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Microencapsulation and Its Uses in Food Science and Technology: A Review

Pedro Henrique Rodrigues do Amaral, Patrícia Lopes Andrade and Leilane Costa de Conto

Abstract

Microencapsulation is a group of technologies aiming to produce small particles called microcapsules that can be released at a specific speed under certain conditions. Microencapsulation technology is used in the pharmaceutical, agrochemical, and food industries; however, microcapsule production is most challenging for applications in the food industry owing to the high costs of the technique, which may make the final product too expensive. Common methods for microencapsulation include spray-drying and coacervation, and different wall materials and filling materials can be used for both techniques. In this review, we summarize current methodologies used for microencapsulation, with a focus on applications in the food industry.

Keywords: microencapsulation, food industry, nutrient enrichment, wall material, core

1. Introduction

Currently, food manufacturers and scientists worldwide are aiming to identify and characterize foods that can be used as sources of beneficial nutrients to promote the health and well-being of consumers. Based on this new paradigm, the development of new food products must combine novel technologies with the use of traditional methods to the control bio-accessibility of certain components in foods. As the interactions among health, nutrition, and genetics are clarified, this approach will become increasingly important.

One effective method for achieving these aims is microencapsulation [1], which was used as early as 1930. The first products containing microencapsulated materials were successfully fabricated in 1954. This advancement promoted further research on the use of microencapsulation in the pharmaceutical industry, wherein researchers found they could use these techniques to achieve controlled release of drugs in the body or in specific organs. Thus, pharmaceutical companies were crucial for developing improved techniques for microencapsulation [2]. In the 1960s, the first studies of microencapsulation in food technology were performed using essential oils; scientists attempted to prevent lipid oxidation, volatile compound losses, and aroma-controlled release. Subsequently, many more studies regarding microencapsulation of food products were published [3].

The goals of microencapsulation are to coat an active compound (core) by an encapsulating agent, also known as wall material, which will isolate the active material, thereby protecting the active material from adverse changes or to hide sensory properties that are not appreciated by consumers. The isolation provided by the encapsulating material will break under the application of a specific stimulus (e.g., pH or heat), releasing the active substance in the specific target location or under ideal conditions [2].

In this review, we summarize the latest applications of microencapsulation and microcapsule production methods in the food industry.

2. Microencapsulation in the food industry

The techniques for producing microcapsules are significantly more challenging in the food industry than in other industries because the sensory qualities of foods cannot be compromised by the addition of encapsulated components. Furthermore, food matrices are more complex than those used in pharmaceutical and cosmetic industries. Moreover, in the food industry, microcapsules must be ingested orally, resist the adverse conditions of the gastrointestinal tract, and exhibit mucoadhesive properties [1]. Several different methods for microcapsule production have been developed, and microcapsules can be fabricated using various materials, which are chosen depending on the function of the microcapsules [4].

Microencapsulation is used to reduce adverse aromas, volatility, and reactivity of food products and to provide food products with greater stability when exposed to adverse conditions (e.g., light, O₂, and pH) [5, 6]. Favaro-Trindade et al. [1] stated that microencapsulation is used in the food industry to reduce the reactivity of the active material in the external environment, reduce the speed of losses and evaporation of the core material into the medium, improve food handling, provide controlled release of the active product, mask unpleasant odor and taste, and allow the encapsulated material to be distributed in a food formulation homogeneously. However, microencapsulation is associated with dramatically increased costs of production, which may limit the economic viability of the method.

Notably, consumers are becoming increasingly aware of the importance of consuming meals that benefit health. Thus, products are being developed to provide health benefits to consumers; microencapsulation of various active compounds, such as vitamins, minerals, essential oils, and omega-3 polyunsaturated fat acids, among others, may be used to protect these compounds from nutrient loss and oxidation reactions and to hide sensory characteristics [2]. Therefore, while there are a wide range of applications of microencapsulation in the food industry, more studies are needed to determine the effectiveness of microencapsulation and the consumer acceptance of products manufactured using microencapsulation [7].

2.1 Microencapsulation processes

Microencapsulation is the science of trapping components (core or active) into a secondary material (encapsulant, wall material, carrier, or cover), producing small solid particles (1–500 µm in size) [8]. These particles are able to release their contents at a specified rate or under specific conditions [1].

The first step in microencapsulation consists of mixing the active material with the encapsulant material, making an emulsion. The mixture can be made with one or two agents. The mixture is then dried, producing microcapsules of different diameters and forms depending on the preparation method and materials used [7].

Physicochemical methods (simple or complex coacervation, separation of organic phase, and liposomal wrapping), physical methods (spray-drying, spray chilling, spray coating, fluidized bed, extrusion, centrifugation with multiple orifices, co-crystallization, and lyophilization), and chemical methods (interfacial polymerization and molecular inclusion) have been developed for microencapsulation [9].

Techniques and materials for microencapsulation are described in **Table 1** [1].

The methods most used by the food industry and which deserve attention are described below.

2.1.1 Coacervation

The oldest microencapsulation technique and one of the most widely used techniques is coacervation, which involves macromolecular aggregates that form a colloidal system with two existing phases: one that is rich in colloids (coacervate) and one that is poor in colloids (supernatant). This method is performed by depositing the encapsulating agent around the active compound through physicochemical changes, such as temperature, polarity, pH, or ionic strength [2, 6].

Methods for encapsulation	Encapsulated materials
<i>Physical methods</i>	
Stationary extrusion	Liquid/solid/gas
Submerged nozzle	Liquid/solid/gas
Centrifugal extrusion	Liquid/solid/gas
Vibrant nozzle	Liquid/solid/gas
Spray-drying	Liquid/solid
Rotating disc	Liquid/solid
Pan coating	Solid
Air suspension	Solid
Spray chilling and spray cooling	Liquid/solid
Fluidized bed	Solid
Co-crystallization	Liquid/solid
Lyophilization	Liquid
<i>Chemical methods</i>	
Interfacial polymerization	Liquid/solid
Molecular inclusion	Liquid
In situ polymerization	Liquid/solid
<i>Physical-chemical methods</i>	
Simple coacervation	Liquid/solid
Complex coacervation	Liquid/solid
Liposomes	Liquid/solid
Lipospheres (solid lipid nanoparticles and nanostructured lipid carriers)	Liquid/solid
Evaporation of the solvent	Liquid/solid

Table 1.
Methods and kind of materials utilized for food products encapsulation.

Coacervation occurs when medium changes make the wall material form polymeric chain units, which then interact with others close chains, forming aggregates. After this step, the aggregates interact with each other through high-intensity attraction forces. Consequently, the aggregated polymer chains will be deposited around the droplets of the hydrophobic phase dispersed in the emulsion, forming a protective film [4].

The microcapsules obtained by coacervation can be classified as mononuclear or multinuclear according to their internal structure. When a drop of core material is encapsulated by coacervation, the particle formed is mononuclear; multinuclear particles are formed by aggregation of various mononuclear microcapsules. Multinuclear microcapsules have a matrix structure, and the core material can be released slowly unless the wall has been broken. However, mononuclear microcapsules have a vessel structure and release all their contents quickly. These particles are also irregular in structure because the wall material is not equally distributed over the surface of the core drop. The thinnest part of the wall layer will be more susceptible to disruption and release of the core.

Therefore, multinuclear microcapsules have greater controlled release and are produced more easily than mononuclear microcapsules [10].

The microcapsules produced by coacervation may have small diameters ranging from 1 to 500 μm for complex coacervation and from 20 to 500 μm for simple coacervation [1]. An example is presented in **Figure 1**. When using lipophilic materials with a hydrophilic coating, high encapsulation efficiency (85–90%) is generally observed [11–15].

Complex coacervation has been used for microencapsulation of sensitive microorganisms and compounds, such as probiotics bacteria, omega-3 products, and bioactive compounds [16–18].

2.1.2 Spray-drying

The use of spray-drying for microencapsulation is another widely used technique due to its low-cost and easy application [19]. Spray-drying technique is used in the food industry for microencapsulating juice, pulp, and vegetal extracts [20, 21], probiotics [22], and fish oil [23].

During spray-drying, a homogeneous mixture of the active material and wall material in aqueous or organic solution is subjected to a hot airstream that promotes the evaporation of the solvent drying the microcapsules. Thus technique generates no solvent residues and does not require a washing process. However, the use of

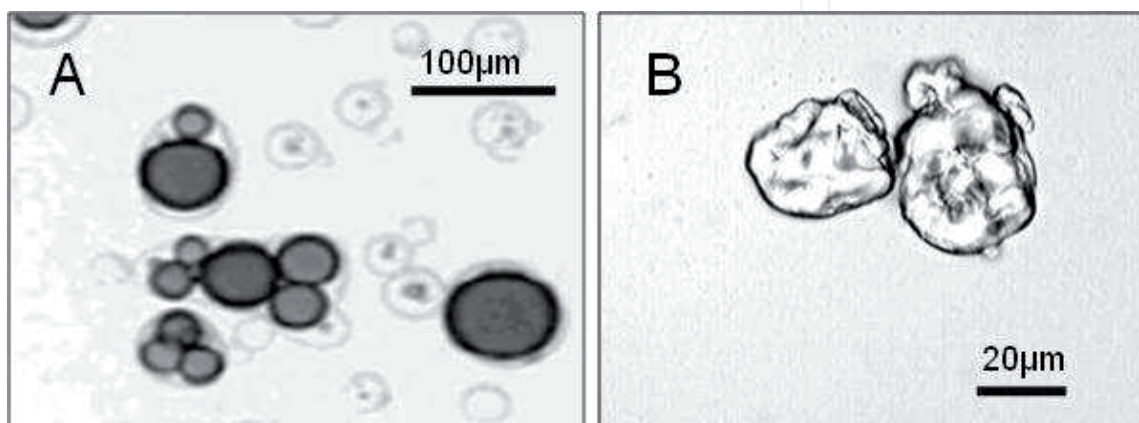


Figure 1. Microcapsules obtained by complex coacervation with gelatin/gum arabic (A) and soybean protein (B). Source: Author.

high temperatures may compromise the integrity of the core and wall materials. This microcapsule production process has a high efficiency rate, which can be affected by the concentration of the wall material, the system speed, and the feed temperature [24]. Moreover, spray-drying is more widely used than other methods owing to its relatively low cost and capacity for large-scale production [25].

However, according to Kolanowski et al. [11], spray-drying results in porous particles, and this characteristic may increase the susceptibility of the core material to oxidation. Additional disadvantages include the requirement for expensive equipment and the irregularity of the produced microcapsules [6, 24].

Table 2 shows some recent studies about spray-drying application on food microencapsulation.

2.1.3 Fluidized bed

In fluidized bed encapsulation, while the particles of the core material are suspended, the wall material is atomized into the chamber, depositing on the core. When the particles reach the top of the ascending column, they are released into a descending column of air which releases them back into the fluidized bed, where they are again coated, dried, and hardened, ensuring a uniform coating. Fluidized bed encapsulation is one of the few technologies that allow particles to be coated with any wall material (polysaccharides, proteins, emulsifiers, fats, etc.). This method has been used, for example, to isolate iron from ascorbic acid in multivitamin formulations or to encapsulate salt and acidulants avoiding, this way, the interaction of such ingredients with others [34].

Regardless of the method for microencapsulation, release of the core material depends on various factors, including pH, temperature, diffusion, medium solubility, mechanical rupture, and biodegradation. Additionally, the thickness of the encapsulating material may alter the stability and permeability of the microcapsules [1].

2.1.4 Molecular inclusion

One of the most promising possibilities of flavor stabilization is the formation of inclusion complex (molecular encapsulation) with β -cyclodextrin. Szente and

Paper	Source
Flavonoid microparticles by spray-drying: influence of enhancers of the dissolution rate on properties and stability	Sansone et al. [26]
Microencapsulation of linseed oil by spray-drying for functional food application	Gallardo et al. [27]
Optimization of microencapsulation of fish oil with gum arabic/casein/beta-cyclodextrin mixtures by spray-drying	Li et al. [28]
Retention of saffron bioactive components by spray-drying encapsulation using maltodextrin, gum arabic, and gelatin as wall materials	Rajabi et al. [29]
Spray-drying microencapsulation of synergistic antioxidant mushroom extracts and their use as functional food ingredients	Ribeiro et al. [30]
Spray-drying microencapsulation of cinnamon infusions (<i>Cinnamomum zeylanicum</i>) with maltodextrin	Santiago-Adame et al. [31]
Influence of different combinations of wall materials on the microencapsulation of jussara pulp (<i>Euterpe edulis</i>) by spray-drying	Santana et al. [32]
Sulfur aroma compounds in gum arabic/maltodextrin microparticles	Uekane et al. [33]

Table 2.
Spray-drying studies for microencapsulated food products.

Szejtli [35] investigating the stabilization of natural and synthetic coffee compounds with β -cyclodextrin, and thermal stability of this carbohydrate, observed the molecular encapsulation with natural and synthetic coffee compounds. They also noted that β -cyclodextrin is thermally destroyed at 260°C.

Inclusion compounds of β and γ -cyclodextrins with essential oils of lemon, orange, and camomile have been studied. Lemon and orange oils resulted in the union with β and γ -cyclodextrin. With camomile oil, the complex observed was only with γ -cyclodextrin [36].

2.2 Wall materials

The wall material should be able to form a film that is cohesive with the core material, be chemically compatible and nonreactive with the core material, and provide the desired coating properties, for example, strength, flexibility, impermeability, and stability [37]. In order to be able to be applied in food, the wall material must be food grade, biodegradable, and capable of forming a barrier between the active agent and the medium [19].

Importantly, some core materials are insoluble in aqueous solutions and may not easily form emulsions [38]. Specific proteins may function as emulsifiers, and polysaccharides contribute to the stability of emulsions; the interactions between proteins and carbohydrates can also help stabilize the emulsions.

Among the polysaccharides utilized as wall materials, gum arabic, maltodextrins, and modified starches are the most usual because of the high molecular weight and the high glass transition temperature [19]. However, other polysaccharides are also used, such as carrageenan, carboxymethylcellulose (CMC), and chitosan.

2.2.1 Polysaccharides

Gums are a group of polysaccharides and polysaccharide derivatives obtained from plants or secreted by bacteria and are commonly used for microcapsule production in the food industry.

Gum arabic has low viscosity in water, provides good retention of volatile compounds (>85%), and protects the core material from oxidation, which is crucial for microencapsulating essential oils and volatile substances [7]. Gum arabic has advantages for having this property emulsifier in a wide pH range, as well as other texturing, training film around the droplets and binding properties [38]. Conto et al. [17] studied the complex coacervation of soy proteins with gum arabic (GA); Renard et al. [39] worked with vitamin E microencapsulated on β -lactoglobulin/GA matrix.

Alternatively, alginate can be used for microencapsulation. This material forms strong, elastic gels with a distinct three-dimensional network. The gel network and homogeneity depends on the cation concentration; excess Ca^{2+} may result in multiple alginate chains having different physicochemical properties.

Alginate can also be used to produce microcapsules and cell immobilization through ionotropic gelation, which involves dropping the concentrated alginate solution into calcium chloride solution, externally gelling the polymer into a microcapsule. The size of the microcapsules formed using external gelation is governed by the size of droplets formed during the extrusion process [40] and ranges from tens of microns to millimeter size. Less commonly, microcapsules may be formed by internal gelation, in which the alginate in solution contains calcium carbonate [41].

Another common use of alginate microcapsules is to reduce the viability losses of probiotic bacteria, like *Bifidobacterium* and *Lactobacillus*. Some works with probiotics alginate encapsulation are presented by Cook et al. [42] and are summarized in **Table 3**.

Encapsulation material	Bacteria	Reference apud Cook et al. [42]
Alginate	<i>Lactobacillus acidophilus</i>	Chandramouli et al. [40]
Alginate coated with palm oil and poly-L-lysine	8 different <i>Lactobacilli</i> and <i>Bifidobacteria</i>	Ding et al. [43]
Alginate and xanthan gum	<i>Lactobacillus acidophilus</i>	Kim et al. [44]
Alginate coated with either chitosan, alginate, or poly-L-lysine-alginate	<i>Lactobacillus acidophilus</i> , <i>Bifidobacterium bifidum</i> , <i>Lactobacillus casei</i>	Krasaekoopt et al. [45]
Alginate	<i>Lactobacillus casei</i>	Mandal et al. [46]
Alginate Alginate and pectin	<i>Lactobacillus casei</i>	Sandoval-Castilla et al. [41]
Alginate coated with whey protein	<i>Lactobacillus plantarum</i>	Gbassi et al. [47]
Alginate coated with chitosan, Sureteric, or Acryl-EZE	<i>Bifidobacterium animalis</i>	Lisserre et al. [48]
Alginate coated with chitosan	<i>Bifidobacterium breve</i>	Cook et al. [42]

Table 3.
 Overview of literature available on the alginate encapsulation of probiotics cited by Cook et al. [42].

Carrageenans are widely used as thickening and stabilizing agents. Previous studies have reported microcapsules produced by carrageenan and oligochitosan polymer, but most reports have described the use of carrageenan for the encapsulation of microbial cells due to its capacity for gelation with the change in temperature from 40 to 45°C [49], suggesting the potential for use in probiotic foods.

Starch and modified starch can also be used as a wall material owing to its low viscosity, outstanding retention volatility (>93%), and ability to stabilize the emulsion with the core material [7]. Starches and their derivatives have been applied for the microencapsulation of vitamins, such as ascorbic acid [50, 51]. Maltodextrin, which is inexpensive and has low hygroscopicity, can be used to prevent particle agglomeration [17] and has antioxidant effects [7].

Chitosan is also commonly used as a gelation agent in the food and pharmaceutical industries. Chitosan also allows concurrent cell permeabilization and immobilization; thus, chitosan-containing complexes of coacervated capsules have been widely explored [52].

Additionally, carboxymethylcellulose (CMC), an anionic water-soluble polymer, is used as an industrial agent owing to its capacity as a thickener, suspending agent, stabilizer, and binder. CMC forms resistant films that can protect against organic solvents, oils, and greases [53].

2.2.2 Protein films

Protein films are excellent oxygen and aroma barriers and can be used to produce microcapsules using coacervation techniques [54] or double emulsions with subsequent reticulation using glutaraldehyde or heat gelation [55]. Usually, proteins have been utilized with other biopolymers; some examples are presented in **Table 4**.

Whey proteins (WPC and WPI) have also been investigated as wall materials for microencapsulation. For example, whey protein has been used for encapsulation of volatile and nonvolatile materials [56], typically through spray-drying, complex coacervation, heat gelation, and enzymatic gelation [57, 58]. Combinations such as whey protein isolate/gum arabic [59], β -lactoglobulin/pectin [60], β -lactoglobulin

(b-Lg)/κ-carrageenan [61], whey protein/chitosan/gum arabic [62], and milk protein products/xanthan [63] are frequently used.

Despite these studies, proteins from plant sources have not been commonly used as carrier or wall materials in microencapsulation applications owing to limitations related to heat instability and organic solvent sensitivity. However, the use of reticulating agents to convert the proteins into a more stable form may improve their industrial applicability [64].

Additionally, soy proteins have the benefits of renewability, low-cost, and healthful effects [65]. Soy protein has high compatibility with gum arabic. SPI has been successfully used for microencapsulation of casein hydrolysate by spray-drying [66], orange essential oil by complex coacervation [13], and fish oil by enzymatic gelation [57]. **Figure 2** presents microcapsules obtained with SPI by complex coacervation and enzymatic gelation.

Gelatin can also be used as a foaming agent, emulsifier, and humectants in food, pharmaceutical, medical, and technical application owing to its surface-active properties. Type A gelatin has a high isoelectric point and can form oil/water emulsions with positive charges at a wide range of pH values [67].

2.3 Core materials

Among core materials, essential oils are highly unstable and are sensitive to variations in light, air, temperature, and humidity. Therefore, new methods are needed to protect oils against these changes in order to increase their shelf life and their chemical stability under adverse conditions [1, 7].

Wall material	Core material	Source
Gelatin/gum arabic	Soybean oil, olive oil, and peanut oil	Rabišková, Valasková [68]
Gelatin/gum arabic	EPA	Lamprecht, Schäfer, Lehr [69]
Gelatin/gum arabic	Fish oil	Jouzel et al. [70]
Whey protein/gum arabic	Sunflower oil, lemon, and orange essential oil	Weinbreck, Minor, DeKuif [58]
Hydroxypropyl methylcellulose	Fish oil	Wu, Chai, Chen [71]
Gelatin/gum arabic	Oleoresin and soybean oil	Alvim [72]
Gelatin/gum arabic	Baking flavor	Yeo et al. [73]
Gelatin/pectin/gum arabic	Oils	Prata [74]
Gelatin/gum arabic	Peppermint oil	Dong et al. [10]
Gelatin	Stigmasterol	Oliveira [75]
SPI/pectin	Casein hydrolyzate	Medanha et al. [76]
b-Lg/pectin	DHA	Zimet, Livney [77]
Gelatin/polyphosphate	Fish oil ethyl ester	Barrow, Nolan, Holub [78]
Gelatin/gum arabic	Flavors	Leclercq, Milo, Reineccius [79]
Gelatin/gum arabic	Soybean oil and paprika resin oil	Célis [80]
Gelatin/gum arabic	1-Dodecanol (C12OH)	Kong et al. [81]
HPMC/NaCMC/SDS	Sunflower oil	Katona, Sovilj, Petrovic [82]
SPI/gum arabic	Orange essential oil	Jun-Xia, Hai-Yan, Jian [13]

Table 4.
Overview of literature available on the proteins as encapsulant material.

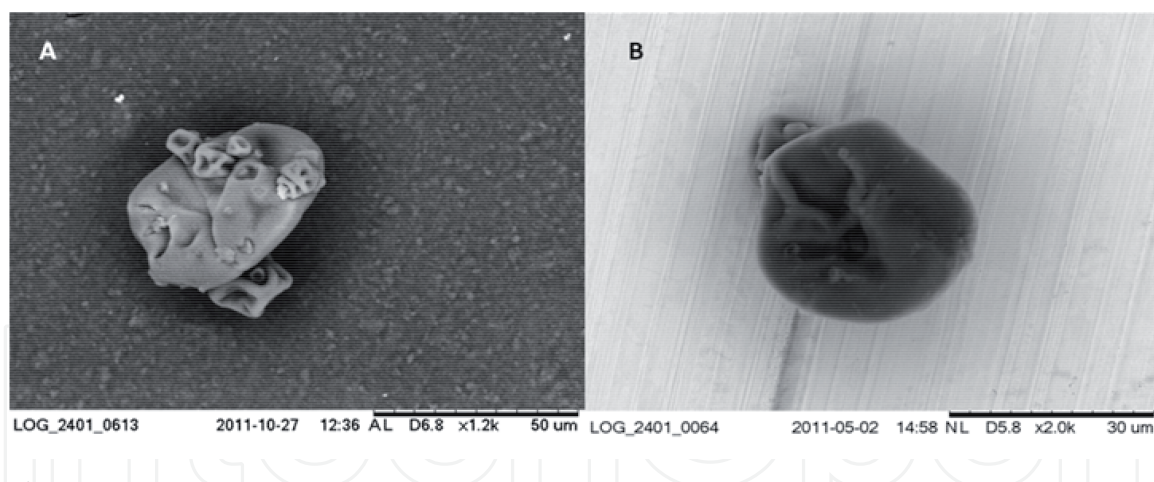


Figure 2. MEV of SPI microcapsules obtained by complex coacervation (A) and enzymatic gelation (B). Source: Author.

Vitamins and minerals are generally added to foods to increase nutritional value, such as cereals, dairy products, infant foods, etc. However, these compounds can cause the food to taste unpleasant or may react with other food constituents, changing their sensory characteristics. Therefore, microencapsulation is widely used to protect vitamins and minerals against adverse conditions, such as temperature and humidity, to prevent undesirable reactions in food [1].

Microorganism microencapsulation has been applied to allow reuse of bacteria during the production of lactic acid and fermented milk products; increase production and cell concentrations in reactors; provide protection against oxygen gas, freezing temperatures, and the unfavorable pH of gastric juices and other acids; remove cell sand stop acidification; provide greater stability and maintain the viability of cultures during product storage; and increase their useful life [1].

Microencapsulation is widely used for enzyme immobilization, allowing reuse of enzymes and providing enzymes with superior stability; these features also reduced costs associated with the relevant processes. Moreover, microencapsulation for immobilization of enzymes is simple and permits the production of microcapsules having a variety of compositions [83].

Many studies have shown that consumption of omega-3 polyunsaturated fatty acids provides multiple health benefits, including reducing the risk of cardiovascular disease. Polyunsaturated fatty acids of the omega-3 group are mainly found in marine animals, such as plankton and fish in cold and deep waters, and fish oil has been the traditional source of these fatty acids. However, fish oil has an undesirable flavor. Thus, microencapsulation has been used for incorporation of fish oil as the core material, hiding the unpleasant sensory characteristics of the oil [17].

In studies with omega-3 microcapsules applied in food products, Chavez-Servín et al. [84] examined the addition of microencapsulated omega-3 fatty acids in infant formulas. Lysine and lactose degradation were observed; however, it was determined that the microcapsules did not affect the sensory acceptance of the final product. Moreover, Yep et al. [85] applied omega-3 microcapsules in bakery products and evaluated the effects of consumption of small daily doses of omega-3 fatty acids by intake of commercial bread compared to supplementation with capsules. They concluded that the effects depended on the amount of EPA and DHA in the blood of the individuals studied. Serna-Saldivar et al. [86] determined the shelf life of bread enriched with DHA and microencapsulated fish oil, showing that the development of off-flavors occurred more quickly in the breads containing liquid fish oil. Davidov-Pardo et al. [87] also observed changes in technological and sensory characteristics of breads containing omega-3 microcapsules.

The encapsulation of acids such as ascorbic, citric, fumaric, and lactic acids is usually carried out to avoid oxidation and allow them to be dissolved under specific conditions. Three specific applications stand out in this case: the dough improver, because the encapsulated ascorbic acid is often used to alter dough strength and improve slicing properties, color, and texture of baked products, being released during the proofing and baking stages, the aroma complement, and as an auxiliary agent in the meat processing, allowing the desired cured meat pigments to form [88].

Some natural colorants, such as *urucum*, β -carotene, and *Curcuma*, have solubility problems. It can be solved by encapsulation processes, which make them easier to handle during the process and improve the solubility and oxidation stability. Another advantage that can be associated with its use is in the shelf life extension, which can exceed 2 years, compared to the 6 months for non-encapsulated ones [88].

3. Conclusion

Foods and other substances microencapsulated exhibit wide applicability, being an effective and extremely important tool in the preservation of various nutritional components, microorganisms, enzymes, dyes, etc., protecting food and other products from the most aggressive processing methods.

Several materials can be used as encapsulants, the most common being carbohydrates and some proteins, due to their higher affinity with various types of materials to be encapsulated. There are several methods of encapsulation by physical, chemical, and physicochemical, the most used being atomization, fluidized bed, and coacervation.

Despite the wide applicability, encapsulation has found little space in the food industry because of the cost. While the pharmaceutical and cosmetic sectors often support the use of high-cost techniques, the food industry works with lower profit margins, reducing production costs. In addition, industries often have strong resistance to the adoption of new technologies, due to the cost of implementation and the need for training.

Development of methodologies for incorporation of functional compounds in foods is needed to improve the health benefits and marketability of foods. Finally, microencapsulation of nutrients is a relatively new technology in the food industry, and further studies are needed to determine how to apply this technology most effectively.

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