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# Microencapsulation: concepts, mechanisms, methods and some applications in food technology

## Microencapsulação: conceitos, mecanismos, métodos e algumas aplicações em tecnologia de alimentos

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**- REVIEW -**

**ABSTRACT**

*Microencapsulation is a process in which active substances are coated by extremely small capsules. It is a new technology that has been used in the cosmetics industry as well as in the pharmaceutical, agrochemical and food industries, being used in flavors, acids, oils, vitamins, microorganisms, among others. The success of this technology is due to the correct choice of the wall material, the core release form and the encapsulation method. Therefore, in this review, some relevant microencapsulation aspects, such as the capsule, wall material, core release forms, encapsulation methods and their use in food technology will be briefly discussed.*

**Key words**: *microcapsules, microencapsulation, controlled release.*

**RESUMO**

*A microencapsulação é um processo em que substâncias ativas são revestidas por cápsulas extremamente pequenas. É uma tecnologia nova, a qual tem sido empregada na indústria de cosméticos, farmacêutica, agrotóxicos e alimentícia e, nesta, é utilizada em aromas, ácidos, óleos, vitaminas, micro-organismos, entre outros. O êxito nessa tecnologia deve-se à correta escolha do material encapsulante, da forma de liberação do núcleo e do método de encapsulação. Dessa forma, nesta revisão, serão abordados, sucintamente, alguns aspectos relevantes da microencapsulação, como a cápsula, o material encapsulante, as formas de liberação do núcleo, os métodos de encapsulação, assim como sua utilização na tecnologia de alimentos.*

**Palavras-chave**: *microcápsulas, microencapsulação, liberação controlada.*

## INTRODUCTION

Microencapsulation may be defined as the packaging technology of solids, liquid or gaseous material with thin polymeric coatings, forming small particles called microcapsules (GHARSALLAOUI et al., 2007). The polymer acts as a protective film, isolating the core and avoiding the effect of its inadequate exposure. This membrane dissolves itself through a specific stimulus, releasing the core in the ideal place or at the ideal time (SUAVE, 2006).

Microencapsulation has numerous applications in areas such as the pharmaceutical, agricultural, medical and food industries, being widely used in the encapsulation of essential oils, colorings, flavorings, sweeteners, microorganisms, among others (AZEREDO, 2005).

Recently, the food industry has demonstrated increasingly complex formulations: as microorganisms in fermented meat; the addition of polyunsaturated fatty acids that are susceptible to auto-oxidation in milk, yogurts or ice creams; and the use of flavor compounds that are highly volatile in instant foods, which often can only be checked by microencapsulation (KHAN et al., 2011; GHARSALLAOUI et al., 2012).

Microencapsulation can serve as an effective means of creating foods that are not only a source of nutrients with sensory appeal but also a source of well-being and health for individuals, such as by increasing the level of calcium to prevent osteoporosis, using microorganism-produced lactic acid to decrease cholesterol and adding phenolic compounds to prevent heart problems (OLIVEIRA et al., 2002; SANGUANSRI & AUGUSTIN, 2006).

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In this review, some relevant aspects of microencapsulation, such as the capsule, wall material, core release forms, encapsulation methods and some of their uses in food technology will be briefly discussed.

Capsule

Generally, capsules can be classified according to their size: macrocapsules (>5,000μm), microcapsules (0.2 to 5,000μm) and nanocapsules (<0.2μm). In terms of their shape and construction, capsules can be divided into two groups: microcapsules and microspheres. Microcapsules are particles consisting of an inner core, substantially central, containing the active substance, which is covered with a polymer layer constituting the capsule membrane. Mononuclear and polynuclear microcapsules can be distinguished by whether the core is divided (FAVARO-TRINDADE et al., 2008).

In contrast, microspheres are matrix systems in which the core is uniformly dispersed and/or dissolved in a polymer network. Microspheres may be homogeneous or heterogeneous depending on whether the core is in the molecular state (dissolved) or in the form of particles (suspended), respectively (SILVA et al., 2003).

Wall materials

The correct choice of the wall material is very important because it influences the encapsulation efficiency and stability of the microcapsule. The ideal wall material should have the following characteristics: not reactive with the core; ability to seal and maintain the core inside the capsule; ability to provide maximum protection to the core against adverse conditions; lack an unpleasant taste in the case of food applicability and economic viability (GHARSALLAOUI et al., 2007; NAZZARO et al., 2012).

According to FÁVARO-TRINDADE et al. (2008), most wall materials do not have all the desired properties; a common practice involves mixing two or more materials. Such materials can be selected from a wide variety of natural and synthetic polymers, including the following that we highlight: carbohydrates: starch, modified starches, dextrins, sucrose, cellulose and chitosan; gums: arabic gum, alginate and carrageenan; lipids: wax, paraffin, monoglycerides and diglycerides, hydrogenated oils and fats; inorganic materials: calcium sulfate and silicates; proteins: gluten, casein, gelatin and albumin.

Controlled core release

According to GOUIN (2004), encapsulation should allow the core to be isolated from the external environment until release is desired. Therefore, the release at the appropriate time and place is an extremely important property in the encapsulation process, improving the effectiveness, reducing the required dose of additives and expanding the applications of compounds of interest. The main factors affecting the release rates are related to interactions between the wall material and the core. Additionally, other factors influence the release, such as the volatility of the core, ratio between the core and wall material, particle size and viscosity grade of the wall material (ROBERTS & TAYLOR, 2000).

The main mechanisms involved in the core release are diffusion, degradation, use of solvent, pH, temperature and pressure. In practice, a combination of more than one mechanism is used (DESAI & PARK, 2005). Diffusion occurs especially when the microcapsule wall is intact; the release rate is governed by the chemical properties of the core and the wall material and some physical properties of the wall. For example, some acids can be released during a process step but protected by another step. In some cases, some preservatives are required at the product surface, but their spread to other parts must be controlled (AZEREDO, 2005).

According to ROSEN (2006), degradation release occurs when enzymes such as proteases and lipases degrade proteins or lipids, respectively. An example is reducing the time required for the ripening of cheddar cheese by 50% compared with the conventional ripening process (HICKEY et al., 2007).

In contact with a solvent, the wall material can dissolve completely, quickly releasing the core or start to expand, favoring release. For example, microencapsulation of coffee flavors improves the protection from light, heat and oxidation when in the dry state, but the core is released upon contact with water (FRASCARELI et al., 2012).

The pH release occurs because pH changes can result in alterations in the wall material solubility, enabling the release of the core. For example, probiotic microorganisms can be microencapsulated to resist the acid pH of the stomach and only be released in the alkaline pH of the intestine (TOLDRÁ & REIG, 2011).

Changes in temperature can promote core release. There are two different concepts: temperature-sensitive release, reserved for materials that expand or collapse when a critical temperature is reached, and fusion-activated release, which involves melting of the wall material due to temperature increase. An example is the fat-encapsulated cheese flavor used in microwave popcorn, resulting in the

uniform distribution of the flavor: the flavor is released when the temperature rises to 57-90°C (PARK & MAGA, 2006).

Pressure release occurs when a pressure is applied to the capsule wall, such as the release of some flavors during the mastication of chewing gum (WONG et al., 2009). Some wall materials and the possible mechanisms for the microcapsules release are listed in table 1.

Some encapsulation methods

The choice of the most suitable method depends on the type of core, the application for the microcapsule, the size of the particles required, the physical and chemical properties of the core and the wall, the release mechanism required, the production scale and the cost (SUAVE et al., 2006). According to CABALLERO et al. (2003), the main encapsulation methods are: spray drying, spray cooling, extrusion, coacervation, lyophilization and emulsification.

Spray drying

This process involves the formation of an emulsion, solution or suspension containing the core and wall material, followed by nebulization in a drying chamber with circulating hot air. The water evaporates instantly in contact with the hot air, and the material encapsulates the core (LAOHASONGKRAM, 2011). Atomization has some advantages over other methods: large equipment availability, possibility of employing a wide variety of encapsulating agents, potentially large-scale production, simple equipment, good efficiency, reduced storage and transport costs and low process cost. The main disadvantage of atomization is the production of non-uniformly sized materials (MADENE et al., 2006).

The spray drying technique is the most common microencapsulation method, has been used for decades to encapsulate mainly flavors, lipids, and pigments, but its use in thermo-sensitive products, such as microorganisms and essential oils, can be limited because the required high temperature causes volatilization and/or destruction of the product (GHARSALLAOUI et al., 2007).

The sumac flavor has been successfully encapsulated by spray drying in sodium chloride in salted cookies, salads and crackers (BAYRAM et al.,

Table 1 - Wall materials and their potential release mechanisms.

| Wall Materials | Release Mechanisms | | | |
|---|---|---|---|---|
| | Mechanic | Thermal | Dissolution | Chemical |
| Soluble in water | | | | |
| Alginate | x | | x | |
| Carrageenan | x | | x | |
| Caseinate | x | | x | |
| Chitosan | x | | | |
| Modified cellulose | x | | x | |
| Gelatin | x | | | |
| Xanthan gum | x | x | | |
| Arabic gum | x | x | | |
| Latex | x | | x | |
| Starch | x | | x | |
| Insoluble in water | | | | |
| Ethylcellulose | x | | | |
| Fatty alcohols | x | x | | x |
| Fatty acids | x | x | | x |
| Hydrocarbon resin | x | x | | |
| Mono, di and triacyl glycerol | x | x | | |
| Natural waxes | x | x | | |
| Polyethylene | x | x | | |

Source: adapted from FAVARO-TRINDADE et al. (2008).

2005). KRISHNAN et al. (2005) microencapsulated cardamom oleoresin by spray drying in arabic gum, maltodextrin and modified starch, the results showing an increase in the oleoresin protection. ANEKELLA & ORSAT (2013) optimized the microencapsulation of probiotics in raspberry juice by spray drying in 91.15%. The encapsulation of lipids in potato starches, tapioca and corn by spray drying has been successful, with no interactions between the encapsulated and wall materials (DRUSCH et al., 2006).

Spray cooling

According to CHAMPAGNE & FUSTIER (2007), spray cooling microencapsulation is based on the injection of cold air to allow solidification of the particle. Microparticles are produced from a mixture containing the core and wall material in droplets. This mixture is nebulized by an atomizer and enters a chamber in which air flows at low temperature. The reduction of temperature results in the solidification of the wall material, enabling the core to be encapsulated.

Spray cooling microencapsulation is considered the cheapest encapsulation technology by employing lower temperatures and with a high potential for scale-up. However, microparticles can present some disadvantages, including low encapsulation capacity and the expulsion of the core during storage. Spray cooling has been used to encapsulate mainly minerals and vitamins (RATHORE et al., 2013).

GAMBOA et al. (2011) microencapsulated tocopherols in a lipid matrix by the spray cooling with values of encapsulation efficiency greater than 90%. WEGMULLER et al. (2006) developed microcapsules by spray cooling that contained iron, iodine and vitamin A to fortify salt using oil hydrogenated palm. The microcapsules obtained were highly stable and no sensory differences were detected. The encapsulating agent maltodextrin was shown to be efficient to prevent the oxidation of linseed oil by spray cooling (GRATTARD et al., 2002).

Extrusion

This method is based on a polysaccharide gel that immobilizes the core when in contact with a multivalent ion. Extrusion involves incorporating the core in a sodium alginate solution, followed by the mixture undergoing drop-wise extrusion via a reduced caliber pipette or syringe into a hardening solution, such as calcium chloride (SWARBRICK, 2004).

The main advantage of this process is the very long shelf life of flavor compounds due to the provision of an almost impermeable barrier against oxygen. One of the drawbacks of this technology is the rather large particles formed by extrusion (typically 500-1,000mm), which limit the use in applications where mouth-feel is a crucial factor. Additionally, a very limited range of wall materials is available for extrusion encapsulation (GOUIN, 2004).

MIRZAEI et al. (2012) microencapsulated ***L. acidophilus*** in a calcium alginate gel and resistant starch by extrusion, resulting in an increased survival rate of ***L. acidophilus*** in Iranian white-brined cheese after 6 months of storage. YULIANI et al. (2006) showed that the microencapsulation of limonene with β-cyclodextrin by extrusion offered an effective means against oxidation.

Coacervation

Coacervation is the technique that involves the deposition of the polymer around the core by altering the physicochemical characteristics of the medium, such as the temperature, ionic strength, pH and polarity (AZEREDO, 2005). It is called simple coacervation when only a single macromolecule is present, whereas when there are two or more molecules of opposite charges is referred to as complex coacervation (FREITAS et al., 2005).

Coacervation is a relatively simple, low-cost process that does not require high temperatures or organic solvents. It is typically used to encapsulate flavor oils (OLIVEIRA et al., 2002). One of the main disadvantages of the coacervation is that occurs only within limited ranges of pH, colloid concentrations and/or electrolyte concentrations (COMUNIAN et al., 2013).

JUN-XIA et al. (2011) microencapsulated sweet orange oil by coacervation with soybean protein isolate, indicating good protection for the core. OLIVEIRA et al. (2007) microencapsulated ***B. lactis*** and ***L. acidophilus*** by coacervation with pectin and casein, demonstrating more resistance of the product to gastric and intestinal juices. ROCHA-SELMI et al. (2013) encapsulated aspartame by coacervation, improving the protection even at 80ºC.

Lyophilization

Lyophilization is a method involving the dehydration of frozen material under a vacuum sublimation process, that is, compound water removal occurs without submitting the sample to high temperatures (CHEN & WANG, 2007).

This method provides excellent quality products because it minimizes the changes associated with high temperature, it is widely used in essences or flavorings. However, its high cost and long process time undermine its commercial applicability (MARQUES et al., 2006). CALVO et al. (2012) microencapsulated extra-virgin olive oil in the presence of maltodextrin, carboxymethylcellulose and lecithin by lyophilization, demonstrating that the oil was unaltered for 9 to 11 months, which increased the shelf life. EZHILARASI et al. (2013) encapsulated garcinia fruit extract in whey protein isolate and maltodextrin by lyophilization and aplicated in bread that exhibited higher volume, softer crumb texture, desirable colour and sensory attributes.

Emulsification

According ZANETTI (2001), in the microencapsulation by emulsification, first the core is dispersed in an organic solvent where the wall material is. Then, dispersion is emulsified in the water or oil, which contains an emulsion stabilizer. The organic solvent is then removed by evaporation under stirring, providing the formation of compact polymer globules in which the core is encapsulated.

This technique has been frequently used because of the simplicity of the procedures involved in producing the particle and the choice of the components of the formulation and preparation conditions. Emulsification has been used to encapsulate mainly enzymes, minerals, vitamins and microorganisms (AZEREDO, 2005). With the use of encapsulated enzymes by emulsification in cheese production, there was an increased rate of proteolysis compared with free enzyme production (KAILASAPATHY & LAM, 2005). SONG et al. (2013) microencapsulated probiotics by emulsification in alginate-chitosan, demonstrating more resistance in simulated gastrointestinal conditions. Some encapsulation methods and their size ranges of the microcapsules are shown in table 2.

## CONCLUSION

Microencapsulation has been applied in a wide variety of products from different areas, and studies have shown an enormous potential to provide the core with advantageous features, resulting in superior quality products, including in the food industry. However, much effort through research and development is still needed to identify and develop new wall materials and to improve and optimize the existing methods of encapsulation for the better use of microencapsulation and its potential applications.

Table 2 - Encapsulation methods and sizes of capsules.

| Encapsulation Methods | Core | Size (μm) |
|---|---|---|
| Physical Methods | | |
| Spray drying | Liquid/solid | 5 – 150 |
| Spray cooling | Liquid/solid | 20 – 200 |
| Fluidized bed | Solid | >100 |
| Co-crystallization | Liquid/solid | - |
| Lyophilization | Liquid | - |
| Physicochemical Methods | | |
| Simple coacervation | Liquid/solid | 20 – 500 |
| Complex coacervation | Liquid/solid | 1 – 500 |
| Solvent evaporation | Liquid/solid | 1 – 5,000 |
| Liposomes | Liquid/solid | 0.02 – 3 |

Source: adapted from FAVARO-TRINDADE et al. (2008).

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