamma rays, X-rays and electron-beam accelerators (WHO 1988).

14.2.1 Gamma Radiation

Gamma radiation is generated by photons emitted from the radioactive isotopes cobalt-60 and cesium-137, with energies of 1.17 and 1.33 MeV (^{60}Co) and 0.66 MeV (^{137}Cs).

Cobalt-60 is the most common source of gamma radiation used for food processing. There are more than 200 large-scale gamma plants operating worldwide and this number is growing. The main advantage of these radioisotopes is the high efficiency due to their penetrating power. Nevertheless, when not being used, the gamma sources have to be stored in a water pool in order to absorb the energy and protect workers. In a gamma facility, the food product can be treated in the same boxes or pallets in which they will be transported and distributed, being thus carried into the irradiator, submitted to the radiation source and taken back out again. The isotope source is constituted most often by multiple pencils in known positions (Fig. 14.1). Gamma irradiators are designed to provide an acceptable distribution of absorbed dose within the product through an arrangement of, or pathway for, products in irradiation containers around the radiation source (Fig. 14.1), which allows the products to absorb the radiation from multiple angles (Ferreira et al. 2018).

14.2.2 Electron-Beam Radiation

Electron-beam radiation is produced from electron accelerators (Fig. 14.2) and it can be used directly or, indirectly, converted to X-rays. The beam energy of e-beam accelerators is usually from 80 keV to 10 MeV.

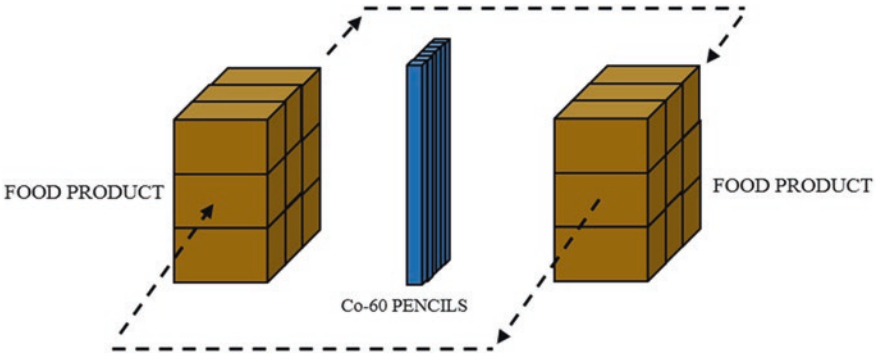

Fig. 14.1 Schematic diagram of a gamma irradiator facility

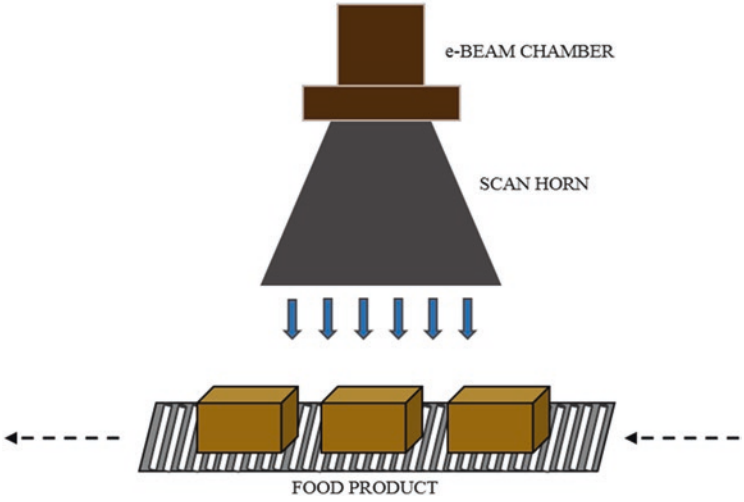

Fig. 14.2 Schematic diagram of an e-beam irradiator

The advantage of electron-beam irradiation is that the source equipment can be switched on and off depending on the necessity and do not rely on a radioactive source that radiates nonstop thus needing to be replaced after some time. In this way, the electron-beam irradiation is a more controlled and energy efficient process and it can also be considered a more environmentally acceptable alternative to ^{60}Co (Ferreira et al. 2018), yet with the disadvantages of a greater complexity of maintenance, lower dose uniformity and lower penetration in the products.

In an accelerator facility, there is a material handling system, the conveyor, that transports the food products to and from the irradiation zone in a precisely and controlled manner (Fig. 14.2) and, in some cases, moves them into and out of the facility (Miller 2005).

14.2.3 X-Rays

X-rays are generated by the same technology that produces electron-beams at or below an energy of 5 MeV, by a process known as Bremsstrahlung conversion (Miller 2005) which happens when electrons are accelerated to a metallic target (e.g. tantalum, tungsten or gold) to be converted to an X-ray steam. Nevertheless, most of the energy required to produce X-rays is lost as heat in the target, turning this process more inefficient and expensive than gamma or electron-beam radiation. Therefore, its throughput efficiency is quite low when compared to that obtained by e-beam. The photons in X-rays are similar to gamma radiation, meaning that they have very high penetration, but without using isotopes as source.

14.3 Dose Range and Dosimetry

Irradiation processing has been studied extensively worldwide and has become accepted as a proven and effective post-harvest treatment to reduce pathogenic contamination, prevent sprouting, eliminate parasites, extend the shelf-life of fresh perishable foods, and provide ready-to-eat food for astronauts and emergency rations and diets for immune-compromised patients, among other applications.

The different purposes on the use of irradiation technology depend on the absorbed radiation dose (Table 14.1), which is the accumulated energy that is transferred to the matter, i.e., absorbed energy per mass unit. The SI unit for dose is the "Gray" (Gy), defined as the absorption of one joule in a mass of 1 kg ($1\ Gy = 1\ J\ kg^{-1}$) of irradiated matter.

Dosimetry provides essential important information in radiation processing. When the food product is exposed to radiation, the absorbed doses and dose rates must be measured prior to its irradiation to determine the performance of the irradiation facility. The dosimetry system consists of the dosimeters, the measurement instruments and their associated reference standards and procedures for correct use. These systems are recommended to characterize the radiation facility for operational qualification, perform dose mappings, quality control and validation of the processes. In this way, the dosimetry systems must be calibrated to ensure that the irradiator is capable of operating and delivering the adequate doses to the product (ISO/ASTM 52303:2015). The minimum (D_{min}) and the maximum (D_{max}) absorbed doses that are applied to a food product in order to achieve the final purpose, maintaining its quality control and assurance, are based on the routine dose monitoring and regulatory limits that may be applicable. The ratio of the maximum dose received by a product stack to the minimum dose received is commonly referred to as the "Dose Uniformity Ratio" (DUR), with DUR = 1.0 as the ideal value. DUR must be optimized by changing, for example, the position of the product in relation to the source or the irradiator design. In food products with high density, the ideal DUR is difficult to achieve.

Table 14.1 Applications of food irradiation and recommended dose ranges

Purpose	Dose range (kGy)	Examples of food treated
Low doses (0.1–1 kGy)		
• Inhibition of sprouting	0.05–0.15	Potatoes, onions, garlic, ginger-root, chestnuts
• Insect disinfestation and parasite disinfection	0.15–0.50	Cereals and pulses, fresh and dried fruits, dried fish and meat, fresh pork
• Delay of physiological process (e.g., ripening)	0.50–1.0	Fresh fruits and vegetables
Medium dose (1–10 kGy)		
• Extension of shelf-life	1.0–3.0	Fresh fish, strawberries, asparagus
• Inhibition of spoilage and pathogenic microorganisms	1.0–7.0	Fresh and frozen seafood, raw or frozen poultry and meat
• Improving technological properties	2.0–7.0	Grapes (increasing juice yield), dehydrated vegetables (reduced cooking time)
High dose (10–50 kGy)		
• Industrial sterilization (in combination with mild heat)	30–50	Meat, poultry, seafood, prepared foods, sterilized hospital diets
• Decontamination of certain food additives and ingredients	10–50	Spices, enzyme preparations, natural gum

Various dosimeters can be used for food processing with a reproducible response to radiation, such as polymethyl methacrylate (PMMA) dosimeters, radiochromic dosimeters and alanine dosimeters. PMMA dosimeters can be measured in a calibrated spectrophotometer at a given specific wavelength to determine the response, the specific absorbance after irradiation and the dosimeter thickness. Alanine dosimeters can be produced in pellets or films and the concentration of free radicals formed during irradiation can be measured by Electron Spin Resonance (ESR). The American Society for Testing and Materials (ASTM) E61 'Radiation Processing' is an international group of experts who have established and maintained standard practices, methods and guides for ionizing radiation processing and dosimetry.

14.4 International Standards on Food Processing

The *Codex Alimentarius*, also known as "food code", is a compilation of all the standards, codes of practice, guidelines and recommendations of the *Codex Alimentarius* Commission, with the objective of protecting consumers' health and ensuring fair practices in the food trade. The proposed guidelines for irradiation are based on findings of the Joint Expert Committee on Food Irradiation (JECFI) composed by experts of the Food and Agriculture Organization, International Atomic Energy Agency and World Health Organization (FAO/IAEA/WHO). In 1981, WHO

published a document stating that irradiation of food at doses up to 10.0 kGy is safe and introduces no further toxicological or nutritional problem (JECFI 1981). In 1997, the FAO/IAEA/WHO Study Group on High-Dose Irradiation (JSGHDI 1999) concluded that food irradiated to any dose appropriate to achieve the intended technological objective is both safe to consume and nutritionally adequate and that no upper dose limit has to be imposed.

Food that has been irradiated must be labeled as "irradiated" or "treated with ionizing radiation" together with the Radura symbol (Fig. 14.3) and the name of the product. The word "radura" derived from radurization, a term composed of the initial letters of the word "radiation" and the terms "durus" (Latin word for "hard" or "lasting") (Maherani et al. 2016). Generally, the symbol is green and resembles a plant in a circle that illustrates the rays from the sources (Fig. 14.3).

Currently, food irradiation is approved for using in more than 50 countries worldwide for over 60 products, including Australia, Belgium, Brazil, Canada, China, India, Russia, South Africa, Thailand, USA and Vietnam. In 2015, more than 700,000 tons of foods were irradiated (Eustice 2018), including spices, meat, fresh fruits and vegetables. Based on the *Codex* standards for the Labeling of Prepackaged Foods (CAC 1985), many countries have developed their own national regulations for irradiated food labeling and in most countries there is still no requirement to use the Radura symbol. Furthermore, the list of products allowed to be irradiated differs from country to country. For instance, in the European Union, the Directive 1999/3/EC only permits the irradiation of dried aromatic herbs, spices and vegetable seasonings (European Union 1999).

Fig. 14.3 The Radura symbol

14.5 Consumers' Acceptance

Perhaps consumers' acceptance is the most difficult barrier to cross on the use of irradiation, making their perception an interesting and important point to take into consideration. Although consumers are always interested in new technologies, the truth is that they confuse irradiated food with radioactive food that can cause damage to human health or environment (Ornellas et al. 2006; Junqueira-Gonçalves et al. 2011; Roberts 2014). Actually, the main worries of the consumers include safety, nutrition, detection and labeling of irradiated food. Even so, the public would consider consuming irradiated food if their benefits are explained or if they are informed about the use of "Radura" symbol, which is believed to transmit confidence and safety. In this way, an effort on the education of consumers has to be done, providing scientific and credible information in order to familiarize them with the principles, aims and benefits of irradiated products, which may lead to change their opinion (Maherani et al. 2016).

14.6 Effects of Irradiation on Food Bioactives

Bioactive compounds are phytochemicals naturally found in fruits, vegetables or whole grains that may provide beneficial health effects, including antioxidant, anti-inflammatory, antimicrobial, anticancer and immunomodulatory activities (Kris-Etherton et al. 2002; Shashirekha et al. 2015). They include different classes of compounds, such as polyphenols, carotenoids, tocopherols, phytosterols, organosulfur compounds, fatty acids, betalains, essential oils (terpenes) and alkaloids. They have different chemical structures and solubility (hydrophilic or lipophilic), distribution in nature, range of concentrations in foods, bioavailability in the human body, sites of action, effectiveness against harmful species, specificity and biological action.

When food is exposed to ionizing radiation, some primary effects are induced in food matrices due to the presence of water molecules, via ionization and excitation, which exponentially increase by the secondary action of the free radicals formed during this process. Due to the high water contents in food, the products that are formed during water radiolysis are considered the main responsible for the potential effects on food composition. These products include several chemical species: hydrated electrons (e^-_{aq}), hydroxyl radicals ($HO^\bullet$), hydrogen radicals ($H^\bullet$), excited water molecules (H_2O^*), ionized water molecules (H_2O^+), hydrogen peroxide (H_2O_2) and diatomic hydrogen molecules (H_2) (Le Caër 2011). The occurring modifications in atoms and molecules are known as the primary or direct effects of radiation, which result in the formation of new chemical compounds and free radicals, both chemically unstable and reactive. These radical species can interact with themselves and/or continue to react with other food components, that could lead to the formation of new compounds that are not present in non-irradiated food (Ferreira et al.

2018). These new reaction products can also interact with the above-mentioned free radicals, representing the secondary or indirect chemical effects of radiation processing (Ferreira et al. 2018).

The irradiation can alter and/or improve the chemical components on food, enhance the extractability of some molecules and change its bioactivity, and these changes may depend on the irradiation conditions (water content, temperature, pH, dose and dose rate). Furthermore, the effects induced by ionizing radiation on the bioactive compounds are also dependent on the food composition. The observed increase in the extractability of compounds can be explained by changes in cellular structures, namely by the depolymerization and dissolution of the cell wall polysaccharides by irradiation (Harrison and Were 2007; Behgar et al. 2011). Thus, the improvement of total phenolic and/or flavonoid contents on irradiated samples can be related with their release from matrix structures, increasing extractability of certain molecules, but also to the degradation of larger compounds into smaller ones by irradiation. The degradation of ascorbic acid, an important vitamin found in different fruits, usually observed after food irradiation can be easily attributed to the reversible oxidation of ascorbic acid to dehydroascorbic acid that can be further hydrolyzed and oxidized irreversibly to 2,3-diketogulonic acid (Deutsch 2000). Oppositely, the increase in the antioxidant activity sometimes produced after irradiation can be associated to an enhancement in enzymes activity (e.g., phenylalanine ammonia-lyase or peroxidase). For instance, stimulation of phenylalanine ammonia-lyase activity may promote the accumulation of anthocyanins, flavonoids and other phenolic compounds (Given et al. 1988; Oufedjikh et al. 2000). It is also pertinent to mention that the increase or decrease in the extraction yield is strongly dependent on the solvents used for the extraction (Pérez et al. 2007; Khattak et al. 2008).

The effects induced by ionizing radiation on food bioactive compounds have been studied for many years and will be reviewed on this section.

14.6.1 Fruits and Vegetables

Fruits and vegetables are colorful, flavorful and nutritious components rich in bioactive compounds, such as polyphenols, carotenoids, vitamins, phytoestrogens, glucosinolates and anthocyanins. Carotenoids and anthocyanins are also important compounds as they are pigments that impart the color to fruits and vegetables.

Some studies have reported the effects of gamma and electron-beam radiation on the major compounds of berry fruits. Barkaoui et al. (2020) compared the impact of gamma and electron-beam radiation on the antioxidants of strawberries (*Fragaria* × *ananassa*), being both treatments capable of preserving the phenolic content and increasing the antioxidant activity of the fruits at 2 kGy. On the other hand, both treatments induced a significant reduction on the amount of L-ascorbic acid (vitamin C). In fact, it is documented that ascorbic acid of berry fruits is sensitive to irradiation (Hussain et al. 2012; Tezotto-Uliana et al. 2013; Elias et al. 2020) and

can be oxidized to dehydroascorbic and, consequently, to 2,3-diketogulonic acid (Deutsch 2000). Maraei and Elsawy (2017) reported an increase of phenolic and anthocyanin contents and antioxidant activity in strawberries irradiated at 600 Gy. Furthermore, gamma irradiation at 0.5 kGy was observed to increase the concentration of anthocyanins (Tezotto-Uliana et al. 2013) and doses up to 2 kGy enhanced the phenolic content and antioxidant activity (Cabo Verde et al. 2013; Guimarães et al. 2013) in raspberries (*Rubus idaeus* L.). Electron-beam radiation at 3 kGy preserved the phenolic content and antioxidant activity of raspberries (Elias et al. 2020), and the total anthocyanin content, the antioxidant activity and ascorbic acid amount in blueberries (*Vaccinium corymbosum*, cvs. Collins, Bluecrop) (Kong et al. 2014).

Different effects of ionizing radiation have been found in tomatoes (*Solanum lycopersicum* var. cerasiforme) depending on the variety and/or the harvest conditions. Guerreiro et al. (2016) reported slight increases on total phenolic content of cherry tomatoes irradiated at 3.2 kGy, while a decrease in the concentration of individual phenolic compounds was observed in round tomato (Schindler et al. 2005), not affecting the anthocyanin content (Singh et al. 2016). A decrease in the levels of lycopene, the most representative carotenoid in tomatoes, was reported with 1.5 kGy electron-beam doses (Madureira et al. 2019), which was associated to the isomerization of lycopene. On the other hand, fluctuations in the content of lycopene were observed by Loro et al. (2018) in tomatoes, with increasing and decreasing values at doses up to 1.5 kGy.

Najafabadi et al. (2017) found that gamma radiation at doses up to 2.5 kGy not only increased the content of the total phenol and anthocyanins, but also improved the amount of vitamin C in jujube (*Ziziphus jujuba* var. vulgaris) fruit, while other water-soluble vitamins, as folic acid, thiamine (B_1) and pyridoxine (B_6), decreased significantly at this absorbed dose. Concerning mangoes (*Mangifera indica* L.), the studies developed by Reyes and Cisneros-Zevallos (2007) suggested that electron-beam radiation at doses up to 3.1 kGy did not affect the total phenolic and total carotenoid contents or the antioxidant capacity even after 18 days of storage, despite the observed increase in the levels of flavonols and phenolic acids and the decrease in ascorbic acid content. Other authors reported an increase in phenolic and carotenoid compounds and a decrease in ascorbic acid concentration in stored mangoes irradiated (1–1.5 kGy) with gamma (El-Samahy et al. 2000) and electron-beam (Moreno et al. 2007) radiation. It was also verified that gamma radiation (1–2 kGy) enhanced the antioxidant properties of peach (*Prunus persica* Bausch, Cv. Elberta), due to the increase in total phenolic content via enhancement of phenylalanine ammonia-lyase (PAL) activity (Hussain et al. 2010), despite a reduction of the ascorbic acid concentration. As for irradiated chestnuts (*Castanea sativa* Mill.), it was reported that electron-beam and gamma radiation could preserve total phenolics but not flavonoid content, and increase the antioxidant potential of the fruit, at 1 and 3 kGy, respectively (Carocho et al. 2012). In another study performed by Carocho et al. (2014), 1 kGy absorbed dose was described as capable of preserving phenolic profile and antioxidant properties of chestnuts, leading to higher values of tocopherols and β-carotene bleaching inhibition.

No antioxidant activity assay exactly reflects the antioxidant capacity of the samples. In fact, it is important to assess the antioxidant potential using methods that take into account different mechanisms of action (Moharram and Youssef 2014; Shahidi and Zhong 2015), including, among others, hydrogen atom transfer (HAT), single electron transfer (SET), reducing power and metal chelation. In a study performed by Kavitha et al. (2015) different tendencies were obtained in the evaluation of antioxidant activity of *Zizyphus mauritiana* Lam. fruit using different methodologies. A significant rise was observed in total flavonoids, 1,1-diphenyl-2-picrylhydrazil (DPPH) scavenging activity and super oxide anion radical scavenging activity, while reducing power, total phenolic content and antioxidant activity measured by thiobarbituric acid reactive substances (TBARS) assay decreased with increasing irradiation doses (0.25–1.0 kGy).

In dry fruits, it was demonstrated that gamma irradiation at 5 kGy was capable of improving the contents of phenolic (>40%) and flavonoid (>56%) compounds, the DPPH scavenging activity (>18%) and the antibacterial potentials of two genotypes of Egyptian date palm fruits, *Phoenix dactylifera* L. (El-Beltagi et al. 2019). Sun dried apricots (*Prunus armeniaca* L.) also underwent a significant increase of total phenols and flavonoids, β-carotene and antioxidant activity after gamma radiation at a dose of 3 kGy (Hussain et al. 2011, 2013). Those authors also analyzed the effect of gamma radiation on individual phenolic acids and flavonoids, observing significant increases in the concentrations of gallic acid (26%), ellagic acid (24%), quercetin (26%) and apigenin (37%) induced by the treatment (Hussain et al. 2013).

The effect of gamma radiation was also evaluated in fresh green vegetables, such as watercress (*Nasturtium officinale* R. Br.) (Pinela et al. 2016, 2018), fenugreek (*Trigonella foenum–graceum* L.) and spinach (*Spinacia oleracea* L.). Gamma radiation at 5 kGy combined with modified atmosphere packaging (MAP) induced an increase on monounsaturated fatty acids (MUFA), tocopherols and total phenolic acids (Pinela et al. 2016, 2018). An increase in total phenols, flavonoids and carotenoids was observed for fenugreek and spinach at doses above 0.75 kGy, which were responsible for increasing the antioxidant activity (Hussain et al. 2016). In spinach, after 1.5 kGy irradiation dose, total phenols, flavonoids and carotenoids increased 3.7%, 15.1% and 21.7%, respectively. Concerning the irradiated fenugreek samples, increases of 2.1%, 3.3% and 8.4% in the concentrations were observed for phenols, flavonoids and carotenoids, respectively. A decrease in the ascorbic acid content and increase on dehydroascorbic acid was also verified after irradiation of both vegetables. Fan (2005) reported that irradiation up to 2 kGy increased the phenolic content and antioxidant capacity of endive (*Cichorium endiva* L), Romaine and Iceberg lettuce (*Lactuca sativa* L) further stored at 7–8 °C for 8 days. After that time, and comparing with non-irradiated samples, the phenolic content increased by 40%, 60% and 25% for Romaine and Iceberg lettuce and endive leaf tissues, respectively. Concerning the antioxidant activity, the increases were 52%, 88% and 34% for Romaine and Iceberg lettuce and endive leaf tissues, respectively. On the other hand, irradiation of baby carrots induced a reduction in the phenolic content of 20% at 1 kGy gamma radiation dose (Hirashima et al. 2013).

Table 14.2 summarizes the documented effects of irradiation on bioactive compounds in fruits and vegetables.

Table 14.2 Effects of ionizing radiation on bioactive compounds in fruits and vegetables

Fruit/vegetable	Radiation source	Applied doses	Main results	References
Fragaria × *ananassa*	γ-radiation	1, 2 and 3 kGy	Preservation of phenolic content; increase of DDPH scavenging activity at 2 kGy; degradation of L-ascorbic acid	Barkaoui et al. (2020)
	e-beam	1, 2 and 3 kGy	Preservation of phenolic compounds; increase of DPPH scavenging activity at 2 kGy; higher reducing power at 1 and 3 kGy; degradation of L-ascorbic acid	
	γ-radiation	300, 600 and 900 Gy	Increase of phenolic and anthocyanin contents and antioxidant activity at 600 Gy and during storage; degradation of ascorbic acid	Maraei and Elsawy (2017)
Rubus idaeus L.	γ-radiation	0.5, 1.0 and 2.0 kGy	Decrease of ascorbic acid levels; increase of anthocyanin content at 0.5 kGy	Tezotto-Uliana et al. (2013)
		0.5, 1.0 and 1.5 kGy	Higher values of phenolics and antioxidant activity at 1.5 kGy	Cabo Verde et al. (2013)
		0.5, 1.0, and 2.0 kGy	Increase of phenolic content and antioxidant activity at 2 kGy after 12 days of cold storage; variable tendency of ascorbic acid depending on the absorbed dose	Guimarães et al. (2013)
	e-beam	3 kGy	Preservation of phenolic content and antioxidant activity; loss of ascorbic acid	Elias et al. (2020)
Vaccinium corymbosum, cvs. Collins, Bluecrop	e-beam	0.5, 1, 2 and 3 kGy	Preservation of anthocyanins after treatment and storage; no significant difference on antioxidant activity and ascorbic acid between non-treated and treated samples, although decreasing with storage	Kong et al. (2014)
Solanum lycopersicum var. cerasiforme	γ-radiation	1.3, 3.2 and 5.7 kGy	Slight increase of phenolic content at 3.2 kGy	Guerreiro et al. (2016)
	e-beam	1.5 and 3.1 kGy	Decrease of lycopene content at 1.5 kGy and preservation of DPPH scavenging activity	Madureira et al. (2019)
Lycopersicon esculentum Mill.	γ-radiation	0.5, 0.75, 1.0, 1.5, 2.0, 3.0 and 4.0 kGy	Preservation of anthocyanins content	Singh et al. (2016)

(continued)

Table 14.2 (continued)

Fruit/vegetable	Radiation source	Applied doses	Main results	References
		0.5, 1.0 and 1.5 kGy	Preservation of ascorbic acid up to 1.5 kGy, although degradation with storage; increase of lycopene at 0.5 kGy	Loro et al. (2018)
		2, 4 and 6 kGy	Radiolytic degradation of ferulic acid, *p*-coumaric acid, rutin and naringenin	Schindler et al. (2005)
Ziziphus jujuba var. vulgaris	γ-radiation	0.5, 1.0, 2.5 and 5.0 kGy	Significant increase in total monomeric anthocyanin (~12%) and total phenolic contents (~6%) up to 2.5 kGy; significant decrease in both parameters at 5 kGy; increase of vitamin C content at 2.5 kGy	Najafabadi et al. (2017)
	e-beam	1, 1.5, and 3.1 kGy	Preservation of total phenolic content, carotenoid content and antioxidant capacity even after 18 days of storage; increase in flavonols and phenolic acids; decrease of ascorbic acid content	Reyes and Cisneros-Zevallos (2007)
Mangifera indica L.	e-beam	1.0, 1.5 and 3.0 kGy	Significant increase of phenolic (55%) and carotenoids (91%) concentrations and antioxidant activity (6%) at 1 kGy	Moreno et al. (2007)
	γ-radiation	0.5, 0.75, 1.0 and 1.5 kGy	Increase of phenolic content with increasing absorbed doses and storage time; increase in carotenoid concentrations with storage, in particular, at 1.5 kGy; slight decrease in ascorbic acid concentration with radiation	El-Samahy et al. (2000)
Prunus persica Bausch, Cv. Elberta	γ-radiation	1.0, 1.2, 1.4, 1.6, 1.8 and 2.0 kGy	Increase of phenolic and anthocyanin contents and PAL activity with irradiation dose and storage until 21 days; reduction of ascorbic acid content with doses higher than 1.6 kGy; improvement of DPPH scavenging activity and FRAP with radiation	Hussain et al. (2010)
Castanea sativa Mill.	γ-radiation	0.5, 1 and 3 kGy	Increase of phenolics (53%), DPPH scavenging activity (71%), β-carotene bleaching inhibition (61%) and TBARS inhibition (83%) at 3 kGy; decrease in flavonoids content (65%) at 1 kGy	Carocho et al. (2012)

(continued)

14.6.2 Beverages

Table 14.2 (continued)

Fruit/vegetable	Radiation source	Applied doses	Main results	References
	e-beam	0.5, 1 and 3 kGy	Increase of phenolics (126%), DPPH scavenging activity (37%), reducing power (60%), β-carotene bleaching inhibition (67%) and TBARS inhibition (84%) at 1 kGy; decrease of flavonoids content (87%) at 1 kGy	
	γ-radiation	1 kGy	Increase of tocopherols, namely γ-tocopherol; increase of β-carotene bleaching inhibition	Carocho et al. (2014)
	e-beam	1 kGy	Increase of α- and γ-tocopherols; increase of β-carotene bleaching inhibition	
Zizyphus mauritiana Lam.	γ-radiation	0.25, 0.5, 0.75 and 1 kGy	Increase of total flavonoid and decrease of total phenolic contents at doses up to 1 kGy; increase of DPPH scavenging activity and reduction of reducing power with increasing doses	Kavitha et al. (2015)
Dried *Phoenix dactylifera* L. fruits	γ-radiation	2.5, 5.0 and 10.0 kGy	Improvement of phenolic (>40%), flavonoid (>56%) contents, DPPH scavenging activity (>18%) and antibacterial potential at 5 kGy	El-Beltagi et al. (2019)
Dried *Prunus armeniaca* L. fruits	γ-radiation	1.0, 1.5, 2.0, 2.5 and 3.0 kGy	Linear increase of β-carotene with increasing absorbed doses	Hussain et al. (2011)
		3 kGy	Significant increase of total phenolics (12%), total flavonoids (16%) and β-carotene (37%); increase in DPPH scavenging activity (23%), FRAP (14%) and β-carotene bleaching inhibition (74%); enhancement of gallic acid (26%), ellagic acid (24%), quercetin (26%) and apigenin (37%) concentrations	Hussain et al. (2013)
Nasturtium officinale R. Br.	γ-radiation	1, 2 and 5 kGy	Preservation of antioxidant activity and total flavonoids at 5 kGy; increase of MUFA, tocopherols and total phenolics at 5 kGy	Pinela et al. (2016)
Trigonella foenum–graceum L.	γ-radiation	0.25, 0.5, 0.75, 1.0, 1.25 and 1.5 kGy	Increase of total phenols (2.1%), flavonoids (3.3%) and carotenoids (8.4%) and antioxidant potential at 1.5 kGy; loss of ascorbic acid and increase in dehydroascorbic acid after irradiation	Hussain et al. (2016)

(continued)

Table 14.2 (continued)

Fruit/vegetable	Radiation source	Applied doses	Main results	References
Spinacia oleracea L.			Increase of total phenols (3.7%), flavonoids (15.1%) and carotenoids (21.7%) and antioxidant activity after 1.5 kGy; reduction of ascorbic acid content and increase in dehydroascorbic acid after irradiation	
Cichorium endiva L.	γ-radiation	0.5, 1 and 2 kGy	Increase of phenolic content (25%) and antioxidant capacity (34%) at 2 kGy, after 8 days of storage	Fan (2005)
Romaine *Lactuca sativa* L.			Increase of phenolic content (40%) and antioxidant capacity (52%) at 2 kGy, after 8 days of storage	
Iceberg *Lactuca sativa* L.			Increase of phenolic content (60%) and antioxidant capacity (88%) at 2 kGy, after 8 days of storage	
Baby carrots	γ-radiation	0.5 and 1 kGy	20% reduction of phenolic content at 1 kGy	Hirashima et al. (2013)

Notes: PAL (Phenylalanine ammonia-lyase); DPPH (2,2-Diphenyl-1-picrylhydrazyl); FRAP (Ferric reducing power); TBARS (Thiobarbituric acid reactive substances); MUFA (Monounsaturated fatty acids)

Juices from vegetables (Song et al. 2006; Lee et al. 2009) and fruits (Girennavar et al. 2008; McDonald et al. 2013; Eissa et al. 2014; Shahbaz et al. 2014; Arjeh et al. 2015; Naresh et al. 2015) showed interesting results when subjected to radiation. In carrot (*Daucus carota* var. *sativa*) juices, after storage of 3 days, an increase in the phenolic content (1912.5 μg/mL of sample) and FRAP value (1349.7 mM FRAP/mL of sample) were observed in irradiated samples when compared to the non-irradiated and non-stored sample (1732.9 μg/mL of sample and 931.3 mM FRAP/mL, respectively, for phenolic and FRAP values) (Song et al. 2006). On the contrary, the levels of total phenolics (9121.7 μg/mL of sample) and antioxidant potential value (5776.2 mM FRAP/mL of sample) in irradiated kale (*Brassica oleracea* var. *acephala*) juice were significantly lower than the control (9635.5 μg/mL and 6251.8 mM FRAP/mL, respectively) (Song et al. 2006), and flavonoid content did not change (Jo et al. 2012). In irradiated tamarind (*Tamarindus indica* L.) juice, higher phenolic content and antioxidant activity were found at 5 kGy (Lee et al. 2009).

Low doses (up to 600 Gy) of gamma radiation caused no effect on ascorbic acid, phenolic content and antioxidant activity of orange juice (*Citrus sinensis* L. Osbeck) (McDonald et al. 2013). In watermelon (*Citrullus lanatus* cv.) and mango (*Mangifera indica* L.) juices (fresh and stored) irradiated at 3 and 5 kGy, both total phenolics and flavonoids contents were higher (15-48%) than in non-irradiated samples, although vitamin C and lycopene contents were lower (Eissa et al. 2014; Naresh et al. 2015). An enhancement in the antioxidant activity in both irradiated juices was also observed with increasing irradiation dose. Furthermore, gamma radiation

induced increases in the levels of individual phenolic acids present in mango juice, as gallic acid, chlorogenic acid and syringic acid by 3.2, 2.3 and 2.5 fold, respectively (Naresh et al. 2015). Shahbaz et al. (2014) observed a decrease in DPPH and ABTS scavenging activities probably associated with a reduction on anthocyanins and total phenolic contents of irradiated (1–2 kGy) pomegranate (*Punica granatum* L.) juice. The same observations were made by Arjeh et al. (2015) for sour cherry (*Prunus cerasus* L.) juice, with reductions in monomeric anthocyanins of more than 60% at 3 kGy after 60 days. In a study performed on grapefruit (*Citrus paradisi* Macf.) irradiated at doses ranging from 1 to 10 kGy and further juiced, electron-beam radiation was found to significantly reduce the vitamin C and lycopene contents, as well as those of nomilin (a terpenoid limonoid) and dihydroxybergamottin (a furanomcoumarin); on the other hand, naringin level was significantly higher in irradiated juice in comparison with the control sample (Girennavar et al. 2008).

Teas are the most widely consumed beverages in the world. In black tea, made from leaves of *Camellia sinensis* (L.) O. Kuntze, gamma radiation up to 2 kGy increased the total phenolic content, with lower water activity having positive influence on the obtained results (Fanaro et al. 2014). Gerolis et al. (2017) studied the effect of gamma radiation on total antioxidant capacity of green tea (*Camellia sinensis* (L.) O. Kuntze), yerba mate (*Ilex paraguariensis* A. St. Hil.) and chamomile tea (*Matricaria recutita* L.), reporting that this technology can reduce the capacity of some antioxidants and improve the capacity of others, which depends on the compounds present, solvents used and also the method of assessment. In another study developed by Janiak et al. (2017), different Bulgarian teas were studied: Mursalski tea (*Sideritis scardica* Gris), Mashterka tea (*Thymus serpyllum* L.), Good Night tea (tea mix), Staroplaninski tea (Balkan tea mix), Trakia tea (tea mix), and Mountain tea (Planinski tea mix). The authors observed improvement in tannin and phenolic contents and antioxidant capacity in some teas but not in others. Tables 14.3 and 14.4 outline the reported effects of ionizing radiation on the bioactivity of beverages.

14.6.3 Meat

Meat is widely consumed as a main source of proteins, vitamins, minerals and fatty acids. Losses of α-tocopherol and thiamine and riboflavin were observed in irradiated beef, lamb, pork and turkey with doses up to 9 kGy (Fox et al. 1995; Lakritz et al. 1995). On the other hand, significant higher levels of riboflavin and niacin were detected in pork chops and chicken breasts irradiated at the dose range 2–4 kGy (Fox et al. 1989). Fatty acids present in beef and ground beef were also significantly increased at doses up to 7 kGy (Yilmaz and Geçgel 2007; Haque et al. 2017). On Table 14.5 the reported effects of irradiation on the bioactives content of meat are collected.

Table 14.3 Effects of ionizing radiation on the bioactivity of beverages

Fruit/ vegetable beverage	Radiation source	Applied doses	Main results	References
Daucus carota var. *sativa* juice	γ-radiation	3 and 5 kGy	Significant increase of phenolic content and FRAP value in irradiated samples, even after 3 days of storage	Song et al. (2006)
Brassica oleracea var. *acephala* juice			Lower phenolic content and FRAP value in irradiated samples	
		1, 3, and 5 kGy	Slight increase of phenolic content with irradiation; no variation of flavonoid content; reduction of ascorbic acid content with irradiation	Jo et al. (2012)
Angelica keiskei Ito juice	γ-radiation	1, 3, and 5 kGy	Slight increase of phenolic content with irradiation; no variation of flavonoid content; reduction of ascorbic acid content with irradiation	Jo et al. (2012)
Tamarindus indica L. juice	γ-radiation	1, 3, and 5 kGy	Higher total phenolic content and antioxidant activity by DPPH and FRAP at 3 and 5 kGy	Lee et al. (2009)
Citrus sinensis L. Osbeck juice	γ-radiation	200, 400 and 600 Gy	No effect on ascorbic acid, phenolic content and antioxidant activity.	McDonald et al. (2013)
Citrullus lanatus cv. juice	γ-radiation	1, 3 and 5 kGy	Increase of phenolics and flavonoids contents with irradiation; reduction of vitamin C at 3 and 5 kGy; lower lycopene content at 1 kGy; enhancement of DPPH scavenging activity	Eissa et al. (2014)
Mangifera indica L. juice	γ-radiation	1, 3 and 5 kGy	Increase of total phenolic content with increasing doses; increase of total flavonoids at 3 and 5 kGy; decrease of ascorbic acid concentration with increasing doses; increase of DPPH scavenging activity with doses; enhancement in the concentrations of individual phenolic at 3 and 5 kGy: gallic acid, protocatechuic acid, syringic acid, chlorogenic acid, and rutin, and ellagic acid and *p*-coumaric acid at 5 kGy	Naresh et al. (2015)
Punica granatum L. juice	γ-radiation	0.4, 1, and 2 kGy	Decrease in total phenolic compounds, DPPH and ABTS scavenging abilities at 1 and 2 kGy	Shahbaz et al. (2014)

(continued)

Table 14.3 (continued)

Fruit/ vegetable beverage	Radiation source	Applied doses	Main results	References
Prunus cerasus L. juice	γ-radiation	0.5, 1.5, 3.0, 4.5, and 6.0 kGy	No significant difference in total phenolic content with irradiation; reduction of monomeric anthocyanins (>60%) at 3 kGy after 60 days; decrease of DPPH scavenging activity at doses >3 kGy (21%) and storage (29%); decrease in FRAP values by 20% after irradiation and 27% after storage	Arjeh et al. (2015)
Citrus paradisi Macf. juice	e-beam	1.0, 2.5, 5.0, and 10.0 kGy	Significant reduction of the vitamin C and lycopene contents at doses >2.5 kGy; preservation of β-carotene; decrease of nomilin and dihydroxybergamottin contents; increase of naringin at doses >2.5 kGy	Girennavar et al. (2008)

Notes: PAL (Phenylalanine ammonia-lyase); DPPH (2,2-Diphenyl-1-picrylhydrazyl); FRAP (Ferric reducing power); TBARS (Thiobarbituric acid reactive substances); MUFA (Monounsaturated fatty acids); ABTS (2,2′-Azinobis-(3-ethylbenzthiazolin-6-sulfonic acid)

14.6.4 Aromatic and Medicinal Plants

The effect of ionizing radiation on the phytochemicals can vary according to the plant material and the applied dose. Pereira and co-workers showed that gamma radiation at 10 kGy increased significantly (>1-fold) the content of polyphenols in ethanolic extracts and infusions of *Mentha* × *piperita* L. (peppermint), *Aloysia citrodora* Paláu (lemon verbena) and *Thymus vulgaris* L. (thyme) in comparison with the control sample (0 kGy) (Pereira et al. 2017, 2018).

In other studies, the same authors observed that an absorbed dose of 10 kGy of gamma radiation could improve the extractability of phenolic compounds (3.5–5.5 fold) and their antioxidant properties in both infusions and methanol/water extracts of *Ginkgo biloba* L. (Pereira et al. 2015a, b). An increase of 35% in total phenolic content was reported in water extracts of *Rosmarinus officinalis* L. irradiated at 30 kGy, but not in methanol or ethanol extracts (Pérez et al. 2007). The authors hypothesized that this increase could be associated with the presence of phenolic diterpenes in rosemary that result in water-soluble quinone-type compounds by gamma radiation. A previous study developed by Horváthová et al. (2007) revealed a slight increase in the total phenolic content of *Origanum vulgare* L. extracts prepared from samples irradiated at 10 kGy. Gamma irradiation of *Petroselinum crispum* (Mill.) Fuss var. neapolitanum induced a reduction in vitamin C content at 2.7 kGy, while increased total polyphenols in doses up to 2.0 kGy (Cătunescu et al. 2017). By contrast, *Salvia officinalis* L. gamma irradiated at 2 and 4 kGy demonstrated lower antioxidant capacity associated to a lower polyphenolic content (30 and 45%, respectively) (Salem et al. 2013).

Table 14.4 Effects of ionizing radiation on teas

Tea	Radiation source	Applied doses	Main results	References
Camellia sinensis L. O. Kuntze	γ-radiation	1.0, 1.5, 2.0, 2.5, 5.0, 7.5 and 10.0 kGy	Increase of total phenolic compounds at 2 kGy (for 0.183 and 0.651 water activity) or at 5 kGy (for 0.924 water activity); preservation of antioxidant activity; increase of caffeine concentration at 10 kGy	Fanaro et al. (2014)
		20 kGy	Decrease of phenolics level; decrease of flavonoids contents in methanolic extracts; no significant difference in ABTS activity between irradiated and non-irradiated samples	Gerolis et al. (2017)
Ilex paraguariensis A. St. Hil.	γ-radiation	20 kGy	Decrease of phenolics content; preservation of flavonoids content; no significant difference in ABTS activity; decrease of DPHH scavenging activity	
Matricaria recutita L.	γ-radiation	20 kGy	Preservation of phenolics content; decrease of flavonoids content in methanolic extracts; increase of β-carotene bleaching inhibition of water infusions with irradiation; decrease of DPPH scavenging activity	
Mursalski tea (*Sideritis scardica* Gris)	γ-radiation	5 kGy	Improvement of FRAP values and ABTS scavenging activity with irradiation	Janiak et al. (2017)
Mashterka tea (*Thymus serpyllum* L.)	γ-radiation		Increase of DPPH scavenging activity in irradiated samples	
Good Night tea	γ-radiation		Preservation of phenolics content and antioxidant activity	
Staroplaninski tea	γ-radiation		Increase of DPPH scavenging activity with irradiation; improvement of FRAP values and ABTS scavenging activity with irradiation	
Trakia tea	γ-radiation		Increase of total phenolics and tannins contents; improvement of FRAP values and ABTS scavenging activity with irradiation	
Mountain tea	γ-radiation		Increase of tannins content; improvement of FRAP values and ABTS scavenging activity with irradiation	

Notes: DPPH (2,2-Diphenyl-1-picrylhydrazyl); FRAP (Ferric reducing power); ABTS (2,2′-Azinobis-(3-Ethylbenzthiazolin-6-sulfonic acid)

Table 14.5 Effects of ionizing radiation on bioactive compounds of meat

Meat	Radiation source	Applied doses	Main results	References
Beef	γ-radiation	2, 4 and 6 kGy	Increase of free fatty acids with irradiation	Haque et al. (2017)
		0.24, 0.47, 0.94, 1.88, 2.81, 5.62 and 9.37 kGy	Loss of α-tocopherol	Lakritz et al. (1995)
			Reduction of thiamine and riboflavin levels with irradiation	Fox et al. (1995)
Ground beef	γ-radiation	1, 3, 5 and 7 kGy	Significant increase of *trans* fatty acids with irradiation	Yilmaz and Geçgel (2007)
Turkey	γ-radiation	0.24, 0.47, 0.94, 1.88, 2.81, 5.62 and 9.37 kGy	Loss of α-tocopherol	Lakritz et al. (1995)
			Reduction of thiamine and riboflavin levels with irradiation	Fox et al. (1995)
Pork	γ-radiation	0.24, 0.47, 0.94, 1.88, 2.81, 5.62 and 9.37 kGy	Loss of α-tocopherol	Lakritz et al. (1995)
			Reduction of thiamine and riboflavin levels with irradiation	Fox et al. (1995)
		0.5, 1.75, 3.50, 5.25 and 7.0 kGy	Loss of thiamine with increasing doses; increase of riboflavin and niacin levels at 4 kGy	Fox et al. (1989)
Lamb	γ-radiation	0.24, 0.47, 0.94, 1.88, 2.81, 5.62 and 9.37 kGy	Loss of α-tocopherol	Lakritz et al. (1995)
Chicken	γ-radiation	0.5, 1.75, 3.50, 5.25 and 7.0 kGy	Loss of thiamine with increasing doses; increase of riboflavin and niacin levels at 4 kGy	Fox et al. (1989)
		0.24, 0.47, 0.94, 1.88, 2.81, 5.62 and 9.37 kGy	Reduction of thiamine and riboflavin levels with irradiation	Fox et al. (1995)

Concerning medicinal plants, Pereira et al. (2014) observed higher contents of phenolics (107.45 mg GAE/g) and flavonoids (and 33.77 mg CE/g) in the methanolic extracts of medicinal plant borututu (*Cochlospermum angolensis* Welw.) irradiated at 10 kGy, which was correlated with an increase in the antioxidant activity. Similarly, the scavenging activity of *Amoora rohitaka* ethanolic extracts improved by 112%, while the phenolic content and reducing power of methanolic extracts was enhanced by more than 30% at 5 kGy (Rajurkar and Gaikwad 2012). A summary of the results regarding bioactive compounds in irradiated aromatic and medicinal plants is collected in Table 14.6.

14.6.5 Legumes, Cereals and Grains

Legumes are an important group of plant foodstuffs, especially in developing countries, being recognized as a cheap source of protein. Zhu et al. (2010) evaluated the effect of gamma radiation (2–10 kGy) on the phenolic compounds of three different genotypes (black, red and white) of *Oryza sativa* L. grains. The authors reported an increase of phenolic acids content in black rice extracts irradiated at 8 kGy (423.3 mg/kg) compared with the control (381.6 mg/kg), although lower doses promoted a decrease on these acids. On the other hand, irradiation at 6 kGy caused a significant increase (378.3 mg/kg) in anthocyanins content, in comparison with the control (346.6 mg/kg). Similarly, distinct effects of irradiation were observed in different genotypes of rice grains depending on the analyzed variable and absorbed doses (Shao et al. 2013).

The extraction of phenolic compounds from *Nigella sativa* L. seeds using different solvents was evaluated by Khattak et al. (2008). The authors reported that a radiation dose up to 16 kGy improved total extraction yields (3.7%, 4.2%, 9.0% and 5.6% for hexane, acetone, methanol and water, respectively), phenolic contents (2.7% in acetone extracts) and DPPH scavenging activity (10.6% and 5.4% in acetone and methanol extracts, respectively). In a study conducted by Bhat et al. (2007), the levels of total phenolics of *Mucuna pruriens* L. seeds were found to be dose-dependent, significantly increasing at doses higher than 2.5 kGy (73.4 g/kg and 116 g/kg for control and 30 kGy, respectively) while doses higher than 7.5 kGy resulted in a tannin concentration increase (2.62 g/kg for control and 5.81 g/kg for 30 kGy). Also, Siddhuraju et al. (2002) found that irradiation (2–6 kGy) after aqueous soaking increased (>1-fold) the phenolics levels in seeds of three different species of *Sesbania* (*Sesbania aculeata*, *S. rostrata* and *S. cannabina*) and one species of *Vigna* (*Vigna radiata*), attributing this increase to a higher extractability by depolymerization and dissolution of cell wall polysaccharides by irradiation.

Concerning the irradiation of soybeans (*Glycine max* (L.) Merr.), an absorbed dose of 1 kGy was enough to increase total phenolics (10%) and tannins (21.6%), which was correlated with an increase in the antioxidant activity (Štajner et al. 2007). Also, the total isoflavone content (genistein, daidzein, genistin and daidzin) increased at an applied dose of 10 kGy (666.1 mg/kg for control sample and 755.2 mg/kg for 10 kGy sample) (Popović et al. 2013). Variyar et al. (2004) observed an increase higher than onefold in aglycon content with the increase in absorbed doses (0.5–5 kGy) and consequent increase in DPPH scavenging activity, associated with the higher levels of isoflavones in treated samples. Gamma radiation at 1 kGy applied in red kidney beans (*Phaseolus vulgaris*), slightly improved phenolic content and antioxidant activity by DPPH free radical scavenging assay and inhibition in lipid peroxidation, keeping constant even after 6 months of storage (Marathe et al. 2016). Table 14.7 summarizes the effects of irradiation on the bioactive compounds in legumes, cereals and other grains.

Table 14.6 Effects of ionizing radiation on bioactive compounds of aromatic and medicinal plants

Aromatic and medicinal plant	Radiation source	Applied doses	Main results	References
Aloysia citrodora L.	γ-radiation	1, 5 and 10 kGy	Increase of verbascoside at 1 kGy	Pereira et al. (2018)
Mentha × *piperita* L.			Increase of eriodictyol-7-*O*-rutinoside, luteolin-7-*O*- rutinoside and rosmarinic acid concentrations at 5 kGy	
Thymus vulgaris L.			Increase of luteolin-7-*O*-glucuronide, luteolin-*O*-glucuronide and eriodictyol-*O*-glucuronide at 5 kGy; increase of apigenin-6,8-*C*-dihexoside and eriodictyol-7-*O*-glucuronide at 10 kGy	
Ginkgo biloba L.	γ-radiation	1 and 10 kGy	Improvement of the extractability of phenolic acids and flavonoids at 10 kGy in infusions and methanol/water extracts.	Pereira et al. (2015b)
			Increase of α-tocopherol levels at 1 kGy; increase of antioxidant activity with irradiation	Pereira et al. (2015a)
Rosmarinus officinalis L.	γ-radiation	30 kGy	Increase of total phenolic content by 35% in water extracts after treatment; increase of antioxidant activity (DPPH scavenging and reducing power) in ethanol and water extracts	Pérez et al. (2007)
Origanum vulgare L.	γ-radiation	5, 10 and 30 kGy	Significant increase of total phenolic content at 10 kGy; preservation of DPPH scavenging activity	Horváthová et al. (2007)
Salvia officinalis L.	γ-radiation	2 and 4 kGy	Decrease of total phenolics (>30%) and antioxidant activity (>11%) with irradiation	Salem et al. (2013)
Petroselinum crispum (Mill.) Fuss Var. Neapolitanum	γ-radiation	0.7, 1.4, 2.0 and 2.7 kGy	Decrease of ascorbic acid with irradiation; increase of total polyphenols at 0.7 and 2 kGy; decrease in radical scavenging activity at 2.7 kGy	Cătunescu et al. (2017)

(continued)

Table 14.6 (continued)

Aromatic and medicinal plant	Radiation source	Applied doses	Main results	References
Cochlospermum angolensis Welw.	γ-radiation	1 and 10 kGy	Increase of tocopherols at 1 kGy; increase of phenolics and flavonoids at 10 kGy; higher antioxidant activity (DPPH scavenging activity, reducing power, β-carotene bleaching inhibition and TBARS inhibition) in infusions irradiated at 10 kGy; higher antioxidant activity (DPPH scavenging activity, reducing power and TBARS inhibition) in infusions irradiated at 10 kGy	Pereira et al. (2014)
Amoora rohitaka (Rxb.) Wight & Arn	γ-radiation	1, 3 and 5 kGy	Increase of total phenolics with irradiation; increase of ABTS scavenging activity; increase of FRAP values in methanol extracts up to 5 kGy; increase of DPPH scavenging activity in aqueous and ethanol extracts	Rajurkar and Gaikwad (2012)

Notes: DPPH (2,2-Diphenyl-1-picrylhydrazyl); ABTS (2,2′-Azinobis-(3-Ethylbenzthiazolin-6-sulfonic acid)

14.6.6 Spices

Even in small quantities, spices are a potential source of natural contamination by mesophylic, sporogenic, and asporogenic bacteria, hyphomycetes, and faecal coliforms into foodstuffs where they are added (Sádecká 2007). Hence, ionizing radiation treatment is mostly used in order to eliminate the microbial contamination. Furthermore, some studies were also performed reporting the impact of this technology on the antioxidant properties of different spices.

Variyar et al. (1998) studied the effect of gamma radiation (10 kGy) in five commercially important spices: cinnamon (*Cinnamomum verum* J. Presl.), clove (*Zyzygium aromaticum*), cardamom (*Elettaria cardamomum* (L.) Maton), nutmeg and mace (*Myristica fragrans* Houtt.). In clove, the concentrations of gallic acid and syringic acid increased considerably (384.9 ± 11.6 and 33.0 ± 4.7 mg/kg dry weight, respectively) in the 10 kGy sample in comparison with the control sample (174.7 ± 7.29 and 7.9 ± 1.3 mg/kg dry weight, respectively). In irradiated nutmeg, the concentrations of some observed phenolic acids, such as syringic acid, caffeic acid + vanillic acid, gentisic acid + *p*-hydroxybenzoic acid, also increased at 10 kGy irradiation dose (five-fold, three-fold and six-fold, respectively). The increased level of these phenolic acids in clove and nutmeg could be justified by the degradation of hydrolysable tannins and consequent higher extractability of phenolic acids.

The antioxidant properties of irradiated (5–30 kGy) black pepper (*Piper nigrum* L.), clove (*Syzygium aromaticum* (L.) Merr. & L.M.Perry) and ginger (*Zingiber*

Table 14.7 Effects of ionizing radiation on bioactive compounds of legumes, cereals and grains

Legume, cereal and grain	Radiation source	Applied doses	Main results	References
Black rice	γ-radiation	2, 4, 6, 8, and 10 kGy	Increase of phenolic acids content at 8 kGy; highest level of anthocyanins at 6 kGy	Zhu et al. (2010)
		2, 4, 6, 8, and 10 kGy	Increase of antioxidant activity with doses 2–8 kGy	Shao et al. (2013)
White rice	γ-radiation	2, 4, 6, 8, and 10 kGy	Decrease of phenolic acids content with irradiation	Zhu et al. (2010)
		2, 4, 6, 8, and 10 kGy	Increase of total phenolics at 10 kGy; significant increase of antioxidant activity at 10 kGy	Shao et al. (2013)
Red rice	γ-radiation	2, 4, 6, 8, and 10 kGy	Decrease of phenolic acids content with irradiation	Zhu et al. (2010)
		2, 4, 6, 8, and 10 kGy	Enhancement of total phenolics at >6 kGy; Significant increase of antioxidant activity at 8 kGy	Shao et al. (2013)
Nigella sativa L.	γ-radiation	2, 4, 8, 10, 12 and 16 kGy	Improvement in the extraction yields (3.7%, 4.2%, 9.0% and 5.6% for hexane, acetone, methanol and water, respectively) at doses up to 16 kGy; increase by 2.7% of phenolic contents at 16 kGy in acetone extracts; enhancement of DPPH scavenging activity by 10.6% in acetone and 5.4% in methanol extracts	Khattak et al. (2008)
Mucuna pruriens L.	γ-radiation	2.5, 5.0, 7.5, 10, 15 and 30 kGy	2-Fold increase of total phenolics for doses >2.5 kGy and tannins concentrations at doses >7.5 kGy	Bhat et al. (2007)
Sesbania aculeate (Willd.) Pers.	γ-radiation	2, 4 and 6 kGy	Increase of total phenolics at 2 kGy; preservation of tannins with irradiation	Siddhuraju et al. (2002)
Sesbania rostrate Bremek. & Oberm			Increase of total phenolics at 2 kGy; preservation of tannins with irradiation	
Sesbania cannabina (Retz.) Pers.			Preservation of total phenolics tannins with irradiation	
Vigna radiate (L.) R.Wilczek			Increase of total phenolics and tannins at 2 kGy; increase of condensed tannins with irradiation	

(continued)

Table 14.7 (continued)

Legume, cereal and grain	Radiation source	Applied doses	Main results	References
Glycine max (L.) Merr.	γ-radiation	1, 2, 4, 6, 8 and 10 kGy	Highest values of phenolic compounds and tannins at 1 kGy; increase (17%) of FRAP value at 1 kGy; increase of DPPH scavenging activity with increasing irradiation doses	Štajner et al. (2007)
		1, 2, 4 and 10 kGy	Significant increase of total isoflavone content at 10 kGy; significant increase of genistein, daidzein, genistin and daidzin concentrations at 4 kGy; significant increase of total phenolic and tannin contents with irradiation; increase of DPPH scavenging activity with irradiation	Popović et al. (2013)
		0.5, 1, and 5 kGy	Decrease of total isoflavones with irradiation; increase (>1-fold) of isoflavone aglycones content with the increasing of absorbed doses; significant increase in DPPH scavenging activity with irradiation	Variyar et al. (2004)
Phaseolus vulgaris L	γ-radiation	0.25, 1.0, 5.0 and 10.0 kGy	Increase of fatty acids at 10 kGy; increase of free phenolic compounds and antioxidant activity at 1 kGy	Marathe et al. (2016)

Notes: DPPH (2,2-Diphenyl-1-picrylhydrazyl); FRAP (Ferric reducing power)

officinale Roscoe) were also evaluated (Suhaj et al. 2006; Suhaj and Horváthová 2007). The results demonstrated that DPPH scavenging activity of black pepper extracts initially decreased with increasing doses, however, after 2 months of storage, a significant increase of those values occurred due to the increase of dry matter content during the storage. On the other hand, a decrease in the reducing power of these samples was observed with increasing dose and during storage (Suhaj et al. 2006). Suhaj and Horváthová (2007) did not detect variations in phenolic compounds, antiradical activity and reducing power of irradiated clove extracts, even after 5 months of storage, except for phenolic compounds, which were observed to decrease. Contrarily, in ginger, all the parameters (phenolics, antiradical activity and reducing power) kept constant immediately after irradiation but significantly increased by 10% with storage, in particular in those irradiated at 10 and 20 kGy. In Table 14.8 the main effects of irradiation on the bioactive compounds of spices are outlined.

14.6.7 Edible Flowers

Edible flowers have been used in many culinary preparations to improve the sensorial and nutritional qualities of food, by adding color, flavor, taste and visual appeal to salads, sauces, garnish, entrees, drinks or desserts. In this way, it is important to improve the conservation and safety of these products.

Viola tricolor L. (heartseases) and *Tropaeolum majus* L. (garden nasturtium) flowers irradiated by gamma and electron beam radiation at 1 kGy demonstrated higher capacity to scavenge DPPH and to inhibit β-carotene bleaching than not irradiated samples, attributed to an increase in the levels of phenolic compounds, despite the content of anthocyanins decreased (Koike et al. 2015a, b). In addition, the authors reported no significant differences in the observed effects between gamma and electron-beam irradiation, concluding that both technologies can be used to preserve the edible flowers quality (Table 14.9).

Table 14.8 Effects of ionizing radiation on bioactive compounds of spices

Spice	Radiation source	Applied doses	Main results	References
Cinnamomum verum J.Presl	γ-radiation	10 kGy	Increase of protocatechuic acid concentration	Variyar et al. (1998)
Zyzygium aromaticum			Significant increase of gallic acid and syringic acid concentrations; decrease of *p*-coumaric acid and ferulic acid + sinapic acid concentrations	
Elettaria cardamomum (L.) Maton			Preservation of phenolic acids contents	
Myristica fragrans Houtt.			Preservation of phenolic acids concentrations	
Nutmeg			Significant increase of gentisic acid + *p*-hydroxybenzoic acid, caffeic acid + vanillic acid and syringic acid concentrations	
Piper nigrum L.	γ-radiation	5, 7.5, 10, 20, and 30 kGy	Significant decrease of DPPH scavenging activity; decrease of reducing power with irradiation	Suhaj et al. (2006)
Syzygium aromaticum (L.) Merr. & L.M.Perry	γ-radiation	5, 10, 20 and 30 kGy	Preservation of DPPH scavenging activity, reducing power and total phenols	Suhaj and Horváthová (2007)
Zingiber officinale Roscoe			Preservation of DPPH scavenging activity and reducing power with irradiation; increase of DPPH scavenging activity during storage at 10 and 20 kGy	

Notes: DPPH (2,2-Diphenyl-1-picrylhydrazyl)

Table 14.9 Effects of ionizing radiation on the bioactive content of edible flowers

Edible flower	Radiation source	Doses applied	Main results	References
Viola tricolor L.	γ-radiation	0.5, 0.8 and 1 kGy	Higher amounts of phenolic compounds at 1 kGy; increase of DPPH scavenging activity and β-carotene bleaching inhibition at 1 kGy	Koike et al. (2015a)
	e-beam	0.5, 0.8 and 1 kGy	Higher amounts of phenolic compounds at 1 kGy; highest DPPH scavenging activity and β-carotene bleaching inhibition at 1 kGy	
Tropaeolum majus L.	γ-radiation	0.5, 0.8 and 1 kGy	Increase of non-anthocyanin phenolics and decrease of anthocyanin concentrations with irradiation; decrease of DPPH scavenging activity with irradiation; increase of β-carotene bleaching inhibition at 1 kGy	Koike et al. (2015b)
	e-beam	0.5, 0.8 and 1 kGy	Increase of non-anthocyanin phenolics and decrease of anthocyanin concentrations with irradiation; decrease of DPPH scavenging activity with irradiation; enhancement of β-carotene bleaching inhibition at 1 kGy	

Notes: DPPH (2,2-Diphenyl-1-picrylhydrazyl)

14.6.8 Mushrooms

Mushrooms are one of the most perishable products and their short shelf-life is an obstacle for the distribution of the fresh product (Fernandes et al. 2013a). In fact, some studies highlighted the use of gamma irradiation as a conservation process since the results showed that this technology is effective in maintaining the chemical characteristics of *Lactarius deliciosus* L., *Boletus edulis* Bull.: Fr and *Hydnum repandum* L.: Fr (Fernandes et al. 2013a, b). Several studies were performed in order to evaluate the effect of gamma and electron-beam irradiation in the antioxidant properties of different wild and cultivated mushrooms species. Fernandes et al. (2013c) demonstrated that gamma radiation at 0.6 kGy was the most efficient method to preserve the chemical composition of fresh *Macrolepiota procera* (Scop.) Singer when compared with other processing treatments such as freezing (at −20 °C) and drying (at 30 °C). Fernandes et al. (2014) studied the effects of electron-beam irradiation on different dried wild mushrooms (*B. edulis* Bull. and *Russula delica* Fr.) on nutritional, chemical and antioxidant parameters. The antioxidant activity was improved significantly at 6 kGy for both mushroom species, attributed to the increased levels of tocopherols. The same effect was observed in the antioxidant activity for *Amanita caesarea* and *A. curtipes* irradiated at 2, 6 and 10 kGy, which was correlated with the increase in the levels of phenolic compounds (Fernandes et al. 2015). Studies in fresh samples of *Agaricus bisporus* Portobello using gamma and electron beam radiation were performed to evaluate the

effectiveness of these technologies on the conservation of this mushroom (Cardoso et al. 2019). In that work, gamma radiation treatment was associated with the increase of ergosterol, monounsaturated fatty acids and β-tocopherol, while e-beam led to higher values of polyunsaturated fatty acids.

Regarding the irradiation of truffles, total phenolics content significantly increased in *Tuber aestivum* after gamma irradiation with 1.0–2.5 kGy (Adamo et al. 2004; Tejedor-Calvo et al. 2020), although ergosterol and total sterols concentrations did not significantly change (Tejedor-Calvo et al. 2020). However, in irradiated *T. melanosporum* no significant changes were noticed in sterols even after storage of 35 days. Moreover, electron beam irradiation at 2.5 kGy enhanced the concentration of phenolic compounds up to 21 days of storage by almost three-fold (Tejedor-Calvo et al. 2019). Table 14.10 gathers the effects of irradiation treatments on the bioactive compounds of mushrooms.

14.7 Valorization of Food Bioactives: A Forthcoming Application of Food Irradiation

Although bioactive compounds are naturally present in many foods, after suitable isolation and purification, they can be used as ingredients on the development of functional foods, on cosmetics and/or on health care products.

Irradiation treatment can improve the food bioactivity, as it was highlighted in the previous sections. This potentiality could be applied to valorize other products through the improved extraction of bioactive compounds from irradiated materials and further incorporation in food, cosmetic or pharmaceutical products. To the best of our knowledge there is no documented application regarding the use of bioactives from irradiated sources in other products, but there are several encouraging outputs on the incorporation of bioactive compounds in food that will be detailed bellow.

The fortification of food through the incorporation of bioactive compounds in order to improve their quality or biological properties has been explored, especially in yogurts, meat, sausages or bread (Dall'Asta et al. 2013; Amirdivani and Baba 2014; Ribas-Agustí et al. 2014; Guiné et al. 2016; Turgut et al. 2017). Green tea (*Camellia sinensis*) infusions used during milk fermentation to produce yogurt were reported to promote the growth of beneficial yogurt bacteria and enhance the antioxidant activity of yogurt (Amirdivani and Baba 2014). Guiné et al. (2016) obtained yogurts enriched with antioxidants extracted from wine, observing an increase in antioxidant activity without affecting acidity. Chestnut flour was utilized in the formulation of functional bread (Dall'Asta et al. 2013), with a ratio of 50/50 (soft wheat/chestnut flour) leading to the highest value of antioxidant capacity in the final product. Moreover, bread produced with wheat flour fortified at 1, 2, and 3% (w/w) with dried leafy vegetable presented higher concentrations of polyphenols and higher values of antioxidant activity than the controls (Alashi et al. 2019). Other study investigated the incorporation of pomegranate peel extract at 0.5% and 1.0%

Table 14.10 Effects of ionizing radiation on mushrooms bioactive compounds

Mushroom	Radiation source	Applied doses	Main results	References
Lactarius deliciosus L.	γ-radiation	0.5 and 1.0 kGy	Reduction of total tocopherols; increase of phenolics at 0.5 kGy; increase of antioxidant activity at 0.5 kGy	Fernandes et al. (2013a)
Boletus edulis Bull.:Fr.	γ-radiation	1 and 2 kGy	Increase of δ- and γ-tocopherols at 1 kGy; increase of MUFA and decrease of PUFA at 1 kGy; preservation of total phenolics at 2 kGy; increase of TBARS formation inhibition; decrease of DPPH scavenging activity	Fernandes et al. (2013b)
Hydnum repandum L.:Fr.			Increase of δ-tocopherol at 1 kGy; increase of MUFA and decrease of PUFA at 1 kGy; higher total phenolic content at 1 kGy; increase of lipid peroxidation inhibition; decrease of DPPH scavenging activity	
Macrolepiota procera (Scop.) Singer	γ-radiation	0.6 kGy	Increase of MUFA content; enhancement of α- and γ-tocopherol contents; decrease of phenolics; increase of β-carotene bleaching inhibition and TBARS inhibition	Fernandes et al. (2013c)
Boletus edulis Bull.	e-beam	2, 6 and 10 kGy	Higher concentration of *p*-coumaric acid at 2 kGy; higher concentration of cinnamic acid at 10 kGy; increase of total tocopherols with irradiation; decrease of total phenolics with irradiation; higher DPPH scavenging activity and β-carotene bleaching inhibition at 6 kGy	Fernandes et al. (2014)
Russula delica Fr.			Higher concentrations of gallic acid and cinnamic acid at 6 kGy; higher values of total tocopherols and total phenolics at 6 kGy; higher DPPH scavenging activity and β-carotene bleaching inhibition at 6 kGy	
Amanita caesarea (Scop.) Pers.	e-beam	2, 6 and 10 kGy	Increase of *p*-hydroxybenzoic acid concentration at 2 kGy; increase of MUFA at 10 kGy; higher values of tocopherols at 10 kGy; increase of total phenolics with irradiation; increase of antioxidant activity with irradiation	Fernandes et al. (2015)
Amanita curtipes Gilbert			Increase of MUFA at 10 kGy; increase of tocopherols; increase of total phenolics at 10 kGy; increase of antioxidant activity at 10 kGy	
Agaricus bisporus Portobello	γ-radiation	1, 2 and 5 kGy	Higher amount of ergosterol at 2 kGy	Cardoso et al. (2019)

(continued)

Table 14.10 (continued)

Mushroom	Radiation source	Applied doses	Main results	References
	e-beam		Higher amount of ergosterol at 2 kGy	
Tuber aestivum	γ-radiation	1.0, 1.5 and 2.5 kGy	Increase of phenolic compounds at 1.5–2.5 kGy	Adamo et al. (2004)
		0.5, 1.0, 1.5 and 2.5 kGy	Increase of total phenolics with irradiation; preservation of total sterols.	Tejedor-Calvo et al. (2020)
Tuber melanosporum	γ-radiation	1.5 and 2.5 kGy	Decrease of total phenolic compounds at 2.5 kGy; preservation of total sterols	Tejedor-Calvo et al. (2019)
	e-beam		Increase of total phenolic compounds at 1.5 kGy; preservation of total sterols	

Notes: DPPH (2,2-Diphenyl-1-picrylhydrazyl); FRAP (Ferric reducing power); TBARS (Thiobarbituric acid reactive substances); MUFA (Monounsaturated fatty acids); PUFA (Polyunsaturated fatty acids)

concentrations in beef meatballs which was effective on retarding lipid and protein oxidations and on preventing rancid odor formation (Turgut et al. 2017). Moreover, Ribas-Agustí et al. (2014) demonstrated that it is possible to produce dry fermented sausages with natural antioxidants from grape seed and cocoa extracts without changing their sensory properties. Recently, a maceration industrial-scale extraction process was demonstrated to be effective on the recovery of bioactive compounds from unripe red grapes (cv. Sangiovese) with a high extraction yield (Fia et al. 2020). The obtained extract showed greater phenolic compound and water-soluble vitamin contents and antioxidant activity than those measured in the traditional product (called "verjuice") obtained from unripe grapes. The process was suitable to be transferred to the wine industry to produce extracts that can be used as ingredients for other industries and enhance the sustainability of the wine sector.

Concerning the pharmaceutical industry, natural bioactive compounds, in particular, omega-3 fatty acids from fish and fish oil, plant sterol esters and/or phenolic compounds from, e.g., green tea or red wine, have been combined with major hypolipidemic drugs. This co-therapy appeared to be safe and effective to prevent or treat cardiovascular diseases progression (Scolaro et al. 2018) even if further research has to be done to understand the biochemical mechanisms involved in these protective effects.

Relating to the use of food bioactive compounds in cosmetics products, there are some *in vivo* studies in the literature with positive results. In a study with 20 volunteers, it was demonstrated that a formulation containing green tea extract was able to inhibit the photoaging and tumor generation (Li et al. 2009). Also, a product developed as a combination of resveratrol, green tea polyphenols and caffeine was evaluated in a 12-week study with 16 volunteers and revealed to reduce facial redness (Ferzli et al. 2013). Black grape seed extract was also efficiently used in a water-in-oil cream as demonstrated to increase the skin elasticity, decrease

erythema effects and/or skin sebum content (Sharif et al. 2015). Nevertheless, it becomes important to continue studying the best way to recover bioactive compounds from food products and also to explain their potential added-value to the producers.

There are some patents on the application of bioactive compounds extracted from food sources in pharmaceutical and functional foods. As an example, a method to produce grape extracts with high values of Oxygen Radical Absorbance Capacity (ORAC) was patented to be added to foodstuffs and nutritional supplements as a beneficial antioxidant (Ying et al. 2011). The development of nutraceutical products from the antioxidants present in different fruits extracts and, in particular, from *Grewia asiatica* L. (phalsa) was patented based on their *in vitro* and *in vivo* antioxidant potential (Choudhary et al. 2014). Furthermore, a nutraceutical composition using resveratrol in combination with other components, e.g. genistein, lycopene, hydroxytyrosol or polyunsaturated fatty acids, was patented for delaying aging and/or for the treatment or prevention of age-related diseases in animals was patented (Raederstorff et al. 2008). Also, the production of pharmaceutical formulations to deliver biologically active compounds in a controlled manner so as to increase the bioavailability of compounds protecting them from *in vivo* degradation, has been the object of patents (Yuhua and Chien 2009). Slavko (2012) also invented a new pharmaceutical formulation that comprises silica earth, resveratrol, grape seeds extract, green tea extract and tomato powder with antioxidant effects.

14.8 Concluding Remarks

Food products are extremely rich in bioactive compounds. Due to the growing demand in society for new ingredients that can be reused on foods, cosmetics or pharmaceuticals, scientific communities are searching and developing new formulations and optimizing extraction processes. The findings presented in this chapter turn evident the potential added-value of the bioactive compounds extracted from irradiated plant and food sources. Nevertheless, further research should be performed on their applicability for the preparation of new formulations in different industries.

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Chapter 15
Influence of High Pressure Processing on Food Bioactives

Gulsun Akdemir Evrendilek

15.1 Introduction

High hydrostatic pressure (HHP) processing is one of the most popular novel food processing technologies as it can be applied to both liquid and solid foods with and without packaging at room temperature and/or slightly higher and lower than that of the room temperature. HHP provides microbiologically safer food with no adverse changes and/or improved quality properties. Compared to other novel processing technologies, HHP is easy to apply and processing is mostly pronounced by pressure, treatment time, and temperature. Pressure is the most important processing factor as it has a major impact on microbial load and enzyme inactivation.

15.2 Application of High Hydrostatic Pressure

15.2.1 Principles of High Hydrostatic Pressure

High pressure processing follows Le Chatelier's principle explained as "In a system at equilibrium, a change in one of the variables that determines the equilibrium will shift the equilibrium in the direction counteracting the change in that variable" (Herrinton 2007). Effect of pressure on chemical and/or biochemical systems at constant temperature is defined by the volume change (ΔV) described as the variation of the molar volume between the final and the initial states (Asano and Le Noble 1978). Equilibrium shifts toward bond rupture with positive ΔV with

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increasing pressure; whereas negative ΔV results in bond formation. Thus, increase in pressure favors decrease in volume, and vice versa. Le Chatalier's principle, on the other hand, valid for pressure processing only at equilibrium conditions meaning that all materials have a positive compressibility (Balny and Masson 1993). Substances also follow microscopical ordering principle under the compression. Increased pressure causes an increase in the degree of substances under compression at constant temperature. Pressure and temperature act antagonistically with respect to molecular processes which explain increase in melting temperature of a solid with increased pressure with the exception of type I ice (Balny and Masson 1993). HHP technology is also explained by isostatic principle expressed as the transmittance of pressure uniformly and instantaneously (independent of size and geometry of food) (Ramaswamy et al. 1999).

15.2.2 Effects of High Hydrostatic Pressure on Chemical Bonds

Effect of HHP on chemical bonds varies in parallel to the power these bonds. Moreover, compounds with low molecular weight mostly responsible for nutritional and sensory characteristics are not affected, but high molecular weight components with tertiary structure determining functionality are sensitive to pressure (Carlez et al. 1994). Heat processing induces chemical reactions accelerating formation of new components related to color and flavor while beneficial components are lost. These reactions cause changes in food composition and formation of unique color and flavor. HHP, on the other hand, does not accelerate chemical reactions in principle, instead induces physical changes in molecules microscopically and/or macroscopically (Yamamoto 2017). From a microscopic aspect, HHP processing, for instance, restricts molecular motions while causing hydrogen bonds to brake and the molecules to be packed together toward filling gaps between the molecules. Air bubbles of food samples present in gas-liquid dispersion system are dissolved in the liquid phase (Yamamoto 2017). Processing time, processing energy, and the risk of over-processing of some parts of voluminous products can be reduced by the momentary pressure transmission (Barba et al. 2015).

15.2.3 Transmission of the Pressure to Food Systems and Importance of the Food Composition

Unlike heat processing where the heat is gradually transferred through the food system, pressure is transmitted instantaneously and uniformly throughout being independent of the size and geometry of the food product during HHP (Barba et al. 2015). HHP systems provide momentary pressure transmission to food product to

be processed so that pressure applied homogenously regardless of the size and the shape of the product (Barba et al. 2015).

Even though pressure transmission to food is independent of the size and geometry of the food product, its composition is important. Water content of the food sample is the most important factor for the HHP of foods because water transmits the pressure, and homogenous distribution of water provides even distribution of pressure. Moreover, the efficacy of HHP processing decreases with reduced a_w, especially in foods with values below 0.9 (Raso et al. 1998). Studies also reported that cellular damage under pressure has strong relationship with water activity. As water activity of the food matrix gets lower, higher protection of spoiling and pathogenic agents against HHP was observed revealing lower values of water activity causing increase in microbial resistance to HHP (Patterson 2005; Black et al. 2007a, b; Hayman et al. 2008; Syed et al. 2016). In contrast to water, the presence of fats, proteins, minerals, and sugars serves as a protector and increases microbial resistance to HHP due to their resistivity to pressure (Molina-Hoppner et al. 2004; Black et al. 2007a). They may act as a protector to bacteria, thus decrease the microbial inactivation. In fact, it was reported that bacteria are more resilient to HHP in complex matrix such as milk or meat, compared to a buffer at the same pH. When treated at 350–450 MPa at 22 °C for 10 min, strong baroprotective effect on *Yersinia enterocolitica* in whole milk compared to phosphate buffer was observed (Chen and Hoover 2003). pH of the pressurized medium is another major stress factor brings variability in the resistance for different microorganisms (Stewart et al. 1997). Studies related to the effectiveness of HHP on pH revealed that more acidic conditions tend to enhance the effect of pressure (Ritz et al. 2006; Kingsley and Chen 2009). Since it is difficult to measure changes in pH under pressure, there is limited information regarding the actual effect of pH during pressure application (Syed et al. 2016).

Pressure is the most important process parameter for food processing as magnitude of pressure determines level of microbial inactivation and changes in food components (Fig. 15.1).

HHP treatment with temperatures at or below ambient temperature is effective for the inactivation of most vegetative pathogenic and spoilage microorganisms at pressures between 200 and 600 MPa with the rate of inactivation being influenced by the peak pressure (Hauben et al. 1996; Pagan and Mackey 2000; Patterson 2005) as well as ribosomal destruction (Niven et al. 1999), enzyme inactivation (Degraeve et al. 1996; Simpson and Gilmour 1997), inactivation of membrane-bound transport systems (Ulmer et al. 2002), and damage to the proton efflux system (Wouters et al. 1998) (Fig. 15.2).

Although initial studies with HHP of food were started with pasteurization purposes and focused on inactivation of both foodborne and food spoilage microorganisms, today HHP applications are extended beyond the current commercial applications for food safety and shelf-life extension. HPP carries a great potential to retain food safety and food preservation, eliminate food contaminants, reduce salt intake by increased saltiness perception, recovers high value and health-related compounds, improve textural properties, preserve healthy lipids, lowers allergenic

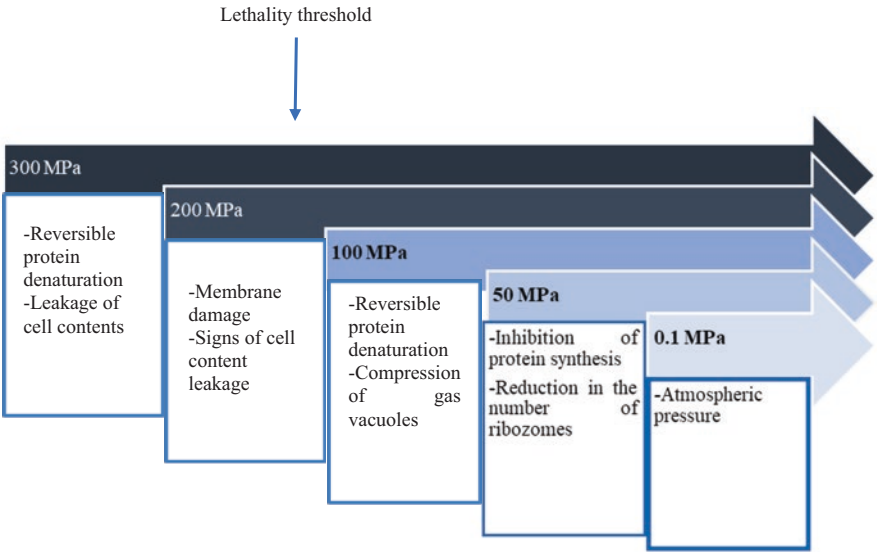

Fig. 15.1 Pressure dependence on biochemical changes and microbial inactivation (Lado and Yousef 2002)

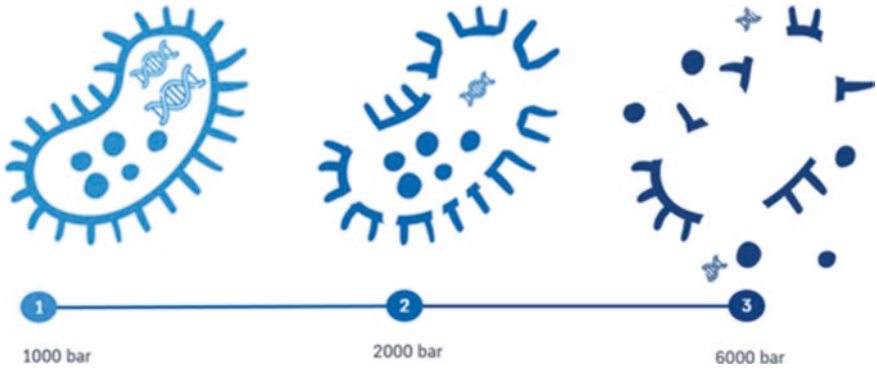

Fig. 15.2 Inactivation of microorganisms in relation to applied pressure (Anonymous 2020)

potential, diminish formation of processing contaminants, enhance health attributes through increased bioavailability of micronutrients and phytochemicals (Fig. 15.3). Among them, recovery of bioactive compounds has attracted a great attention as it affects both bioavailability and bioaccessibility of the food components (Barba et al. 2015).

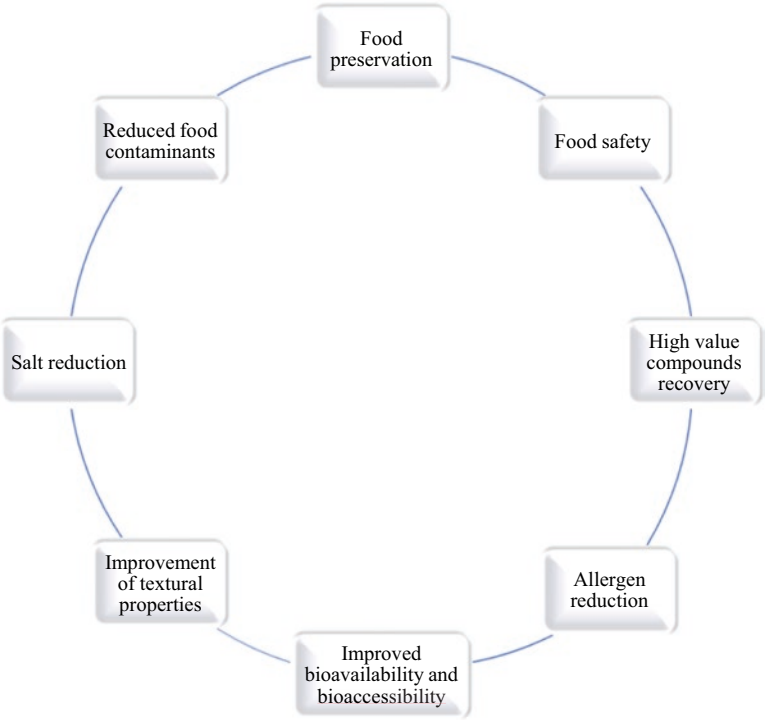

Fig. 15.3 Potential applications of HHP treatment (Barba et al. 2015)

15.3 Bioaccessibility and Bioavaliability

Bioaccessibility, defined as the fraction or the quantity of the compound released from the food matrix in GI tract, is one of the important terms for explaining the use of a bioactive compound by the human body (Heaney 2001). Food needs to go through digestive transformations to be converted into materials ready for assimilation, absorption/assimilation into the intestinal epithelium cells, and lastly, the presystemic metabolism (both intestinal and hepatic) in order for the bioaccessibility of a component. Solely absorption-based definition, on the other hand, misses the beneficial effects of unabsorbed nutrients such as binding of bile salts by calcium in the tract. Thus, *in vitro* digestion procedures for some nutrients generally simulating gastric and small intestinal digestion, sometimes followed by Caco2 cells uptake is used (Courraud et al. 2013). Bioaccessibility alone is not sufficient to determine the utilization of a specific nutrient, the term bioavailability, therefore, is also described as the utilization of a nutrient. Bioavailability refers to the fraction of ingested nutrient or compound that reaches the systemic circulation (Wood 2005). Intercellular compounds of which having bioactive properties are extracted from the target cell and become available by tissue permeabilization. Heat treatment may cause degradation of thermo-sensitive compounds, thus alternative treatments such as HHP is on demand.

15.4 Effect of High Pressure Processing on Food Constituents

15.4.1 Effects on Proteins

The three-dimensional native structure of the proteins is formed by the delicate balance between stabilizing and destabilizing interactions, within the polypeptide chains, and with the solvent resulted from the compact folding of the polypeptide chain formed by the volume of constitutive atoms (compositional volume), the volume of internal cavities, plus a contribution due to solvation (Balny and Masson 1993).

Although folding of isolated native proteins may be a spontaneous process, folding process involves enzymes and protein chaperones in the cell. Depending on the environmental conditions of solvents, salts, and pH; native proteins are stable in a narrow zone and pressure disturbs the balance of intramolecular and solvent-protein interactions (Jaenicke 1991; Balny and Masson 1993). Among the four structures of the proteins, the quaternary structure is mainly held by hydrophobic interactions which are very sensitive to pressure (Weber 1986). Even moderate pressures (<1.5 kbar) favor the dissociation of oligomeric proteins. This phenomenon is always accompanied by negative volume changes, sometimes large, such as for the dissociation of lactic dehydrogenase (Schade et al. 1980). Aggregation or precipitation of formed subunits are followed by dissociation (Masson et al. 1990). Dissociation of oligomers, on the other hand, takes place at pressures lower than those at which unfolding of monomers is observed (<150–200 kPA). Unfolding of proteins and reassociation of subunits from dissociated oligomers are realized with the pressures higher than 150–200 kPA. Time dependent conformational changes occur on the pressure dissociated subunits and the renaturation of oligomers such as hysteresis after pressure release and can occur very slow (Weber 1986; Balny and Masson 1993). Beyond 300 kPa pressure application, significant changes on tertiary structures of protein start to happen. Denaturation of protein is a very complex process including intermediate forms leading to multiple denatured forms (Li et al. 1976). Depending on the rate of the compression and the secondary structure rearrangement, irreversible denaturation of the secondary structure, on the other hand, takes place at very high pressures, above 700 kPa (Carrier et al. 1990) (Fig. 15.4).

Denaturation of proteins by pressure is important as it also affects enzymes catalyzing a reaction pathway with a succession of steps (substrate binding, protein isomerization, and chemical steps). The effects of pressure on proteins can be summarized as on the reaction itself, on enzyme(s) conformational changes, or on enzyme(s) dissociation into subunits (Balny and Masson 1993).

Whey protein concentrate (WPC) contains minimum of 35% protein, and applied temperatures greater than 75 °C causes protein denaturation during spray-drying process applied to produce whey powder. Heat denaturation results in negative effects on the functional properties of the protein revealing reduction in emulsifying capacity and foaming properties (Pittia et al. 1996). Improvements in functional

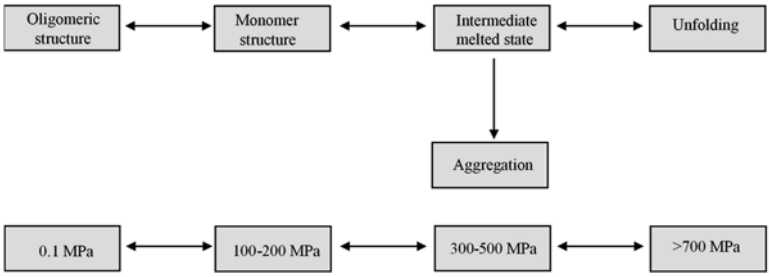

Fig. 15.4 Scheme of protein structure modification by high hydrostatic pressure (adapted from Bolumar et al. 2016)

properties may be achieved by modifying the protein structure using physical treatments instead of heat (Kato et al. 1983).

Pressures up to 200–300 MPa usually result in reversible pressure-induced partial denaturation, whereas pressures greater than 500 MPa result in irreversible and extensive effects on proteins, including denaturation due to unfolding of monomers, aggregation, and formation of gels (Balny et al. 1989). Pressure greater than 300 MPa applied more than 30 min induces irreversible denaturation of β-LG resulting in increased hydrophobicity and formation of protein aggregates (Pittia et al. 1996). Buried hydrophobic and SH groups undergo some structural changes and thus increase the flexibility (Pittia et al. 1996).

HHP-induced β-LG dimers tend to be surrounded by hydrophobic amino acid residues, resulting in an increase of hydrophobic affinity of β-LG at the surface hydrophobic sites (Yang et al. 2003). It is possible that pressure treatment probably induces partially reversible unfolding of the β-LG and this unfolding results in the unmasking of buried hydrophobic groups and an increase in the hydrophobicity of the protein. Therefore, HHP provides an increase in hydrophobicity of β-LG, an expected enhancement of some functionality in food systems. Enhancement of whey protein functional properties provide desirable functionality to a wide range of food products, including improved appearance, body, texture, and consistency and thus solubility (Morr and Ha 1993).

Processing treatments used to manufacture WPC may result in heat induced protein denaturation, which then reduces whey protein solubility. While native whey proteins are soluble at around pH 7, heat-induced denatured whey proteins are less soluble than native whey proteins (Morr and Ha 1993). Therefore, protein solubility of WPC is a useful tool to estimate protein denaturation (Morr and Foegeding 1990).

Effect of HHP on WPC protein solubility changes with applied pressure and exposure time. Compared to control samples, protein solubility did not decrease in 6% whey protein isolate (WPI) solution after HHP treatment at 400 MPa for 10 min (Kanno et al. 1998). However, solubility of 1% WPC decreased as HHP treatment time increased from 5 to 30 min at 690 MPa (Lee et al. 2006). Thus, HHP treatment conditions are important to maintain protein solubility in potential food product formulations. HHP also affects emulsifying properties of WPC. In fact, HHP

conducted at 690 MPa for 5 min provided an increased emulsifying activity of WPC (Lee et al. 2006). HHP caused an increase of the surface hydrophobicity, attributed to the molten globule state, resulting in partial unfolding of whey proteins (Liu et al. 2005; Lee et al. 2006). The molten globule state provided by HHP retains protein solubility and increases hydrophobicity to enhance foam stability of whey proteins (Lim et al. 2008).

HHP also induces conformational changes in the pork batter's proteins, with alteration of some of the epitope structures (Hajós et al. 2004). HHP at 600 MPa provided modification of the IgE immunoreactivity of the proteins in the sausage batter (Hajós et al. 2004). HHP applied in the range of 100–400 MPa to rice grains (*Oryza sativa* L. Japonica var. akitakomachi) immersed in distilled water exhibited solubilization and subsequent release of ice allergenic proteins. Approximately 0.2–0.5 mg per gram of rice proteins was released in the pressure range of 300–400 MPa (Kato et al. 2000) (Fig. 15.5).

HHP applied at 100–300 MPa for 15 min to soy "whey", a by-product from the manufacture of tofu, provided reduction in the immunoreactivity of soy proteins to the antibodies against Gly m 1, an important allergen of soybean that causes allergy by inhalation (Peñas et al. 2006). Compared to control, HHP-treated soy sprouts obtained from seeds showed significantly lower immunoreactivity (Peñas et al. 2011). This positive effect of HHP enables dairy whey hydrolysates obtained by pepsin and trypsin in combination with HHP treatment to be used as a source of peptides in hypo-allergenic infant formula (Peñas et al. 2006).

HHP of soybean seeds with 300 MPa at 20 °C for 25 min provided the release of basic 7S globulin, an important allergenic protein, in soybean seeds soaked in water. A decrease in the allergen Gly m 1 content of soybean whey was also reported after

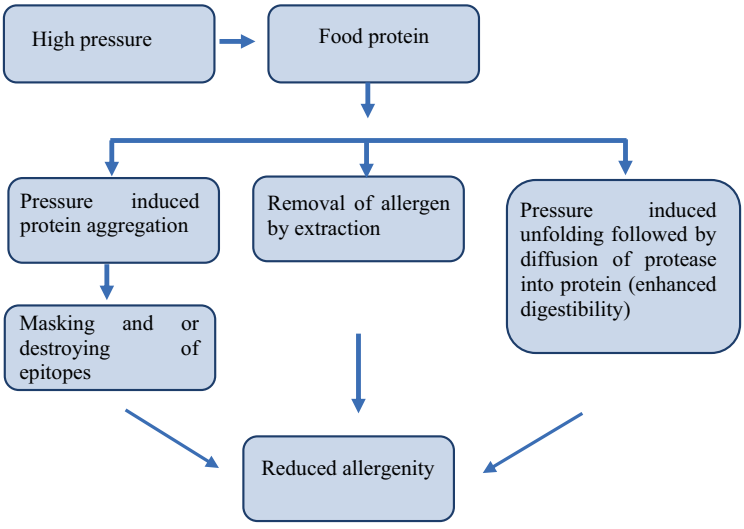

Fig. 15.5 Effect of pressure processing on conformational changes regarding to allergenity (adopted from Rahaman et al. 2016)

HHP at 100, 200 and 300 MPa (Peñas et al. 2006). Considerable amount of protein was released from the polished rice grains when immersed in water and pressurized at 100–400 MPa. Two major allergens of 16 kDa albumin and 33 kDa globulin were released after HHP (Kato et al. 2000). Conformational changes observed in the tertiary and secondary structures of SPI are obvious at pressures above 200 MPa (Messens et al. 1997; Wang et al. 2008). As a result, HHP treatment significantly influenced the free SH content and hydrophobicity of SPI, which are closely associated with the allergenicity of SPI (Breitenede and Radauer 2004).

15.4.2 Effect on Polyphenols, Carotenoids, Isothiocyanates, and Glucosinolates

HHP processing has been applied to various food products including pomegranate juice (Varela-Santos et al. 2012; Chen et al. 2013), vegetables (tomato, green pepper, green celery, onion, lemon and olive oil), beverages (Barba et al. 2010), mulberry juice (Wang et al. 2017), apple juice (Kim et al. 2012), cactus juice (Moussa-Ayoub et al. 2017), jabuticaba juice (Inada et al. 2018), orange juice (Esteve and Frigola 2008; Polydera et al. 2003, 2005; Júnior et al. 2015), cashew apple (*Anacardium occidentale* L.) juice (Lavinas et al. 2008; Queiroz et al. 2010), honeydew melon juice and honeydew melon juice milk, milk and dairy products (Borad et al. 2017; Demazeau et al. 2018; Marciniak et al. 2018; Parrón et al. 2018; Du 2016), sweet potato flour (Cui and Zhu 2019), fermented rice bran (Kim and Han 2012), exotic fruit and their products (Rawson et al. 2011), and açaí pulp (de Jesus et al. 2020) for preservation or extraction of bioactive compounds polyphenols, carotenoids, glucosinolates, micronutrients and polynutrients as well as minerals with respect to their bioavailability and bioaccessibility.

Extraction and preservation of bioactive properties of carotenoids may change depending on processing parameters, especially with applied pressure. HHP at 600 MPa for 15 min provided a significant increase (3%) in total carotenoids in carrot purée (Patras et al. 2009). HHP of vegetable beverage conducted at 40 °C with 400 MPa for 1 min revealed a significant increase in β-carotene, γ-carotene, lutein, lycopene, and lycopene-epoxide (Plaza et al. 2006). Similarly, carotenoid extraction was significantly increased by pressure processing in the range of 400–600 MPa for 2–15 min at 20–25 °C from orange juice, persimmon purée, and tomato products (Sanchez-Moreno et al. 2004, 2005). Significant increase in extraction of β-criptoxanthin, zeaxanthin, lutein, β-carotene, and α-carotene in orange juice with pulp by 400 MPa, 40 °C treatment temperature for 1 min (Sánchez-Moreno et al. 2005) and extractable carotenoids (β-criptoxanthin, −criptoxanthin-5,8-epoxide, β-carotene, lutein, zeaxanthin, lycopene, neolycopene) in papaya slices (cv Sunrise) by 400 MPa, 25 °C treatment temperature for 1 min (De Ancos et al. 2007), and extractable carotenoids (β-carotene, γ-carotene, lutein, lycopene, lycopene-epoxide) from Gazpacho soup by 400 MPa, 60 °C treatment temperature for 15 min (Plaza et al. 2006) were also observed.

HHP processing of tomato purée with 500 MPa pressure at 20–25 °C temperature for 2–12 min provided 60% and 21% increase in lycopene extraction (Krebbers et al. 2003; Qiu et al. 2006). Besides water, different solvents such as water:ethanol, chloroform:ethanol were also utilized along with HHP to increase the extraction rate. In fact, extraction of lycopene from tomato paste waste by 100–600 MPa at room temperature for 1–10 min in chloroform:ethanol (95:5, v/v) and ethanol:water (45:55–95:5, v/v) provided up to 92% recovery (Jun 2006). Increase in the extraction of carotenoids could be attributable to various factors such as permeabilization of the plasma membrane cell and denaturation of the carotenoid-binding protein induced by HHP (Barba et al. 2015).

Polyphenols due to their antioxidant properties carry great importance for consumers (Barba et al. 2012) and thus, studies focused on HP-assisted extraction of phenolic compounds from different plant sources. HHP provided significant increase on phenolic compounds from orange juice with pulp with 400 MPa pressure at 40 °C for 1 min (Sánchez-Moreno et al. 2005) and this was found to be useful for extraction of flavonoids from propolis (Shouqin et al. 2004, 2005) and *Rhodiola sachalinensis* (Zhang et al. 2007). HHP not only provides improved extraction yield but also reduces extraction time compared to other extraction methods such as ultrasonication, leaching, Soxhlet, and reflux (Shouqin et al. 2005). Compared to heat reflux at 85 °C for 45 min, extraction at room temperature, and ultrasonic extraction with 250 W at 20–40 °C for 90 min; extraction of total phenolic compounds (TPC) from tea leaves using different solvents (acetone, methanol, ethanol and water) with 100–600 MPa pressure at 20 °C for 1–10 min provided significant increase (Xi et al. 2009). Another study conducted with HHP extraction (100 MPa up to 600 MPa) of green tea for the extraction of four major catechins (epicatechin gallate, epigallocatechin, epigallocatechin gallate, epicatechin) and gallic acid revealed that the extraction yields of the catechins with HP extraction of 400 MPa for only 15 min to be the same as those of organic solvent extraction for 2 h (Jun et al. 2010). HHP extraction (380 MPa pressure at room temperature for 10 min) of green tea polyphenols compared with ultrasonic extraction, leaching extraction, reflux extraction with water, methanol and ethanol as extraction media showed that the achievable ratio (mass of essential components/mass of raw herb) of HHP was the highest (Shouqin et al. 2004).

Extraction of polyphenols from grape by-products have also been studied extensively as they are good sources of polyphenols and can be extracted in large quantities. Compared to conventional extraction methods, HHP provided significant increase in the total as well as individual anthocyanins from grape byproducts (Corrales et al. 2008a; Corrales et al. 2009; Corrales et al. 2008b). Moreover, studies concluded that anthocyanin extraction by HHP is influenced by several factors such as their chemical nature, the extraction method employed, sample particle size, storage time and conditions, as well as presence of interfering substances. HP-assisted extraction of caffeic acid, catechin and ferulic acid with 500 MPa pressure at 60 °C for 5–15 min significantly increased extraction of phenolic compounds from Korean black raspberry when it is compared to conventional extraction at 100 °C for 24 h using a reflux condenser (Seo et al. 2011). Similarly, HP assisted extraction of gallic

acid, corilagin, ellagic acid, and total phenolic compounds from longan fruit pericarp was significantly higher. Moreover, HP-assisted extraction (200–500 MPa/30–70 °C/2.5–30 min) of corilagin and total phenolic yield were significantly higher than that of the conventional ethanolic extraction during 12 h (Prasad et al. 2009a; Prasad et al. 2010). HP extraction yield of polyphenols from litchi fruit pericarp applied with 200–500 MPa for 2.5–30 min at 30–90 °C was superior than that of the other conventional extraction methods (Prasad et al. 2009b).

Myrosinase enzyme action provides formation of various isothiocyanates such as sulforaphane, phenethyl isothiocyanate, and benzyl isothiocyanate with beneficial properties (Dinkova-Kostova and Kostov 2012) from glucosinolates. However, myrosinase is inactivated by heat processing, and thus, transformation of the glucosinolates into the beneficial products is inhibited, therefore research has focused on nonthermal processing technologies that have no adverse effects on myrosinase. Studies with pressure-treated broccoli juice and heads revealed that HHP applied at 100–500 MPa and 20–40 °C for 15–35 min provided catalytic reactions that converts glucosinolates into isothyocyanates by enhancing cell disruption or membrane permeabilization (Van Eylen et al. 2007, 2009).

Treatment time has significant adverse effect on glucosinolates degradation due to leaching, and thus, glucosinolate hydrolysis gets even higher at elevated pressures as a result of cell permeabilization. Permeabilization rate increases during pressure treatment with increasing pressure. Due to the complex myrosinase–glucosinolate interactions with 100-500 MPa pressures at 20–40 °C for 15–35 min, an increase in isothyocianate formation occurs after HHP. Degradation rates of isothiocyanate sulforaphane and phenylethyl isothiocyanate were reported to be lower when HHP was applied in comparison to thermal treatment (30–100 °C), concluding that myrosinase activity might be controlled by HHP treatment (Barba et al., 2015). Compared to blanching conducted at 90–95 °C for 3 min, a significant increase was observed in total isothiocyanates after 200–600 MPa pressure application at 20–40 °C for 5 min (Alvarez-Jubete et al. 2014).

Although no significant modification in indole-3-carbinol and indole-3-acetonitrile was detected in naturally fermented cabbage and sauerkraut produced by fermentation with *Lactobacillus plantarum* and *Lactobacillus mesenteroides* after pressure processing (300 MPa/40 °C/10 min) and during 3-month refrigerated storage; significant losses in the range of 33–67% in ascorbigen content was detected after HP and 3-month storage (Peñas et al. 2013).

15.5 Effect of High Pressure Processing on Bioavailability and Bioaccessibility of the Bioactives

Studies related to determination of the effect of HHP on bioaccessibility and bioavailability of the nutrients are mostly conducted *in vitro* using simulated gastrointestinal digestion (Cilla et al. 2012; McInerney et al. 2007; Van Buggenhout et al.

2010). HHP conducted at room temperature with 400–600 MPa for 2 min did not show any significant effects on bioaccessibility of carotenoids in carrot. However, 600 MPa pressure treatment slightly increased lutein bioaccessibility in green beans and reduced β-carotene bioaccessibility in broccoli (McInerney et al. 2007).

HHP treatment did not adversely affected antioxidant capacity and total carotenoid content of different vegetables. Depending on the type of the vegetables and magnitude of the pressure, minor alterations in carotenoids were observed after HHP (McInerney et al. 2007). While mild HHP provided 1.2 times higher bioaccessibility of β-carotene in carrots compared to mild thermal pasteurization, intense HHP and pressure-assisted thermal sterilization (600 MPa/117 °C/Fo equivalent of 3 min) resulted in over 1.2 and 2.5 times lower bioaccessibility of β-carotene compared to intense thermal pasteurization and thermal sterilization (Fo = 3 min), consequently. Bioaccessibility of thermally sterilized β-carotene in carrot was improved by about 80%; however, pressure-assisted sterilization resulted in about 30% reduction compared to the raw sample (Knockaert et al. 2011).

It was reported that bioaccessibility of a compound is related to tissue hardness and, in fact bioaccessibility of β-carotene in carrot tissue is inversely related to hardness (Lemmens et al. 2009). Mild thermal pasteurization and mild HHP pasteurization provide reduction in firmness and turgor pressure loss thus, softening the carrot tissue. Tissue softening provided by HP pasteurization may explain the higher bioaccessibility of β-carotene in the sample compared to mild thermal pasteurization. Tissue softening changes with thermal treatment conditions and it is proven that at the more intense pasteurization and sterilization conditions, thermal processing resulted in a softer tissue compared to HP treatment resulting in higher bioaccessibility in the thermally treated samples (Knockaert et al. 2011).

Carrot puree with added olive oil was treated with thermal pasteurization ($^{10°C}P_{90°C}$ = 10 min: equivalent to 90 °C/10 min at Z = 10 °C) and HHP treatment (600 MPa/45 °C/20 min) to determine effect of both treatments on bioaccessibility of β-carotene. HHP treatment did not improve the bioaccessibility of β-carotene in the HHP-homogenized puree; whereas an equivalent thermal pasteurization increased the bioaccessibility more than double (Knockaert et al. 2012a). Extractability of lycopene in tomato juice by thermal processing (93 °C/60 s), HHP (500–700 MPa/30 °C/0–10 min) and pressure-assisted thermal processing (PATP) (500–700 MPa/100 °C/10 min) increased by 12% after HHP and PATP, however, this was not translated into improvement in bioaccessibility (Gupta et al. 2011). All processes provided only less than 0.5% of lycopene in micellar form and that was bioaccessible. Pressure treated samples had more prominent lycopene crystals, but the crystals appeared to be enveloped in all the processed samples. Compared to control samples, higher extent of all-trans-β-carotene micellarization was observed in processed juice. Hot break juice subjected to PATP came up with 15–30% improvement in all-trans-β-carotene micellarization compared to raw juice that had undergone the same process (Knockaert et al. 2012b).

Besides other bioactive compounds, effects of HHP on release of minerals from tissue were also investigated. As HHP disrupts the cell wall, minerals are released

into the extracellular phase. However, studies revealed that bioaccessibility was not dependent on the concentration of the minerals in the extracellular content (Barba et al. 2015). HP treatment at 500 MPa for 2–10 min provided significant increase in the content of minerals by 2–303% for Ca, 4–11% for Fe, and 8–28% for Zn (Briones-Labarca et al. 2011). On the other hand, calcium dialysability was significantly lowered by HHP; whereas dialysability of iron and zinc were slightly increased. HHP of algarrobo (*Prosopis chilensis*) seeds at 500 MPa for 10 min caused a decrease in the concentration of calcium, iron, and zinc but improved the bioaccessibility of iron and zinc (Briones-Labarca et al. 2011). HHP also affected mineral concentration of milk. Calcium concentration between the micellar and serum phases is changed by HHP. Increased concentration of Ca and P in the serum phase 1–2 h after application of 300 MPa was observed in raw skim milk (Regnault et al. 2006). HHP at 400 MPa was also found to be effective for increasing the initial concentration of Ca, P, and Mg in serum phase in bovine, caprine, and ovine milk (López-Fandiño and Olano 1998). Similarly, 12% increase in ionic calcium after pressure treatment followed by incubation at 30 °C for 15 min was observed, however the concentration of ionic calcium was reversed within 4 h upon storage at 20 °C (Zobrist et al. 2005). Results regarding the concentration of the minerals change with applied pressure and type of the milk as well as the complexity of the milk system, and dynamic nature of the micelles (Orlien et al. 2006, 2010). HHP conducted at 400 MPa and 36 °C for 5 min to milk-based (whole, skimmed, and soya) fruit (orange, pineapple, kiwi, and mango) beverages positively modulated Ca and P bioaccessibility as well as P bioavailability (Cilla et al. 2011). However, Ca solubility, transport, and Caco-2 uptake did now show parallel trends. It is possible that other factors influenced the Ca uptake even though the Ca may be in a solubilized form, and available for absorption in the body (Cilla et al. 2011).

15.6 Conclusions

Studies related to preservation of bioactive compounds and their functionality revealed that compared to most common processing technologies such as thermal treatment, HHP provides better preservation of bioactive with functionality. Effect of HHP on individual bioactive may change depending on the structure of the bioactive compounds and HHP parameters with pressure being more detrimental. Besides, functionality of proteins may be changed by HHP depending on their structure and processing parameters applied. Bioavailability and bioaccessibility of the individual compounds also have revealed both properties are changed by the chemical structure and processing parameters. Since these studies are independent from each other and some of them have contradictory results, more comprehensive studies need to be conducted with bioactives regarding changes in their chemical structures, their functionality as well as their bioavailability and bioaccessibility.

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Chapter 16
Influence of Ultrasound Treatments on Food Bioactives

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16.1 Introduction

Ultrasound (US) is a well-known emerging technology in the food industry and it may improve several phenomena like mass and energy transfer (Gallego-Juarez 2017). US is a high reproducible technique that allows completing food processes in seconds or minutes, minimizing the processing cost, reducing manipulation and work-up. The final products produced with US treatments usually have higher quality, purity and safety. It eliminates the post-treatment of waste-water and consumes less time and energy as compared to conventional processes (Chemat et al. 2011). It is a safe, non-polluting and environmentally friendly technology (Gallego-Juarez 2017).

US can be defined as sound waves having frequency that exceeds the hearing limit of the human ear (>16 kHz) (Hecht 1996). The types of ultrasonic spectrum are as follows; low frequency (20–500 kHz) high power (>1 W/cm^2) and high frequency (>100 kHz) low power (<1 W/cm^2) US depending on its frequency and intensity (Mason et al. 2011). The representative range for the frequency that is generally used in ultrasonic technologies is between 20 kHz-500 MHz (Yusaf and Al-Juboori 2014).

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High-frequency or low power US can be used for non-invasive and non-destructive analyses and monitoring of various food materials (fresh fruits and vegetables, raw and fermented meat products, fish, poultry, oils, cereals etc.) during processing and storage to ensure high quality and safety (Awad et al. 2012). It generally serves to obtain information on the physicochemical properties of foods. Examples of evaluated parameters are acidity, firmness, sugar content, ripeness etc. On the other hand, low frequency or high power US is used for the changes in the physical and chemical properties of foods (Soria and Villamiel 2010) by inducing pressure, shear and temperature difference in the medium through which they propagate and is capable of producing cavitation that inactivate microorganisms in foods (Piyasena et al. 2003). It is used in number of processes in the food science and technology such as surface cleaning and destruction of contaminants, inactivation of microorganisms and enzymes, cooking, degassing, defoaming, bleaching, crystallization, filtration, homogenization, meat tenderization, emulsification, drying, extraction of foods and bioactive compounds, and freezing (Awad et al. 2012; Chemat et al. 2011). The application of US on the above cited unit operations can influence food bioactives as well as their functionality (Lafarga et al. 2019; Cilia et al. 2018). Some studies showing the effect of US treatment on food bioactives in different food processing conditions are given in Table 16.1.

In addition, reports showed that US affects food allergens (Wang et al. 2020; Nayak et al. 2017) and helps in the development of functional food ingredients (Ozuna et al. 2015). This chapter investigates the existing reports on the applications of US in food science and technology as well its influence on food bioactives. The advantages and disadvantages of US treatment on food bioactives in different food processes are given in Fig. 16.1.

16.2 Influence of Ultrasound on Food Bioactives in Different Processes

16.2.1 Juice Processing

Fruit juices are widely consumed food products all around the world that can easily be consumed by different age consumer groups including infants, children and adults. They are important products for the nutritional needs of people as they contain high amounts of vitamins, minerals, dietary fibre, phytochemicals such as carotenoids, phenolic acids, flavanols, showing health promoting activities (Gómez et al. 2011). In order to increase the shelf life of fruit juices, thermal pasteurization is a common method to prevent microbial growth. The major drawback of thermal processing is decreased quality of the final product due to undesirable biochemical and nutritional changes. Keeping nutritional, quality, flavour losses at the minimum level is the major goal of fruit juice industry due to preference of consumers to fresh, high-nutritional, high-quality products with extended shelf life (Paniwnyk

Table 16.1 Some studies showing the effect of ultrasound treatments on food bioactives in different food processing conditions

Food matrix	Food process	Treatment conditions	Results	Reference
Strawberry juice	Fruit juice processing	Frequency: 25 kHz Duration: 15 or 30 min	Ascorbic acid content ↑, TPC ↑ twofold with 30 min of US	Bhat and Goh (2017)
Cantaloupe melon juice	Fruit juice processing	Amplitudes: 20, 60, 100% Power intensities: 75, 226, 376 W/cm^2 Duration: 2–10 min	Improved colour parameters, TPC ↓ by 30% (376 W/cm^2–10 min)	Fonteles et al. (2012)
Brown seaweed	Extraction	Frequency: 20 kHz Power intensities: 7,35,75 W/cm^2 Duration: 25 min	Enhanced extraction of bioactives especially HMW phenolic compounds	Kadam et al. (2015)
Purple potato	Extraction	Frequency: 20 kHz Amplitudes: 30, 50,70% Duration: 5 min	Amount of extracted anthocyanins ↑	Mane et al. (2015)
Tomato pulp	Pulp processing	Frequency: 24 kHz Power intensity: 105 W/cm^2 Duration: 15, 30, 60 min	Lycopene bioaccessibility ↓	Anese et al. (2013)
Celery	Enzymatic hydrolysis	Power intensity: 40–99 W Duration: 0 to 60 min	Luteolin yield ↑ 26.1-fold Apigenin yield ↑ 32.2-fold at 80 W, 30 min	Zhang et al. (2011)
Rapeseed	Enzymatic hydrolysis	Frequency: 20 kHz Power intensities: 200–1200 W Duration: 3, 6, 9, 12, 15, 18 min	ACE inhibitory activity of protein hydrolysates ↑	Wali et al. (2017)

ACE: angiotensin-I-converting enzyme; GAE: gallic acid equivalent; HMW: higher molecular weight; TPC: total phenolic content

2017). Therefore, food industry and researches seek alternative non-thermal methods to give the minimum sensorial and nutritional damage to fruit juices. US is an effective method of great interest to keep the highest quality, increase the shelf life and retain the bioactive compounds of fruit juices. Besides, US is a technology that meet The Food and Drug Administration (FDA) requirement of 5 log reduction in microorganisms in fruit juices ensuring the safety of the products (Aadil et al. 2013).

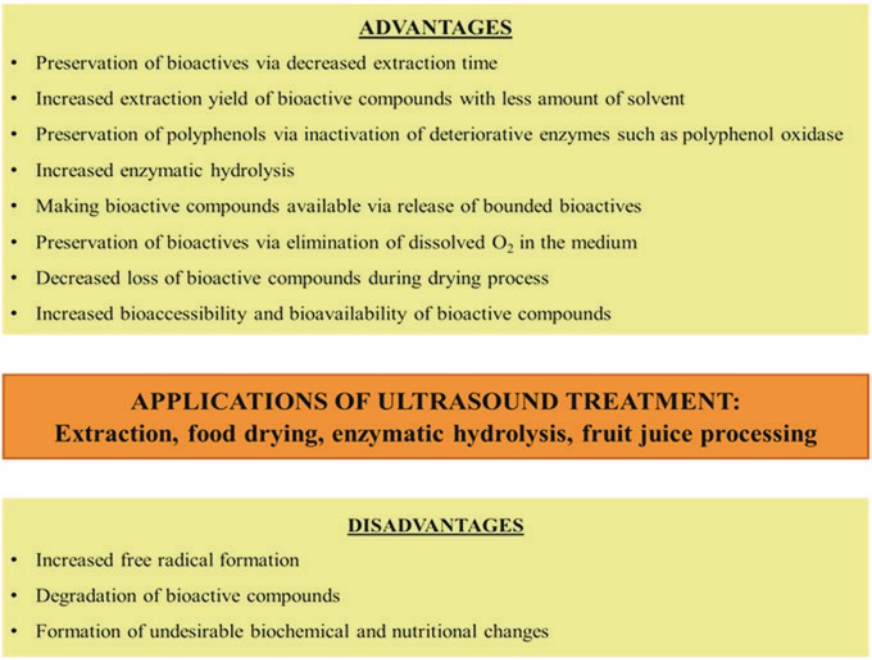

Fig. 16.1 Advantages and disadvantages of US treatment on food bioactives in different food processes

Many studies have been conducted on many fruit juices such as strawberry (Bhat and Goh 2017), apple (Abid et al. 2013), orange (Guerrouj et al. 2016), grapefruit (Aadil et al. 2017), carrot (Martínez-Flores et al. 2015), red grape (Margean et al. 2020), watermelon (Rawson et al. 2011) juices, in order to determine the effect of US treatment on the quality characteristics of the products. In terms of the effect of US treatment on bioactives in fruit juices, studies focus on the change in the amount of carotenoids, total phenolics, phenolic acids, flavonoids, anthocyanins, ascorbic acid as well as the antioxidant capacity of the products. Two-sided differing results have been obtained depending on the treatment time, degree of the amplitude, temperature, and US intensity, emerging either improved (Aadil et al. 2013; Zafra-Rojas et al. 2013; Bhat and Goh 2017) or worsened quality (Rawson et al. 2011; Fonteles et al. 2012; Radziejewska-Kubzdela et al. 2020; Silva et al. 2020).

The food matrix and processing conditions are the most important variables for these different results (Fonteles et al. 2012). In studies, a few theories have been suggested for the variabilities of US treatment. Firstly, sonication process can produce free hydroxyl radicals that can attach to the aromatic ring of phenolic compounds. Especially, enhanced antioxidant activity of phenolics was observed, when hydroxyl group is added to *ortho*- or *para*-positions (Aadil et al. 2017; Bhat and Goh 2017; Wang et al. 2019). Second, formation of free radicals may affect the phenolics in juices as hydroxyl radicals result in oxidative degradation (Fonteles

et al. 2012). Third, cavitation eliminate the dissolved O_2 in the medium which is a major risk factor for ascorbic acid degradation. Therefore, increased ascorbic acid levels were reported in many studies (Bhat and Goh 2017; Margean et al. 2020). Furthermore, US treatment inactivates enzymes such as polyphenol oxidase that is responsible for the enzymatic browning reactions leading to improved antioxidant activity of fruit juices (Nadeem et al. 2018). Lastly, US is a mechanical process that causes cell wall breakage, surface peeling, erosion, particle breakdown resulting in the release of bound form of bioactives, making them available as well as allowing extraction of more bioactives (Bhat and Goh 2017; Wang et al. 2019).

In the study of Aadil et al. (2013), US applied for 4–16 min to strawberry juice samples with 20 kHz frequency, 400 W power. As a result, the highest values of total antioxidant capacity, total phenolic content, 2,2-diphenyl-1-picryl hydrazyl (DPPH) radical scavenging activity and the amount of catechin, gallic acid, ellagic acid were reported at 12 min, and then a sharp decrease was observed in all parameters at 16 min due to degradation of antioxidant compounds. Approximately, 80% increase was reported for total phenolic content at 12 min (95.8 mg/100 ml GAE) as compared to control (57.6 mg/100 ml GAE). Whereas ascorbic acid content continued to increase to 22.8 mg/100 ml at 16 min. Application of 400 W power of US for 30, 60 and 90 min in apple juice increased the total phenolic content, total flavonol content, antioxidant capacity and vitamin C content. Also non-enzymatic browning increased proportionally to time due to breakdown of colouring pigments. However, this colour change was not visible with naked eye (Aadil et al. 2013). Abid et al. (2013) applied US at 25 kHz frequency for 30, 60 and 90 min in apple juice. After 30 min vitamin C content increased and the antioxidant capacity increased by 25% in 90 min. In all applications total phenolic, total flavonol and total flavanol contents increased. Total phenolic content increased to 829 μg GAE/g from 757 μg GAE/g at 90 min.

Watermelon juice was sonicated at 20 kHz frequency, 24.1–60 μm amplitude level between 2–10 min. It was shown that ascorbic acid level reduced by time indicating the degradation of ascorbic acid at prolonged processing at higher power levels. Total phenolic content did not change up to 6 min but a significant decrease was reported after 10 min with 41.56%. A decrease in lycopene content at higher amplitudes after 5 min was another result of the study due to hydroxyl radical formation (Rawson et al. 2011). In purple cactus pear juice long duration (10, 15, 25 min) and short duration (3, 5, 8 min) of US was exerted at 20 kHz frequency and 40%, 60% and 80% amplitudes. In all treatments total phenolic content, ascorbic acid content and DPPH radical scavenging capacity values were higher than the control. The highest values were obtained at 80% amplitude either at 15 or at 25 min (Zafra-Rojas et al. 2013).

In another study, orange juice was enriched with xylooligosaccharide and the stability of the prebiotic in the functional food was tested after sonication at power of 300, 600, 900 and 1200 W, and 20 kHz frequency. Although the stability of xylooligosaccharide was not affected, high energy treatments caused a significant reduction in ascorbic acid content, total phenolic content, and ferric reducing antioxidant power (FRAP) values (Silva et al. 2020). In the study of Radziejewska-Kubzdela

et al. (2020), the effect of thermal processing, enzymatic pre-treatment and sonication of mash was evaluated in barberry juice. Interestingly, the lowest amount of ascorbic acid was detected with sonication as compared to other treatments. Besides, antioxidant capacity, and total flavonoid content were decreased by 55% and 22%, respectively (Radziejewska-Kubzdela et al. 2020).

Pasteurization and sonication was compared in red grape juice at the amplitude of 50% and 70%, at 20 kHz frequency for 5 or 10 min. The total phenolic content affected by amplitude level but not with the treatment time and the highest retention was observed at 70% amplitude and 10 min. Alternatively to pasteurization 70% amplitude and 5 min of US treatment was recommended to increase the flavonoids including rutin, epicatechin, resveratrol and quercetin. On the other hand pasteurization decreased phenolic content from 25% to 30%. High amplitude treatments at 70% for 10 min increased ascorbic acid content as compared to untreated samples. In a shelf life study of carrot juice for 20 days at 4 °C, US treatment increased antioxidant capacity, retained carotenoids up to 12 days, ascorbic acid >90% after 12–14 days. Slight changes was reported after 20 days in total phenolic content. US with 58 °C heat treatment retained 98% of carotenoids and 100% of ascorbic acid (Martínez-Flores et al. 2015). Guerrouj et al. (2016) reported increased total phenolic content, total flavonoid content, DPPH radical scavenging activity, ascorbic acid content, and carotenoid content in all sonicated samples. Total phenolic content increased from 42.7 mg/100 ml GEA to 69.5 mg/100 ml GEA after 30 min.

16.2.2 Extraction

The most common method used to extract molecules from animal, plant and microorganisms is ultrasound-assisted extraction (UAE) (Dong et al. 2010). Using this technique presents many advantages including of being an energy-saving, cost effective technique that uses low or moderate temperatures, which is important for molecules with low thermal stability. However, in order to make sure the extraction is successfully conducted, some factors such as the ultrasonic power, temperature, frequency, reactor characteristics and interaction solvent-substrate should be taken into consideration (Esclapez et al. 2011). In this system, the propagation of the waves leads to the disintegration of bubbles that generates a disorder, particles collisions and destabilization of the microporous elements of the sample. The result of this set of reaction is the rapid flow of solute from the sample to the extraction solvent (Azmir et al. 2013). According to Esclapez et al. (2011), the most beneficial period for extraction of bioactives from plants is generally during the first few minutes. The attention is mostly given to plants because of the fact that they contain several polar and non-polar bioactives such as phytochemicals (phenolic acids, flavonoids, anthocyanins, etc.), lipids (carotenoids, tocopherols, polyunsaturated fatty acids, sterols etc.), flavors, fragrances of pigments that are generally exploited in the cosmetic, food and pharmaceutical industries (Esclapez et al. 2011).

The application of UAE in the extraction of plant bioactives significantly increased the antioxidant activity of its extract (Şahin Ercan and Soysal 2013; Altemimi et al. 2016; Nkhili et al. 2009). In contrast, other researchers reported that unsuitable conditions may lead to the degradation of phenolic compounds (Dahmoune et al. 2013; Babazadeh et al. 2017). The recovery of these compounds may be influenced by the nature of the solvent used, extraction time, extraction temperature, frequency and power of US and particle size of the analyzed compound (Chemat et al. 2017; Khan et al. 2010; Ma et al. 2009).

In a study regarding the extraction of bioactive compounds from *Malva sylvestris,* it was shown that the amount of gallic acid, genistein, quercetin, apigenin and myricetin was significantly higher in all cases with different solvents using UAE compared to that of agitated bed extraction (ABE). The ABE method showed extraction concentrations of 55.4–114.3, 0–112.4, 84.4–150.4, 0–131.1 and 78.2–118.20 mg/g for gallic acid, genistein, quercetin, apigenin and myricetin, respectively, whereas the values obtained with the UAE technique were 124.1–146.5, 115.9–153.2, 175.7–183.9, 145.8–173.3 and 124.1–131.2 mg/g, respectively (Bimakr et al. 2017). Accordingly, Li et al. (1994) obtained 15 and 30% increase in total extract and saponin content, respectively, from *Panax ginseng* using ultrasonic irradiation (UI) extraction compared to extraction without UI. Altemimi et al. (2016) used UAE method to optimize the isolation of phenolic compounds from peaches and pumpkins. Their research showed that the best optimal environments given as temperature, power and time, respectively, for the abstraction of those active compounds from pumpkins were 41.45 °C, 44–60% and 25.67 min, while those of peach they were 41.53 °C, 43.99% and 27.86 min. They also mentioned that the extraction processes were significantly better with UAE compared to solvent extraction without UAE.

The impact of ultrasonic extraction on non-polar compounds was studied by Hashemi et al. (2016), it was shown that tocopherol and tocotrienol contents of *Pistacia khinjuk* hull oil significantly increased with amplitudes of UAE. Variations in amplitudes (25–50%) and pretreatment durations (15–45 min) led to concentrations ranging between 41.1–60.6, 57.4–76.8, 4.33–4.42, 61.3–84.5, 136.5–162.5 and 78.8–99.7 mg/g respectively for α-tocopherol, β-tocopherol, δ-tocopherol, γ-tocopherol, α-tocotrienol and β-tocotrienol while the initial values (amplitude: 0%, pretreatment time: 0 min) of these same parameters were 37.4, 52.3, 4.0, 55.5, 119.2 and 73.2 mg/g, respectively. As far as the carotenoids were concerned, Lima et al. (2020) showed that bath-type ultrasound-assisted extraction (BUAE) significantly increased the total carotenoid contents compared to maceration extraction (ME) while probe-type ultrasound-assisted extraction (PUAE) had the lowest extraction power compared to BUAE and ME. The carotenoid contents of GPE (Guava pulp powder) and GWE (Guava waste powder) were 160.4 and 130.6 mg/100 g with BUAE, 135.6 and 79.0 mg/100 g with ME, 89.4 and 65.1 mg/100 g with PUAE, respectively. Idris and Sulaiman (2017) investigated the extraction performance of conventional and UAE of gallic acid (GA) from *Labisia pumila* and found that the GA concentration was enhanced 1.23-fold with US compared to the conventional method. Similarly, the impact of different ultrasonic

devices and sonication time on the amount of extracted phenolic acids and flavone glucosides was reported by Dent et al. (2015). The mass fractions of total phenols, rosmarinic acid, hydroxycinnamic acid, hydroxybenzoic acid, flavonoids, luteolin glucosides and apigenin glycosides ranged between 6893–7224, 3623–3549, 38.6–42.3, 25.4–39.4, 1570–1412, 1434–1550 and 194–218 mg/100 g, respectively, with a power of 100 W for a period varying between 8–12 min. These changed to 5834–5596, 2462–3700, 90.2–115.2, 110.6–100.5, 1552–1914, 1047–1620 and 144.3–216.1 mg/100 g, respectively, with an US power of 500 W. Šic Žlabur et al. (2016) compared the extraction power of classical methods and US in the isolation of some bioactives from lemon balm leaves. Results showed that the total phenols (1334 mg/L), non-flavonoids (751 mg/L) and flavonoids (581 mg/L) contents of samples extracted using classical methods were significantly higher compared to those extracted with US as given as 138.1–891.8, 80.3–507.9, and 57.8–391.6 mg/L, respectively. When UAE methods were compared in themselves, it was found that the extraction power of bioactives significantly increased from 5 to 25 min. This was justified by the extraction time that plays a key function in the separation of molecules (Koubaa et al. 2015; Šic Žlabur et al. 2015). The report of Radziejewska-Kubzdela et al. (2020) that investigated the effects of different processing methods on bioactive concentrations of *Berberis amurensis* juice, showed a significant increase in anthocyanin content in juice from processed mash using US (261 mg/100 g) compared to those from thermal (231 mg/100 g) and enzymatic processing (224 mg/100 g). It is important to specify that the values for the same parameter in juice from mash without pretreatment and raw material were 156 and 217 mg/100 g, respectively. The increase in anthocyanin values was found in pretreated samples explained by better extractability facilitated with the pretreatments. Similar observations were reported by Buchert et al. (2005) and Borowska et al. (2009). The extraction yield of phenolic compounds isolated from walnut shell using US compared to standard shaking method gave an extraction yield of 51.2 mg GAE/g dry weight (DW) with ultrasonic probe, which was two times higher than that of the shaking (20.6 mg GAE/g DW) and ultrasonic batch (25.8 mg GAE/g DW) methods (Han et al. 2018). In most of the cases presented above, the variations in extraction yield of bioactives are justified by the fact that UAE facilitate the break of the cell walls, reduce particle size and increase the mass transfer via the solid elements by the hydrodynamic process of cavitation bubbles (Veličković et al. 2006).

16.2.3 Emulsification

This is one of the first food processing methods of US application. The use of US in food emulsification was reported for the first time by Wood and Loomis (1927). Emulsification is a process by which two substances with different natures (generally oil and water) are mixed with the aid of an emulsifying agent into a homogenous solution (Awad et al. 2012). This process requires a source of energy which can be provided through mechanical or US agitations to facilitate the breakdown of

drops into small droplets (Thompson and Doraiswamy 1999). It is an important way to deliver the lipophilic bioactive molecules into a variety of food products (Chemat et al. 2011). The advantage of using US emulsification is that it requires less quantity of surfactants. The produced droplets are stable and smaller with this method (Abismail et al. 1999; Juang and Lin 2004; Canselier et al. 2002).

Also, ultrasonication generates the breakdown of cavitation leading to high energy microjets near interfaces and facilitates emulsification (Thompson and Doraiswamy 1999). US is required in the food industry during the production process of foods such as fruit juices, ice cream, butter, mayonnaise, margarine, tomato ketchup, milk homogenization, bioactives or aroma encapsulation (Wu et al. 2000; Karabacak et al. 2019). Gaikwad and Pandit (2008) demonstrated that enhancing ultrasonic irradiation power facilitates the dispersion and reduces droplets size. They also mentioned that the observed effects were proportional to the viscosity of the oil being analyzed and the tension at the interface. US emulsification is cheap, easy to use and can be incorporated for the quality improvement the emulsified foods products (Soria and Villamiel 2010). Reports from the literature showed that US nano-emulsification is capital for encapsulation and retention of lipid-soluble or non-polar bioactives. Encapsulation is a way to stabilize active principles through the organizing systems capable to preserve their chemical, physical and biological attributes as well as their delivery under appropriate conditions (Burgain et al. 2011). The non-polar molecules that are generally encapsulated after emulsification are lipid-soluble vitamins (A, D, E, and K), polyunsaturated fatty acids (docosahexaenoic acid, eicosapentaenoic acid, linoleic acid, linolenic acid, arachidonic acid, oleic acid etc.), terpenes (essential oils compounds: sesquiterpenes, monoterpenes etc.) (Banasaz et al. 2020). It was shown that US has been intensively used to make emulsions with lesser droplet sizes (40 nm), under appropriate conditions (Liang et al. 2017). Meghani et al. (2018) used an ultrasonic homogenizer (20 kHz, 10 min, 400 W) to encapsulate vitamin D using tween 80 as surfactant, cinnamon oil, phosphate buffer saline, water and Dulbecco's modified eagle's medium and obtained particle sizes of 166.2, 118.0, 170.8 and 40.5 nm. In a similar study carried-out by Bush et al. (2017), it was reported that US emulsification of docosahexaenoic acid in algae using soy lecithin as surfactant lead to droplet sizes of 258 nm. Lane et al. (2016) obtained particle sizes of 192 and 182 nm, after emulsification with US (24 kHz) and rotor-stator homogenizer (4000 rpm for 2 min), respectively, when flaxseed/high docohexaenoic acid algae oil encapsulated using soy lecithin and tween. Abismail et al. (1999) compared oil/water emulsions obtained using US (20 kHz, 130 W) and mechanical agitation (170 W) using identical systems (water, polyethozylated 20 EO sorbitan monostearate and kerosene) and found that the drop dimensions produced using US were significantly smaller and stable as compared to those of mechanical agitation. Besides, US required less amount of surfactants. However, detrimental effects of UAE can be observed, especially in products that are rich in polyunsaturated fatty acids. In the study of Juang and Lin (2004) availability and delivery of nutrients and non-polar bioactives was facilitated though US emulsification showing a deteriorative effect on oil quality. Chemat et al. (2004) reported the appearance of metallic and rancid odour in insonated sunflower oil.

Additionally, hexanal and hept-2-enal detected as a result of the sono-oxidation of sunflower oil. This suggests that the polyunsaturated fatty acids found in this oil were oxidized to produce the secondary oxidation products and off-odor. Similarly, Halim and Thoo (2018) determined that US treatment catalyzed lipid oxidation in sunflower and palm oils. As a result, it was recommended to carry-out US emulsification under suitable conditions for better results and less detrimental effects.

16.2.4 Enzymatic Hydrolysis

Enzymes play a very important role in food technology, as they act as reaction catalysts that enable them to convert raw materials into better food products. They are specific to their substrates, their catalytic efficiency and an increase rate of about 10^{10} or more compared to chemical reactions when working under appropriate physicochemical conditions. Enzymes have a various range of applications in food processing and production of all types of food products. They modify and improve nutritional, sensory and functional characteristics of foods. About 75% of industrial enzymes are hydrolytic enzymes amongst which proteases, lipases and carbohydrates dominate the market with 70% of sales (Chaudhary et al. 2015). They found their application in the dairy, baking, juice brewing industries; meat and starch processing, and extraction of bioactives (Chaudhary et al. 2015; Liang et al. 2017; Nag and Sit 2018). Enzymatic hydrolysis can be defined as a process by which enzymes enable the breakdown of bonds in macromolecules by releasing a molecule of water (Yin et al. 2012). Enzymatic hydrolysis is a capital in human and animal nutrition, through its role in the digestion of foods for assimilation or absorption. The method is also of great importance for the food industry where it is exploited to enable the release of more free nutrients and bioactives that can be exploited for nutrition or technological purposes.

US has been used to catalyze or enhance the enzymatic hydrolysis of macromolecules and bioactives from foods in order to boost the release of specific reaction product for further use (Du et al. 2018).

Lunelli et al. (2014) studied the enzymatic hydrolysis of sugarcane bagasse into fermentable sugars with and without US. They found that the concentration of fermentable sugar obtained using the sonication method was 0.26 g [sugar]/g which was the double of the one obtained without ultrasound under similar conditions (temperature: 50 °C, 10% mass of enzyme, moisture: 75% after 240 min). The synergistic influence of glucoamylase and US on the hydrolysis of starch was investigated by Wang et al. (2017). The results indicated an increase in the hydrolysis extend with reaction time, which was optimal with US treatment (7.20 W/mL at 10 min). They also found that the US application has no effect on the optimal temperature of the enzyme, but it accelerated the thermal inactivation of glucoamylase.

From the investigation of Vidal et al. (2018) on the enzymatic breakdown of pepsin catalyzed with and without US and the impact on the properties of

hydrolysates obtained from collagens, it was found that the treatment that produced the best antioxidant capacity for the fiber sample was with 4% enzyme and assisted ultrasound (40.7%) leading to 21.7% hydrolysis. Accordingly, Du et al. (2018) enhanced the enzymatic breakdown of corncob by pretreating them through soaking in an aqueous ammonia solution assisted by ultrasound (USAA). They noticed that the highest cellulose and sugar recovery yields as well as the delignification registered under optimum conditions (10 W/mL, sonication time: 11.66 min) were respectively 83.8, 77.6 and 84.7%. They also indicated that a pretreatment using USAA selectively take out lignin, hemicellulose and have not effect on cellulose. The authors explained this by the synergistic impact of ammonia and hydroxyl radicals generated during cavitation that increased the depolymerization of lignin and breakdown of lignin-hemicellulose bonds (Ramadoss and Muthukumar 2014).

US was also used in the hydrolytic degradation of lard to produce free fatty acids under the catalytic action of two lipases (1,3-specific lipase from *Rhizomucor miehei* abbreviated pRML and a non-specific mono- and diacylglycerol lipase from *Penicilium cyclopium* abbreviated MDL) in hydrophilic medium (Huang et al. 2020). Results of their investigations showed that the highest hydrolytic degradation rate of the fat after 6 h at 45 °C using pRML and MDL only was 39.9 and 8.5% respectively. The combination of pRML and MDL led to a hydrolysis rate close to 78.1%. However, the combination of pRML and MDL accompanied by 5 min ultrasound treatment before the reaction led to a hydrolytic degradation rate of 97%. The different in hydrolysis rate recorded was attributed to the cavitation occurrences generated by ultrasound that can improve the dissolution of substrates, facilitate mass transfer, destroy weak connections, modify protein conformations, thereby enhancing the degree of product formation (Zhu et al. 2003; Lerin et al. 2014).

The influence of UAEH on the antioxidant capacity and physicochemical of protein hydrolysates from corn was investigated by Liang et al. (2017). These authors found that ultrasound increase the production of short chain peptides (MW: 200–3000 Da), specifically those containing hydrophobic amino acids. Additionally, 40 suspected antioxidant peptides were detected in the sonicated hydrolysates, showing the potential importance of this method in the preparation of antioxidant peptides from corn. Nag and Sit (2018) with UAEH of polyphenols from pomegranate peels reported similar information. The values of total phenolic content, total flavonoid content and antioxidant activity were found 19.77 mg GAE/g, 17.97 mg QE/g and 74.2% respectively, under optimal conditions (US duration: 41.45 min, temperature: 44.85 °C, viscozyme concentration: 1.32 ml/100 ml, incubation: 1.82 h).

16.2.5 Drying

Drying or dehydration is one of the famous and oldest techniques used in the preservation of food. It uses thermal energy such as hot air, convection frying, sunlight, smoking etc. (Cohen and Yang 1995). For years and for preservation or processing

purposes, humans have been drying foods such as meat, fruits, vegetables, fish, prawns, tubers etc. to ensure their availability during out of season times for food security reasons (Pakbin et al. 2014). Nowadays, there is a section only for dehydration almost in all food processing companies in the world. The aim of this technique is to eliminate the water from the food in order to inhibit the biological and chemical alterations.

However, during this process, practical difficulties related to the slow mass exchange can occur depending of the nature of the food and processing conditions (Başlar et al. 2016). US treatments are used in the drying process to accelerate the mass transfer and address these challenges. Additionally, it uses low temperatures which are good for food quality preservation (Başlar et al. 2016; Cárcel et al. 2011). Reports have shown that US can be used to pretreat fruits and vegetables before air or and osmotic dehydration (Garcia-Noguera et al. 2014). Accordingly, it was demonstrated that water diffusion is enhanced after US treatment, thereby reducing the drying time by 16% (Opalic et al. 2009). The rise of water diffusivity circulation has been estimated to 28.8% when submitted to ultrasound for 20 min. This was attributed to the creation of microchannels in the structural organization of the substance due to ultrasonic effects (Fabiano 2008). Therefore, the fact that US can increase the circulation of water during air and osmotic dehydration makes this technique an important process complementary to the common drying methods (Oliveira et al. 2011).

US has also been proven to be used to make dried fruits and vegetables with reduced sugar content (Brncic et al. 2010). This can be explained by the release of water soluble entities in the bath during the treatment (Cui et al. 2010).

The effect on ultrasonic drying on food bioactives have also been reported in the literature. In one study, (Lagnika et al. 2019) evaluated the effect of ultrasound-assisted-drying (UAD) on the total phenolic (TP), total flavonoid (TF), vitamin C and DPPH radical scavenging activity of cashew apple. Results demonstrated that UAD preserve better the active principles and increase their antioxidant activity of cashew apple. The TP, TF, vitamin C contents and antioxidant of cashew apple dried with US were respectively 2.41 mg/ml, 0.24 mg/ml, 5.7 mg/100 g and 82.89% while the value of these same parameters on samples dried without treatment were 1.80 mg/ml, 0.12 mg/ml, 3.01 mg/100 g and 74.46% respectively. The authors explained these differences with the increase in extractability of TP, TF and vitamin C by US which lead to the appearance of pores in plant tissues and that enhance the extraction of polyphenols (Amami et al. 2017). Vallespir et al. (2019) studied the impact of US-assisted low temperature dehydration on kiwifruit bioactives. They found that drying at 15 °C without pretreating with US promotes bioactive losses of 39–54%. However, the application of ultrasonic treatments during dehydration at 15 °C reduces the losses to 15–14%. Similar results were reported by Ren et al. (2017) who demonstrated that ultrasonic pretreatment (20 kHz) of onion before hot-air and freeze drying enhance quercetin retention compared to untreated samples. Vallespir et al. (2019) justified their findings with the short drying time that reduced the exposure of kiwifruit and consequently their active principles. Moreno et al. (2017) reported that US can initiate a reaction mechanism in the tissue that lead to

the production of new phenolic compounds through combination of present molecules but also through stimulation of secondary metabolism. Fernandes et al. (2015) estimated the water diffusion for air drying method with and without ultrasonic pretreatment. An increase in diffusivity by 33–89% was recorded when applying ultrasound. In the same line, a significant increase in vitamins B1, B2, B3, B6 and B5 as well as protein was registered with ultrasound treatment. The authors attributed this to the release of these molecules from the cell membranes. UAD pretreatment was used to incorporate microencapsulated polar and non-polar nutrients into foods (Rojas et al. 2019). It was found that pretreatment with US rises the iron content of pumpkin and apple more than 1000 times compared to untreated samples. In the same line, the carotenoid concentration was improved in about 430% with US while the untreated sample exhibited a carotenoid percentage of 65. US treatment can be beneficial for the integration of nutrients into the food matrix.

Though US is used for food preservation, if control is not applied, it can degrade the quality of the final product, rending it improper by changing its organoleptic (color, taste, flavor, texture, odor) and nutritional properties (loss of essential fatty acids, destruction of vitamins) (Zhang et al. 2006). It can also lead to the reduction of bioactives retention (Vallespir et al. 2019). Conditions should be well defined in order to preserve these nutrients and active principles.

16.3 Effect of Ultrasound on Bioaccessibility/Bioavailability of Food Bioactives

Fruits, vegetables and other classes of foods are rich in bioactives with multiple beneficial biological and physiological effects. However, in order to exert their role *in vivo*, these bioactives must have good retentions during industrial processing and nutrition (resistance to digestion) (Lafarga et al. 2019). Bioaccessibility is the magnitude of a specific molecule or substance that is released from the food to the intestine for absorption (Dima et al. 2020). As far as bioavailability is concerned, it gives an idea of the amount of molecule or substance that is ingested to produce systemic effects (Toutain and Bousquet-Melou 2004). It is well known that processing of foods can change the bioaccessibility of molecules with beneficial effects on health (Rodríguez-Roque et al. 2015). In previous sections, it was shown that US facilitates the release of cells content through the disruption of the cell barrier and therefore increase the amount of active principles extracted. Therefore, the application of US treatments on foods can enhance the bioaccessibility and bioavailability of their active principles.

In one study, Lafarga et al. (2019) investigated the influence of ultrasonic processing on the bioaccessibility of phenolic antioxidants (PA) of lettuce, tomato, red pepper, green pepper and zucchini. They found a significant increase in bioaccessibility of PA in green pepper and lettuce while no change was recorded with the other vegetables. As far as lettuce is concerned, the initial phase (methanolic

extraction) gave total phenolic contents (TPC) of ~10 and ~6.80 mg/100 g for sonicated and untreated lettuce respectively; the TPC from the sonicated extract being significantly higher than that of the untreated sample. At the gastric phase which represents the stomach conditions, no difference was recorded in the TPC of both samples. However, at the intestinal phase, phenolic antioxidant obtained from the sonicated lettuce (~18.69 mg/100 g) was significantly higher (more available) than that of the untreated sample (~11 mg/100 g). Concerning the green pepper, initially, the TPC in sonicated and untreated samples were ~ 50 and 35 mg/100 g respectively. At the gastric phase, the TPC in the sonicated sample (~120 mg/100 g) was significantly higher than that of the untreated sample (~50 mg/100 g). At the intestinal phase, the TPC in the sonicated sample (~60 mg/100 g) was still significantly elevated compared to the untreated one (~35 mg/100 g). The sonicated samples also exhibited highest antioxidant activities in almost all the cases. It is important to note that TPC and antioxidant activities at the gastric phases were significantly higher compared to the intestinal phase. The authors attributed the higher TPC and antioxidant activities after the gastric and intestinal stages to the rise in polyphenolic compounds when compared to the initial stage. Generally, the TPC and antioxidant activities at the intestinal stage were lower than that of the gastric one. The authors explained this by the fact that phenolic compounds are very sensitive to alkaline conditions and could have been decomposed due to the alkalinity of the intestine (Bermúdez-Soto et al. 2007). According to Jamali et al. (2008), different pH values can affect the availability and biological activity of phenolic compounds and may render them more reactive under acidic conditions. In summary, a few studies are available on the effect of US treatment on the bioaccessibility and bioavailability of food bioactives.

16.4 Effect of Ultrasound on Food Allergy

Food allergy is a situation where the immune system response is abnormal or excessive due to the consumption of specific foods and additives (Nayak et al. 2017). According to the National Institute of Allergy and Infectious Diseases (NIAID), food allergy is an adversative health effect that arises reproducibly on contact to proteins, or antigens, which are constituents of food mediums, thereby producing a specific immune response (Nayak et al. 2017). Reports from the literature show that approximately 170 foods or their constituents are allergenic (Nayak et al. 2017). The major foods that are involved in these side effects are fish, milk, eggs, soybeans, peanuts, wheat, shellfish and tree nuts (FALCPA 2004).

For food safety reasons, the removal or reduction of allergens from food is gaining great interest. Among the techniques that have attracted researchers' attention is US, even though it is still in its infancy. As previously explained, high intensity US (20–100 kHz) works through mechanical waves initiating physical and chemical changes through the production of sonication cavitation bubbles in foods leading to compression and rarefaction until collapse when the bubble size is critical. The rise

in temperature and pressure around the collapsed holes is the foundation for destroying the structure of allergens and their action. The exerted chemical and mechanical effects can modify the structure of the native allergenic protein leading to the establishment of inter- and intra-molecular connections (Mawson et al. 2011; Soria and Villamiel 2010; Lee et al. 2009). It is important to note that with low intensity US the mechanism involved is acoustic streaming. Here, there is no production of bubbles (Leighton 2007; Alzamora et al. 2011). Microstreaming when applied with force could breakdown low energy bonds such as hydrogen and Van der Waals interactions in the polypeptide chains, thereby denaturing the protein (Tian et al. 2004). Zhen-xing et al. (2006) studied the effect of high intensity US on shrimp allergens. They found that high intensity US reduces the allergenicity of shrimp allergen (ASA). The decreases in ASA determined using ELISA with pool serum of shrimp allergic patients were reducing with US treatments (from 0–180 min) ranging from 100–18.7% (for shrimp allergen) and 100–31.1% (for shrimp muscle allergen), while with polyclonal antibodies against shrimp allergens, the decrease was from 100–25.3%. These findings are in line with those of Wang et al. (2020) who reported that US treatment significantly reduces the allergenicity of fresh milk and that it could help in the production of hypo-allergic cow milk.

16.5 Development of Functional Food Ingredients

Consumers are more interested today with foods not only containing essential nutrients, but also substances which may have beneficial technological and health effects (Hernández-Carrión et al. 2014). Functional foods are products containing several biologically active principles and which after consumption a contemporary diet have a beneficial impact on the overall condition of the body such as improving health and decreasing the possibility to contract some disorders (Bigliardi and Galati 2013). They are foods containing vitamins, fatty acids, mineral elements, dietary fibers, and foods with bioactives such as phytochemicals (antioxidants and others) and probiotics (Butnariu and Sarac 2019). The development of functional foods involves the addition or insertion of selected active principles with beneficial biological actions. Specific peptides with good biological activities compared to their mother protein, since in many studies, their ability to reduce the risk to develop chronic disorders as well as their beneficial contribution to human health has been studied (Udenigwe and Aluko 2012).

Several studies have been conducted so far in order to produce new constituents and methods that will contribute to the expansion of functional foods for improving consumers' health. Amongst the emerging methods used to develop these products figures high-intensity ultrasound (HIU) (Ozuna et al. 2015). For example, HIU has been used several times in the generation of protein hydrolysates and active principles as functional food constituents. US treatments have been used to increase the extraction yield of bioactives such as phenolic compounds, to facilitate the enzymatic hydrolysis of proteins and produce peptides with functional attributes. HIU

affects the low energy bonds (hydrogen, Van-der-Waals, hydrophobic, ionic interactions etc.) and destroy the structure of macromolecules such as proteins and bound phenolic compounds through cavitation. These structural transformations may lead to an increase in the concentration of bioactives (Ozuna et al. 2015). Stefanović et al. (2014) demonstrated that high intensity pre-treatment (15 min at 25 °C) at 40 kHz and 21.3 W of proteins from egg white respectively led to a significant increase in the degree of equilibrium and initial rate of alcalase hydrolysis by 14 and 140% while after 60 min, had a negative effect. This was justified by the aggregations of proteins that reduce access of unfolded and aggregated proteins to their specific proteases. Uluko et al. (2015) investigated the influence of HIU (800 W, 1–8 min) on the peptide profiles and angiotensin-converting enzyme (ACE) inhibition of MCP hydrolyzates. They found that the number of peptides with molecular weight of 0.1–2 kDa increased with the application of high US pretreatment for 5 min and was correlated to the rise in ACE inhibitory activity. After designing a pilot plant in order to enhance the antioxidant activity of MPC hydrolysates which integrated HIU pretreatment, ultra- and nano-filtrations, spray and freeze drying, they noticed that the pilot plant enhances the isolation of ACE inhibitory and antioxidative peptides in complex protein hydrolysates. They also found that the fraction with 0.2–3.5 kDa exhibited highest ACE inhibitory activity and the fraction with 3.5–8 kDa exhibited highest antioxidant activity. Xu et al. (2013) demonstrated that HIU treatment at 400 W increased the antioxidant capacity of denatured soybean meal hydrolysate using neutrase. In their report, the *in vivo* anti-fatigue property and *in vitro* antioxidant potential increased with the reduction in molecular weight of the meal hydrolysate. In contrast, Yang et al. (2011) demonstrated that HIU pretreatment inhibit the alcalase hydrolysis of soybean sauce lees proteins. Similarly, Ren et al. (2013) showed that sweeping frequency ultrasound (SFU) and fixed frequency ultrasound (FFU) enhance the DH of zein by about 11.5% compared to the control. The ACE-inhibitory activity of zein hydrolysates was increased by 12.3 to 114.7% by SFU and FFU. They also reported that pretreating zein with SFU increase the α-helical conformation of polypeptides by 3.4% while the β-sheets and turns were raised by 24.4%.

The US treatment influence the bioactive content of foods as well as their bioaccessibility and bioavailability. It is clear that processing methods may affect the functionality of active compounds, this by modified their concentration, structure, physicochemical parameters etc. Several reports from the literature show the impact of US treatments on the functionality of bioactives. A few information are available on the effect of US on functionality of food bioactives. It will be important to focus more on this aspect with more specificity to see how the functionality of bioactives is affected by some changes such as structural, and physicochemical modifications.

16.6 Conclusion

The present chapter aimed at gathering information on the effect of US treatment on food bioactive and their importance in food science and technology. Data show that US treatments represents a fast, efficient and consistent option to enhance food quality, this by facilitating processes such as extraction of nutrients and bioactives from foods, making them accessible and available; emulsification; enzymatic hydrolysis; drying and reduction of food allergens. The method is also significantly useful in the development of new ingredients and products with a unique functionality. On the other hand, if the processing conditions are not well respected, US treatments can instead damage the food products, by promoting chemical degradation reactions such as lipid oxidation. Monitoring the physicochemical and composition properties of foods during treatments and storage is capital for the manufacturing good and stable foods. US is simple to use, rapid, portable and cheap. It is of great importance today in renowned research laboratories, large scale food industries and pilot plants. It is a good method for the extraction and preservation of food bioactives.

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Chapter 17
Influence of Membrane Separation Processes on Food Bioactives

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17.1 Introduction

Nowadays, membrane processing is widely used in food processing, especially in beverage industry over the last 40 years (Grandison 2003). A membrane, in general terms, can be defined as a selective thin layer of a semipermeable material that under a potential gradient can separate fractions from a solution (Asad et al. 2020). Membranes are selective and allow some materials to pass through (the permeate) and other materials to keep retained (the retentate) on it. Sometimes it is the retentate that is desired, in others the permeate stream, while sometimes both products are of value (Grandison 2003). The potential gradient can be generated by pressure, temperature, electrical or concentration difference and the separation can be based on their sizes or affinity (Asad et al. 2020). The use of membranes in the beverage industry has a lot of advantages compared with other thermal and nonthermal techniques. Membrane technology provides higher selectivity, minimum effect on temperature, it is easier to install and remove and consumes less utilities. What is more, thermal techniques can reduce the product's quality and induce changes in its chemical compounds, whereas nonthermal techniques as centrifugation produces a loss in volatile compounds needing longer times. These disadvantages are overcome by membranes, that have the capability of separate, concentrate, clarify, dealcoholize, pH stabilize, sugar regulate and cold sterilize beverages (Peyravi et al. 2019).

Briefly, membranes can be classified attending to three main characteristics: material, configuration and pore size. According to its material, membranes can be organic or inorganic. Organic ones are the most common membranes used, mainly made of polymeric materials. Examples of that are polysulfone, polyethersulfone

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or polyvinylidene fluoride, usually hydrophilic materials that have two key advantages: they have higher resistance to attachment of organic material on their surface and they become wet easily, which makes them more permeable for a given pore size (Voutchkov and Voutchkov 2017). On the other hand, inorganic membranes can be metallic or zeolite-based, but the most used for food applications are ceramic membranes. These membranes have superior mechanical, thermal and chemical properties but are more expensive than organic membranes, so a balance between these factors must be done in order to select the more appropriate (Benfer et al. 2004).

Regarding the configuration, there are a wide variety of geometries available in the market. According to the membrane geometrical configuration, there are principally two types of ceramic membranes available, flat and cylindrical membranes. Flat ceramic membranes can be in a disc form with low packing density, usually used in small-scale pilot plant or laboratory applications. Also, they can be in a sheet form, the most common. They are around 6 mm of thickness and can be mono or multi-channelled. Usually they are packed in modules which makes it easier to replace that the disc form. This shape can be used with higher turbidity feeds. Cylindrical membranes do not have to be obligatorily of circle form, other forms as hexagons are included in this type of tubular membranes. In general, they can be of three types: single-channel, multichannel or hollow fibre membranes. Single-channel membranes have mean duct diameter of 10–25 mm and are suitable for fluids with high turbidity and large amount of suspended solids. They are very easy to clean because of their diameter and robustness. Also, this type of membranes permits high cross-flow velocities to control fouling. Multi-channel membranes arose as an improvement to increase the packing density of tubular membranes. They are also called monoliths with a surface/volume ratio up to 782 m^2/m^3, and have similar advantages as single-channel membranes. The exterior diameter of this type of membranes is variable and sometimes can be limited if the running configuration is inside-out. This is due to the increment of the pressure drop in the monolith because of the long and tortuous path that the liquid is forced to pass from inside to the outer surface. Some enterprises solve this problem by adding extra conducts in the tubular membrane through which the permeate can flow till the collection (Lee et al. 2015). Finally, hollow fibre membranes are usually made of ceramic materials with 2–4 mm diameter or lower, depending on the maker. This novel system consists in more compact modules with higher effective membrane surface, avoiding the problem found with multi-channel membranes allowing the use of lower transmembrane pressures to drive permeate flow (Voutchkov and Voutchkov 2017). All the membranes described are exposed graphically in Fig. 17.1.

Regarding the pore size, we should differentiate between dense and porous membranes. For food applications the most used are porous membranes such as microfiltration (0.1–10 μm), ultrafiltration (0.01–0.1 μm), nanofiltration (0.001–0.01 μm) and membrane distillation (0.2–1.0 μm) ones, which perform separation processes according to sieving mechanisms in which molecules larger than the membrane pores are rejected while smaller ones pass through (Asad et al. 2020).

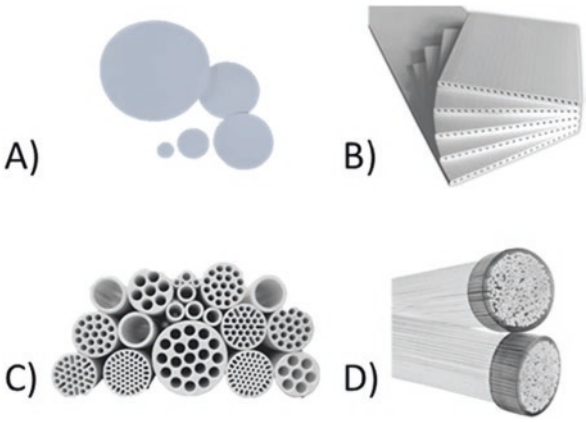

Fig. 17.1 Ceramic membranes configurations: (**a**) flat disc membranes; (**b**) flat sheet membranes; (**c**) mono and multi-channel membranes; (**d**) hollow-fibre membranes (Lee et al. 2015)

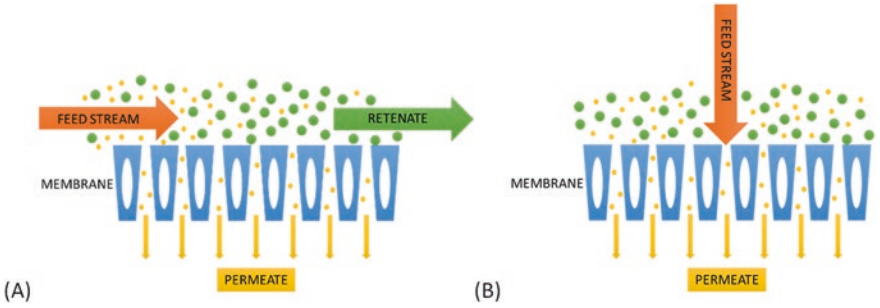

Fig. 17.2 Schematic diagram of membrane filtration modes: (**a**) cross-flow; (**b**) dead-end filtration

In general, membrane performance is modulated by two parameters: flux and selectivity. The flux is the volume of a substance that permeates through the membrane per unit of area for a period of time, meanwhile the selectivity (rejection) is the fraction that is retained on the membrane. Darcy's law describes roughly the flux through a porous membrane:

$$J = A\Delta p,$$

where J is the volumetric flux and Δp is the transmembrane pressure, i.e. the difference between the pressure in the feed side and the permeate side. A is the hydraulic permeability constant and depends on the membrane morphology (material characteristic, porosity and configuration) (Asad et al. 2020).Cross-flow and dead-end filtration are the two types of filtration modes according the direction between the feed flow and the permeate flow. They are schematically described in Fig. 17.2. The main difference between them is the direction in which the feed crosses the membrane. When the direction is tangential to the membrane surface and perpendicular to the permeate it is cross-flow filtration, whereas when the direction of the flux is perpendicular to the membrane surface it is called dead-end filtration. The second one is

more prone to accumulate particles on its surface and trigger fouling, meanwhile in cross-flow filtration the direction of the flux respect to the membrane helps remove deposited particles on the membrane layer (Asad et al. 2020).

The principal limiting factor in membrane filtration is fouling, that includes (i) adsorption, (ii) pore blockage, sealing or constriction, (iii) precipitation and (iv) cake formation. Fouling takes place when particles are deposited on the membrane surface or in the pores of the membrane. The first and easy way to minimize fouling is implementing cleaning cycles to restore its productivity, which must be optimized to minimize costs and plant shut-downs. Reducing the permeate flux also has been reported to help to minimize the fouling (Chen et al. 2018; Stoller and Ochando-Pulido 2015; Stoller et al. 2017). To mitigate foulant agglomerations and to increase the effective surface, structural modifications of the membrane surface have been demonstrated to be useful by inducing secondary flow that produce eddies and in consequence reduce fouling (Barambu et al. 2019). Specifically, when using rotating disk membranes, the fouling is easy to be decreased by increasing the rotation speed and transmembrane pressure. It is due to the high centrifugal force and the radial components of drag on particles near the membrane surface (Engler and Wiesner 2000). Besides, sometimes particles added on membrane filtration systems or suspended in the feed flow can as well behave as fouling mitigation agents (Wang et al. 2020). It is similar to the new concept of using a "dynamic membrane" made of particles deposited via permeation grand on the membrane surface. They act as a secondary membrane minimizing fouling enabling higher fluxes (Anantharaman et al. 2020). Bubbling is another technique used to enhance the filtration process by reducing fouling by injecting gas into the feed stream (Cui and Taha 2003). Novel approaches report that artificial intelligence and machine leaning are crucial techniques for better control membrane fouling in filtration processes and that reduce cleaning costs (Bagheri et al. 2019). Anyway, membrane processes in the industry need to have three types of service support facilities and equipment: backwash, CIP and feed systems chemical cleaning (Voutchkov and Voutchkov 2017).

Membrane technology is actually widely used in food and beverage industry thank to the improves in the last years in module designs, advances in membrane's materials and the better understanding of the fouling phenomena (Cheryan and Alvarez 1995).

17.2 Membrane Separation in Non-alcoholic Beverages

17.2.1 Membrane Filtration and Juice

A juice is an unfermented, but fermentable liquid obtained from the edible part of fruits or vegetables in good condition, duly ripe and fresh or that have been kept in good condition by appropriate procedures. Some juices can be made together with their seeds and peels, which are not normally incorporated into the juice. The juices

are prepared using appropriate procedures that maintain the essential physical, chemical, organoleptic and nutritional characteristics of the fruit juices from which they come. They may be cloudy or clear and may contain restored components of volatile aromatic and flavouring substances, all of which must be obtained by appropriate physical procedures and which must come from the same type of fruit (FAO/OMS). This liquid is contained in fruits and vegetables tissues and can be extracted from them by pressure or including an intermediate process such as cooking, grinding or centrifugation. Fruit juices are consumed worldwide because of their flavours, tastes and freshness. However, their beneficial health effects have made them a regular consumption product. As sources of polyphenolic compounds and carotenoids, fruit juices are interesting to introduce them in the daily diet. An example are purple grape juices that have shown inhibitory effects on low-density lipoprotein oxidation at the same way as the red wine with high antioxidant activity (Frankel et al. 1998). More extensively, there are a lot of in vitro and in vivo studies on cells, animal or humans in which it is confirmed that fruit juices act as potent free-radical scavenger and metal chelator by increasing the activity and expression of endogenous antioxidant enzymes, such as catalase and supper-oxide dismutase, and thereby reducing plasma free-radical release and damage to DNA. These effects have been attributed to its content in polyphenolic compounds as resveratrol, gallic acid, quercetin and catechins (Zielinski et al. 2014).

A general elaboration process schedule of fruit juice is exposed in Fig. 17.3. Although some pre-treatments are still necessary before membrane filtration, such as enzymatic treatments, this technology has been proposed and investigated for juice clarification and high sensory quality drink production. The main purpose to introduce membrane filtration in this step is to retain compounds of high molecular weight as pectin, proteins and colloids, letting the permeation of compounds of low molecular weight as sugars, acids and salt with water. Apart from that, membrane filtration has also been examined to stabilize fruit juice microbiologically. In this case, the objective is to produce a clarified juice free of spoilage microorganisms, letting a retentate with fibrous concentrated pulp. For both applications, microfiltration and ultrafiltration seem to be the better options for juice processing, while nanofiltration in non-alcoholic beverage is only used for the separation of contaminants or concentrate some interesting compounds as bioactive compounds (Reis et al. 2019). For lime juice, membrane ultrafiltration has been found to be better than microfiltration in terms of flux. Also, molecular weight cut-off membranes have been proved instead of only size discrimination but the effect is insignificant on the quality of filtered juice (Rai et al. 2006). The clarification of prickly pear juice has been carried out using a hollow fibre membrane with high permeate flux, high retention of turbidity, with low loss of total soluble solids, carbohydrates, polyphenols, betalains and antioxidant activity, and without changes in colour, maintaining a product rich in bioactive compounds with good technological properties (Castro-Muñoz et al. 2018). On the other hand, for apple juice, the lower the pore size, the lower the flux rate. Comparing different ceramic membranes at the same "optimal" pore size (0.2 μm) hydrophilic polyvinylidene fluoride and polyethersulfone seem to be the most efficient materials upon crossflow filtration (Mondor et al.

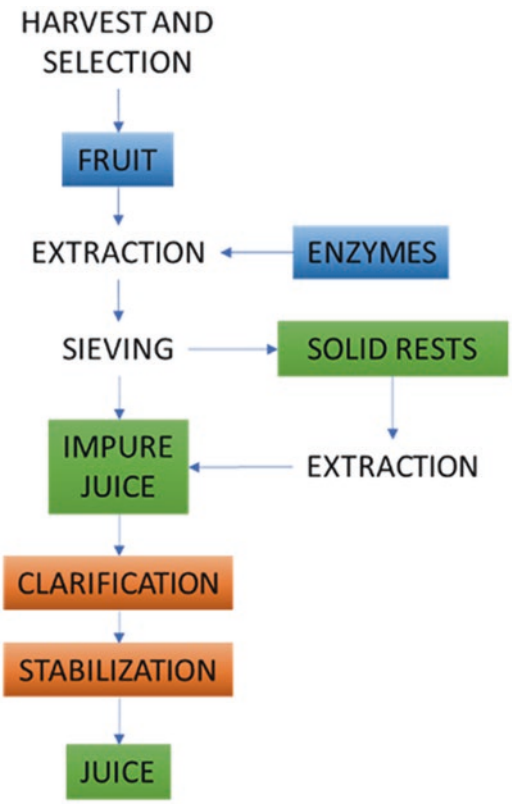

Fig. 17.3 Juice elaboration process

2000). Nylon nanofibrous membranes as substitutes to commercial polyamide membranes also are a good alternative for clarifying apple juice. They remove the turbidity, colour and bitter phenolic compounds, maintaining intact the antioxidant capacity (Fuenmayor et al. 2014). Asymmetric stainless steel membranes also has been studied in apple juice with very good potential prospects for its results (Qiangbing et al. 2015). For obtaining a high-quality apple juice, in this clarification step with membrane, filter aids have been studied for improving it, giving higher permeate fluxes and lower membrane fouling. Bentonite is the better filter-aid followed by polyvinylpolypyrrolidone. Although with the second one, the best tannin rejection is obtained, bentonite increases the permeate flux and improves the juice colour without changes of pH, total acidity, and keeping the contents of total sugar, organic acid and vitamin C, which are important quality indices in fruit juice (Youn et al. 2004). Usually, enzyme hydrolysis and pasteurization are pre-treatments followed by the clarification of the apple juice using membrane filtration. The possibility of eliminate these pre-treatments has been studied. When the juice is only pasteurized the flux of the membrane filtration is lower due to gelatinization at the membrane surface caused by cross-linking between deposited pectin and starch molecules. However, in this context, the permeate flux can be increased when the

transmembrane pressure, the cross-flow velocity, or the temperature are increased. So, when eliminating the enzyme hydrolysis pre-treatment, the concentration and the viscosity of the feed are the major factors influencing the permeate flux in the membrane filtration of the apple juice. Taking care of this, the clarified juice obtained has very good sensorial qualities without starch, pectin and thermoacidophilic bacteria, letting eliminate this pre-treatment without detriment in the quality of the final product (He et al. 2007). Also, the elimination of the pasteurization step has been studied, but only with selective microorganism in apple juice. Species of genus *Alicyclobacillus* may cause serious problems in fruit juices as this thermophilic bacterium produces endospores that survive mild heat treatments, and can germinate in fruit juices. So, to eliminate those spores in apple juice nitrocellulose membrane filters can be used (Lee et al. 2007). Biofilms of yeast strains (*Candida* and *Rhodotorula*) naturally present in apple juice also can be eliminated by membrane filtration process, specifically by polyvinylidene fluoride ultrafiltration membranes (Agustín et al. 2019). Other juices as pineapple juice can be processed (clarified and stabilized) by polymeric hollow fibre membranes of 0.20 μm without microbial growth or loss of stability and physicochemical characteristics during storage at various temperatures after 6 months (Laorko et al. 2013). In passion fruit juice it has been demonstrated that the predominant fouling mechanism depends on the applied pre-treatment. Comparing centrifugation, enzymatic liquefaction and chitosan coagulation, the last one gives the highest reductions of turbidity and enables the highest permeate flux with the posterior microfiltration with hollow fibre membranes of 0.4 μm. Upon the other two pre-treatments, cake formation is the major fouling factor, while in chitosan coagulation internal pore blocking is the driving fouling mechanism (Domingues et al. 2014). Polyvinylidene fluoride membranes with pore size of 0.22 and 0.45 μm can accomplish the clarification of pomegranate juice satisfactorily. However, in both cases the permeate flux decreases over time as a result of the membrane fouling (Mirsaeedghazi et al. 2010). When ultrasound technology is added to the enzymatic pre-treatment of pomegranate fruit juice, in the consecutive membrane filtration with hydrophilic mixed cellulose ester membrane with a pore size of 0.45 μm, the intensity of the cake formation is reduced. So, ultrasound waves can increase the capability of membranes performance (Aliasghari Aghdam et al. 2015). The clarification of watermelon juice by tangential cross flow microfiltration with ceramic membrane of pore size of 0.1 μm let obtain a juice with almost 100% of the turbidity eliminated and without loss of the lycopene present and its antioxidant activity (Gomes et al. 2013). However, in red beet juice the clarification with a hydrophilic membrane of 0.45 μm pore size not only decreased the turbidity, but also the total phenolic content, total soluble solids, juice colour, antioxidant activity and betacyanin and betaxanthin content (Amirasgari and Mirsaeedghazi 2015). Apart from that, it has been demonstrated in red beet juice that the best efficiency of membrane processing is at high transmembrane pressure and feed flow rate. Under this conditions the predominant fouling mechanisms are standard blocking and cake resistance (Dos Santos et al. 2016).

Also, membrane filtration has been used in purple sweet potato juice for obtaining an anthocyanin enriched juice. Although anthocyanins retention was observed

during the filtration process, purple sweet potato microfiltered juices generate better permeate with compromise between appearance transparency and anthocyanin content. Thus, this technique could be an interesting tool to clarify sweet potato juices, rich in anthocyanins, and to obtain products with high sensory quality (Zhu et al. 2017). Membrane ultrafiltration is used combined with electrodialysis for concentrate cranberry juice for obtaining an extract with natural phenolic antioxidant compounds such as proanthocyanins and anthocyanidins, in addition to another cranberry juice, enriching it. Cranberry is known for its benefits in human health and its nutraceutical potential and this enrichment improves their functionality, enhancing it health benefits. With the use of membrane filters the antioxidant capacity of the enriched cranberry juice can increase by almost 20% (Bazinet et al. 2009). Polyvinylidene fluoride and polyethersulfone membranes have been demonstrated to be the best options for concentration of cranberry juice without affecting the sugar concentration, and specially filtration membranes with moderate negative charges which seem to facilitate the electro-transfer of anthocyanins due to the attraction between opposite charges (Husson et al. 2013). However, in consequence the citric and malic acid content of the enriched juices decrease significantly with effects in its taste (Bazinet et al. 2012). Ultrafiltration with membranes of 100 kDa molecular weight cut-off in pequi juice has been used for obtaining a concentrate of carotenoids and polyphenols with the retention of almost 100% of the present bioactive compounds (Gomes et al. 2014). Membrane microfiltration followed by membrane nanofiltration has been used for concentrating bioactive compounds from strawberry juice. Microfiltration pre-treatment is needed for clarify the juice and remove suspended particles, and this can be achieved by a polyamide membrane with 0.4 μm pore size. Nanofiltration with polyvinylidene difluoride membrane can reach a retention up to 95% of the major anthocyanin compounds of strawberry juice (pelargonidin-3-O-glycoside) with it antioxidant activity intact and maintaining the red colour and the rest phenolic compounds in the juice (Arend et al. 2017). Similar to this, crossflow microfiltration with a ceramic multichannel of 0.2 μm in melon juice let obtain a juice with almost all the composition intact which has not undergone any thermal treatment and a glowing orange retentate rich in carotenoids, provitamin A (Vaillant et al. 2005).

In addition, in some juices, membrane distillation has been applied for concentrating or purifying some compounds with good performance results. In apple juice it has been discovered that using polyamide membranes the temperature influences the flux significantly. An apple juice concentrated till 50% in solids is the higher that can be obtained without loss of productivity and biological value (Gunko et al. 2006). For the concentration of blackcurrant juice with hollow fibre membranes the influence of the temperature has also been confirmed. The juice concentrated by this way can reach at least 58 °Brix protecting the juice from deterioration (Kozák et al. 2009). Regarding pear juice, hollow fibre membrane made of polypropylene has been reported to be the better one for recovering the main pear aroma compounds (Diban et al. 2009). Membrane filtration is used as the first step for producing inulin from chicory juice, simplifying this process, increasing the juice yield, improving the product quality and reducing the cost and waste volume. The most common

method used is rotating disk module, in which at high rotating speeds, the permeate flux increases with membrane pore size and transmembrane pressure. At low rotating speeds, permeate flux is independent of membrane type and transmembrane pressure due to a thick deposited fouling layer as a dominant filtration resistance. Talking about the carbohydrate transmission, at higher transmembrane pressure it decreases due to the resistance built by the cake layer. The better carbohydrate transmission has been discovered to be at high rotating speed and with a polyvinylidene fluoride membrane of 0.45 μm pore size (Zhu et al. 2013; Luo et al. 2013). A sequential microfiltration and nanofiltration can be used to purify and concentrate prebiotic sugars (fructooligosaccharides) present in artichoke juice. Microfiltration membrane that presents the lowest flux decline, less solid deposition on its surface and almost 100% of prebiotic sugars recovered in the permeate stream is made of polyethersulfone with pore size of 0.05 μm. For the next step, 100% retention of prebiotic sugars is obtained with a polyamide membrane with molecular weight cut off of 150–300 kDa. By this way it has been demonstrated that it can be obtained a functional ingredient that can be added on foodstuff applications (Machado et al. 2016). Membrane filtration has been used in fresh alfalfa juice, for separating and obtaining a soluble protein concentrate by removing 80–90% of the water and non-protein components (Knuckles et al. 1975; Zhang et al. 2016). Also, membrane ultrafiltration is used for purifying and concentrating sugar beet juice (Kawa-Rygielska et al. 2013). For this matrix, the filtration productivity and selectivity depended on the membrane polymer more than on other parameters. With regenerated cellulose membranes the filterability is better but with polyethersulfone membranes the retention of impurities (colorants, proteins and colloids) is improved, leading to a higher juice purity (95–96%) (Zhu and Mhemdi 2016). The use of membrane filtration in this matrix as well as in sugarcane juice can eliminate the usage of chemicals for obtaining **sucrose**. However, some technical problems, such as low permeate flux, high sucrose loss in membrane retentate and serious membrane fouling, are impeding this technological upgrading in sugar industry (Luo et al. 2016). For sugarcane juice the fouling mechanism is the combined cake filtration-complete blocking model (Shi et al. 2019). Because of this, it has been developed an integral membrane filtration consisting of a tubular loose ultrafiltration membrane, spiral-wound tight ultrafiltration and spiral-wound nanofiltration. This system has been reported to reach a colour removal of more than 95% and the recovery of most of sugar, leading a high sucrose recovery of up to 98% (Luo et al. 2016). Also, for sugar cane juice ceramic, membranes of pore size 20 μm have been found to provide the best filterability and retention of impurities (Shi et al. 2019).

17.2.2 Membrane Filtration and Milk

Milk is an opaque whitish nutritious secretion produced by the secretory cells of the mammary glands of mammals. Its main function is to nourish the young until they are able to digest other foods, in addition to protecting their gastrointestinal tract

against pathogens, toxins and inflammation and contributing to their metabolic health by regulating the processes of obtaining energy, especially the metabolism of glucose and insulin. In general, milk contains a lot of components as proteins, peptides, lipids, carbohydrates and salts (Messer and Urashima 2002). Not all mammalian milks have the same properties. As a general rule, it can be said that milk is a slightly viscous, matt white liquid, whose composition and physicochemical characteristics vary significantly according to animal species, and even according to different races. These characteristics also vary in the course of the lactation period, as well as in the course of its treatment. Human is almost the unique mammal that continue drinking milk during all life because provides calcium, vitamins A and D and fatty acids. Milk, depending on the target commercial application, can go through a large number of processes, known as purification processes. These ensure the sanitary quality of the milk, and are listed below. The most common consumed milk for adults are obtained from cow or goat, and both are usually summited to the same elaboration process, that is presented in Fig. 17.4. Membrane separation technology has successfully been incorporated to the dairy industry in multistage, from raw milk concentration to milk microbial stabilization. But for the functional molecules present in milk, membrane separation can be a particularly suitable technology in order to fractionate and concentrate various functional components (Chen et al. 2018).

The surface morphology and internal microstructure of the membrane has an important role in the filtration of milk. In skim milk, internal fouling proceeds by protein-polymer and protein-protein interaction forming a gel layer on the surface of the membrane. This layer is slightly compressible and densifies as it grows. The fouling starts very short after the beginning of the filtration time (James et al. 2003). So, interactions between the milk components and the membrane must be taken into account. For example, it has been discovered that removing lipids does not

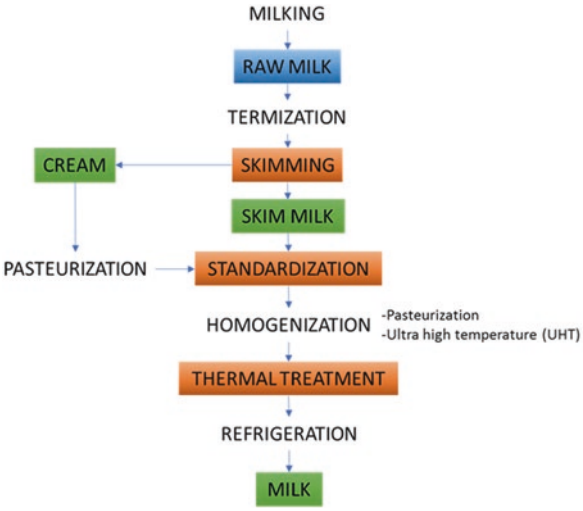

Fig. 17.4 Milk elaboration process

significantly improve the filterability (Batina et al. 1997). In the fouling formed in the membranes, it is possible that gelation caused by acids present naturally in fresh milk occurs, and that remains in the retentates. It can be avoided by diafiltration at 80 °C for 15 min (Li and Corredig 2020). To reduce membrane fouling minimizing the number of cleaning cycles, different methods have been developed. The immersion precipitation technique has been proved for improving polyethersulfone and polyvinylidenefluoride membranes in order to have better results, thereby integrating them in the milk pasteurization step. With this upgrade, milk permeation flux is considerably improved during 100 °C treatment for 20 min with no change in protein rejection and with the highest performance and antifouling properties (Rahimpour et al. 2009). Membrane filtration of pasteurized skim milk by a 0.6 μm pore size membrane has been set up in a dynamic filtration system with overlapping membrane and metal blind disks as a novel procedure, but the retentates have more than 15% of proteins, so it does not work for the dairy industry, but can be useful upgraded for the protein concentrates industry (Schäfer et al. 2018). For minimizing the formation of a casein micelle gel layer in milk membrane filtration, high cross-flow velocity is always used. Uniform transmembrane pressure and graded permeability microfiltration membranes are examples of strategies for fouling control that can be easily implemented at industrial scale (Cheryan and Alvarez 1995). Also, innovative techniques as the use of rotating disks (Engler and Wiesner 2000; Ding et al. 2002), air slugs (Cui and Taha 2003; Cui and Wright 1996) or vibrating modules (Al-Akoum et al. 2002) increase shear force close to the membrane surface to control fouling. Other techniques as electric fields and ultrasonic waves also are being recently implemented in milk membrane filtration (Chen et al. 2018).

Minerals have greater influence in membrane processes because they interact dynamically with the other milk components. At the normal pH of milk, approximately 30% of the calcium, 65% of the magnesium and 45% of the phosphorus are in the soluble form, and there are other minority minerals as Zn, Fe, Cu and Mn. By membrane filtration it has been reported that the ratio of soluble minerals/total divalent cations can be decreased down to 7%. The loss of these minerals during membrane processing is influenced not only by the pore size, also by pH and temperature. With membrane ultrafiltration at normal pH, the total divalent cations and soluble minerals increase in the retentate with loss of micellar calcium, and in consequence the ratio soluble minerals/total divalent cations decrease as predicted before. When the pH is reduced from 6.7 to 5.1 the total divalent cations increase in the permeate and the soluble minerals increase both in permeate and retentate. When the temperature is increased the total divalent cation and soluble minerals content in permeate decrease (Lin et al. 2015).

In the milk elaboration process, it is very important the step of thermal treatment because it achieves a microbial stability. The most commonly used treatments are pasteurization or ultra-high temperature (UHT) for destroying the pathogens and deactivate spoilage microorganisms in order to extend the shelf life of milk and for being stored at ambient temperature (Muir 2011). However, high temperature processes can produce protein denaturation (Anema and Li 2003) or other physicochemical changes in milk, as damage in sensory attributes or the creaming properties

(Fox et al. 2015). The majority of the bacteria present in the raw milk (up to 80%) can be removed by membrane filtration. The somatic cells (Pettipher et al. 1980) and other organism associated with normal and mastitis udders can be removed by membrane microfiltration (Schipper and Dakota 1966). Salmonella and Listeria, the most common pathogenic bacteria in milk, can be removed with a polyethersulfone membrane with pore size of 0.45 μm (Jin et al. 2020; Madec et al. 1992). *Clostridium tyrobutyricum* spores from milk can be eliminated by 0.8 μm porosity membrane (Bourgeois et al. 1984). In goat milk, *Clostridium tyrobutyricum, Clostridium sporogenes, Clostridium bifermentans, Clostridium perfringens* and its spores can be removed with nitrocellulose membrane filter of 0.8 μm pore size. Also with this filter, the butyric acid bacteria in cow milk can be removed (Reindl et al. 2014). *Bacillus cereus* spores also can be removed from milk with membrane filtration. In fact, a heat treatment of the milk at 72 °C during 5 min can improve the filterability of the milk and the retention of psychrotrophic spores (Christiansson et al. 1997; Hoffmann et al. 2006). *Paenibacillus spp.*, and mayoritary *Paenibacillus amylolyticus* and *Paenibacillus odorifer* as the predominant species, are others spore-forming bacteria that adversely affect the quality of dairy products. They can be removed from raw milk with a membrane filter with 0.65 μm pore size (Ohkubo et al. 2019). The smaller the pore diameter (0.8–0.45 μm) the higher the bacterial removal according to several studies. So, it is a fact that pathogens and their spores can be removed from milk by ceramic membranes, extending the shelf life from 12 to 45 days at refrigerated temperatures (Chen et al. 2018). In this approach of membrane filtration also fouling can occur due to the block driven by bacteria and spores in addition to rest of proteins. Traditionally an uniform transmembrane pressure system is used for maintaining a high permeate flux with high cross-flow velocity (Shigematsu 2004). It leads to less compact fouling layers by recirculating the permeate in a concurrent direction (Chen et al. 2018). The most novel approach is to use an inhomogeneous ceramic membranes with a gradient of membrane resistance by varying the porosity of the membrane of the thickness of the selective front layer of the membrane (García and Rodríguez 2015). A difference with the thermal treatment is that membrane filtration must be applied in skim milk with this purpose, because flat globules have larger size that bacteria, and makes membrane fouling more pronounced (Chen et al. 2018).

Membrane filtration technologies are widespread unit operations in the dairy industry, often employed to obtain ingredients with tailored processing functionalities. An example of that are milk protein concentrates powders that are increasingly utilized. The presence of soluble casein improves the rehydration properties of the powders and impacts their thermal stability. But and acidification of the milk previously to the membrane filtration can induce partial dissociation of casein micelles and modify the natural equilibrium of calcium and phosphate between the micelles and the serum phase. When the milk protein concentrate powder is made with acidified skim milk, it exhibits poor thermal stability with lower soluble calcium and phosphate. So, the integrity of the casein micelles and the amount of dissociated, non-sedimentable caseins play a major role in determining the thermal stability of milk protein concentrates powders (Eshpari et al. 2017). The membrane filtration

can be used for fractionating the protein of milk as it has been reported to achieve transmission of whey protein but not of beta-casein, and can be useful for obtaining whey protein-depleted milk without depletion of beta-casein (Holland et al. 2011). Tubular ceramic membranes with a uniform transmembrane pressure system based on applicating a waterproof coating on the external monolith surface has been developed for separating the micelles of casein from skim milk. This system does not yield a higher flux than the conventional tangential flow filtration but it permits to reduce fouling in long term experiments (Springer et al. 2011). However, when the process is at cross flow microfiltration, the ceramic membrane pore size and the filtration temperature influence the protein fractionation of the skim milk. The transmission of proteins increases when increasing the pore size. For instance, 0.20 μm microfiltration pore size let obtain higher concentration of native whey proteins compared with 0.05 and 0.10 μm pore sizes, but a significant amount of caseins can permeate the 0.2 μm membrane resulting in casein distribution similar to skim milk. However, for the production of native whey protein concentrates, it is preferred to start from a permeate free from casein, and the 0.05 and 0.10 μm membranes are able to retain all caseins. When temperature is 50 °C the permeate has more native whey proteins than when the temperature is 60 °C. At 60 °C the native whey protein transmission is reduced due to higher casein deposited on the membrane. So, optimal protein fractionation of skim milk into a casein-rich retentate and a permeate with native whey proteins can be obtained by 0.10 μm MF at 50 °C (Jørgensen et al. 2016). With a traditional crossflow membrane filtration using a molecular weight cut off of 80 kDa and a plate and frame pilot scale system at temperatures below 10 °C, the retentate obtained is nearly free of whey proteins and with approximately 20% of beta-casein removed. When the temperature is 25 °C the skim milk obtained has undergone more than 80% of casein retention. This free-casein skim milk technologically when is reconstituted to the original casein volume fraction, has no statistically significant differences in gelation behaviour (Holland et al. 2011). The fractionation of casein micelles and whey protein beta-lactoglobulin of skim milk by crossflow membrane filtration has been accomplished with 0.1 μm pore size in ceramic monochannel membranes. Membrane length and resistance have been reported to have a crucial role in this process. When low resistance membranes are used, this results in low whey protein permeation due to a deposit layer in the large parts of the membrane, even at lower transmembrane pressure. The increment in the membrane resistance makes decrease the deposit layer and increase the protein transmission at lower permeate flow rate (Piry et al. 2012). On the other hand, the partial or total removal of whey protein from milk has influence on the heat stability of the concentrates obtained. 80 kDa polysulfone membrane retentates show higher thermal stability than 30 kDa cellulose membrane, due to lower retention of whey protein, and not attributable to ionic composition differences or pH, but to the type and number of complexes formed in the serum phase (Renhe and Corredig 2018). However, for obtaining a complete fractionation of caseins and whey proteins from skim milk by membrane microfiltration with a novel method with spiral-wound membranes, several factors must be investigated. The transmembrane pressure is the only significant influence on the length-dependent filtration performance in this

novel system, and washing steps adding a diafiltration medium seems to be needed (Hartinger et al. 2020). In general, for obtaining this integral separation, at lower feed concentration, the concentration of whey proteins in the permeate is restricting, meanwhile, upon higher feed concentration, more severe fouling reduces the flux although the permeate protein concentration is higher. Flux and protein permeation are independent of the casein to whey protein ratio (Hartinger and Kulozik 2020). Bovine skim milk can be modified using membrane filtration with ceramic 0.1 μm membranes followed by polyethersulfone 10 kDa cut-off membranes to produce whey protein with casein profile similar to human milk (McCarthy et al. 2017). From reconstituted skim milk (3.2% protein), serum protein concentrates can be obtained using a tangential flow filtration at 3–4 °C with polyvinylidene-difluoride membranes (0.1 μm and 0.45 μm pore-size), and a polyethersulfone membrane (1000 kDa cut-off). However, there are some differences between them. With the polyethersulfone membrane the starting flux is the highest but decreases later by >40%, although obtaining a permeate composed by beta-casein in >97% of present casein. The same occurs with the polyvinylidene-difluoride membrane of 0.1 μm, meanwhile for the one with 0.45 μm pore size the flux experiments a lower decrease (<20%) and the retentate has higher beta-casein/alfa-casein ratios and minor whey proteins as lactoferrin relative to the other serum proteins (Crowley et al. 2015).

Also, membrane filtration can be used in whole milk for separating the cream and fractionate fat globules at the same time. This two steps are traditionally done by centrifugation and a posterior homogenization for achieving the globule fat size that has a profound impact on the stability and organoleptic characteristics of dairy products (Timmen and Patton 1988). Membrane filtration is able to fractionate flat globules without disrupting the fat globule membranes according to their size. Specifically, ceramic microfiltration membranes with large range of diameter has been developed for fractionating fat globules into two fractions, lower than 2 μm and higher than 2 μm. The posterior milk or derivate products has more finer characteristics (Goudédranche et al. 2000).

In the cheese industry membrane ultrafiltration or microfiltration is used for standardise the milk composition and quality, which is varied by the season, lactation stage, variation in diet or weather. In concrete, low-concentration factor ultrafiltration is now widely practised (Soodam and Guinee 2018). Also, in this industry membrane filtration has been used for recovery of proteins and polar lipids present in milk fat globule membrane from cheese whey because of their technological and nutritional properties. Tangential filtration technique with polyethersulfone and cellulose acetate membrane with 0.15 μm pore size has been implemented. When thermocalcic aggregation whey pre-treatment is applied, the clarification of the whey with both of these membranes results in low permeate fluxes and high retention of ask and whey proteins. When the pH and temperature are increased, polar lipid retention is increased while the fouling formation is minimized (Rombaut et al. 2007). The treatment of the dairy effluents with membrane filtration for recovery all these interesting components has been studied with emphasis in the membrane shear rate, finding that the better results regarding lower membrane fouling is up to 3000 kPa of transmembrane pressure (Frappart et al. 2008).

17.3 Membrane Separation in Alcoholic Beverages

17.3.1 Membrane Filtration and Beer

According to FAO/WHO, beer is a beverage that can be alcoholic or non-alcoholic, made of malty cereals fermented (mainly barley), water and hop. This definition is the most different from the most known international classifications because includes the non-alcoholic beer. More extensively, it is an undistilled alcoholic beverage, with a bitter taste, which is made from sprouted barley grains and other cereals whose starch is fermented by yeast (mainly Saccharomyces cerevisiae) in water, and is often flavoured with hop (RAE: Real Academia Española). Several types are known with a wide range of nuances due to the different ways of elaboration and the ingredients used. It generally has an amber colour with shades ranging from golden yellow to black through reddish browns. It is considered "gaseous" (it contains CO_2 dissolved in saturation that manifest as bubbles at ambient pressure) and is usually topped with a more or less persistent foam. Its appearance may be crystalline or cloudy. Its alcoholic graduation can reach up to about 30% vol, although it is mainly between 3% and 9% vol. It is a beverage rich in phytochemicals as flavonoids, hydroxycinnamates and phenolic acids. These compounds, also present in a variety of foods, are widely reported for their protective effect in chronic disease development (Collins et al. 2010). Specially the female inflorescences of *Humulus lupulus L.* (Cannabaceae), commonly named hop, which confer bitterness, aromas and antiseptic properties to the beer, is composed by terpenes, sesquiterpenes and prenylated phenolic compounds, mainly acylphloroglucinols (bitter acids) from the series of alpha acids. They have shown numerous biological activities as antimicrobial, sedative and estrogenic properties (Bocquet et al. 2018). A study carried out in hypercholesterolemic patients after coronary bypass surgery revealed that a diet supplemented with 330 mL of beer per day decrease the risk factors of coronary atherosclerosis with its markers of process (Gorinstein et al. 2007). Talking about type of beers, the most commonly consumed are lager, ale and stout. Ale has been found to contain the highest level of polyphenols and to possess the highest antioxidant activity, meanwhile Stout was found to be the one which produced a moderate inhibitory effect on cancer cell proliferation (Collins et al. 2010). The antioxidant activity of beer in general has been correlated to its content on flavanols and flavonoids and to a lesser extent with it content in polyphenols (Gorinstein et al. 2007). But in general, all type of beers are summited to the same process of elaboration, that is presented in Fig. 17.5.

Membrane separation processes are interesting alternatives that may be utilised in several steps of beer production and may replace some traditional and time-consuming techniques (Ambrosi et al. 2014). In the beer production the steps of clarification and stabilization are essential for obtaining the final product. Beer clarification is carried out in order to eliminate yeast and colloidal particles responsible for haze, ensuring the biological stability of beer. It is usually performed using standard filtration that consists in the retention of solid particles (yeast, cells,

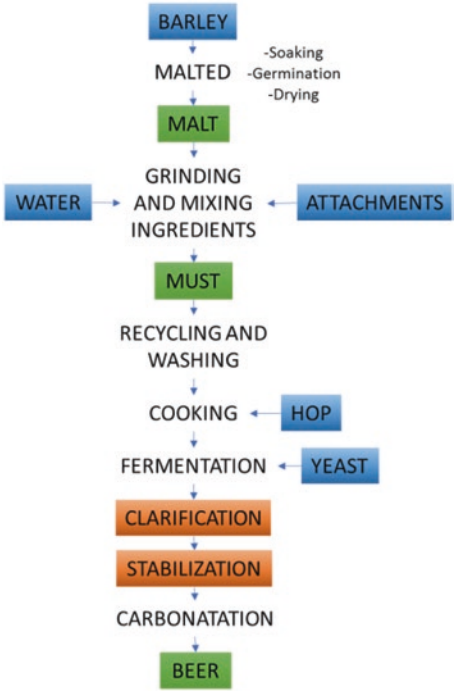

Fig. 17.5 Beer elaboration process

macrocolloids, suspended matter) and solutes responsible for haze, being the most difficult to control. By this way, membrane filtration is an alternative to conventional filtration with filters-aids such as diatomaceous earth. This promising technology must be able to produce a clear and bright beer, to perform separation in a single-step without additives, to operate at low temperature (0 °C), and to achieve economic flux (Fillaudeau and Carrère 2002). A membrane is capable to produce a clear beer in terms of turbidity while solid contents, in particular larger molecular substances, are retained. For selecting the membrane filter for clarification in beer, two criteria must be taken into account, the retention of solids and the permeability. In general, the quality of the resulting beer is determined by the interactions of the technical filtration parameters and its selectivity (Lindemann et al. 1991). During beer filtration with membranes convective and diffusive transport are the two main forms of transport through the membrane. The convective transport attaches to the large molecules, meanwhile the diffusive transport in very important in the elimination of alcohol molecules. The retention of one or other molecules depends on the membrane type, beer flow rate and solute molecule size (Petkovska et al. 1997). A large pore size does not necessarily result in a better permeability because of clogging due to similar pore and particle sizes (Schneider et al. 2005). Tubular ceramic membranes with a pore size of 0.2 μm has been reported to reduce the turbidity in more than 95% but with a significant colour reduction of more than 20% (Alicieo et al. 2008). In general, membranes with a pore size smaller than 0.5 μm can provide

a clear beer with a turbidity below 10 EBC (Schneider et al. 2005). Ceramic hollow fibre membranes with pore size 0.2 μm can achieve a turbidity lower than 1 EBC unit but with reduction in density, viscosity, colour, and foam in different ways. On another hand, 0.8 μm asymmetric membrane modules lead to lower reduction in those parameters with good turbidity in the permeate, meanwhile the ones with 1.4 μm pore size let the obtention of a steady state permeation flux and a minimum loss of permeate quality parameter with the inconvenient of higher turbidity. So this type of membrane with a pore size between 0.45–0.65 μm seems to be the better one (Cimini and Moresi 2016). Comparing among organic membranes for this matrix, cellulose nitrate of 0.45 μm membranes display the highest rejection of suspended particles and proteins, meanwhile 0.45 μm nylon membranes provide the highest rejection of polyphenols and colour compounds. Cellulose acetate membranes with higher pore size (1.2 μm) has higher permeability but in detriment of no adequate separation of proteins and suspended particles. Membranes made of polytetrafluoroethylene with pore size of 0.45 μm are the least selective due to its hydrophobicity producing less interaction with the hydrophilic proteins (Yazdanshenas et al. 2019). A hydrophobic polytetrafluoroethylene-membrane has a higher permeability for wort than a hydrophilic polyamide-membrane because the beer in this step has higher content of hydrophilic compounds, which can bind to the hydrophilic material (Schneider et al. 2005). The selectivity of separation is not dependent only of the membrane's pore size but rather by the formation of the fouling layer affected directly by the beer flow.

Fouling in beer processes by membranes is a fact, and the way in which it occurs depend on the combination of beer composition and membrane characteristics (van der Sman et al. 2012). In general, the detriment in the permeate flux in beer filtration is produced by two fouling mechanisms, an internal pore fouling conforming the standard blocking model, and an external surface fouling conforming the cake filtration model (Blanpain-Avet et al. 1999a). In the clarification step, starch has been found as the major foulant compound forming a layer from the first seconds of processing when using a cellulose nitrate membrane of 0.2 μm. With this pore size ethanol is not rejected (Eagles and Wakeman 1997). When using multi-channel ceramic membranes with a pore diameter of 1.40 μm it has been reported that there are several kinds of fouling resistance in beer: irreversible and reversible external or internal, and that they majority depend on the flux velocity, especially on reversible external resistance. For minimizing those fouling resistances, changes in membrane properties of hydrodynamic methods could be of interest (Fillaudeau and Lalande 1998). Reversible fouling and specifically gel and stationary cake layers contribute more than 95% to the hydraulic resistance, meanwhile the rest is attributed to irreversible fouling where pore fouling produce 0.2% of the hydraulic resistance (Yazdanshenas et al. 2010). In the clarification of the beer the most predominant fouling mechanism for flux decline is the complete blocking of the membrane pores followed by the formation of a cake layer of yeast cells, meanwhile in the posterior stabilization of a clarified beer only the reversible internal fouling occurs. Despite that, ceramic cross-flow membranes can obtain a beer quality similar to the traditional filtration and pasteurization (Kazemi et al. 2013; Cimini and Moresi 2014). It

has been reported that membrane fouling and the initial period of internal fouling is influenced strongly by the initial turbidity of the beer mainly produced by gelatine and tannic acid. Lower turbidity result in lower final total resistance and later internal fouling (Czekaj et al. 2001). Hydrodynamic methods above the membrane surface has been studied in crossflow microfiltration with clarified beer with colloids and macromolecules and a rough beer with yeast cells. Oscillatory flow as hydrodynamic method has been demonstrated to have the capability of decreasing membrane fouling resistance in the rough beer with a yeast cake layer (up to 100%) on the membrane surface in contrast with steady flow, meanwhile having no effect in the membrane clogging of the clarified beer. In both hydrodynamic methods, the transmembrane pressure has been found to have a prejudicial effect (Blanpain-Avet et al. 1999b). The permeate flux can be improved by making some modification on the membrane surface. It has been studied with pure beer yeast suspension through smooth and stamped ceramic membranes, both with pore size of 0.20 μm. Helically stamped surface has been reported to have more advantages than smooth surfaces as higher flux velocity and lower power consumption per volume unit (Stopka et al. 2001). Some other flux enhancement techniques for beer filtration can be considered for increasing the permeation rate. However, it has been reported that using increased bulk-flow turbulence or superimposed helical flow pattern are almost totally ineffective. The introduction of a high frequency backflush program has been reported to achieve a flux increase higher than 400%. It can be attributed to a reduce of the fouling produced by a reversible particle holding and releasing due to the high frequencies (Gan 2001). Using infrasonic high frequency pulses at lower pressure than the transmembrane pressure is possible to remove a portion of the foulant cake. And it has been reported effectively that the permeate flux depend in the pulses frequency (Czekaj et al. 2001). But apart from all those methods to help improve the permeate flux by reducing fouling, the membrane cleaning is also an essential step for maintaining the permeability and selectivity of the membrane for clarifying beer. There is a synergistic relationship between the fouling appeared with the cleaning conditions and the composition of the cleaning agent. It has been formulated and optimized a chemical cleaning method for ceramic microfiltration membranes used in beer industry. It consists on a three step cleaning mechanism that include a simultaneous caustic cleaning and oxidation method which has been reported to restore up to 90% of the original membrane permeability (Gan et al. 1998).

The filterability of beer is influenced by filtration technology as well as beer composition. The loss of large molecules due to the formation of a fouling layer on the membrane and its inner pores as beta-glucans and proteins can have consequences in the beer quality. It can be negative in the case of losing foam enhancing substances (proteins) or bioactive components as beta-glucans. How the filtration is performed (e.g. transmembrane pressure difference) determines the fouling and thus has an impact on both filter resistance and permeate quality (Jin et al. 2004). Using a polysulfone membrane with a molecular weight cut-off of 100 kDa the components responsible of colour, flavour, proteins and bitterness can be kept retained in the membrane during the clarification meanwhile at the same time the

turbidity is removed (de Oliveira and de Barros 2011). With ceramic membranes, beer components that have higher flux decline capacity are the following ones, in impact order: mixture of maltose and sucrose < amylase < pure beer yeast < alpha-bitter acids < catechin (Stopka et al. 2000). When using a 0.2 μm polycarbonate membrane under cross-flow microfiltration it has been demonstrated that the protein retention is closely related to the fouling mechanism. Firstly, with the internal pore blocking the protein retention is lower meanwhile with the posterior fouling layer the retention is very higher. The proteins help form it in association with polyphenols and beta-glucans making a loss of them too (Blanpain-Avet et al. 1999a). It has been reported that light scattering complexes of protein linked to polyphenols cause fouling in a higher measure than each for separated (Czekaj et al. 2001). The influence of fatty acids of beer in general and medium chain fatty acids ethyl esters on crossflow membrane filtration using polyethersulphone membrane has been studied. When antifoam agent is added to the beer, containing high amounts of fatty acids, the filterability decreases by 20% and the pressure rises faster. By this way large and medium chain fatty acids keep retained in the membrane due to their high size, decreasing the filterability and increasing the transmembrane pressure. So, the presence of fatty acids in beer have a negative effect in membrane filtration (Kupetz et al. 2015a). Also, the transmembrane pressure is increased when the beer contains high number of beta-glucans which keeps retained (up to 150 mg/L) in the membrane. For this, the influence of beta-glucan in beer elaboration process has been studied too. When barley enriched with B-glucan in 300 mg/L is used for preparing the beer, the filtrate flux decreases more than 40% during membrane filtration. Meanwhile when the enrichment is carried out with yeast also in 300 mg/L the flux decreases more than 95% during membrane filtration. However, in this context pure medium chain fatty acids ethyl esters of beer have no effect on filterability (Kupetz et al. 2015b). These studies demonstrate a synergistic effect on filterability with polysaccharides and fermentation lipid by-products. And that known β-glucan thresholds regarding filterability must be reconsidered (Kupetz et al. 2017). In general, the presence of arabinoxylan and beta-glucans decrease the filterability of the beer, meanwhile it is not caused by the presence of dextrin. It is mainly attributed to their molecular weight, but the decrease in filterability is in a less important way than the other components of the beer (Sadosky et al. 2002).

Otherwise, after clarification, biological stabilization is necessary to ensure the microbiological stability of the final product during its shelf life. It consists on a heat treatment during a short period of time, call pasteurization. However organoleptic modifications are produced in this process. Thus, membrane filtration also appears interesting to apply instead of this thermal step and allows the elimination of the organoleptic problems. It can produce a microbial free beer without deterioration in beer quality by operating at low temperature (close to 0 °C), thus ensuring beer stability (biological, colloidal, colour, aroma and flavour, foam stability), and enhancing economical flux (Fillaudeau and Carrère 2002). The choice of pore diameter of the membrane is a critical factor. The pore size is going to determine the sterilization and the loss of components of the beer. For crossflow 0.5 μm ceramic microfiltration membrane a bright filtrate is achieved with no loss of components

and near to complete sterility. With higher pore size the sterilization is lost and the filtrate clarity is poorer, although the rates improve (Burrell and Reed 1994). However, the antimicrobial performance of using membrane filtration instead of traditional pasteurization depends on the potential antimicrobial effect of the hop and the presence of beer adapted lactic acid bacteria. When precultures in beer, *Lactobacillus brevis* and *L. lindneri* strains significant decreased their cell size distribution towards shorter rods. These morphological changes are mainly attributed to the hop bitter acids presents in the beer which induce the expression of hop resistance genes in bacteria membranes. In consequence, these bacteria have an increased penetration rate through filters, being dangerous in non-pasteurized beers. In consequence, the selection of adequate test strains are suggested to be important for the rigorous and standardized evaluation of membrane filtration performance (Asano et al. 2007). With nylon-6 nanofibrous membranes it has been achieved a total remove of *Sacharomyces cerevisiae* and wild bacteria as Flavobacterium *johnsoniae* and *Iodobacter fluviatilis* during clarification of beer. In this context, it has been reported that yeast can form cakes with lower resistance than spoilage bacteria, which form cakes with higher density, smaller interstitial space and higher resistance flow (Lemma et al. 2015).

In addition, clarification and stabilization of beer by membrane filtration has been carried out at the time or with other purposes. With the additional purpose to recover the surplus yeasts, microfiltration with ceramic membranes has been described to be a good option if fouling mechanisms are evaded (Stopka et al. 2000). These membranes also have been used with the purpose to recover beer from tank bottoms, with the surprise of obtaining a beer richer in total nitrogen, polyphenols and diacetyl but without influence on beer quality (Bugan et al. 2000). For the selective removal of substances as flavours, spoilage compounds or riboflavina from beer, membranes with incorporated molecularly imprinted polymer particles seem to be a new and very promising tool (Borrelli et al. 2011). Beer as an alcoholic beverage can be dealcoholized by membrane distillation method. It has been reported that during an osmotic membrane distillation of beer 77% of higher alcohols, 99% of esters and 93% of aldehydes volatile compounds are eliminated with loss of bitterness, foam and stability, with post treatments needed for improving the product quality (Liguori et al. 2015). However, those alcohol free beer do not exhibit difference with original beers in terms of colour, gravity and polyphenol content (Liguori et al. 2016). On the other hand, polyamide non-porous membranes has been reported to reach this purpose without losing other nutrients or flavouring components as maltose or glycerol, reducing the alcohol content from 5% vol. to 2.45% vol in 6 h (Purwasasmita et al. 2015). Pervaporation is another membrane technique that let separate volatile organic compounds and that could be useful in beer industry, helping non-alcoholic beer to improve their organoleptic characteristics by enriching them with extract of volatile compounds extracted from other beers (Paz et al. 2017).

Some authors have tried to create an optimal adaptive scheduling and control of beer membrane filtration, attending to its sensibility trying to create a continuous process without stops to minimize the economic costs. An example of this is the model carried out by van Willigenburg and co. (2015), who converted the process to

a simple mathematical problem which was resolved numerically. The model proposed computes and gains insight in the costs and control associated with beer membrane filtration, providing clues for operators to improve manual control that is currently being used. They achieved a money saving of 25% and described that the membrane clogging is the principal problem that can occur. For the best functioning of the membrane filtration and the highest savings, they considered explicitly the opportunity to remove fouling by removing heavily fouled beer which might further improve performance of beer membrane filtration. This has not been considered by the industry previously and is an innovative approach to make the process really continuous. However, nowadays this measure is not implemented, so in a pilot plant or industrial scales it is necessary to use some type of test to determine the filter efficiency and performance of beer during membrane filtration in order to keep the stops of the process at minimum, as occurs in traditional beer filtration (Niemsch and Heinrich 2000). In this context, filter efficiency describes the ratio of filtered beer volume between two filter periods, which are interrupted by an intermediate cleaning step of the membrane. Test most commonly used consist on an usual beer cross flow membrane filtration till blockage. After that, the membrane is rotated and cleaned with distilled water oppositely to the filtration direction. Thereafter the membrane is turned back in the filtration direction and then the previous steps are repeated three times. These cleaning cycles should simulate an intermediate membrane cleaning in industrial scale. With the times of each cycle of beer filtration, using mathematical modelling equations and the help of software, filter efficiencies for the first and second, respectively second/third and third/fourth filtration periods can be determined. Although a lot of tests have been optimized for usual beer filtration (Stewart et al. 2000), only a few have been approached to the membrane filtration. By this way, Kupetz and co. (2018) developed a test that can be used to optimize the process and to investigate the interactions between components and membrane, predicting the filterability. Hajipour and co. (2010) also simulated numerically and verified experimentally the cross flow microfiltration of non-alcoholic beer with tubular membranes, developing a model that can be used to design and optimize non-alcoholic beer clarification.

17.3.2 Membrane Filtration and Wine

Wine is a drink obtained from the grape (*Vitis vinifera* species), through alcoholic fermentation of its must or juice. Fermentation is produced by the metabolic action of yeasts, which transform the natural sugars of the fruit in ethanol and gas in the form of carbon dioxide. The sugar and acids in the fruit, *Vitis vinifera*, are sufficient for the development of fermentation. However, wine is a sum of environmental factors: climate, latitude, altitude, hours of light and temperature, among several others. More concretely, the name "wine" is given only to the liquid resulting from the total or partial alcoholic fermentation of the grape juice, without the addition of any substance. In many laws, only the fermented drink obtained from *Vitis vinifera* is

considered as wine. Knowledge of the particular science of winemaking is called oenology (without considering the vine growing processes) (Robinson 2006). Regular moderate wine consumption is often associated with reduced morbidity and mortality from a variety of chronic diseases in which inflammation is the root cause. It is due to its content in bioactive compounds and the most numerous are resveratrol, hydroxytyrosol and melatonin. Resveratrol and hydroxytyrosol are polyphenols, meanwhile melatonin, recently described in wine, is an indoleamine. Resveratrol seems to be one of the most promising compounds due to its bioactivity, with wine being the main source of resveratrol in diet. Hydroxytyrosol, whose main source in diet is olive oil, has been also found in both red and white wine in considerable amounts. Melatonin has been found in wine in low amounts. However, both high bioactivity and bioavailability have been attributed to it. They show antioxidant, cardioprotective, anticancer, antidiabetic, neuroprotective and antiaging activities (Fernández-Mar et al. 2012).

Without entering in major details, there are mainly four different types of wines, the most known as traditional wines: red, white, rose and sparkling. All of them are summitted to a similar elaboration process described in Fig. 17.6.

Membrane filtration has demonstrated to be a promising process to achieve simultaneous clarification and microbiological stabilization in wines, as well as a useful technique to help eliminate defects. It can be a substitute of conventional processes that imply several filtration steps on diatomaceous earth (Boissier et al. 2008; El Rayess and Mietton-Peuchot 2016). Cross flow filtration membrane systems can bring a lot of advantages in wine production, reducing and fluxing the strain of the wine and increasing its lifetime, when smaller diameter membranes are

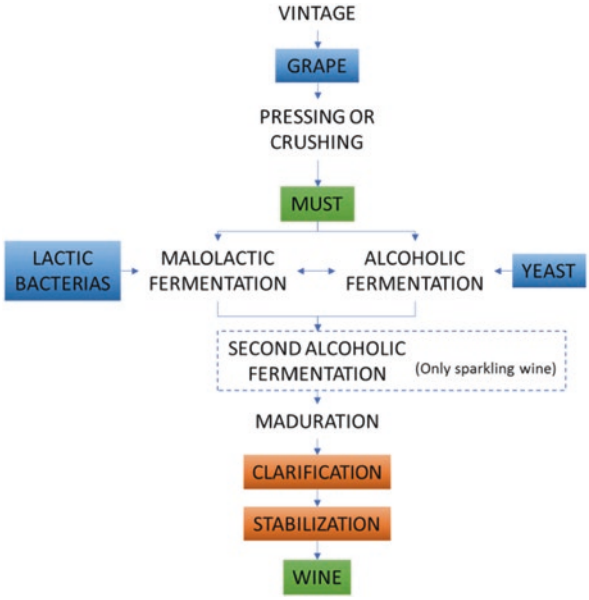

Fig. 17.6 Wine elaboration process

used. When it was evaluated the use of cross flow membranes for clarifying red and white wines it was found that this treatment can replace the pre-treatment with chemical usually done for clarifying wine (Manninger et al. 1998).

In general, membranes made of hydrophilic polymers like polyethersulfone give higher flux and process wines with better taste (Anon 1998). Comparing three types of membranes with the duo material-pore size for wine: cellulose-nitrate microfiltration membrane with pore diameter of 0.2 μm (MF 0.2), a polyethersulfone ultrafiltration membrane (UF 100), and a cellulose-acetate membrane (UF 20), the main differences found between them are the colloidal and phenolic fractions, with consequences on taste and colour. Colloids and phenolics (70–80% of all fraction, including anthocyanins) keep totally retained with the UF 20 membrane, phenolics moderately retained (20%–30% retention) by the UF 100 membrane, and both colloids and phenolics fully permeated through MF 0.2 membrane. Although it is well known that a partial remove of tannins from the red wine is beneficial in terms of taste, the anthocyanins removed by the UF 20 and 100 membranes give an unacceptable loss of red wine colour. Concerning the turbidity reduction, all three membranes offer acceptable results when operating on the slightly cloudy white wine, while the performance of the MF 0.2 membrane is inadequate on highly turbid red wine. However, taking all comparison into account, MF 0.2 membrane is largely superior to the other two membranes because it does not retain anything and is not plugged by colloids, so the optimum pore size could be around 0.1 μm, the lower range of pore diameter of the classical microfiltration processes (Peri et al. 1988). Another study compares polyethersulfone, cellulose mixed esters, cellulose acetate, polypropylene and nylon membranes, and the best one for clarifying wine seems to be the cellulose acetate ones with pore size 0.2–0.45 μm if from the less flux reduction point of view. If only the physic-chemical properties of the wine are taken into account, the best pore size is 0.8 μm (Urkiaga et al. 2002). Hollow fibre membrane system has been reported to achieve a higher turbidity reduction than using a traditional clarification with bentonite clay (95% of reduction vs. 66%) without changes in quality, colour, aroma or flavour (Severo et al. 2007). With a membrane of 500 kDa cut-off it can be achieved a wine with turbidity of 0.11 NTU, more than acceptable for a wine. However, it also carries a loss up to 11% in the protein content, up to 43% of fatty acids and alcohol esters and up to 26% of higher alcohols (Prodanov et al. 2019). Deeper, it must be taken into account the effect of the membrane material (pore size constant of 0.2 μm) combined with the wine composition (crude and previously filtrated) on wine clarification effect. When filtrating a previously filtered wine, the permeability of hydrophilic polyethersulfone membrane is superior to hydrophobic polytetrafluoroethylene membrane, but in both it could be obtained high fluxes under standard operating conditions. With this type of composition, the main problem is that with both membranes irreversible fouling is dominant. For a crude wine, the hydrophilic polyethersulfone membrane has a stronger mechanical effect than the other one, which is reflected by a more complex balance between reversible and irreversible fouling. So, the propensity to form irreversible or reversible fouling is an effect modulated by the interactions and dependence of the membrane and the matrix. More specifically, the factors that affect fouling seem

to be the molecules/membrane interactions in the hydrophobic polytetrafluoroethylene membrane, and the balance between the hydrodynamics of the system and the deposited material for the hydrophilic polyethersulfone membrane (El et al. 2016). In wine there are three main fouling mechanisms: pore blocking, pore constriction and caking, being the last one the dominant one. Usually the cake build-up appears after the pore constriction (Li et al. 2010).

Some authors have studied deeper these fouling mechanisms attending to the components that form it and the components that keep retained on the membrane as a consequence. With a monochannelled tubular alumina cross flow membrane with pore size of 0.2 μm, colloidal material deposited on the surface of the membrane after few minutes. This material was initially characterized as mixtures of high molecular weight moderately acidic grape proteoglycans and low molecular weight acidic moieties (Belleville et al. 1992). With a filter of 0.65 μm for clarifying a Cabernet Sauvignon wine, it has been found a decrease in the concentration of tannins, anthocyanins and total polyphenolic index linked with a loss in colour intensity index, also affecting the wine in a sensorial and not only a chemical way, caused by the adsorption in the membrane filter in the initial stages of filtration (Arriagada-Carrazana et al. 2005). In general, polysaccharides, long chain proteins and some phenolics seems to be the mains responsible for fouling (Li et al. 2010). Proteins are also responsible for wine instability resulting in haze formation, meanwhile glycoproteins prevent this protein aggregation and precipitation, so the first one must be eliminated but not the second one (de Bruijn et al. 2011). Polysaccharides and polyphenols from wine have been reported to induce the loss of membrane permeability from the first minutes of the process operation. In this context, fouling is caused by physical-chemical interactions between the membrane and wine constituents and by wine constituents with wine constituents, all promoted by hydrodynamic conditions. In general, aggregates of polyphenols and polysaccharides have the higher influence to the adsorptive fouling. While the impact of polyphenols is largely dependent of the membrane properties, polysaccharides play a primary role in flux decline in a relative independent way from the membrane characteristics (Vernhet and Moutounet 2002). With hydrophilic organic microfiltration membranes, it has been reported that mannoproteins are the main wine polysaccharide that make permeate flux decrease by promoting fouling. Pectic polysaccharides present in wine have been highlighted to develop a protective effect against this flux detriment induced by mannoproteins. So, flux decline due to polysaccharides does not depends on the total amount of them but to the respective amount of each polysaccharide (Vernhet et al. 1999). Comparing between polypropylene and polyethersulfone membranes with the same pore size (0.2 μm), the first one show only marginal adsorption of polyphenols and polysaccharides meanwhile the other one shows stronger adsorption (Ulbricht et al. 2009). Ranking the colloids components in wine on the basis of their effect in fouling, the first one is pectin followed by tannins and mannoproteins. Pectin causes the formation of gel layer and tannins a cake layer (El Rayess et al. 2012).

In order to prevent and reduce this fouling, different methods could be applied such as back- flushing, backshocking or infrasonic pulsing (Urkiaga et al. 2002).

The impact in the formation of these deposited structures from wine fines (lactic bacteria and colloidal aggregates) and yeasts through membrane cross-flow microfiltration has been studied as well. Yeast cells always form reversible deposits that can be avoided by maintaining a low flux, meanwhile fines form a coherent and adherent cake, an irreversible deposit. By this way, when there are yeast and fines, the transmembrane pressure has a great influence in the composition, final resistance and reversibility of the deposit. So, an abrupt rise of the transmembrane pressure allows the formation of a mixed deposit which extent likely be dependent on the yeast to fine ratio, and in which the presence of yeast favoured the deposit reversibility (Boissier et al. 2008). Setting membrane type and pore size, also the critical operating conditions can be determined for limiting fouling only caused by wine fines not only for the continuity of the process, but also for not losing some of these valuable compounds from wine. However, it cannot be avoided the fact that membrane fouling in presence of colloids as tannins, pectin and mannoproteins occurs from the first minute of filtration. The mechanism of fouling is slightly different for each compound: for mannoproteins the phenomenon is an adsorption on the membrane material with formation of a deposit layer, and for pectin a gel layer compaction or deformation under high pressures. So, the only possible solution compatible at industrial level is determine a "threshold flux" in which a certain degree of fouling is let and acceptable (Stoller and Ochando-Pulido 2015; Stoller et al. 2017; El et al. 2011). Other options are the use of specific enzymes in order to hydrolyse pectin chains or the use of bentonite for finning the wine by precipitating unstable tannins (El Rayess et al. 2012).

Respect to microorganism growth, some authors report that a membrane porosity of 0.45 μm is the better for removing yeast and bacteria (Castro-Muñoz et al. 2018), but others suggest that a porosity of 1 μm or more is enough for removing yeast (Mondor et al. 2000), meanwhile 0.30 μm of porosity is needed for removing bacteria (Renouf et al. 2007). In contrast with the general opinion of the smaller the pore size, the more microbes are eliminated, winemakers seem to try to adopt filtration membranes with porosities as large as possible to sustain adequate flow rates and keep intact the aromatic, flavour and phenolic profile. One of the principal microorganisms' enemy of wine is *Brettanomyces*, a yeast that induces in wine an unpleasant smell of horse stables, leather, sweat, or even urine. It has the ability of survive and grow in wine bottles, so several authors have studied in the last years the most effective method to remove it before bottling without losing volatile phenols. Also, while sulphites from wine help limit growth of the spoilage yeast, SO_2 has been reported to decrease cell size of Brettanomyces, potentially decreasing the porosities of filtration membranes required for removal (Millet and Lonvaud-Funel 2000; Agnolucci et al. 2010). So, for removing this yeast membrane filters with porosities $\leq$0.8 μm are needed, and when the membranes used have large porosity, additional SO_2 must be added to curtail potential spoilage by cell Brettanomyces that pass through the filtration medium (Umiker et al. 2013).

In addition, membrane filtration has been incorporated to wine process elaboration for other purposes than those described before. When there is copper naturally present or added to wine in order to eliminate the hydrogen sulphide, it reacts with

the sulphites and form complexes around or below 0.2 μm of size that must be eliminated from wine. Membrane filtration is an option for this purpose. Depending on the type of membrane, the filterability can be better or worse. Polyethersulfone and nylon membranes remove up to 40–90% of sulphide-bound copper, meanwhile cellulose, Teflon and glass fibre membranes only remove minimal sulphide-bound copper. These complexes are removed by adsorption in the membrane rather than by particle size discrimination and the presence of compounds as polysaccharides and proteins inhibit part of this adsorption so its content must be limited in this case for not losing them and reach eliminate the sulphide-bound copper (Kontoudakis et al. 2018).

The maximum permitted dealcoholisation of wine by the laws of the European Union is 2%, although in some cases higher dealcoholisation levels are necessary. This restriction is due to avoid decreasing the organoleptic quality of wine. However, there are few studies about the dealcoholisation of wine by membrane technology in which the European authorities can be based on (Lisanti et al. 2013). The dealcoholisation of wine can be carried out by nanofiltration membranes, but it must be taken in consideration that the rejection of the main compounds as acids, sugars and tannins could be very high. With flat sheet polymeric membranes, it can be achieved a wine with 5% (v/v) alcohol removed with less than 15% loss of each solute (Labanda et al. 2009). However, the major differences from the 5% alcohol removed to the non-dealcoholized ones are the higher astringency and the loss of olfactory compounds (Lisanti et al. 2013). Polypropylene hollow fibre membranes have been reported for being used to partial dealcoholize wine, reaching a partial dealcoholisation of 2% (v/v) with acceptable aroma losses without sensorial perception of the quality of the product (Diban et al. 2008). The loss of aroma compounds is reduced below 20% (usual losses range from 30% to 50%) (Diban et al. 2013). With this type of membranes only a loss of monomeric anthocyanins occurs due to the adsorption on the membrane surface and to the oxidation of wine when in contact with air during the treatment. Although this loss does not affect the colour parameter of wine, it can modify the astringency properties, so the dealcoholisation must be carried out under limiting conditions of oxygen (Gambuti et al. 2011). Polyamide membranes has been reported to reach a wine dealcoholisation of 7% (v/v) of the present alcohol. However, for maintaining the organoleptic properties, the addition of an aromatic extract after the process is necessary (Catarino and Mendes 2011). Other study reported that a wine dealcoholisation by membrane processes of 7.8% (v/v) is sensorially acceptable without significant changes in colour, taste, flavonoids and phenolic compounds (Corona et al. 2019). A slight wine alcohol reduction also has been achieved by reducing the sugar content in musts by membrane nanofiltration, although with loss of colour and aroma compounds (García-Martín et al. 2010; Mihnea et al. 2012). A two-stage nanofiltration for sugar reduction and dealcoholisation has been reported to achieve a white wine with a very similar content to the original must (Salgado et al. 2017).

Anion-exchange membranes have been reported to be used in wines for a reagent-free pH correction and tartrate stabilization. However, polyphenols and

polysaccharides are the main affected components of the wine by these membranes, because the treatment among 10 h result in the fouling layer formation and electro-convection reduction (Sarapulova et al. 2018).The deposition of these compounds on the surface increase the hydrophobicity of the membrane and their accumulation into the polymer decrease the ion-exchange capacity and the electrical conductivity (Bdiri et al. 2020).

Microfiltration with polymeric membranes has also been applied for producing bioactive extracts from winemaking wastes. However, as the most important parameters for this purpose are the membrane type and pore size, it has not been found the perfect membrane which shows preferential rejection of phenolic compounds in general over sugars in order to separate them (Arboleda Meija et al. 2019). In red wine lees, polyvinylindenefluoride hollow fibre membranes have been applied getting suspended solids retained while bioactive compounds (polyphenols, anthocyanins and resveratrol) are in the permeate. After that an extract of anthocyanins (higher than 93%) can be achieved with flat sheet nanofiltration membranes (Cassano et al. 2019).

17.4 Conclusions

As summarized in this chapter, membrane separation technology is a green and sustainable tool that could be applied in food industries for different purposes. The possibility to configure the membrane equipment using different molecular weights cut-off (MWCO) gives the possibility to use this technology to enrich the foods in targeted compounds as nutrients or bioactive compounds. At the same times, it is possible to use the membrane filtration to enhance the shelf-life of products thanks to the decrease of bacterial content. However, beside that its use in food industry is consolidated, further investigations will be needed in order to improve the processes. In fact, the new materials used for the production of membranes and the possibility to produce selective/functionalized membranes, will open new frontiers in terms of the application of membrane filtration in food industries.

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Chapter 18
Effect of Ozonation and Plasma Processing on Food Bioactives

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Abbreviations

CAPP	Cold atmospheric pressure plasma
CP	Cold plasma
DBD	Dielectric barrier discharge
O_3	Ozone
OH	Ohmic heating
RNS	Reactive nitrogen species
ROS	Reactive oxygen species
TPC	Total phenolic content

18.1 Introduction

Thermal and nonthermal processing technologies are the main physical disinfection treatments are used in the food industries (Bovi et al. 2019). The classification has been done, depending on whether the heat is applied or not. Among thermal treatments, ohmic heating (OH), microwave, radiofrequency, infrared, and inductive heating are common ones (Alves Filho et al. 2020). Conventional thermal

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processing also includes, cooling, freezing, and drying (Chizoba Ekezie et al. 2017). Moreover pasteurization (Hou et al. 2019), and sterilization (Alves Filho et al. 2020), belong to the traditional thermal processing, that are widely used for extending the shelf life of food, although these techniques may affect the quality and safety of the products. Furthermore, certain food components are thermally sensitive, and heating may cause losses in nutritional, physicochemical, rheological, and sensory characteristics (Soares et al. 2017) as thermal treatment decreases the bioavailability of some nutrients (Petruzzi et al. 2017). It is known that consumers are looking for safe and high-quality food products (Alves Filho et al. 2020). Although thermal processing is still the major food processing technique (Pankaj et al. 2018), and the most cost-effective tool to ensure microbial safety and enzyme deactivation (Petruzzi et al. 2017), the distinct nature of the food industry requires the adoption of new technologies in order to ensure, safety assurance, shelf-life extension and sustainability (Misra et al. 2015a).

Non-thermal technologies such as ultraviolet (UV) light, pulsed electric fields (PEF), radiofrequency (RF) electric fields, ultrasound, supercritical carbon dioxide, pulsed X-rays, membrane technology, ozone, plasma and high hydrostatic pressure (HPP), are technologies where heat is not applied (Almeida et al. 2015), although HPP can be also combined with heat. All these technologies represent a promising change in the scenario of food processing and they have contributed to shelf life extension of foods and better preservation of nutrients and sensory properties compared to thermal methods (Porto et al. 2020).

Bioactive compounds are phytochemicals found in food that are able to regulate metabolic processes, thus promoting better health. These compounds are mainly found in plant foods such as fruit, vegetables, and whole grains and typically occur in small amounts. Antioxidant action, inhibition or induction of enzymes, inhibition of receptor activities, and induction and inhibition of gene expression are some of the basic health properties. Although bioactive compounds constitute a class of compounds, they differ in their chemical structures having a hydrophilic or lipophilic structure, in the distribution in nature in the range of concentrations in both food and in the human body, in the possible site of action, in the effectiveness against oxidizing species, in the specificity and in biological action. Among them, polyphenolic compounds, carotenoids, tocopherols, phytosterols and organic sulfates are important groups in the human diet (Carbonell-Capella et al. 2014).

Plasma processing is a promising, novel, non-thermal technology which is efficiently applied in food industry with diverse forms for utilization. Microbial decontamination of food products, including sporulating and spoilage/pathogenic organisms, packaging material processing, and functionality modification of food materials are the main goals of plasma application (Chizoba Ekezie et al. 2017). In addition, the preservation of the above mentioned valuable compounds in the highest possible extent consist one of the major target of plasma processing. The effect of plasma processing in food quality parameters is well-documented (Hou et al. 2019). Ozone treatment is increasingly used as an alternative modern method of inducing stress and increasing the health-promoting potential of plant materials. Ozone can increase the plant's resistance to stress by stimulating the system of

neutralizing reactive oxygen species in cells or increasing the synthesis of antioxidant enzymes. The plant defense response depends on the interrelationship between many complex signaling pathways and metabolic signals (Ludwikow and Sadowski 2008). The ozone sensitivity of fruit and vegetables varies depending on the genus, species and cultivar (Horvitz and Cantalejo 2014; Segade et al. 2019). This effect also depends on the form and method of ozonation and the ozone dose used. Higher concentrations and longer contact times can cause oxidation of biologically active compounds and increased oxidative stress which degrades the quality of the plant raw materials (Sachadyn-Król and Agriopoulou 2020). In the present chapter, recent developments for plasma and ozone techniques effects in main food bioactives are summarized.

18.2 Basic Food Bioactives

Various bioactive compounds are extracted from natural sources, such as fruits, vegetables, legumes, oils, nuts, whole grains cereals and food processing residues with many beneficial results on human health. Bioactive foods are foods that contain at least one bioactive compound that has a positive effect on its consumption by providing improved health (Fernandes et al. 2019; Recharla et al. 2017). In addition, foods of plant origin, including fruits and vegetables due to their high concentration of bioactive ingredients (phenolic compounds, carotenoids, tocopherols, vitamin C, vitamin E, dietary fibers, essential minerals, fatty acids, and others), are of great interest for the development of functional food as the health benefits are enormous (Carbonell-Capella et al. 2014; Kongkachuichai et al. 2015; Agriopoulou et al. 2020a). Bioactive compounds have anti-inflammatory, antioxidant, antiviral, antibacterial, antithrombotic, anti-cancer, anti-allergic, anti-diabetic anti-tumor, anti-obesity properties and immunomodulatory activities (Tran et al. 2020).

Increased global demand for nutritious and functional foods has shifted the food industry's interest in the search for bioactive ingredients for future industrial applications by developing new value-added and novel food products (Trigo et al. 2020). Recent studies have demonstrated the value of valorization of fruit and vegetable wastes in order to recover valuable bioactive ingredients (Campos et al. 2020; Hussain et al. 2020).

Preliminary studies conducted by Gómez-García et al. (2021) demonstrated that several waste by-products, such as melon peels and seeds, could be used by the food industry to produce functional foods for a variety of applications, such as food fortification, enhancers, food preservatives, colorants, dyes, due to their richness in bioactive compounds, in particular polyphenols (flavonoids and phenolic acids), carotenoids (α, β-carotene and β-cryptoxanthin) and fatty acids (oleic, linoleic and palmitoleic acids), among other compounds. In particular, using natural phenomena (polarity, solubility, density, pH or selectivity and affinity) with no use of solvents, for the extraction of bioactive compounds, melon by-products showed significant levels of value-added compounds, 15 polyphenols (mainly hydroxycarbonate and

hydroxybenzoic acid and flavonoids) and four carotenoids (especially β-carotene and β-cryptoxanthin) which, in turn, are responsible for a variety of biological activities (antimicrobial, provitamin A, antioxidant activity, among others) promoting health. The basic food bioactive compounds are presented in Fig. 18.1.

18.2.1 Carotenoids

Carotenoids are natural pigments known as provitamin A, also called tetraterpenoids, found in fruits and vegetables, and consisting a group of bioactive compounds with antioxidant properties that help reduce many degenerative diseases (Langi et al. 2018; Augusta et al. 2019). Depending on their function, they can be classified into two groups: carotenes, such as α-carotene, β-carotene and lycopene, as well as xanthophylls, including lutein and zeaxanthin (Langi et al. 2018). Lutein and zeaxanthin, bioactive substances of carotenoids, are present in high concentrations in egg yolks (Langi et al. 2018) and protect against eye diseases. Their release from food and their absorption by the human body ranges from 5 to 30% due to their low oral bioavailability, making it necessary for the design of functional foods/ingredients (Augusta et al. 2019).

Edible flowers can be eaten fresh (salads), dried or canned in sugar, as well as incorporated into cakes, snacks, as their consumption is associated with multiple

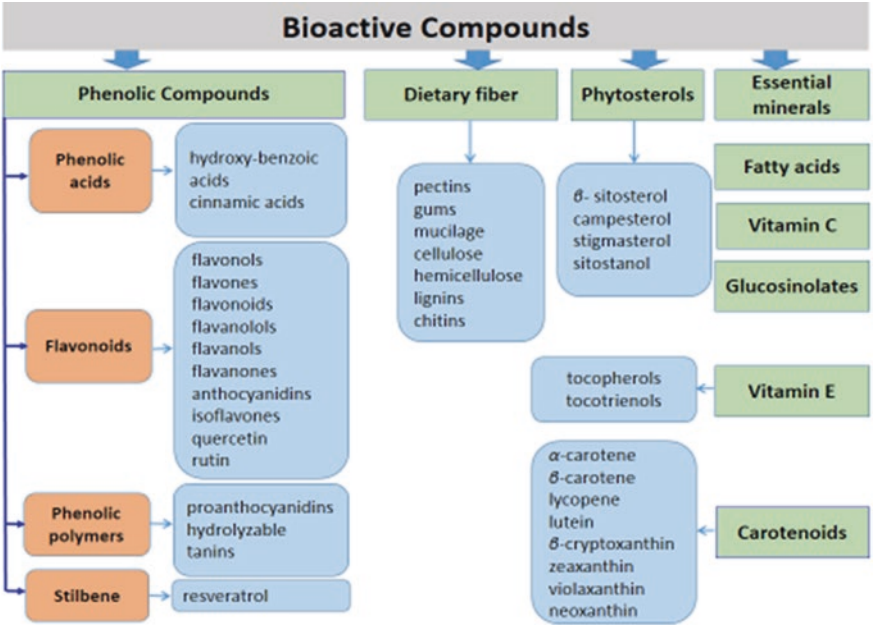

Fig. 18.1 The basic food bioactive compounds

health benefits (antibacterial, anticancer, anticoagulant, diuretic properties). According to Pires et al. (2021) the combination of *Vaccinium myrtillus* L. with rose petals and calendula, dehydrated apples and goji berries has led to the creation of new bioactive food products that provide healthy choices to consumers. The encapsulation of materials in enhancing the stability of bioactive compounds is a widely used technique. In particular, according to Nurhadi et al. (2020) the red ginger extract powder produced using encapsulant materials maltodextrin: gum arabic in a ratio of 5:5 resulted in the best stability of the bioactive compounds during storage (Nurhadi et al. 2020).

To extend the life of the product, encapsulation is a promising technique in which bioactive compounds are released protecting the active ingredients from unwanted agents, such as light, heat, water, enzymes, pH, which can adversely affect its stability. In addition, with this technique, the organoleptic characteristics of the product are improved by releasing characteristic ingredients, without changing their taste, aroma and texture (Coelho et al. 2021). In addition, the exploitation of fruit and cereal processing can create new products with added biological value. The addition of edible petals, calendula and rose, to bilberry snacks improved the content of organic acids and tocopherols. Also, in the same research, the addition of apple and goji fruits resulted in the highest content of phenolic acids and anthocyanins contributing to the development of healthy snacks (Pires et al. 2021). Experimental studies have proved the direct amplification of processed foods (cereal bakery products) with various minerals using polymeric and non-polymeric coating materials for micro/nanoencapsulation of the main target minerals, resulting in the production of enriched foods with extended shelf life and reduced losses of nutritional and aesthetic quality (Gharibzahedi and Jafari 2017). Bioactive compounds are also found in traditional edible seaweeds. Recent research suggests that brown seaweeds provide a rich and sustainable source of macro- and micronutrients in the human diet, therefore they are a valuable source of bioactive compounds. They are used as food in Asian countries such as China, Japan and Korea where about one-fifth of meals contain seaweeds (Ristivojevi et al. 2021).

18.2.2 Polyphenolic Compounds

Phenolic compounds or polyphenols consist of an aromatic ring and a benzene ring associated with at least one hydroxyl group, including phenolic acids (hydroxybenzoic acids and hydroxy-trans-cinnamic acids), coumarins, flavonoids (flavones, flavonols, flavanones, flavanols, and anthocyanidins) isoflavonoids, lignans, stilbenes, and phenolic polymers (proanthocyanidins and hydrolyzable tannins) (Carbonell-Capella et al. 2014; Varzakas et al. 2016). Flavanols (eg catechins, proanthocyanidins) are abundant in grapes, with a small portion being transported to the must during winemaking. Significant amounts of phenolic compounds remain in the by-products of wine, making them bioactive products. A great source of

proanthocyanidins is grape seed extract with protection against peroxidative damage and the ability to neutralize free radicals (Varzakas et al. 2016).

Polyphenols are secondary metabolites with beneficial effects on health. They are found in abundance in a variety of foods such as cereals, soy, oats, red fruits such as strawberries, grapes and plums, seeds, fruits, vegetables, herbs, and beverages such as wine, coffee, tea, and cocoa. Their anti-cancer, antioxidant, antimicrobial, anti-inflammatory properties make them of utmost importance for human nutrition. However, their bioavailability is affected by many physicochemical factors such as the type of bioactive compounds, their polarity, molecular mass, solid state (crystalline versus amorphous) plant matrix, solid state (crystalline versus amorphous) and digestion by gastrointestinal enzymes and absorption in intestinal (Hosseini and Jafari 2020; Ozdal et al. 2016; Agriopoulou and Stamatelopoulou 2017).

Quercetin is a natural polyphenolic compound belonging to the group of flavonoids and has a number of bioactive properties benefits for humans (antioxidant, anticancer and antiviral properties) (Li et al. 2019). It is a hydrophobic compound with bitter-tasting, low stability and low solubility in water, and low bioavailability (Hosseini and Jafari 2020; Li et al. 2019; Wang et al. 2021). It is mainly found in tomatoes, lettuce, onions, strawberries, grapes and apples (Hosseini and Jafari 2020; Wang et al. 2021). Carvacrol is a phenolic monoterpene, with a wide range of biological effects, such as antimicrobial, insecticide, antioxidant, anti-angiogenic and anticancer, is mainly found in oregano, thyme, and peppermint. It has low water solubility, volatility, low stability, and it is not immediately bioavailable because it is excreted in the urine (Shinde et al. 2020). Table 18.1 shows some recent studies with bioactive compounds, their occurrence in food, their determination techniques and their health-promoting properties.

18.3 Ozone Technology

18.3.1 Basic Principles of Ozone Technology

O_3 (ozone) is a three atomic form of oxygen. Solid ozone has a black-violet color, liquid ozone has a dark blue color, while the gaseous form is blue in color and has a pungent smell (Laszlo and Jeno 2016). It is highly unstable in all states, highly explosive in both solid and liquid forms, and decomposes quickly in gaseous form. Ozone has a short half-life in aqueous solutions; therefore, it should be used immediately after production. The half-life ranges from a few seconds to several hours; this difference is due to many factors. Ozone is easily detectable even at very low concentrations (0.01–0.05 ppm). It is heavier than air; in the gaseous form, it is partially soluble in water, and the solubility increases with a decrease in temperature (Coelho et al. 2015).

To obtain ozone, a large amount of energy must be provided to split the molecular oxygen into a pair of free radicals, which react with the available oxygen to

Table 18.1 Bioactive compounds, their occurrence in food, their determination techniques and their health-promoting properties

Bioactive compounds	Food origin	Determination techniques	Health-promoting properties	References
Polyphenols (phlorotannins and bromophenols), carotenoids (fucoxanthin and astaxanthin), and n-3 long-chain polyunsaturated fatty acids	Brown seaweeds	High-performance thin-layer chromatography (HP-TLC)	Antibacterial, anticoagulant, antiviral, antitumor, anti-hyperlipidemic, antitoxic, immunoregulatory, hepatoprotective, antiaging, and antioxidant effects and reduced risk of hypertensions and cardiac heart disease	Ristivojevi et al. (2021)
Non-acylated anthocyanins and acylated anthocyanins	Purple potato flour	Ultra-high performance liquid chromatography with quadrupole time of flight mass spectrometry (UPLC-Q/TOF-MS)	Anti-cancer, aging delay, enhancing immunity and vision, purifying the cardiovascular and cerebrovascular	Gong et al. (2021)
Picrocrocin, safranal and crocin	Spice saffron	Measuring absorbance at 257, 330 and 440 nm, respectively	Anti-cancer effects	Rajabi et al. (2015), Tarantilis et al. (1994)
Trans-resveratrol	Grape skin powder	High-performance liquid chromatography (HPLC)	Against neurodegenerative diseases such as Alzheimer's, Parkinson's and Huntington's diseases	Rai et al. (2021)
Tocopherols and anthocyanins	Bilberry fruit-based snacks	HPLC	Antioxidant, antibacterial and hepatotoxic properties	Pires et al. (2021)
Phytosterols, polyphenols, carotenoids, flavonoids and dietary fiber	Date palm	NA[a]	Treatment and prevention of various diseases like diabetes, cancer, hypertension, immune deficiency	Maqsood et al. (2020)

(continued)

Table 18.1 (continued)

Bioactive compounds	Food origin	Determination techniques	Health-promoting properties	References
Gallic acid, chlorogenic acid, epicatechin, rutin, caffeic acid p-coumaric acid, ferulic acid, quercetin, cinnamic acid	Dried jujube	HPLC	Antioxidant, anti-inflammatory and antineoplastic properties and liver protective function	Li et al. (2021)
Pectin and κ-carrageenan	Extruded rice starch	A miniature twin-screw extruder	Antioxidant, anti-inflammatory, anti-mutagenic and anti-carcinogenic activities	Maqsood et al. (2020), He et al. (2021)
Phenolic compounds	Pigeon pea	Ultra-high performance liquid chromatography coupled with triple quadrupole mass spectrometry (UPLC- QqQ-MS/MS)	Against numerous human diseases, respiratory infections, reproductive system infections, dysentery, diabetes, hepatitis, measles, jaundice, ulcers, sores, and menstrual disorders	Gai et al. (2021)
Phenolic compounds	Flours obtained from grape pomace	HPLC	Antitumor, anti-aging, antimicrobial, and anti-inflammatory effects	Monteiro et al. (2021)
Polyphenols, inulin, oligofructose and sesquiterpene lactones	Chicory	NA[a]	Hepatoprotective, anti-inflammatory, antioxidant, sedative, immunological, cardiovascular, hypolipidemic, antidiabetic, anticancer, gastroprotective, antimicrobial	Perović et al. (2021)
Carvacrol	Oregano, thyme, peppermint	HPLC	Antimicrobial, antioxidant, anti-angiogenic and anticancer potential	Shinde et al. (2020)

[a]*NA* Not available in the publication

produce O_3. In the natural environment, this substance is formed by the action of ultraviolet rays from the sun on oxygen molecules (Kim et al. 2003). Under controlled conditions, depending on the desired concentration, ozone can be obtained by photochemical, electrolytic, or silent discharge methods (Alias et al. 2012). For economic and technical reasons, the last of the abovementioned methods is frequently used, which gives the highest concentration of ozone. O_3 is a strong oxidant

with high reactivity and a high oxidation-reduction potential of 2.07 V. It can inactivate viruses, bacteria, fungi and mycotoxins more effectively than chlorine or chlorine dioxide (Sahab et al. 2013; Agriopoulou et al. 2016). The microbial destruction mechanism involves the oxidation of sulfhydryl groups, amino acids, peptides, and proteins to shorter peptides by ozone as well as the oxidation of polyunsaturated fatty acids to acid peroxides. This results in cell disruption and leakage of cellular content, and the oxidation of intracellular proteins causes cell death (Nath et al. 2014). Ozone effectively destroys gram-positive and gram-negative bacteria. The presence of ozone also inhibits the formation of mold and destroys spore forms of bacteria (e.g., *Bacillus* and *Clostridiu*m) (Ziarno and Zaręba 2015). Hence, ozone is used as a disinfectant for disinfection of equipment or surfaces that come into contact with food. Gaseous ozone reduces the number of microorganisms by approximately 7.5–2.5 log cycles (Piechowiak et al. 2019). The first report on ozone appeared in 1840, but only after several dozen years, it was used for the first time to disinfect drinking water in 1906 and as a disinfectant for packaging meat products in 1910. In 1982, the Environmental Protection Agency (EPA) conducted tests to confirm the safe disinfection effect of ozone, because of which the gas received the Generally Recognized As Safe (GRAS) status. In 2001, the FDA (Food and Drug Administration) approved liquid and gaseous ozone as a harmless substance suitable for direct contact with food.

Ozonation is the process of purifying products with ozone. It is used in all industries for cleaning equipment or packaging as well as for shelf-life extension of food. There are two main types of ozonation: water and gas phase. The method of water ozonation consists of producing an aqueous solution of ozone with a specific concentration. This solution is obtained by introducing the gas generated by the ozonizer into the water. As ozone with a high oxidation-reduction potential is soluble in water, this solution can be used as a disinfectant. The main advantage of this method is that at ambient temperature, this water has a similar activity to hot water. Another form is gas ozonation, i.e., direct exposure of products to ozone produced by the generator. The raw materials are placed in a modified atmosphere enriched with ozone. Usually, the products are periodically cooled with a stream of ozone of a certain concentration (Balawejder and Piechowiak 2019). This method requires the use of devices that intensify the mass exchange between the disinfected product and the stream of produced gas. Because of the high toxicity of ozone, it is necessary to use closed apparatus during gas ozonation, which makes it more difficult than ozonation in water. The effectiveness of ozonation depends on many factors, among which most important ones are: pH value, relative humidity, processing temperature, and the amount of chemicals consuming ozone (Bechlin et al. 2020). These factors can primarily affect the solubility, stability, and reactivity of ozone. The pH value mainly affects its stability, which decreases as the alkaline nature of the environment increases. Furthermore, with the increase in the relative humidity of the environment, an improvement in the antibacterial effect of ozone is observed. Increasing the temperature of the process worsens the stability and solubility of ozone, but improves its reactivity. The presence of ozone-consuming substances mainly reduces its reactivity by causing various chemical reactions (Laszlo and

Jeno 2016). To obtain the best results during ozonation, all these factors should be carefully controlled. Ozone is most commonly used in two ways which can have very different results: long-term storage in an atmosphere containing a low concentration of ozone or a single high-dose ozone treatment.

18.3.2 Ozone—A Tool for Enriching Food Bioactive Composition?

During storage, the fruit is exposed to the loss of large amounts of biologically active compounds, mainly polyphenols and antioxidants. It is hypothesized that ozone may be an abiotic factor that induces defense mechanisms in plants. The result of these interactions may be an increase in the content of secondary metabolites and improvement of the associated health-promoting properties of plant products. In products of plant origin, ozonation is performed in two possible ways: preharvest and postharvest treatment. Preharvest treatment with ozone gas is expensive, while postharvest treatment requires proper selection of ozone form, concentration, and time of exposure depending not only on the type and quantity of the food product but also on the initial quality of each particular commodity. The review of Sachadyn-Król and Agriopoulou (2020) shows that in general, fruits are more sensitive to the oxidizing effects of ozone than vegetables and that ozonated water is very mildly reactive in comparison with ozone gas.

Ozone can affect compounds contained in plants in two ways: by direct reaction with them or by reaction with radicals formed during its decomposition (Khadre et al. 2001). Ozone molecules can enter plants through open stomata and then react with the elements of the aqueous apoplast in the form of ROS (reactive oxygen species)–mainly hydroxyl, hydroperoxyl, and superoxide radicals (Karaca and Velioglu 2014). As a result of the interaction with ozone, the components of the calcium and ion channels of the plasma membrane are damaged. This destruction almost immediately leads to the production of ROS and some enzymes, including peroxidase (POD) and superoxide dismutase (SOD) (Oksanen et al. 2004). This condition is called oxidative stress and can cause many physiological changes in plants. The use of ozone improves the resistance of plants to stress by stimulating the system of scavenging oxygen free radicals and thus increases the synthesis of antioxidant enzymes (Sachadyn-Król and Agriopoulou 2020). However, the impact of the ozonation process on the antioxidant state depends on the selection of appropriate parameters of this technique, the quality of the tested fruits, and the conditions of their storage (Carletti et al. 2013; Botondi et al. 2015).

Ali et al. (2014) studied the effect of ozonation during the storage of berry fruits and found that the use of high ozone concentration may significantly decrease the content of antioxidants. Zhang et al. (2011) observed that the use of low doses of ozone reduces the loss of antioxidant compounds in stored fruits, and in some cases, it even increases them compared to the initial value. Many studies have been

conducted on the effect of ozone on the content of polyphenols; unfortunately, the results have been ambiguous. However, on the basis of these results, it could be stated that the method of ozone administration is very crucial. Long-term exposure of fruits to ozone can significantly reduce the content of polyphenols, while the use of short-term ozonation helps to preserve these metabolites (Botondi et al. 2015). The influence of ozonation on antioxidants is also related to their location in the cell, as ozone mainly stimulates the antioxidants located in the apoplast (Karaca and Velioglu 2014).

The increase in the content of flavonoids and phenolic acids may result from the modification of the plant cell wall due to contact with ozone. Damage to the cellular structure releases some polyphenols (Najda et al. 2018). The increase in the amount of these metabolites may also be associated with a change in the activity of enzymes caused by the action of ozone, such as polyphenol oxidase (PPO) and POD, which are responsible for the oxidation of phenolic content (Zhu et al. 2019). Superoxide dismutase, ascorbate peroxidase (APX), and catalase (CAT) are also enzymes considered as the main stress-induced response mechanisms caused by ozone (Gutiérrez et al. 2018). In the study of Zhu et al. (2019), the expression of POD was rapidly increased in ozone treatment during storage, which favors protection against the cell membrane damage. Phenylalanine ammonia-lyase (PAL) is involved in the biosynthesis of flavonoids and could be stimulated by various abiotic stresses. The decrease in the content of phenolic compounds is mainly due to the oxidizing potential of ozone (Sachadyn-Król and Agriopoulou 2020). Their degradation is the result of various chemical reactions, including nucleophilic substitution, leading to the oxidation of organic compounds and the formation of hydroxylated and quinone compounds as the formation of aliphatic compounds originates from the breakage of the aromatic ring (Asokapandian et al. 2018). Garcia-Mateos et al. (2019) examined the effect of ozonation (24 mg O_3 L^{-1} min^1) on drinking juice for 7 min, and they noted a significant decrease in the content of metabolites and the related antioxidant effect. Glowacz and Rees (2016) also obtained similar results during the ozonation of chili peppers. Scientists have observed a decrease in the antioxidant activity of green chili peppers exposed to ozone. Sachadyn-Król et al. (2019) conducted ozonation of extracts from the pericarp of paprika and observed that the ozone treatment caused slight changes in the content of secondary metabolites. Hourly ozonation treatment reduces the metabolic changes related to the concentration of polyphenols in pepper fruits.

Piechowiak et al. (2018) analyzed the effect of ozonation on the content of flavonoids and the total antioxidant activity in the fruits of highbush blueberry (*Vaccinum corymbosum*). On the basis of the obtained results, they found that the use of ozone gas limited the reduction of flavonoids as compared to that in the control, non-ozonized sample. Ali et al. (2014) investigated the effect of ozone of stored papaya fruit on the stability of antioxidant compounds. Their results suggest that the application of an ozone dose of 1.5, 2.5, and 3.5 ppm successively for 95 h increased the antioxidant capacity by 0.03%, 30.9%, and 21.9%, respectively, compared to that in the non-ozonized sample. Slightly different results were obtained by Giuggioli et al. (2015) who examined the effect of ozonation on the antioxidant activity of

raspberries. The researchers concluded that ozone activity did not have a significant effect on the ability to eliminate radicals. However, the authors suggested that the ozone dose used may have been too low. According to Piechowiak et al. (2019) the degradation of polyphenolic compounds during fruit storage is the result of enzymatic reactions related to the activity of oxidoreductase enzymes. The authors argue that ozone is likely to inactivate oxidative enzymes and thus reduce or completely limit the breakdown of polyphenols. Moreover, Barth et al. (1995) noted that regardless of the time of ozonation, the activity of the enzyme PPO in stored blackberry fruits was inhibited. Ali et al. (2014) found that the abovementioned action of ozone is due to the activation of enzymes involved in the biosynthesis of polyphenolic compounds.

Piechowiak et al. (2018) studied the effect of ozonation on the microbiological quality and antioxidant activity of blueberries stored under refrigerated conditions, but they did not find any significant changes in the content of flavonoids and phenolic compounds until the seventh day of storage. Scientists, however, observed a significant increase in antioxidant capacity. Other studies by Piechowiak et al. (2019) on the effect of the ozonation technique on the microbiological quality and antioxidant activity of raspberries stored at room temperature proved that ozone reduces the degradation of polyphenols. Moreover, an increase in the DPPH and ABTS radical quenching capacity was observed during daily storage in a cold storage facility.

18.3.3 Influence of Ozonation on Bioactive Compounds in Food Processing

The versatile effect of ozone and its effectiveness in combating microorganisms and contaminants make it suitable for use in all industries, including the food industry. Many studies have been conducted to confirm the beneficial effect of ozone in inhibiting the development of microorganisms in fruits and vegetables, and it has also been proven that ozone is more effective than organic acids or chlorine (Ziarno and Zaręba 2015; Białoszewski et al. 2012; Antos et al. 2013). The abovementioned results have popularized the use of ozone as a disinfectant in terms of storing products in an ozone-rich atmosphere, washing raw materials with ozonated water, or adding ozone directly to liquid products. Today, ozonated water is being increasingly used in fruit and vegetable processing. It is mainly used in the purification of fresh raw materials and for extending their shelf-life. Ozone, as an ethylene inhibitor, slows down the aging processes of plants (Cullen and Norton 2012). This method offers a great opportunity for the use of ozone in the processing of soft fruits, including raspberries, strawberries, blueberries, and currants, as it delays their softening processes (Shah et al. 2019). Ozone is also used in fruit and vegetable processing as a potential pesticide (Balawejder and Piechowiak 2019; Antos et al. 2013), and fungicides reduction agent (Antos et al. 2018; Gabler et al. 2010).

Al-Antary et al. (2019) observed that the use of ozone may also be an effective method for cleaning vegetables. Their study on lettuce leaves clearly indicated that ozonation reduces the content of pesticides. Vegetables are more susceptible to ozone than fruits. This is due to the different structure of tissues and the surface area interacting with ozone. Ozonation of lettuce leaves with a large surface area enabled a much more effective reduction of pesticides than that achieved for most fruits, where ozone penetration is limited due to the dense pericarp (Pandiselvam et al. 2020). The pesticide residue degradation mechanism is based, among others, on photolysis, photocatalysis, and oxidation and reduction reactions. The different forms of chemical oxidation are the basis for the removal of pesticide residues as well as for reducing the risk of microbial contamination in food (Velioglu et al. 2018).

Another sector of the food industry in which ozonation is being increasingly used is meat processing. Here, ozonation is used to eliminate bacteria such as *Campylobacter, E. coli*, *Listeria*, *Clostridium*, and *Salmonella* (Crowe et al. 2012; Novak and Yuan 2004). Another study confirmed that the use of tea polyphenol coating combined with washing with ozonated water improved the storage quality of black sea bream. Ozone is a promising disinfectant in the fish industry, especially when it is combined with other disinfection technologies. The use of ozone in the dairy industry should also be mentioned. Unfortunately, less research has been conducted on this topic to date. An important attempt was the use of ozone to limit the growth of *Cronobacter* in skim milk powder (Torlak and Sert 2013).

The latest reports focus less on microbiological aspects of ozone treatment but rather on the quality of products and explaining the mechanisms. Research results indicate that ozone can be used as an abiotic elicitor of plant defense mechanisms in low-processed food products of plant origin by enhancing the content of secondary metabolites and antioxidant activity. Despite the known mechanisms of action of ozone, there are still many unknowns, especially regarding its action on different food categories. The most commonly used technique of ozonation for plant products is long storage in an atmosphere containing a low concentration of ozone (Tzortzakis et al. 2007). The second approach involves a high dose ozone single treatment. The comparison of short- and long-term treatment was made by Botondi (Botondi et al. 2015), who showed that these two approaches can have a very different result. Ozone long term treatment (additionally 0.5 g/h for 4 h each day until 35% weight loss) resulted in significant decrease in phenolic and anthocyanin contents, while ozone shock treatment (1.5 g/h for 18 h) preserved polyphenols, anthocyanins and carotenoids in wine grapes during postharvest dehydration.

Perhaps the future of food processing is to combine ozonation with other methods of extending life, such as coating. In the study of Bambalele et al. (2021) the effect of ozone and coating on antioxidant activity and biochemical properties of mango fruit was investigated. The parameters measured by ascorbic acid, phenolic content, and antioxidant capacity (FRAP and DPPH) indicated that ozonation (0.25 ppm for 24 and 36 h) and coating (with moringa leaf extract and carboxymethyl cellulose) enhanced the antioxidants of mango fruit during storage. Furthermore, this combine treatment can preserve the membrane integrity, antioxidants, and enhance the fruit quality (Table 18.2).

Table 18.2 Recent studies investigating the effects of ozone processing on food bioactives

Food matrix	Bioactive compound	Treatment	Effect of processing	Reference
Black mulberry fruit	Ascorbic acid, anthocyanins	0.64 and 5 mg/m^3, 6 days	Increase in ascorbic acid content, no change in anthocyanin content	Tabakoglu and Karaca (2018)
Grape tomatoes	Phenolic compounds, lycopene, vitamin C	3.43 mg/L and 6.85 mg/L, 2 and 4 h	Reduction in the content of phenolic compounds, lycopene and vitamin C	Wang et al. (2019)
Hot red pepper fruits	Phenolic compounds, antioxidant activity	2 mg/L for 3 h	Increase of antioxidant activity and phenolic compounds	Sachadyn-Król et al. (2019)
Kiwi	Phenolic compounds, flavonoids, antioxidant activity	300 ppb, 60 days	Increase of antioxidant activity with no changes in the content of flavonoids and a decrease in the concentration of phenolic compounds	Goffi et al. (2020)
Melon	Carotenoids, phenolic compounds, antioxidant activity	10.0 ± 4.8 and 38.0 ± 8.1 g/L for 30 and 60 min	No changes in carotenoids, decrease in phenolic compounds and antioxidant activity	Miller et al. (2018)
Melon Peel	Total phenolics, chlorophylls, vitamin C, and antioxidant activity	152 ± 71 (30 min) and 369 ± 193 ppm (60 min) at 15 °C	Increase of vitamin C, total phenolics, and chlorophylls after 30 min of exposure. After 60 min almost 100% increase in the total phenolics	Miller et al. (2021)
Orange juice	Ascorbic acid, total phenolics	600 mg/h for 30 min	Decrease in ascorbic acid and total phenolics	Shah et al. (2019)
Rocket leaves	Phenolic compounds, antioxidant activity, content of chlorophylls, carotenoids and ascorbic acid	1, 5 and 10 ppm, 10 min	No changes	Gutiérrez et al. (2018)
Raspberries	Succinate dehydrogenase, cytochrome C and H+ -ATPase oxidase, antioxidant activity	8–10 mg/L, 30 min every 12 h for 3 days	Increased activity of enzymes involved in oxidative phosphorylation, higher antioxidant activity	Piechowiak et al. (2021)
Strawberry	Phenolic compounds, Anthocyanin	0.1 ppm O_3 in water for 2 min	Increase of phenolic compounds and content of anthocyanins	Nayak et al. (2020)
Watermelon juice	Total phenolic content, lycopene, ascorbic acid	1 L/min for 5, 10, 15, 20 and 25 min	The content of ascorbic acid and lycopene degraded significantly, total phenolic content decreased	Lee et al. (2021)

18.4 Limitations and Advantages of Ozone Technology

Despite many years of use of ozone in food production worldwide, it is still used mainly for sanitary purposes in Europe. In the European Union countries, including Poland, this technique has aroused much distrust among food producers, and therefore, it is still not very popular (Ziarno and Zaręba 2015). Despite its many advantages, ozone treatment has several obstacles that make it still a less commonly used method.

The advantages of ozone treatment include rapid and effective action in combating harmful microorganisms and pollutants. Ozone exhibits biocidal activity against molds, viruses, bacteria, and yeasts as well as against fungi and bacterial spores (Ziarno and Zaręba 2015; Karaca and Velioglu 2020). It is one of the most powerful disinfectants. Another advantage is the lack of hazardous decomposition products and byproducts of the ozone molecule in the finished food product; thus, its use does not harm the produced food. Furthermore, ozone is produced on site, thus eliminating the possible harmful effects associated with its transport or storage on human health. In addition, food producers using the ozone method in their plants can save on the supply of detergents, as the use of ozone reduces the use of hot water and sanitizing agents in their purification installations.

The main disadvantage of ozone is its toxicity. The production or storage of ozone in high concentrations is harmful to human health. Both direct and indirect effects of ozone can damage the immune and respiratory systems and can cause pulmonary congestion. The harmfulness of ozone depends on its route of administration, concentration, and exposure time. Ozone concentration exceeding 0.1 ppm may damage the cornea of the eyes, the mucous membranes of the throat and nose, and the ciliated cells of the lungs (Ziarno and Zaręba 2015). According to US standards, ozone residues in bottled water should not exceed 0.4 mg g^{-1} (Tabakoglu and Karaca 2018). However, there are no legal provisions on the method to measure the residual ozone content in foods. Moreover, long-term exposure to high ozone concentration may change the shape of the food packaging and reduce its barrier effect. Ozonation may adversely affect the sensory and quality characteristics of food. This process primarily changes the aroma of the product. Nadas et al. (2003) studied the effect of ozonation on the quality of stored strawberries, and they observed a change in the smell of fruit. This was caused by a significant loss of volatile compounds due to their oxidation by ozone. The use of ozone may also adversely affect the color of the product. Das et al. (2006) noted that ozone gas at 30 ppm concentration caused discoloration of the surface of cherry tomatoes (Wang et al. 2019). Ozone treatment also leads to discoloration of fresh plants with a high water content (Goffi et al. 2020). Similar observations were noted by Glowacz and Rees (2016) when determining the effect of ozonation on the color of chilies. In this case, the use of a high concentration of ozone caused a visible change in the green color of chilies. Most likely, this phenomenon is related to the accelerated degradation of chlorophyll due to the action of ozone. Ozonation may also deteriorate the nutritional value of the product. Alothman et al. (2010) noted that the use of a high dose of ozone may result

in a significant reduction in vitamin C (Miller et al. 2018). Similar results were obtained by Wang et al. (2019) who analyzed the effect of ozonation on the quality of grape tomatoes. The loss of ascorbic acid content might be related to the direct reaction of ozone or indirect reaction of secondary oxidant free radicals on the activity of several enzymes such as APX and ascorbate oxidase (AO) (Yeoh et al. 2014). Velioglu et al. (2018) found that the use of ozone to degrade pesticides can also produce compounds that are more harmful than the parent compounds. Another obstacle in the use of ozone in food production is its limited storage and transport time; thus, it usually has to be produced on site (Guzel-Seydim et al. 2004). The high costs associated with the need to purchase complex equipment discourage entrepreneurs from using ozone in food production plants.

18.5 Plasma Technology

Plasma is characterized as the fourth state of matter, next to solids, liquids and gases, which is constituted of a large number of ionized gas, electrons, free radicals, ions, in their fundamental or excited states with a net neutral charge (Chizoba Ekezie et al. 2017; Pankaj et al. 2018). The transition from one state to another is done by addition of heat. These active species are capable of initiating reactions in any system as they have sufficient energy to break the covalent bonds, playing an important role in microbial inactivation by damaging the proteins, lipids and nucleic acids (Gupta et al. 2017). Plasma was discovered by Crookes in 1879 (Ozen and Singh 2020) but the plasma technology and plasma research has evolved at a rapid rate in various areas at the late 1990s (Gupta et al. 2017).

Different plasma applications are developing rapidly in various specific fields, such as biology, medicine and agriculture, and mainly in food and medicine industries (Babajani et al. 2019). Nowadays plasma treatment has been used as a potential bio-decontamination technology for microbial and agrochemicals residues on food (Sarangapani et al. 2017; Moutiq et al. 2020) and in modification of packaging properties (Romani et al. 2020; Zhu et al. 2020). Plasma is also used to modify raw materials in order to provide a product with added value (Charoux et al. 2020). Especially the use of plasma in food decontamination can be reach in some cases up to a 5-log reduction in microorganism count (Rodríguez et al. 2017). The efficacy of the plasma treatment mainly depends on the penetration of plasma in foods and the physiological changes initiated by reactive species (Misra et al. 2016a).

18.5.1 Basic Principles of Plasma Processing

In order to produce plasma it is necessary to supply energy in a gas to cause its ionization (López et al. 2017). Although any kind of energy which can ionize the gases, such as electrical, thermal, optical (UV light), radioactive (gamma radiation)

and X-ray electromagnetic radiation can be used for plasma generation, electric or electromagnetic fields are the most widely used. In food processing, dielectric barrier discharge (DBD), and jet plasma are the most commonly used plasma generation sources (Pankaj et al. 2018). In DBD the generation of plasma takes place between two metal electrodes covered with one or more dielectric layers, that limit the charge and prevent an arc-like discharge (Moutiq et al. 2020). In plasma jet devices there are two concentric electrodes, where the inner electrode is typically connected to a radio frequency power at high frequency causing ionization of the feed gas, which exits the nozzle and gives a 'jet-like' appearance (Pankaj et al. 2018).

18.5.2 Plasma Chemistry and Technology

Plasma can be divided in different categories, based on different parameters such as, plasma sources, electron density, pressure, temperature, ionization, collision frequency, etc. According to their temperature, plasma is divided in thermal plasma and non-thermal plasma. The temperature of the plasma generally refers to the temperature of the electrons ions and neutral composing it (Charoux et al. 2020). Thermal plasma can reach temperatures of up to several thousand Celsius degrees and non-thermal plasma can reach temperatures between 30 and 60 °C (Alves Filho et al. 2020; López et al. 2017). Contrary to thermal plasma, cold plasma is produced at low levels of pressures and power, and has a non-equilibrium thermodynamic temperature distribution between the electrons and ions, thus designated as non-equilibrium plasmas (Das et al. 2006) while thermal plasma designated as equilibrium plasma.

According to the pressure, plasma is divided in low-pressure plasma (<1 Pa), moderate pressure plasma (≈100 Pa) and atmospheric pressure plasma (100 kPa) (Lee et al. 2021). In addition, the ionized gas that can be used, varies from atmospheric air to oxygen (O_2), nitrogen (N_2), carbon dioxide (CO_2) and noble gases, including helium (He), argon (Ar), and also the combinations of different gases and gas with humidity (Babajani et al. 2019; Zhao et al. 2019). The use of He as a supply gas on the one hand reduces the cost of the process, as He ionizes more easily than other gases, requiring smaller quantities of voltage and electric consumption. On the other hand He is very expensive gas, compared with air or other gas mixtures of nitrogen and oxygen, and this is a reason for its use in experimental cold plasma systems, and not in commercial scale systems (Alves Filho et al. 2020).

The reactive oxygen species (ROS), like hydrogen peroxide (H_2O_2), ozone (O_3), superoxide anion ($O_2^{\bullet}$), hydroxyl radicals (•OH), singlet oxygen ($^1O_2$), alkoxyl ($RO^{\bullet}$), peroxyl ($ROO^{\bullet}$), hydroperoxyl ($HO_2^{\bullet}$) and carbonate anion radical ($CO^{\bullet}{}_3^-$), the reactive nitrogen species (RNS), like excited molecules of N_2 and N_2O and other nitrogen oxide species (NO, NO_2, N_2O_4 and N_2O_5), the energetic ions, and charged particles, that are produced by the plasma discharging in air, act as very strong oxidizers, and effect on food components, promoting different chemical reactions and imparting changes in the food composition (Feizollahi et al. 2020; Puligundla et al.

2020). These reactive species are produced by applying energy at specific frequencies to a gas or a gas mixture (Ramazzina et al. 2015).

Another important characteristic of plasma is the electron density which represent the plasma density itself. Electron density is the number of free electrons per unit volume. Plasma is also divided in fully ionized (≈100%) and partially or weakly ionized (<1%) (Charoux et al. 2020).

18.5.3 Cold Plasma (CP) and Cold Atmospheric Pressure Plasma (CAPP)

Cold plasma (CP), or non-thermal plasma (NTP), is an innovative, green technique which is gaining considerable interest with its use as non-thermal technology for processing foodstuffs, with significant advantages. In fact this technique is cheap, flexible and environmentally friendly, offering many potential applications, as is suitable for inactivation of bacteria, fungi, spores, and viruses (Pankaj et al. 2018). Cold plasma is widely used in food processing, as it has strong antimicrobial effects against major pathogenic and spoilage microorganisms, such as *Escherichia coli* (Bermúdez-Aguirre et al. 2013) *Salmonella typhimurium* (Fernández et al. 2013) and *Listeria monocytogenes* (Ukuku et al. 2019) in fresh produce. Cold plasma can also be used for mycotoxins elimination (Puligundla et al. 2020; Agriopoulou et al. 2020b), for decontamination of both foodstuffs and food packaging materials (Pankaj et al. 2014) and for enzyme inactivation (Misra et al. 2016b). The composition of the food remains stable during and after the cold plasma treatment, which is relatively inexpensive in terms of required energy, while the relative processing, ranges from seconds to a few minutes depending on the product (Gupta et al. 2017).

Cold atmospheric pressure plasma (CAPP) induced by dielectric barrier discharge (DBD) has many industrial applications (Porto et al. 2020). CAPP has been used as an alternative food disinfection method and can be used to improve microbial quality and enhance the food safety (Bovi et al. 2019). CAPP operates at mild processing temperatures, high efficiency with inactivation levels above 5–7 $\log_{10}$ for a plethora of microorganisms, short duration of treatment, low set up and operating costs, and low environmental impact due to limited energy requirements and no generation of toxic by-products or residues (Corradini 2020).

18.5.4 Influence of Plasma Processing on Bioactive Compounds in Food Processing

The effect of plasma processing in food bioactives, in maintaining quality attributes, and in nutritional value depends on different parameters, and is undoubtedly strongly dependent on the food matrix and on the experimental conditions such as

voltage, frequency, flow rate, treatment time and plasma generation method. The type of gas that is used is one of the most the most crucial parameter (Pankaj et al. 2013). The positive, the negative or the no significant effect of plasma treatment in food bioactive are contradictory in many publications. Plasma treatment have been used in many categories of plant products, mainly focused on the preservation of their qualitative characteristics.

Cold plasma is a promising non-thermal technology for the decontamination and preservation of fresh and fresh-cut produce. The review of Bovi et al. (2019) shows that in general, the use of cold atmospheric pressure plasma has a great potential as an alternative decontamination method for food and can be used to improve microbial quality and enhance the food safety of berries. In another study, blueberries submitted to cold plasma using air as ionized gas, showed an increase in phenolics and flavonoids after 1 min of processing for applied voltages (60 and 80 kV) (Nadas et al. 2003). As it concerns the fresh produce a number of physicochemical and biochemical modifications mainly affect the taste, color and texture of fresh-cut fruits and vegetables. Tappi et al. (2016) studied the use of gas plasma for the treatment off resh-cut apples. Main quality and metabolic parameters such as PPO activity, respiration and heat production were assessed. From the obtained results regarding enzymatic browning, inhibition PPO residual activity decreased linearly by increasing the treatment time (up to about 42%), proving that this technique is very encouraging for fresh-cut apple stabilization. Mahnot et al. (2020) evaluated the microbial and quality aspects in-package cold plasma decontamination of fresh-cut carrots. Plasma processing was effective in reducing natural microflora of carrots, as 2 log10 CFU/g reduction in the population of both, total aerobic mesophiles, and yeast and mold were observed. Moreover the changes in pH, colour, texture, and total carotenoids of cold plasma treated carrots were minor. Lacombe et al. (2015) conducted a study on atmospheric cold plasma inactivation of aerobic microorganisms on blueberries and effects on quality attributes using with a mixture of 4 cubic feet/minute (cfm) of Cold plasma jet and 7 cfm of ambient air. The obtained results showed that the use of atmospheric cold plasma significantly reduced total aerobic plate count, and a significant reduction in anthocyanins was observed after 90 s. A decrease in the carotenoids content of cut kiwi fruit upon subjecting to DBD plasma treatment in air was reported by Ramazzina et al. (Ramazzina et al. 2015).

Juices are another category of plant products to which the use of plasma is applicable. The critical step in juice production is pasteurization, the most widely applied technique for successful inactivation of vegetative microorganisms and enzymes, used for prolongation of the juice shelf life. Plasma treatment could be used alternatively compared to to traditional thermal pasteurization. Garofulic et al. (2015), studied the effect of gas phase plasma treatment on the anthocyanin and phenolic acid content of sour cherry juice, and found that short treatment (3 min) of larger volume of the juice (3 mL) resulted in the highest concentration of both anthocyanins and phenolic acids. Herceg et al. (2016), studied the gas phase plasma impact on phenolic compounds in pomegranate juice and found an increase of 33.03% in total phenolic content (Kovacevic et al. 2016). Rodríguez et al. (2017), studied the effect of indirect cold plasma treatment on cashew apple juice and in general found

that a low N_2 plasma flow rate promoted an increment of the vitamin C, total polyphenol content and in the antioxidant activity.

Grains and flours during storage can be contaminated by major food-borne pathogens with numerous changes in the chemical and physical properties. Several studies have reported that expect from thermal treatment for pathogens inactivation, plasma processing has been effectively used. Lee et al. (2016) studied the evaluation of cold plasma treatments for improved microbial and physicochemical qualities of brown rice. From the obtained results, the microbial safety of brown rice against microorganisms of *Bacillus cereus, Bacillus subtilis*, and *E. coli* O157:H7, as a 2.30 log CFU/g reduction was observed, parallel to the slight changes to its physicochemical quality.

Misra et al. (2015b) investigated the effect of atmospheric pressure cold plasma treatments (60–70 kV, 5–10 min) on structural and functional properties of hard and soft wheat flours. An improvement in the dough strength and optimum mixing time for both flours was observed, attributed to oxidation of protein sulfhydryl groups and subsequent disulphide bond formation between cysteine moieties. Bahrami et al. (2016) reported that atmospheric pressure cold plasma treatments (air, 15 V and 20 V) for 60 or 120 s. reduced total free fatty acids and phospholipids and the results were dose dependent, proving the potential of cold plasma as a tool to modify flour functionality.

Dried food ingredients such as spices, herbs, powders and seeds need to be disinfected for food safety and quality reasons. The review of Charoux et al. (2020) shows that in general, cold plasma enhances seed quality and germination and improve antioxidant profile without causing oxidative damage to the seed (Yeoh et al. 2014). Kim et al. (2017) evaluated the microbial decontamination of onion powder using microwave-powered cold plasma treatments. The growth of *B. cereus*, *Aspergillus brasiliensis*, and *E. coli* O157:H7 in the treated onion powder was assessed during storage at 4 and 25 °C as well as the physicochemical and sensory properties of the powder such as antioxidant activity, quercetin content, and color properties. According to the obtained results improved microbiological safety in terms of *B. cereus* and *E. coli* O157:H7 and retarded the growth of *A. brasiliensis* in onion powder, was observed. Moreover all treatments did not significantly affect the physicochemical and flavor properties.

In a very recent study, Beyrera et al. used cold plasma in powdered *Spirulina* algae for spore inactivation and preservation of bioactive compounds. The results of this study showed an increase in total phenolic content (TPC) by a factor of up to 2 in a plasma nitrogen treatment (Beyrer et al. 2020). Table 18.3 summarizes recent studies on the effects of plasma processing on food.

Table 18.3 Recent studies investigating the effects of plasma processing on food bioactives

Food matrix	Bioactive compound	Determination method	Treatment	Effect of processing	Ref
Coconut water	Peroxidase POD, phenolic compounds	NMR spectroscopy and chemometric analysis	Atmospheric cold plasma processing (ACP)	Inactivation of peroxidase POD (enzymatic reduction), reduction of some phenolic compounds, increased the content of others, not affect total soluble solids, titratable acidity, and color	Porto et al. (2020)
Minimally processed kiwifruit	Ascorbic acid, polyphenols, pigments	Radical scavenging assay	Atmospheric double barrier discharge (DBD) plasma treatment	Immediate loss of pigment and visual quality, improvement in color retention, no significant changes in antioxidants (ascorbic acid and polyphenols)	Ramazzina et al. (2015)
Jujube slices	Procyanidins, flavonoids, phenolics	Scanning electron microscopy (SEM), thin-layer drying mathematical models	Cold plasma pretreatment time and drying temperature	Improving the drying rate, improvement of the content of procyanidins, flavonoids and phenolics, reduce the production of 5-hydroxymethylfurfural, inhibit the degradation of antioxidants	Bao et al. (2021)
Tomato pomace	Phenolic compounds	Ultra-performance liquid chromatography (UPLC)	High voltage atmospheric cold plasma (HVACP) with different working gases (air, Ar, He and N_2)	Enhancement of the extraction of phenolic compounds, increase the antioxidant capacity of the extracts	Bao et al. (2020)
	Antioxidant capacity	UV/VIS spectroscopy			
Mandarins	Ascorbic acid,	High-performance liquid chromatography (HPLC)	Antimicrobial washing and in-package atmospheric dielectric barrier discharge cold plasma (ADCP)	Enhancing the shelf life of mandarins in plastic packaging by inhibiting the growth of *Penicillium digitatum* by 77.1% in fruit, while minimizing changes in fruit quality during storage	Bang et al. (2020)
	Total phenolic content, antioxidant capacity	UV/VIS spectroscopy			

(continued)

Table 18.3 (continued)

Food matrix	Bioactive compound	Determination method	Treatment	Effect of processing	Ref
Spirulina microalgae	Total phenolic content	UV/VIS spectroscopy	Surface micro-discharge cold atmospheric pressure plasma (SMD-CAPP)	Increase in total phenolic content	Bao et al. (2021)
Radicchio leaves	Antioxidant compounds	ABTS (2,2-azinobis-(3-ethylbenzthiazoline-6-sulfonic acid) and ORAC (oxygen radical absorbance capacity) antioxidant assays	Dielectric barrier discharge cold plasma device	No change in antioxidant activity	Bermudez-Aguirre (2020)
Camu-camu juice	Antioxidant compounds, ascorbic acid and total monomeric anthocyanins	UV-vis spectroscopy	Cold plasma food processing	Improving concentration of bioactive compounds (antioxidant compounds, ascorbic acid and total monomeric anthocyanins), inactivation of degradative enzymes, negative result in color	Castro et al. (2020)
Fresh sliced apples and potatoes	Polyphenol oxidase (PPO), peroxidase (POD)	UV–Vis spectroscopy	Plasma processed air	Reducing the activity of polyphenol oxidase (PPO) and peroxidase (POD)	Bußler et al. (2017, 2020)
Cashew apple juice (*Anacardium occidentale* L)	Total phenolic content, total flavonoid content	UV–Vis spectroscopy aluminum chloride assay	Benchtop plasma system	Increase in polyphenol and flavonoid	Agriopoulou et al. (2020b)
	Vitamin C	UV–Vis spectroscopy		Increase in vitamin C	
Blueberries	Anthocyanin content	High-performance liquid chromatography (HPLC)	In-package high voltage dielectric barrier discharge plasma reactor	Decrease in anthocyanin content	Sarangapani et al. (2017)
	Total phenolic content, total flavonoid content	UV–vis spectroscopy		Increase in phenols and flavonoids	
Fresh and dried walnut	Total phenolic contents	UV–vis spectroscopy	Plasma jet treatment	Increase in total phenolic content after 15 and 30 days of storage (4 °C)	Amini and Ghoranneviss (2016)

18.6 Limitations and Advantages for Plasma Technology

Plasma technology as non-thermal food processing technology is very attractive for the products which are heat-sensitive as its working temperature is near to room temperature. Plasma can be used for the inactivation of pathogenic microorganisms both in fresh and processed food, ensuring food safety to the consumer as it is a technology that is both chemical and water-free, as well as environmental friendly (Gupta et al. 2017).

Although plasma is a rapid, effective and economically sustainable technology, which requires low energy input and investment, several factors need to be optimized in order to achieve successful application of this technology in food (Charoux et al. 2020). Plasma technology is at its infancy, and there is no data on the dosage or the toxicity of this technology. Also there is no standard for determining of plasma dosage (Ozen and Singh 2020). The difference between plasma treatments can be derived from the different structures of the food and different storage conditions (Amini and Ghoranneviss 2016).

18.7 Conclusions

Nowadays, the tendency for consumption of fresh, natural and safe food products with higher quality and longer shelf life without using the chemical preservatives has been increasing. From this point of view, increasing the storage time with preservation of the nutritional quality of the products is worth considering. The preservation of the organoleptic characteristics of food is a key goal of the food industry. The inactivation of food spoilage and pathogenic organisms and enzymes without appreciably altering the critical nutritional, organoleptic and functional characteristics of food is a top priority. Ozone and plasma treatment have been used as a potential bio-decontamination technology for microbial and chemical risks associated with food products, aiming to retain the quality attributes of food. Treatment time and power density, are mainly determine the success of each treatment. As these techniques except for advantages have some disadvantage, in order to produce high-quality products, with ozone and plasma treatment, more target compounds investigations are required, and each food matrix must be evaluated regarding its quality and composition after these treatments.

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Chapter 19
Influence of Nano-based Food Processes on Food Bioactives

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19.1 Introduction

Nanotechnology has been qualitatively explained as the nano-scale control and manipulation of matter, usually at the molecular and/or atomic level, and in dimensions ranging from 1 to 100 nm for the innovative enhancement or improvement of their functionality and geometrical characteristics or generally for the finesse fabrication of novel materials and process methodologies (Chellaram et al. 2014; Kehinde et al. 2020a, b, c, d). It has been applied in integration with other technological fields of medicine and health, pharmaceutical, cosmeceutical, food and agriculture, mechanical systems and material fabrication, and in electronics and mechatronics, amongst several others. Accordingly, it has become part of the

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everyday life but especially in the indispensable aspects of food and agriculture, nanotechnology has been applied in subjects such as aquaculture, food packaging, unit operations in food and feed processing, and water treatment procedures (He et al. 2019).

In 1902, the Austrian-Hungarian chemist Prof. Richard Adolf Zsigmondy (1865–1929) teamed with Henry Siedentopf (1872–1940) and developed the ultramiscope. Later, in 1912, Prof. Zsigmondy would use the equipment for immersion ultramicroscopy for the nanoparticulate observation of suspended fluidic materials; a feat that aided his obtainment of the Nobel Prize in Chemistry in 1925. In addition, he coined the terms nano- and meter to form nanometer for the definite particulate characterization and dimensional evaluation of aqueous colloidal gold (Hulla et al. 2015). Thereafter, neoteric nanotechnology which goes beyond nanoscale characterization to the manipulation level was birthed by Richard Phillips Feynman (1918–1988), an American quantum physicist, who in December 1959, gave a presentation to American Physical Society titled "Plenty of Room at the Bottom". He sparked the scientific curiosity of writing the whole 24 volumes of Encyclopedia Brittanica in their entirety on the head of a sixteenth inch pin and further on the technological manipulation and control of atoms and molecules for the fabrication of new materials (Ray and Gupta 2018). Well over a decade later, the Japanese semiconductor physicist Prof. Norio Taniguchi (1912–1999) coined the terms nano- and technology into nanotechnology, and expounded its employment for the separation, processing, deformation and consolidation of materials by a molecule or atom (Hulla et al. 2015).

Nanotechnology has been incorporated into processing of cosmeceuticals based on specific influences such as increased efficacy, active transport of functional ingredient, controlled release, sustained physical stability, enhanced entrapment efficiency, and improved occlusive attributes (Kaul et al. 2018). Products such as anti-wrinkle creams, toothpastes, deodorants, shampoos, aftershave lotions, perfumes and sunscreens have been developed for daily applications and therapeutic usage against quick skin aging, skin dryness, hair infections, and body spots, amongst several others (Melo et al. 2015; Kehinde et al. 2020a, b, c, d). Such therapeutic products involving the subjects of pharmaceutics and cosmetics are referred to as cosmeceuticals and when processed on the nanoscale, they are termed as nanocosmeceticals. Nanocosmeceuticals can be in the form of liposomes, nanoemulsions, nanoniosomes, nanocapsules, nanocrystals, nanospheres, nanocapsules, nano-cubosomes, nanodendrimers and nanofibrils (Duarah et al. 2016). These nanomaterials have special characteristics with nanoemulsions found to possess distinctive attributes of tactility, texture and transparency, nanopigments observed to enhance the efficiency of sunscreen products and also possess transparency, nanoliposomes bearing little vesicles comprising of cosmetic assortments that shield light-sensitive ingredients and formulants prone to oxidation, and nanocapsules widely utilized in preparing skin care products (Lohani et al. 2014).

Pharmaceutical nanotechnology subsumes the employment of nanoprocessing on pharmaceutical materials for molecular scale applications such as drug delivery, biosensing, diagnostic imaging and therapeutic management of bodily disorders.

The chief benefits of nanotechnology in pharmacology are related to the enhancement of the delivery and effectiveness of administered drugs through mechanisms such as improved dissolution rate, elevated surface area, increased solubility, biodelivery to targeted sites of action, boosted oral bioavailability, controlled release, reduction in dosage required to effect changes, quicker onset of therapeutic effects, and conservation of functionality (Maravajhala et al. 2012). Nanopharmaceuticals are usually in the form of nanosuspensions, nanoemulsions, nanomicelles, Solid lipid nanoparticles, protein nanoparticles, Nanocrystals, Biopolymeric nanoparticles, Metallic nanoparticles, inorganic nanoparticles, and polysaccharides nanoparticles. Pharmaceutical nanoemulsions are prepared using techniques such as sonication, microfluidization, high pressure homogenization, and phase inversion, and for nanomicelles, procedures such as grafting polymerization, solvent pH Change Solubilization, and Co Precipitation are applied (Thakur and Agrawal 2015). Drug-related nanoparticles are prepared by methodologies such as in situ polymerization (dispersion polymerization and emulsification polymerization), precipitation, salting-out, emulsification-diffusion, emulsification-evaporation and interfacial polymerization functionality (Maravajhala et al. 2012). Protein nanoparticles are prepared from insoluble (gliadin and zein) and water-soluble (human and bovine serum albumin) natural protein substrates; dendrimers are prepared either from their cores with an outward extension or by converging from outwards to the inner core; polymeric micelles are produced by precipitation of a singular block through solvent addition or by the simple dissolution of a solvent-based polymer with a subsequent dialysis; and liposomes are synthesized using solvent injection, thin layer hydration, surfactant solubilization, and mechanical agitation (Patra et al. 2018).

In building and construction engineering, nanotechnology has been described to be potentially effective for increasing the durability and strength of composites made with cement, producing nanosensorial materials having abilities to self-repair and sense environmental changes, fabrication of corrosion-free and affordable steel, production of materials with remarkably higher thermal insulation attributes relative to conventionally available counterparts, production of thin films and coats having the abilities to clef-clean and self-color change for energy optimization, and more importantly, production of materials with minimized environmental pollution (Pacheco-Torgal and Jalali 2011). Nanomaterials are being studied and used in concrete mixes for these purposes. Silica fume and nanosilica having high strength (flexural, compressive and tensile), low permeability and enhanced durability; Iron III oxide nanoparticles having electrical properties that are useful for sensing of damage and stress; nanotitanium dioxide with good stability, affordability and of minimal environmental pollution outcomes; nanoclay for the reinforcement of cement-originated composites; nanocalcium carbonate for the development of concrete strength and its hydration; and Carbon nanotubes having a hexagonal lattice and a resultant tensile strength greater than steel by a factor of five (Silvestre et al. 2015). Furthermore, nanocoatings for buildings have been studied with some of their special functionalities such as self-healing capabilities, wear and scratch resistances, flame retardation, corrosion retardation, anti-graffiti, and energy efficiency

using nanopreparations such as nanochromic materials (such as oxides of nickel, vanadium, titanium and tungsten) and oxides of other metals including aluminium, silver, and zirconium (Boostani and Modirrousta 2016).

The neoteric field of nanomedicine which is basically composed of nanotechnology and medicine has improved in recent years especially in the aspect of research. This is comprehensively allied to the special strengths of nanomaterials such as quicker biochemical reactions and effects, improvable biocompatibilities and more importantly, their very minute sizes (Boisseau and Loubaton 2011). The stability of nanomaterials make them useful for optimized biodistibution, their versatility make them suitable for increased targeted efficiency through adjustment of physicochemical properties such as shape, size and architecture, and their large surface area implies that they have the potentials to bear large quantities of biological materials (Barkalina et al. 2014). Their operations span across various innovative tasks such as gene therapy, phagokinetic studies, tissue and DNA engineering, tumor detection, isolation and purification of biological molecules, drug delivery systems and cancer therapies, thus making the healing process faster and managing metabolic syndrome disorders (Jena et al. 2013; Sharma et al. 2020). As medical implants, nanomaterials can function in enhancing bone-to-bone contacting, improving osteointegration, improving cellular responses by enhancing protein adsorption and increasing mechanical characteristics such as wear resistance (Saji et al. 2010).

19.2 Nanotechnology in Food and Agricultural Processing

19.2.1 Nanosensor Development

Nanosensor technology has been recently studied in food and agricultural processing for their abilities to detect and offer quantitative analyses on target materials which could be adulterants, toxins, pathogens, organic substances, gases, ingredients, pathogens or other foreign bodies based on their rapid responses, finesse detection limits, compatibility for batch and continuous processing, and their versatility. In agriculture generally, nanosensors are being studied for usage as soil condition monitors, and for detection of pests at their early growth stages before they create economic loses (Duncan 2011). For food products, they have been used for food contact packaging as plasmonic, fluorescent and magnetic nanosensors for the detection of pesticides and toxins with others being constructed with materials such as graphene, Tin zinc oxide and Polyvinylidenefluoride used for humidity detection, pathogen detection and sensory (taste) analysis (He et al. 2019). These sensors bear the capabilities to dramatically transform the efficiency of analytical detections by governmental regulatory agencies or quality assurance personnel in the food industry for the timely discovery of toxicants and allergens and the eventual enhancement of food safety (Duncan 2011). In addition to quality assurance and food safety benefits, the integration of nanosensors will be beneficial for improving organoleptic,

textural and nutritional attributes of processed food. These sensors are usually fabricated to work with electrochemical, biological, biochemical mechanisms that would create specific responses to gases, pathogens or other targeted materials at the nanoscale and still offer quantifiable results (Banerjee et al. 2016). Nanosensors can work through non-invasive mechanisms to sense gases emitted as a result of biological agents such as spoilage and/or pathogenic microbes, insects, or rodent attacks simply by interpreting changes in their optical, electrical or chemical properties.

19.2.2 Nanofluids

Food processing comprises of several unit operations that involve heat transfer such as pasteurization, blanching, sterilization, drying, refrigeration, dehydration, and freezing for purposes such as shelf-life extension, value addition, weight reduction, or enhancement of safety. However, several heat exchanging equipment fabricated over the years bear numerous limitations related to efficient energy utilization arising from thermal loses. Nanofluids have been studied in recent years to possess high thermal conductivity and heat transfer coefficients thus leading to maximal energy utilizations within short time durations, retainment of organoleptic attributes of foods and other sensorial characteristics, and minimized degradation of bioactive components in foods (Jafari et al. 2017) They have been described as colloidal suspensions of optimal stabilities, embodied with nanoscale particles of metallic or non-metallic natures dispersed in liquid phases (Choi 2009). Their relative potencies lie in their abilities to facilitate heat transfer due to their inherent characteristics, especially their higher surface-area-to-volume-ratio. Nanofluids can be prepared through the one-step technique which involves the concurrent production and dispersion of nanoscale particles in a base fluid to achieve nanofluid formulations of better stabilities (Salari and Jafari 2020) Nanofluid formulations such as Graphene nanosheets/water, Ag/water, TiO_2/ethylene glycol-water, and Al_2O_3/water have been studied to potentially have their thermal conductivities enhanced from around 7 to as high as 34% and with the possibilities of having their viscosities heightened to up to 215% (Salari and Jafari 2020).

19.2.3 Nanowater Treatment

Nanoprocessing has been gainfully employed for the purification and overall quality enhancement of potable water using nanomaterials such as silver nanoparticles and carbon nanotubes amongst others for the microbial disinfection of water through mechanisms such as disruption of microbial cell walls, alteration of DNA synthesis, retardation of enzymatic activity, and the photocatalytic syntheses of reactive oxygen species (ROS) for the damage of cellular organelles of microbes (Ahmed et al. 2013). In addition, they can function as antimicrobials by direct interruption of

trans-membrane electron transfer and causing significant imbalance for the required metabolic processes necessary to maintain the viability of the microorganism. Other nanomaterials such as self-assembled monolayers on mesoporous silica, dendrimers, nanosorbents, nanofillers, single enzyme nanoparticles have been used for the fabrication of methodical filters and membranes with enhanced features such as desirable flux rates, reusability, efficient purification, enhanced durability, selective permeability and operational with cost and energy effectiveness (Dhakras 2011). Nanomanufactured membranes are constructed with composites of nanomaterials making them workable to selectively absorb target contaminants specific to the water source along with improved resistance to the fouling constraint which would help reduce costs in the long run. Nanomaterials are usually applied as integrated composites with other materials to augment their mechanical attributes or other lacking functionalities when formulating such membranes. Some examples of such membranes include nanoreactive membranes such as Zero-valent Fe laden cellulose acetate membrane, Silica and cellulose-based membranes, and Polymer-impregnated ceramic TiO_2 filters, and Nanostructured membranes which includes the Nanocapillary array membranes (Theron et al. 2008).

19.2.4 Nanopesticides

The incorporation of nanotechnology with agropesticide processing has shown to address common limitations of conventional preparations such as stability on exposure to environmental conditions such as sunshine and rainfall, high dosage necessities for significant impact, toxicity to plants and animals, weak penetration of insects and microbes, and high costs of ingredient formulation and pesticide manufacturing (Hayles et al. 2017). Correspondingly, nanopesticides can be viewed as agents which act for the protection of plants from undesirable biological agents with all their components in nanosizes and with potentially enhanced attributes associated with their component dimensions (Kah and Hofmann 2014). Through nanoprocessing, active ingredients of pesticidal mixes can be nnanoencapsulated for a slow and controlled release over a proportionately longer time period and can even provide a targeted delivery against deleterious phytopathogens such as *Megnaporthe grisea, Fusarium culmorum, Rhizoctonia solan, Botrytis cinereal, Scalerotinia sclerotiorum* and *Biploaris sorokinniana*, amongst several others (Chhipa 2016). In addition, these nanopesticides have a homogenous coverage and with their suspension stabilities, they reduce farm losses through reduced transportation to non-target areas and reduced photolysis and also improve bio-interactions through boosted uptakes (Kah et al. 2018). Nanopesticides have been made into preparations such as nanospheres, nanodispersions, nanoemulsions, nanosuspensions, nanometals, nanoclays, and nanocapsules with research and models such as nanodispersed triclosan, nanosphere insecticides, nanopermethrin, polymeric stabilized bifenthrin, and porous hollow Si-encaged validamycin with intensified capabilities including more rapid decomposition in soil, defense against untimely disintegration and higher pest

destruction at lower dosages (Kookana et al. 2014). Ingredients used in manufacturing conventional pesticides are lipophilic and have the huge limitation of minimal solubility. The nanoprocessing of active ingredients into nanoparticles presumably with the concurrent change in solid form will induce an increased solubility with an eventual increase in their bioavailabilities (Kah et al. 2013).

19.2.5 Nanofertilizers

Nanofertilizers have been illustrated as nanomaterials that can intensify plant growth and yield either directly by providing nutrient(s) to them or through indirect contribution by promoting the delivery of typically used fertilizers (Liu and Lal 2015). They have relatively higher dissolution rates and could be small enough to gain entrance into plant cells through their cell wall structures or probably through interactions with other cellular parts such as the cytoplasm and the membrane, though more studies are being undertaken to fully understand the schematics of such mechanisms (Nair et al. 2010; Liu and Lal 2015). Conventional fertilizers used for nitrogen supply are not usually fully biodelivered to the target plants and are lost by leaching and volatilization, causing environmental degradation through eutrophication issues for coastal and freshwater ecosystems with the formation of harmful macroalgal and cyanobacterial blooms (Conley et al. 2009; Kah et al. 2018). Nanofertilizers can be prepared either to supply micronutrients, micronutrients or have their matrices used as carriers for the productive biodelivery of nutrients to plants with the goals of curbing nutrient losses to the environment, improved nutrient delivery to plants during uptake, and a general minimization of unwanted changes to the environment in the distant future. Metals and metal oxides such as manganese, zinc, copper, molybdenum, copper oxide, iron oxide, and zinc oxide have been processed into nanofertilizers and applied in different concentrations ranging from 0.05 ppm to 2000 ppm for crops such as cucumber, soybean, pea, rye grass, chick pea and lettuce seeds to induce beneficial effects such as increase in chlorophyll content, root elongation, enhanced photosynthesis, and increased seed weight (Chhipa and Joshi 2016).

19.3 Nanotechnological Techniques for Processing Food Bioactive Materials and Products

Conventional technological processes such as cryoconcentration of solutions, conductive-convective drying of grain, electromagnetic intensifier extraction, thermal processing of viscous food products, energy-optimized drying, and low-temperature pasteurization with targeted decrease in energy consumption and process intensification have been improved with the innovative addition of

nanotechnology (Burdo 2005; Kehinde et al. 2019). The application of nanoprocessing in food systems can be viewed based on the position of the processed nanomaterials used as nano-inside and nano-outside where the internal usage is related to additives and formulation ingredients in general while the outside is associated with coatings and packaging (Ravichandran 2010) (Table 19.1). Unlike macroscopic attributes, the nanoscale biological, chemical, physical and chemical characteristics can be nanotechnologically adapted by altering the atomic and molecular configuration for a cheaper, sustainable, functional and precise design and development of novel food formulations (Ravichandran 2010). The nanoprocessing of bioactive constituents in food materials commonly involves their encapsulation (Fig. 19.1). This ensures the conservation of their original characteristics and their safe delivery to the site of action subsequent to ingestion. In addition, these nanoscale processing techniques work for both hydrophilic and lipophilic bioactive materials, thus making it possible to develop a wide range of functional foods by food processors to meet the dietary, nutritional, and health needs of the modern man (Sharma et al. 2019). Nanoencapsulation techniques can either take the top-down approach or the bottom-up design for the effective actuation of the processing targets. Top-down approach includes emulsification and emulsification-solvent operation while the bottom-up methodologies include supercritical fluid technique, inclusion complexation, nanoprecipitation, and coacervation. Procedural schematics applied for nanoprocessing of food have also been similarly classified based on their methods of preparative occurrence as top-down synthesis or separate (or ex situ) and the bottom up or joined synthesis. The ex situ mode involves the detached preparation of nanomaterials with a subsequent integration into the system of interest through size techniques involving reduction of the average particle sizes such as mechanical milling, microfluidization, extrusion, spray drying, etc. (Vasile 2018; Kehinde et al. 2021). The alternative joint synthesis involves an inclusive preparation of the nanomaterial(s) in their systems of interest using procedures such as precipitation, sol-gel refining, and chemical syntheses (Oliveira and Machado 2013; Kehinde et al. 2020a, b, c, d).

19.3.1 Nanoemulsion and Nanoemusification

They are emulsions with dimensions of the nanoscale, thermodynamically stable, distinct from microemulsions on the basis of particulate shape and size dispersed in the continuous phase and bearing the potentials to deliver bioactive components. Oil-in-water or water-in-oil nanoemulsions can be further complexed into emulsions of emulsions termed as double nanoemulsions such as water-in-oil-in-water or oil-in-water-in-oil, with the former having water droplets dispersed in a relatively bigger oil droplet which is subsequently dispersed in an aqueous continuous phase (Jafari 2017). Jaiswal et al. (2017) summarized their advantages as

Table 19.1 Effect of nanoprocessing of functional food ingredients

Nano preparation	Bioactive component	Carrier matrix	Nanomaterial characteristics	Study target	Results	Ref
Nanoliopsome	Fish gelatin hydrolysate	Nanoliposomal vesicles	With an initial peptide content of 0–10 mg/ml, average particle size in the range 134–621 nm, ζ-potential of 0.06–8.65 mv and a polydispersity index of 0.27–0.49	Examination of encapsulation efficiency (EE) and antioxidant activity of Liposomal nanovesicles	Antioxidant activity peptide fraction observed with a maximum EE (84.5%) at peptide concentration of 1 mg/ml. Effective hydrophobic interaction and hydrogen bonding between phosphatidylcholine and peptidic fraction.	Hosseini et al. (2017)
Nanoemulsion	Lipophilic bioactive compounds	Barley protein	Nanoencapsulates having sizes of 20–30 nm	Biodelivery of bioactive component in simulated in vivo model	Nano-encapsulations delivered and steadily released β-carotene to a simulated human intestinal tract intact	Wang et al. (2011)
Double nano-emulsions (W1/O/W2)	Gallic acid	Emulsifiers used include Polyglycerol polyricinoleate (PGPR), and pectin-WPC or/ Tween 80 and delivery systems are pectin, PGPR, soybean oil and deionized water	Size of W1/O emulsions was 15.1 and 17.4 nm for 5% and 4% PGPR and Particle size of double emulsions ranging from 100–200 nm.	Comparative examination of stability of prepared emulsions during long term storage	Z-potential of the double emulsions prepared with WPC-pectin resulted in a higher stability during the long-term storage	Gharehbeglou et al. (2019)

(continued)

Table 19.1 (continued)

Nano preparation	Bioactive component	Carrier matrix	Nanomaterial characteristics	Study target	Results	Ref
Nanoemulsion w/o	Gallic and ascorbic acid	Olive oil	Mean droplet diameter for ascorbic acid nano emulsion ranging from 201 to 401 nm, and for gallic acid nanoemulsion, 195–401 nm	Examination of nanoemulsion stability	Emulsion stability index for ascorbic acid nano emulsion ranging from 98.4 to 99.8% and for gallic acid nanoemulsion ranging from 98.4 to 100%	Katsouli et al. (2018)
Mesoporous silica nanoparticles	Cinnamon essential oil	Potato starch films	Film thickness of 25.93 ± 0.83 nm and tensile strength of 56.12 ± 1.39 MPa	Antimicrobial effect against post-harvest white mushrooms molds	Inhibition of (Agaricus bisporus) from CNRMA 03.0371 strain (Mucor circinelloides) and FJ09 species (Mucor sp.)	Zhang et al. (2019)
Nanocomplexes	Orange peel oil	Whey protein concentrate and pectin	Particle sizes at pH = 3, 6 and 9 were 360, 182 and 185 nm, respectively	To determine emulsion stability and encapsulation efficiency	At pH values of 3, 6, 9 EE was 88%, 84% and 70%, and Z-potential +1.7, −19, and − 30.3 mv respectively.	Ghasemi et al. (2017)
Nanoliposomes	Melatonin, phosphatidylcholine, and cholesterol	Supercritical carbon dioxide	Particle sizes ranging from 66.19 to 137.3 nm for carbon dioxide pressures of 10–22 MPa	Liposome stability during storage and against degradation	Stability constant of the liposomes found to be 0.068 and 0.091 after 1 and 2 months of storage respectively. Liposomes were found to be resistant to degradation in a simulated gastric environment.	Situ et al. (2017)

Nano preparation	Bioactive component	Carrier matrix	Nanomaterial characteristics	Study target	Results	Ref
Nano-complexes	Folic acid	7S and 11S globulins obtained from defeated soy flour	7S-FA (5.5–7.0) having a zeta average of 33.86 ± 1.4 d nm and 11S-FA (3.0–7.0) having 51.33 ± 1.3 d nm	Nanocomplex stability and folic acid release for biomass formation	7S-FA (5.5–7.0) and 11S-FA (3.0–7.0) having poly dispersity indices of 0.498 ± 0.36 and 0.851 ± 0.40 respectively. Both globulins showed higher growth for Lactobacillus casei BL23 relative to control treatment.	Ochnio et al. (2018)
Double W1/O/W2 nanoemulsion	Folic acid	Pectin and whey protein concentrate		Modeling and release profile of folic acid at different pH values	Folic acid nano-capsules made with Span as the surfactant had the lowest release rate in acidic conditions (pH = 4) and highest release in the alkaline conditions (pH = 11)	Assadpour et al. (2017)
Nanooligo-hyalurosomes nano-delivery system	Curcumin and resveratrol	Oligo-hyaluronic acid	Average particle size 134.5 ± 5.1 nm and Zeta potential −29.4 ± 1.2	Stability and controlled release of antioxidant on subjection to in vitro gastrointestinal conditions	Perfect stability and outstanding sustained release character with a dose-dependent antioxidant activity	Guo et al. (2018)

(continued)

Table 19.1 (continued)

Nano preparation	Bioactive component	Carrier matrix	Nanomaterial characteristics	Study target	Results	Ref
Nanoparticles	Catechin	Horse chestnut starch (HSC), lotus stem chestnut starch (LSC), and water chestnut starch (WSC)	Average particle size of HSC, WSC and LSC based nano-particles were 322.7, 559.2 and 615.6 nm	Release behaviour and bioactivity retention	Encapsulation efficiency of 59.09, 48.30, and 55.00% for HSC, WSC and LSC nanoparticles. Antidiabetic and antiobesity potentials were retained at in encapsulated catechin upon in-vitro digestion	Ahmad et al. (2019)

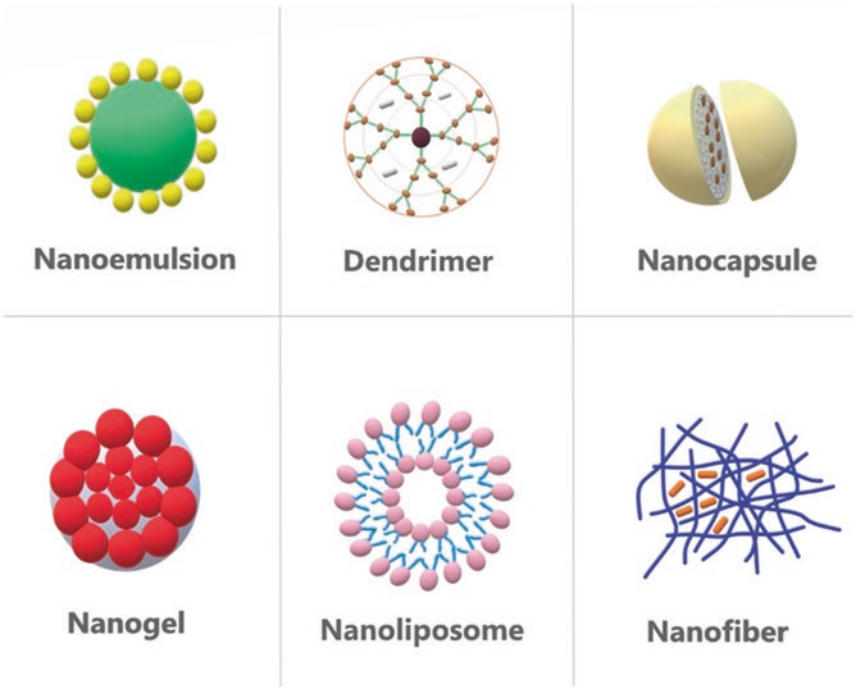

Fig. 19.1 Different nanocarriers for encapsulation of food bioactive compounds

1. Having enhanced physical stability and being processible into different formulations such as creams, foams, sprays and liquids
2. Bearing the potential to protect and deliver bioactive components to target sites
3. Adjustment of sensor qualities of formulations
4. Aiding the solubilization of lipophilic materials and functional as alternatives to vesicles and liposomes
5. Biocompatible with no toxicity or irritation

Moreover, immobilization of biologically functional food components in matrices of nanoemulsions has been reported to enhance their dispersibility in solutions of different phases and minimizing the possibility of separation, prevent deterioration peculiar to those bioactive materials and ensure their proper interaction with other food ingredients while conserving their functionality, and maintaining the organoleptic profile of the food products where employed (Donsì et al. 2011; Kehinde et al. 2020a). Food-based bioactive ingredients such as long-chain fatty acids (linolenic acid, linolenic acid, docosahexaenoic acid, and oleic acid), antioxidants (tocopherol, ascorbic acid, isoflavones, and other phenolics), antimicrobial peptides (lysozyme, casecidins and nisin), short-chain fatty acids, fat and water soluble vitamins, prebiotics and probiotics have been processed into nanoemulsions and found to exhibit superior attributes such as better physical stability and smaller droplet sizes relative to macroemulsions (Lohith Kumar and Sarkar 2017; Sosalagere et al. 2021).

More notably, their processing into nanoemulsions have been found to remarkably enhance their bioavailability and absorption as a result of their subcellular dimensions which enables their transportation across cell membranes on the basis of concentration gradient (Porter et al. 2008).

19.3.2 Electrospraying, Electrospinning, and Nanofibers

Electrospraying involves the electrical production of particles from a polymeric solution and atomized through a nozzle and electrospinning also involves a polymeric solution but requires a high voltage potential and a spinneret to produce nanofibers (Esfanjani and Jafari 2016). Though with similar principles of operation, these techniques vary on the basis of polymer concentration; when the polymeric solution is of low concentration, fine electrosprayed droplets are produced from a destabilized jet nozzle, at higher polymer concentration however, the spinneret is stabilized and electrospun nanofibers are formed (Esfanjani and Jafari 2016; Shishir et al. 2018). Important parameters to be considered in the elctrospinning process have been categorized as ambient conditions of atmospheric pressure, humidity and temperature, processing conditions such as needle diameter, separation length between the needle and collector, Volume feed rate, and Applied voltage, and the solution properties such as surface tension, elasticity, electrical conductivity, and viscosity (Ghorani and Tucker 2015). Based on their non-thermal nature, they are useful for the nanocarriage of heat labile bioactive components in food preparations and have been reported to be more efficient relative to spray drying (Pérez-Masiá et al. 2015; Shishir et al. 2018). Ghorani and Tucker (2015) outlined the boons of elctrospun nanoprocessing of food bioactives into nanofibers as

1. A more effective procedure to process protein-based bioactive components such as peptides, oligopeptides and polypeptides with a reduced tendency for denaturation
2. Occurring at room temperature, thus reliable for processing of heat-sensitive nutraceuticals
3. Alterable geometry of final product by simply changing the combination of processing parameters
4. Efficient encapsulation by electric force rather than mechanical which might create frictional heat
5. Sustained release of encapsulated bioactive component over time

19.3.3 Coacervation

Coacervation is a nanoprocessing methodology that encompasses the phase separation of a composite or singular polymeric solution through three peculiar phases viz.: preparation of immiscible phases in the course of mixing for continuous liquid phase, coating and core materials; synthesis of the encapsulating wall to circumvent the bioactive component; and a terminal solidification of the encapsulates by cross-linking, desolvation or heating (Bakry et al. 2016; Shishir et al. 2018). When only type of polymer is used, it is referred to as simple coacervation and in the case of more than one, it is termed as complex coacervation. Ezhilarasi et al. (2012) itemized factors affecting the characteristics of complex coacervates and the interaction of biopolymers as

1. Processing acidity or pH
2. The nature of the polymers on the basis of their charge, its flexibility and its molecular mass
3. Concentration of the polymers processed
4. The tendency of the polymers to dissociate into ions and interact together
5. The ratio of the polymers in the coacervate formulation
6. Hydrophilicity and hydrophobicity of polymers
7. Polarity of polymers and their tendency to form hydrogen bonds.

These factors have also been reported to affect the stability of the coacervates and are required to be optimized for proper their usage in food products. This is of importance because complex coacervation has been found to be more functional than spray drying or simple coacervation as a result of its low temperature requirement which makes it useful for heat labile bioactive components, more durable controlled release of encapsulated components, higher core loading capacity, and an overall protection of bioactive compounds (de Souza Simões et al. 2017). Functional food-borne ingredients such as anthocyanins, folic acid, β-carotene, antimicrobials and other oil-based materials have been nanoencapsulated in studies with desirable outcomes reported such as tolerance to simulated gastrointestinal digestion, enhanced bioavailabilities, improve stability against thermal degradation, and efficient loading capacities (Arroyo-Maya and McClements 2015; Shishir et al. 2018).

19.3.4 Nanoprecipitation

This technique is also referred to as solvent shifting, antisolvent precipitation, solvent displacement, and desolvation and it occurs in three phases viz.: nucleation, growth and nanoprecipitate aggregation (Aubry et al. 2009; Kehinde et al. 2020b). The particle nucleation takes place at high saturation beyond that of the critical limit for the polymer solution resulting in an interfacial breakage between the polymer and solvent, while the particle growth takes place through coagulation or

condensation of the particles to the core after an energy release which also forms the basis for their aggregation (Bareras-Urbina et al. 2015). The efficacy of a nanoprecipitation is related to parameters of the procedure used and polymer and involved such as such as interfacial tension, solubility, diffusion, agitation technique, drying technique and polymer concentration (Beck-Broichsitter et al. 2010). Ezhilarasi et al. (2012) and Bareras-Urbina et al. (2015) Relative advantages of nanoprecipitation in comparison with other nanoprocessing methodologies for food bioactive components include:

1. Higher bioavailability and cellular uptake from in vivo investigations
2. Not having a requirement for a precursor emulsion unlike other encapsulation techniques
3. Requires a single step and simpler facility setup to develop nanoparticles, thus requiring lesser energy and costs.
4. Though it requires water soluble solvents, it is methodical for the encapsulation of hydrophobic bioactive components.
5. Nanoprecipitated encapsulates have better encapsulation efficiency and better stability against degradation
6. Can be performed through various mechanisms such as flash nanoprecipitation (for reproducible and small particles), two-step nanoprecipitation (a remedy for constraint of choosing solvent of high potential solubility for bioactive ingredients) and ouzo-effect nanoprecipitation (versatile for different solutes).

For nanoprecipitation, biopolymers such as polysaccharides (starch and chitosan), polyesters (Polylactic acid), protein (Bovine serum albumin and gelatin) have been prepared as nanoparticles by nanoprecipitation and the process of their formulation depends on encapsulation efficiency and size variables which are also reliant on parameters such Organic/aqueous phase ratio, polymer concentration, Organic phase flow rate, and stirring rate (Rivas et al. 2017).

19.3.5 Nano-spray Drying

Spray drying basically involves the conversion of a liquid fluid into a dried powder through the use of a heated and drying medium and is a prominent technique for the encapsulation of hydrophobic and hydrophilic bioactive ingredients subsequent to an emulsification procedure (Assadpour and Jafari 2019). It is useful for the processing of different bioactive ingredients and food additives such as peptides, probiotics, colorants, flavors, minerals, vitamins, carotenoids, protein concentrates and isolates, and polyphenols, amongst several others based on the rapid nature of the process and the protection of heat sensitive materials (Arpagaus et al. 2017; Kaur et al. 2020). A typical spray drying process is fundamentally comprised of liquid feed atomization, sprayed droplet drying, and separation and collection of the dried product from the drying medium, with the use of food grade biopolymers such as proteins (albumin and collagen), carbohydrates (chitosan, lactose, dextrose,

maltodextrin, etc.), and gums (carrageenan, guar gum, and Arabic gum) as wall materials (Arpagaus et al. 2017). Arpagaus et al. (2017) summarized that the encapsulation operation by spray drying is achieved by emulsifying, dispersing or dissolving the core material in a solution matrix of the wall material and then spray-drying the mixture under optimally adjusted conditions of moisture content, particle morphology, drying gas inlet temperature, polymer concentration, solid concentration, spray mesh size, solvent type and concentration, surfactant type and concentration, and the drying gas flow rate. Assadpour and Jafari (2019) outlined some advantages of spray drying for encapsulation of food bioactives as

1. Workability with several experimental designs with an appreciable encapsulating efficiency
2. Occurs rapidly, thus requiring lesser energy and saving costs
3. Can be used for organic or aqueous solvent through closed cycle or open cycle designs
4. 4.Sticky foods are processible simply by the use of surfactants and drying enhancers
5. A simple operation process requiring less technical knowledge
6. Can be scaled-up to fit commercial demands

On the other hand, nano spray drying has been appraised as being advantageous over the traditional spray drying especially for nanoencapsulation needs due to the requirement of lesser sample quantity and the production of smaller-sized particles of remarkable bioavailability (Arpagaus et al. 2018; Kehinde et al. 2020c).

19.4 Effects of Nanoprocessing on Food Bioactives

19.4.1 Nanodelivery of Bioactive Such as Probiotics, Peptides and Polyunsaturated Fatty Acids (PUFAs)

Several food bioactive materials have their specific sites of actions where they maximally impart their functionalities (Kehinde and Sharma 2018; Sharma et al. 2021). For example, probiotics have the large intestine as their body site of action since the small intestine is too close to the stomach and only trace amounts of rod-shaped and Gram-positive bacteria are able to adapt there. A probiotic food has been described as one containing an adequate amount of probiotic microbes per serving and the eventual delivery of the microbes of such food to the large intestine in adequate amounts is the desirable target for producers and consumers. Nanoprocessing of foods have been studied to influence the adsorption, distribution, metabolism, and excretion (ADME) of their concomitant bioactive components (Card et al. 2011). Borel and Sabilov (2014) summarized the size charge and hydrophobicity characteristics of food-based nano-systems for optimal absorption, distribution, and excretion as:

1. For the absorption of the bioactive component which implies its interaction with the epithelial cells and gut mucosa, its size should be less than 100 nm, of neutral charge and of hydrophobic nature.
2. For an efficient distribution or biodistribution through bodily organs such as kidney, liver, spleen, brain, lungs and heart, an optimal systemic circulation would also require a size and charge of less than 100 nm and neutrality, but of hydrophilic nature.
3. For excretion, sizes of >500 nm for fecal matter and < 5 nm for urine will be optimal.

In addition, factors such as the surface chemistry of the nanodelivery system, and the physical, morphological and chemical attributes of the bioactive components have been understood to influence its location and time of release, as well as its gravity, method and rate of uptake (Sahay et al. 2010; Borel and Sabliov 2014).

19.4.2 Nanoencapsulation of Essential Fatty Acids, Vitamins, Phenolics and Other Antioxidants

Nanoencapsulation systems such as nanoemulsions, multiple nanoemulsions, nano-liposomes, solid lipid nanoparticles, nano-structured lipid carriers, complexed biopolymer nanoparticles have been used to encapsulate bioactive components and food additives for diverse reasons related to their eventual efficiencies. Assadpour and Jafari (2018) highlighted some mechanisms and benefits for these such as:

1. Improving the bioavailability of phenolic compounds and other antioxidants through the protection of such within food formulations and a subsequent enhancement of their permeation, absorption, and solubility in the body.
2. Nanosystems such as solid lipid nanoparticles, nano-liposomes, complexed biopolymer nanoparticles, and nano-structured lipid carriers (NLCs) offer environments of suitable hydrophobicity for the carriage and delivery of typical flavonoids such as quercetin in foods of low- or non-fat compositions.
3. Nanocarrier bodies have been studied to protect bioactive compounds against suboptimal conditions of temperature, oxygen, light exposure and pH.
4. Nanoemulsion encapsulation systems have the potentials to improve the physical and chemical stability of fish oils and also offer controlled release of essential fatty acids.
5. Food vitamins prone to degradation through handling and processing operations such as cooking and blanching and exposure to light can be nanoencapsulated for protection against such environmental conditions.

19.5 Conclusion

The rising demands for functional foods and effective processing methodologies have necessitated the birthing and global adoption and integration of nanotechnological procedures into food processing. Accordingly, these finesse techniques have shown formidable advantages especially in the conservation and delivery of functional properties of consumed foods. Nano-processed food materials have been studied to be effective carriers of one or more functional bioactive compounds in their matrices with an eventual delivery of such at their desired sites of action. Furthermore, processing considerations such as material and energy requirements, industrial floor spaces and more importantly the environmental impacts of industrial-scale manufacturing are being optimally designed and factored through nano-scale food processing. The conservation of the food and therapeutic attributes of functional food materials subjected to nano-processing techniques would make such procedures the soothing solution to the modern-day requirement of food processing.

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