Chapter 4
Microencapsulation Technologies

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Abstract Microencapsulation involves the packaging of a gaseous, liquid or solid substance (i.e., the core or active) within a secondary material in small capsules in the range of about 0.5−2000 μm. Microencapsulation protects and stabilizes the encapsulated substance until it is released at a desired site and time by conditions that trigger its release from the microcapsule. By appropriate formulation and processing, microencapsulated ingredients may be designed to achieve the desired properties that make them superior to the neat bioactive core in the intended application. The design of a microencapsulated ingredient requires a multidisciplinary approach that includes considering the physico-chemical properties of the core and the materials to be used as encapsulants, the design and formulation of the microencapsulated ingredient, and the choice of technology for processing the microcapsules. The technologies available for the microencapsulation of various food bioactives and the properties of selected microencapsulated ingredients are discussed.

4.1 Introduction

Microencapsulation may be defined as a technology that involves the packaging of a gaseous, liquid or solid substance (i.e., the core or active) within a secondary material (i.e., matrix, encapsulant) in small capsules in the range of about 0.5−2000 μm. Microencapsulation protects and stabilizes the encapsulated substance and allows controlled delivery of the encapsulated substance at a desired site and rate when the capsule is exposed to specific conditions which provide a trigger or stimulus for the breakdown of the capsule. Many types of microencapsulated formulations can be developed for the delivery of food bioactives and desirable microorganisms. For food

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applications, the whole delivery system has to be created from food grade ingredients. The inherent properties of the bioactive and the encapsulating material, including their solubility in oil and aqueous systems and their affinity for interfaces, their resistance to degradation in various environments (e.g., when exposed to oxygen, high temperature, enzymes), and the interactions between the bioactive and the encapsulant matrix are some of the major factors that govern the choice of the formulation and the technology for the processing of the microencapsulated bioactive (Augustin and Hemar 2009; Garti and McClements 2012; Nazzaro et al. 2012).

Microencapsulation technologies have been used in the pharmaceutical and chemical industries for some time. Microencapsulation is comparatively new to the food industry, but as there are similar requirements for stabilization and controlled delivery in the food and pharmaceutical industries, the food industry has adapted many of the technologies from the pharmaceutical and chemical industries when developing microencapsulated food ingredients. However, a factor that needs to be taken into account when adapting the technology for commercial application in the food industry is the low margins in the food industry compared to the pharmaceutical and fine chemical industries, which can tolerate higher cost technologies. Another difference is that the pharmaceutical and chemical industries have the option to use a wider range of materials and chemical polymers as encapsulants, as they are not restricted to food materials or materials that have GRAS (generally regarded as safe) status. Selecting the appropriate process for the production of microcapsules requires consideration of (1) technical issues, such as the inherent properties of the core, the encapsulant matrix, the size and morphology of the particle, the mechanisms triggering the release of the core and the format of delivery; (2) the cost, consistency and sustainability of the raw materials and formulation; and (3) the economics of the microencapsulation technology, increasingly including the associated environmental footprint of the process.

The focus of this chapter is the methods used for the processing of microcapsules and the process variables that can be manipulated to achieve the desired properties of the microcapsules. However, the method of choice for processing microcapsules cannot be detached from the materials and formulation used for the design of the systems. Therefore some aspects of formulation are also considered from the perspective of material-process interactions for improving the amenability of the formulation to be transformed into a microencapsulated ingredient in the desired format with the required functional performance.

4.2 Ingredients Used in the Formulation of Food-Grade Microencapsulated Products

The formulation of the microencapsulated product is the first consideration once the target application has been defined. Table 4.1 lists some of the major bioactives of interest to the food and nutraceutical industries. Materials that have commonly been used in microencapsulated formulations as the carrier or encapsulant matrices for food bioactives include a range of proteins (e.g., caseins and whey proteins, soy

Table 4.1 Selection of bioactives of interest to the food and nutraceutical industries

Bioactive	Examples	Sources
Omega-3 fatty acids	α-linolenic acid (ALA)	Flax, perilla, chia
	Eicosapentaenoic acid (EPA) and Docosahexaenoic acid (DHA)	Fish oil, marine algae, krill oil
Probiotics	Lactobacilli, Bifidobacterium	Cultured microorganisms
Prebiotics	Inulin	Chicory root, Jerusalem artichoke, jicama
	β-glucan	Barley, oats
Carotenoids	β-carotene	Carrots, sweet potato, palm oil, algae
	Lycopene	Tomato, water melon, red grapefruit
	Lutein and zeaxanthin	Nasturium (yellow flowers), kale, spinach
Phenolic compounds and polyphenols	Resveratrol	Japanese knotweed, wine
	Curcumin	Tumeric
	Flavonoid (quercetin and rutin)	Onions
	Flavonoids (hesperitin)	Orange juice
	Catechins and epicatechins	Cocoa, chocolate, tea

proteins, wheat proteins), sugars and syrups, starches, gums (e.g., gum acacia, alginate, pectin, carrageenan, cellulosic material), chitosan, oils and fats, phospholipids and food-grade low molecular weight emulsifiers (e.g., Tweens).

The development of a delivery system should achieve the following attributes of performance in the intended application: (1) the stabilization of the original food bioactive after isolation from a food source; (2) the protection of the bioactive against degradation when it is in an encapsulated ingredient format and when it is incorporated into a manufactured food product; (3) the masking of tastes (if required; for example, with bioactives such as polyphenols which have an astringent tastes) so that the fortified food product has sensory appeal and is acceptable to the consumer; and (4) the delivery of the bioactive to the intended site in the gastrointestinal tract after consumption. The ability to meet all the demands of performance of a delivery system presents several scientific and technical challenges in developing a successful delivery system for a bioactive. When more than one bioactive is to be co-delivered in one encapsulation system, the challenges are even greater depending on the inherent physical and chemical properties of the bioactives, as well as the interactions between them and with the matrix.

Depending on the technology or method used for microencapsulation, different formats, morphologies, structures and other properties can be produced. The desired properties and functionality can be tailored to suit the target application. Therefore, it is important to define the criteria that will define a successful microencapsulated ingredient. The defined criteria allow the proper selection of the microencapsulation technology. Each of the available technologies can create microcapsules within a

Table 4.2 Encapsulation Processes

Physical processes	Chemical processes
Drying (Spray drying, freeze drying)	Coacervation (Simple and complex)
Spray chilling	Inclusion complexation
Fluidized bed coating	Hydrogels, beadlets
Extrusion	Biopolymeric particles
Spinning disk coating	Emulsion-based systems including layer-by-layer deposition
Supercritical fluid processing	Liposomes

particular range of size and bioactive payload type and quantity. Often, an approach based on a retro-fit design is used as a guide to the formulation and design of a microencapsulation system that is suited for the performance required in the final application (Ubbink and Krueger 2006). It can also sometimes be an iterative process where a formulation is first developed, its performance checked, and re-formulation carried out to optimize the microencapsulated formulation (Augustin and Sanguansri 2012). However, although it may appear to be deceptively simple to formulate and process a microencapsulated product with all the desirable attributes, it should be appreciated that the development of a microencapsulated product requires a multi-disciplinary approach and significant scientific knowledge of each component and process used in its development. Once the formulation has been developed, the bioactive core and the carrier matrix/encapsulant material are then made into the microencapsulated ingredient using the appropriate microencapsulation technology. The common processes employed for microencapsulation of bioactives for food application are listed in Table 4.2, and described in the following section.

4.3 Microencapsulation Technologies for Bioactive Delivery

Microencapsulation technologies generally refer to processes or methods applied to produce a microencapsulated ingredient or bioactives, with various types of morphologies and structures (Fig. 4.1). These are classified into physical or chemical processes, and often a combination of both physical and chemical processes is applied in order to achieve the desired functionality in the final microcapsule.

Depending on the technology used for microencapsulation, different formats, morphologies, structures and other properties can be produced. The desired properties and functionality can be tailored to suit the target application. The complexity of the food matrix and the stresses that the microencapsulated ingredients are exposed to during the manufacture of food make the choice of the appropriate formulation and technology more challenging. Therefore, it is important to define the criteria required for a successful microencapsulated product/ingredient (Table 4.3). These criteria allow for the proper selection of the microencapsulation technology.

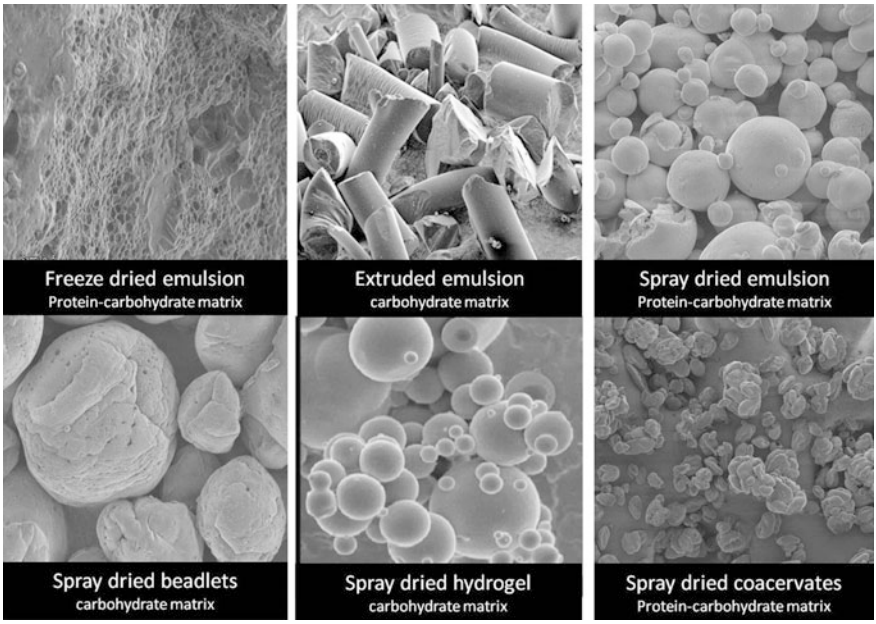

Fig. 4.1 Microstructure of dried encapsulated ingredients

Table 4.3 Criteria for selection of microencapsulation technology based on desired performance of the microencapsulated ingredient in final applications

Desired performance	Technology	Format	Particle size (μm)	Payload (%)
Liquid to powder conversion	Spray drying	Powder, granules, agglomerates	10–400	5–50
	Freeze drying	Powder	20–5000	5–50
	Spray chilling	Powder	20–200	10–20
Solubilization	Emulsion	Liquid	0.2–5000	1–20
	Nanoemulsion	Liquid	0.01–0.1	Various
Stabilization	Spray drying	Powders	10–400	5–50
	Emulsion	Liquid	0.2–5000	1–25
	Coacervation	Powder	10–800	40–90
	Inclusion complexation	Powder	0.001–0.01	5–15
	Biopolymeric particles	Liquid, powder	10–1000	20–50
	Fluid bed coating	Powder, granules, agglomerates	5–5000	5–50
Taste masking	Inclusion complexation	Powder	0.001–0.01	5–15
	Binding to biopolymers	Liquid, powder	10–1000	20–50
	Fluid bed coating	Powder, granules, agglomerates	5–5000	5–50

(continued)

Table 4.3 (continued)

Desired performance	Technology	Format	Particle size (μm)	Payload (%)
Visual clarity	Nanoemulsion	Liquid	0.01–0.1	Various
Controlled release	Liposome entrapment	Liquid, powder	10–1000	5–50
	Extruded microsphere	Beadlets, powder	200–5000	20–50
	Emulsified microspheres	Beadlets, powder	10–1000	20–50
	Nozzle coextrusion	Beadlets, powder	150–8000	70–90
	Rapid expansion of supercritical fluid	Powder	10–400	20–50
	Fluid bed coating	Powder, granules, agglomerates	5–5000	5–50
Shear resistance	Spray drying	Powder	10–400	5–50
	Extrusion	Powder, pellets	200–5000	5–40
Compression resistance	Spray drying	Powder	10–400	5–50
	Extrusion	Powder, pellets	200–5000	5−40

4.3.1 Physical Processes

4.3.1.1 Drying

Drying converts liquid materials into solid or semi-solid particles. The process results in the production of powders, granules or dry agglomerates. For food applications, powder formats are generally preferred over slurries, emulsions or suspensions. Powders are much easier to handle and transport, have longer storage stability, and may be readily added to a range of food formulations. The most common drying methods for the preparation of microencapsulated ingredients are spray drying and freeze drying.

Spray Drying

Of the drying methods, spray drying is the preferred method, as it is cost-effective and is also suitable for the drying of heat-sensitive bioactives (Gouin 2004; Murugesan and Orsat 2012).

The major stages in a spray drying process are (1) atomization of the liquid feed or slurry; (2) the contact of the atomized droplets with the drying medium; (3) the evaporation of water from the droplet; and (4) the separation of the dried product from the air. The rapid evaporation of water during spray drying keeps the product temperature relatively low (below the outlet temperature), and hence, it is possible to use spray drying technology to produce microencapsulated products with heat sensitive cores.

Spray drying has been used for microencapsulated vitamins, minerals, flavors, antioxidants (Murugesan and Orsat 2012), omega-3 oils (Augustin et al. 2006), fruit

fibers with bioactives (Chiou and Langrish 2007) and probiotics (Silva et al. 2011). The formulation (bioactive core, encapsulation material, ratio of core: encapsulant), the processes (e.g., homogenization conditions) for preparing the feed and process parameters used in spray drying, such as inlet and outlet temperatures, feed rates, feed temperature, airflow rates and type of atomizer or nozzle, all impact the properties of the final microencapsulated product (Murugesan and Orsat 2012). For example, in the preparation of spray dried lycopene with gelatin and sucrose as wall materials (core:wall ratio of 1:4 and gelatin:sucrose ratio of 3:7), it was found that increasing the feed temperature from 35 to 55 °C increased encapsulation yield and encapsulation efficiency, but further increase in the feed temperature to 65 °C was detrimental. This was possibly due to the need to balance good atomization, which improves with lower viscosity at higher feed temperatures versus the build of particles on the chamber wall due to the trajectory of the atomized particles when the feed viscosity is too low (Shu et al. 2006). These authors also showed that inlet temperature affected encapsulation yield and encapsulation efficiency within the range of inlet temperatures of 170–210 °C, and the optimum inlet temperature was 190 °C (Shu et al. 2006). However, increasing the inlet temperature from 145 to 175 °C decreased the lycopene content of spray dried watermelon powders with maltodextrin as the encapsulating agent (Quek et al. 2007).

Freeze Drying

The freeze drying process has three major stages: (1) the freezing of the liquid formulation which results in nucleation and growth of ice crystals and the formation of a glassy matrix; (2) the primary drying where the ice crystals are sublimed under low pressure; and (3) the secondary drying where the unfrozen water is desorbed by increasing the temperature or reducing the pressure during drying, depending on the material sorption isotherm. The freezing step is important as freezing affects ice formation and therefore the morphology of the product and reconstitution time. Fast freezing rates should be avoided if there is protein in the formulation. The primary drying rate affects the resistance of the dried product. The secondary drying step allows removal of water to the desired water content (Kasper and Friess 2011). As freeze drying is carried out at low temperature, the process enables high retention of volatile components and minimal degradation of heat-sensitive bioactives. Freeze drying is the most commonly used commercial method for production of dried probiotics. It has been used for the microencapsulation of many other bioactives including polyphenols, vitamins, omega-3 oils, citrus extracts, herbal extracts, spice oils and limonene.

The method of drying and the drying conditions affect the stability of the encapsulated bioactives. This was demonstrated in the early work of Desorby et al. (1997), where β-carotene encapsulated in maltodextrin was most stable to degradation when the emulsion was freeze dried compared to when it was spray dried or drum dried, with most degradation occurring in the drum-dried samples. Whether spray drying or freeze drying had a more detrimental effect on the survival of probiotic bacteria during storage was dependent on the source and strain of the

probiotic bacteria and on the encapsulation formulation (Chávez and Ledeboer 2007; Ying et al. 2010).

4.3.1.2 Spray Chilling

The spray chilling process comprises two major steps: (1) the addition of a bioactive into a molten fat, and (2) the atomization of the molten material in a heated atomizing nozzle into a refrigerated chamber, which solidifies the molten carrier material to form solid lipid microparticles. The residence time in the spray cooling chamber is short (usually a few seconds), after which the microparticles are collected. The core is typically a lipophilic bioactive, but it can also be a hydrophilic or an amphiphilic core (e.g., a peptide), and the carrier is often a lipid that is solid at room temperature (e.g., hydrogenated or fractionated edible oils with high melting points, or beeswax).

Spray chilling is a low-cost technology that is suitable for heat-sensitive ingredients such as omega-3 fatty acids, enzymes and probiotics, but it suffers from low encapsulation efficiency, and the bioactive is sometimes expelled during storage (Okuro et al. 2013). In cases where the lipid is the encapsulant, the release of the bioactive core may be triggered by increasing the temperature to above the melting point of the fat or digestion of the fat by lipases in the gastrointestinal tract after ingestion (Okuro et al. 2013). However, it is difficult to obtain delayed release greater than 30 min with a water-soluble core in a food with high water activity (Gouin 2004).

4.3.1.3 Fluidized Bed Coating

Fluid bed coating technology involves the suspension of solid particles in a moving stream of air, which may be heated or cooled. The coating is then sprayed onto the particle through an atomizer. The coating may be a molten material (e.g., fat) or a material (e.g., proteins, polysaccharides, complex formulations, emulsifiers) dissolved in a solvent (e.g., water, aqueous ethanol). Where a coating liquid is used, it is rapidly evaporated by hot air to form the outer layer of the coated particle. Where the coating is a lipid, it solidifies when in contact with the cool air as it reaches a temperature below its melting point. The particle may be passed repeatedly through the spray coating and the drying air for many coating cycles to obtain uniform coating, and in addition, multiple types of coatings may be used sequentially. This process may be used to control the coating thickness, the particle size and the release properties of the bioactive core. A pre-requisite for film coating is good adhesion properties between the coating and the core. Variables such as solvent evaporation rate, spray rate, droplet size and temperature influence the properties of the coated particle (Kuang et al. 2010). Apart from the formulation, factors such as relative humidity, fluidizing gas flow rate, coating spray rate and the composition of the fluidization gas need to be controlled for an optimized process. This also

requires an understanding of the thermodynamics of the coating process, and psychometric charts are invaluable for providing a rational basis for optimizing the coating process (Gouin 2005).

4.3.1.4 Extrusion

Extrusion—Dripping Technologies

One of the oldest methods for encapsulation is the formation of microbeads. It has been widely used for the encapsulation of cells as it results in minimal injury to cells, and in high viability of probiotic bacteria, as well as of high retention of heat sensitive bioactives (e.g., carotenes). The most common material used as the matrix for extrusion of microbeads containing bioactives is alginate. Where alginate is used, the steps involve (1) preparing an alginate solution; (2) adding the bioactive core (usually as an emulsion) into the alginate solution; and (3) dripping the resultant emulsion or suspension into a hardening bath through an orifice (e.g., needle or nozzle). Calcium chloride is generally used as the hardening solution as calcium ions cross-link the alginate molecules, forming a gel network that entraps the loaded bioactive. The microbeads can then be dried to the desired moisture. Although this process is simple, there have been difficulties in scaling up due to the slow process required for the formation of microbeads. However, advances in technologies such as laminar jet break-up technology have increased the productivity of the process and enabled a narrower dimensional range and high encapsulation efficiency. In this process, a laminar jet of a polymer solution is broken up with a vibrating nozzle (Del Gaudio et al. 2005).

Extrusion Cooking

Extrusion cooking is an energy-efficient, low-cost process that is used for producing ready-to-eat cereals and snack products. Notably, there is increasing interest in developing nutritious snacks (Brennan et al. 2013). During extrusion cooking, ingredients are mixed, cooked, texturized and formed. Oils and nutritive ingredients may be added to the formulation and these ingredients become embedded (or encapsulated) within the matrix of the extruded product.

Yilmaz et al. (2001) examined the encapsulation of sunflower oil in a starch matrix. These authors showed that the formulation and the processing conditions during extrusion influenced the properties of the microencapsulated oil. The size of the oil droplets in the extruded product decreased with increasing HLB (hydrophilic-lipophilic balance), increasing screw speed, increasing melt temperature and decreasing throughput. The release kinetics will depend on adequate mixing and dispersion of the encapsulant within the matrix (Yilmaz et al. 2001).

Sensitive nutrients such as vitamins are degraded during extrusion. The extent of degradation depends on the type of vitamin, the formulation, and the processing conditions used (Killeit 1994). Nutrient degradation during extrusion may be

reduced by introducing protected pre-encapsulated vitamin products. This has been suggested for animal feed preparations (Putnam 1986). Others have suggested that phytochemicals (β-carotene in medium-chain triglyceride oil) should be added after the plasticization of starch ingredients, to minimize exposure time to thermal and mechanical stresses, but losses are still 30 % and higher (Emin et al. 2012). However, with the introduction of modern twin-screw extruders, extrusion encapsulation is becoming an attractive alternative, as the process is flexible, and exposure to high temperatures can be avoided by introducing bioactives into the last port of the barrel (Abbas et al. 2012).

4.3.1.5 Spinning Disk Systems

Spinning disk coating uses rotational forces to create a thin film around a core. The core ingredient is suspended in a wall material and dropped onto the rotating disk which throws the droplets out towards the circumference of the disk where the wall material solidifies through drying or chilling, depending on the properties of the encapsulant material used and on the conditions. (Mason and Sparks 1987; Sparks et al. 1995). As the spinning rate can be carefully controlled, the disk process is able to yield narrow particle size distributions between 20 microns and several millimeters (Labelle 2002). For example, with spinning disk atomizers, hydrogels (alginate) and microbeads of narrow particle size (300–600 μm) distribution are obtained (Senuma et al. 2000).

4.3.1.6 Supercritical Fluid Encapsulation

With supercritical fluid encapsulation, the core material and the encapsulating material (typically a polymer) are dispersed and/or dissolved in a supercritical fluid, such as carbon dioxide. The supercritical fluid is ejected from a nozzle in the form of a spray. The carbon dioxide flashes off very rapidly, leaving residual particulate material. The solvent and anti-solvent properties of supercritical carbon dioxide can be exploited for the micronization and encapsulation of bioactive cores. Depending on the solubility of the compounds to be encapsulated, the SAS (supercritical anti-solvent) or RESS (rapid expansion of supercritical solutions) processes can be used. Santos and Meireles (2013) have recently discussed the applications of SAS for micronization of quercetin and β-carotene and RESS for the encapsulation of bixin-rich and anthocyanin-rich extracts in polyethylene glycol as the encapsulation material. Ethanol maybe used as a co-solvent. The properties of the ingredients in the formulation, the core:encapsulant ratio, the solubility of the bioactive in the supercritical fluid and the processing conditions used influence the properties of the encapsulated product. Xia et al. (2012) prepared pro-liposomes of lutein and hydrogenated phosphatidylcholine using SAS and showed that high lutein loading liposomes with encapsulation efficiency of >90 % could be produced. An interesting application is the use of supercritical carbon dioxide for the production of β-glucan aerogels for carrying flaxseed oil. The aerogels were impregnated with a

high concentration of flaxseed oil (65 %) by using supercritical carbon dioxide as the medium for mass transfer. It was suggested that an additional coating may be required to protect the oil against degradation due to the high porosity of the aerogels (Comin et al. 2012).

4.3.2 Physico-chemical Processes

4.3.2.1 Coacervation

Coacervation has been used in the food industry for encapsulation of flavors, omega-3 oils and other ingredients (Gouin 2004; Lamprecht et al. 2001). Simple coacervates are formed from one type of polymer, and those formed from two or more types of polymer (often from oppositely charged materials) are called complex coacervates. Coacervation may involve the preparation of an oil-in-water emulsion with the lipophilic bioactive being present in the oil phase. Subsequently under turbulent mixing, two liquid phases are separated under appropriate conditions. The concentrated polymer rich phase is the coacervate containing the bioactive, and the other phase is called the equilibrium solution. Coacervates are stabilized following a change in the conditions of the environment such as temperature, pH and ionic concentration. The formation of more permanent structures with well-defined sizes and shapes requires cross-linking of the biopolymer using a chemical or enzyme, and alternatively another biopolymer may be adsorbed around the droplets for stabilization (Jones and McClements 2010). The coacervates can be dried into a free flowing powder. A variation of complex coacervation involving the addition of a controlled agglomeration step and the formation of an outer shell that surrounds the agglomerates was developed by Barrow et al. (2007) for encapsulation of omega-3 oils.

Complex coacervates formed by electrostatic binding between proteins and polysaccharides (protein-polysaccharide complex) are most commonly used in the food industry (De Kruif et al. 2004). Protein-polysaccharide complexes exhibit better functional properties in a number of applications than proteins and polysaccharide combination without complexation (Schmitt et al. 1998). The use of coacervates and protein-polysaccharide complexes as delivery systems for bioactives or sensitive molecules in food is attractive due to the variety of biopolymer combinations that may be adapted to the various requirements (e.g., mechanical properties and permeability) of the delivery system (Schmitt and Turgeon 2011). For example, a gelatin-sodium alginate based polyelectrolyte complex used as a carrier for ascorbic acid has enhanced its thermal stability compared to each of the encapsulant materials on their own (Devi and Kakati 2013).

The mechanical strength and diffusion characteristics of chitosan-based coacervates can be influenced by the type of chitosan used in forming the complex. Chitosan hydroglutamate-based complexes in combination with kappa carrageenan or with alginate have higher mechanical strength than complexes based on acid soluble chitosan. In addition, higher diffusion release characteristics of a model dye

have been shown in acid soluble chitosan coacervated microcapsules, when kappa carrageenan was used compared to alginate (Pandya and Knorr 1991). Omega-3 oil loaded coacervates have been prepared with gelatin as the capsule wall followed by addition of edible salt to precipitate the gelatin rich phase (salting-out), and using transglutaminase as the cross-linking agent for hardening the capsule wall (Soeda et al. 2003). Vitamins and polyunsaturated fatty acids that are substantially insoluble in boiling water for at least 3 min have been encapsulated by cross-linking a protein, sugar and a water-soluble salt, by heating the mixture (Chaundry et al. 1992).

4.3.2.2 Inclusion Complexation

Inclusion complexes may be formed between an active and a host molecule such as cyclodextrin or starch. Inclusion complexation takes place at a molecular level and utilizes the host molecules as the encapsulating medium.

In the case of cyclodextrins, the internal surface is lipophilic, while the external surface is hydrophilic. This structure characteristic makes cyclodextrins suitable for encapsulation of less polar molecules (e.g., essential oils) into the apolar internal cavity through hydrophobic interactions (Bhandari et al. 1999). β-cyclodextrin complexes have been used for the encapsulation of oils (onion and garlic oils, omega-3 oils) to enhance their stability to temperature, light, oxidation, polymerization or double bond migration, and mask undesirable taste and odor (Dziezak 1988; Djedaini et al. 2000; Kim et al. 2000). A complex formed between DHA and cyclodextrin prepared by a twin-screw kneader has high resistance to oxidation in fishmeal paste application even without an antioxidant in the formulation (Yoshii et al. 1997). The inclusion of polyphenols in cyclodextrins improved their water solubility, especially for the less water-soluble phytochemicals (Fang and Bhandari 2010).

A new way to prepare cyclodextrin-based supramolecular systems has been reported for encapsulation of bioactive materials with altered functions in which the process of molecular assembly can be controlled (Chen and Liu 2010). The supramolecular assembly of vitamin B6 with beta-cyclodextrin was successfully prepared using different methods (kneading, co-precipitation and freeze drying), and showed that the vitamin B6 probably enters the cyclodextrin torus when forming the β-cyclodextrin-vitamin B6 inclusion complex (Borodi et al. 2009). The use of ligand-binding proteins has been explored as an alternative to cyclodextrins for inclusion complexation (Jones and McClements 2008).

Starch inclusion complexes have been used for controlled delivery of bioactives (Lesmes et al. 2008). Examples inlcude the complexation of genistein with high-amylose starch (Cohen et al. 2011) and long chain fatty acids with amylose

(Zabar et al. 2009). Hydrophobically modified starch has also been used in the molecular inclusion of polyphenols, such as curcumin (Yu and Huang 2010). The curcumin-starch complex showed a 1670-fold increase in solubility, possibly reflecting the hydrophobic interaction and hydrogen bonding between curcumin and the hydrophobically modified starch, and the encapsulated curcumin revealed enhanced in vitro anti-cancer activity compared to the free form (Yu and Huang 2010).

4.3.2.3 Liposome Entrapment

Liposomes have been used for the encapsulation and delivery of bioactive and biological agents. Liposomes stabilize bioactive materials from changes in the surrounding environment, including enzymatic and chemical modification, as well as buffering against extreme pH and changes in ionic strength. The production of liposomes using a simple low cost process without contamination of the product is of much interest to liposome producers. Different methods of liposome preparation and loading, their applications in food, cosmetics and pharmaceuticals, the main analytical methods for their characterization, and the mechanisms of liposome targeting were recently reviewed by Maherani et al. (2011). In the food industry, liposomes have been used for flavors and nutrients, as well as for antimicrobial delivery to protect food products against the growth of spoilage and pathogenic microorganisms (Taylor and Davidson 2005). While hydrophilic bioactives are typically encapsulated in their internal aqueous phase, liposomes have also been used to encapsulate lipophilic bioactives, by dissolving the bioactive in the phospholipid before water is added and sonicated to form the encapsulated material. The use of liposomes for encapsulation of antioxidants, enzymes, proteins, vitamins and minerals has been reported; however, their lack of stability under gastrointestinal conditions (low pH and enzymes), insufficient loading of bioactives and the high cost of materials remain a challenge to liposome-based encapsulation in food applications (Liu 2013).

Bioactives entrapped in liposomes are protected from oxidation and the preparation can be added directly to food products, as a dispersion, or be dried into a free flowing powder (Haynes et al. 1991). Food-approved pro-liposomes are now available, and can be used for the microencapsulation of food ingredients. They are much simpler to use, but remain expensive. Takahashi et al. (2006) successfully prepared a thermodynamically stable liposome-encapsulated curcumin (up to 85 % entrapment efficiency) from commercially available low-cost lecithin using a mechanochemical method. In an animal study, the liposome encapsulated curcumin had significantly higher absorption in the gastrointestinal tract, leading to enhanced bioavailability and bioactivity (Takahashi et al. 2009). Soy lecithin (Mohan et al. 2016) or egg lecithins (Nii et al. 2003) are food-grade materials which have been used in the formulation of liposomes. Milk-fat-globule-membrane phospholipids isolated from buttermilk have been used to prepare liposomes using ascorbic acid as a model bioactive (Farhang et al. 2012).

4.3.2.4 Biopolymeric Particles

Biopolymeric particles can be produced using food-grade proteins or polysaccharides and used as structured carriers for both hydrophilic and hydrophobic cores for controlled release and encapsulation applications (Chang and Chen 2005; Gupta and Gupta 2005; Ritzoulis et al. 2005) and for protection and delivery of probiotics (Huq et al. 2013). Biopolymeric particles or matrices may be formed by promoting self-association or aggregation of single biopolymers or by inducing phase separation in mixed biopolymer systems, for example, using aggregative (net attractive) or segregative (net repulsive) interactions (Weiss et al. 2006). Bioactives can be encapsulated in particles designed to release in response to specific environmental triggers.

Biopolymeric matrices have been produced using simple or complex coacervation methods involving proteins or protein and polysaccharide mixtures to create transparent solid matrices designed for controlled release applications (Renard et al. 2002). Biopolymer nanoparticles or microparticles can also be formed by controlled thermal treatment of electrostatic complexes of globular proteins and ionic polysaccharides. The size, charge, and stability of the biopolymer particles can be manipulated by controlling the holding temperature, holding time, initial protein concentration, polysaccharide type, protein-to-polysaccharide ratio, co-solvent composition, pH and ionic strength (Jones and McClements 2011). The use of biopolymeric particles formed by heat treatment of beta-lactoglobulin/beet pectin was investigated for application in food products as delivery systems, clouding agents, texture modifiers, or as fat droplet mimetics (Jones and McClements 2008).

Co-precipitation or freeze thaw methods have been used to produce biopolymeric particles. A co-precipitation method has been used to encapsulate quercetin using a hydrophobic protein in aqueous medium, resulting in enhanced molecular stability of the zein:quercetin particles (zein:quercetin ratio of 25:1 and 50:1 wt/wt) to alkaline pH and UV irradiation (Patel et al. 2012). The freeze–thaw method followed by alginate gelation via ionic interactions between the polymer and the cross-linking ions was used to encapsulate D-limonene in alginate/poly(vinyl alcohol) (20:80) mixture, resulting in extended release of the encapsulated D-limonene in comparison to the free aroma (Levica et al. 2011).

4.3.2.5 Emulsion-Based Systems

Emulsion-based systems are used to deliver lipophilic bioactives (oil-in-water emulsions) or hydrophilic bioactives (water-in-oil emulsions). Different emulsion systems (e.g., simple or complex emulsion structures) can be formed depending on the emulsifier and process used during emulsion preparation (Fig. 4.2). Emulsion-based systems have typically been used to encapsulate flavors, omega-3 fatty acids, carotenes, lycopene, lutein and other lipid soluble bioactives and vitamins (McClements et al. 2009). Conventional emulsions are widely used in the food industry. However, the delivery of bioactives in a stable emulsion-based

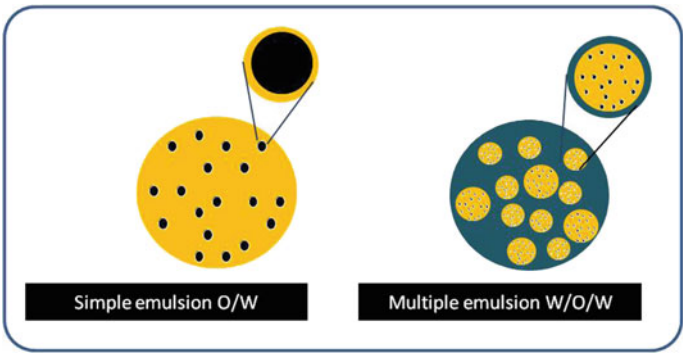

Fig. 4.2 Structure of a simple and complex emulsions

system to food and beverage may require more sophisticated design to meet the desired success criteria for particular applications. Examples of more complex emulsion based systems include multiple emulsions, micro emulsions, multilayered emulsions, solid-lipid particle emulsions, colloidosomes and internally self-assembled emulsion droplets (ISAsomes).

Simple Emulsions

A simple emulsion used for delivery is a dispersion of one immiscible phase, such as oil (containing the bioactive), in another phase (the continuous phase), such as water. During preparation of a simple oil-in-water emulsion, the oil phase is dispersed into fine droplets using an emulsifier to stabilize the oil-water interface. High shear homogenization or micro fluidization processes are used to disperse the oil into small droplets forming the emulsion. The type of emulsifier, the total solids, the pH, and the other formulation components including homogenization and storage conditions influence the emulsion properties and stability during storage (Fig. 4.3).

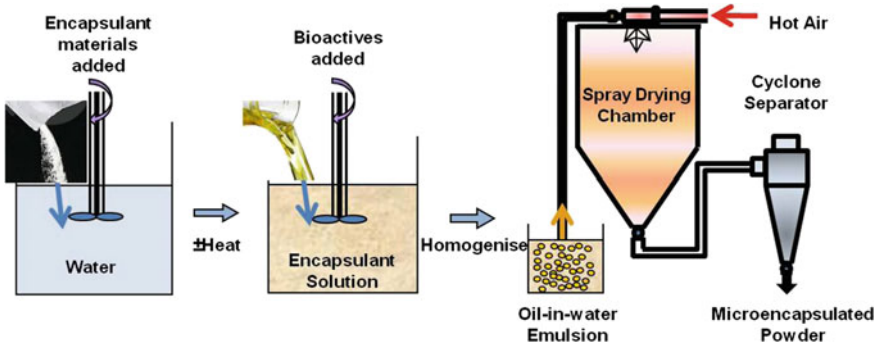

Fig. 4.3 Method for manufacture of spray dried emulsion

Multiple Emulsions

Multiple emulsions used for delivery may be in the form of water-in-oil-in-water (W/O/W) emulsions consisting of small droplets of water containing soluble bioactives, dispersed within larger oil droplets that are dispersed in an external aqueous continuous phase. W/O/W may be tailored to improve encapsulation, protection and release characteristics over simple emulsions. Bioactives can be added in both the inner water phase and the oil phase. Multiple emulsions are prepared either by one-step emulsification or by a double emulsification process. In a one-step emulsification process, the W/O emulsion is formed with the right balance of hydrophilic–lipophilic emulsifiers and heat. It can then be inverted to form a multiple emulsion. In the double-emulsification process, the primary W/O emulsions are formed under high shear conditions, and are subsequently dispersed in a secondary water phase with mild shear to avoid disruption of the already formed primary emulsions. A double emulsification and an enzymatic gelation method using transglutaminase cross-linked protein encapsulant has been suggested by Cho et al. (2003) to improve the storage stability of fish oil and achieve controlled release.

W/O/W emulsions contain a much lower amount of fat than O/W emulsions [e.g., 14 g less fat per 100 g emulsion was reported in one case (Poyato et al. 2013)], which is an aspect to be taken into account when they are used as ingredients for functional foods (Poyato et al. 2013). Although there is widespread recognition of the value of W/O/W emulsions in contributing to the development of reduced-fat products and as vehicles for the delivery of nutrients, the potential for double emulsions in food technology has yet to be fully elucidated (Dickinson 2011). de Ciriano et al. (2010) reported the need for the use of antioxidants in highly unsaturated O/W emulsions in order to control lipid oxidation. The type of antioxidant used and the phase in which it is added need careful consideration. In olive oil and linseed oil emulsions, Poyato et al. (2013) found that depending on the type of emulsions (W/O/W or O/W) the hydrophilic antioxidant (*Melissa officinalis* extract) was more efficient in O/W emulsions, whereas the lipophilic antioxidant (BHA) was more effective in W/O/W emulsions. Rutin and anthocyanins have been successfully encapsulated within the internal aqueous phase of W/O/W multiple emulsions in the (outer droplet) size range of 13−15 μm with encapsulation efficiency of >80 % (Akhtar et al. 2013).

Multilayered Emulsions

Multiple layered emulsions are emulsion produced using electrostatic layer-by-layer deposition technologies. For example, a secondary layer with an opposite electrostatic charge may be deposited onto the interfacial layer of a primary emulsion (e.g. deposition of chitosan onto the interface of droplets in lecithin-stabilized emulsions). Multilayered emulsions offer improved stability over conventional emulsions. However, their preparation is more difficult than that of conventional

emulsions as it requires extra ingredient and processing steps. Crosslinking of polymer layers adsorbed at the interface of oil-in-water emulsions is often required to enhance the stability of multilayered emulsions. Zeeb et al. (2012) demonstrated that laccase could be used to covalently crosslink pectin onto gelatin interfacial membrane, showing that pectin remained attached to the surface after increasing the pH from 3.5 to 10 and then lowering the pH again, making it robust enough for some applications. Multilayered emulsions have a thicker interfacial layer that is more resistant to disruption compared to simple emulsions. For example, lipid droplets coated by a three-component interfacial layer (β-lactoglobulin–ι-carrageenan–gelatin) were more stable to repeated freeze–thaw cycling than those coated with either a one-component (β-lactoglobulin) or two-component (β-lactoglobulin–ι-carrageenan) layer (Gu et al. 2007a). In another example, multilayered emulsions containing 1 % (w/v) κ-carrageenan with similar values for d_{32}, ζ-potential and the rheological properties at both pH values 3.5 and 7 were produced, having improved stability of against environmental stresses such as pH values around the isoelectric point of the protein (Perrechil and Cunha 2013). A protein/polyphenol microcapsule based on (-)-epigallocatechin gallate (EGCG) and gelatin (type A) was produced using the layer-by-layer (LbL) assembly containing ∼30 % w/w EGCG that retained its antioxidant activity (Shutava et al. 2009).

Multilayered emulsions formed by mixing an oil-in-water emulsion containing relatively large anionic droplets ($d_{32} \sim 0.6$ μm, β-lactoglobulin (β-Lg)–pectin coated, pH 4) with another oil-in-water emulsion containing relatively small cationic droplets ($d_{32} \sim 0.2$ μm, β-Lg coated, pH 4) were unstable to droplet aggregation at intermediate small-droplet concentrations (due to bridging flocculation) and also at high small-droplet concentrations (due to depletion flocculation); however, relatively stable particles could be made over a range of low small-droplet concentrations, which resulted in large droplets surrounded by small droplets (Gu et al. 2007b).

Solid Lipid Particle Emulsions

Solid lipid particle emulsions contain partially emulsifier-coated solid lipid particles dispersed in an aqueous continuous phase. This type of emulsion enables better control of the release of encapsulated bioactive, improved stability, higher payloads, ability to include both lipophilic and hydrophilic bioactives in the same system, and may be easier to scale up to large-scale production (McClements et al. 2009). This has originally been used in drug delivery, but has been explored more recently for delivery of bioactives in functional foods.

Internally Self-assembled Emulsion Droplets

Internally self-assembled emulsion droplets (ISAsomes) whose core comprises lipophilic molecules such as monoglycerides, are self-assembled in well-defined liquid crystalline phases. The structure of these ISAsomes can be tuned by

temperature variation and/or the addition of oils (Glatter et al. 2010). ISAsomes are potential carrier systems for hydrophilic, amphiphilic and/or lipophilic molecules. Immobilization of particles is accomplished by the addition of a polysaccharide at elevated temperatures while in a fluid state, and the system can simply be solidified by lowering the temperature at the right moment (Guillot et al. 2009). The addition of polymers (κ-carrageenan or methylcellulose) gelifies the continuous aqueous phase, forming a hydrogel loaded with ISAsomes. Self-assembled thermo-gelling emulsions were prepared by blending ISAsomes with thermo-reversible hydrogels of methylcellulose, κ-carrageenan, and their 1:1 mixture. The ISAsomes remained practically intact during their embedding into the hydrogel matrix, retaining their internal self-assembled structure and functionality (Tomsic et al. 2009). These authors found that methylcellulose was able to stabilize the ISAsomes at higher temperatures (up to 90 °C), and their most interesting results were obtained for the ISAsome-loaded (1:1) methylcellulose:κ-carrageenan system, with a narrow intermediate temperature window where it is a sol. This specific thermal behavior allows for easy temperature tuning of the system's aggregate state and of the internal self-assembled structure (Tomsic et al. 2009), offering the potential for controlled release applications.

4.4 Nanoencapsulation

Nanoencapsulation has been increasingly used for the delivery of food bioactives (Ezhilarasi et al. 2013). Nanoencapsulation typically uses micellar or vesicular systems in which the active ingredient is confined to a cavity surrounded by a unique surfactant or polymeric membrane. The preparation of nanocapsules containing a membrane forming molecule, a co-emulsifier, and a lipophilic component has been used to encapsulate a range of food ingredients, including omega-3 fatty acids. The nanocapsules were 40–80 nm in size and were suitable for delivery of components into clear liquid drinks, as well as other beverages and foods (Weder et al. 2000). Hydrophobic nanospheres of flavour compounds encapsulated in pH-sensitive or moisture-sensitive microspheres have been shown to improve shelf life of foods and beverages and prolong the sensation of flavor (Shefer and Shefer 2003).

Nanoemulsions and nanoparticles are emerging technologies that may aid in the delivery of bioactives. Nanoemulsions are submicron emulsions comprising a liquid-in-liquid dispersion of small droplets at about 20–200 nm range (Solans et al. 2005). Their small size offers potential advantages over conventional emulsions, e.g., higher stability to droplet aggregation and to gravitational separation, high optical clarity, ability to modulate product texture, and, increased bioavailability of lipophilic components (McClements and Rao 2011). Nanoemulsions may be formed using emulsifiers and very high shear resulting in emulsified fine droplets of the bioactive that is stable against sedimentation or creaming (Gonnet et al. 2010). Addition of a large amount of emulsifiers is often required; however, using high-shear homogenization helps stabilize nanoemulsions with lower levels of surfactants in the final formulation (McClements and Li 2010; Gutierrez et al.

2003). One of the difficulties in forming nanoemulsions from triglyceride oils compared to n-alkane oils is the much higher viscosity of the triglyceride oil. The size of the triglyceride oil nanoemulsion droplets was dramatically reduced by the addition of polyethylene glycol to the water phase (Wooster et al. 2008).

Nanoparticles are dense colloidal submicron particles (often polymeric), forming a homogeneous dispersion (Anton et al. 2008). Nanoparticles have been used for the delivery of a range of bioactives. The loading of hydrophobic nutraceuticals into Maillard-conjugate based nanoparticles provides protection of the nutraceuticals against degradation and enable delivery in clear beverages (Markman and Livney 2012). Lipophilic bioactives have been delivered with lipid nanoparticles to improve their bioavailability (Yao et al. 2014). Curcumin has been loaded into lipid nanoparticles (nanoemulsions), protein nanoparticles (zein nanosuspensions) and phospholipid nanoparticles (nanoliposomes) to protect curcumin from chemical degradation, increase the solubilisation of curcumin in intestinal fluids and improve oral bioavailability (Zou et al. 2016).

The future of nanoencapsulation in food requires further consideration. Consumers are concerned due to the potential and unknown possible toxicity related to nanoparticles. With the oral ingestion of nanoemulsions, there is the possibility to change the biological fate of bioactive components within the gastrointestinal tract and the potential toxicity of some of the components used in their fabrication (McClements and Rao 2011). In dry formats, nanoparticles present high risk of inhalation and may have explosive properties (Sleigh and Barton 2011). On the other hand, nanodelivery systems for nutraceuticals and other bioactives offer many advantages, including better colloidal stability, better sensory properties (suppression of undesired attributes of certain nutraceuticals), improved bioavailability, improved stability to degradation of the bioactive, and more.

4.5 Emerging Trends

Developments of microencapsulation technologies for stabilization of bioactives have been established in the last few decades. More recently, the development has focused on new microencapsulation technologies that can control the release of the bioactive at target sites in the body. Microencapsulation of bioactive components offers benefits in the development of food products with health promoting or disease preventing effects (de Vos et al. 2010). With the increasing demand for functional foods with associated health benefits, the demand for more robust microencapsulation technologies and formulations that can resist bioactive degradation during processing, shelf life and digestion are expected to grow. More robust formulations will facilitate the incorporation of bioactives into various foods and beverages.

The development of microencapsulation technologies that capitalize on applying novel combinations of different technologies to achieve the desired functionalities in the microcapsules will continue to emerge. It is, however, important to bear in mind that the more complex the technology is, the more costly it will usually be.

Spray drying and cooling have the lowest cost, fluid bed coating and spinning disk have medium cost, while coacervation and certain chemical processes have the highest cost. The question of necessity for development of more sophisticated microencapsulation technologies therefore needs to be asked. The goal of promoting human health would better be served by developing simple encapsulation technologies, and addressing bioavailability and safety concerns, to facilitate wider incorporation of nutraceuticals in food and beverages.

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