

SHORT OVERVIEW OF ENCAPSULATION TECHNOLOGIES FOR DELIVERY OF BIOACTIVES TO FOOD

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Abstract

Encapsulation strategy has emerged as a mean to protect sensitive bioactive compounds, to improve their stability and to deliver their active forms to the targeted place. A number of bioactives can be encapsulated – cells, enzymes, vitamins, minerals, aromas and flavours, food colourants, antioxidants, etc. Over the years various encapsulation technologies were employed in encapsulation of bioactive compounds intended to the application in food industry. This paper gives a short overview of commonly used technologies for the encapsulation of food bioactives.

Key words: *encapsulation, bioactives, food applications*

1. INTRODUCTION

The awareness of modern consumers related to the nutritional profile of food products and requirements laid in front of food producers regarding development of such products, have enormous impact on since community who is constantly searching for the new ways to enrich the traditional foods. Human diet has changed tremendously; food is not consummated only to satisfy the basic nutritional needs, but it is expected to enhance the human health, to prevent development of various diseases, and to help in isolated from natural sources can be employed, such as plant polyphenols, probiotic bacteria, omega-3-fatty acids, vitamins, minerals [1]. These compounds usually face the problem of low stability against the light, moisture, changes of pH or temperature; all this negatively affects the biological activity of bioactive compounds, and in long term lowers their bioavailability and reduce the shelf-life of food products [2].

To overcome these limitations, encapsulation of bioactive compounds and application of encapsulated bioactives instead of free forms can

treatment of developed ones. All of that led to the expansion of research in the domain of functional food products, with improved nutritional value and biological activity.

In order to develop new food products, it is necessary to find a way to bring the functionality to the selected foods. The common way to achieve this is to enrich the foods with the natural compounds with a high biological activities and proven beneficial impact on human health. In that respect, various actives

be utilized. Encapsulation technology implies the entrapment of bioactive substances within the other substance – e.g. polymeric carrier material, where the particles of different diameters are produced. Many different encapsulation technologies exist today; this paper will provide short overview of most commonly used encapsulation technologies in the food industry.

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2. SPRAYING TECHNOLOGIES

Most of the active compounds are in the liquid form, therefore the majority of encapsulation technologies implies some sort of drying. In that respect, various technologies can be applied, such as spray-drying, spray-chilling, spray-cooling, melt injection, fluidized bed coating [3, 4, 5].

Spray-drying is the encapsulation technique most widely used for the application in food industry. The polymer/active compound mixture is sprayed in the hot chamber, at the high velocities of emulsion feed and under the high inlet air temperatures. It is cost effective process that allows continuous production of beads [5]. The production rates achieved with this technique are high, so it is highly applicable on industrial level; the utilization of multi nozzle spray-drying equipment can help with increment of production rates even to higher extent [1]. Spray-drying is usually used for the encapsulation of hydrophobic compounds, but lately some hydrophilic compounds were successfully encapsulated as well [6, 7, 8]. The produced spray-dried powder-beads are spherical in shape, with a mean diameter up to 300 µm, and good stability [1]. However, the main drawbacks of the spray-drying process are reflected in following: (1) it is difficult to control particle size and to produce the beads of desired diameter, and (2) the heat-sensitive compounds can be destroyed during the spray-drying, if the inlet air temperature is too high. Despite this, most of encapsulates used in the food industry (around 80-90%) are the ones prepared by spray-drying [9, 10].

Spray-cooling/Spray-chilling techniques rely on principle quite opposite to the one behind spray-drying process – the mixture of polymer/bioactives solution is cooled down to the certain temperature. The differences between the cooling and chilling processes are in the carrier material used, most precisely in its melting point. Thus, as a carrier material for spray-cooling vegetable oils with melting point of 45-122 °C are used, while spray-chilling process employs vegetable oils with melting point of 32-42 °C [1]. Encapsulates produced with this techniques are up to 200 µm in size, with a very high encapsulation efficiency [1]. Spray-

cooling/chilling techniques are regarded as environmentally friendly techniques since they do not require any harmful organic solvents, or high process temperatures (which also lowers the operational costs), as opposite to for example spray-drying technique [11].

Fluidized bed coating is the technique used to apply an additional coating over the previously formed powder particles; this can be performed in continuous or batch conditions [1]. It is a potent technique, where the obtained particles have improved properties, particularly related to the release of actives [12]. The advantage of this technique is controllable particle size [1]; however, the problem that might occur is the agglomeration of particles [13].

3. EXTRUSION TECHNOLOGIES

Extrusion technologies involve dripping of a polymer/actives mixture into the gelling solution, through a dripping tool, where the collected beads were left to harden for a certain period of time. Depending on the type of dripping tool, various extrusion technologies can be distinguished: (1) electrostatic extrusion, (2) coaxial air flow extrusion, (3) vibrating jet/nozzle extrusion, (4) spinning disk atomization and (5) jet-cutting [14]. The particles produced via extrusion technologies are usually in the size range of 0.2 to 5 mm; although particle diameter is mainly influenced by dripping tool, the polymer used as a carrier material displays some influence too, particularly its viscosity and rheological behaviour [15, 16].

In general, extrusion technologies are performed under mild conditions (pressure and temperature are generally lower than 700 kPa and 118 °C) [18], they do not utilize any harmful solvents and it is possible to use these technologies for encapsulation of bioactives in both anaerobic and aerobic conditions; all listed make them applicable for encapsulation of sensitive bioactives, such as heat-sensitive products, as well as living cells. In addition, the microbeads produced by extrusion technologies have less porous structure when compared to those produced for example by means of spray-drying process [19]. However, the major drawbacks of extrusion

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technologies are reflected in difficulties to achieve high production capacity on industrial level [1]. Furthermore, in some cases, size of produced beads may be too bulky for application to certain food products, as it can be perceived in the mouth [20].

Electrostatic extrusion technique employs electrostatic potential in the production of microbeads. Namely, applied electrostatic potential leads to the disruption of the liquid surface at the top of the needle, which in turn results in formation of a stream of charged droplets [15, 17]. The main advantage of electrostatic extrusion is possibility to produce particles small in diameter and with very uniform size distribution [17].

Coaxial air flow technique includes development of droplets under the impact of compressed air. In this way abruption of drops from the nozzle tip is faster when compared to the simple dripping when detachment of droplets is governed only by gravitational force [21]. The diameter of produced particles is up to 200 µm. The main precedence of this technique is high reproducibility, which is important from the operational point of view [22].

Vibrating jet/nozzle extrusion, spinning disk atomization and jet-cutting techniques are the extrusion techniques that can gain higher production rates. When compared to electrostatic extrusion and coaxial air flow technique, the techniques relying on vibrational, as well as jet-cutting technology can increase the productivity up to 50 times, hence they are more relevant when production on industrial level is concerned [21].

4. COMPLEX COACERVATION

Coacervation is a promising encapsulation technology, especially from the industrial point of view, since it can result in the high payloads (up to 99%) [22]. Coacervation process relies on the phase separation of hydrocolloids (one or more) from the initial solution, followed by the deposition of developed coacervate phase around the suspended or emulsified active compound(s) [22]. If necessary, hydrocolloid capsule can be additionally crosslinked using enzymatic or chemical cross-linker. Commonly, the coacervation process is conducted between proteins and polysaccharides, but lately protein-protein coacervates are also in development.

The coacervation as encapsulation technique yields in excellent encapsulation efficiency (up to 100%) [1]. Furthermore, produced microcapsules have very good controlled/sustained release properties, they are resistant to heat and mechanical stress [23, 24, 25, 26]. Nevertheless, limitations of coacervation are mostly related to the type of core material that can be encapsulated. Namely, coacervation is mainly used for the encapsulation of hydrophobic actives, while the hydrophilic compounds are difficult to encapsulate. To achieve the encapsulation of hydrophilic compounds, it is necessary to introduce some changes to the encapsulation procedure; usually, it implies formation of the double emulsions at the beginning of coacervation process [27, 28]. At the end, even though the microencapsulates obtained by complex coacervation exhibit numerous favourable properties, this technology still isn't fully accepted and applied in the food industry, mostly due to the its complexity, as well as high process costs.

5. EMULSION-BASED TECHNOLOGIES

Emulsification processes for microgels production. Hydrogel biopolymer particles can be produced via emulsification method. Classic emulsion processes involves formation of oil-in-water emulsions, where slightly hydrophobic polymer is emulsified in water, using oil-in-water emulsifier; gelling or cross-linking agent can be also added in order to promote gelation. The particles obtained via this technology are in the size range 10-1000 nm, and with a good sphericity [30, 31]. An alternative way to produce biopolymer particles is the novel inverse emulsion technique [32, 33, 34]. This technique is called inverse since it implies production of water-in-oil emulsions, by dispersing the water solution of hydrophilic polymer in hydrophobic oil phase, with the assistance of water-in-oil type of emulsifier. These techniques are easier to scale-up to industrial level than for example extrusion processes, but disadvantage of emulsification processes is reflected in difficulties to control the process and to obtain particles of desired size [1]. In addition, the produced microgel particles have to be separated from the liquid, which may increase the costs of process.

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Emulsions are colloidal delivery systems, which are fundamentally consisted of an oil, surfactant, and water. They are already widely used in the food industry; some of the food products that are basically emulsions are mayonnaise, milk, ice-creams (oil-in-water emulsions), as well as butter and margarine (water-in-oil emulsions) [1]. More complex emulsion-type delivery systems are double (or multiple) emulsions. In these systems small droplets of one phase (water or oil) are surrounded with bigger droplets of other phase (oil or water); these delivery systems are also applied in the food products, such as dressings, aromatic mayonnaise and ice-creams [35].

Emulsions can be produced via different production procedures. Most easy way is spontaneous emulsification, where upon mixing of oil, water, surfactant and if necessary co-surfactant, emulsions are produced. In addition to this, it is possible to apply mechanical mixing (blending, microfluidization, or sonication) or temperature changes, in order to obtain the desirable size of emulsion droplets [36]. These, so called *low energy methods* are favourable when encapsulation of thermo-sensitive compounds is concerned. Nevertheless, the systems used in the food industry are usually too complex for the low energy methods. In that respect, *high energy methods* for the production of food emulsions are more applicable [37]. Most commonly used method is high pressure homogenization, where droplets in the size range between 0.3 and 1 μm , with a narrow size distribution can be produced [37]. Double emulsions are usually produced through a two-step method, where two surfactants, hydrophobic and hydrophilic, are used.

In recent years, alternative delivery systems based on the emulsions are developed. Namely, double or multilayer emulsions are coated with the layer(s) of (bio)polymers, where the interactions are usually formed by the electrostatic forces [38]. As biopolymers for coatings various materials can be used; some of them are proteins (with amphiphilic properties, such as milk proteins) and polysaccharides (with the opposite charge to that of emulsions) [39]. Such systems provide improved properties of emulsions, primarily those related to the emulsion stability.

6. LIPOSOMAL TECHNOLOGY

Although the liposomal technology has been mostly employed in delivery of drugs, recently it arises as an interesting strategy for delivery of bioactives into foods. Liposomes are lipid vehicles formed on the basis of hydrophilic–hydrophobic interactions between polar lipid compounds (usually phospholipids) and water molecules. Namely, these interactions occurs between hydrophilic heads and lipophilic hydrocarbon tails of polar lipids, resulting in the formation of lipid bilayers; in addition to that, interactions between formed bilayers and water molecules govern the arrangement of lipids into spherical vesicles, i.e. liposomes [40]. Due to their structure, liposomes are able to encapsulate both water-soluble and lipid-soluble bioactives, as well as amphiphilic compounds, which make them suitable for the encapsulation of wide array of bioactive compounds.

There are a number of classical methods for liposomes production, such are thin-film hydration method, reversed-phase evaporation [41], solvent injection method [42, 43] and heating-based methods [44]. However, these methods have some drawbacks, usually related to the large liposomal particles produced, so it is necessary to apply additional processes to reduce the size of liposomes. Also, classic methods frequently involve utilization of organic solvents, which should be omitted in food applications. In that respect, development of new methods for the production of liposomes is necessary, in order to overcome listed difficulties of standard method. Some of the newly developed liposome production methods employ different membranes in the production procedure – those are so called membrane contactor-based methods [45, 46, 47]. Also, methods based on the freeze drying can be used for the production of liposomes; namely, freeze drying of double emulsions with addition of cryoprotectants proved to be promising method for the liposomes production [48]. Finally, proliposome method has been establishing as a suitable method for the production of stable liposomes, small in size and with a narrow size distribution and with a good encapsulation efficiency [49, 50]; what is more important, proliposome method is easy to scale up to

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industrial level of production, which is necessary for the applications in food industry.

7. MOLECULAR INCLUSION

Encapsulation of bioactives by molecular inclusion generally implies inclusion of bioactive compounds in the cyclodextrin molecules. Cyclodextrins (CDs) are a group of natural oligosaccharides containing six, seven or eight glucose residues, interlinked by $\alpha(1\rightarrow4)$ glycoside bonds in a structure shaped like cylinder; they are labelled as α -, β - and γ -cyclodextrins, respectively [51]. The depth of the cylinder-like cavity of natural CDs is ~ 0.8 nm, but the diameter can vary.

In general, all the methods used to achieve inclusion complexation rely on simple principle of co-precipitation [52, 53]. However, the procedure of encapsulation, as well as conditions necessary to achieve the complexation are different and unique for each and every guest molecule, i.e. bioactive compound to be encapsulated [54]. Usually, it is required to stir, sonicate or heat the mixture of CDs solution and bioactives in order for complexation to occur [55, 56]. If preparation of complexes implies co-evaporation, the treatment by heat in vacuum-oven or under the vacuum in a rotary evaporator can be used [57, 53].

Encapsulation in CDs has number of advantages, but probably the most important one is that it enhances the solubility of poorly water-soluble bioactives [56, 58, 59, 60], in that way making them more suitable for the applications in food industry. In addition to that, it was proved that β – CDs enable controlled release of encapsulated compounds [57, 61, 62].

8. CONCLUSION

Despite the fact there is a number of encapsulation technologies successfully applied on the lab-scale level, there are still some constraints to their application in the actual industry. When applying encapsulated bioactive compounds into the real food products, there are a lot of factors affecting the process itself, and subsequently the final food product: the nature of active compounds (hydrophilicity or hydrophobicity), the particle size of obtained encapsulates, compatibility with the food

matrix; in addition, it is necessary to take into account the influence of mixing process during the food product preparation on the stability of encapsulates, as well as the impact of added encapsulates on the rheological, textural and sensorial properties of final food product. On top of all, costs of implementation of new process and development of new product have to be justified.

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