



Contents lists available at ScienceDirect

Trends in Food Science & Technology

journal homepage: www.elsevier.com/locate/tifs

Review

Micro and nano encapsulation, retention and controlled release of flavor and aroma compounds: A critical review

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ARTICLE INFO

Keywords:

Flavor
Aroma
Microencapsulation
Nano-encapsulation
Stability
Industrial application
Food

ABSTRACT

Background: Encapsulation of flavor and aroma in an appropriate form is an important concern for a long time. Encapsulation is the most successful way not only to preserve or mask flavor and aroma compounds but also to enhance their thermal and oxidative stability, overcome the limitations of high volatility, to control the fast release and improve the poor bioavailability, as well as to increase their application in food systems.

Scope and approach: This review focuses on the recent advances in micro and nano-encapsulation of flavor and aroma compounds. We comprehensively highlight the suitability of micro and nano-encapsulation approaches for the retention of flavor and aroma including emerging techniques, new formulations and novel encapsulate systems, to illustrate the flavor release mechanisms, and depict the industrial applications of encapsulated flavor and aroma compounds.

Key findings and conclusions: Nano-encapsulation has attracted more attention compared to microencapsulation; showing better encapsulation efficiency, enhanced stability of capsule, and more control on flavor release. Recently, spray chilling and spray freeze drying as an alternative to spray drying, can overcome the thermal loss of flavor, promising for the microencapsulation of heat sensitive compounds. In contrast, electro-spraying and electro-spinning in combination with the emulsion or coaxial approach are novel and promising for the nano-encapsulation of flavor and aroma compounds. The combination of carrier materials, e.g. polysaccharide with protein can improve the encapsulation efficiency and capsule functionality. However, application of micro and nano-encapsulates into different food and gastrointestinal systems needs to be explored in order to expose their release mechanisms and application efficiency.

1. Introduction

Encapsulation is a common technique for creating an external membrane or coating of one material over another material, which is applied for the protection and/or preservation of bioactive, volatile, and easily degradable compounds from biochemical and thermal deterioration. It is also used for masking undesirable flavors and aromas. Encapsulation was first developed around 60 years ago as a technology for coating solids, liquids and gaseous compounds. The coating enables controlled release of compounds at specific rate under certain conditions (Desai & Park, 2005; Shishir, Xie, Sun, Zheng, & Chen, 2018). The

encapsulated materials can be pure substances or a mixture, which are also called the coated material, core material, actives, fill, internal phase or payload. On the other hand, the coating materials are known as packing material, capsule, wall material, film, membrane, carrier or outer shell (Fang & Bhandaria, 2010). The coating materials are usually made of natural or modified polysaccharides, gums, proteins, lipids, and synthetic polymers (de Souza Simões et al., 2017). The selection of coating material depends upon the nature of the core material, encapsulation process, and final use of the product. The morphology of the resulting microcapsule depends upon on the arrangement of core material and deposition process of the coating material, which can be

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<https://doi.org/10.1016/j.tifs.2019.02.030>

Received 2 December 2017; Received in revised form 4 September 2018; Accepted 6 February 2019

Available online 10 February 2019

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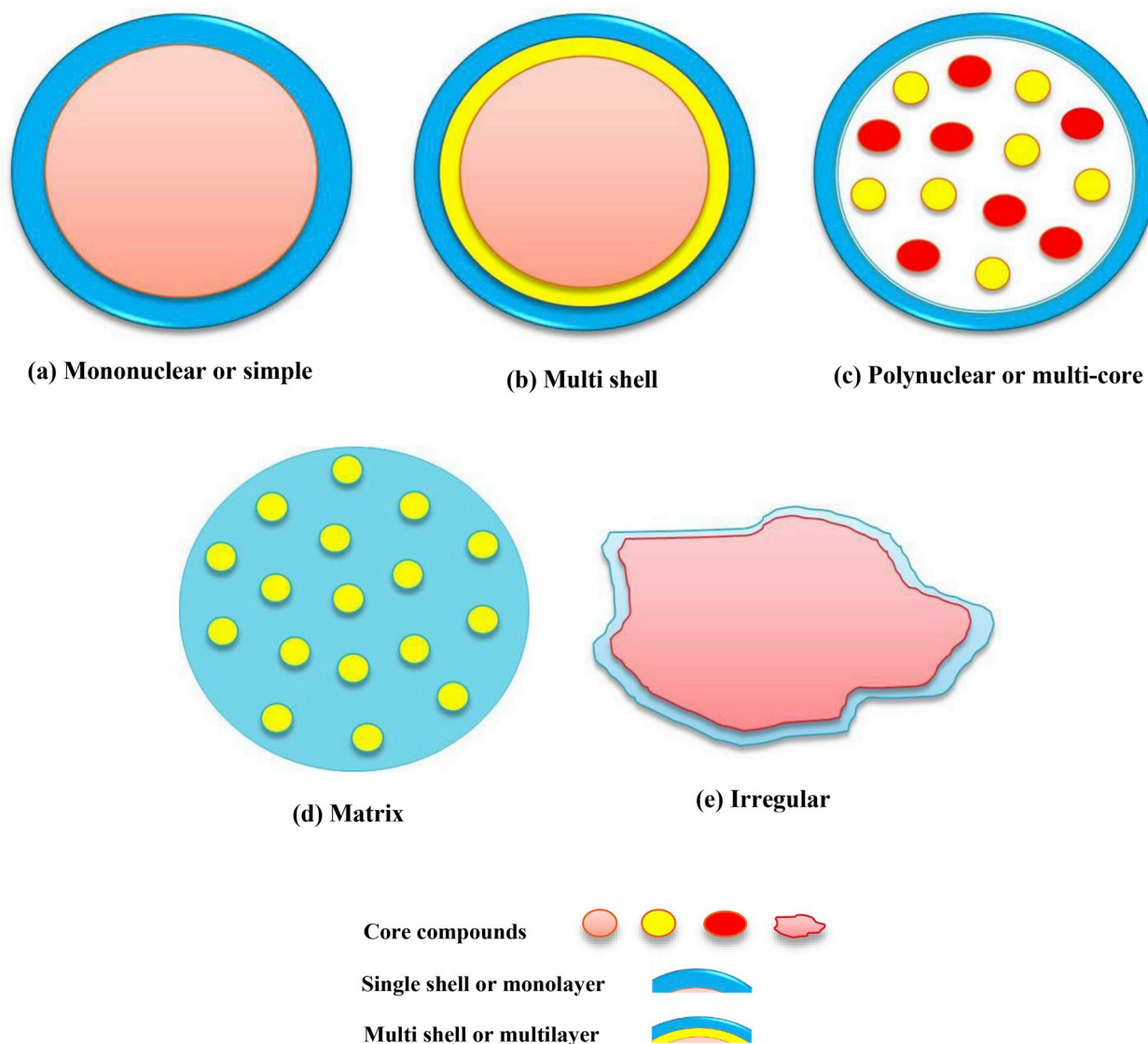


Fig. 1. Various form of capsules: (a) Mononuclear or simple, (b) Multi shell, (c) Polynuclear or multi-core, (d) Matrix, (e) Irregular.

divided into mononuclear, polynuclear, and matrix. Mononuclear capsules contain an outer shell around the core, while polynuclear capsules have many cores which are entrapped into a shell, and matrix capsules have the core material uniformly distributed within the shell material. In addition, it is possible to have a multi-shell mononuclear capsule or a capsule with non-spherical shape/irregular shape (Kim et al., 2004). Fig. 1 represents different forms of capsules.

Nano-capsules and microcapsules are the most functional and desirable size in encapsulation processing. Even though, nano scale and micro scale refers 1–1000 nm and 1–1000 μm , respectively, nano-encapsulation ranges the capsule size from 1 nm to several hundred nanometer in diameter and microencapsulation ranges from 1 μm to several hundred micrometers in diameter (Katouzian & Jafari, 2016; Rodríguez, Martín, Ruiz, & Clares, 2016; Shishir et al., 2018). Furthermore, particle size in between the range of nano and micro encapsulation is called as submicron particles, while the particle size above the microencapsulation range is referred as macro-particles (Koo, Cha, Song, Chung, & Pan, 2014; Lević et al., 2015; O'Toole et al., 2012). Microencapsulation is the most common and widely used in food and pharmaceutical industries. Recently, nanoencapsulations have attracted rising interest for their unique feature in terms of efficient encapsulation, enhanced stability, and better controlled release of encapsulated materials (Katouzian & Jafari, 2016; Shishir et al., 2018).

Flavors and aromas are organic molecules having low molecular weight. They are relatively volatile and very sensitive to air, heat, light, and moisture (Bakry et al., 2016). Therefore, encapsulation is a common practice in the flavor and fragrance industry to preserve both the flavor and aroma. Depending on the applied encapsulation technique, the materials obtained from the encapsulation process can be a powder, a paste or a liquid (Prost, Poinot, Rannou, & Arvisenet, 2012). In processed food products the degradation of flavor and aroma occurs during processing and storage of the product. To reduce the level of degradation and preserve the originality of flavor and aroma, pre-encapsulated volatile ingredients can be used in foods and beverages (Madene, Jacquot, Scher, & Desobry, 2006). However, the type of flavor and aroma preservation technique depends on the type of product it is going to be used in or the processing conditions through which it will pass through. For example, in the bakery industry, powder forms of encapsulated flavor and aroma compounds are mostly used as it is very stable, easy to weigh and store. In bakery applications, starch, wheat flour or soy flour are used as carrier substances (Cappelle, 2002). Liquid forms (e.g. emulsion) of encapsulated compounds are also used in different bakery products as it helps to improve the texture of the final product along with preserving the flavor and aroma compounds (Cappelle, 2002). In frozen products such as ice cream, flavorings are added directly to the other ingredients in the ice cream mixture

followed by passing through homogenizer and forms an oil-in-water type emulsion, or flavor is added to the ice cream mixture during agitation/maturation before freezing (Clark, 2009; Fiol et al., 2017). These emulsion particles preserve the flavor compounds and the temperature during processing and storage also assist in retaining the flavor until consumption. In beverages, flavor and aroma compounds are generally encapsulated in the form of micro and nano-emulsions before mixing with other ingredients. Different antioxidant compounds are also used to preserve flavor and aroma compounds in food products by preventing oxidation and off-flavor production. However, due to the taste profile and intense colors of some antioxidants it limits their use in food industry (Weerawatanakorn, Wu, Pan, & Ho, 2015).

To date, a variety of encapsulation techniques have been reported as novel and promising for the encapsulation of flavor compounds. These techniques, in the form of micro-capsule, micro-emulsion, nano-emulsion, nano-capsule, nano-fiber, nano-tube, solid lipid nanoparticle, inclusion complex, and bead encapsulation offer numerous benefits over conventional methods (e.g. spray drying) (Fuciños et al., 2017; Hussein et al., 2017; Koupantsis, Pavlidou, & Paraskevopoulou, 2016; Lević et al., 2015; Rodenak-Kladniew, Islan, de Bravo, Durán, & Castro, 2017; Saberi, Fang, & McClements, 2016; Tampau, Gonzalez-Martinez, & Chiralt, 2017; Zhang et al., 2017). Therefore, in this review, we comprehensively discuss the recent progress on micro and nano-encapsulation of flavor and aroma compounds. Several recent review articles focused on the encapsulation of food bioactive compounds and bioactive oils (Bakry et al., 2016; de Souza Simões et al., 2017; Katouzian & Jafari, 2016; Rodríguez et al., 2016), but from our best observation, there is particularly no recent review on the advances in micro and nano-encapsulation of flavor and aroma compounds. Hence, the aim of this review is to highlight the suitability of micro and nano encapsulation approaches for the retention of flavor and aroma compounds including emerging techniques, new formulations and novel encapsulate systems. Additionally, we aim to illustrate the controlled release mechanisms of encapsulated flavors, and to expose the industrial applications for encapsulated flavor and aroma compounds. This review enhances our understanding of the production of flavor and aroma capsules for industrial applications, and therefore provides some insight for future potential.

2. Roles of encapsulation for flavor and aroma compounds

Food flavor is a sensory attribute of food material. Chemically it is the combination of taste and aroma. Flavors and aromas are the most important attributes of food material. They may present in the food inherently, may develop during processing or be added during processing according to consumers' demands. Aromas and flavors give the first impression of a food material, which can change the consumers' intention for further use or consumption. Flavor stability with an appropriate level of strength is a great concern of food processors. This is because it has a strong relationship with the quality and acceptability of food materials, but it is difficult to control (Jun-xia, Hai-yan, & Jian, 2011; Madene et al., 2006). In fact, flavors are volatile in nature, and therefore very prone to release from food materials during manufacturing, transportation, and storage. Furthermore, flavors are very susceptible to change by the action of pH, moisture, acid, salt, and enzymes, and thereby may produce off-flavors (Feng et al., 2018; Fuciños et al., 2017). To increase the availability of flavor compounds and limit flavor degradation, encapsulation is the most effective and commonly practised approach (Khoshakhlagh, Koocheki, Mohebbi, & Allafchian, 2017; Sanchez-Reinoso, Osorio, & Herrera, 2017). Encapsulation protects the flavor from oxidative and thermal degradation and allows for efficient processing (Feng et al., 2018; Fuciños et al., 2017). For example, dry, granular or powder food products avoids dissolution of particles into a matrix. Encapsulation also creates a protective layer around the flavor droplet to protect and control its release at certain conditions, with potential to mask undesirable tastes

or very strong odors (Ezhilarasi, Karthik, Chhanwal, & Anandharamkrishnan, 2013). The droplets with diameters ranging from a few nanometers to a few millimeters are formed during encapsulation (Wandrey, Bartkowiak, & Harding, 2010). Encapsulation of food ingredients in micro scale is the common practice. However, nano-encapsulation of flavor offers greater benefits on stability, encapsulation efficiency, and control release over time in comparison with microencapsulation (de Souza Simões et al., 2017; Ezhilarasi et al., 2013).

Nanoparticles offers number of benefits over micro-particles. For example, nanoparticles create a more stable system as they have less tendency for particle aggregation or gravitational separation, particularly in the case of emulsification encapsulation. Very fine nanoparticles scatter light weakly and look transparent or less turbid. Therefore, nano encapsulated flavor and aroma compounds are very suitable for use in crystal clear beverage and other food without any negative impact on color attributes. Nanoparticles provide unique rheological properties in food (Chen, Khemtong, Yang, & Gao, 2011; McClements, 2012; Mozafari, 2006; Saifullah, Ahsan, & Shishir, 2016; Tadros, Izquierdo, Esquena, & Solans, 2004). They show higher bioaccessibility and are more easily absorbed in the body; hence it shows greater bioavailability (He & Hwang, 2016; Salvia-Trujillo, Martín-Belloso, & McClements, 2016).

3. Factors affecting flavor and aroma retention during encapsulation

Flavor retention during processing and flavor stability in processed products is a major concern in the food industry. Flavor represents both the unique characteristics and the quality of a food product. Almost all flavor and aroma compounds are very active and highly volatile in nature. They mix easily with air molecules and diffuse very quickly. Therefore, flavor demands significant attention during processing of food products, since it is very susceptible to degradation and/or loss in the presence of air, light, moisture, high temperatures or in the presence of certain other components within the food material. Encapsulation techniques protect the flavoring from most of these factors preventing flavor loss during processing and storage of food materials. Flavor stability and flavor retention are proportionally interrelated terms. When the flavor stability increases, flavor retention also increases (Given, 2009; Ma, Tan, Dai, & Zhou, 2013).

Retention of flavors during the encapsulation process depends upon the stability of flavor within the capsules. It can be defined as the mass ratio of total flavor (oil) in the powder to the theoretical quantity in the powder assuming ideal retention (Ma et al., 2013; Penbuditkul et al., 2012). Simply, it calculates the flavor losses during the production of encapsulates. Percent flavor retention can be calculated by the following equation.

$$\text{Flavor retention (\%)} = \frac{\text{Total flavor in capsules}}{\text{Flavor in the feed mixture}} \times 100 \quad (1)$$

This formula can also be used to determine the flavor retention in food stuff after processing. Of those, the total flavor content in encapsulated droplets or particles can be estimated by different techniques, such as distillation (Baranauskienė, Venskutonis, Dewettinck, & Verhe', 2006; Chen, McGillivray, Wen, Zhong, & Quek, 2013), extraction and evaporation (Chen et al., 2013; Li et al., 2013), gas chromatograph (GC) analysis (Ko, Jeon, & Park, 2012), and absorbance measurements (Li et al., 2013; Ramoneda, Ponce-Cevallos, del Pilar Buera, & Elizalde, 2011). It should be noted that flavor retention during encapsulation process depends on many factors, which could be classified into five major categories: flavor characteristics, carrier characteristics, sample preparation before encapsulation, method of encapsulation, and operating conditions of the encapsulation method (Gupta et al., 2016; Jafari, Assadpoor, He, & Bhandari, 2008). Fig. 2 represents the factors affecting flavor retention during encapsulation. A brief discussion on flavor retention affecting factors during encapsulation has been

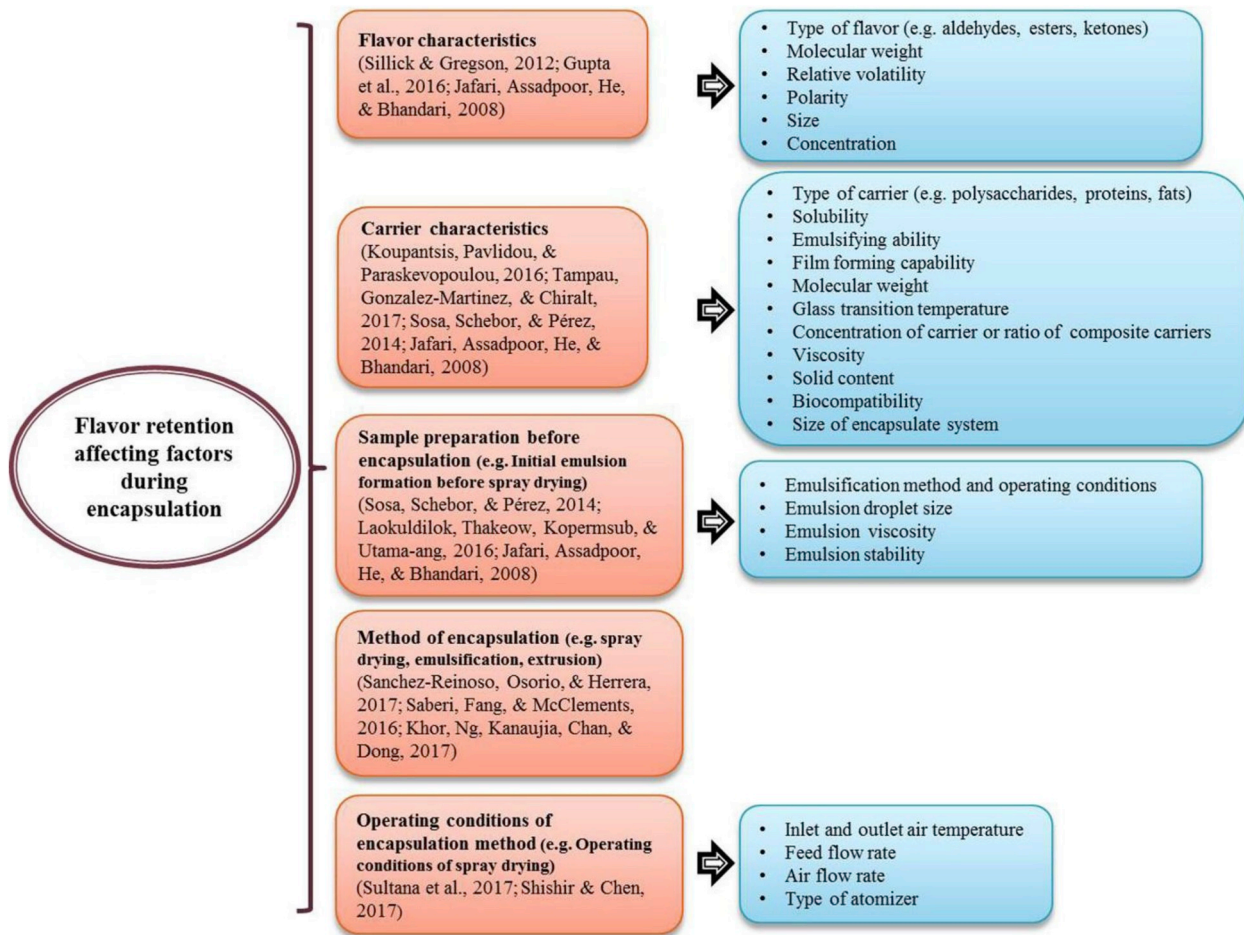


Fig. 2. Flavor retention affecting factors during encapsulation process.

addressed in this section.

3.1. Flavor characteristics

Understanding on flavor characteristics is very important for successful encapsulation of flavor compounds. Variations in flavor characteristics, i.e. molecular weight, relative volatility, polarity, type of flavor, size of flavor core and concentration of flavor may change the retention of flavor during the encapsulation process. Among the flavor characteristics, molecular weight and relative volatility are the key factors affecting flavor retention during encapsulation (Jafari, Assadpoor, He, et al., 2008). Molecular weight (MW) of flavor compounds is correlated to molecular size, which can influence the diffusion process of flavor molecules. Flavor compounds with higher molecular size exhibit a comparatively slower diffusion rate and require greater time to reach the atomized droplet surface during drying as compared to the compounds with lower molecular size. Therefore, higher molecular weight (MW) flavor compounds usually show better retention during the encapsulation process (Gupta et al., 2016). In contrast, volatility of flavor compounds refers to the ability of how fast a flavor compound can shift to the gaseous state from a liquid or solid phase. Relative volatility of a compound is generally measured on the basis of the volatility of water. Higher relative volatility of a flavor compound means the flavor has a high tendency to be volatilized and diffused (phase transition); therefore it may show less retention during the process (Jafari, Assadpoor, He, et al., 2008). Jørgensen et al. (2012) encapsulated eight flavor compounds with a variation in molecular weight (44.05–152.26) and boiling point (21–229 °C). Molecular weight (MW) has been found to have a close correlation with boiling point

(Bp). The Bp of flavor compounds increases with the increasing molecular weight of flavor compounds. While Bp is an indication of phase transition of a compound from liquid to gaseous phase. Therefore, relative volatility of flavor compounds is positively correlated with their MW (Jørgensen et al., 2012). Previous studies showed that the flavor compounds with high MW and Bp, i.e. citral (MW: 152.26, Bp: 220–229 °C), octanol (MW: 130.26, Bp: 196 °C) and butyric acid (MW: 88.1, Bp: 162 °C) had less evaporation (higher retention) during the process of either encapsulation with or without starch as compared to the flavors of acetaldehyde, dimethyl sulphide, diacetyl, allyl isothiocyanate, and ethyl butyrate, which have lower MW and Bp (MW:44.05–116.18, Bp: 21–121 °C) (Jørgensen et al., 2012). The retention of flavor compounds also depends on their polarity. Higher polarity compounds are relatively more soluble in water. In cases of encapsulation through drying process, the flavor loss increases with the water solubility of flavor compounds. This is because, the flavor diffuses with water and is lost during the evaporative process (Jafari, Assadpoor, He, et al., 2008). In contrast, corn starch–aroma compound inclusion complexes were prepared using co-precipitation method by Zhang et al. (2017). They reported that polar aroma compounds (linalool, heptanol and menthol) exhibited better inclusion ratio of aroma in comparison with nonpolar or weak polar aroma compounds (heptanolide, carvone and menthone). The aroma inclusion capacities were attributed to the solubility of aroma compounds and group structure properties of the inclusion complex. In addition, the thermo-stability results showed that inclusion complexes of polar aroma compounds had higher enthalpies than those of nonpolar or weakly polar aroma compound inclusion complexes, suggesting that the polar compounds need greater energy for the phase transition and were more stable than the nonpolar or weakly polar

Table 1
Recent studies reported on microencapsulation of flavors and aroma compounds.

Flavor compounds	Encapsulation method	Carrier agent	Encapsulate system	Major findings	References
Cocoa aroma compounds	Spray drying	Maltodextrin (MD) and Hi-Cap 100 (HC)	Microcapsule	<ul style="list-style-type: none"> ● The process yield reached up to 58.77% ● HC showed a higher retention of cocoa aroma (22.6–32.5%) and better micro-structural properties compared to MD in cocoa aroma microcapsules 	Sanchez-Reinoso et al. (2017)
D-limonene and ethyl hexanoate	Spray drying	<i>Saccharomyces cerevisiae</i>	Microcapsule	<ul style="list-style-type: none"> ● The encapsulation rate constant of D-limonene and ethyl hexanoate were 0.69/h ● Flavor retention of D-limonene and ethyl hexanoate were 5–48% and 24–45%, respectively ● The encapsulation efficiency and yield varied from 39% to 47% and 45%–50%, respectively ● All anylose-flavor inclusion complexes exhibited excellent thermal stability ● Amylose-n-heptanol inclusion complex showed greater stability than other complexes 	Sultana et al. (2017)
Aliphatic and aromatic alcohols	Sealed heating and freeze drying	Amylose	Molecular Inclusion Complex	<ul style="list-style-type: none"> ● Sodium caseinate-CMC microcapsules with the addition of glycerol exhibited greater loading capacity and encapsulation efficiency reached up to ~55% and ~90%, respectively ● Glycerol addition enhanced the encapsulation efficiency by two folds, while tannic acid was unsuccessful ● Glycerol addition showed higher retention of volatile compound in microcapsules during storage at low relative humidity (RH) and temperature (0% RH, 25 °C) 	Feng et al. (2018)
β-pinene	Complex coacervation	Sodium caseinate, whey protein isolate, carboxymethylcellulose (CMC), and reticulating agents (i.e. glycerol and tannic acid)	Microcapsule	<ul style="list-style-type: none"> ● Corn starch–linalool complex exhibited a higher inclusion ratio than the other complexes ● The study exposed that the polar aroma compound inclusion complexes required more energy for the phase transition and were more stable than the nonpolar or weak polar aroma compound inclusion complexes 	Koupanitsis et al., 2016; Koupanitsis & Paraskevopoulou, 2017
Six aroma compounds (Heptanolid, carvone, menthone, linalool, heptanol and menthol)	Co-precipitation	corn starch	Molecular Inclusion Complex	<ul style="list-style-type: none"> ● The addition of β-CD revealed better masking properties and encapsulation efficiency of curcuminoids ● The optimal encapsulation process was 5% of core loading mass with addition of 20 g/L of β-CD exhibiting high curcuminoids encapsulation with low volatile release, moisture content and hygroscopicity ● Ultrasound emulsification showed high emulsification efficiency and stability ● The supercritical fluid extraction process resulted in a considerable loss of oleoresin by dissolution and promoted a droplet volume expansion ● The study exhibited high retention of pungent and volatile compounds around 96% and 75%, respectively ● Lipid carriers with higher concentration of saturated fatty acids showed better retention of ginger oleoresin 	Zhang et al. (2017)
Turmeric flavor (Curcuminoids)	Homogenization and spray drying	Brown rice flour (BRF) and beta-cyclodextrin (β-CD)	Microcapsule	<ul style="list-style-type: none"> ● The study showed maximum encapsulation efficiency of cinnamic aldehyde and limonene are ~90% for the loading capacity of 10% 	Laokuldilok et al. (2016)
Pepper oleoresin spicy flavor (Capsaicinoids)	Ultrasound emulsification, supercritical fluid extraction and freeze drying	Hi-Cap 100 modified starch	Microparticle		de Aguiar, Silva, de Rezende, Barbero, & Martínez (2016)
Ginger oleoresin	Spray chilling	Palmitic acid with oleic acid or palm fat	Solid lipid microparticles		Oriani et al. (2016)
Five flavor compounds (Limonene, nicotine, methyl salicylate, cinnamic aldehyde and neobee)	Spray chilling	Erythritol	Crystalline particles		Sillick and Gregson (2012)

(continued on next page)

Table 1 (continued)

Flavor compounds	Encapsulation method	Carrier agent	Encapsulate system	Major findings	References
D-limonene	Emulsification and electrostatic extrusion	Sodium alginate	Calcium alginate bead	<ul style="list-style-type: none"> The encapsulation efficiency decreased steadily with increasing oil load (~63% efficiency for 30% of load) Crystalline particles exhibited significant protection to flavor compound from volatilization until melting of the erythritol at ~120 °C The study showed efficient immobilization of D-limonene within Calcium-alginate beads (immobilization efficiency 50–77%) Calcium-alginate beads can provide thermal protection to D-limonene up to 200 °C 	Levic et al. (2015)
Cardamom flavor (D-limonene, 1,8-cineole, myrcene, terpineol, linalool)	Emulsification and freeze drying	Whey protein isolate (WPI) guar gum (GG), and carrageen (CG)	Microcapsule	<ul style="list-style-type: none"> Microcapsules containing only WPI exhibited the highest flavor entrapment (7.5%) and microencapsulation efficiency (98.5%) The 30% WPI and 30% WPI + GG formulations showed better retention ability of 1,8-cineole and D-limonene during storage The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Mehyar et al. (2014)
Bitterness of quercetin	Hot-melt extrusion	Carnauba wax, shellac and zein	Microcapsule	<ul style="list-style-type: none"> The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Khor et al. (2017)
Orange terpenes	Hot melt counter-rotating extrusion	Maltodextrin and sucrose	Microcapsule	<ul style="list-style-type: none"> The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Tackenberg et al. (2015)
Orange flavor (terpenes, carvacrol)	Batch mixing	Maltodextrin and sucrose	Microcapsule	<ul style="list-style-type: none"> The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Tackenberg, Marmann, Thommes, Schuchmann, and Kleinebudde (2014)
Citral	Emulsification and spray drying	Sucrose, trehalose maltodextrin, and modified starch (Capsul)	Microcapsule	<ul style="list-style-type: none"> The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Sosa et al. (2014)
Sweet orange flavor	Molecular inclusion complex	β-cyclodextrin	Microcapsule	<ul style="list-style-type: none"> The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Zhu et al. (2014)
Vanilla oil	Complex coacervation	Chitosan	Microcapsule	<ul style="list-style-type: none"> The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Yang et al. (2014)
Rose hip seed oil	Coaxial electrospinning	Zein prolamine	Fibrous zein film	<ul style="list-style-type: none"> The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Yao et al. (2016)
Peppermint oil	Coaxial electrospinning	Alginate and pectin	Microcapsule	<ul style="list-style-type: none"> The microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH 6.8 in the order of zein > carnauba wax > shellac <i>In vitro</i> bitterness analysis ensured the good taste masking efficiency of the microcapsules Maximum loading of orange terpenes of 6% was obtained with the encapsulation efficiency of 86.7% The orange terpenes remained unchanged within the carrier matrix during the storage of 12 weeks The process conditions affected significantly the retention of orange terpenes and carvacrol The retention of active compounds between 6% and 100% correlated inversely with the vapour pressure of the pure active compounds The mixture of maltodextrin and trehalose offered higher glass transition temperatures Trehalose revealed promising performance in carrier formulation for the encapsulation citric flavors The orange flavor-β-cyclodextrin inclusion complexes can form large aggregates in water The thermogravimetric curve of flavor-β-cyclodextrin inclusion complex showed downward sloping from room temperature to 2501C The formulation of vanilla oil and chitosan (2:1) exhibited the highest encapsulation efficiency The studies of thermostability and release profile of flavor microcapsules verified improved thermostability and long residual action of vanilla oil The fibrous oil loaded zein films showed the mean diameter ranging from 700 nm to 2.9 μm The optimal loading capacity and encapsulation efficiency of rose hip seed oil in zein matrix (35%) was 12.24% and 90.16%, respectively The study revealed microcapsules with a particle size ranging from 1.58 to 3.25 μm The microcapsules were more stable produced with higher ratios of alginate and pectin 	Koo et al. (2014)

(continued on next page)

Table 1 (continued)

Flavor compounds	Encapsulation method	Carrier agent	Encapsulate system	Major findings	References
Caffeine	Desolvation and spray drying	Sodium alginate	Microparticle	<ul style="list-style-type: none"> The maximum encapsulation efficiency was 85.15% obtained with alginate-pectin matrix of 80:20 Caffeine loaded alginate micro-particles showed improved stability in digestive system, and sustained release profile in simulated gastric fluid 	Bagheri, Madadiou, Yarmand, and Mousavi (2014)
Sweet orange oil	Complex coacervation and spray drying	Soybean protein isolate (SPI) and gum Arabic (GA)	Microcapsule	<ul style="list-style-type: none"> The highest coacervate yield was observed in SPI/GA ratio of 1:1 The core material load of 10% exhibited the highest encapsulation efficiency and yield The addition of sucrose (Sucrose: SPI = 1:1) increased the encapsulation yield by 20% GC-MS analysis revealed better retention of flavor compounds 	Jun-xia et al. (2011)
Menthol	Fluidized bed coating	Gelatin, oil-gelatin emulsion, modified starch, and aquacoat	Microparticle	<ul style="list-style-type: none"> Fluidized bed coating showed better encapsulation efficiency of menthol than spray drying Fluidized menthol particles exhibited controlled release of menthol in water than spray-dried particles Gelatin emulsion carrier was more effective and showed enhanced control on menthol release and maximum encapsulation efficiency (~95%) 	Sun et al. (2013)

compounds, when subjected to heat (Zhang et al., 2017). Therefore, better retention of polar flavor compound depends on encapsulation method. Flavor retention with various functional groups can be in the sequence of acids < aldehydes < esters ≤ ketones ≤ alcohols (Gupta et al., 2016; Jafari, Assadpoor, He, et al., 2008).

3.2. Carrier characteristics

Flavor and aroma compounds are volatile, thermo-labile and light sensitive. Therefore choosing suitable carrier materials and the core-to-carrier ratio are very important for efficient encapsulation of flavor compounds. The carrier materials should be good emulsifiers with low viscosity at high concentrations. They should also have good dissolution and network-forming characteristics, the ability to preserve flavor compounds from adverse processing and storage conditions, as well as the capability to control the flavor release profile in certain mediums (Bakry et al., 2016). From a health and safety point of view, “generally recognized as safe” (GRAS) materials are permitted for food applications, including natural biopolymers (Robin & Sankhla, 2013). The majority of biopolymers used for flavor encapsulation are polysaccharides, proteins, and lipids. A variety of biopolymers associated in encapsulation of flavor compounds have been presented in Table 1 and Table 2. Maltodextrins, cyclodextrins, pectin, sodium alginate, gums, whey protein concentrate, and zein are commonly used for the encapsulation of flavor and aromas either as a single carrier or in combination with each other, known as a complex form. Recently, complex forms have achieved greater attention due to their multi-benefits in retention, loading capacity and controlled release profile (Esfanjani, Jafari, & Assadpour, 2017; Jun-xia et al., 2011; Kayaci, Sen, Durgun, & Uyar, 2014; Koupantsis et al., 2016; Laokuldilok, Thakeow, Kopermsub, & Utama-Ang, 2016; Mehyar, Al-Isamil, Al-Ghizzawi, & Holley, 2014). Apart from the carrier materials, the physical or physicochemical interactions between flavor compounds and carrier materials, such as development of insoluble complexes, and molecular association of the carrier material with flavor compounds through hydrogen bonding, may also affect the flavor retention (Gupta et al., 2016). Jørgensen et al. (2012) reported that low molecular weight aroma compounds (i.e. acetaldehyde, dimethyl sulphide, diacetyl, allyl isothiocyanate and ethyl butyrate) binding to native starch granules exhibited positive retention of flavor as compared to pure aroma. While, the high molecular weight aroma compounds (i.e. citral, octanol and butyric acid) showed a negative retention, i.e. starch-induced aroma evaporation. The matter has been attributed to the effect of starch-mediated exposure from a large surface area or a partial destruction of starch granules from which aroma compounds can evaporate (Jørgensen et al., 2012).

3.3. Sample preparation before encapsulation

Sample preparation is the pre-encapsulation step that can also play a significant role in flavor retention. In general, the formation of core-carrier emulsions is used as a primary step before encapsulation and it is also used as an encapsulation technique. The detailed mechanisms of emulsification has been discussed under the section of 4.1 Emulsification. Spray drying and freeze drying are the most common techniques for encapsulation, in which emulsification is used for sample preparation as reported for turmeric flavor, cardamom flavor, and citral (Laokuldilok et al., 2016; Mehyar et al., 2014; Sosa, Schebor, & Pérez, 2014). Furthermore, several recent studies also added an emulsification step in electro-spraying, electro-spinning and electrostatic extrusion (Khoshakhlagh et al., 2017; Levic et al., 2015; Tampau, González-Martinez, & Chiralt, 2017). The key benefit of this pre-encapsulation step is that it allows the encapsulation of a variety of immiscible compounds particularly flavor, aromas and essential oils. The formation of an emulsion provides a protective shell to flavor compounds that can reduce the oxidative and thermal loss during the encapsulation process.

Table 2
Recent studies reported on nano-encapsulation of flavors and aroma compounds.

Flavor compounds	Encapsulation method	Carrier agent	Encapsulate system	Major findings	References
Menthone	<i>In situ</i> nanoprecipitation	Starch	Starch nanoparticles (SNPs)	<ul style="list-style-type: none"> The menthone loaded SNPs exhibited spherical shape particle sizes ranging from 93 to 113 nm The results showed maximum encapsulation efficiency of 86.6% and loading capacity of 0.19 ml/g The menthone loaded SNPs formed at 90 °C revealed high crystallization and thermal stability SLNs exhibited spherical shape and mean diameters in the range of 90–130 nm with narrow size dispersion Linalool loaded SLNs revealed more than 80% encapsulation efficiency in all tested formulations <i>In vitro</i> study ensured controlled release profiles of linalool loaded SLNs for at least 72 h 	Qiu et al. (2017)
Linalool	Melting and Sonication	Myristyl myristate (MM), cetyl esters (CS) and cetyl palmitate (CP)	Solid lipid nanoparticles (SLNs)	<ul style="list-style-type: none"> D-limonene loading of 10 and 20% revealed high encapsulation efficiency (around 87–93%) Enhanced thermal stability of D-limonene nano-capsules was observed 	Rodenak-Kladniew, Isian, de Bravo, Durán, & Castro (2017)
D-limonene	Emulsion electro-spraying	<i>Alyssum homolocarpium</i> seed gum, Tween 20	Nano-capsule	<ul style="list-style-type: none"> PCL carrier systems exhibited nano-fiber with some beads, while starch systems showed nanoparticle PCL systems revealed better carvacrol encapsulation efficiency than starch systems Maximum encapsulation efficiency (> 80%) was achieved at 15% PCL 	Khoshkhalagh et al. (2017)
Carvacrol	Emulsion electro-spinning	Polar corn starch-sodium caseinate, (CS-NaCas) and non-polar poly-ε-caprolactone (PCL)	Nano-fiber and nanoparticle	<ul style="list-style-type: none"> The study exhibited highly stable caffeine loaded α-lactalbumin nanotubes 	Tampau, Gonzalez-Martinez, & Chiralt (2017)
Caffeine	Self-assembly and freeze drying	α-lactalbumin	Nanotube	<ul style="list-style-type: none"> The nanotubes revealed excellent encapsulation efficiency near to 100% and loading capacity of ~10% 	Fuciños et al. (2017)
Carvacrol	Desolvation/High shear homogenization	Maltodextrin and Tween 20	Nano-capsule	<ul style="list-style-type: none"> The study revealed the encapsulation efficiency of 49.3–76.4% and loading capacity of 48.7–69.2% 	Hussein et al. (2017)
Caffeine	Heat-induced denaturation	β-lactoglobulin	Nanoparticle	<ul style="list-style-type: none"> Nano-encapsulation of carvacrol was more efficient than nano-emulsion The particle size ranged from 200 to 300 nm Maximum caffeine encapsulation of 13.54% was found at caffeine to β-lactoglobulin ratio of 50:1 The study revealed rapid nanoparticle degradation, increased polydispersity, and little caffeine release at stomach conditions Caffeine completely released from nanoparticles at intestinal conditions 	Guo, Harris, Kaur, Pastrana, and Jauregi (2017)
Ethylvanillin	Electro-spraying	Stearic acid	Nanoparticle	<ul style="list-style-type: none"> The nanoparticles ranged from 60 to 70 nm The study revealed the polydispersity index and encapsulation efficiency of ~21% and ~70%, respectively Exhibited consistent release profile of ethylvanillin with diffusion through a lipid membrane 	Ethayeb, Stride, Edirisinghe, and Harker (2016)
Ethylvanillin	Electro-spraying	Ethylcellulose	Nanoparticle	<ul style="list-style-type: none"> The mean particle size varied between 45 and 85 nm and the polydispersity index between 16 and 34% The loading capacity and encapsulation efficiency ranged from 67 to 81% and 71–84%, respectively The release profile of ethylvanillin from the nanoparticles exhibited sustained release over 15 h 	Ethayeb, Stride, and Edirisinghe (2015)
Vanillin	Emulsion-solvent evaporation	Poly lactic acid (PLA)	Nanoparticle	<ul style="list-style-type: none"> The study exhibited homogeneous particle size of ~240 nm with vanillin encapsulation efficiency of 41% 	Dalmolin et al. (2016)

(continued on next page)

Table 2 (continued)

Flavor compounds	Encapsulation method	Carrier agent	Encapsulate system	Major findings	References
Peppermint oil	Dispersion and freeze drying	Zein and gum arabic	Nanoparticle	<ul style="list-style-type: none"> <i>In vitro</i> release study showed a slow and sustained release of vanillin PLA nanoparticles offered good physical and chemical stability during 90 days of storage in room temperature Peppermint oil loaded zein/gum arabic nanoparticles exhibited the mean particle size of ~128 nm Maximum encapsulation efficiency (~89%) was found for the formulation of zein: peppermint oil = 4: 1 The gradual release of peppermint oil from freeze-dried samples was found at pH 2.0–8.0 The study revealed the smallest emulsion size of 36 nm Nanoemulsions exhibited a good physical stability and strong antimicrobial activity in comparison with free β-limonene The encapsulation method successfully produced nano-emulsions, while Tween 40 showed optically transparent and small droplets ($d < 30$ nm) Emulsions produced at optimum condition of oil phase composition and surfactant type can show thermal stability to coalescence up to over 80 °C The study exhibited stable nano-capsules with < 200 nm in size 	Chen and Zhong (2015)
β -limonene	High pressure homogenization	Monostearin and medium chain triglyceride oil	Organogel-based nanoemulsion	<ul style="list-style-type: none"> Nanoemulsions exhibited a good physical stability and strong antimicrobial activity in comparison with free β-limonene 	Zahi et al. (2015)
Limonene	Spontaneous emulsification	Medium chain triglyceride oil and several types of Tween (40, 60, and 80)	Nano-emulsion	<ul style="list-style-type: none"> The encapsulation method successfully produced nano-emulsions, while Tween 40 showed optically transparent and small droplets ($d < 30$ nm) Emulsions produced at optimum condition of oil phase composition and surfactant type can show thermal stability to coalescence up to over 80 °C 	Saberi et al. (2016)
Eugenol and thymol	Anti-solvent precipitation	Zein, Sodium caseinate	Nanocapsule	<ul style="list-style-type: none"> The study exhibited stable nano-capsules with < 200 nm in size Eugenol and thymol loaded nano-capsules showed controlled release in 24 h Thymol nano-capsules revealed better loading capacity, encapsulation efficiency, and controlled release than eugenol nano-capsules 	Chen, Zhang, and Zhong (2015)
Roasted coffee oil	Mini-emulsification solvent Evaporation and sonication	Poly L-lactic acid (PLLA) and poly hydroxybutyrate-co-hydroxyvalerate (PHBV)	Nanocapsule	<ul style="list-style-type: none"> The study showed successful encapsulation of major aroma compounds of coffee oil in nanocapsules Sonication yielded the highest oil recovery for PLLA systems, while high shear homogenization led to the highest oil recovery for PHBV system 	Freiberger et al. (2015)
Geraniol (loaded into cyclodextrin inclusion complex)	Electrospinning	γ -cyclodextrin (γ -CD), and polyvinyl alcohol (PVA)	Nanofiber	<ul style="list-style-type: none"> Geraniol/γ-CD inclusion complex loaded PVA nanofibers exhibited the mean diameter of ~290 nm The study showed high thermal stability of nano-fibers Geraniol/γ-CD inclusion complex loaded PVA nanofibers revealed enhanced thermal stability for 2 years with a minimal loss of geraniol (~10%) 	Kayaci et al. (2014)
Eugenol	Emulsion-ionic gelation crosslinking	Chitosan	Nanoparticle	<ul style="list-style-type: none"> The results revealed the loading capacity of 12% and encapsulation efficiency of 20% with a spherical shape having the size of < 100 nm The extrusion of eugenol loaded chitosan nanoparticles ensured the improved thermal stability of encapsulated eugenol up to 155 °C compared to free eugenol 	Worannuch and Yoksan (2013)
Saffron extract (crocin, safranal, and picrocrocin)	Multiple emulsification	Whey protein concentrate (WPC), pectin and maltodextrin	Multiple emulsion	<ul style="list-style-type: none"> The multiple emulsions produced by WPC-pectin-maltodextrin along with 5% inner aqueous phase showed a high stability and low release profile Emulsions showed a high protection of crocin, safranal, and picrocrocin in the gastric condition 	Esfanjani et al. (2017)

It can also improve the encapsulation efficiency, loading capacity, and controlled release profile (Khoshakhlagh et al., 2017; Tampau et al., 2017).

3.4. Methods of encapsulation and their operating conditions

One of the most important factors affecting flavor retention during encapsulation is the selection of encapsulation method and optimum processing conditions. For example, fish oil microcapsules were produced from four combinations of carrier materials (maltodextrin and soybean soluble polysaccharide) using various drying methods, i.e. spray granulation, spray drying, and freeze drying. Spray granulation exhibited excellent encapsulation efficiency and produced highly stable fish oil microcapsules (Anwar & Kunz, 2011). Similarly, a spicy flavour, pepper oleoresin (Capsaicinoids), was encapsulated with Hi-Cap 100 carrier material using ultrasound emulsification, and supercritical fluid extraction with freeze drying. Of these methods, ultrasound emulsification showed a higher emulsification efficiency and stability, while the supercritical fluid extraction process resulted in a considerable loss of oleoresin by dissolution and promoted droplet volume expansion (de Aguiar, Silva, de Rezende, Barbero, & Martínez, 2016). In a similar fashion, the processing or operating conditions of the encapsulation method can also affect flavor retention. Getachew and Chun (2016) studied coffee oil flavor encapsulation by polyethyleneglycol using supercritical carbon dioxide. They observed that temperature, pressure and polymer-to-oil ratio have significant effect on coffee oil flavor encapsulation. Therefore, they optimized the processing conditions and found that a temperature of 40 °C, pressure of 260 bar and polymer-to-oil ratio of 6.5:1 was the best combination of process parameters that exhibited the highest oil retention of 79.78%. Likewise, in the study of essential oil-loaded starch nanoparticles formed by short glucan chains using *in situ* nano-precipitation method, the encapsulation efficiency significantly increased with an increase in complexation temperature from 30 °C to 90 °C (Qiu et al., 2017). In order to provide a clear understanding of encapsulation methods and their operating conditions; we have reported a vigorous and thorough discussion concerning the most important encapsulation techniques, processing mechanisms, and recent improvements or innovations under the following section “Encapsulation approaches”.

4. Encapsulation approaches

Various techniques are available for the encapsulation of food ingredients into the micro or nano scale. The methods include spray drying, spray chilling or spray cooling, freeze-drying, fluidized bed-coating, extrusion, co-crystallization, emulsification, molecular inclusion, rotational suspension separation, and coacervation (de Souza Simões et al., 2017). Broadly, these methods are divided into three categories, i.e. chemical methods (e.g. *in situ* polymerization, molecular inclusion); physico-chemical methods (e.g. emulsification, coacervation), and physico-mechanical methods (e.g. spray drying, freeze drying) (Jafari, Assadpoor, He, et al., 2008). The selection of method for encapsulation depends upon the properties of core and wall materials, expected release rate, processing steps, particle size, and the final application of the encapsulated particles (de Souza Simões et al., 2017). There are a number of methods that have been developed for micro encapsulation; however, for nano-encapsulation, careful selection of the method is required and the number of method is limited. Moreover, the techniques used for achieving nano-encapsulation are more complex than those of microencapsulation (Bakry et al., 2016; Ezhilarasi et al., 2013). However, in flavor encapsulation, around 80–90% encapsulates are produced by spray drying, followed by spray chilling (5–10%), melt extrusion (2–3%), and melt injection (~2%) (Gupta et al., 2016). Therefore, the most important techniques involved in micro and nano-encapsulation of flavor compounds using different carrier materials have been presented in Tables 1 and 2 and they have been

comprehensively discussed in the following section in terms of their process suitability, encapsulation efficiency, recent improvements and limitations.

4.1. Emulsification

Emulsification is a process of mixing two immiscible solvents and the resultant product is known as an emulsion. It is the most frequently used encapsulation technique. Emulsification is also used as a preparatory step for encapsulation methods, e.g. spray drying and freeze drying (Laokuldilok et al., 2016; Mehyar et al., 2014). Usually, emulsions consist of oil, water and stabilizer. For an oil-in-water emulsion, oil is known as the dispersed phase and water as the continuous phase, while the stabilizer acts as an interface between the water and oil phase, creating a protective layer around the oil droplets. The classification of an emulsion is in accordance to its dispersed phase, i.e. when oil is dispersed in water is known as oil in water (O/W) emulsion; on the other hand, when water is dispersed in oil is known as water in oil (W/O) emulsion (Bakry et al., 2016; Saifullah et al., 2016). Most flavor emulsions are oil in water type emulsions, since flavorings are mostly lipid in nature. Flavor emulsions are used mainly in liquid and semi-solid food products (e.g. ice cream, soft drink). Based on oil droplet size, emulsions can be classified as macro emulsions, micro emulsions and nano emulsions (Piorkowski & McClements, 2014). Micro and nano emulsions are ultrafine emulsions that show better functionality than macro emulsions in terms of stability, optical clarity, and control of release. Ultrafine emulsions can be prepared by using either a high energy approach (e.g., high-pressure valve homogenizers, micro-fluidizers, and sonication methods) or low energy approach (e.g., phase inversion and spontaneous emulsification) (Ezhilarasi et al., 2013; Lu, Kelly, & Miao, 2016; Saifullah et al., 2016). The high energy approaches require specially designed mechanical equipment, and therefore they can be expensive. On the contrary, low energy approaches do not require any sophisticated equipment, and they are simple, inexpensive and more energy efficient (Anton & Vandamme, 2011). However, the low energy approaches require high amounts of stabilizer, allow limited types of oil and surfactant, and produce unstable droplets at elevated temperatures (Saber et al., 2016).

4.1.1. High-pressure homogenization (HPH)

High-pressure homogenization (HPH) is commonly applied high energy method in the food industry. At first, a coarse emulsion is prepared by high shear mixer, and then this emulsion is passed through a narrow orifice with high speed under high pressure (100–2,000 bar). This step exposes the emulsion to a combination of intense disruptive forces (i.e. turbulence, shear, cavitation) to facilitate droplet disruption thus converting the coarse or large droplets into smaller ones, resulting in a final emulsion (Piorkowski & McClements, 2014; Saifullah et al., 2016). Lu et al. (2016) developed a *D*-limonene flavored organogel-based nano emulsion of monostearin and a medium chain triglyceride oil using HPH. According to the study, monostearin, medium chain triglyceride oil, and *D*-limonene were mixed at the weight ratio of 1.5:8.5:20 (w/w/w). *D*-limonene organogel was used as the oil phase, and Milli-Q water containing different amounts of Tween 80 surfactant was used as the water phase. The study revealed the smallest emulsion size of 36 nm. The nano emulsions exhibited a good physical stability and strong antimicrobial activity in comparison with free *D*-limonene (Zahi, Liang, & Yuan, 2015).

The carvacrol nanoemulsions (O/W) and nanocapsules were prepared using a high-pressure homogenization technique with maltodextrin and Tween 20. The study exposed the encapsulation efficiency of 49.3–76.4% and loading capacity of 48.7–69.2%. Nano-encapsulates showed better efficiency than nano-emulsions in order to encapsulate the carvacrol (Hussein et al., 2017). However, the desired characteristics of emulsions and better efficiency in flavor retention can be achieved by controlling the homogenization conditions. Recent

studies show that the emulsion droplet size may decrease with increasing homogenization pressure or number of passes, decreasing interfacial tension, increasing emulsifier adsorption rate, and decreasing continuous phase viscosity (Lu et al., 2016; Yuan, Gao, Zhao, & Mao, 2008; Zahi et al., 2015). There is usually a linear relationship between the logarithm of homogenization pressure (P) and the logarithm of the droplet diameter (d) i.e. $\log d \propto \log P$. To increase the emulsification efficiency and stability of an emulsion, the addition of sufficient amounts of emulsifier is also very important. At optimal conditions (e.g. high emulsifier levels, low interfacial tensions, and appropriate viscosity ratios of the dispersed and continuous phases), HPH with multiple passes can produce an emulsion with a droplet size of less than 100 nm (Piorkowski & McClements, 2014).

4.1.2. Microfluidization

The working principle of microfluidization is quite different from HPH. In this method, initially, the coarse emulsion is pumped through a channel under high pressure (up to 20,000 psi) (Ezhilarasi et al., 2013). The channel is designed in a pattern, which divides the main flow into two streams that are then made to impinge on each other at high velocity in an interaction chamber. When two fast-moving emulsion streams are collided in the interaction chamber, intense disruptive forces are generated, which leads to breakdown of coarse emulsion droplets, forming a fine emulsion. Several parameters, i.e. homogenization pressure, number of passes, emulsifier concentration, and phase viscosity ratio, can affect the emulsion droplet size similar to HPH processing (Piorkowski & McClements, 2014). α -limonene sub-micron emulsion particles were developed using microfluidization and ultrasonic techniques by Jafari, He, and Bhandari (2007a). In this study, spray drying was used to convert the α -limonene loaded emulsions into particles. Maltodextrin combined with modified starch, whey protein or Tween 20 surfactant were used. The results showed that microfluidization exhibited better performance in the production of small droplets (700–800 nm), narrow distributions, and a stable emulsion, as well as the highest retention (86.2%) of α -limonene in the powder (Jafari et al., 2007a). Some other studies also exposed that microfluidization is more efficient for the encapsulation of α -limonene and fish oil compared to sonication (Jafari, Assadpoor, Bhandari, & He, 2008; Jafari, He, & Bhandari, 2007b).

4.1.3. Ultrasonic technique

Food grade flavor emulsions with micro and nano-sized droplets can also be formed using ultrasound. High-intensity ultrasonic waves (frequency > 20 kHz) are used to create intense disruptive forces, resulting in a fine emulsion droplet formation (Anandharamkrishnan, 2014; Jafari et al., 2007a). Similar to other high energy approaches, a pre-mix coarse emulsion is used in the ultrasound technique. An ultrasonic probe is dipped into the sample or set in the flow stream of the sample to be homogenized. The probe produces intense disruptive forces at its tip through a combination of cavitation, turbulence, and interfacial waves. Ultrasonic homogenization can be done continuously or in batches (Leong, Wooster, Kentish, & Ashokkumar, 2009; Piorkowski & McClements, 2014). For lab-scale production, batch ultrasonic homogenization is suitable. On the contrary, continuous ultrasonic homogenizers are commonly used for the large-scale production of fine emulsions (Piorkowski & McClements, 2014). Roasted coffee oil mini-emulsions were prepared using poly L-lactic acid (PLLA) and poly hydroxybutyrate-co-hydroxyvalerate (PHBV) as carrier material through HPH and sonication. The results showed the successful encapsulation of major aroma compounds of coffee oil in nanocapsules. Sonication yielded the highest oil recovery for PLLA systems, while HPH led to the highest oil recovery for PHBV systems (Freiberger et al., 2015). However, the ultrasonic technique is particularly suitable for low viscosity fluids over highly viscous systems (Piorkowski & McClements, 2014).

4.1.4. Spontaneous emulsification

In this method, when two liquids (usually an organic phase and an aqueous phase) are mixed together at a particular temperature, the spontaneous formation of emulsion occurs. The emulsion is formed through the rapid diffusion of surfactant and/or solvent molecules from the dispersed phase to the continuous phase with the help of the chemical energy released due to the dilution process (Ghai & Sinha, 2012; Piorkowski & McClements, 2014). The emulsification operation of this method can be altered in different ways, such as changing the environmental conditions (e.g. temperature, pH, and ionic strength) and the mixing conditions (e.g. stirring speed, rate of addition, and order of addition). The droplets formed through this emulsification process can be controlled by varying the compositions of the two initial phases, as well as the mixing conditions (Piorkowski & McClements, 2014). The spontaneous emulsification approach was used to encapsulate limonene by using medium chain triglyceride oil and several types of Tween (40, 60, and 80). The study successfully produced nano-emulsions, while Tween 40 showed optically transparent and small droplets ($d < 30$ nm). Emulsions produced at optimum condition of oil phase composition and surfactant type can show thermal stability to coalescence when they were heated over 80 °C (Saberi et al., 2016). Furthermore, orange oil, food grade oil, and crocin loaded nano-emulsions were also successfully prepared by this approach (Chang & McClements, 2014; Komaiko & McClements, 2015; Mehrnia, Jafari, Makhmal-Zadeh, & Maghsoudlou, 2016).

4.1.5. Phase inversion emulsification

Phase inversion emulsification is a simple method that involves the slow addition of increasing amounts of an aqueous phase into an organic phase to induce a catastrophic phase inversion from a W/O to O/W form or vice versa by either changing the temperature (called phase inversion temperature method) or changing the composition (called phase inversion composition method or emulsion inversion point method) (Solans & Solé, 2012; Thakur, Villette, Aubry, & Delaplace, 2008). The phase inversion temperature (PIT) method includes a transitional phase inversion. The driving force in the PIT method is the alteration of physicochemical properties of the surfactant molecules (e.g. molecular geometry and solubility) by the action of temperature change. In contrast, emulsion inversion point (EIP) method involves a catastrophic phase inversion, where the ratio of the oil-to-water phases is altered, while the surfactant properties remain unchanged (Piorkowski & McClements, 2014). Flavor oils can be encapsulated by the EIP method. The nano-emulsions produced by this method showed the size of emulsion is $d < 200$ nm. The size of the emulsions depended on a number of factors, including oil type, surfactant type, surfactant-to-oil ratio, and initial surfactant location. The optimization of processing conditions is required for the production of fine emulsions using low surfactant concentrations (Ostertag, Weiss, & McClements, 2012). However, very few studies employ the phase inversion emulsification approaches for the encapsulation of flavor, and therefore there is a huge scope to work with those.

4.1.6. Miscellaneous emulsification techniques

Some other novel approaches, such as emulsion-solvent evaporation, emulsion ionic gelation crosslinking, and multiple emulsification are able to produce stable nano particles. Using emulsion-solvent evaporation, the vanillin-loaded polylactic acid nanoparticles produced exhibited a homogeneous particle size of ~240 nm with vanillin encapsulation efficiency of 41% (Dalmolin, Khalil, & Mainardes, 2016). An *in vitro* release study showed a slow and sustained release of this encapsulated vanillin. Furthermore, the vanillin-loaded polylactic acid nanoparticles offered good physical and chemical stability during 90 days of storage at room temperature (Dalmolin et al., 2016). The emulsion-ionic gelation crosslinking approach produced eugenol-loaded chitosan nanoparticles of < 100 nm. The results revealed a loading capacity of 12%, encapsulation efficiency of 20% and a

spherical shaped particle. The extrusion of eugenol-loaded chitosan nanoparticles ensured the improved thermal stability of encapsulated eugenol up to 155 °C compared to free eugenol (Woranuch & Yoksan, 2013). Ionic gelation/ionotropic gelation forms inter and intramolecular cross-linkages when positively charged polymers come in contact with specific polyanions (Janes, Calvo, & Alonso, 2001). The multiple emulsification method can also produce highly stable nano-emulsions with controlled release profile using whey protein concentrate (WPC), pectin and maltodextrin. The resultant nano-emulsions containing crocin, safranal, and picrocrocin were protected from gastric conditions (Esfanjani et al., 2017). In addition, Fioramonti, Rubiolo, and Santiago (2017) recently reported the multilayer emulsion of flax seed oil with maltodextrin (MD) and whey protein isolate. This encapsulation method provided high encapsulation efficiency (> 90%), low water activity (0.14–0.33).

4.2. Spray drying

Spray drying is the most widely used, and economic technique for the encapsulation of food bioactive compounds (Shishir & Chen, 2017; Shishir, Taip, Aziz, Talib, & Sarker, 2016). Spray drying is used for the production of flavor powders. It offers a high production rate with minimum operating costs, which allows the spray drying to be used in an industrial scale for the encapsulation of flavor compounds (Sanchez-Reinoso et al., 2017; Sultana et al., 2017). Furthermore, spray-dried flavor powder is used in a wide range of food materials in order to enhance product acceptability in the eyes of the consumer (Nedović, Kalušević, Manojlović, Petrović, & Bugarski, 2013).

The basic principle of spray drying includes the atomization of a feed sample into a drying chamber through atomizer (e.g. spray nozzle), drying of liquid droplets inside the drying chamber, and powder recovery through a cyclone separator (Masters, 2002; Shishir & Chen, 2017). For preparation of the feed sample for spray drying encapsulation, core materials are dissolved in a dispersion of a single or a combination of carrier materials in order to form an emulsion or a suspension. The dispersed carrier materials must have good solubility in water since only water-based dispersions are useful for spray drying purposes. Furthermore, carrier materials must be low viscosity at high concentration, provide good emulsification and film-forming characteristics, and have efficient drying properties (Shishir, Taip, Saifullah, Aziz, & Talib, 2017; Sultana et al., 2017). The mixing of core compounds and carrier materials can be performed through high-speed mixing or high-shear emulsification using colloid mills in order to form a coarse emulsion. Then the emulsion can be further processed using different types of mechanical means, such as high-pressure homogenization, microfluidization, and ultrasonic emulsification. Before beginning spray drying, the formed emulsion must be stable over a certain period of time (Liu et al., 2001). The common carrier materials used in spray drying are mainly hydrophilic polysaccharides (e.g. maltodextrins, chitosan, alginate and different types of gums) and proteins (e.g. whey protein), whereas the core materials can be active hydrophobic or hydrophilic molecules (Bakry et al., 2016; de Souza Simões et al., 2017).

The formed emulsion is used as the sample feed during spray drying. It is pumped into the drying chamber through the atomizer. There are two types of atomizer which are widely used: a high-pressure nozzle or a centrifugal wheel (Masters, 2002; Phisut, 2012). The atomizer breaks down the liquid feed into small droplets and sprays it into hot air. The powder particle size depends on the atomizer pore size. Three types of air flow are used inside the drying chamber: co-current flow, counter-current or mixed flow type. However, co-current type air flow is generally applied for the encapsulation of food flavor compounds (Jafari et al., 2008). Finally, the encapsulated dry particles are separated through cyclone separator and deposited into the receiver. The total time required for altering the liquid droplet into powder form is few milliseconds to few seconds (Masters, 2002; Phisut, 2012), which is

very suitable for heat sensitive compounds (e.g. flavor).

The feed rate and viscosity of feed have significant effects on the spray drying process. Emulsions with higher viscosity affect the atomization and consequently the drying process. Highly viscous emulsions can be the cause of nozzle blockages, increased wall deposition, and poor yield. The retention of flavor compounds during spray drying is affected by the composition and properties of the emulsion and by the drying conditions (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). The temperature of drying medium (hot air) is usually set at different levels according to the nature of the core material. The rapid drying of droplets keeps the core temperature below 100 °C in spite of the high temperatures (> 150 °C) used in the process (Jafari et al., 2008). It is very important to keep the temperature low for flavor core materials because flavor may contain various components with low boiling points, and therefore it is possible to lose certain aromatics during the drying process (Apintanapong & Nookhorm, 2003).

A successful encapsulation shows maximum retention of core material and minimum deposition of core material in the powder particle wall. Spray drying still shows limited retention of volatile flavor compounds particularly those have low boiling point, even if optimum drying conditions are followed (Reineccius, 2004). There are two theories have been developed in this regard and the first one is the selective diffusion theory. According to this theory, when the emulsion droplets become dry to a moisture content of about 23 to 7% ($a_w < 0.90$), the dried surface acts as a semi-permeable membrane allowing the continuous evaporation (or diffusion) of water and the efficient retaining of flavor molecules (Reineccius, 2001, 2004). The diffusivity of flavor dramatically reduces compared to water molecules after the formation of dry surface. Hence, more losses of flavor usually occur at the initial of drying process. The second theory is the relative volatility of flavor compounds compared to water. The flavor loss is proportional to the relative volatility of flavor and thus more volatile flavor losses occur at a higher rate in the initial stage of drying (Reineccius, 2001, 2004). In spray drying, there are three points where flavor losses occur. Firstly, flavor losses occur during atomization, as it creates large surface area and higher turbulence in emulsion system. Secondly, flavor losses occur when the droplets are exposed to the drying chamber and start drying. At this stage, flavor molecules diffuse through the outer membrane of the droplets with water molecules, since the droplet wall is not hard enough to entrap the flavor molecules. Thirdly, the final flavor losses occur when the water droplets exceed their boiling point and form bubbles inside the droplet that mostly burst out to the surface together with volatile compounds. It is shown that losses during this third stage (i.e. during morphological development) are greater than during atomization and the beginning of surface drying (Hecht & King, 2000).

In recent studies, cocoa aroma compounds were microencapsulated through spray drying using maltodextrin and Hi-cap 100. The study revealed that the process yield reached up to 58.77%. The carrier material Hi-cap 100 showed a higher retention of cocoa aroma (22.6–32.5%) and better micro-structural properties in cocoa aroma microcapsules compared to maltodextrin (Sanchez-Reinoso et al., 2017). Similarly, Sultana and coworkers reported the maximum retention of β -limonene and ethyl hexanoate by spray drying microencapsulation were 48% and 45%, respectively, with *Saccharomyces cerevisiae* used as a carrier material (Sultana et al., 2017). The results revealed that more than 50% of flavor and aroma compounds were lost during spray drying microencapsulation. On the contrary, maximum microencapsulation efficiency by spray drying was reported up to 68.91%, 87.34% and 80.2% for white champaca extract, sweet orange oil flavor, and oregano flavor in skimmed milk powder, respectively (Samakradhamrongthai, Thakeow, Kopermsub, & Utama-ang, 2016; Liu, Xu, & Wang, 2012; Baranauskiene et al., 2006).

In the case of nano-encapsulation, spray drying was only used to convert a suspension of colloidal nanoparticles into nanostructured powder form (de Paz et al., 2012). Jafari et al. (2007a) studied the

encapsulation of nano-emulsion particles of D-limonene by spray drying. Maltodextrin in combination with a surface active biopolymer, i.e. modified starch (Hi-Cap 100), whey protein concentrate or a surfactant (Tween 20), was used as the wall material. Hi-Cap exhibited better retention of D-limonene by around 86.2% in spray-dried particles. However, nano-spray drying has recently been applied for the development of nano-encapsulated bioactive products. Nano-spray drying can offer nano scale particle sizes with enhanced stability and improved bioavailability (O'Toole et al., 2012; Pérez-Masiá et al., 2015).

4.3. Spray chilling/cooling

Spray chilling is one of the most promising emerging methods in the microencapsulation of food flavor compounds. It solidifies the atomized liquid droplets into particles from a few microns to millimeters. This encapsulation technique is also referred to as spray cooling, spray congealing, or prilling (Lopes, Speranza, & Macedo, 2016; Oxley, 2012). The basic principle of spray chilling is similar to spray drying, which includes atomization, particle development and particle collection (de Souza Simões et al., 2017). The key difference of spray chilling from spray drying is the use of a cooling chamber instead of a drying chamber. The temperature of the cooling chamber is kept below the melting point of the carrier material used, which can overcome some limitations of spray drying, particularly the thermal loss of flavor. Another difference from spray drying and freeze drying is that spray chilling allows only lipid-based carrier materials, i.e. fats, waxes, polyethylene glycols, fatty acids, and fatty alcohols (Đordevic' et al., 2015; Sillick & Gregson, 2012). Therefore, it offers extra benefits to microcapsules with enhanced stability at gastrointestinal conditions (Arslan-tontul & Erbas, 2017). Although spray chilling and spray cooling approaches can alternatively be recognized (Oxley, 2012), theoretically they have distinct temperature range of the cooling chamber on the basis of carrier materials used. The most common encapsulating materials used in spray chilling are fractionated or hydrogenated vegetable oil with a melting point of 32–42 °C. In contrast, spray cooling includes vegetable oils or other materials with a melting point of 45–122 °C (Đordevic' et al., 2015).

Ginger oleoresin flavor-loaded lipid microparticles were developed by using saturated fatty acids, i.e. palmitic acid with oleic acid or palm fat through the spray chilling technique (Oriani et al., 2016). At first, saturated fatty acids were melted at 85 °C using a hot water bath. Afterwards, five formulations of fatty acids, i.e. palmitic acid (75–90%), oleic acid (5–15%), and palm fat (5–15%) were prepared. Then, 10% of ginger oleoresin was added in each formulation and mixed properly before being fed into the spray chiller. Solid lipid microparticles were formed within the cooling chamber, where the inlet air temperature was 7 °C. The study exhibited a high retention of pungent and volatile compounds around 96% and 75%, respectively. Lipid carriers with a higher concentration of saturated fatty acids, i.e. palmitic acid and palmitic acid with palm fat, showed better retention of ginger oleoresin. The particles exhibited a spherical shape with rough surface and narrow size distribution ranging from 39.8 to 43.2 μm, as well as having ginger oleoresin dispersed over the entire particle (Oriani et al., 2016). Sillick and Gregson (2012) produced crystalline particles for the encapsulation of five flavor compounds, i.e. limonene, nicotine, methyl salicylate, cinnamic aldehyde and neobee using the spray chilling technique with anhydrous sugar alcohol (erythritol) as a carrier material. The study showed maximum encapsulation efficiency of cinnamic aldehyde and limonene at around 90% for the loading capacity of 10%. The encapsulation efficiency decreased gradually with increasing oil load to around 63% of encapsulation efficiency for 30% of oil load. Crystalline particles exhibited significant protection to flavor compounds from volatilization until melting of the erythritol at ~120 °C. This encapsulation approach also offered very low melt viscosity and no drying requirement (Sillick & Gregson, 2012).

Therefore, spray chilling or spray cooling is a simple, economic and easy to scale up technique, which does not require the use of organic solvents or the application of high inlet air temperature (Đordevic' et al., 2015). Furthermore, around 5% of encapsulated flavors are spray chilled products due to the minor or no loss of flavors by dispersion (Lopes et al., 2016). However, spray chilling does not have enough control on particle size and yields are moderate for small batches (Lopes et al., 2016). Moreover, spray chilling only permits lipid-based carrier materials, which limits its extensive application (Đordevic' et al., 2015). Hydrophobic flavors are easily miscible with lipid carrier materials, and therefore creates a poor barrier which can cause the degradation of many flavor compounds (Benczedi, 2002). The application of non-miscible carrier materials, e.g. sugar alcohol (erythritol) can enhance the barrier properties of encapsulated compounds (Sillick & Gregson, 2012).

4.4. Electro-spinning and electro-spraying

Electro-spinning and electro-spraying are novel approaches for encapsulation of flavor compounds, which are simple, economic and scale up-able techniques, as well as have versatile application (Kayaci et al., 2014; Koo et al., 2014). In the electro-spinning approach, nano-fibers are produced from a polymer solution in a spinneret by a high voltage potential, while particles are produced from a polymer solution in the nozzle through liquid atomization by electric forces (Esfanjeni & Jafari, 2016). The principles of these techniques are the same, but different only in the concentration of polymer solution. In the case of high concentrations of polymer, the jet from the spinneret is stabilized and lengthened, and therefore nano-fibers are developed by electro-spinning. On the contrary, in electro-spraying, the jet with a low polymer concentration from the nozzle is destabilized, and therefore produces fine droplets or particles (Esfanjeni & Jafari, 2016). These approaches are non-thermal treatments and there is no chance of thermal degradation for heat sensitive compounds. Therefore, they are very suitable for the encapsulation of flavor compounds. They allow the production of fibers and particles with a narrow size distribution. They prevent droplet agglomeration and coagulation complications. They are able to produce both micro and nano-scale encapsulate systems that offer a versatile range of applications in food and pharmaceutical industries (Esfanjeni & Jafari, 2016; Koo et al., 2014; Tampau et al., 2017; Yao, Chang, Ahmad, & Li, 2016). However, electro-spinning does not permit many proteins to be used alone, and needs surfactant or plasticizer with a protein solution to develop electro-spun fibers. Oppositely, electro-spraying does not have such limitations (Tarhini, Greige-Gerges, & Elaissari, 2017). Electro-spraying can also create smaller particle sizes compared to nano-spray drying (Pérez-Masiá et al., 2015). Beads and porous structures made by electro-spinning can weaken the sustained release delivery system (Esfanjeni & Jafari, 2016). Moreover, low throughput of electro-spinning approach can bound the huge scale production and commercial application (Yang et al., 2017).

To date, several novel approaches have been introduced into electro-spinning and electro-spraying, i.e. coaxial and emulsion approaches. The successful applications of these novel approaches for the encapsulation of flavor compounds have also been reported by several recent studies, e.g. rose hip seed oil by coaxial electro-spinning (Yao et al., 2016), peppermint oil by coaxial electro-spraying (Koo et al., 2014), carvacrol by emulsion electro-spinning (Tampau et al., 2017), and D-limonene by emulsion electro-spraying (Khoshakhlagh et al., 2017). The coaxial approach comprises two concentrically aligned capillaries for the development of fibers or particles with a core-shell structure. This innovative approach offers one-step co-encapsulation of multiple compounds with a diverse solubility, higher encapsulation efficiency, higher stability, and enhanced controlled release profile of encapsulated compounds compared to single nozzle electro-spinning or electro-spraying (Matsuura & Maruyama, 2017; Yao et al., 2016). The emulsification approach allows the encapsulation of immiscible

compounds with high encapsulation efficiency, high loading capacity, enhanced thermal stability, and sustained release profile of flavor compounds (Camerlo, Vebert-nardin, Rossi, & Popa, 2013; Khoshakhlagh et al., 2017; Tampau et al., 2017).

4.5. Freeze drying

Freezing of water content in a solution or suspension and followed by evaporating the water molecules from the solution or suspension at low temperature is the basic principle of freeze drying or lyophilization technique (Anandharamakrishnan, Rielly, & Stapley, 2010). The whole drying process occurs under low temperature and pressure. Hence, it is very suitable for heat-sensitive food compounds including flavors, aromas, enzymes etc. It shows higher retention of volatile compounds and natural colors, as well as produces powder with rapid solubility (Anandharamakrishnan, 2014; Madene et al., 2006; Raja, Taip, Azmi, & Shishir, 2017). Freeze drying offers more suitable attributes to flavors in comparison with other drying and encapsulation methods (Buffo & Reineccius, 2001; Chen & Zhong, 2015). There are four basic steps involved in freeze drying: freezing, primary drying, secondary drying, and final treatment (Abdul-Fattah, Kalonia, & Pikal, 2007).

Freezing: This is the first step of the freeze drying process. The substance (raw material) is frozen to a crystallized ice form. The size of crystal depends upon the cooling rate and initial intensity of cooling temperature and end temperature of cooling. All of the water molecules in the food material form crystalline ice, while food components remain in an amorphous and glassy state, i.e. do not form crystal (Abdul-Fattah et al., 2007).

Primary drying: This step refers to the removal of ice by sublimation under vacuum that occurs, when the necessary energy for latent heat is supplied to the frozen food material. Sublimation starts at the ice surface and occurs at the interface between the frozen and dried part of the material. At the beginning, the drying rate becomes high because of the low resistance to both heat and mass transfer in the initial stages of drying. As the drying process proceeds towards the core of the material, the heat and mass transfer rate decreases, since the dry layer around the frozen portion of the material acts as an insulation material, and therefore blocks the heat transfer to the ice front and the mass transfer from the ice front (Abdul-Fattah et al., 2007).

Secondary drying: This step begins when all of the ice crystals are removed from the material and the bound water in the drying material remains. The temperature and pressure in the drying chamber should be the same as the primary drying step. This step takes a longer time and usually is one-third of the drying time (Anandharamakrishnan, 2014). The drying process is continued until the moisture content of the product is reduced to less than 5% (for food materials).

Final treatment: The dried food material should be preserved properly to prevent rehydration and lipid oxidation reactions. Therefore, after finishing the drying process, the drying chamber is filled with an inert gas, particularly nitrogen gas which is preferred for food materials (Abdul-Fattah et al., 2007).

There are two major factors which help to produce higher quality freeze-dried flavor powders. Firstly, the whole drying process is carried out under vacuum conditions, so there is a practical absence of air that does not allow oxidative degradation. Secondly, the drying temperature is lower than the ambient temperature. Hence, the compounds (e.g. flavors) that are very susceptible to oxidative and thermal degradation can be dried under vacuum with minimal physical and chemical damage (Chen & Zhong, 2015). The sample preparation for freeze drying is almost similar to spray drying. The flavor and aroma compounds, stabilizer, and water are homogenized to obtain the desired particle size. Since freeze drying alone cannot produce micro or nanoparticles, it is just a technique of water removal from droplets, and therefore the final characteristics of freeze-dried particles rely on a suitable encapsulation method (e.g. emulsification) to form the required size of droplets, or to break down the droplets into micro or nano size form

(Ezhilarasi et al., 2013; Varshosaz, Eskandari, & Tabbakhian, 2012).

Aliphatic and aromatic alcohols were encapsulated into an amylose molecular complex by sealed heating and freeze drying (Feng et al., 2018). At first, a dispersed amylose suspension is prepared by heating and was cooled. The flavor compounds were then added to the amylose suspension and allowed to react. Then it was taken for ethanol washing and centrifugation. Finally, the precipitates were treated by freeze drying in order to get flavor powder. The results showed that the encapsulation efficiency and yield varied from 39% to 47% and 45%–50%, respectively. All amylose-flavor inclusion complexes exhibited excellent thermal stability. The amylose-n-heptanol inclusion complex showed greater stability than other complexes (Feng et al., 2018). Cardamom flavors, i.e. α -limonene, 1,8-cineole, myrcene, terpineol and linalool, were encapsulated using whey protein isolate (WPI), guar gum (GG), and carrageenan (CG) through emulsification and freeze drying (Mehyar et al., 2014). The study revealed that the microcapsules containing only WPI exhibited the highest flavor entrapment (7.5%) and microencapsulation efficiency (98.5%). The 30% WPI and 30% WPI + GG formulations showed better retention ability of 1,8-cineole and α -limonene during storage (Mehyar et al., 2014). Similar recent studies have also been reported on pepper oleoresin spicy flavor (de Aguiar, Silva, Alves De Rezende, Barbero, & Martínez, 2016), caffeine (Fuciños et al., 2017), and peppermint oil (Chen & Zhong, 2015).

However, higher energy requirements and long processing time (more than 20 h) are the major disadvantages of freeze drying (de Souza Simões et al., 2017). Moreover, freeze drying produces open porous structured irregular particles that is not effective, when prolonged release of flavor compound is required (Hundre, Karthik, & Anandharamakrishnan, 2015).

4.6. Spray-freeze-drying

In the recent times, researchers show their interest on spray-freeze drying as alternative technique, which offers the benefits of freeze drying and spray drying (Anandharamakrishnan, 2014; Hundre et al., 2015). There are three main steps involved in SFD: (i) atomization of a liquid sample, (ii) solidification of liquid droplets by freezing, (iii) sublimation at low temperature and pressure in order to dry the frozen droplets into powder particles (Ishwarya, Anandharamakrishnan, & Stapley, 2015; Leuenberger, 2002). In this technique, feed solution can be directly sprayed into a cold dry gas, a cryogenic liquid or a cryogenic liquid containing gaseous headspace. The particles size distribution is directly affected by the atomization method as droplet size does not change as it falls in the drying chamber. Hence, the final product particle size distribution is almost identical with the atomized liquid droplet size distribution (Ishwarya et al., 2015). Spray-freeze dried vanillin flavor microcapsules were prepared by using β -cyclodextrin (β -CD), whey protein isolate (WPI) and their combinations (β -CD + WPI) and were compared with both spray-dried and freeze-dried flavor microcapsules. Spray-freeze dried vanillin microcapsules prepared with WPI exhibited better thermal stability than the spray-dried and freeze-dried microcapsules. However, spray-freeze dried vanillin microcapsules showed numerous fine pores on their surface (Hundre et al., 2015). Ishwarya, Anandharamakrishnan (2015) reported spray-freeze drying of a coffee solution gave 93% volatile aroma retention in the final powder; however, for spray drying and freeze drying this figure was 57% and 77% respectively. Despite having advantages over spray drying and freeze drying in terms of product quality and higher encapsulation efficiency, the high fixed and operating cost is a major limitation of this method. The operating cost of spray-freeze drying for a product drastically increases for frozen solutions that have low eutectic (T_e) or glass transition temperatures (T_g) and hence require low drying temperatures (Ishwarya et al., 2015).

4.7. Extrusion

The encapsulation of flavor compounds by extrusion is a relatively new approach compared to spray drying. Extrusion processing includes the passing of a carrier solution mixed with active compounds through a nozzle into a gelling environment. In laboratory scale production, the solution-containing carrier and active compounds are loaded into a syringe and passed through a needle into gelling conditions to form gelation (Jia, Dumont, & Orsat, 2016). In this process, the pressure and temperature are generally kept below 100 psi and 118 °C, respectively. Therefore, extrusion is very suitable for heat sensitive compounds, and it has intensive applications for the microencapsulation of flavor compounds (Khor, Ng, Kanaujia, Chan, & Dong, 2017; Rodríguez et al., 2016; Tackenberg et al., 2015). Furthermore, microcapsules produced by extrusion generally show less porous structures than spray-dried microcapsules. In extrusion, several mechanisms are used to form droplets based on the interaction of gravitational, surface tension, impulse, and frictional forces. Therefore, the extrusion technique can be classified into simple dripping or prilling, electrostatic extrusion, coaxial airflow, vibrating jet/nozzle, jet cutting, spinning disk atomization, and melt extrusion. After the formation of droplets, they are instantly hardened to capsules by either physical (e.g., cooling or heating), or chemical process (e.g. gelation) (Rodríguez et al., 2016; Đorđević et al., 2015).

Calcium alginate/D-limonene beads were produced by electrostatic extrusion (Lević et al., 2015). According to the study, D-limonene was firstly added into Na-alginate solutions under vigorous mixing in order to form D-limonene/Na-alginate emulsions. Then the emulsions were taken into an electrostatic immobilization unit equipped with a high voltage unit, magnetic stirrer and protective cage. The electrostatic extrusion system altered the emulsions into spherical droplets or calcium alginate beads through a blunt stainless steel needle using a syringe pump. In order to create dried forms of beads, they were further air-dried at 25 °C for 48 h. The study exhibited efficient immobilization of D-limonene within calcium alginate beads (immobilization efficiency 50–77%). Furthermore, calcium alginate beads were able to provide thermal protection to D-limonene up to 200 °C (Lević et al., 2015). Orange terpenes were encapsulated in maltodextrin-sucrose carrier matrix using counter-rotating twin screw extrusion process. Around 67% of flavor retention was reported for the loading capacity of 4.1%. The authors explained the reason of flavor loss was due to the insufficient mixing during the process and flavor evaporation after extrusion (Tackenberg, Krauss, Schuchmann, & Kleinebudde, 2015). In a further study, three factorial optimization design was performed to investigate the effect of barrel temperature, powder feed rate, and active pharmaceutical ingredient content on the retention of flavor compounds. The results revealed that a maximum of 6% loading of orange terpenes was obtained with the encapsulation efficiency of 86.7%. The orange terpene microcapsules were also highly stable during storage of 12 weeks (Tackenberg et al., 2015).

Quercetin is a natural flavonoid having excellent health promoting and medicinal benefits, but it has very bitter taste that discourages the consumers' attraction to quercetin enriched products. Furthermore, it is very sensitive to oxidative degradation (Kawabata, Mukai, & Ishisak, 2015). Recently, the bitterness of quercetin was encapsulated by using carnauba wax, shellac and zein through hot-melt extrusion. The quercetin loaded microcapsules exhibited significantly reduced dissolution rate in the simulated salivary pH (6.8) in the order of zein > carnauba wax > shellac. *In vitro* bitterness analysis by electronic tongue ensured the good taste masking efficiency of the quercetin loaded microcapsules (Khor et al., 2017). However, the extrusion process is two times more expensive than spray drying. The use of screw extruders at high pressure can cause flavor loss, and therefore optimization is required. Moreover, the particle size produced by extrusion is larger and the loading capacity of flavor is relatively low compared to spray drying that can limit its commercial application (Rodríguez et al., 2016).

4.8. Coacervation

Coacervation has been well known as an encapsulation method for long time, while recently it has been used in food industries for encapsulation of food flavor compounds. Coacervation involves liquid-liquid phase separation of colloid particles from a solution and creates a new phase, which is called coacervate (Shishir et al., 2018; Yang et al., 2014). The phase separation can be classified as aqueous and non-aqueous phase separation depending on whether water is used as the solvent. In the case of flavor encapsulation, aqueous separation is the most common, since flavor components are hydrophobic in nature. The basic mechanism involved in this method is the formation of an emulsion and subsequent precipitation of the continuous phase around the droplets of the discontinuous phase. This method involves a three-phase system, which includes a manufacturing vehicle (solvent), the material to be encapsulated, and the coating material. There are three major stages in coacervation processing: formation of the three immiscible phases, deposition of the wall material around the core material, and solid microcapsule formation through shrinkage and solidification (Bakry et al., 2016).

Formation of the three immiscible phases: Phases are formed during mixing. Core material, coating material, and continuous liquid phase are separated in this step. In cases of food flavor and aroma encapsulation, the selection of coating materials are strictly limited by food additive regulations and gelatin is used in most instances.

Deposition of the wall material around the core droplets: This step involves interfacial sorption of the hydrophilic phase on the droplets of core material. The adjustment of pH and temperature of the solution is needed for the development of capsules, which allow the encapsulant coming out from the solution, coagulation at the surface of the droplets and formation of the wall coating. The droplet wall coating remains in liquid form and needs hardening at this stage.

Shrinkage and solidification: In order to form a solid and hardened capsule, heating, desolvation, or cross-linking techniques can be used.

The coacervation technique is classified as simple or complex according to number of colloidal solutes in the system. When the system contains only one colloidal solute (e.g. only gelatin) it is called simple coacervation. On the other hand, while the system contains more than one solute (e.g. gelatin and gum acacia) it is called complex coacervation (Bakry et al., 2016; de Souza Simões et al., 2017). In simple coacervation, a nonsolvent or another chemical is added, which competes for solubility with a colloidal solute, and consequently, protein precipitation takes place and a protein-rich coacervation phase is formed. In contrast, complex coacervation involves the addition of oppositely charged hydrophobic colloids. The droplet shell of complex coacervates is composed of a negatively charged hydrocolloid molecule (e.g. gum arabic) and a positively charged hydrocolloid molecule (e.g. gelatin) (Koupantsis et al., 2016; Shishir et al., 2018). Complex coacervation has better encapsulation functionalities than that of simple coacervation, and therefore complex coacervation is a better alternative for food flavors (Bakry et al., 2016). The basic steps of coacervation method presented in Fig. 3.

Sutaphanit and Chitprasert (2014) developed holy basil essential oil-loaded alginate microcapsules by using the simple coacervation approach. The optimal design using response surface methodology (RSM) was found at a gelatin concentration of 11.75% (w/v) and oil amount of 31 mL, which ensured the highest yield, oil content, and encapsulation efficiency of 98.80%, 66.50%, and 95.41%, respectively. Microcapsules also exhibited less loss of oil content during storage at 60 °C for 49 days equivalent to 25 °C for 18 months. However, the particle mean diameter was around 392 µm with a sponge-like structure containing a large number of micron-sized pores. On the contrary, β-pinene was encapsulated using sodium caseinate, whey protein isolate, carboxymethylcellulose (CMC), and reticulating agents (i.e. glycerol and tannic acid) through complex coacervation (Koupantsis et al.,

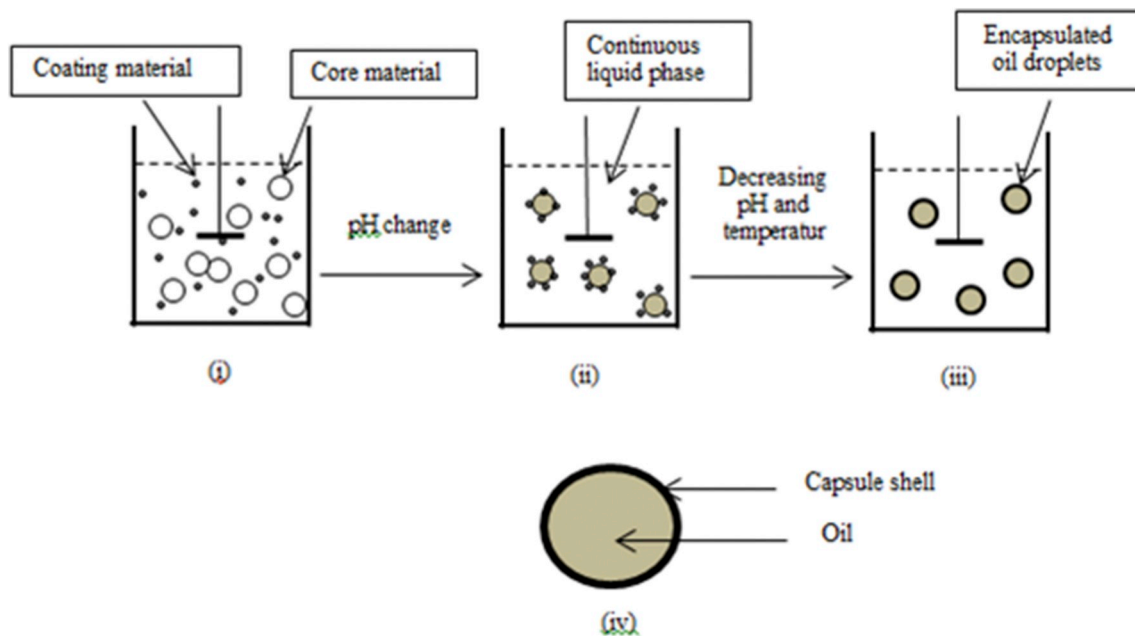


Fig. 3. The basic steps of coacervation method: (a) mixing of the three immiscible phase, (b) deposition of the coating material around the core material droplets, (c) formation of capsule wall, (d) single droplet produce by coacervation method.

2016). The results showed that sodium caseinate-CMC microcapsules with the addition of glycerol exhibited greater loading capacity and encapsulation efficiency reached up to ~55% and ~90%, respectively. Glycerol addition enhanced encapsulation efficiency two fold, while tannic acid was unsuccessful. Moreover, glycerol addition showed higher retention of volatile compounds in the microcapsules during storage at low relative humidity (RH) and temperature (0% RH, 25 °C) (Koupantsis et al., 2016; Koupantsis & Paraskevopoulou, 2017). Other studies also reported the successful encapsulation of vanilla oil and sweet orange oil by complex coacervation with high loading capacity, enhanced flavor retention, and improved thermal stability (Jun-xia et al., 2011; Yang et al., 2014).

Therefore, the coacervation approach is considered more efficient in flavor retention than spray drying because it does not require high temperature, offers high core loading capacity, high encapsulation efficiency, and a greater controlled release of bioactive compounds (de Souza Simões et al., 2017). However, the technique also has some limitations, i.e. high production cost depends on many factors (e.g. pH, ionic strength), there is inadequate stability in different aqueous solutions, and less control on particle size and particle accumulation (Jia et al., 2016; Joye & McClements, 2014).

4.9. Fluid bed coating

Fluid bed coating is also known as air suspension coating, fluidized bed processing or spray coating, in which a coating is applied on to the particles suspended in air (Bakry et al., 2016). Batch and continuous are two types of fluid bed coating methods. There is a wider choice of coating material compared to spray drying. The coating materials include aqueous solutions of cellulose or starch derivatives, proteins or gums, but also emulsifiers (Bakry et al., 2016). There are three basic steps involved in this process: Firstly, there is fluidization of the particles to be coated into the coating chamber, with the help of an air stream. Then, the coating material is sprayed by a nozzle on to the particles. Finally, the solvent of coating material is evaporated by hot air, and therefore the coating material adheres to the particles (Bakry et al., 2016; Madene et al., 2006). This technique can be used to develop a second coating to spray-dried products or to products with a sensitive core, such as oils, flavors and aromas (Nedović et al., 2013).

This technique allows a greater range of coating materials in comparison with traditional spray drying, and is more economic in operational cost and time compared to freeze drying (Đorđević et al., 2015; de Souza Simões et al., 2017). However, this technique is not heavily explored for the encapsulation of flavor compounds compared to spray drying and freeze drying. Recently, Anwar and Kunz (2011) studied the effect of different drying methods, i.e. fluidized bed coating, spray drying and freeze drying on the stabilization of fish oil microcapsules with maltodextrin, soybean soluble polysaccharide (SSPS), hydroxypropyl beta-cyclodextrin (HPBCD) and octenyl succinic anhydride starch (OSA-starch). Fluidized bed coating was reported as the most suitable technique that ensured maximum ranges of encapsulation efficiency (96.10–99.39%) in all matrices. Microcapsules produced with SSPS and OSA-starch showed the higher stability in the order of fluidized bed coating > spray drying > freeze drying during the storage of 5 weeks at 21 °C. Similarly, fluidized bed-coated menthol particles were produced using gelatin, oil-gelatin emulsion, modified starch, and aquacoat (Sun, Zeng, He, Qin, & Chen, 2013). Fluidized bed coating showed better encapsulation efficiency of menthol than spray drying. Fluidized menthol particles exhibited controlled release of menthol in water than spray-dried particles. Among the carrier systems, the gelatin emulsion carrier was more effective and showed enhanced control of menthol release and maximum encapsulation efficiency (~95%) (Sun et al., 2013). However, it takes a relatively long coating time and a substantial amount of coating materials to ensure complete covering (Nedović et al., 2013).

4.10. Molecular inclusion in cyclodextrins

Cyclodextrins (i.e. α -, β -, γ -cyclodextrin) are an enzymatically modified form of starch molecules. The most common type of cyclodextrin is β -cyclodextrin (β -CD) (Kfoury, Hadaruga, Hadaruga, & Fourmentin, 2016). A typical application of cyclodextrin is to protect the unstable components and to provide high added value, particularly to flavor compounds by means of encapsulation. The molecular structure of cyclodextrin is like a hollow truncated cone. The size of the inner cavity is in the nano range. The inner lipophilic cavity of cyclodextrin entraps the flavor or aroma molecules of the right size via hydrophobic interactions (so-called molecular inclusion), whereas its

external surface shows hydrophilic characteristics, and helps to dissolve it in water (Kfoury et al., 2016; Zuidam & Heinrich, 2010). Zhu, Xiao, Zhou, and Zhu (2014) developed a β -CD molecular inclusion complex in order to encapsulate sweet orange flavor. According to the study, β -CD is dissolved in warm water maintained at 35 °C followed by the slow addition of the sweet orange flavor to the warm β -CD solution. The mixture is then continuously stirred for 3 h at 35 °C. When its temperature decreases spontaneously to room temperature, the solution is kept overnight at 5 °C. The cold precipitated material is then recovered by vacuum filtration. The precipitate is washed by alcohol and dried in a convection oven at 50 °C for 24 h. Thus, the sweet orange flavor-loaded β -CD molecular inclusion complexes are prepared. The study revealed that the orange flavor β -CD inclusion complexes can form large aggregates in water. The thermogravimetric curve of the flavor- β -CD inclusion complex was downward sloping from room temperature to 250 °C (Zhu et al., 2014). There are a number of studies have been reported on the improved characteristics of flavor and aroma after encapsulation with different cyclodextrins, e.g. carvacrol in α -CD, β -CD, and HP- β -CD (Liang, Yuan, Vriesekoop, & Lv, 2012), Cinnamaldehyde in α -CD and β -CD (Chun, Jo, Bjrappa, Choi, & Min, 2015), Eugenol in β -CD (Hill, Gomes, & Taylor, 2013), Vanillin in β -CD (Hundre et al., 2015), Thymol in β -CD (Del Toro-Sánchez et al., 2010), and Ethyl benzoate in HP- β -CD (Yuan, Lu, & Jin, 2014).

5. Flavor release-affecting factors and release mechanisms

Flavor release or release rate may be defined as the migration of flavor molecules from one environment or state to another environment or state over a certain period of time. Release rate of flavor and aroma compounds is very important for food products. Even before the food is placed in the mouth, people can estimate the food's quality and taste. The volatile flavors mixed with air molecules can enter the nasal cavity, which comes into contact with receptor cells in the olfactory epithelium through the orthonasal route, and the human brain perceives the kind of food it is, its quality and taste. Some flavoring compounds release in the mouth cavity during chewing of the food stuff and is mixed with the air that moves to the nasal cavity that interacts with odor proteins to trigger olfactory transduction (Laing & Jinks, 1996). There are a number of factors that influence the release of flavor and aroma compounds (i) thermodynamic factors (e.g. partitioning coefficient); and kinetic factors (e.g. diffusion and mass transfer) that control flavor release; (ii) the chemical structure of flavor molecules (size and weight); (iii) the physicochemical properties of aroma compounds that are related to their availability for perception (e.g. volatility and hydrophobicity); (iv) the initial strength or concentration of flavor (v) the effect of major food components (e.g. lipid, protein, carbohydrate); that interact with the aroma compounds; (vi) oral processing of food that take physiological factors into account (e.g. chewing rate, saliva flow, swallow frequency) (Yang, 2012; Zuidam & Heinrich, 2010). However, according to the literature, we have summarized the most important factors involved in flavor release as shown in Fig. 4.

Molecular weight and chemical composition of a wall material influence its barrier properties. For example, a higher molecular weight carrier material minimizes the aroma diffusion (Goubet, Le Quere, & Voilley, 1998). However, higher polymerization shows lower retention of flavor compounds, which may occur due to lower degrees of interactions between the aroma and carrier material (Zuidam & Heinrich, 2010). When an amorphous coating material is in a glassy state, its molecules may have very less mobility, which is very similar to a crystalline phase (Carolina, Carolina, Zamora, & Jorge, 2007; Soottitantawat et al., 2004; Ubbink & Schoonman, 2003). The transformation from a glassy to rubbery state is characterized by the glass transition temperature (T_g). Below the T_g , a coating material remains in its glassy state. At T_g , coating materials reduce viscosity and increase their molecular mobility (Roos, 1995), and therefore show poor barrier characteristics. The value of glass transition temperature depends on

the molecular weight of the amorphous material. T_g increases with increasing molecular weight. Hence, amorphous coating materials with higher molecular weight show better protection against flavor release at temperatures over the T_g . However, below the T_g , low molecular weight matrices provide better storage stability than high molecular weight matrices, since the first have a lower residual open and a closed porosity (Ubbink & Schoonman, 2003).

The capsule morphology depends upon the properties of carrier and core materials. Most of the flavors and aromas are hydrophobic in nature and may not mix well with hydrophilic carrier materials. Thus, they present as oil droplets. The release mechanism of the interior content of the capsules can be divided into five categories: diffusion, dissolution or melting, fracturation, swelling, and erosion & degradation as shown in Fig. 4 (Madene et al., 2006; Moran, Yin, Cadwallader, & Padua, 2014; Rodríguez et al., 2016).

The encapsulated flavoring compounds follow the specific mechanism depending upon the nature of core and wall materials, end-use environment, and the factors involved with the process (Shay & Reineccius, 1994). The release rate of flavor and aroma compounds can be determined by Avrami's equation (Desai & Park, 2005; Moran et al., 2014; Yoshii et al., 2001):

$$R = \exp(-kt)^n \quad (2)$$

Where R is the retention of the aroma compounds during release, t is the time, n is the parameter representing the release mechanism, and k is the release rate constant. The parameter n has a value of 1 for first-order kinetics and 0.54 for diffusion limiting reaction kinetics.

Taking logarithm of both sides and simplified form of equation (2)

$$\ln(-\ln R) = n \ln k + n \ln t \quad (3)$$

Using this equation the parameter n can be determined as the slope by plotting $\ln(-\ln R)$ against $\ln t$, and the release rate constant k from the interception at $\ln t = 0$. When the flavor release phenomena is mainly controlled by flavor diffusion inside the carrier matrix then the n value is usually equivalent to 0.5, which is known as half order release. On the other hand, when core material is actually a solution then first order release occurs and n is 1. However, $n > 1$ occurs at the initial period of flavor release and at the time of burst of the flavor emulsion (Furuta, Soottitantawat, Neoh, & Yoshii, 2010). An important point is that this release mechanism classification is only valid for the case when flavor release occurs from a single microcapsule. But in reality a matrix of encapsulated powder contains particles varying in size and wall thickness (Furuta et al., 2010). For example freeze dried and spray dried powder includes particles with different properties and the value of n in equations (2) and (3) for an encapsulated powder matrix varies according to the property of the powder. Furuta et al. (2010) reported that equation (2) is basically comparable to the equation of Kohlrausch-Williams-Watt (KWW) (Williams & Watts, 1970). That was expressed as:

$$G(t) = G_0 \exp \left[-\left(\frac{t}{\lambda} \right)^\beta \right] \quad (4)$$

Here, $G(t)$ represents the residual quantity of flavor in powder at time t , G_0 is initial flavor content, λ is the relaxation time, which reflects the inverse of the flavor release rate, β is the relaxation constant and t is time.

Since an encapsulated powder matrix (i.e. spray-dried powder) containing powder particles with various release characteristics, the sum of the KWW relaxation equation of an individual particle i might be its total (overall) release performance (Furuta et al., 2010). This can be expressed as-

$$G(t) = \sum_i G_i \exp \left[-\left(\frac{t}{\lambda_i} \right)^{\beta_i} \right] \quad (5)$$

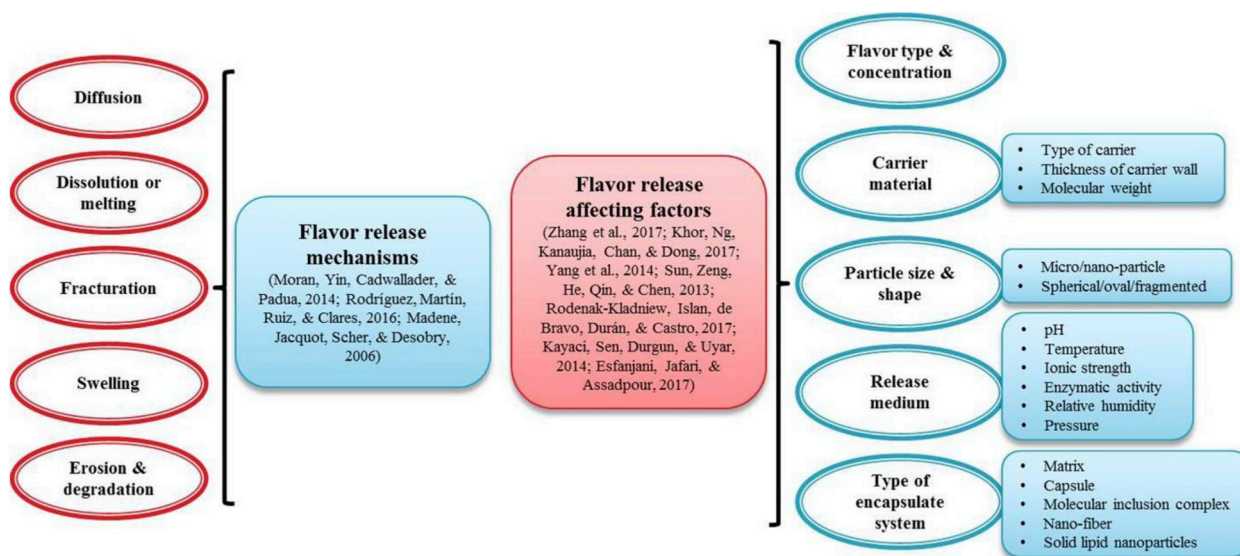


Fig. 4. Flavor release affecting factors and release mechanisms.

5.1. Diffusion

Diffusion is the dominant flavor release mechanism from the capsule. The vapour pressure of volatile compounds on each side of the capsule wall is the key driving force inducing diffusion (Dumay, Lalignant, Zasytkin, & Cheftel, 1999). The steps involved with this mechanism are the diffusion from the internal matrix to the surface of the capsule and then its movement to the surrounding food component. The molecular weight of flavoring compound influences the diffusion process. In fact, the diffusion rate through a polymer matrix decreases with an increase of molecular weight, decrease of volatility, and increase of $\log P$ (flavor-water partition coefficient; which indicates relative hydrophobicity (+) or relative hydrophilicity (-)) of the flavor compounds (Goubet et al., 1998). The diffusion rate increases when an amorphous matrix is converted from a glassy to rubbery state, which enhances the relative mobility (Moran et al., 2014). The pore size, thickness of the coating wall (diffusive layer), particle size and shape, as well as polarity can also affect the diffusivity of flavor compounds. Furthermore, the homogeneous or heterogeneous distribution of active compounds in a polymer matrix can influence flavor diffusivity (Huang & Brazel, 2001). In a steady state condition, Fick's law of diffusion expresses the amount of flavor and aroma compound to diffuse through or be transferred per unit area, per unit time or by mass flow of the compounds in a certain time. Considering a one-dimensional spherical coordinates similar to a microsphere morphology (Seuvre & Voilley, 2017; Vasisht, 2014); Fick's first law of diffusion is:

$$J = \left(\frac{1}{A}\right) \cdot \frac{dC}{dt} = -D \frac{dC}{dr} \quad (6)$$

Here, J is the diffusion flux, D is the diffusion coefficient (m^2/s), dC/dr is the concentration gradient of the compounds inside and outside of the microcapsule, r is the radius of the capsule and A is the surface area of the capsule. The negative sign in the equation represents that diffusion happens in the opposite direction of increasing concentration (Vasisht, 2014).

The simplified form of the equation with information about the concentration difference between the inside and outside of the capsule is:

$$\frac{dC}{dt} = -D \cdot A \cdot (\Delta C)/R$$

$$\text{or, } J = -D(\Delta C)/R$$

Here, $(\Delta C) = C_{mo} - C_{mi}$; and where, C_{mo} is concentration of the compounds outside of the microcapsule, C_{mi} is concentration of the compounds inside of the capsule and R is the thickness of the microcapsule.

5.2. Dissolution or melting

The mechanism of dissolution or melting involves dissolution of the coating material in a suitable solvent or the melting of the coating material through the influence of heat and/or moisture. Water-soluble coating materials easily release flavor and aroma compounds in water or in the presence of moisture. However, thermal release involves a coating material using fat. For that case, the coating material releases the core materials in the surrounding environment upon heating. There are also few coating materials which are dissolved in water at certain temperatures. This type (melting or dissolution upon heating) of flavor release occurs during cooking or baking. However, the active compound may not completely be present inside the coating material but also at the surface. Therefore, diffusion can also affect the dissolution mechanism during the release of flavor compounds to the surrounding medium from the matrix surface (Rodríguez et al., 2016; Siegel & Rathbone, 2012).

5.3. Fracturation

During fracturation, flavor release occurs by means of physical rupture. The coating shell may be broken or fractured by the action of external forces (e.g. pressure, shearing, grinding, extrasonics) and internal forces (e.g. expansion). Water-insoluble encapsulates (made from fats and waxes) can be released by physical rupture within a shorter time in comparison with other release mechanisms (Shahidi & Han, 1993). For instance, chewing is the most common fracturation means for the release of flavor compounds (Moran et al., 2014).

5.4. Swelling

During Swelling, the coating materials are placed in a thermodynamically compatible medium. The polymer swells because of absorption of fluid from the medium. The flavor and aroma compounds in the swelling part of the matrix then release easily by means of diffusion (Fan & Singh, 1989). The degree of swelling can be controlled by the rate of absorption of water or other solvents, such as glycerin or propylene glycol (Gibbs, Kermasha, Alli, & Mulligan, 1999).

5.5. Erosion and degradation

This mechanism is referred to as a combination of degradation, dissolution, and diffusion processes. Through erosion and degradation mechanisms, the wall material becomes thin, thus diffusion through the capsule wall increases. The capsule matrices or coating wall may also become degraded. For example, lipid coatings may be degraded by the action of lipases (Barbosa-Cánovas, Ortega-Rivas, Juliano, Yan et al., 2005). In general, two types of erosion processes are observed, i.e. bulk erosion and surface erosion. When the entrance of release medium inside the matrix is faster than the splitting of chemical bonds of the matrix, it is referred to as bulk erosion. In contrast, surface erosion refers to the slow entrance of the release medium inside the matrix in comparison with matrix chain breakage. Furthermore, matrix shape may also affect the release mechanism of erosion and degradation (Rodríguez et al., 2016).

6. Application of micro and nano-encapsulated flavor and aroma compounds in food industry

Flavor and aroma compounds are the most important active food components. The enhanced retention and controlled release of flavor and aroma is the highest priority of a food processor. Therefore, encapsulation is a common practice in food industries for the preservation of flavor compounds (Saifullah et al., 2016). However, there are a variety of encapsulation methods available that produce different forms of encapsulates, such as paste, powder, capsules, granules, and emulsion droplets. Most of the methods use flavor encapsulation to produce powder forms. The desired form depends upon the end use of flavor. Flavor powder is used in the preparation of a wide range of food products, such as confectionery products (cakes, bread, biscuits, cookies, chocolates, candies), milk powder, instant desserts, instant beverages, baked foods, instant drinks, extruded snacks etc. (Madene et al., 2006). Encapsulated flavor powders produced by different methods are suitable for different types of products, since the encapsulation process and/or coating material properties are different from each other. For illustration, flavor powder produced by spray drying is mostly used where flavor release requires room temperature and an aqueous solution. In contrast, powder produced by extrusion requires the flavor to release with higher temperatures compared to spray dried powders. Oxygen sensitive flavor compounds, i.e. citrus oils might have better stability in encapsulates prepared by melt extrusion than prepared by other methods because of the barrier properties it can provide (Bakry et al., 2016; Ubbink & Schoonman, 2003). Flavor encapsulated by molecular inclusion in β -cyclodextrin is used for the preparation of hard candy, fruit leathers, and angel food cake reported by Reineccius, Reineccius, and Peppard (2004). Flavor emulsions are used in ice creams and beverages. Micro and nano-emulsion shows unique optical clarity, therefore suitable for the application in transparent and turbid soft drinks (Bakry et al., 2016; Kfoury et al., 2016; McClements, 2015).

Nano and micro sized encapsulate particles facilitate well the distribution of flavor compounds in food products and help in prolonging the release of flavoring. In food processing operations, there are several factors (e.g. temperature, pH, enzyme), which may cause flavor degradation. Therefore, flavoring ingredients are added at different stages of processing depending upon the types of products made. For instance, in some processes, flavor is added in the last step of the process to reduce thermal loss during processing. Besides, encapsulated flavor and aroma compounds in suspension and paste forms are also used in foods differently, depending on the processing operation and the food product.

7. Conclusion and future trends

Flavors and aromas are the most important bioactive food ingredients, which represent the taste and quality of a food product.

Encapsulation allows flavors to be in a stable form and makes them suitable for application in a wide range of food products. Nano-encapsulation approaches provide a greater loading capacity and encapsulation efficiency, enhanced stability, and better control on flavor release profile compared to microencapsulation. In recent, spray chilling is gaining increasing research interest as an alternative to spray drying because it overcomes heat-induced flavor losses by spray drying. However, spray chilling shows less control on particle size. On the contrary, nano-capsules produced by electro-spraying and nano-fibers produced by electro-spinning are emerging and promising approaches showing high efficiency in nano-encapsulation. The combination of two encapsulation approaches for obtaining desired characteristics of micro or nano-capsules has also recently been observed, i.e. addition of emulsification with spray drying, freeze drying, extrusion, electro-spraying, or electro-spinning offers a combined benefits. The combination of carrier materials, e.g. a combination of polysaccharides, polysaccharide with protein, or a polysaccharide with lipid also provides an advantageous effect on flavor retention and release characteristics. Therefore, the recent advances on flavor and aroma encapsulation are a remarkable contribution as discussed in this review. Future studies could be emphasized on flavor release mechanisms and their application efficiency in different food or biological (gastro-intestinal) systems, wall material properties and storage behavior of encapsulated powder in order to integrate with the recent advances and to accelerate their commercialization.

Conflicts of interest

The authors declare that there are no conflicts of interest.

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