MICROENCAPSULATION: A REVIEW OF APPLICATIONS IN THE FOOD AND PHARMACEUTICAL INDUSTRIES

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ABSTRACT

Microencapsulation is a technique that has been widely used in the food and pharmaceutical industries. This technique can be used to reduce the cost of production, to increase the stability of compounds, to mask undesirable tastes, and to improve the release properties of compounds in food industries. Currently, microcapsules are utilized in beverage, bakery, meat, poultry, and dairy products. Moreover, microencapsulation has been used to increase stability, to mask bitter taste, to improve the release properties of drugs, and to provide specific drug delivery in pharmaceutical industries. The challenge of microencapsulation is in selection of the appropriate conditions for producing highly effective microcapsules. Many factors affect the quality of microcapsules, including preparation techniques, types of core material, and types of wall material. We provide an overview of the current research on the applications of microencapsulation in food and pharmaceutical industries, the selection of suitable conditions for developing high efficiency microcapsules, and future trends in microencapsulation.

Keywords: application, core material, food industries, microencapsulation, pharmaceutical industries, wall material

1. Introduction
1.1 Definition of microencapsulation

The microencapsulation process is used to entrap small particles of liquids, solids, or gases in one or two polymers. As presented in Fig. 1, the particle component is referred as the "core material", and polymers are called variously such as "wall material", "shell", "coating", "carrier", or "encapsulant" (Luzzi, 1970; Desai and Jin Park, 2005). The purpose of microencapsulation is to protect the core material from environmental factors (such as light, moisture, temperature, and oxygen), to extend shelf-life (Shahidi and Han, 1993; Gouin, 2004), and to improve the release properties of compounds (Müller et al., 2002). Microencapsulation has been applied in the design of new materials not only for the food industry but also for pharmaceuticals, cosmetics, and textiles, where the stability, efficiency, and bioactivity of compounds are required (Koo et al., 2014; Dias et al., 2015).

1.2 Classification of microcapsules

The selection of the core, wall material, and microencapsulation technique affects the properties of microcapsules, including morphology. Based on the various properties of the core, wall material, and microencapsulation technique, different types of particles (Fig. 2) can be obtained (Gharallahou et al., 2007). The morphology of microcapsules can be described as: mononuclear (Fig. 2a), poly/multinuclear (Fig. 2b), matrix (Fig. 2c), multi-wall (Fig. 2d), and irregular (Fig. 2e).

1.3 Microencapsulation techniques

Microencapsulation techniques are divided into three classes: chemical, physical, and physico-chemical methods (Jyothi et al., 2010). Chemical methods include in situ polymerization and use of liposomes; physical methods include spray-drying and fluidized bed coating, and physico-chemical methods include coacervation and sol-gel encapsulation (Gibbs, 1999; Gouin, 2004). The microcapsules produced by each method are different (Fang & Bhandari, 2010).

1.3.1 Chemical methods

In situ polymerization

The in situ formation of a hydrogel has recently been recognized for its potential biomedical and biotechnological applications
including controlled-release drug delivery, three-dimensional (3D) cell culture, and injectable tissue engineering, since it is a simple method (Smeds and Grinstaff, 2001; Ko et al., 2013).

Hydrogels are 3D polymeric networks with high water content that remain insoluble in aqueous solutions because of chemical or physical crosslinking of individual polymer chains (Lin and Metters, 2006; Ko et al., 2013). To produce hydrogels, bioactive compounds or cells are premixed in an aqueous solution (sol) and injected to a target site, to form a hydrogel depot through a sol-to-gel transition mechanism. This sol-gel transition can be generated by changing the environmental conditions including pH, temperature, light, and ionic strength (Hou et al., 2008; Ko et al., 2013).

**Liposomes**

Liposomes are often used in the pharmaceutical and food industries for entrapping an aqueous solution within a membrane of phospholipids. Liposomes are microscopic vesicles composed of phospholipid bilayers surrounding aqueous compartments.
As presented in Fig. 3, the liposome encapsulates a hydrophilic drug within an aqueous medium, the hydrophilic region, while also entrapping the lipophilic drug within the lipid bilayer, the hydrophobic region (Barenholz, 2003; Nii and Ishii, 2005).

Liposomal entrapment can protect bioactive materials from environmental and chemical stresses, including the presence of enzymes or reactive chemicals, and exposure to extreme pH, temperature, and high ion concentrations (Foged et al., 2007).

1.3.2 Physical methods

Spray-drying

Spray-drying is a widely used technique in the food and pharmaceutical industries (Giunchedi and Conte, 1995; Gibbs, 1999; Jain, 2000; Bruschi et al., 2003; Reineccius, 2004), because it presents several advantages including those of low cost, easy adaptation to the industrial scale, and high drug-loading efficiency (Baranauskienė et al., 2006). In the spray-drying process, core material is mixed or homogenized in a solution of wall material, to form a stable emulsion. This emulsion is fed into a spray dryer and formed into a dried particle (Loksuwan, 2007) by hot air (Turchiuli et al., 2005).

Wall materials can be selected from a variety of polymers, depending on the core material and the desired characteristics of the final product. For the spray-drying process, the wall material must be soluble in water at an acceptable level (Gouin, 2004) and possess good properties of emulsification, film forming, and drying (Reineccius, 2004).

Fluidized bed coating

Fluid bed coating is commonly used for coating solid particles (Ronsse et al., 2007). The mechanism of formation of microcapsules using this technique can be divided into three stages: nucleation, transition, and ball growth. In the beginning of the microencapsulation process, particles are suspended in the coating chamber. Droplets of the polymer solution are then sprayed, increasing the probability of particle-droplet impact and spread on the particle surface. At the end of this process, droplets are evaporated and formed into microcapsules (Teunou and Poncelet, 2002).

This manufacturing technique is ideal for the food industry, because of its high versatility, relatively high batch size, and simplicity (Deppere et al., 2009).

1.3.3 Physico-chemical methods

Coacervation

Coacervation can be divided into “simple” and “complex” coacervation (Wang et al., 1999). This method involves the phase separation of one or many hydrocolloids from a polymeric solution layer around the core material that is suspended in the same reaction media (Gouin, 2004; Desai and Jin Park, 2005).

In simple coacervation, only one coating material (typically pectin) is used (Luzzi, 1970). The simple coacervation method is dependent on conditions such as pH, ionic strength, temperature, and structure of the macromolecules. For example, when the pH is adjusted to a value near the isoelectric point (pI) of gelatin at low ionic strength, the net charge of gelatin becomes balanced. The molecules unfold and sediment to form microcapsules (Schmitt et al., 1998). Complex coacervation is mostly dependent on pH and the concentration of polymer. This process involves the reaction between two oppositely charged polymers (protein and polysaccharide) and is called the “polymer-polymer interaction method”. Negatively charged polysaccharides (such as acacia, pectin, alginate, and carboxy methyl cellulose) interact with positively charged proteins (such as gelatin, soy protein isolate;
SPI, and whey protein isolate; WPI) via electrostatic interactions (Saravanan and Rao, 2010). The mixtures of protein and polysaccharide form an electrostatic complex in a specific pH range, relying on the pI of protein and pKa of polysaccharide. At a pH below the pI of the protein and above the pKa of the polysaccharide carboxyl group, they can effectively interact via electrostatic interactions (Giancone et al., 2009).

**Sol-gel encapsulation**

The sol-gel encapsulation technique involves adsorption on glass surfaces, entrapment in a polymer matrix, or incorporation in porous glass powders, because entrapment is based on the growing of siloxane polymer chains around the biomolecule within an inorganic oxide network (Flora and Brennan, 2001; Gupta and Chaudhury, 2007).

The advantages of the sol-gel method are low cost, simplicity, safety, and an absence of vacuum requirement. However, this method also has disadvantages; entrapment in a sol-gel glass may change the chemical and biological properties of the entrapped compounds because of reduced degrees of freedom and interactions with the inner surface of the pores (Zink et al., 1994; Lin and Brown, 1997).

2. Application Fields of Microencapsulation

Microencapsulation technology is widely used in several industries, especially food and pharmaceutical industries, since it can increase solubility, enhance stability, and improve the controlled release properties of compounds such as essential oils, antioxidants, enzymes, drugs, etc. Therefore, this section focuses on the applications of microencapsulation in these industries.

2.1 Applications in the food industry

The food industry utilizes functional ingredients to improve flavor, color, and texture properties and to extend the shelf-life of products. Moreover, ingredients that have functional health benefits, such as antioxidants and probiotics, are of great interest (Borgogna et al., 2010). However, most of these ingredients have low-stability and are easily decomposed by environmental factors. Thus, the preparation of high-stability bioactive compounds is important. Microencapsulation is one way to address these issues. In recent years, there has been a great deal of research on the production of high efficiency microcapsules and their applications in the food industry.

2.1.1 Beverages

Burin et al. (2011) evaluated the stability of anthocyanin, which was encapsulated within different carrier agents in an isotonic soft drink system. Anthocyanins are water-soluble pigments obtained from plants. These pigments are generally used as colorants in foods and drinks, because they have high colorant power, low toxicity, and high water solubility (Ersus and Yurdagel, 2007). Moreover, many studies have shown that anthocyanins have important antioxidant and anticarcinogenic properties (Wang and Xu, 2007; de Rosso et al., 2008). Nevertheless, anthocyanins are unstable pigments and can be decomposed to colorless compounds by many factors including pH, temperature, light, oxygen, and the food matrix (Wang and Xu, 2007). Therefore, microencapsulation has been used to increase the stability of these compounds (Giusti and Wrolstad, 2003). In their study, the spray-drying technique was used to encapsulate anthocyanins originated from Cabernet Sauvignon grapes. They found that the obtained microcapsules presented uniform particle sizes and a spherical surface. Moreover, a combination of maltodextrin (MD) and gum Arabic (GA) resulted in increased protection of the anthocyanin pigments.

Aditya et al. (2015) prepared microcapsules of curcumin and catechin using water-in-oil-in-water emulsions (W/O/W). This study aimed to prevent the degradation of both curcumin and catechin in beverage systems. The biological activities of curcumin and catechin increase when they are used in combination (Xu et al., 2010; Manikandan et al., 2012). In the food industry, these two compounds are used to develop functional food and drink products, because they are effective bioactive compounds that can prevent several diseases such as cancer, obesity, infection, and cardiovascular ailments (Stammier and Volm, 1997; Nayak et al., 2010; Aditya et al., 2013). However, curcumin and catechin are unstable compounds. They are easily degraded in the presence of oxygen, alkaline pH, and high temperature (Green et al., 2007). In this study, it was found that the stability of encapsulated curcumin and catechin, either individually or in combination, increased, in a model beverage system.

Kausadkar et al. (2015) encapsulated lemon oil with maltodextrin by the spray-drying technique. Lemon oil has a sharp, fresh smell. Therefore, it is mainly used as a flavoring agent in food and beverages. However, the high amounts of unsaturated and oxygen-functionalized compounds in this oil affect oxidation during storage. Thus, the microencapsulation technique has been used to address this problem. The stability of samples was examined in terms of sensory characteristics in forms of instant ice tea premix at various storage temperatures (4, 28 and 45 °C). It was found that encapsulated lemon oil showed a good odor/taste profile and the appearance did not change over the storage time at all storage conditions. These results indicated that encapsulated lemon oil can be stored for 6 months.

2.1.2 Baked goods

Rocha et al. (2012) produced microcapsules of lycopene by spray-drying, using a modified starch as the encapsulating agent. The functionality of microcapsules was determined by applying them to cake. Lycopene is a carotenoid present in several fruits and vegetables. It is widely used as a red food colorant.
Nevertheless, lycopene is easily decomposed by oxidation during the storage process, because of its high number of conjugated double bonds. In this study, microencapsulation was expected to increase the stability of lycopene. The results showed that cake made with microcapsules was more pigmented than standard cake.

O’Brien et al. (2003) encapsulated vegetable shortening to increase oxidative stability and convert fat into a stable powder for use in short dough biscuit production. Currently, most ingredients used in commercial biscuit production are in dry form. However, the fat ingredient must be added in the form of liquid (oil) or block (fat), which requires an additional manual step. The objective of this research was to develop microcapsules of high-fat powders and evaluate their effect on biscuit quality compared to the quality of a control biscuit produced with hydrogenated vegetable fat. It was found that microencapsulated vegetable fat produced at a low homogenization pressure, with whey protein concentrate (WPC) containing 5% protein as the encapsulating agent, could be used for producing biscuits with acceptable characteristics. Therefore, microencapsulated high-fat powders could be used as a replacement for fat/oil in commercial biscuit production.

2.1.3 Meat and poultry

Muthukumarasamy and Holley (2006) were interested in the use of probiotics in dry fermented sausages, for increasing nutritional value. However, many studies have reported that probiotic organisms have poor survival in fermented foods (Shah et al., 1995; Kailasapathy and Rybka, 1997; Lücke, 2000; Shah and Ravula, 2000). To improve the viability of bacterial cells, microencapsulation technique was used to retain them within a protective polymer membrane or matrix (Audet et al., 1993). The results showed that microencapsulated Lactobacillus reuteri could be used in dry fermented foods, because it can prevent the loss of cell viability after drying without altering the sensory quality of products.

Jiménez-Martín et al. (2016) used microcapsules of omega-3 fatty acids from fish oil for enriching frozen chicken nuggets and investigated the effect of time of frozen storage on the oxidative stability and sensory properties of this product in comparison to that with bulk fish oil addition. It was found that time of frozen storage had no effect on the sensory quality of chicken nuggets enriched with omega-3 fatty acids. Microencapsulation of omega-3 fatty acids from fish oil could be used for the enrichment of pre-fried frozen meat products with fish oil, improving the oxidative shelf-life and preserving the sensory quality characteristics of the enriched products.

Comunian et al. (2014) evaluated the effect of encapsulated ascorbic acid on physicochemical and sensory stability of chicken frankfurters. Ascorbic acid is a natural antioxidant derived from fruits and vegetables. However, it is very unstable. It is easily decomposed by various factors including, heat, light, high oxygen concentration, and high water activity. Ascorbic acid is commonly used in frankfurters to replace sodium erythorbate. Hence, this study aimed to encapsulate ascorbic acid in frankfurters, because this technique allows for the incorporation of an effective antioxidant with vitamin functionality and improves stability of the product. The results showed that it was possible to produce frankfurters with acceptable sensory characteristics, when using ascorbic acid as an antioxidant.

2.1.4 Dairy products

Anjani et al. (2007) developed microcapsules of Flavourzyme with various wall materials, for use in cheese production. Low-moisture cheese varieties, such as cheddar, require longer ripening to develop the desired flavor, texture, taste, and aroma characteristics. Since traditional cheese maturation occurs at a very slow rate (McSweeney, 2004), exogenous enzymes are added to increase the maturation rate. However, direct addition of enzymes results in loss of enzyme, poor enzyme distribution, reduced yield, and poor cheese quality. Microcapsules added to milk during cheese manufacturing allowed for good distribution of Flavourzyme.

Champagne et al. (2015) evaluated the effects of microencapsulation by the spray-drying technique on the stability of probiotic bacteria in ice cream. Recently, several functional ice creams have been produced by adding probiotic bacteria. However, processing and storage conditions affect the viability of probiotic bacteria (Champagne et al., 2005). Therefore, microencapsulation has been used to enhance the survival of probiotic bacteria (Sheu et al., 1993; Ahmadi et al., 2014). The results showed that the encapsulated probiotic bacteria had higher survival rates compared to the non-encapsulated culture.

2.2 Application in pharmaceutical industries

The microencapsulation technique has been widely used in the pharmaceutical industry for controlled release of drugs, enhancing stability, and flavor-masking (Mendanha et al., 2009).

Arimoto et al. (2004) explored the use of microcapsule formulations for the colon-specific delivery of a water-soluble peptide drug. In general, peptides are heat-sensitive and have low permeability through polymeric membranes. Thus, this study aimed to preserve the stability of heat-sensitive drugs and the desired permeability allowing for a delayed-release profile of macromolecular drugs. The results showed that poly(EA/ MMA/HEMA) with a molar ratio of 95:8:5:40 exhibited good film-formability at 40°C. These conditions could be proposed as an appropriate way to prepare delayed-release microcapsules containing water-soluble drugs for colon-specific delivery.

Chen et al. (2014) produced doxorubicin and heparin co-loaded microcapsules using chitosan for synergistic cancer therapy. Researchers have tried to eliminate the adverse effects of the toxic chemotherapeutic agent doxorubicin (DOX) by using microencapsulation to protect...
normal tissue. Moreover, heparin (HEP) is negatively charged, making it difficult for it to undergo cellular uptake. Therefore, chitosan (CHI), which is a positively charged polymer, was used to form an HEP/CHI multilayered capsule. CHI can protect HEP from heparanase, which facilitates the intracellular delivery of HEP. The anticancer drug DOX is also encapsulated for the combination therapy. In this study, the researchers found that DOX-loaded (HEP/CHI) microcapsules had high stability in heparanase solution. Moreover, the microcapsules of DOX and HEP showed a synergistic effect against human pulmonary carcinoma (A549) cells.

Janczyk et al. (2010) used an electronic tongue for the detection of bitter taste-masking microencapsulation of active pharmaceutical ingredients (APIs), namely ibuprofen and roxithromycin, popular non-steroidal anti-inflammatory and antibiotic drugs, respectively. It was found that chemical images obtained by the measurements of pure API solutions and those by measurements of API encapsulated with taste-masking additives were significantly different. Moreover, the character of change obtained from microencapsulation was the same in both APIs.

2.3 Different viewpoints between food and pharmaceutical applications

Currently, microcapsules play important roles in the food and pharmaceutical industries. Table 1 shows the similarities and differences in the purposes of microencapsulation in the food and pharmaceutical industries. In food industry, microencapsulation is used to reduce cost, increase stability of compounds, mask undesirable tastes, and improve the release properties of compounds. In pharmaceutical industry, microencapsulation is used to increase stability, mask bitter taste, and improve the release properties of drugs, while providing for specific drug delivery. The main objective in the food industry is to produce high efficiency microcapsules with a low cost of production. However, the aim of pharmaceutical industries is to obtain high efficiency microcapsules that can deliver drugs to specific organs, regardless of the cost.

3. Challenges of Microencapsulation: Important Factors in the Microencapsulation process

The challenges associated with microencapsulation are to determine the appropriate microencapsulation technique, to select the encapsulating materials (core and wall), and to optimize the conditions for producing high-efficiency microcapsules.

3.1 The selection of microencapsulation techniques

Saikia et al. (2015) compared two microencapsulation techniques, spray- and freeze-drying, for encapsulating phenolic compounds from Averrhoa carambola pomace. Freeze-drying is a suitable technique for microencapsulating heat-sensitive compounds; however, this method is expensive (Lopez-Quiroga et al., 2012). Spray-drying is a commonly used technique in the pharmaceutical and food industries, for the encapsulation of polyphenols (Solohub and Cal, 2010). It was seen that freeze-dried microcapsules had a higher encapsulation efficiency and greater release of phenolic compounds in gastric fluid than spray-dried microcapsules.

Alvim et al. (2016) used spray-drying and spray-chilling techniques to produce microcapsules of ascorbic acid for use in biscuits. It was found that microcapsules prepared using these two methods had different mean diameters, owing to the structure formation in each method; however, they had similar encapsulation efficiencies. Microcapsules can prevent the formation of dark spots on biscuits, which may occur during the baking process. Moreover, microcapsules of ascorbic acid, produced by spray-drying and spray-chilling had a high stability during the baking process. These results indicate that both microencapsulation methods may be used for protecting active substances in baked products.

3.2 Selection of core material

Saravanan and Rao (2010) encapsulated various types of drugs, including metronidazole hydrochloride (MH), diclofenac sodium (DS), and indomethacin (IM), using pectin-gelatin and alginate-gelatin complex coacervation. MH is a water-soluble drug and is soluble at an acidic pH. DS is soluble at an alkaline pH and insoluble at an acidic pH. IM is insoluble in water. This study aimed to find the appropriate conditions for encapsulating drugs that have different water solubility. Water-soluble MH yielded microcapsules with poor encapsulation efficiency. IM produced microcapsules with an irregular shape. DS resulted in physical changes to the microcapsules, because of crystallization of free acid during the coacervation process. These results indicate that IM is an ideal core for encapsulation via complex coacervation, in terms of stability and prolonged release.

**Table 1: Purposes for the use of Microencapsulation in Food and Pharmaceutical industries**

<table>
<thead>
<tr>
<th>Industries</th>
<th>Cost reduction</th>
<th>Increasing stability</th>
<th>Taste masking</th>
<th>Improving release-properties</th>
<th>Specific drug delivery</th>
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<tr>
<td>Food</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
<td>x</td>
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<tr>
<td>Pharmaceutical</td>
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✔ = necessary factor
x = unnecessary factor

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Recently, we have studied the effect of core material on the properties of microcapsules of bioactive compounds from mulberry leaf, produced using the protein-polysaccharide interaction method (Peanparkdee et al., 2016). In this study, 60% ethanolic mulberry leaf extract (60E) and 95% ethanolic mulberry leaf extract (95E) were compared, to determine the more suitable core material. It was found that microcapsules prepared using 60E as a core material had higher phase separation and encapsulation efficiency, in addition to enhanced chemical properties, compared to microcapsules prepared using 95E. These results indicated that 60E, which has high hydrophilicity, could be used as an effective core material for producing microcapsules using the protein-polysaccharide interaction method.

3.3 Selection of wall material

de Barros Fernandes et al. (2014) evaluated the effects of replacing GA with modified starch, MD, and inulin, on the characteristics of rosemary essential oil microencapsulated by spray-drying. GA is considered an excellent wall material for entrapping essential oils; however, it is a costly polymer. Hence, polymers that can be used to replace GA have been assessed. A mixture of modified starch and MD had good properties, including high retention of volatile compounds. Moreover, the combination of modified starch and inulin was also shown to be a feasible substitute for GA in foods due to the prebiotic properties of inulin which can provide health benefits.

Carneiro et al. (2013) studied the encapsulation efficiency and oxidative stability of flaxseed oil microencapsulated by spray-drying, using different combinations of wall materials, including maltodextrin (MD), whey protein concentrate (WPC), gum Arabic (GA), and modified starches. The aim of this research was to evaluate the potential of MD combined with GA, WPC, and modified starch as alternative materials, for microencapsulation of flaxseed oil by spray-drying. From the result, the combination of MD and modified starch showed the best encapsulation efficiency. However, the mixture of MD and WPC performed better in protecting the active material against oxidation during storage. According to the results, a mixture of MD and WPC or MD and modified starch could be suggested as a suitable wall material for microencapsulation of flaxseed oil.

4. Future Trends

4.1 Food industries

The expansion in functional foods seems to be a long-term trend with important market potential. Therefore, new innovations have been introduced in the food industry (Bigliardi and Galati, 2013; Santiago and Castro, 2016). Microencapsulation is one of the innovations that are currently of interest. Moreover, many researchers continue to develop novel components for use as functional ingredients, preservatives, colorants, and flavors in food products using microencapsulation techniques.

4.2 Pharmaceutical industries

Drug microencapsulation has tremendous potential for use in the pharmaceutical industry, because it allows for the sustained and controlled release of drugs for various medical applications. However, encapsulated drugs still have limitations in organ-specific drug delivery. Obtaining high reproducibility of microencapsulated drugs remains a challenge (Lam and Gambari, 2014).

Furthermore, microencapsulation is used in other industries, including the textile and cosmetic industries, as shown in Table 2.

References


<table>
<thead>
<tr>
<th>Food Industry</th>
<th>Pharmaceutical Industry</th>
<th>Other industries</th>
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<tbody>
<tr>
<td>Functional foods</td>
<td>Specific drug delivery</td>
<td>Textile industries</td>
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<tr>
<td>- Probiotics</td>
<td>- Oral drug delivery</td>
<td>- Fragrance finishing</td>
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<td>- Antioxidants</td>
<td>- Transdermal drug delivery</td>
<td>- Color change materials</td>
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<td>- Vitamins</td>
<td>- Stomach-specific drug delivery</td>
<td>- Fire retardants</td>
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<td>- Dietary fibers</td>
<td>- Colon-specific drug delivery</td>
<td>Cosmetic industries</td>
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<tr>
<td>- Food preservatives</td>
<td>- Small intestine-specific drug delivery</td>
<td>Essential oils</td>
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<td>- Food colorants</td>
<td>- Bitter taste-masking</td>
<td>Polyphenolic compounds</td>
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<td>- Food flavors</td>
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