



Recent microencapsulation trends for enhancing the stability and functionality of anthocyanins: a review

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Abstract

Anthocyanins (ACNs) are water-soluble pigments in various fruits and vegetables known for their high antioxidant activity. They are used as natural food colorants and preservatives and have several medicinal benefits. However, their application in functional foods and nutraceuticals is often compromised by their low stability to heat, oxygen, enzymes, light, pH changes, and solubility issues. Spray drying has emerged as an effective microencapsulation technique to enhance the shelf life, quality, and stability of ACNs. This manuscript reviews the latest scientific developments in spray drying microencapsulation of ACNs-rich fruit extracts. Process optimization and the stability and physicochemical properties of the spray-dried, microencapsulated ACNs-rich powders are discussed. This review also covers functional food and nutraceutical applications and introduces novel encapsulation methods, such as freeze-drying, supercritical carbon dioxide (SC-CO₂), coacervation, drum drying, and electrospraying, highlighting their potential in improving the utility of ACNs-rich fruit extracts.

Keywords Anthocyanins stability · Encapsulating agents · Functional food value · Optimized spray drying · Powder product

Abbreviations

ABC	ATP-binding cassette
ACNs	Anthocyanins
ANN	Artificial neural networking
AVIs	Vacuolar inclusions
BTL	Biltranslocase
CHI	Chitosan
CMC	Carboxymethylcellulose
CO ₂	Carbon dioxide
CSG	Cress seed gum
DE	Dextrose equivalents

EC	Encapsulating carbohydrates
ER	Endoplasmic reticulum
FAs	Fatty acids
FD	Freeze-drying
GA	Gum arabic
GGM	Galactoglucomannan
GRAS	Generally recognized as safe
GSTs	Glutathione S-transferases
GX	Glucuronoxylan
HMP	High methyl pectin
HPH	High-pressure homogenization

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IN	Inulin
IG	Ionic gelation
kDa	Kilodalton
KON	Konjac
MATE	Multidrug and toxic compound extrusion
MC	Modified chitosan
MD	Maltodextrin
mPa	Millipascal
Mw	Molecular weight
OSA	Sodium octenyl succinate
PAG	Prosopis alba exudate gum
PEC	Pectin
PEG	Polyethylene glycol
PVP	Polyvinylpyrrolidone
RBC	Rice bran concentrate
RSM	Response surface methodology
SA	Sodium alginate
SC-CO ₂	Supercritical carbon dioxide
SMP	Soy milk powder
SNARE	Soluble N-ethylmaleimide-sensitive factor attachment protein receptors
SPI	Soy protein isolates
SPP	Soy protein powder
TA	Total anthocyanins
Tg	Glass transition temperature
TP	Total phenolics
TS	Tapioca starch
WPI	Whey protein isolates
WS	Waxy starch
XG	Xanthan gum
ZG	Zedo gum
°Bx	Brix

Introduction

In the present era of optimal nutrition, the growing demand for healthier foods has led to the consumption of natural antioxidant-containing foods, such as polyphenol-rich fruits and vegetables. Anthocyanins (ACNs) are water-soluble phenolic compounds renowned for their potent natural antioxidant properties. Their unique structure, characterized by a flavonoid backbone, multiple hydroxyl groups, conjugated double bonds, chelation sites, glycosylation, and varied substitution patterns, collectively contributes to their exceptional antioxidant properties. These structural attributes enable ACNs to effectively neutralize free radicals, inhibit metal ion-induced oxidative reactions, and protect cells and tissues from oxidative damage, making them valuable natural antioxidants (Enaru et al., 2021; Tarone et al., 2020).

ACNs are responsible for plants' orange, red, violet, and blue colors and thus hold great potential as natural colorants (Azman et al., 2022a, b). Grapes, blackcurrants, blueberries,

and bilberries are among the most popular ACNs-rich fruits. In contrast, red cabbages (Machado et al., 2022), eggplants (Demiray et al., 2023), and red onions (Ali et al., 2016) are typical examples of ACNs-rich vegetables. In addition to antioxidants, ACNs provide a broad spectrum of therapeutic effects and biological activities, including anticarcinogenic, inflammatory, immune-stimulating, antibacterial, antiallergic, and antiviral properties (Nawawi et al., 2023a; Riberio et al., 2019; Salehi et al., 2020). However, the biological properties and nutraceutical value of ACNs are often compromised by their instability. The stability of ACNs is affected by multiple factors such as light, temperature, pH, enzymatic reactions, oxidation, presence of metal ions, humidity, microorganisms, moisture, solvents, and the presence of co-pigments, causing degradation or partial loss of these compounds (Azman et al., 2022a, b; Feitosa et al., 2023; Nawawi et al., 2023a; Nawawi et al., 2023b). In this regard, different delivery processes have been developed to improve the stability of ACNs and coloring pigments, such as microencapsulation, nanoencapsulation, and protein complex formation (Feitosa et al., 2023).

Microencapsulation is a viable alternative that efficiently preserves thermally sensitive compounds such as phenolic compounds (Abdel-Aty et al., 2022; Gawalek, 2022). This technique entraps bioactive compounds within the encapsulating agent and transforms liquids into powders for easier handling (Rocha et al., 2019; Singh et al., 2023a). Encapsulating agents such as maltodextrin (MD), gum arabic (GA), carrageenan, alginate, waxes, and phospholipids are added to facilitate powder production, protect bioactive compounds from oxygen, light, or other unfavorable conditions, and increase stability (Li et al., 2017; Ligarda-Samanez et al., 2023).

Many investigations on the microencapsulation of ACNs-rich fruit extracts, such as berries, pomegranates, jaboticaba, and cherries, have focused on storage, color stability, and the quality of the final fruit powder (Gawalek, 2022; Halahlah et al., 2023; Mahdavi et al., 2016b; Shwetha and Preetha, 2016). Spray drying, freeze-drying, and supercritical carbon dioxide (SC-CO₂) technology are commonly used microencapsulation techniques, and spray drying is the most popular among food industries owing to its cost-effectiveness, efficiency, and high yield of good-quality powder products (Ravichandran et al., 2023; Da Rosa et al., 2019). Over the years, various parameters of spray-drying microencapsulation techniques have been studied for ACNs from various fruit types. New updates are required to determine which parameters can be applied to best suit specific fruits.

Therefore, the primary goal of this review was to assess the current trends and advances in spray drying microencapsulation of ACNs-rich fruit extracts. The effects of different parameters, such as types of encapsulating agents, the ratio of encapsulating agent to feed, drying temperature, and feed

flow rate, on the physiochemical and stability characteristics and functional food quality of the end-use ACNs powder product are discussed. Furthermore, an overview of innovative microencapsulation techniques, such as freeze-drying, SC-CO₂, coacervation, drum drying, and electrospraying, is provided.

An overview of ACNs

The word ACNs has been coined from two Greek words, namely *Anthos* (flower) and *kyanos* (blue) (Koop et al., 2022). As a class of polyphenols and sub-class of flavonoids, ACNs are widely distributed in different parts, especially leaves, fruits, and flowers of various plants belonging to the *Rosaceae*, *Vitaceae*, *Saxifragaceae*, *Ericaceae*, *Cruciferae*, *Fabaceae* and *Caprifoliaceae* families, among others (Li et al., 2021; Mazza and Miniati, 2018). ACNs tend to be located in the cells of plant vacuoles and cell walls and can be acquired through extraction (Ijod et al., 2022) (Fig. 1). ACNs yield in fruits depends on several factors such as processing and extraction methods, growing location and weather, fruit maturity levels, and storage conditions before analysis (Mazza and Miniati, 2018; Nawawi et al., 2023a).

The concentration of ACNs varies from 0.1 to 1.0% across various plant materials (Ercoli et al., 2021). Vegetables (e.g., red potato, purple sweet potato, eggplant, carrot, purple corn, red onion, and red cabbage) and fruits (e.g., berries,

cherries, blood oranges, blackcurrants, pomegranate, grapes, plums, and apples) are essential sources of ACNs for human nutrition (Karacabey et al., 2023; Mohammed and Khan, 2022; Tan et al., 2023). Berries, grapes, blackcurrants, and some tropical fruits have been identified as abundant sources of ACNs (Khoo et al., 2017). Due to their broad range of nutra-pharmaceutical attributes and chemo-preventive effects, as evidenced by clinical trials, ACNs have emerged as promising natural compounds with the potential to replace synthetic additives or colorants (Thakur et al., 2023).

ACNs consist of two aromatic rings attached to three carbons in an oxygenated heterocycle and a chromane ring with a second aromatic ring (Tarone et al., 2020) (Fig. 2). ACNs have sugar at R₃ positions called oligosaccharide side chains, normally in a single glucoside unit. The amount of hydroxyl and methoxy groups indicates the level of intensity and type of color of the ACNs. The higher amounts of hydroxyl and methoxy groups contribute to the extracts' intense bluish and redness colors, respectively (Enaru et al., 2021).

ACNs are also known as the glycosylated (aglycone) forms of anthocyanidins. Chemically, anthocyanidins are sugar-free counterparts of ACNs. As natural dyes, these compounds are responsible for the color of many fruits (Khoo et al., 2017). There are six main types of glycosides derivative ACNs: pelargonidin, cyanidin, delphinidin, peonidin, petunidin, and malvidin. Interestingly, pelargonidin displays a red-colored pigment in fruits and berries but appears orange in flowers. Besides, cyanidin appears reddish-purple

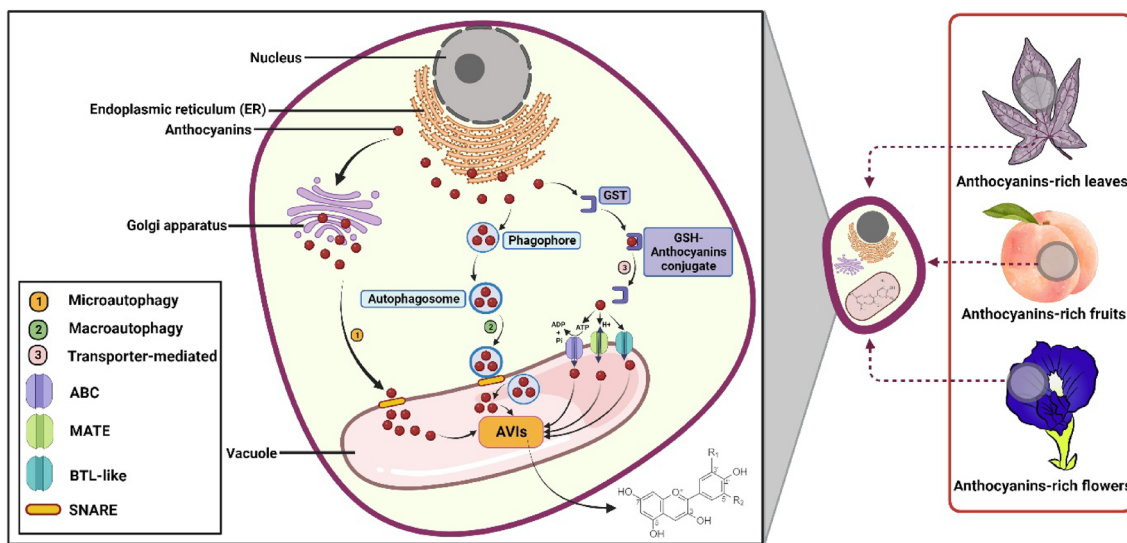


Fig. 1 ACNs move from the synthesis site, the endoplasmic reticulum (ER), to the vacuole for storage. ER-derived vesicles facilitate anthocyanin transport to the vacuole, where they bind to the membrane via soluble N-ethylmaleimide-sensitive factor attachment protein receptors (SNARE) and release ACNs during the micro (1) and macro (2) autophagy processes. Several membrane proteins (multi-drug and toxic compound extrusion (MATE), ATP-binding cassette

(ABC), and bilitranslocase (BTL-like transporters) aid in the transport of ACNs into vacuoles and their sequestration in vacuolar inclusions (AVIs) in the membrane transporter-mediated pathway (3), Glutathione S-transferases (GSTs) mediate the conjugation of ACNs to generate the glutathione-ACNs conjugate, which serves as an intact and efficient means of transport from the ER to the vacuole. Adapted from Nistor et al. (2022) with modification

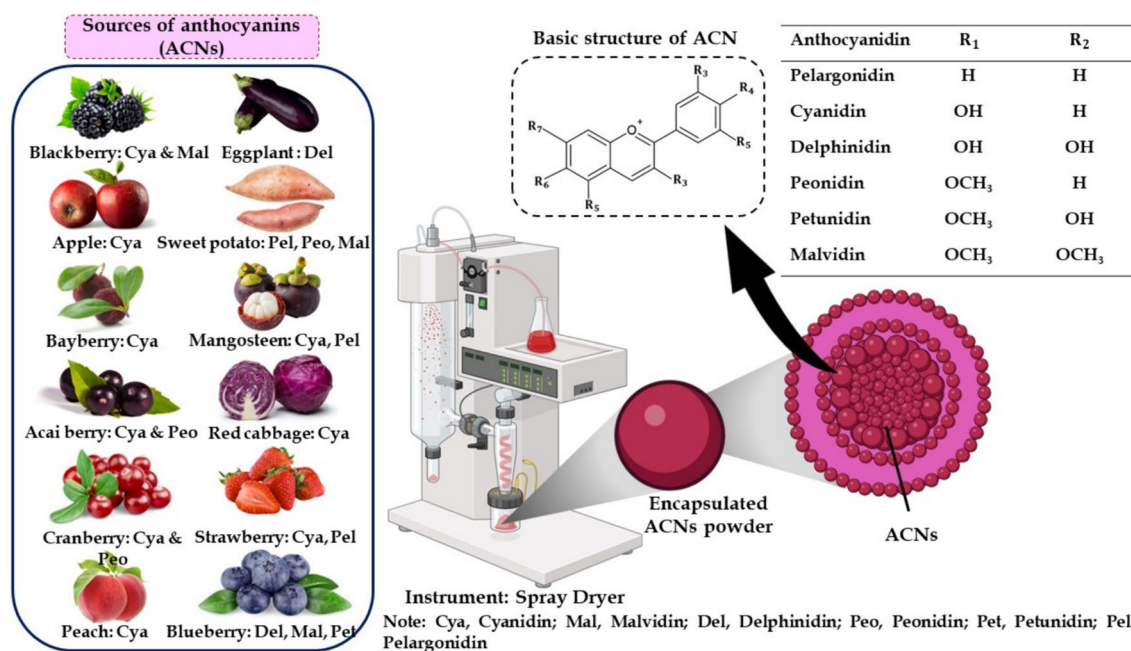


Fig. 2 Microencapsulation of ACNs extracts from various food sources and general chemical structure of ACNs and their anthocyanidins. R₃=sugar (glucose, arabinose, galactose, etc.). Adapted from Kozłowska and Dzierzanowski (2021) with modification

or magenta, while delphinidin causes a blue-reddish or purple pigment in plants. Examples of methylated anthocyanidins include peonidin, malvidin, and petunidin. Peonidin contributes to the magenta color in grapes, berries, and red wines. Also, malvidin displays as a darker rusty red pigment in red wine, while petunidin appears as a dark red or purple pigment in blackcurrants and purple petals of the flower (Khoo et al., 2017). These color variations can result from the complex interplay between pH, co-pigments, metal ions, genetics, environmental factors, chemical modifications, and concentration levels (Enaru et al., 2021).

Stability of ACNs

ACNs, which are hydrophilic, are highly unstable compounds and are quickly degraded due to different factors. The stability of ACNs is influenced by their concentration, pH, storage temperature, chemical structure, the presence of enzymes, proteins, flavonoids, metal ions, oxygen, and light (Enaru et al., 2021; Jafari et al., 2016). Their stability is also affected by the presence of hydroxyl or methoxy functional groups in the structure, as the presence of these clusters decreases the stability of the compound in an organic solvent or aqueous solution (Khoo et al., 2017).

The quality of ACNs degrades during processing and storage, thereby reducing the effectiveness of their potential role in the food and pharmaceutical industries. Therefore, preventive measures must be employed to maintain the

quality of these compounds, particularly during thermal processing (Ijod et al., 2022). The degradation of ACNs occurs when they are exposed to temperatures exceeding 60 °C for prolonged periods, typically exceeding 60 min. Such conditions can break covalent bonds within ACN molecules and induce oxidation processes, resulting in a loss of color and potential health benefits (Ali et al., 2016). In blanched purple potatoes (95–97 °C/2 min), a 63% loss in total monomeric ACNs was recorded compared to that in fresh potatoes (Karacabey et al., 2023). Also, hot drying (60°C, 10 h), hot water blanching (5 min), and steaming (100°C, 5 min) of red cabbages resulted in ACNs losses of 60, 23 and 13%, respectively (Tan et al., 2023).

Improving ACNs stability: chemical and biological approaches

The therapeutic applications of ACNs are often limited because of their reduced stability and low solubility in aqueous and organic media. Interestingly, ACNs can be transformed into acylated or glycosylated derivatives using enzymatic, chemical, or chemoenzymatic approaches. For example, converting ACNs into bioconjugates via fatty acid (FAs) acylation may offer the opportunity to positively modify these compounds' physicochemical properties and biological functionalities (Khoo et al., 2017). Bioconjugates, a novel class of hybrid materials consisting of a synthetic macromolecule linked to a biomolecule/

biological entity such as peptides or proteins, vitamins, and nucleic acids, are gaining increasing importance in the fields of medicine, biotechnology, and nanotechnology (Li and Mahato, 2017).

The stability of ACNs can also be improved through intramolecular copigmentation, wherein the color intensity of anthocyanidins/ACNs is increased or reinforced in the presence of other flavonoids as cofactors or copigments (Azman et al., 2022a, b). In this phenomenon, due to color intensification, an increase in color intensity with spectral shifts towards higher wavelengths can be observed with the addition of a copigment to acidic (preferably), neutral, and even slightly alkaline ACNs aqueous solutions.

Improving ACNs stability: physical approaches

In addition to bioconjugation and copigmentation, the stability and functional properties of ACNs can be improved using microencapsulation. Microencapsulation is a viable alternative for enhancing the stability and practical uses of ACNs on an industrial scale. In this process, ACNs, being the core material (active agent), are entrapped within another substance (wall material/coating material) or encapsulants such as starches, gelatin, GA, and MD (Estupiñan-Amaya et al., 2023; Mahdavi et al., 2016a; Rocha et al., 2019).

Optimization of various parameters (type and concentration of the encapsulating agent, concentration of the active agent, ratio of coating/active material, process time, temperature, etc.) is required to establish the best conditions for microencapsulation, offering better yield and good quality of the end-use powdered product, which has been extensively reviewed by Tarone et al. (2020). In this context, conventional optimization methods are now being replaced by modern approaches, such as response surface methodology (RSM) and *in silico* (computer simulation) modeling studies, to devise an optimized microencapsulation process (Machado et al., 2022; Mahdavi et al., 2016a; Tarone et al., 2020).

Microencapsulation provides many advantages to the final product by diminishing the surroundings and protecting bioactive compounds/ACNs from side effects caused by air, light, moisture, and heat. This process can deteriorate the vapor of the inside material to the outside environment and modify the physical characteristics to make ACNs or related bioactive materials more convenient to use. Also, microencapsulation is efficient in masking the inside material's flavor and forming two phases when mixed with liquid or semi-solid products (Ray et al., 2016). Powdered particle size can be categorized as macro (> 5000 μm), micro (1–5000 μm), and nano (< 1 μm) (Jafari et al., 2008).

Spray drying microencapsulation

Spray drying, due to its versatility, cost-effectiveness, and ease of operation, is the most common and widely applicable technique for the encapsulation of bioactive such as ACNs-rich extracts (Da Rosa et al., 2019; Ravichandran et al., 2023) (Fig. 2). Spray drying process is the traditional method for forming powders from semi-solid or liquid forms. Most of the 15,000 industries have used spray dryers to form products, such as luminescence materials, oxides, chemicals, fertilizers, and dried foods (Nandiyanto et al., 2019).

Additionally, it is a process in which the industry can manage acceptable levels of deterioration and decomposition of volatile compounds such as fruit juice. Spray drying, which involves encapsulation by creating protective 'walls' around sensitive ingredients, converts liquids into solids and enhances their shelf-life and color stability while safeguarding them against oxidation (Vasile et al., 2023). The reduced volume resulting from spray drying also simplifies the handling and storage of the compounds.

The principles of spray drying include preparation, homogenization, atomization, dispersion, and dehydration of the liquid solution. A critical issue during this process is wall deposition, which may affect the quality and quantity of the product that needs to be achieved. The occurrence of wall deposition depends on the spray dryer's type, size, and operating parameters. Appropriate measures and controls are needed to avoid wall deposition, thus preventing the high maintenance cost and lowering the powder yield (Tarone et al., 2020). The efficiency of spray drying depends on the selection of parameters, such as the encapsulating agent and its concentration added to the feed (active agent), inlet and outlet temperatures, atomization speed or pressure, and feed flow rate (Gawalek, 2022; Pan et al., 2022; Vasile et al., 2023). These factors must be controlled and manipulated for acceptable physical properties and a higher powder yield.

Encapsulating agents

The limitation of the spray drying technique is the use of high temperatures for drying and air access (Bednarska and Janiszewska-Turak, 2019), which may decompose thermally sensitive ACNs (Gawalek, 2022). Therefore, encapsulating agents are introduced in the spray drying to facilitate powder production, especially for ripe fruits with high °Bx value. This high °Bx value corresponded to a low glass transition temperature (T_g). When ripe fruits dehydrate above T_g , they may exhibit stickiness and adhere to dryer walls, resulting in a reduction in the final yield of

the product (Zotarelli et al., 2017). To prevent stickiness, one option is to control the glass transition temperature so that it remains below $T_g + 20$ °C. High molecular weight encapsulating agents can also improve the product's glass transition (Machado et al., 2022).

Encapsulating agents can be divided into groups of carbohydrates, proteins, or a combination of both. Carbohydrates act as a protective barrier from the external environment to the inside material, such as gums, starch, modified starches, dextrans, and cellulose (Halalah et al., 2023; Lacerda et al., 2016; Villacrez et al., 2013). The most essential characteristics of this group are their emulsifying activity and solubility properties. These agents should exhibit characteristics such as low viscosity, non-hygroscopic, bland flavor/tasteless, non-reactive with core materials, soluble in aqueous solvents, inexpensive, food-grade, flexible, rigid, thin, and pliable (Tan et al., 2015).

MD has good water solubility and low viscosity and is produced by enzymatic or acid hydrolysis of starch (Lacerda et al., 2016). The low emulsifying properties of MD can be counteracted by substituting it with sodium octenyl succinate (OSA) starch. OSA starch introduces lipophilic elements, resulting in amphiphilic properties that enhance emulsification (Lacerda et al., 2016; Sweedman et al., 2013). Another encapsulating agent in carbohydrates is inulin, which is a polysaccharide. This polysaccharide consists of fructose units linked by β -(2,1) bonds with glucose in the chain. Inulin is derived from chicory and has dietary fiber and prebiotic effects on consumers (El-Kholy et al., 2020; Lacerda et al., 2016).

Examples of protein types include whey protein isolates (WPI) and soy protein isolates (SPI) (Robert and Frede, 2015). These proteins excel as encapsulating agents because of their distinct attributes and adaptability. Whey protein isolates sourced from whey, a byproduct of cheese production, are renowned for their high nutritional quality as complete proteins containing all essential amino acids. They also exhibit exceptional emulsifying properties, stabilize emulsions, and efficiently encapsulate lipophilic compounds. Moreover, WPI has a low allergenicity, making it suitable for a wide array of applications, and its neutral flavor accommodates the encapsulation of diverse ingredients. On the other hand, SPI, derived from soybeans, offers versatility by encapsulating a broad spectrum of ingredients, including flavors, vitamins, minerals, and lipids. With its high protein content, SPI enhances the nutritional value of encapsulated products. In contrast, its functional properties, such as emulsification and film formation, further contribute to its effectiveness in encapsulation applications. Being gluten-free and sustainably sourced from soybeans, SPI aligns with dietary preferences and environmentally conscious practices (Bian et al., 2022).

The selection of encapsulating agents depends on the solubility of the bioactive compound of interest, which is either hydro- or lipo-soluble. GA is the best encapsulating agent for liposoluble bioactive compounds, and hydro-soluble compounds such as ACNs and MD with different dextrose equivalent (DE) values, GA, or modified starch are commonly used. Due to its low hygroscopicity, the prominent MD used in spray drying is MD 10-DE (De Souza et al., 2015).

Encapsulating agents from natural polymers can prevent the degradation of ACNs and aid in their delivery to the human body for nutraceutical applications (Vergara et al., 2020). Combining OSA starch, inulin, and MD as encapsulating agents with an encapsulating carbohydrates (EC) ratio of 2:1:1 (2/3:1/6:1/6) produced jussara pulp microparticles with favorable properties. Specifically, these microparticles exhibited excellent color, antioxidant activity, and ACNs content, making them a promising encapsulation approach for preserving the quality and functionality of the jussara pulp (Lacerda et al., 2016).

The production of ACNs-rich powders from various fruit sources using spray drying is summarized in Table 1. Overall, the SPI encapsulation technique was highly efficient for polyphenols (catechins, ellagitannins, gallitannins, and quercetin glycosides). This is due to the nature of the charge of bioactive compounds, where polyphenols and ACNs have negative and positive charges, respectively. Also, the polyelectrolyte structure (density and charge type) contributes to the interaction of SPI with the bioactive polymer. Meanwhile, MD was more effective in entrapping ACNs owing to the production of larger and smoother particles of fruit powder than GA, which produced smaller particles and wrinkled surfaces, thus making it easy to be exposed to oxygen (Ferrari et al., 2013).

Specific inlet and outlet temperatures are needed to maximize encapsulation efficiency and prevent the acceleration of ACNs, polyphenols, and antioxidant degradation (Gawalek, 2022). Higher inlet temperatures may produce lower-quality ACNs powder, such as a dense surface layer, thereby reducing the efficiency of powder reconstitution (Jafari et al., 2017). In some instances, spray drying using a high inlet temperature and aspiration rate may contribute to the high yield of microparticles. Consequently, this can reduce the stickiness of fruit powder on the cyclone wall (Yingngam et al., 2018). A more significant loss of ACNs was detected when the air inlet temperature was higher than 180°C (Gawalek, 2022). A higher inlet temperature with a lower concentration of encapsulating agents below 30% (w/w) leads to decreased ACNs stability due to non-enzymatic browning and pro-anthocyanidin deterioration (De Souza et al., 2015).

A combination of encapsulating agents, such as protein-based products, protects ACNs better than a single

Table 1 Production of ACNs-rich powder from different fruits using a spray drying technique

ACNs-rich fruits	Encapsulating agent	Dissolution medium for an encapsulating agent	Spray drying parameters	Stability enhancement	References
Blackcurrant (<i>Ribes nigrum</i> L.)	MD: DE11, DE18, DE21 & Inulin	Blackcurrant extract	Inlet/outlet temperature: 150/70 °C, 160/70°C, 180/85°C, 205/100°C	- Air inlet temperatures (> 180 °C) caused more polyphenol and ACNs losses - The highest TA and TP (86%) was obtained with MD DE 11 at 150 °C - MD gave higher TA and TP than Inulin	Bąkowska-Barczak and Kolodziejczyk (2011)
Jaboticaba (<i>Myrciaria jacobinaca</i>) peel	MD, GA, GA & MD, or Capsul™ & MD	Distilled water	Inlet temperature (140, 160, and 180 °C), feed flow rate (360 mL/h), aspirator air rate (28 m ³ /h)	- High preservation of TA and antioxidants in MD or GA when an inlet temperature of 160 °C was used	Silva et al. (2013)
Blackberry (<i>Rubus</i> spp.)	MD, GA, or both 7% (w/w)	Blackberry juice	Inlet air temperature (145 °C), outlet temperature (75–80 °C), relative humidity (32.8%), flow rate (0.49 kg/h)	- High TA after encapsulated with 7% of MD - 7% MD caused long shelf-life during storage for 150 days at 25 °C - The combination of MD and GA improved shelf-life during storage for 150 days at 35°C	Ferrari et al. (2013)
Bordo Grape (<i>Vitis labrusca</i>)	MD (10–30%) (w/w)	Bordo grape extract	Inlet temperature (130, 150, and 170 °C), feed flow rate (44 mL/min), airflow (40 L/min)	- Increased stability during storage (120 days) at 25 °C	De Souza et al. (2015)
Barberry (<i>Berberis vulgaris</i>)	MD: GA (3:1) & MD: gelatin (3:1)	Hot distilled water	Inlet temperature (150 °C), outlet air temperatures (100 °C), feed flow rate (800 mL/h)	- Longest half-life (7 months) in 4 °C for a combination of MD and GA	Mahdavi et al. (2016b)
Jamun (<i>Syzygium cumini</i>)	MD & GA (1:1) (w/w)	Distilled water	Inlet temperature (110 °C), outlet temperature (60–75°C), feed flow rate (1.5–20%)	- Increased stability at 70 °C	Shwetha et al. (2016)
Pomegranate (<i>Punica granatum</i>)	GA & Capsul™ (1:1)	Pomegranate juice	Inlet temperature (162–170°C), outlet temperature (89–93 °C), mass flow rate (1 kg/h), air flow rate (500 m ³ /h)	- Improved stability during storage (25 °C) for 3 months by preserving ~90% of ACNs	De Araújo Santiago et al. (2016)
Blackberry (<i>Rubus</i> spp.)	MD DE 18–20	Ultrapure water	Inlet temperature (150 °C), feed flow rate (470 L/h), aspirator rate (35 m ³ /h)	- Prolonged half-life of pure ACNs at 4 °C (224 days) and 35 °C (151 days)	Weber et al. (2017)

Table 1 (continued)

ACNs-rich fruits	Encapsulating agent	Dissolution medium for an encapsulating agent	Spray drying parameters	Stability enhancement	References
Maqui (<i>Aristotelia chilensis</i> (Mol.) Stuntz)	MD & SPI (2:1) (w/v)	Distilled water	Inlet temperature (120–180 °C), feed flow rate (1 mL/min)	- Decreased oxidation for 69–75 days	Fredes et al. (2018)
Maoberry (<i>Antidesma puncticulatum</i> Miq.)	MD (DE10) 1:5 (w/w)	Distilled water	Inlet temperature (140 °C), feed flow rate (6 mL/min), aspirator rate (29 m ³ /h)	- Preserved ACNs at 4 °C and 25 °C during 30 days of storage	Yingngam et al. (2018)
Elderberry (<i>Sambucus nigra</i> L.)	MC, SA, GA 1% (w/v)	Elderberry extract	Inlet temperature (115 °C), outlet temperature (58 °C), feed flow rate (4 mL/min)	- Faster release of active substances in MD followed by GA and SA - Better stability of ACNs in encapsulated than non-encapsulated sample	Ribeiro et al. (2019)
Red-Fleshed Apple (<i>Malus niedzwetzkyana</i>)	GA 6% (w/v) & MD 4% (w/v)	Distilled water	Inlet temperature (150 °C), outlet temperature (100 °C), feed flow rate (15 mL/min)	- Improved heat stability - Improved light stability during 12 days of storage	Xue et al. (2019)
Pomegranate (<i>Punica granatum</i>)	GA (15% w/v) & XG (0.075% w/v)	Emulsion	Inlet temperature (170 °C), outlet temperature (85 °C), flow rate (20 mL/min)	- Decreased oxidation at 25 °C and 60 °C for 30 days	Yekdane and Goli (2019)
Blueberry (<i>Vaccinium</i> spp.)	MD	Blueberry extract	Inlet temperature (120, 140, and 160 °C), outlet temperature (79.75, 100, and 108.25 °C), feed flow rate (0.45 L/h)	- Stabilized at 140 °C for 115.47 days	Da Rosa et al. (2019)
Blackberry (<i>Rubus fruticosus</i>)	MD (1:1) (w/w)	Distilled water	Inlet temperature (170 °C), outlet temperature (105 °C), feed flow rate (0.5 L/h)	- High stability, shelf-life, and low ACNs degradation in lower pH - Effective to minimize loss of TA up to pH 5	Santos et al. (2019)
Chokeberry (<i>Aronia</i> spp.)	MD DE 10, MD DE 15, GA and MD DE 10 & GA or MD DE 15 & GA	Chokeberry juice	Inlet temperature (160 or 200 °C), outlet temperature (80–160 °C or 100–200 °C)	- High TA during storage for 2 months at 4 °C in MD 10 with an inlet temperature of 160 °C - High TA during storage for 2 months at 25 °C when GA and MD DE 10 (3:1) with an inlet temperature of 160 °C	Bednarska and Janiszewska-Turak (2019)

Table 1 (continued)

ACNs-rich fruits	Encapsulating agent	Dissolution medium for an encapsulating agent	Spray drying parameters	Stability enhancement	References
Purple potato (<i>Solanum tuberosum</i> L.)	MD	Distilled water	Inlet temperature (92–188 °C), air flow rate (600 L/h), feed flow rate (3 mL/min)	<ul style="list-style-type: none"> - Increased bioaccessibility of ACNs during in vitro digestion - Preserved 48% of ACNs during storage for 138 days at 60 °C 	Vergara et al. (2020)
Black seedless barberry (<i>Berberis vulgaris</i>)	MD (7.5 & 15%), GA (3 & 6%), WPI (8 & 16%)	Barberry juice	Inlet temperature (130 °C), outlet temperature (80 °C), air flow rate (600 L/h),	<ul style="list-style-type: none"> - Increased WPI and MD-enhanced encapsulation efficiency - Preservation of TA and TP were optimized 	Mirzaei et al. (2021)
Blackcurrant (<i>Ribes nigrum</i> L.)	WPI 10% (w/w)	Deionized water	Inlet temperature (120 °C), outlet temperature (85 °C)	<ul style="list-style-type: none"> - High TA in encapsulated sample - TP and antioxidants were preserved better than control 	Wu et al. (2021)
Red cabbage (<i>Brassica oleracea</i> L. var. <i>capitata</i> L. f. <i>rubra</i>)	MD & GA	Raw extract or concentrated extract	Inlet temperature (130 °C), feed flow rate (8 mL/min), aspirator rate (75,031 m ³ /h), air pressure 0.14 mPa	<ul style="list-style-type: none"> - High stability of microencapsulated TA - MD is better than GA in heat stability 	Machado et al. (2020)
Mangosteen (<i>Garcinia mangostana</i> L.) pericarp	GA & MD or IN & MD or PEC & MD (3:37) (w/w)	Distilled water	Inlet temperature (150 °C)	<ul style="list-style-type: none"> - MD and IN exhibited superior physicochemical properties - MD and IN increased the stability of ACNs during storage at 4 °C 	Sakulnararat et al. (2021a)
Rabbiteye blueberry (<i>Vaccinium virgatum</i>)	WPI (10%), SPI (4%), HMP & WPI (10:1, 10:2, 10:4) or SPI & HMP (4:1, 4:2, 4:4) (w/v)	Distilled water	Inlet temperature (165 °C), outlet temperature (70 °C), feed flow rate (4 mL/min)	<ul style="list-style-type: none"> - Combination of HMP and SPI increased encapsulation efficiency - Increased stability of ACNs in combination wall than single wall during 60 days of storage 	Pan et al. (2022)
Chokeberry (<i>Aronia melanocarpa</i> L.)	MD DE 11 (60%)	Distilled water	Inlet temperature (150, 155, 160, 165, 170, 175, 180 and 185 °C), outlet temperature (89 °C), feed flow rate (10–15 L/h), drying air flow rate (460 m ³ /h)	<ul style="list-style-type: none"> - Highest TPC preserved after encapsulated with inlet temperature 150–150 °C - High stability of TA in the encapsulated sample when exposed to 150–165 °C 	Gawalek (2022)

Table 1 (continued)

ACNs-rich fruits	Encapsulating agent	Dissolution medium for an encapsulating agent	Spray drying parameters	Stability enhancement	References
Roselle (<i>Hibiscus sabdariffa</i> L.)	MD, GA, KON, MD & GA (1:1) or MD & IN (1:1) or MD & KON (1:1) (w/w)	Roselle extract	Inlet temperature (150 °C), outlet temperature (91 °C), feed flow rate (500 mL/h)	- MD and KON enhanced TP, TA, and antioxidants in the encapsulated sample - High preservation of ACNs after encapsulating with MD and GA - The highest TPC, proanthocyanidin, and ACNs content were obtained in SPI than TS	Nguyen et al. (2022)
American elderberry (<i>Sambucus nigra</i> subsp. <i>canadensis</i>) pomace and juice	SPI 8% & TS 8% (w/v)	Elderberry juice or pomace extract	Inlet temperature (120 °C), outlet temperature (67–77 °C), feed flow rate (10 mL/min)	- Increased storage and digestion stabilities - Highest stability of MD during 25 °C	Ravichandran et al. (2023)
Purple corn (<i>Zea mays</i> L.)	MD, MD & GA (1:1) or MD & WPI (3:7)	Purple corn extract	Inlet temperature (140 °C), outlet temperature (50 °C), peristaltic pump speed (350 r/h)	- High ACNs content and recovery (~97%) in the MD sample - MD:GA combination caused high TPC, scavenging activity, and recovery of TPC	Deng et al. (2023)
Andean blueberry (<i>Vaccinium meridionale</i> Sw)	MD, GA or MD & GA (1:1) (w/w)	Andean blueberry juice	Inlet temperature (170 °C), outlet temperature (80 °C), inlet air flow (32 m ³ /h), compressed air caudal (414 L/h)	- High ACNs content and recovery (~97%) in the MD sample - MD:GA combination caused high TPC, scavenging activity, and recovery of TPC	Estupiñan-Amaya et al. (2023)
Native potato clones (<i>Solanum tuberosum</i> spp. <i>andigena</i>)	MD & GA (9:1) (w/w)	Distilled water	Inlet temperature (120 °C), inlet air flow rate (141 L/h)	-~90% of encapsulation efficiency - Increased thermal stability - Increased stability of ACNs, phenolic and antioxidants	Ligarda-Samanez et al. (2023)
Bilberry (<i>Vaccinium myrtillus</i>)	GX, GGM, GA, GGM + CMC (1:0.7) or GX + CMC (1:0.7) (w/w)	Hot water	Inlet temperature (150 °C), outlet temperature (70 °C), feed flow rate (10–15 mL/min), compressed air pressure (5–8 bar)	- High TA, TP, and antioxidant activity in samples encapsulated with GX or GGM than GA, GGM + CMC, and GX + CMC	Halalah et al. (2023)
Roselle (<i>Hibiscus sabdariffa</i> L.)	MD (100%), MD & PAG (95:5, 85:15 & 70:30), MD & GA (85:15) (w/v)	Roselle extract	Inlet temperature (180 °C), outlet temperature (80 °C), feed flow rate (12 rpm)	- High antioxidant, TP and TA in sample encapsulated with PAG and MD - Improved stability of antioxidants when PAG was added during storage (60 days) at 25 °C and exposed to light	Vasile et al. (2023)
Grape (<i>Vitis</i> spp.)	MD DE 10, RBC	Grape juice	Inlet temperature (140 °C), outlet temperature (79 °C), feed flow rate (4.7 mL/min)	- The incorporation of RBC with MD increased TA, TP, and antioxidants in the encapsulated sample	Almeida et al. (2023)

Table 1 (continued)

ACNs-rich fruits	Encapsulating agent	Dissolution medium for an encapsulating agent	Spray drying parameters	Stability enhancement	References
Oxalis extract tubers (<i>Solanum tuberosum</i>)	Taro starch (15–33.1%) (w/v)	Distilled water	Inlet temperature (90–160°C), pump flow rate (7 mL/min)	<ul style="list-style-type: none"> - Better release of ACNs during in vitro study - High TA and encapsulation efficiency when 20.9% (Taro starch) and inlet temperature (125°C) were used 	Rosales-Chimal et al. (2023)
Tart cherry (<i>Prunus cerasus</i>)	MD DE 9–12 & GA (30% w/v)	Distilled water	Inlet temperature (175 °C), outlet temperature (85 °C), pump flow rate (6 mL/min)	<ul style="list-style-type: none"> - Increased TP, TA, and antioxidants were obtained when MD (15%) was combined with GA (15%), followed by micronization using HPH - Micronized sample had a higher encapsulation efficiency compared to the non-micronized sample 	Singh et al. (2023b)
Pink pepper (<i>Schinus terebinthifolia</i>)	MD, MD & GA (1:1) (w/w)	A mixture of pink pepper and green propolis extract	Inlet temperature (180 °C), outlet temperature (90 °C), pump flow (33.39 L/h)	<ul style="list-style-type: none"> - Combination of MD and GA increased the encapsulation efficiency - High antioxidants were observed after the combination of MD and GA - Combination of MD and GA caused the slow release of TP and TA in simulated gastric fluid 	Laureanti et al. (2023)

MD Maltodextrin; DE Dextrose equivalent; SPI Soybean protein isolates; GA Gum arabic; SPP Soy protein powder; PEC Pectin; IN Inulin; HMP High methyl pectin; WPI Whey protein isolates; MC Modified chitosan; SA Sodium alginate; CMC Carboxymethylcellulose; GGM Galactoglucomannan; GX Glucuronoxylan; PAG Prosopis alba exudate gum; RBC Rice bran concentrate; KON Konjac; TA Total anthocyanins; TP Total phenolics; TS Tapioca starch; HPH High-pressure homogenization

encapsulating agent. Combining encapsulating agents is assumed to create a strong interaction, leading to a synergistic effect in improving stability. The temperature and presence of light also influence the stability of ACNs during storage. Low temperatures and dark conditions during storage improve the stability of ACNs and prolong their shelf-life. Overall, optimizing spray drying conditions, particularly drying temperature, is crucial to ensure polyphenol encapsulation, as presented in Table 1.

Physicochemical, functional food, and nutraceutical attributes of encapsulated ACNs-rich powder

Moisture content, water activity, and particle size are the most critical parameters for producing microencapsulated ACNs powders. The low moisture content of microencapsulated fruit powder is vital to achieve excellent stickiness, flowability, and storage stability and prevent microbial growth. This is due to the higher water activity, which provides more free water space for microbial growth. Notably, the water activity level of the microencapsulated powder produced by spray drying is below 0.3, effectively inhibiting microbial growth (Todorović et al., 2022).

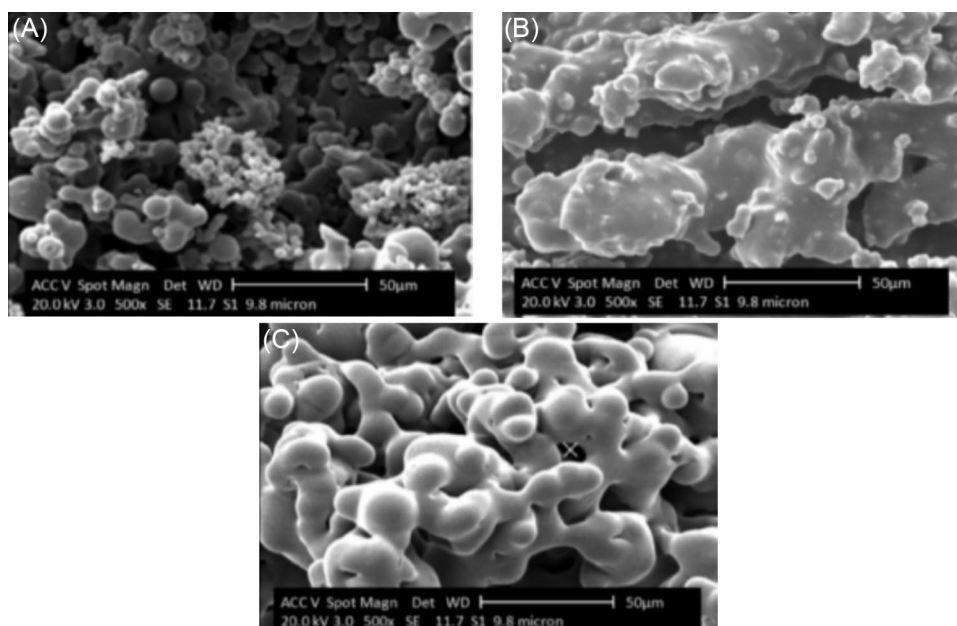
Furthermore, the particle size of the powder affects the texture and nutritional properties of the food product. The optimal size range for microencapsulated powder produced by spray drying is 10–100 μm (Da Rosa et al., 2019). Solubility is also salient because it provides desirable properties such as dispersibility, solubility, and wettability. These properties contribute to the rehydration of food ingredients; thus,

a low moisture content of the powder is desired (Mahdavi et al., 2016a). As shown in Fig. 3, good solubility of the microencapsulated pomegranate powder was achieved when GA was used as an encapsulating agent. However, its optical properties are poor compared to encapsulation with waxy starch and MD and without cellulose (Yousefi et al., 2010).

Santos et al. (2019) found that using MD in blackberry spray drying efficiently preserved the physicochemical characteristics of the end-use powder product. This was due to the MD characteristics having a less hygroscopic nature, which resulted in improved ACNs retention, low moisture content, and excellent powder reconstitution. With these properties, this spray-dried blackberry powder is easily soluble in water and can be effectively applied to produce juice fruit powder.

The microencapsulation of ACNs provides multiple benefits to human health. For example, the addition of encapsulated ACNs powders into yogurt, ice cream, or other desserts can serve as a prebiotic, assisting in addressing digestive system issues. Using inulin to encapsulate ACNs in cornelian cherry fruit extract has an anti-diabetic effect at 1 mg/mL. It is suitable as a food ingredient for diabetic consumers (Enache et al., 2020). Besides, this product suits a vegan diet and consumers with lactose intolerance and diabetic problems (Dias et al., 2020; Enache et al., 2020). In the simulated gastrointestinal studies by da Rosa et al. (2019), microencapsulated blueberry extracts successfully improved ACNs digestion than unencapsulated blueberry extracts. Oancea et al. (2018) also reported that using WPI as an encapsulating agent could facilitate the release of ACNs into the intestine. The presence of various enzymes and different pH levels

Fig. 3 Effect of encapsulating agent on the microstructure of microencapsulated ACNs powder with (a) GA (12%, w/v), (b) waxy starch (12%, w/v), and (c) MD (12%, w/v) without cellulose from Yousefi et al. (2010)



in the human digestive system makes it a complex system. This affects the stability and bioavailability of the ACNs. Combining encapsulating agents, as opposed to single encapsulating agents, is a successful approach. The synergistic effects of these encapsulating agents improve the stability and bioavailability of the ACNs. This implementation enables a controlled-release mechanism that efficiently administrates ACNs to targeted organs or systems, thereby promoting the overall improvement of human health (Fig. 4) (Da Rosa et al., 2019; Enache et al., 2020; Mansour et al., 2020; Oancea et al., 2018). Given these potential health advantages, producing microencapsulated ACNs powder with high solubility is essential and possesses sustained-released properties for its effective use in dry mixes or instant health foods.

Other innovative microencapsulation techniques

In addition to spray drying, various microencapsulation approaches have been studied and verified for their efficiency in encapsulating ACNs. Promising techniques for ACNs include freeze-drying, SC-CO₂, coacervation, drum-drying, and electrospraying microencapsulation. Each approach has unique benefits regarding quality, compound preservation, and encapsulation effectiveness. The following section will discuss their principles, benefits, and prospective uses for protecting the durability and efficacy of ACNs in detail.

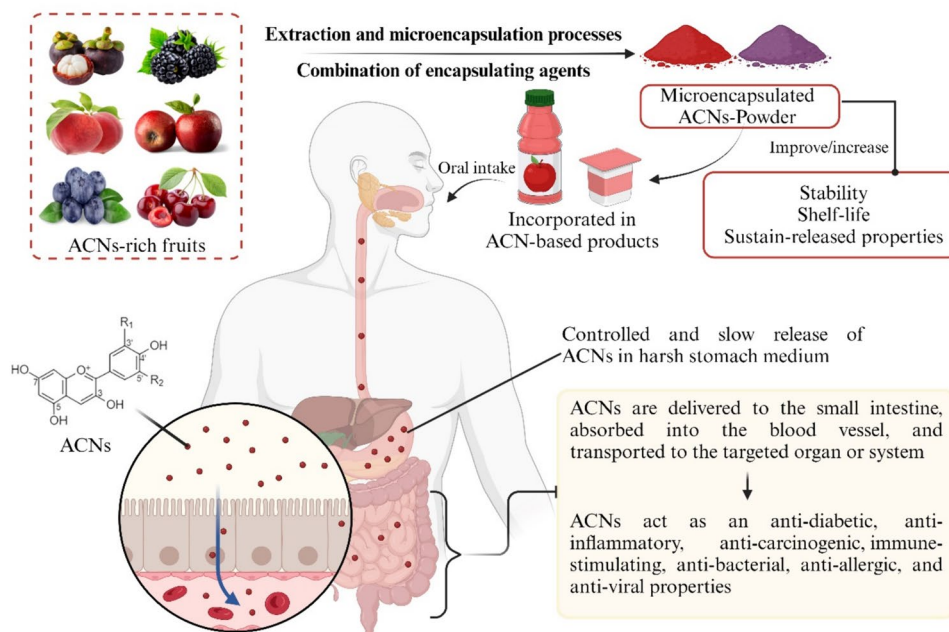
Freeze-drying

The freeze-drying technique (Fig. 5a) relies on the sublimation of water from frozen material and has been explored as an alternative approach for encapsulating ACNs (Fredes et al., 2018). This process involves freezing, sublimation, desorption, and storage (Bhatta et al., 2020). The sublimation phase efficiently extends shelf life and preserves the quality of heat-sensitive food materials. As a simple technique for encapsulating water-soluble essences such as ACNs and other natural aromas or medications, freeze-drying is one of the most convenient methods for drying thermosensitive substances that are unstable in aqueous solution (Azarpazhooh et al., 2018; Estupiñan-Amaya et al., 2020) (Table 2).

As shown in Table 2, MD frequently provided excellent ACNs retention and stability results, particularly at low DE values. A low DE generates low hygroscopicity in the ACNs-rich powder, which minimizes moisture absorption and accelerates powder deterioration. Moreover, MD is highly soluble, blending effortlessly with water and producing potent combinations when combined with ACNs.

The concentration of the encapsulating agent affects ACNs stability, as a high amount of the encapsulating agent serves as a solid wall to protect the core molecule. Regular integration of encapsulating agents improves ACNs retention during storage. This combination initiates the construction of a dual-property wall that regulates compound delivery while simultaneously increasing compound stability. This synergistic effect was observed when MD was combined with the different encapsulation agents.

Fig. 4 Oral intake and mechanisms of microencapsulated ACNs in the human digestive system



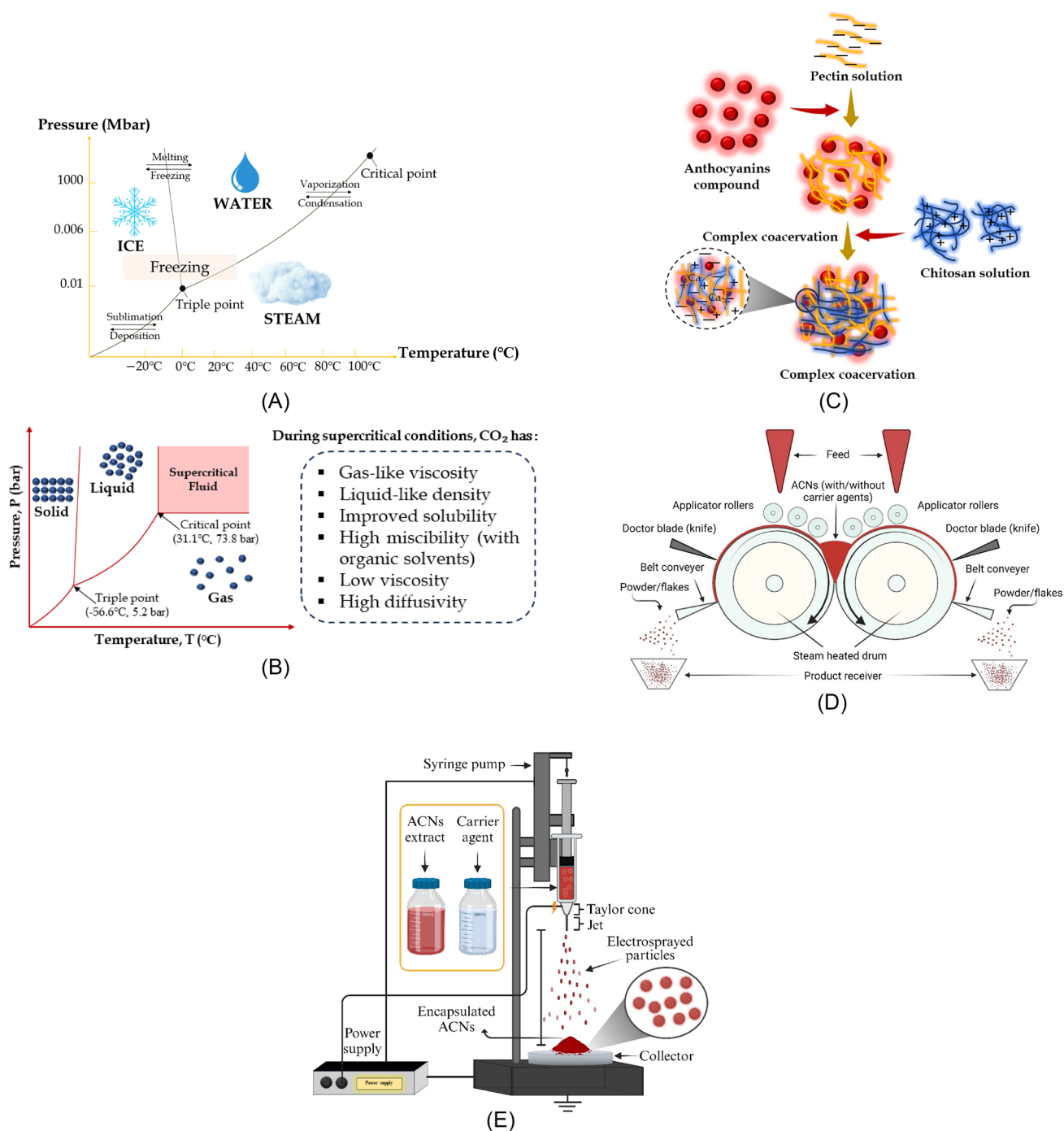


Fig. 5 Schematic diagram of (a) a typical freeze-drying mechanism, (b) supercritical conditions, (c) ACNs coacervation, (d) double drum drying mechanism, (e) a typical electrospaying mechanism. Adapted from Bigazzi et al. (2020) with modifications

Supercritical carbon dioxide (SC-CO₂)

SC-CO₂ is an emerging method that is inert, non-toxic, non-flammable, low cost, environmentally friendly, versatile, and free from toxic excess in the yield of the product formed (Fig. 5b) (Da Fonseca Machado et al., 2018). SC-CO₂ has excellent solvents such as carbon dioxide (CO₂),

ethane, water, propane, and dimethyl ether, which can be categorized as having gas-like low viscosity, intermediate diffusivity, and liquid-like high density. This technique may overcome the disadvantages of the conventional spray drying technique by applying the processing medium with the conditions above its critical point, 31.1 °C and 7.4 mPa (Jang and Koh, 2023), to precipitate and encapsulate the ACNs

Table 2 The encapsulation of ACNs-rich fruit by using freeze-drying, SC-CO₂, coacervation, drum drying, and electrospaying with different encapsulating agents

ACNs-rich fruits	Encapsulating agents	Dissolution medium for encapsulating agents	Techniques	Outcomes	References
Jabuticaba (<i>Myrciaria cauliflora</i>) skins	PEG	Ethanol	SC-CO ₂	<ul style="list-style-type: none"> - Encapsulated powder is more stable to light and temperature than free extract - Encapsulation through the conventional method stabilizes ACNs better than SC-CO₂ 	Santos et al. (2013)
Blackberry (<i>Rubus</i> spp.)	MD (DE 10 & 20) (3:7) (w/v)	Concentrated blackberry extract	FD	<ul style="list-style-type: none"> - High ACNs retention in MD 10 DE (76%) than in MD 20 DE (68%) - MD 10 DE showed better physicochemical analyses 	Yamashita et al. (2017)
Jucara Palm (<i>Euterpe edulis Maritius</i>)	MD & GA (1:1) (w/w)	Acidified pulp solution (pH 2)	FD	<ul style="list-style-type: none"> - Improved thermal stability - Increased ACNs retention when using 2:3 ratio of ACNs to encapsulating agents 	Mazuco et al. (2018)
Blackberry (<i>Rubus</i> spp.) residues	PVP	Blackberry ethanolic extract	SC-CO ₂	<ul style="list-style-type: none"> - High purity of ACNs and improved TA were obtained 	Da Fonseca Machado et al. (2018)
Blackberry (<i>Rubus</i> spp.) residues	PVP	Blackberry ethanolic extract	FD	<ul style="list-style-type: none"> - High TA and antioxidant capacities were attained 	Da Fonseca Machado et al. (2018)
Black raspberry (<i>Rubus occidentalis</i> L.)	GA & gelatin (1:1)	Emulsion	FD	<ul style="list-style-type: none"> - Improved stability of ACNs during 2 months of storage at 37 °C 	Shaddel et al. (2018)
Pomegranate (<i>Punica granatum</i>) peel	MD (5, 10, 15%) & calcium alginate (0.1%) (w/v)	Distilled water	FD	<ul style="list-style-type: none"> - Highest ACNs, phenolics, and antioxidant activities in MD 15% than 5% and 10% - Low degradation rate of ACNs in MD 15% than MD 5% & MD 10% during 42 days of storage 	Azarpazhooh et al. (2018)
Red-fleshed apple (<i>Malus niedzwetzkyana</i>)	GA 6% (w/v) & MD 4% (w/v)	Distilled water	FD	<ul style="list-style-type: none"> - Improved heat stability - Improved light stability during 12 days of storage 	Santos et al. (2019)
Red raspberry (<i>Rubus idaeus</i>)	SPI, GA or SPI & GA (1:1) (w/v)	Distilled water	FD	<ul style="list-style-type: none"> - Highest encapsulation efficiency in the combination of GA (2.5%) + SPI (2.5%) - GA showed higher antioxidant activity - Higher ACNs (48%) in GA + SPI (1:1) during 60 days of storage 	Mansour et al. (2020)

Table 2 (continued)

ACNs-rich fruits	Encapsulating agents	Dissolution medium for encapsulating agents	Techniques	Outcomes	References
Andean blueberry (<i>Vaccinium meridionale</i> Sw.) juice	MD (30–50%) (w/v)	Andean blueberry juice	FD	- MD 30 and 50% improved TA, TP, and antioxidants in the encapsulated sample - Long half-life in combination of ZG:Gelatin and CSG:Gelatin compared to unencapsulated at 50, 60, and 80 °C - CSG has higher thermal stability compared to ZG	Estupiñan-Amaya et al. (2020)
Blue barberry (<i>Berberis integrifolia</i>)	ZG 1.6% and CSG 1.6% (w/v), gelatin 5% (w/v)	Distilled water	Coacervation		Gharanjig et al. (2020)
Karonda (<i>Carissa carandas</i>)	CHI (40, 50, 60 mg/mL) and PEC (5, 6, and 7 mg/mL) (w/v)	Distilled water and acetic acid solution	Coacervation	- Bioaccessibility of ACNs in coacervated was ~1.6 and ~2.0 fold-higher than purified and crude extract - High antioxidant activity in coacervated (~1.0 and ~1.19 fold-higher) than purified and crude extract	Sarkar et al. (2020)
Blackcurrant (<i>Ribes nigrum</i> L.)	WPI (10%) (w/w)	Deionized water	FD	- High preservation of TP and antioxidant - High encapsulation efficiency was achieved	Wu et al. (2021)
Raspberry (<i>Rubus idaeus</i> sp.) juice	MD, GA, & WS (1:10) (w/v)	Raspberry juice	FD	- GA produced the highest yield - TP better preserved in WS than GA and MD - Highest ACNs in GA > WS > MD	Nthimole et al. (2022)
Roselle (<i>Hibiscus sabdariffa</i> L.)	MD, GA, KON, MD & GA (1:1) or MD & IN (1:1) or MD & KON (1:1) (w/w)	Roselle extract	FD	- Highest TP, TA, and antioxidants were attained in KON as an encapsulating agent - The combination of MD + GA caused high ACNs retained in the carrier core	Nguyen et al. (2022)
Bilberries (<i>Vaccinium myrtillus</i> L.)	MD, GA, and MD & GA (1:1) (w/w)	Distilled water	FD	- TP increased during 21 days of storage at 20°C in MD (70%), GA (80%), and MD + GA (80%) (1:1) - MD and MD + GA preserved ACNs better than GA during 21 days of storage at 20°C	Todorović et al. (2022)

Table 2 (continued)

ACNs-rich fruits	Encapsulating agents	Dissolution medium for encapsulating agents	Techniques	Outcomes	References
Aronia (<i>Aronia melanocarpa</i>)	MD (DE 16.5–19.5) (30%), XG (0.5%), GA (30%), CMC (1%) (w/v)	Distilled water	FD	- MD + CMC and MD + XG enhanced retention of ACNs during 100 days of storage at 25°C - Combination of encapsulating agents caused high TP, TA, and antioxidants - Encapsulation efficiency of ACNs higher after solvent ethanol–water extraction	Jang and Koh (2023)
Pomegranate (<i>Punica granatum</i>) peel	MD & <i>Lepidium perfoliatum</i> (Qodume Shahri) seed gum (0.5:4.5) (w/w)	Distilled water	FD		Zahed et al. (2023)
Pink pepper (<i>Schinus terebinthifolia</i>)	MD, MD, & GA (1:1) (w/w)	A mixture of pink pepper and green propolis extract	FD	- High encapsulation efficiency, TP, and TA were obtained in combination with MD and GA compared to a single MD	Laureanti et al. (2023)
Red cabbage (<i>Brassica oleracea</i> L. var. <i>capitata</i> L.f. <i>rubra</i>)	MD20 & GA (100:0, 80:20, 60:40, 40:60, 20:80, 0:100) (w/w)	Distilled water	Drum drying	- MD20:GA (20:80) showed high retention of ACNs during thermal stability at 60°C - Addition of encapsulated ACNs using MD20:GA (20:80) in the Thai desert ‘Aluar’ improved antioxidant and shelf life during storage	Sakulnararat et al. (2021b)
Lamduan (<i>Melodorum fruticosum</i> Lour., <i>Annonaceae</i>)	MD20 & GA (100:0, 80:20, 60:40, 40:60, 20:80, 0:100) (w/w)	Distilled water	Drum drying	- Combination of MD20:GA (60:40) exhibited the highest ACN content - Addition of lamduan ACNs-powder in gummy jellies using MD20:GA (60:40) increased antioxidant, color, and ACNs retention during storage	Sakulnararat and Koneczak (2022)
Black carrot (<i>Daucus carota</i> L.)	Gelatin 8% (w/w) & CHI (low Mw: 25 kDa, intermediate Mw: 350 kDa & high Mw: 600 kDa)	Acetic acid 20% or 80% (v/v)	Electrospraying	- Combination of chitosan and gelatin increased encapsulation efficiency - Low Mw chitosan exhibited slow release of ACNs in food simulants	Atay et al. (2018)
Blueberry (<i>Vaccinium corymbosum</i> L.)	WPI, zein, agave fructans (10, 20 & 30%) (w/v)	Distilled water or 70% ethanol	Electrospraying	- ACNs encapsulated with Zein showed higher stability during electrospaying	González-Cruz et al. (2020)

PEG Polyethylene glycol; SPI Soy protein isolate; GA Gum arabic; MD Maltodextrin; PVP Polyvinylpyrrolidone; WS Waxy starch; PEC Pectin; CMC Carboxymethylcellulose; XG Xanthan gum; WPI Whey protein isolate; KON, Konjac; IN Inulin; CHI Chitosan; ZG Zedo gum; CSG Cress seed gum; FD Freeze-drying; SC-CO₂ Supercritical carbon dioxide; Mw Molecular weight; kDa Kilodalton

(Da Fonseca Machado et al., 2018). A supercritical fluid is above its critical point where gas and liquid exist in the equilibrium phase, and this fluid is known as a pure substance (Wang et al., 2020). This technique's efficiency depends on the active ingredient's thermodynamic properties, encapsulating agents, suitable co-solvents, and the materials used.

As shown in Table 2, the ACNs exhibited more excellent stability when encapsulated in polyethylene glycol (PEG) and polyvinylpyrrolidone (PVP) using CO₂ as the solvent and ethanol as a co-solvent. It is worth noting that these two solvents are generally recognized as safe (GRAS). The improved ACNs retention during SC-CO₂ encapsulation can be attributed to the solubility of PEG and PVP in ethanol. PVP and PEG are both soluble in ethanol, which facilitates their combination with ACNs to create a stable solution before encapsulation.

Coacervation

Coacervation microencapsulation involves the separation of one or more hydrocolloids from the original solution. Following this separation, the newly created coacervate phase surrounds and encapsulates the active ingredient, which is either suspended or emulsified within the same reaction medium (Fang and Bhandari, 2010) (Fig. 5c). Coacervation encapsulation can be accomplished using a single colloidal solute such as gelatin, or through a more intricate method involving substances such as gelatin and gum acacia. Although complex coacervation often lacks specific shapes and is deemed an expensive method for encapsulating food items, it is important to weigh its potential advantages. Specifically, they can be valuable for encapsulating sensitive and high-value functional ingredients, including ACNs (Devi et al., 2023).

Devi et al. (2023) also found that using the dual emulsion method followed by complex coacervation with gelatin and acacia gum enhanced the microencapsulation of ACNs from black rice bran. This approach improved the encapsulation efficiency and thermal stability and ensured better stability of ACNs during storage at both 7°C and 37°C. The microcapsules exhibited decreased moisture content, hygroscopicity, and solubility. Additionally, their appearance was characterized by smooth, circular, or intact surfaces and firm and agglomerated structures. The findings in Table 2 indicate that the coacervation formulation effectively extended the shelf life of ACNs, especially under high-temperature conditions, compared to ACNs that were not encapsulated within a coacervation complex. This indicates that the coacervation formulation formed between the different encapsulating agents strengthens their heat stability, thereby improving the protection of the core materials. Also, the selection of gum-based materials significantly influences the complexity and

stability of coacervation formulations. This factor is crucial for dealing with sensitive compounds.

Drum drying

Drum drying is widely employed in food and chemical sectors to produce powdered or granular substances. This process involves spreading a liquid or slurry in a thin, even layer on the surface of a heated revolving drum to dry the material. The material underwent drying upon contact with the internally heated surface of the rotating drum (Fig. 5d) (Sakulnarmrat et al., 2021b; Sakulnarmrat and Konczak, 2022). Encapsulating agents are often used to protect ACNs during microencapsulation. Meanwhile, Senevirathna et al. (2021) produced purple sweet potato powder with the addition of citric acid rather than encapsulating it with encapsulating agents. In comparison to the control, the powder made with 0.6% citric acid had a higher concentration of ACNs, antioxidant activity, and an intense red color. As a result, it can be determined that factors such as steam pressure, drum rotation speed, and citric acid content influence the powder quality, which can be optimized using RSM.

Based on the findings in Table 2, it can be summarized that even though a similar combination of encapsulating agents was incorporated during drum drying, the encapsulation efficiency was different, probably because of the different sources of ACNs, which possess different properties and interactions with encapsulating agents. Therefore, optimization must be performed to select the best concentration of both encapsulating agents to address this issue.

Electrospraying

Electrospraying encapsulation involves the formation of nanodroplets by using a high-voltage electric field. The voltage, solution feed rate, solution properties, humidity, temperature, and separation from the needle tip to the collector are only a few variables that might influence the ultimate output (Atay et al., 2018). The electrospraying equipment included a syringe pump, voltage power supply, collector, and syringe (Fig. 5e). The electrospraying process requires injecting a syringe-fed mixture of ACNs and encapsulating agents into a liquid medium before an electric field is applied at the nozzle. Therefore, it overcomes the surface tension and produces a cone-shaped droplet called a Taylor cone. Increasing the electric field causes the Taylor cone to become fragile and expels tiny droplets of encapsulated ACNs. Therefore, the solvent evaporates in the air, causing the droplets to coagulate as small particles and accumulate in the collector (Atay et al., 2018; González-Cruz et al., 2020).

The solution properties, including viscosity, surface tension, pH, and electrical conductivity, are crucial elements that must be considered to ensure that ACNs can undergo

electrospraying. Improper solution properties cause undesirable particles to form, eventually reducing encapsulation efficiency (Atay et al., 2018). As reported by González-Cruz et al. (2020), the addition of 10–20% zein successfully encapsulated ACNs from blueberries; further increases in concentration caused instability and clogging in the nozzle. Adding ACNs to 10–30% agave fructans or WPI resulted in instability during the electrospraying process. Based on Table 2, it can be inferred that various factors must be considered during the encapsulation of ACNs by electrospraying. Using a combination of encapsulating agents has improved protective properties and the controlled release of ACNs. Nonetheless, when employing a single encapsulating agent, the concentration and molecular weight of the agent play critical roles in achieving high-quality encapsulated ACNs.

Encapsulation efficiency across various microencapsulation techniques

The encapsulation efficiency (EE) is a crucial parameter that determines the ability of the microencapsulation process to protect targeted compounds. The data presented by various authors in Table 1 and Table 2 for various microencapsulation techniques employed for encapsulating ACNs were used to illustrate the potential of their encapsulation efficiency (Fig. 6). SD and drum drying consistently showed high EE

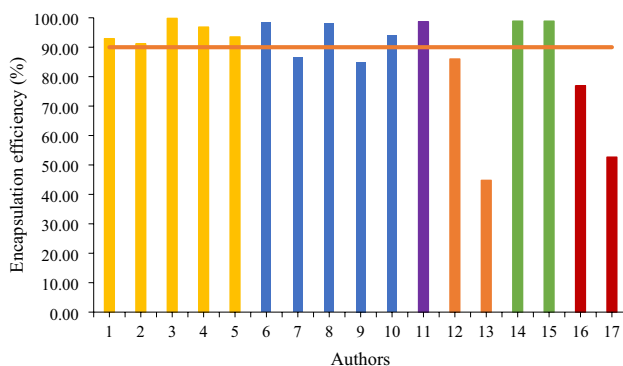


Fig. 6 Percentage encapsulation efficiency (EE) of various encapsulated ACN powders by referring to the optimal operation of different encapsulation techniques as reported by different authors. The straight line indicates 90% of EE. Different bar colors indicate the different techniques. Yellow, spray drying; blue, freeze-drying; purple, SC-CO₂; orange, coacervation; green, drum drying; dark red, electrospraying; numbers 1–17 referred to different authors. 1 – Fredes et al. (2018), 2 – Yingngam et al. (2018), 3 – Ribeiro et al. (2019), 4 – Xue et al. (2019), 5 – Pan et al. (2022), 6 – Laureanti et al. (2023), 7 – Zahed et al. (2023), 8 – Jang and Koh (2023), 9 – Nguyen et al. (2022), 10 – Xue et al. (2019), 11 – Santos et al. (2013), 12 – Gharanjig et al. (2020) 13 – Sarkar et al. (2020), 14 – Sakulnarmrat and Konczak (2022), 15 – Sakulnarmrat et al. (2021b), 16 – Atay et al. (2018), 17 – González-Cruz et al. (2020)

values ranging from 91.14%–99.80% and 98.85%–98.86%, respectively. In contrast, FD exhibited slightly more variability in EE, ranging from 85.00 to 98.33%. Although FD yielded a high EE in some studies (98.33%), some authors reported values lower than 90%, indicating an inconsistency in achieving at least 90% EE. Other techniques, such as SC-CO₂, showed an EE higher than 90%. Coacervation and electrospraying display varying degrees of efficiency, indicating the influence of the process parameters and formulation characteristics. The aforementioned techniques had the lowest EE, ranging from 44.77%–86.00% and 52.65%–76.90%, respectively. Considering the EE data, SD is the best and most promising choice for ACNs encapsulation, owing to its consistently high efficiency, relatively straightforward process, and cost-effectiveness.

Current status of product application with microencapsulation techniques

The increasing awareness and demand for healthy products among consumers requires the food industry and researchers to determine a solution for incorporating and protecting bioactive compounds, such as ACNs, in products. Therefore, microencapsulation is a promising solution for protecting ACNs. Many researchers have compared and determined the effects of different parameters and microencapsulation techniques on the stability of ACNs after incorporation into the product (Mihalcea et al., 2020; Sakulnarmrat et al., 2021a; Santos et al., 2022). For instance, Sakulnarmrat and Konczak (2022) incorporated ACNs from lamduan into gummy jellies after double-drum drying with different encapsulating agents. The combination of MD and GA (60:40) was selected as the best combination and applied to gummy jellies. The shelf-life stability of the gummy jellies was studied for eight weeks at different temperatures (25°C and 35°C). The highest lamduan encapsulated powder (30 g/kg) added to gummy jellies showed the most extended shelf life and retention of ACNs at both temperatures after eight weeks of storage. These findings corroborated the idea that encapsulating ACNs before application to food products retained and enhanced their stability and functionality. Other examples of ACNs sources, microencapsulation techniques, and their applications in various products are shown in Table 3.

Advantages and disadvantages of spray drying compared to other methods

Like any other method, spray drying has unique strengths and limitations compared to alternative techniques. It is quicker, more affordable, versatile, and appropriate for large-scale production than freeze-drying, SC-CO₂, coacervation,

Table 3 Application of encapsulated ACNs-rich fruits into products using microencapsulation techniques

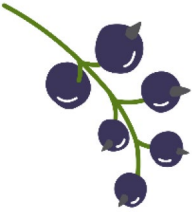







Sources of ACNs	Microen-capsulation Techniques	Food/beverage Products	Outcomes	References
	SD and FD	Yogurt	- High retention of TA (> 80%) in the SD and FD encapsulated powders after 35 days of storage at 5°C - Improved bioaccessibility of ACNs in both SD (44.1%) and FD (43.8%) technique than that in the non-encapsulated sample (35.2%)	Fredes et al. (2018)
Maqui (<i>Aristotelia chilensis</i> (Mol.) Stuntz) 	SD	Yogurt	- High ACNs retention in MD 10 DE (76%) than in MD 20 DE (68%) - MD 10 DE showed better physicochemical analyses	Sakulharmrat et al. (2021a)
Mangosteen (<i>Garcinia mangostana</i> L.) pericarp 	FD	Formulated jelly	- The addition of FD powder caused a slow release of ACNs during simulated gastric digestion and a significant release of bioactive compounds into the gut - A significant decrease in firmness, cohesiveness, and springiness in formulated jelly was observed after the addition of microencapsulated grape	Mihalcea et al. (2020)
Băbească Neagră grape (<i>Vitis vinifera</i> L.) 	SD, FD, and IG	Yogurt	- Higher bioavailability of Cya-3-Glu in the yogurt samples added with SD than that in the FD and IG powders	Santos et al. (2022)
Blackberry (<i>Rubus fruticosus</i>) pomace				

Table 3 (continued)

Sources of ACNs	Microen-capsulation Techniques	Food/beverage Products	Outcomes	References
 Red cabbage (<i>Brassica oleracea</i> L. var. <i>capitata</i> L.f. <i>rubra</i>)	Drum drying	Thai dessert 'Aluar'	- Encapsulated pomegranate peel using MD20:GA (20:80) increased the overall quality of the Thai dessert 'Aluar' - The addition of 30 g/kg of encapsulated pomegranate peel in Thai dessert showed the highest half-life in the model food	Sakulharmrat et al. (2021b)
 Roselle (<i>Hibiscus sabdariffa</i> L.)	SD	Marshmallows	- Marshmallows had an attractive red color and higher acceptability of appearance in the sensory test	Hoang et al. (2024)
 Camu-camu (<i>Myrciaria dubia</i> (Kunth) McVaugh)	FD and SD	Grape must and yogurt beverages	- Camu-camu encapsulated with MD and WPI (1:1) exhibited the highest global acceptance in yogurt, while incorporating camu-camu powder using only MD in grape must beverages yielded higher global acceptance than the control	García-Chacón et al. (2024)
 Blackberry (<i>Rubus fruticosus</i> L.) juice	Coacervation	Candies	- The addition of pea protein/tragacanth at the beginning and end of the candy-making process caused the significant release of phenolic compounds during the digestion phase	Vergara et al. (2023)

Cya-3-Glu Cyanidin-3-O-glucoside; *GA* gum arabic; *FD* Freeze-drying; *FD* Freezing-drying; *MD* maltodextrin; *SD* Spray drying; *TA* total anthocyanins; *WPI* whey protein isolate

drum drying, and electrospraying microencapsulation. However, the potential deterioration of heat-sensitive substances and low encapsulation efficiency during the encapsulation process are noteworthy disadvantages of spray drying. However, the choice of method always depends on the specific properties of the product being dried and the desired characteristics of the final dried product.

Conventional spray drying is the most popular and practically viable technique for microencapsulating ACNs and other bioactive components. High-quality powders with low moisture content, water activity, particle size, and morphology can be produced using spray drying. The selection of encapsulating agents may vary from case to case and mainly depends on the nature of the feed material/active agent to be microencapsulated. Various encapsulating agents are used on specific fruits at specific concentrations or ratios. It can be concluded that the best encapsulating agent is MD, with an inlet temperature of < 180°C. This was due to MD characteristics with low viscosity, moisture, hygroscopicity, and good solubility. These can help reconstitute and entrap bioactive compounds from deterioration during processing. Thus, it can easily be used as a food ingredient. Although there are many emerging technologies for microencapsulation, spray drying is still widely practiced in most industries due to its low operation cost, high yield, speed, and efficiency.

Overall, spray drying is more effective than other techniques for microencapsulation due to its ability to encapsulate bioactive compounds rapidly and individually. However, ACNs extraction requires longer, and the solvents are expensive and risky. Moreover, it is recommended that appropriate encapsulating agents for specific fruits be used to increase the stability of ACNs-rich powder, which is costly. Storage surroundings are also necessary for stabilizing the ACN compound; thus, suitable storage, such as vacuum-pack packaging, can be used to prevent oxidation. Therefore, it is recommended to add an antioxidant agent, such as tocopherol, into the fruit extract and encapsulating agents before the spray drying. Studies have focused on powdered products' physicochemical and nutritional characteristics derived from the spray-drying microencapsulation of ACNs-rich fruits. However, functional foods and nutraceutical qualities are frequently overlooked. Therefore, there is a need to analyze and explore the functional food and nutra-pharmaceutical prospects of ACNs-rich microencapsulated powdered products for specific food applications. Moreover, it can be noted that different process variables for spray drying microencapsulation of ACNs-rich fruits have been optimized by the researchers using conventional methods. However, with new software development, modern tools such as RSM and computer simulation techniques are needed to allow rapid evaluation and optimization of the plans and design of the spray drying-based microencapsulation process. Using

artificial neural networking (ANN) to validate such designs can provide more value for optimizing such processes.

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Declarations

Conflict of interest The authors declare that they have no competing interests.

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