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Microencapsulation of Flavours

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Abstract—*Microencapsulation technology allows a compound to be encapsulated inside a tiny sphere known as microsphere/ microcapsule, having an average diameter as small as 1 mm to several hundred micro meters. Encapsulation provides an effective method to protect flavour compounds. These micro-capsules have a number of benefits such as converting liquids to solids, separating reactive compounds, providing environmental protection, improved material handling properties. Active materials are then encapsulated in micron-sized capsules of barrier polymers (gelatin, Lecithin, Gum arabic). Flavors play an important role by adding to the functionality, quality and taste acceptance of a food. To help meet this challenge, the flavor industry is active in developing new encapsulation technologies that provide flavor release under specific conditions, adding valuable flavor impact and/or extending flavor shelf life. Encapsulation entraps/coats a flavor with material and processes it to create a protective matrix or shell that completely surrounds a flavor. Encapsulation of flavours has been attempted and commercialized using many different methods such as spray drying, spray chilling or spray cooling, extrusion, freeze drying, coacervation and molecular inclusion .This article gives an overview on the general aspects of microencapsulation of flavours and its applications with reference to two of the most common techniques such as Spray drying and extrusion used for the same.*

1. INTRODUCTION

Microencapsulation is a process by which very tiny droplets or particles of liquid or solid material are surrounded or coated with a continuous film of polymeric materials. In a relatively simplistic form, a microcapsule is a small sphere with a uniform wall around it. The coated material is called active or core material, and the coating material is called shell, wall material, carrier or encapsulant. The size of particles formed through encapsulation may be classified as: macro (>5000 mm): micro (1.0–5000 mm); and nano (<1.0 mm) (king, 1990). Two main structures are single-core and multiple-core microcapsules (Figure 1). The former one is typically produced by complex coacervation, fluidized bed drying, droplet co-extrusion, and molecular inclusion, and has high core loading (e.g., 90% of total capsule weight) (Risch,1995;Thies,2001;Wu *et al*.,2005). In multiple-core capsules, which are produced principally by spray drying, the core material is dispersed throughout the wall material and the central area is occupied by the void resulting from expansion of particles during the later drying stages (Liu, 1995; Reineccius, 2001; Teixeira *et al*., 2004). Microcapsules with this structure often have a core loading of 20–30% of total capsule weight. Flavour plays an important role in consumer satisfaction and influences further consumption of foods. Food manufacturers are usually concerned about the preservation of aromatic additives, since aroma compounds are not only delicate and volatile, but also very expensive.

Encapsulation provides an effective method to protect flavour compounds from evaporation, degradation, and migration from food (Madene *et al*., 2006). Namely, creating a suitable microenvironment around flavours reduce the volatility and/or mobility of the flavour constituents (Juteau *et al*., 2004) and provides a better retention during the baking process. A vast majority of the flavour compounds used in the food industry are mainly in the form of liquid at room temperature. Conversion of liquid flavours to dry powders is an important application of microencapsulation in the food industry (Reineccius, 1994; Sharma *et al*., 1999).One of the key aims for the microencapsulation of food flavours is to control the release of these active ingredients until the right time (Reineccius, 1995). Examples of commonly used encapsulated flavours are artificial or natural flavours, spices. Many factors linked to aroma affect the overall quality of the food, examples are physico-chemical properties, concentration and interactions of volatile aroma molecules with food components (Landy *et al*., 1995). To limit aroma degradation or loss during processing and storage, it is beneficial to encapsulate volatile ingredients prior to use in foods or beverages.

Following the first commercial use of microencapsulation in 1954 to create a carbonless copy paper (Dziezak 1998; Shahidi 1993) different encapsulation techniques were developed.

2. ENCAPSULATION TECHNIQUES

Encapsulation of flavours has been attempted and commercialized using many different methods such as spray drying, spray chilling or spray cooling, extrusion, freeze drying, coacervation and molecular inclusion. The choice of appropriate microencapsulation technique depends upon the

end use of the product and the processing conditions involved in the manufacturing product. Of these the two major industrial processes used are spray drying and extrusion (Beristain *et al*., 1996; Goubet *et al.,* 1998).

Table 1: Applications of different encapsulation method in food industry. (Madene *et al*.,2006)

Encapsulation technique	Encapsulated form	Application area
Coacervation	Paste/powder/capsule	Chewing gum, toothpaste, baked foods
Spray drying	Powder	Confectionery, milk powder, instant desserts, food flavours, instant beverages.
Fluid bed drying	Powder/granule	Prepared dishes, confectionery
Spray cooling/chilling	Powder	Prepared dishes, ices
Extrusion	Powder/granule	Instant beverages, confectionery, teas
Molecular inclusion	Powder	Confectionery, instant drinks, extruded snack

The choice of wall materials depends upon a number of factors including: expected product objectives and requirements; nature of the core material; the process of encapsulation; economics and whether the coating material is approved by the Food and Drug Administration (US) or European Food Safety Authority (Europe) (Amrita *et al*., 1999).

Table 2: Characteristics of the wall material used for encapsulating flavours (Madene *et al*., 2006)

Wall material	Interest
Maltodextrin (DE < 20)	Film forming
Corn syrup solid (DE > 20) reductability	Film forming,
Modified starch Gum arabic Forming	Very good emulsifier Emulsifier, film
Modified cellulose	Film forming
Gelatin forming	Emulsifier, film
Cyclodextrin emulsifier	Encapsulant,
Lecithin	Emulsifier
Whey protein	Good emulsifier
Hydrogenated fat water	Barrier to oxygen and

3. CONTROLLED FLAVOUR RELEASE:

Controlled release may be defined as a method by which one or more active agents or ingredients are made available at a desired site and time and at a specific rate (Pothakamury & Barbosa-Canovas, 1995). For matrix systems encapsulating volatile compounds, release depends on several mutually dependent processes such as diffusion of the volatile compound through the matrix, type and geometry of the particle, transfer from the matrix to the environment, and degradation/dissolution of the matrix material (Pothakamury & Barbosa- Canovas, 1995). De Roos (2000) showed that two factors control the rate of flavour release from products, the comparative volatility of the aroma compounds in the food matrix and air phases under equilibrium conditions (thermodynamic factor) and the resistance to mass transport from product to air (kinetic factor). The mechanism of release for the capsule may be based on solvent effects, such as melting, diffusion, degradation, or particle fracture (Table 3).

Table 3: The mechanistic of flavour-controlled release (Richard & Benoıt, 2000)

Encapsulation Technique	Controlled release Mechanistic
Simple coacervation	Prolonged release
Complex Coacervation	Prolonged release (diffusion) and started release (pH, dehydration, effect mechanical, dissolution or enzymatic effect)
Spray drying	Prolonged release and started release
Fluid bed drying	Started release (pH or heat treatment)
Extrusion	Prolonged release

The advantages of controlled release are: The active ingredients are released at controlled rates over prolonged periods of time; Loss of ingredients during processing and cooking can be avoided or reduced; Reactive or incompatible components can be separated (Dziezak, 1988; Brannon-Peppas, 1993).

4. MICROENCAPSULATION BY SPRAY DRYING

Spray drying is the most commonly used encapsulation technique in the food industry (Reineccius,2004) and one of the oldest encapsulation methods, used to prepare the first encapsulated flavours using gum Arabic as the wall material . Carbohydrates, milk proteins, and new emerging biopolymers make up the three main classes of wall materials generally available and suitable for spray drying microencapsulation (Sheu ,1995; Reineccius ,1991; Lee *et al*.,2005). The suitable wall material, must be rehydrated (sometimes with heating) in water (Brenner, 1983) for the surface-active biopolymers to exhibit their emulsifying capabilities during emulsion formation (McClements, 2005). It is desirable to use a pre-determined infeed solids level that is optimum for each wall material composition. When the wall material has been hydrated, the core material must be added to make a coarse emulsion, usually via high-speed mixing or high-shear emulsification by colloid mills. A 20–25% flavour load based on total solids of the wall solution is traditional in spray drying microencapsulation was reported (Soottitantawat *et al*.,2001; Soottitantawat *et al* .,2003; . Shiga et al., 2004) Then, final emulsion will be prepared by other emulsification methods including high-pressure homogenization, e.g., microfluidization (Jafari *et al*., 2007). Following the preparation of the infeed emulsion, it will be pumped to the drying chamber of the spray drier. Two types of atomizers are

widely used: the high-pressure nozzle; and the centrifugal wheel (Finney *et al*.,2002). For the spray drying encapsulation of food flavours generally, co-current air flow is applied. The rapid evaporation of water from these droplets during surface film solidification keeps the core temperature below 100 ºC in spite of the high temperatures (>150 ºC) used in the process. The particles exposure to heat is in the range of a few seconds at most (McKernan, 1972; Sharma and Tiwari , 2001; Rosenberg *et al*., 1990) because core materials such as flavours, may contain many various components with different boiling points, it is possible to lose certain low boiling point aromatics during the drying process (Brooks, 1965; Rosenberg and Sheu,1996; Apintanapong *et al*., 2003). Spray-dried encapsulated powders typically have a very small particle size (generally less than 10 mm) with a multiple-core structure. In optimizing the process, there are at least four group of criteria that can be considered: properties of the wall materials; characteristics of the core materials; specifications of the infeed emulsion; and conditions of the spray drying.

Encapsulated material	Wall material	Parameters	Remarks
Black pepper oleoresin. (Javed et al. ,2004)	Gum arabic and modified starch	The inlet temperature - 105–110 °c outlet air temperature 110 ± 5 °C, respectively. Feed rate - 300 ml/h.	The piperine content of oleoresin decreased from 39.1– 36.2% over a period of six weeks at 30 °C. Gum arabic was found to be a better wall material than modified starch.
Cardamom oleoresin (Savitha ,2005)	Gum arabic, maltodextrin,	Pressure -5 bar. Inlet temperature - 178 °c outlet temperature 120°c. feed rate-300 g/h.	Gum arabic was found to be better wall material than maltodextrins and modified starch. The 1,8-cineole content decreased from 28.58–24.50% and α-terpinyl acetate content decreased from 50.80–46.61% over a period of six weeks at 25°c
l-menthol (Apinan et al. ,2004)	Gum arabic (GA) and modified starch (CAPSUL, HI-CAP 100)	inlet air temperature: 180 °C, outlet air temperature: 100°C±6°c, feed rate: 45 ml/min, air flow rate: 100 kg/h Rotational speed of atomizer: 30,000 rpm.	At the high aw (0.96) HI-CAP 100 was found to be a better wall material than GA and the retention of flavor was improved with increasing in wall material concentration.
Sumac flavour (Ozgur et al., 2004)	Sodium chloride, sucrose, glucose and starch	Inlet air temperature 200°C. Exit air temperature 100°C. Air velocity - 2.12 m/s.	Increasing the soluble solids content (upto 25%) increased the retention of flavor. Sucrose, glucose and starch were not suitable for spray drying of sumac due to their caramelization properties and heterogeneous form.
1-octene-3-ol(Agaricus bisporus) (Zhi-qiang Liu,2004)	Soybean hydrolyzing protein, Arabic gum, dextrin.	Feed temperature of 50–60 °C. air inlet temperature- 130–140 °C.	Best ratio of the carriers is: soybean hydrolyzing protein -10%, Arabic gum-1%, dextrin -15%.
Sweet orange oil (Jun-xia et al, 2010)	Soybean protein isolate/ gum Arabic	Inlet temperature: 160 °C, outlet temperature : 90 °C, atomizing pressure 12 MPa, air flow 0.72 m3/min, and liquid feeder pump flow 320 ml/h.	Addition of sucrose significantly improves the microencapsulation efficiency and microencapsulation yield.
Bayberry polyphenols (Zheng et al.,2010)	Ethyl cellulose	air inlet temperature : 120 °C and output temperature: 100 °C . Feed rate: 25 ml/min	The storage stability of bayberry polyphenols was also remarkably improved . The microcapsules can be used as a natural antioxidant in the food field.

Turmeric oleoresin (Kshirsagar *et al*.,2008)	Gum arabic and malto-dextrin	Inlet and outlet temperatures : 140 ± 2 °C and 88 ± 4 °C, respectively. Feed rate : 150 g/h.	Gum Arabic supplemented with 1% pullulan proved to be a better wall material in terms of stability and film forming ability for encapsulation of turmeric oleoresin
Peppermint oil (Dong *et al*.,2011)	Gelatin/ gum Arabic	Inlet and outlet air temperature are 190 ± 3 and 90 ± 3 °C Feed rate: 6 kg/h.	Transglutaminase is used as hardening agent.

5. MICROENCAPSULATION OF FLAVOR BY EXTRUSION

Encapsulation of flavours via extrusion in glassy carbohydrate matrices has been used for volatile and unstable flavours. The principal advantage of the extrusion method is the stability of the flavour against oxidation. Carbohydrate matrices have very good barrier properties and extrusion is a convenient process enabling the encapsulation of flavours(Gouin,2004). Hitherto, beads from 200 to 2000 microns have been produced by various extrusion techniques with a maximal flavour load of up to 20 % w/w [1]. The electrostatic extrusion technique was applied to immobilize an aromatic compound in calcium alginate gel microbeads. Ethyl vanilline (3-ethoxy-4-hydroxybenzaldehyde) was used as the aroma agent (in the further text vanilline). Vanilline is an important food additive as a flavour enhancer. This compound is widely used to contribute to the fragrance of commercial foods such as candies, cookies, chocolate and beverages.

ENCAPSULATED MATERIAL	WALL MATERIAL	PARAMETERS	REMARKS
Lemonade, Butter (Zasypkin and Porzio 2003)	octenylsuccinic acid anhydride (OSAN)-modified starches	2"co-rotating twin-screw extruder. steady-state pressure -not to exceed 70 kg cm^2, product temperature -not to exceed 113°C and setting time of the product in a cold airflow (13°C) -not to exceed 4 s.	Flavor retention and surface oil : 5.4 to8.8 (per 100g of product) and 23-92(per g of product).
d-Limonene (Sri Yuliani et al. ,2005)	Corn starch containing five levels of b-cyclodextrin (0 5%)	Barrel diameter -16mm. Dry feed rate - 16 g/min and Water feed rate – 4 g/min.	Flavor retention - 76.5% to 128.35% Expansion ratio -1.14 to 1.83. Water absorption index (WAI)-.22 to 7.84 g/g and Water solubility index (WSI) - 14.97% to 39.79%.
Ethyl vanilline (Verica et al. ,2008)	Alginate gel	Collecting solution was calcium Chloride concentration- 0.015 g/ml n. Voltage -4.5 kV. flow rate of the polymer solution - 25.2 cm^3/h.	Vanilla release begins at a temperature of approx. 225ºC and rapidly finishes at 247ºC. Rupture of weak bonds between alginate chains and water molecules occurs in the 50-150 °C temperature range. Polymer dehydration is most rapid at about 112 ºC.

6. CONCLUSION

This study has characterized and evaluated flavour encapsulation compositions utilizing melt extrusion. It was demonstrated that the new, essentially bland enzymatically or/and acid hydrolysed OSAN-starches can eliminate off-notes inherent with the earlier dextrinized OSAN-starches. This carrier blandness is especially critical for encapsulation of weak and delicate flavours. Comparing the identical

compositions for both extrusion and spray drying encapsulation, it has been shown that higher flavour loads, lower surface flavor and higher glass transition temperature of the compositions resulted from spray drying. The real advantage of the glassy extrusion products are related to the larger particle sizes important for a number of applications and the potential for controlled release of flavours. Electrostatic extrusion appears to be a convenient technique for the immobilisation of vanilla into small, monodisperse alginate microbeads. The understanding and control of the complex behaviour of aroma compounds in thermally processed foods require research in both domains: engineering of the matrix with a suitable microstructure and texture properties, as well as the development of the process for the manufacture of microcapsules.

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