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REVIEW PAPER

Spray Drying for the Production of Nutraceutical Ingredients—A Review

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Abstract Contributions of spray drying to food processing applications are increasing as compared to other conventional drying methods. Spray drying has not only contributed in drying of fluids but also has played a vital role in encapsulation and microencapsulation of valuable foods and functional–nutraceutical ingredients. Microencapsulation by spray drying is a cost-effective one-step process as compared to other encapsulation methods. Encapsulation using spray drying is mainly used in the food sector to protect bioactive compounds or functional foods from light, temperature, oxidation, etc. This paper reviews the work done in past years in the functional food and nutraceutical sector using spray drying. The paper focuses on the role of spray drying in vitamins, minerals, flavouring substances, antioxidant compounds and fatty acids encapsulation.

Keywords Spray drying · Nutraceutical · Encapsulation · Food ingredients

Introduction

The first spray dryers were manufactured in the USA in 1933 (Hayashi 1989). Spray drying is one of the best drying methods to convert, in a single step, fluid materials into solid or semi-solid particles. The evaporation takes place during the fluid feed contact with hot air. As the rapid evaporation keeps the droplets' temperature relatively low, the product quality is not significantly or negatively affected (Roustapour et al. 2009). However, spray drying has been frequently described as a harsh drying method due to its often high temperature of operation. Under high temperatures, thermolabile components such as vitamins and enzymes, among other food components, can be damaged or inactivated during the spray drying process (Yoshii et al. 2008).

Spray drying involves complex interactions of process, apparatus and feed parameters which all have an influence on the final product quality (Chegini et al. 2008). The spray drying process can produce good quality final product with low water activity and reduced weight resulting in easier storage and transportation. The physicochemical properties of the final product mainly depend on feed flow rate, particle size, viscosity, spray dryer inlet and outlet temperatures, pressure and type of atomizer (Tonon et al. 2008). Spray drying is often selected as it can process material very rapidly while providing relative control of the particle size distribution (Obón et al. 2009).

The schematic diagram of a spray dryer is shown in Fig. 1. Spray drying consists of four main stages, which include atomization of fluid feed, drying medium and spray contact, drying of feed and separation of product from air. These four phases and their operational parameters have major impact on the drying efficiency and the final product properties (Cal and Sollohub 2010). The fluid feed can be in the form of a solution, a paste or a suspension. The final product can be in the form of granules, powders and agglomerates, which mainly depends on the physicochemical properties of the feed and process operating parameters (Filkova et al. 2007; Masters 1985).

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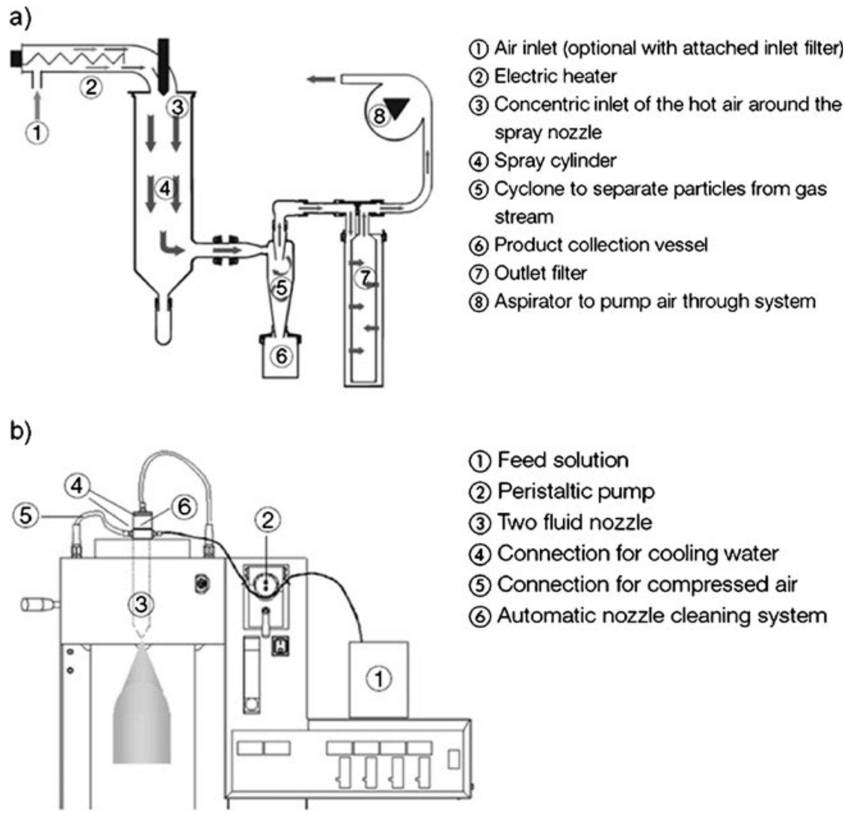

Fig. 1 A schematic diagram of a spray dryer. **a** air flow, **b** feed delivery (Buchi Corporation)

The atomization process generally refers to the formation of powder or liquid suspension in a gas as well as subsequent reduction in particle size (Cal and Sollohub 2010). An effective atomization process converts the fluid feed to tiny droplets with equal size which leads to uniform heat and mass transfer during the drying process. Due to the subsequent reduction in particle size and dispersion of the particles in the drying gas, the surface area of the particles increases exponentially. This increment in surface area of the particles helps to dry the feed in seconds. With the small size of droplets and the even distribution of the fluid feed, the moisture removal occurs without disturbing the integrity of the material. The atomization is achieved by atomizers which are generally classified as rotary atomizers, pressure nozzles, pneumatic nozzles and sonic nozzles (Cal and Sollohub 2010). Atomizers are classified based upon the type of energy which acts upon the bulk fluid. For example, rotary atomizers use centrifugal energy to atomize the feed, while pressure nozzles use a pressure buildup (Filkova et al. 2007; Masters 1985). Atomizers are selected based upon the feed which needs to be dried and targeted final properties of the dried product as well as the particle size.

During atomization, the feed is exposed to the drying gas. In most cases, the drying gas generally refers to atmospheric air. During the spray drying process, the atmospheric air is filtered through a filtering system and subsequently preheated according to the operating parameters. Sometimes, nitrogen or other inert gases are used based upon the feed being dried and its instability, or sensitivity to oxygen (Cal and Sollohub 2010). The inlet air temperature plays an important role in removal of moisture from the dispersion. Performance and efficacy of the spray drying process depend upon the humidity and temperature of the inlet air, while the air temperature is the controlled variable during the spray drying process (Baker 1997).

The fluid spray and air contact time are important components of spray drying as they determine the drying rate and the intensity of drying. The duration of stay of each droplet inside the drying chamber is determined by the spray's flow rate and size of drying chamber which governs the air contact time. Hence, it is important to design the drying chamber and air dispenser in such a way to create easy flow of the product to prevent deposition of partially dried product on the chamber wall and on the atomizer. The main reason for the wall deposit is the overly rapid travel of the droplets, which leads to a reduction of the contact time with the drying air which prevents adequate escape of moisture into the drying air. Generally, the spray and air contact patterns are classified as cocurrent, countercurrent and mixed flow (Cal and Sollohub 2010; Masters 1985).

The drying of feed droplets in a spray drying process is a result of simultaneous heat and mass transfer. The heat from the drying medium is transferred to droplets by convection and then converted to latent heat during the evaporation of the droplets' moisture content. The rate of heat and mass transfer depends upon the droplet diameter and the relative velocity of the air and droplets. The initial drying period starts in spray drying once the droplet comes in contact with the drying medium. This is followed by the falling rate period where the rate of drying begins to decrease, and the period ends once the droplets reach their critical moisture content (Filkova et al. 2007; Masters 1985).

Cocurrent spray dryers are most common and widely used dryers when compared to other systems (Zbicinski et al. 2002). Drying kinetics and particle behaviour in cocurrent systems are well unknown as compared to countercurrent or mixed flow systems. In cocurrent dryers, the atomizer and the drying gas stream inlet are placed in the upper part of the drying chamber (Fig. 1). The feed droplets travel in the same direction of the drying gas flow while losing moisture content. In countercurrent dryers, the air inlet is placed opposite to descending dispersion droplets. In other words, the drying gas is provided from the bottom of the drying chamber and the atomization occurs in the top of the drying chamber.

Countercurrent systems are less commonly used method, and relatively very few products are dried by using this method. This method of drying accounts for only 5% of all spray dryers used (Rahse and Dicoi 2001). The hydrodynamics of recirculation of continuous phase and particle agglomeration is complicated in countercurrent drying system. Due to this complicated flow, the drying phenomena in these systems are not well understood (Piatkowski and Zbicinski 2007).

In mixed-flow dryers, the feed is atomized upwards, in the direction of the top of the chamber, while the drying air inlet is placed in the upper part of the chamber; thus, the drying air passes countercurrent relative to the atomized feed. This method produces fair-sized particles. This is the most economical method which is used to dry thermostable products (Cal and Sollohub 2010).

Following the drying process, the dried particles fall towards the bottom of the drying chamber or travel along with the outgoing air. In most cases, the separation takes place in a cyclone or bag filter. Recovery of food powder during the spray drying process is often a challenge due to the amorphous nature of food materials which leads to a rubbery and sticky final product (Woo et al. 2009). Low-molecular-weight sugars and some organic acids are the major components present in fruits. Due to their low glass transition temperature and hygroscopic nature, the dried products become sticky (Jaya and Das 2009). The stickiness of particles during the spray drying can lead to many operational problems as well as be responsible for the formation of agglomerations inside the chamber which may reduce product recovery (Gianfrancesco et al. 2010).

The spray-dried final product is categorized into two major classes according to their physical characteristics. Generally, spray-dried products are classified into sticky or non-sticky products (Adhikari et al. 2004; Goula and Adamopoulos 2005). Normally, non-sticky materials can be dried over a wide range of operating conditions during spray drying as the powder is easily recovered. Maltodextrins, gums and other hydrocolloids, and proteins are categorized under the non-sticky product category. Sugar and acid-rich natural fruit and vegetable juices, honey, etc. belong to the sticky product category which require special operating conditions to reduce the quantity of unrecoverable powder which sticks to the walls of the drying chamber (Adhikari et al. 2004).

Spray drying is the most often used encapsulation technique by the food industry (Ré 1998; Reineccius 2004) and one of the oldest methods, since the 1930s, to encapsulate flavours (Shahidi and Han 1993). As compared to all microencapsulation techniques and methods, the spray drying process is relatively inexpensive, straightforward, and well established. The one disadvantage of spray drying microencapsulation is limited availability and high cost of the wall or encapsulating materials (Gouin 2004; Rosenberg et al. 1990).

Adequate levels of dietary sourced micronutrients are important to keep the body in healthy condition. Inadequate supply of micronutrients has led to many chronic disorders and diseases (Ames 2001). Most food nutrients like vitamins and minerals are damaged or denatured during produce handling and subsequent processing. So, it is important to preserve these nutrients in the foods supplied to consumers, and this is where spray drying encapsulation can play an important role.

Microencapsulation of essential nutrients using spray drying is increasing in popularity with its ease of operation and cost-effectiveness. Extensive research has been done on the microencapsulation of functional food components, and an overview is presented in this paper.

Encapsulating Methods and Materials

Different methods are available for encapsulation purposes. Those methods can be classified into three major categories, namely physical, chemical and physicochemical as listed in Table 1 (Shahidi and Han 1993). The selection of microencapsulation method depends upon specific application and parameters such as required particle size, physicochemical properties of the core and coating materials, release mechanisms, process cost, etc. (Ré 1998; Shahidi and Han 1993). Hence, it is important to select an appropriate encapsulation method for better process efficiency which can produce high-quality microcapsulated products.

Capsules produced by different encapsulation processes are classified according to their size. Microcapsules usually

Table 1 Microencapsulation methods (Ré 1998)

Category	Methods
(1) Physicochemical processes	(1.1) Simple or complex coacervation (aqueous phase separation)
	(1.2) Emulsion–solvent evaporation (organic phase separation)
	(1.3) Emulsion–solidification
	(1.4) Liposome entrapment (liposome casing)
(2) Chemical processes	(2.1) Interfacial polymerisation (chain organization in solution)
	(2.2) Molecular inclusion (chemically bond structures)
(3) Physical processes	(3.1) Spray drying (atomization)
	(3.2) Spray coating (sprayed material onto a surface)
	(3.3) Prilling (melted material dripped to solidify in midair)
	(3.4) Extrusion, etc.

have a size between 0.2 and 5,000 μm, and macrocapsules are bigger than 5,000 μm. Capsules smaller than 0.2 μm are referred to as nanocapsules (Balassa et al. 1971; Baker 1987).

Spray drying can effectively be used in the microencapsulation of vitamins, minerals, flavour compounds and antioxidants by using a specific and appropriate carrier or encapsulating material. With careful selection of the carriers or encapsulating materials, selective diffusion can be achieved to control the release of the encapsulated core product or compound (Chiou and Langrish 2007).

The microencapsulation by spray drying consists of two major steps. The preparation of the dispersion or emulsion which needs to be processed is the first step in spray drying encapsulation followed by subsequent spray drying of the emulsion. Preparation of the suspended solids matrix solution is a very important step in the encapsulation process. This matrix is selected according to the microcapsules' final application. The active core material which needs to be encapsulated is added to the wall material solution followed by adequate mixing. Then, the core and wall material solution is fed into the spray dryer where it is subjected to four major phases of the spray drying process. The successful microencapsulation mainly depends upon high retention of core material during processing as well as during storage.

Different encapsulating materials are used to enclose/encapsulate different core materials. Every encapsulation material has different encapsulating properties and release characteristics of the core materials. Hence, the selection of encapsulation materials for each core product is an important step in the successful encapsulation spray drying process (Kim and Morr 1996). However, the release rate of core materials depends upon various additional parameters which are listed in Table 2.

Encapsulating materials, used in the food industry, are generally considered as biomolecules. The biomolecules are usually derived from various origins such as plants, animals, microbial, etc. These biomolecules are classified into three major categories, i.e. carbohydrate polymers, proteins and lipids. Carbohydrate polymers are the most abundantly used encapsulating materials when compared to proteins and lipids. An overview of various encapsulating materials and their origins is given in Table 3.

Carbohydrate polymers are further classified into five subcategories, i.e. starch derivatives, cellulose derivatives, plant exudates and extracts, marine extracts and microbial and animal polysaccharides. Generally used encapsulating materials such as maltodextrins and gum arabic belong to the polysaccharide category (Wandrey et al. 2010). Carbohydrate polymers such as starches are modified chemically, biochemically and physically to produce specific purpose encapsulating materials.

Many functional derivatives of starch such as cross-linked, oxidized, acetylated, hydroxypropylated and partially hydrolyzed molecules are available in the market, providing an array of techno-functional properties. Malltodextrins are the best example in this class and are manufactured by starch hydrolysis. Hydrolysis of starch is performed by chemical or biochemical process. According to the degree of hydrolysis, the derivatives are assigned a dextrose equivalent value (DE). The higher the DE value is, the shorter the glucose chain and the higher the sweetness and solubility.

Table 2 Parameters affecting the release rate of core materials (Shahidi and Han 1993)

Coating properties	Density, crystallinity, orientation, Solubility, plasticizer level, cross-linking, Pretreatments
Capsule properties	Size, wall thickness, configuration, Conformity, coating layers, posttreatment
Environmental conditions	Temperature, pH, moisture, solvent type, Mechanical action, partial pressure Differential (inside and outside of coating)

Table 3 Microencapsulating materials overview and their sources (Wandrey et al. 2010)

Origin	Carbohydrate polymer	Protein	Lipid
Plant	Starch	Gluten (corn)	Fatty acids/alcohols
	– Derivatives	Isolates (pea, soy)	Glycerides
	Cellulose		Waxes
	– Derivatives		Phospholipids
	Plant exudates		
	– Gum arabic		
	– Gum karaya		
	– Mesquite gum		
	Plant extracts		
	– Galactomannans		
	– Soluble soybean		
	Polysaccharides		
Marine	Carrageenan		
	Alginate		
Microbial/animal	Xanthan	Caseins	Fatty acids/alcohols
	Gellan	Whey proteins	Glycerides
	Dextran	Gelatin	Waxes
	Chitosan		Phospholipids (Shellac)

Spray Drying in Nutraceutical Applications

Vitamins

Vitamins are important micro nutritional substances which are involved in many biochemical functions in the body. Vitamins cannot be synthesized by the body; hence, they have to be supplied through diet. Insufficient supply of vitamins can lead to many deficiency diseases like scurvy, pellagra, ariboflavinosis, dermatitis, enteritis, etc. Use of multivitamin supplements has indicated a reduction in cases of certain diet-related disorders (Pocobelli et al. 2009).

Vitamins are completely or partially denatured or damaged during the cooking and processing of foods. Vitamin C, folate and vitamin B6 seem to be less stable during high-temperature processing as compared to retinol, thiamine, riboflavin and niacin. Loss of vitamins also occurs during chilling, heating, reheating and storage (Williams 1996). The losses of micronutrients are mainly due to excess use of heat, moisture changes, excessive trimming, cutting, chopping, slicing, washing, soaking, etc. (Lawson et al. 1983). Hence, it is important to take precautionary measures to preserve the vitamins during the subsequent processing steps in the preparation of quality foods.

Micro- and nanoencapsulation are best to preserve vitamins. There are many different methods available for the effective encapsulation of compounds such as spray drying, extrusion, pan coating, etc.; however, since vitamins are thermolabile, they may get damaged during the drying and encapsulation process. The efficacy of three different processes was studied by Desobry et al. (1997) and evaluated in terms of retention of β-carotene (vitamin A). Spray drying, drum drying and freeze drying were considered for the experiment. Pure β-carotene was encapsulated using maltodextrin (25 dextrose equivalent). The inlet temperature of the spray dryer was set to 150 °C and outlet temperature was maintained at 100 °C. Encapsulated β-carotene stabilization was measured at different temperature and relative humidity (RH) for each drying process. During storage, RH was found to have no effect on encapsulated β-carotene. The least degradation of β-carotene was observed in freeze drying (around 8%), followed by spray drying (11%) and drum drying (14%; Desobry et al. 1997).

Vitamin C not only acts as a dietary supplement but also possesses valuable antioxidant properties. It has been accepted and recognized by the US Food and Drug Administration as one of four major dietary antioxidants along with vitamin E, the vitamin A antecedent ß-carotene and selenium (Carr and Frei 1999). Lack of a sufficient supply of vitamin C leads to diseases like scurvy (Levine 1986), while an adequate intake of vitamin C can be beneficial against many chronic diseases (Gey 1998; Weber et al. 1996).

Ascorbic acid losses begin with harvesting and continue through storage (Erdman and Klein 1982). Vitamin C encapsulation was investigated by Esposito et al. (2002) with the assistance of methacrylate copolymers in drugs. The results showed that methacrylate is effective in encapsulating vitamin C. The encapsulation efficiency was around 98% to 100% with good morphology and size distribution, but it showed poor controlled release of vitamin C. A similar research was conducted with different encapsulating materials by Desai and Park (2005) for oral intake. They used chitosan as the encapsulating material with the cross-linking agent tripolyphosphate for spray drying encapsulation of vitamin C. Inlet temperature was set to 175 °C, and flow rate was maintained at 3 mL/min. The final product was evaluated in terms of loading efficiency, surface morphology, particle size and distribution. The encapsulating efficiency was found to be around 45% to 55% with particle size of 6.1–9 μm.

Ascorbic acid encapsulation process parameters were analysed with different methods by (Uddin et al. 2001). Thermal phase separation, melt dispersion, solvent evaporation and spray drying were considered. The studied spray drying parameters were inlet and outlet temperatures kept at 200–300 °C and 70–95 °C, respectively. The loss of ascorbic acid during encapsulation was found to be minimum in spray drying (less than 2%) as compared to the other methods tested; however, the encapsulation percentage was less than 50%. Use of starch and β-cyclodextrin as encapsulating materials in spray drying delayed the degradation of ascorbic acid during storage and showed improved results over unencapsulated ascorbic acid. No colour change was observed in encapsulated ascorbic acid even after exposure to air for 1 month (Uddin et al. 2001).

Cactus pear (*Opuntia streptacantha*) juice was encapsulated using commercial maltodextrin with dextrose equivalents of 10 and 20 (Rodríguez-Hernández et al. 2005). Two different inlet temperatures of 205 °C and 225 °C were used in the spray drying experiment. The final powder was analysed in terms of retention of vitamin C and other physicochemical properties. Maltodextrin with dextrose equivalent of 10 showed better results than maltodextrin with dextrose equivalent of 20. The maltodextrin (DE 10) showed higher vitamin C retention at spray drying pressure and temperature combinations of 0.1 MPa and 205 °C (Rodríguez-Hernández et al. 2005).

Interestingly, ascorbic acid encapsulation was also carried out using pea protein by Pierucci et al. (2006). The encapsulation efficiency and size distribution were evaluated. The results were compared with encapsulation using carboxymethylcellulose and blends with maltodextrin. Pea protein encapsulation produced rough morphological particles as compared to carboxymethylcellulose and blends of maltodextrin. However, the ascorbic acid retention was higher in cases using pea protein as the encapsulating material (Pierucci et al. 2006).

Similar work has been carried out with other encapsulation materials by Trindade and Grosso (2000). Gum arabic and rice starch, along with gelatin, were used as encapsulating materials in this experiment. The inlet and outlet temperatures of the spray drying were maintained at 150 °C and 80 °C, respectively. The feed flow rate and air pressure was 15 mL/min and 49.03 N/cm^2, respectively. Gum arabic-encapsulated ascorbic acid seemed to be more stable and produced better morphology as compared to rice starch-encapsulated ascorbic acid. The encapsulated material was stored at 45 °C, 60–65% RH and 90% RH. The gum arabic-encapsulated ascorbic acid showed higher retention even in higher relative humidity as compared to the rice starch-encapsulated one (Trindade and Grosso 2000).

Cashew apple juice was encapsulated using maltodextrin (DE 10) and cashew apple tree gum by Oliveira et al. (2009). The ascorbic acid retention of the powder was measured. The spray dryer inlet and outlet temperatures were maintained at 185 °C and 90 °C. The feed flow rate and air flow rate was 840 mL/h and 3.75×10^4 L/h, respectively. The ascorbic acid retention was found to be around 95% as the encapsulation material to juice ratio was 5:1. The ascorbic acid in cashew apple juice was successfully protected by the spray drying process (Oliveira et al. 2009).

Arachidonic acid with ascorbic acid was successfully encapsulated, and its oxidative resistance during storage was studied by Watanabe et al. (2004). Gum arabic, maltodextrin and soluble soybean polysaccharides were used as encapsulating materials. The spray drier inlet and outlet temperatures were 200 °C and 100–110 °C. Gum arabic and soluble polysaccharide encapsulates had good oxidative stability (Watanabe et al. 2004).

Minerals

Iron, iodine, potassium, sodium, phosphorus, magnesium and calcium are the main dietary minerals. Mineral deficiency can lead to many disorders and diseases like anaemia, hypokalemia, hypochloremia, hyponatremia, etc. The best method to overcome these mineral deficiencies is to fortify our food with a stable supply of these essential minerals. Mineral-fortified foods can help ensure adequate nutrition to consumers. The bioavailability of these mineral substances mainly depends upon the absorption mechanisms, reactivity with other substances and proper release of materials. Microencapsulation should ensure the reduction of mineral substance reaction with other ingredients and appropriate release and dietary uptake of necessary minerals (Oneda and Ré 2003).

Calcium micro particles were successfully encapsulated by spray drying with the help of derivatives of cellulose and neutral polymethacrylate by Oneda and Ré (2003). The spray dryer was set at inlet air temperature 120–128 °C, outlet air temperature 91–95 °C and a flow rate of 3 mL/min. The encapsulated final product was evaluated for its calcium content, size distribution, morphology and release profiles. The results showed that the final product properties vary with the polymer concentration and its type (Oneda and Ré 2003).

Microencapsulation efficiency of sodium caseinate with different dextrose equivalent carbohydrates was investigated by Hogan et al. (2001). The results showed that the encapsulation efficiency increased with an increase in dextrose equivalent but decreased with the higher core to encapsulating materials ratio (Hogan et al. 2001).

Antioxidants' Colours

Food drying processes usually lead to colour deterioration. This decolouration of foods is principally due to pigment degradation, Maillard reactions, oxidation, etc. These adverse reactions depend upon drying parameters such as temperature, air velocity, etc. (Aversa et al. 2009). For example, Heras-Ramírez et al. (2011) found that blanching of apples increased the retention of colour, total polyphenolic content and total flavonoid content when compared with fresh unblanched pomace. Temperatures 50 °C, 60 °C, 70 °C and 80 °C were used as drying temperatures in cabinet drying with air velocity of 3 m/s (Heras-Ramírez et al. 2011). However, drying process significantly reduced the polyphenol and flavonoid content with increasing process temperature, which leads to the reduction of total antioxidant activity.

Lycopene is responsible for the red/orange colour familiar to tomatoes and has many established health benefits. Lycopene intake can help in the prevention of cardiovascular diseases and certain cancers (Clinton 1998). Lycopene is a naturally occurring carotenoid, which has the highest physical quench rate constant with singlet oxygen, and it has a higher plasma level as compared to β-carotene (Di Mascio et al. 1989). Lycopene was encapsulated using gelatin and sucrose, and the results showed that encapsulation yield and encapsulation efficiency were affected by the gelatin to sucrose ratio, homogenization pressure, spray drying inlet temperature, feed temperature, lycopene to encapsulating material ratio and lycopene purity. The optimum results were found at the feed temperature of 55 °C, air inlet temperature of 190 °C, homogenization pressure of 40 MPa, a gelatin to sucrose ratio of 3:7 and a lycopene to encapsulating material ratio of 1:4 with a lycopene purity of not less than 52% (Shu et al. 2006).

Anthocyanins are known as natural antioxidants and belong to the flavonoid family, and are widely dispersed in flowers, fruits and vegetables (Wang et al. 1997). Anthocyanin pigments from black carrot (*Daucus carota* L.) juice were microencapsulated by spray drying. Maltodextrins with different dextrose equivalents were used in the experiment as encapsulating materials. The tested inlet temperatures were 200 °C, 180 °C and 160 °C, and the outlet temperatures were 131 °C, 118 °C and 107 °C. Maltodextrin with dextrose equivalent of 20–21 retained higher anthocyanin content, while higher temperatures triggered the anthocyanin loss. During storage of encapsulated anthocyanin at 25 °C and 4 °C with maltodextrin (DE 20–21), the higher storage temperature (25 °C) led to higher anthocyanin loss (from 600 mg/100 g to 400 mg/100 g approximately) as compared to the lower temperature (4 °C) with minimal loss (from 600 mg/100 g to 500 mg/100 g approximately; Ersus and Yurdagel 2007).

Watermelon juice with 3% and 5% maltodextrin was spray dried at different inlet temperatures of 145 °C, 155 °C, 165 °C and 175 °C in a study conducted by Quek et al. (2007). The maltodextrin used in the experiment had 8–11 dextrose equivalents. Lycopene and β-carotene contents of the spray-dried powder were determined and compared with raw fruits' lycopene and β-carotene contents. The results showed that the increase in inlet temperature decreased the lycopene and β-carotene contents. β-carotene was found to be more sensitive to the temperature than lycopene. The β-carotene loss was around 27% as compared to a 24% loss for lycopene with same inlet temperature (Quek et al. 2007).

In an experiment conducted by Robert et al. (2003) for the carotenoid encapsulation from rosa mosqueta (*Rosa rubiginosa*), gelatin showed better results as compared to starch in terms of protection of the carotenoid pigments. *Trans*-β-carotene recovery was around 99% in the case of encapsulation with gelatin, but with starch, it was only around 71%. The trend continued in *trans*-lycopene recovery with 72% recovery in gelatin encapsulation and 54% recovered in starch encapsulation. In the case of *trans*-rubixanthin, there was no difference in recovery between starch and gelatin encapsulation (Robert et al. 2003).

The encapsulation of bixin, a pro-carotenoid found in annatto (*Bixa orellana* L.) was studied using spray drying with the help of different encapsulating materials such as gum arabic and maltodextrin (Barbosa et al. 2005). The air inlet and outlet temperatures were maintained at 180 °C and 130 °C with an inlet pressure of 49.03 N/cm^2. The results indicated that bixin encapsulation with gum arabic can be three to four times more stable as compared to maltodextrin (Barbosa et al. 2005).

Bayberries (*Myrica rubra* Sieb. et Zucc.) juice was spray dried and micro encapsulated using maltodextrin (DE 12 and 19), with the inlet and outlet temperature ranges of 140–160 °C and 65–85 °C (Gong et al. 2008). The juice powder quality indicated the importance that the inlet temperature played in the colour of the final product (Gong et al. 2008). Red–purple food colourant from *Opuntia stricta* fruits was produced by spray drying (Obón et al. 2009). Liquid glucose (DE 29) was used as the drying fluid. The inlet temperature of the spray dryer was maintained at 160 °C, and feed flow rate was 0.72 L/h. More than 98% of colour was retained with the recovery of 58% using 20% (*v*/*v*) juice with 10% (*w*/*v*) glucose syrup (Obón et al. 2009).

Still today, very limited research has been done to use spray drying to produce natural food colours from food materials such as fruits and vegetables. Extensive research is needed to explore the preservation of natural colourants from agricultural materials using spray drying and combinations of encapsulating matrices. In their spray drying of cactus pear (*Opuntia ficus indica*) with maltodextrin or inulin, Saenz et al. (2009) pointed out to the combined colour and antioxidant potential of their produced cactus pear powder.

Flavours

More than 90% of the flavours available in the market are produced or encapsulated by spray drying. Oleoresins which are good antioxidants (Singh et al. 2005) are the substances responsible for most of the spice flavours. They are very reactive and unstable in light, temperature and oxygen. Their sensitivity can be overcome by effective encapsulation. Gum arabic, maltodextrin and modified starch have been used as encapsulating materials to encapsulate cardamom oleoresins (Krishnan et al. 2005). Results have showed that 4/6, 1/6, 1/6 proportion blends of gum arabic, maltodextrin and modified starch can effectively encapsulate material as compared to gum arabic (100%) alone; however, gum arabic was found to be the best encapsulating material as compared to other encapsulating materials when all are used alone (Krishnan et al. 2005). The inlet and outlet temperatures of the spray dryer were maintained at 178±2 °C and 120±5 °C. Similar work was carried out on pepper oleoresins by Shaikh et al. (2006). Gum arabic offered the best encapsulation results as compared to the other encapsulating material tested, namely modified starch. During the spray drying experiment, the inlet and outlet temperatures were maintained at 178±2 °C and 110±5 °C, and flow rate was maintained at 300 mL/h (Shaikh et al. 2006).

Oleoresin of rosa mosqueta (*R. rubiginosa*) was encapsulated using gelatin or starch by Robert et al. (2003). The encapsulated oleoresin was investigated after storage at different temperatures such as 25 °C, 40 °C and 55 °C with absence of light. The inlet temperature for starch was maintained at 150±5 °C, and for gelatin, it was 100±5 °C. The outlet temperatures were 70±5 °C and 65±5 °C, respectively. Gelatin and starch both provided the same carotenoid pigment degradation rate; however, gelatin showed better stability for trans-β-carotene (Robert et al. 2003).

Encapsulation of I-menthol using modified starch and gum arabic was studied by Soottitantawat et al. (2005b). The spray drying parameters set the inlet temperature at 180 °C, with corresponding outlet temperature of 100±5 °C, feed rate of 45 mL/min and air flow rate of 100 kg/h. The results confirmed that the increase in encapsulating material concentration, i.e. modified starch or gum arabic, increased the retention of menthol. The highest I-menthol retention was found at the mass ratio of I-menthol/encapsulating materials of 1:9. At this ratio, gum arabic showed around 72% I-menthol retention, while modified starch showed a maximum retention of 87% (Soottitantawat et al. 2005b).

Encapsulation of coffee extracts with cashew gum and gum arabic was studied by Rodrigues and Grosso (2008). The cashew gum (*Anacardium occidentale* L.) from Brazil was used in the experiment to study aroma protection, external morphology and size distribution. No significant difference was found between gum arabic and cashew gum, suggesting that cashew gum can be used as an alternative for gum arabic in encapsulation applications (Rodrigues and Grosso 2008).

Sumac berries extract was spray dried at an inlet temperature of 200 °C, outlet temperature of 100 °C and feed rate of 125 mL/h. The flavour retention in the spray-dried powder was examined using different carriers, namely sodium chloride, sucrose, glucose and starch. Among the carriers studied, sodium chloride was found to be the best carrier for encapsulation of flavour from sumac berries as the other carriers caramelized, making them unsuitable. The carrier to core material, i.e. the sumac flavour extract, was fixed to 4:1 g/g for all samples. The solution was spray dried with different concentrations of sumac berries extract with soluble solids content starting from 5%, 10%, 15%, 20% and 25%. It was found that the solid concentration played an important role in flavour retention, and higher soluble solids content produced higher flavour retention (Bayram et al. 2005).

D-limonene was encapsulated by spray drying with gum arabic, maltodextrin and modified starch as encapsulating materials. A spray drying inlet temperature of 200 °C, outlet temperature of 110±10 °C and feed flow rate of 45 mL/min were maintained during the experiment. The D-limonene retention in the encapsulated powder was affected by emulsion size and powder size. Larger powder size showed higher stability and best retention of flavour (Soottitantawat et al. 2005a).

Citrus peel oil is an important flavouring substance in many industries which include food, pharmaceutical, chemical and cosmetic manufacturing. This oil comprises highly volatile and nonvolatile components like terpene, oxygenated hydrocarbons, pigments and waxes. The oxygenated parts which include alcohols, aldehydes and ketones contribute more towards flavouring as compared to the terpenes (Stuart et al. 2001). Orange peel oil was encapsulated with maltodextrin and mesquite gum mixtures used in 1:4, 2:3, 3:2 and 4:1 (*w*/*w*) ratios in solution (30%). The inlet and outlet temperatures were set at 200±5 °C and 110±5 °C. The highest encapsulation percentage of orange peel oil (84%) was found at 3:2 ratio of maltodextrin to mesquite gum (Beristain et al. 1999).

Cardamom essential oil was also encapsulated using mesquite gum. The spray drying inlet and outlet temperatures were maintained at 200±5 °C and 110±5 °C. The gum was added to the essential oil in the proportion of 5:1, 4:1 and 3:1. The highest volatile retention was found at a 4:1 ratio (Beristain et al. 2001).

Caraway (*Carum carvi* L.) essential oil was encapsulated by spray drying. Whey protein concentrate, skimmed milk powder and waxy corn starch were used in combination with maltodextrin as encapsulating materials in the experiment. The inlet and outlet temperatures of 180±5 °C and 90±5 °C were used in the spray drying process. The combination of whey protein concentrate+maltodextrin/waxy maize starch (9:1) gave higher encapsulation efficiency of 85.88%, with highest total oil retention of 87.85% (Bylaitë et al. 2001).

The encapsulation of essential oil of oregano (*Origanum vulgare* L.), aroma extracts of citronella (*Cymbopogon nardus* G.) and sweet marjoram (*Majorana hortensis* L.) was studied with different encapsulating materials, namely, whey protein concentrate and skimmed milk powder. The spray dryer was operated with an inlet air temperature of 190±5 °C and an outlet air temperature of 90±5 °C. Skimmed milk powder showed better results as compared to whey protein concentrate in terms of encapsulation efficiency and total oil recovery. The encapsulation efficiencies with skim milk powderof oregano essential oil, citronella aroma extract and marjoram aroma extract were 80.2%, 69.4% and 67.9% as compared to whey protein concentrate with encapsulation efficiencies of 71.8%, 65.8% and 54.3%. The total oil content in the encapsulating material found in skim milk powder were 81.3%, 80.6% and 84.7%, while in whey protein concentrate, the oil contents were 73.2%, 81.0% and 76.4% (Baranauskiene et al. 2006).

Applications of spray drying in the production of flavours are well developed as they have wide applications in the food industry. Fewer applications have been studied for nutraceutical ingredients. However, with the increasing demand from consumers for healthier-for-you processed foods, novel spray drying applications are being studied and developed in this particular sector.

Essential Fatty Acids

Essential fatty acids like *n*–6 fatty acids and *n*–3 fatty acids are important for normal growth and development. The proper and adequate nutritional intake of fatty acids prevents many diseases such as coronary artery disease, hypertension, diabetes, arthritis, etc. (Simopoulos 1991, 1999). To ensure availability of the right fatty acids, developments are taking place for their use as food ingredients for food fortification.

Avocados are a rich source of many phytochemicals and fatty acids. They are rich in monosaturated fatty acids, vitamin E, lutein, glutathione, β-sitosterol, folate, potassium, magnesium and fibre (Duester 2000). The consumption of these nutrients may help in the prevention of cardiovascular disease and cancer (Rainey et al. 1994). Avocado oil was encapsulated with whey protein isolate and maltodextrin (dextrose equivalent of 5). The whey protein to maltodextrin ratio of 1:9 gave higher microencapsulation efficiency of around 66.18±3.82%. Since avocado oil is very stable in ambient and refrigerated conditions, no significant difference was found in terms of oil stability due to the encapsulation (Bae and Lee 2008).

Fish oil is rich in many nutrients, especially omega-3 fatty acids. Consumption of fish oil has proven to help reduce several risk factors of cardiovascular disease (Nestel 2000) and to lower blood pressure (Knapp and FitzGerald 1989). Fish oil was encapsulated using alginate and starch blends by Tan et al. (2009). The results were interpreted by researchers in terms of microsphere morphology, yield and microencapsulation efficiency. The spray drying inlet and outlet temperatures were 150 °C and 80 °C, respectively. The use of alginate as encapsulating material increased the oil-holding capacity and decreased the oxidation of fish oil as compared to unencapsulated fish oil (Tan et al. 2009).

Similar work was carried out on fish oil which had 33% omega *n*–3 fatty acids with different encapsulating materials. Sugar beet pectin and glucose syrup with dextrose equivalent of 38 were used as encapsulating materials in this experiment. The sugar beet pectin used in the experiment was found to have acetylation of 18% and esterification of 58% with a 5% protein content. The results indicated that a 1–2% pectin content is sufficient to produce a stable emulsion during spray drying. Sugar beet pectin is recommended as a replacement for gum arabic in the encapsulation of lipophilic materials (Drusch 2007).

Short-chain fatty acids were successfully encapsulated using gum arabic and maltodextrin. The inlet and outlet temperatures used in the spray drying process were 180 °C and 90 °C. Five per cent gum arabic alone, 10% maltodextrin alone and combinations of 5% gum arabic and 5% maltodextrin were tested. The results showed that the size distribution and structure of the encapsulated substance were affected by the type and concentration of encapsulating materials used. The encapsulating material combination of 5% gum arabic and 5% maltodextrin produced a smooth surface and homogeneous size distribution (Teixeira et al. 2004).

The oxidation of encapsulated linoleic acid was studied and investigated. Gum arabic and maltodextrin were used as encapsulation materials. The inlet and outlet temperatures of spray drying were maintained at 200 °C and 100±10 °C. The flow rate of feed and air was 3 kg/h and 7.5 m^3/min, respectively. Gum arabic produced a better material than maltodextrin in terms of size, stability and oxidation resistance. The encapsulation efficiency of the gum arabic was found to be higher as compared to maltodextrin (Minemoto et al. 2002).

Conclusion

Spray drying is an important processing technology used in many functional food and nutraceutical applications. Spray drying is used to produce a large number of dried and encapsulated ingredients such as vitamins, minerals, flavours, etc.; however, the contribution of spray drying to the production of antioxidant-rich food products or incorporation of antioxidants in foods by encapsulation is still minimal when compared to other technologies, principally based on the important initial investment cost of the technology. Further limitation is explained by the high inlet temperature of spray drying which is not suitable for thermosensitive materials. However, the optimization of spray drying process, at lower temperature, for preserving antioxidant substances such as lycopene, anthocyanins and β-carotene has been possible as reported by some researchers (Ersus and Yurdagel 2007; Quek et al. 2007; Shu et al. 2006). Further application development could strongly establish spray drying as an effective method to preserve functional and nutraceutical properties of particular food materials.

More research is required to explore the possibility to preserve or microencapsulate many different sources of antioxidants such as polyphenols, flavonoids, etc., playing with process parameters such as inlet and outlet temperatures, air flow and feed rates, nozzle design and complex encapsulating materials. The research should be done for effective preservation of functional components and exploration of efficacy of various encapsulating materials other than the normally used encapsulating materials. This approach would avoid the bottlenecks in availability of certain encapsulating materials such as gum acacia, and there would be more flexibility to choose different encapsulating materials for specific purposes.

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