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MICROENCAPSULATION IN FOOD PRODUCTS

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Abstract

In the food industry, encapsulation process greatly contributed to the development of functional foods. Functional foods are defined as being the foods that in addition to nutrients, supply the organism with components that contribute to cure the diseases, or to reduce the risk of developing them. Thus, functional foods can contain bioactive components such as: vitamins, peptides, minerals, fatty acids, poly-unsaturated fatty acids, phytosterols, lycopene, antioxidants, enzymes and living cells such as probiotics. The extreme sensitivity of many of these desired compounds leads to their deterioration, at conditions prevailing during food processing and storage, and thus significantly compromises our capability to incorporate them into foods. In this context, encapsulation of food ingredients are made: to protect the bioactive components against the some physical-chemical agents (temperature, pH, moisture, enzymes, oxygen, redox potential, UV light) during the storage; to prevent the reaction of bioactive componentsonents with other components in food products; for masking the bad tasting or smelling; to prevent the evaporation and degradation of volatile active components; to promote the conversion of liquid active compounds into a powder; to assure the controlled release of biocompounds etc. This paper aims to provide a short overview of commonly used processes to encapsulate food active components.

Keywords*: microencapsualation, food ingredients, wall materials, bioactive components.*

INTRODUCTION

Encapsulation is defined as a physic-chemical process in which solids, liquids and gaseous substances are surrounded by a coating, or embedded in homogeneous or heterogeneous particles, to give small capsules with diameters ranging from a few nm to a few mm. In scientific literature, the term of encapsulation can be easily confused with the immobilization one or encapsulation is sometimes considered as being a technique of immobilization (Champagne and Fustier, 2007).

Some researchers distinguish between the two processes (de Vos et al., 2010). The most important difference is the relationship between the core material and wall material. By encapsulation the active components are embedded and entirely covered by the encapsulating material, while considering the immobilization, the active components are adsorbed to the material surface, so that there is always the risk that they drain the system (de Vos et al., 2010).

In terms of morphology, microcapsules can be of two types (Figure 1):

- microcapsules type reservoir (or capsules) in which the active substance is included in a homogeneous space;
- microcapsules type matrix, called microspheres (micro beads). In microspheres, the active substance is dispersed in polymeric network spaces.

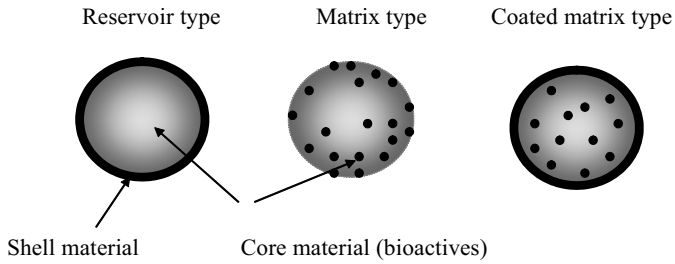

Figure 1. Schematic representation of microcapsules morfology (Dima et al., 2014a)

MATERIALS USED FOR ENCAPSULATION

Considering the selection process of shell materials used for encapsulation of food ingredients, some general properties should be considered (Zuidam et al., 2010; Brownlie et al., 2007):

- to be food grade (certified as "generally recognized as safe" (GRAS) materials)
- to be accepted by the governmental agencies such as the European Food Safety Authority (EFSA) or Food and Drug Administration (FDA) in the USA
- to provide maximal protection of the active material against environmental conditions (oxygen, water vapour)
- to hold actives within capsules structure during processing or storage under various conditions
- do not react with core material
- to have good rheological characteristics at high concentration ($\eta < 500$ cps la $c \geq 45\%$)
- to have good emulsification activity
- to have a good sensory quality
- to require low cost production and to be available in large quantities.

The scientific literature has addressed the various criteria for classifying the coating materials (Augustin and Sanguansri, 2007; Zuidam et al., 2010).

At encapsulation can be used a single material or a mixture of materials (de Vos et al., 2010; Thies, 2004).

Table 1 presents the lists with groups of biomolecules frequently called as materials for encapsulation in food products.

Table 1. Materials used to encapsulate bioactive food components
(adapted from de Vos et al., 2010)

Chemical classes (IUPAC)	Chemical compounds
Carbohydrates	Starch and derivatives (maltodextrins, syrups, dextrins, cyclodextrins) Cellulose and derivatives (ethyl-, methyl-, hydroxipropil-, carboxymethyl- ... cellulose) Plant exudates and extracts (Arabic, gum karaya, Gum tragacanth, Mesquite gum, pectins, guar, locust bean, tara) Marine extracts (Alginate -brown seaweed, Carrageenan -red seaweed) Microbial and animal polysaccharides (xanthan, gellan, dextran, chitosan)
Proteins	Vegetable proteins (gluten, soy protein, zein, rice protein) Animal proteins (gelatine, milk proteins, whey proteins)
Lipids	Glycerides, fatty acids and fatty alcohols, phospholipids, waxes (beeswax, carnauba wax, candelilla wax)
Other materials	Polyvinilpyrrolidone, polylactic acids and derivatives, shellac, paraffin, inorganic materials

METHODS AND TECHNOLOGIES

Encapsulation research has an interdisciplinary character based on knowledge from different fields: colloids and surfaces, physical chemistry, science of polymers, foods science, biotechnology, and engineering sciences. The success of microcapsules manufacture having specific properties for predetermined application areas largely depends on choosing the method of preparation and the coating material. As a result of creative imagination, today there are thousands of ways to make encapsulation. Most of them are applied only in the laboratory. The number of processes with industrial application is more limited, but large enough to satisfy the huge market request of microcapsules. In order to protect from the action of physicochemical and technological agents, the food components are encapsulated through different techniques such as: complex coacervation, ionotropic external/internal gelation, molecular inclusion, extrusion, freeze drying, spray drying, spray chilling/cooling etc. (Table 2) (Zuidam et al., 2010; Dima et al., 2014b). Many of autors have reviewed the preparation and application of microcapsules for food products (Champagne and Fustier 2007; Augustin et al., 2009; Brownlie, 2007;

Zuidam and Shimoni 2009; Dima, 2009; Burgain et al., 2011).

Table 2 shows the methods of classification and techniques of microencapsulation depending on the processes used to realize the entrapment of bioactive components:

Table 2. Usual technologies used for microencapsulation of food ingredients (adapted from Zuidam and Shimoni 2009; Burgain et al., 2011)

Methods	Technology	Types of microcapsules	Particle size (μm)	Bioactive components encapsulated
Chemical and physic-chemical processes	Complex coacervation	Reservoir	10-800	Flavors, essential oils, drugs, antioxidants;
	Emulsification W/O emulsions O/W emulsions W/O/W emulsions O/A/O emulsions Multilayers microcapsules (L/B/L)	Reservoir and matrix	0.2-5,000	Salts, bitter hydrophilic molecules (proteins); dietary fat, antioxidants (lycopene, β-carotene), vitamins (E), sterols, flavors; enzymes, flavors, cells;
	Ionotropic external gelation (extrusion, droping)	Matrix	200-5,000	Probiotics, minerals;
	Emulsification/ionotropic internal gelation	Matrix	10-1,000	Probiotics, enzymes, salts, vitamins;
	Emulsification/enzymatic gelation (rennet-gelation)	Matrix	10-50	Probiotics, enzymes;
	Emulsification/intefacial polymerisation	Reservoir	0.5-20	Lipids, pesticides, cells;
	Co-extrusion	Reservoir	150-8,000	Lipids, essential oils, proteins, confectionery products;
	Liposomes	Reservoir	0.2-1,000	Fatty acids, enzymes, proteins, flavors, vitamins, minerals, bacteriocins;
	Encapsulation using Supercritical Fluids: Rapid Expansion of Supercritical Solutions (RESS)	Matrix	5-400	Essential oils, lipids, vitamins;
	Inclusion (cyclodextrins)	Molecular inclusion	0.001-0.01	Flavors, esential oils, vitamins;
Mechanical processes	Spray drying	Matrix	10-400	Probiotics, flavors, antioxidants, lipids,
	Spray chilling/cooling	Matrix	20-200	Probiotics, enzymes, flavors, antioxidants, vitamins;
	Freeze drying	Matrix	20-5,000	Probiotics, lipids, vitamins, flavors, cells;
	Fluid bed coating	Reservoir	30-5,000	Probiotics, solid particles: minerals, salts, nutraceuticals;
	Extrusion	Matrix	50-2,000	Probiotics, flavors, proteins.

Emulsification/Extrusion external gelification method is the simplest method used to produce the microspheres loaded with food ingredients. The principle of this technique is the external gelation of hydrocolloids using different gelation agents (calcium chloride solution for alginate, potassium chloride for carrageenan and tripolyphosphate for chitosan, transglutaminase for caseinate). A suspension of bioactive compond and hydrocolloid solution is extruded through a needle to produce droplets that are collected in a bath where gelation occurs (ionotropic or thermal). The extrusion technique produces the spherical polymer beads, ranging from 2 to 3 mm diameter. Using additional drag forces (coaxial flow, electrostatic field) the small polymer beads are obtained (down to 100 μm). The extrusion technologies represent an adequate method for encapsulation of living cells, because they do not involve deleterious solvents and they can be accomplished under both aerobic and anaerobic conditions (Dima et al., 2014a).

The most common hydrocolloid used in order to produce the probiotic microcapsules is represented by alginate. Alginate is a linear polymer of two uronic acids: β-D-manuronic acid and α-L-guluronic, obtained by extraction from brown algae. During gelation, the calcium ions occupy the space between two alginate polymer chains, and the strong

interchain binding results in a conformation called the „egg-box" model. (Dima et al., 2014a, 2014b).

The size of alginate beads depends on different parameters, such as: the alginate structure, the alginate and calcium concentration, the gelation time, the needle diameter and finally the distance between the outlet and the coagulation solution.

Emulsification and internal ionic gelation represents a chemical technique used to encapsulate the food components in microspheres with less than 100 µm.

The principle of this method is based on emulsification of hydrocolloid (alginate), calcium carbonate and probiotic cells aqueous suspension into mineral or vegetable oil, in the presence of lipophilic surfactant (Span 80). It will be obtain a water-in-oil emulsion in which the internal phase contains droplets of both alginate and living cells (Figure 2). In order to achieve the internal gelation, the acetic acid (100 µL/100 mL, diluted in a small amount of oil) is added into the formed emulsion. The water-in-oil emulsion is destroyed by a $CaCl_2$ solution (0.05M) and probiotic alginate beads remain inside the aqueous phase. The alginate beads are then separated from oil and washed (Dima et al., 2014b).

During emulsification/internal gelation process, a large number of parameters may influence the characteristics of the microspheres, such as: internal phase ratio, emulsifier concentration, calcium/alginate molar ratio and alginate concentration.

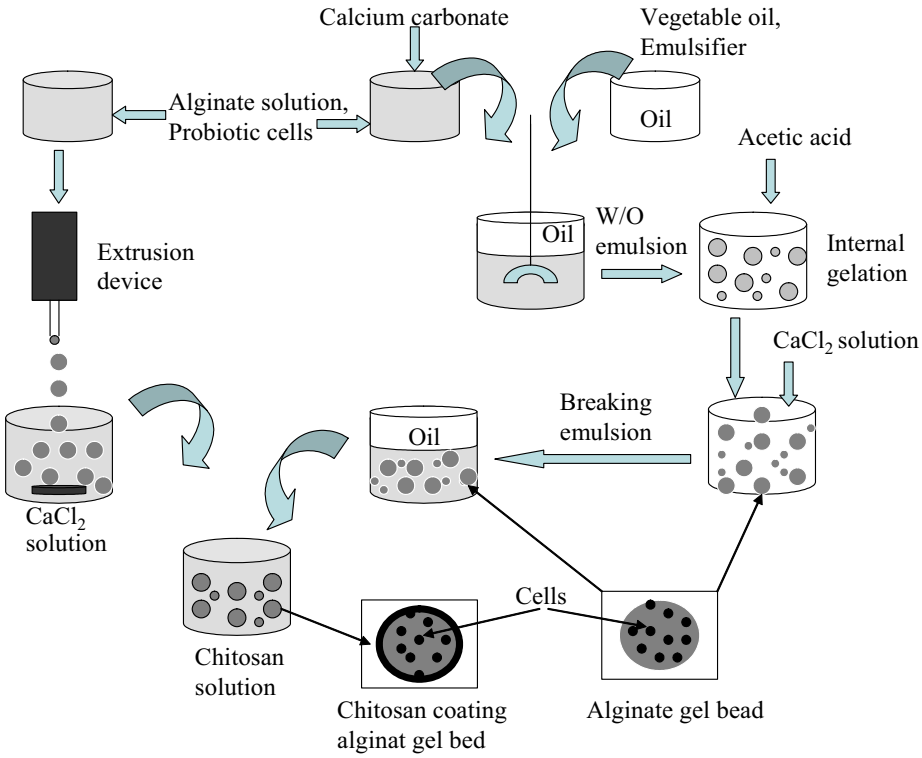

Figure 2. Schematic presentation of the microencapsulation of probiotic cells by extrusion and emulsification/internal gelation

Spray-drying method represents one of the oldest and the most widely used encapsulation technique used in the food industrial area (Augustin et al., 2009; Champagne et al., 2007). In the beginning, this process was applied to flavours in order to protect them from degradation/oxidation and also to dry solid suspensions, but it is presently also applied to bioactive molecules and probiotics (Bourgain et al., 2011).The method presents the following advantages: it can be applied on industrial scale, it is a fast high-yield, it is applied to various biocomponents encapsulation, microcapsules are readily dispersible in water.

The disadvantages of this method are: the complexity of the equipment, the non-uniform conditions in the drying chamber, the method is

limited to shell materials soluble or dispersible in water, spray-dried capsules carry a lower loading (20-30%).

Microencapsulation of food ingredients by spray-drying method is done with great care, following carefully: drying temperature, drying time, type of atomization, shell materials, storage conditions (Figure 3).

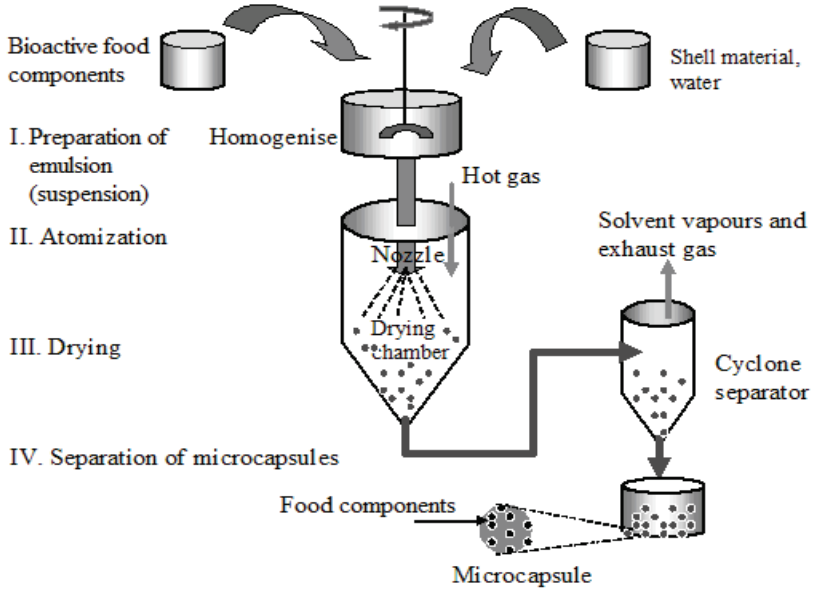

Figure 3. Schematic presentation of the spray-drying encapsulation technique (Dima et al., 2014a)

Spray freeze drying is a similar method to spray drying. The difference is represented by how the droplets produced by atomization are strengthening. In freeze drying method the droplets with probiotic cells in shell material are frozen into the vapours of a cryogenic liquid such as liquid nitrogen. Frozen droplets are then dried in a freeze dryer (Dima, 2009; Zuidan, 2010; Burgain et al., 2011).

The disadvantage of this method is the high energy consumption which implies a much higher price cost than spray drying method.

Fluid bed coating or spray coating method represents an encapsulation technology that utilises a spray process to deliver film material to a core particle, and fluidizing air to circulate materials. Fluid-bed technology is restricted to solid core materials ranging from 30 μm to several centimetres in diameter (Brownlie, 2007).

Solid microcapsules obtained by spray-drying or freeze-drying are moved by the fluidizing air and a liquid coating material is sprayed through a nozzle over the core material in a hot environment.

The complex coacervation is a phase separation process that manifests at the simultaneous dissolution of two polyelectrolytes with opposite charges when the conditions of reaction are changed. Proteins and polysaccharides are the most commonly used compounds for coacervation.

The encapsulation through complex coacervation of flavour compounds has a series of advantages such as: it uses a simple technology, it has an encapsulation efficiency of over 90% and it does not use organic solvents. The microcapsules obtained through complex coacervation are stable at high temperature and enable to controlled release of components (Dima et al., 2014)

Inclusion in cyclodextrins of biomolecules is an elegant encapsulation method. Cyclodextrins (CDs) are cyclic oligomers of α-D-glucopyranose produced by starch degradation under the action of cyclodextrin glucosyl transferase produced by *Bacillus macerans*. The cyclodextrins are formed by the link of six, seven and eight units of glucopyranose, also known as α-CDs, β-CDs and γ-CDs (Figure 4). CDs have o hydrophobic interior and hydrophilic exterior and can include a great variety of apolar molecules in hydrophobic cavity. In this case, a complex called ”*inclusion complex*” is formed. In the food, CDs have been widely used for

stabilisation of flavours, vitamins and essential oils, for elimination of undesired tastes, for the protection of lipophilic food components, to solubilise food colouring and vitamins etc. (Dima, 2009).

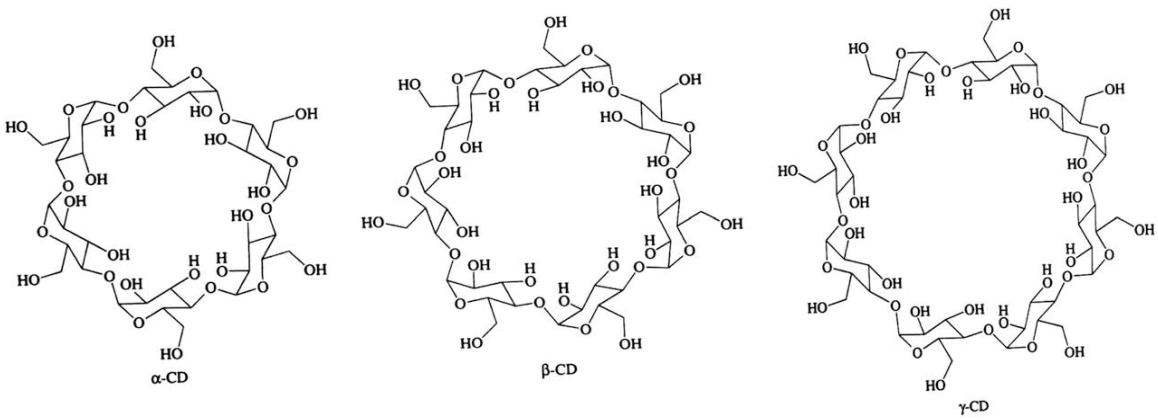

Figure 4. Chemical structures of α-CDs, β-CD and γ-CD

CONCLUSIONS

In the last years more and more consumers prefer foods that provide various benefits for their health status. The use of bioactive food components encapsulated offers processors the opportunity to improve the nutritional and health qualities of their food products.
Microencapsulation allows the protection of a wide range of food components, from small molecules (salts, oils, vitamins, flavours, colorants) and protein (enzymes, hormones) to probiotic bacteria.

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