

Review

Flavour encapsulation and controlled release – a review

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Summary Flavours can be among the most valuable ingredients in any food formula. Even small amounts of some aroma substance can be expensive, and because they are usually delicate and volatile, preserving them is often a top concern of food manufacturers. Encapsulation describes different processes to cover an active compound with a protective wall material and it can be employed to treat flavours so as to impart some degree of protection against evaporation, reaction, or migration in a food. 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. This overview describes each method cited above in terms of the basic chemical and/or physical principles involved and covers mechanisms of flavour release from food matrices.

Keywords Core retention, inclusion, microcapsules, shelf-life, volatile molecules.

Introduction

Flavour plays an important role in consumer satisfaction and influences further consumption of foods. Most available aroma compounds are produced via chemical synthesis or extraction. Foodstuffs containing synthetic flavour are often avoided, because the consumers suspect that these compounds are toxic or harmful to their health (Teixeira *et al.*, 2004).

Flavour stability in different foods has been of increasing interest because of its relationship with the quality and acceptability of foods, but it is difficult to control. Manufacturing and storage processes, packaging materials and ingredients in foods often cause modifications in overall flavour by reducing aroma compound intensity or producing off-flavour components (Lubbers *et al.*, 1998). Flavours form very complex systems because there are many variables. Some are more stable in carbohydrates which are water soluble

and some are more stable in lipid-based coating. 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.

Encapsulation is the technique by which one material or a mixture of materials is coated with or entrapped within another material or system. The coated material is called active or core material, and the coating material is called shell, wall material, carrier or encapsulant. The development of microencapsulation products started in 1950s in the research into pressure-sensitive coatings for the manufacture of carbonless copying paper (Green & Scheicher, 1955). Encapsulation technology is now well developed and accepted within the pharmaceutical, chemical, cosmetic, foods and printing industries (Augustin *et al.*, 2001; Heinzen, 2002). In food products, fats and oils, aroma compounds and oleoresins, vitamins, minerals, colorants, and

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enzymes have been encapsulated (Dziezak, 1988; Jackson & Lee, 1991; Shahidi & Han, 1993).

The process for encapsulation of sensitive compounds consists of two steps: the first is often emulsification of a core material, such as the 'lipid-*aroma*' system, with a dense solution of a wall material such as a polysaccharide or protein. The second is drying or cooling of the emulsions. A schematic illustration of encapsulation of a flavour is shown in Fig. 1. Encapsulation can be employed to retain aroma in a food product during storage, protect the flavour from undesirable interactions with food, minimize flavour-flavour interactions, guard against light-induced reactions and/or oxidation, increase flavours shelf-life and/or allow a controlled release (Reineccius, 1991; Tari & Singhal, 2002). The maximum flavour yield of the different coating techniques is reported in Table 1. The retention of flavour is governed by factors related to the chemical nature of the core, including its molecular weight, chemical functionality, polarity and relative volatility, to the wall material properties and to the nature and the parameters of the encapsulation technology. The rational design of encapsulation systems requires a physico-chemical understanding of the

mechanisms by which compounds are encapsulated and released (Chang *et al.*, 1988; Whorton, 1995; Whorton & Reineccius, 1995; Goubet *et al.*, 1998). There are growing numbers of opportunities in the food industry where greater demands are being made on the integrity of the capsules so as to provide controlled delivery of the core material at the desired time and site (Pothakamury & Barbosa-Canovas, 1995; Reineccius, 1995; Pszczola, 1998; Brazel, 1999; Gibbs *et al.*, 1999a; Augustin *et al.*, 2001).

Incorporation of small amounts of flavours into foods can greatly influence the finished product quality, cost, and consumer satisfaction. The food industry is continuously developing ingredients, processing methods, and packaging materials to improve flavour preservation and delivery (Zeller & Salieb, 1996). The stability of the matrices is an important condition to preserve the properties of the flavour materials. Many factors such as the kind of wall material (Imagi *et al.*, 1992), ratio of the core material to wall material (Minemoto *et al.*, 1999), encapsulation method (Minemoto *et al.*, 1997), and storage conditions (Minemoto *et al.*, 1997; Yoshii *et al.*, 1997) affect the anti-oxidative stability of encapsulated flavour.

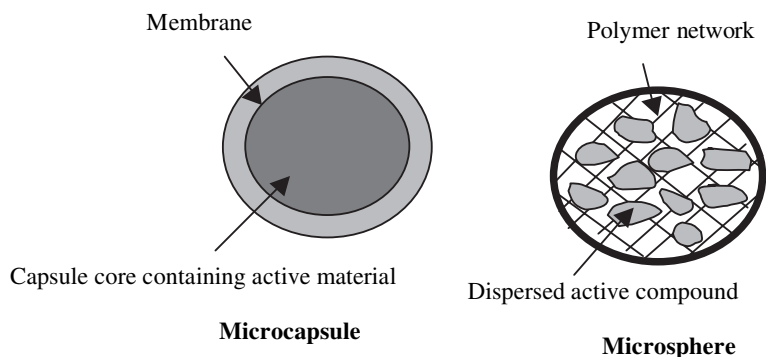


Figure 1 A schematic illustration of flavour encapsulation.

Table 1 Characteristics of encapsulation processes

	Encapsulation method	Particle size (μm)	Max. load (%)	References
Chemical techniques	Simple coacervation	20–200	<60	Richard & Benoit, 2000
	Complex coacervation	5–200	70–90	Richard & Benoit, 2000
	Molecular inclusion	5–50	5–10	Uhlemann <i>et al.</i> , 2002
Mechanical techniques	Spray-drying	1–50	<40	Richard & Benoit, 2000
	Spray chilling	20–200	10–20	Uhlemann <i>et al.</i> , 2002
	Extrusion	200–2000	6–20	Uhlemann <i>et al.</i> , 2002
	Fluidised bed	>100	60–90	Richard & Benoit, 2000

According to the encapsulation process used, the matrices of encapsulation will present various shapes (films, spheres, particles irregular), various structures (porous or compact) and various physical structures (amorphous or crystalline dehydrated solid, rubbery or glassy matrix) that will influence diffusion of flavours or external substances (oxygen, solvent) as well as the food product stability during storage.

The first aim of this paper is to describe the applications and the advantages of the encapsulation technologies and to give a critical industrial perspective. The second aim is to review types and applications of encapsulants and to describe the flavour release mechanism.

Microcapsule system

The flavour compound being protected is locked up in a membrane that isolates it from the external medium. The size of microcapsule formed can vary from a few millimetres to $<1 \mu\text{m}$ (Crouzet, 1998). The simplest of the microcapsules may consist of a core surrounded by a wall of uniform or non-uniform thickness. The core material may be composed of just one or several different types of ingredients and the carrier may be single or multilayered. The microcapsules are generally additives to a larger system and must be adapted to that system. Consequently, there are a number of performance requirements placed on microcapsules. A limited number of encapsulating methods exist (Versic, 1988), but an enormous range of different materials can be used. These include proteins, carbohydrates, lipids, gums and cellulose (Brazel, 1999). Each group of materials has certain advantages and disadvantages. For this reason, many coatings are actually composite formulations of any or all the above. 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).

For encapsulation of the flavour compounds, the carrier material must have no reactivity with the core material; be present in a form that is easy to handle, i.e. with low viscosity at high concen-

Table 2 Characteristics of the wall material used for encapsulating flavours

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

trations; allow a complete elimination of solvent in any processes requiring a phase of desolvation; give the maximum protection of the active ingredient against the external factors; ensure good emulsion-stabilization properties and effective re-dispersion behaviour in order to release the flavour at the times and the place desired (Trubiano & Lacourse, 1988; Shahidi & Han, 1993). A good knowledge of the physico-chemical interactions occurring between aroma compounds and the main constituents of foods such as lipids (Solms *et al.*, 1973; Maier, 1975), polysaccharides (Langourieux & Crouzet, 1994), and proteins (Kinsella & Damadoran, 1980; O'Neill, 1996), is required for food flavouring control. Characteristics of the major wall materials used for flavour encapsulation are reported in Table 2.

Carbohydrates

Carbohydrates are used extensively in spray-dried encapsulations of food ingredients as the encapsulating support, that is the wall material or carrier (Reineccius, 1991; Kenyon, 1995; McNamée *et al.*, 1998). The ability of carbohydrates, such as starches, maltodextrins, corn syrup solids and acacia gums, to bind flavours is complemented by their diversity, low cost, and widespread use in foods and makes them the preferred choice for encapsulation (Dziedzak, 1988; Mutka & Nelson, 1988). In addition, these materials have properties, such as low viscosities at high solids contents and good solubility that are desirable in an encapsulating agent. Starch and products derived from it, such as maltodextrins and β -cyclodextrin, are known to encapsulate aroma compounds and

interactions between flavour and these polysaccharides have been the subject of considerable studies (Reineccius, 1991; Goubet *et al.*, 1998).

Starch

Starch and starch-based ingredients (modified starches, maltodextrins, β -cyclodextrins) are widely used in the food industry to retain and protect volatile compounds. They can act as carriers for aroma encapsulation, fat replacers and also emulsion stabilizers. Many researchers have created new microporous starch materials with the aim of improving aroma retention (Smelik, 1991; Golovnya *et al.*, 1998; Glenn & Stern, 1999; Thomas & Atwell, 1999). Granules of some varieties of starches have naturally occurring 1–3 μm diameter surface pores. Zhao & Whistler (1994) showed that small starch granules have the ability to combine into interesting and potentially useful porous spheres, when spray dried with small amounts of bonding agents such as proteins or a wide range of water-soluble polysaccharides. Thus treating starch granules with amylase enzymes can create a more highly porous structure (Karathanos & Saravacos, 1992; Fannon *et al.*, 1993; Zeller *et al.*, 1999; Yamada *et al.*, 1995). The binding of volatile compounds to starch has been classified into two types. On the one hand, the flavour compound surrounded by the amylase helix through hydrophobic bonding is known as an inclusion complex. On the other hand, polar interactions have been determined which involve hydrogen bonds between the hydroxyl groups of starch and aroma compounds (Arvisenet *et al.*, 2002; Boutboul *et al.*, 2002). It was demonstrated that amylose is able to form inclusion complexes with a wide spectrum of ligand molecules, for instance with flavour compounds (Kuge & Takeo, 1968; Solms, 1986; Escher *et al.*, 2000).

Maltodextrin

Maltodextrins are formed by partially hydrolysing cornflour with acids or enzymes and they are supplied as dextrose equivalents (DEs), the DE value is a measure of the degree of starch polymer hydrolysis. They manifest the ability to form matrices that are important in forming wall

systems (Kenyon & Anderson, 1988; Shahidi & Han, 1993). In selecting the wall materials for encapsulation, maltodextrin is a good compromise between cost and effectiveness, as it is bland in flavour, has low viscosity at a high solid ratio and is available in different average molecular weights (Apintanapong & Noomhorm, 2003). Their major shortcomings are a virtual lack of emulsifying capacity and low retention of volatile compounds (Reineccius, 1991).

Nevertheless, Bangs & Reineccius (1981) showed that retention of twelve flavour compounds depends on the DE of the maltodextrins. The maltodextrins with a DE of 10 had the best retention and, as DE increased (DE 15, DE 20, DE 25 and DE 36.5), flavour retention decreased. Moreover, the flavour retention during storage increased with the DE value of maltodextrin (Yoshii *et al.*, 2001). Anandaraman & Reineccius (1986) observed that the high DE maltodextrin protected encapsulated orange peel oil against oxidation, illustrating the influence of DE to the functionality of the wall system. Desobry *et al.* (1997) confirmed this finding when examining the oxidation of β -carotene encapsulated in a maltodextrin matrix system.

However the use of the DE value has been shown to be inadequate to predict product performances in various applications (Chronakis, 1998). Recently, it has been clearly shown that the molecular weight distribution is a much more accurate tool to predict the fundamental properties of maltodextrins but it has only been applied to particular, specific issues so far (Striegel *et al.*, 1998; Wang & Wang, 2000; White *et al.*, 2003). Jouquand *et al.* (2004) studied the retention of aroma compounds by polysaccharides at three temperatures (60, 70 and 80 °C) by using phase ratio variation method. In a maltodextrin solution with a DE of 5 (10%, w/w) it was shown that the retention depended on the hydrophobicity of flavour and it was favoured by the increase of temperature (from 60 to 80 °C).

Gums

Gums and thickeners are generally bland or tasteless, but they can have a pronounced effect on the taste and flavour of foods. In general,

hydrocolloids decrease sweetness, with much of the effect being attributed to viscosity and hindered diffusion (Godshall, 1997).

Gum arabic is the gum that is most often used as a flavour-encapsulating material. Its solubility, low viscosity, emulsification characteristics and its good retention of volatile compounds make it very versatile for most encapsulation methods. In addition, the wall material is ideally suited to the encapsulation of lipid droplets as it fulfils the roles of both surface-active agent and drying matrix, thus preventing the loss of volatiles in contact with the atmosphere. However, its application within the food industry is limited, because gum arabic is more expensive than maltodextrin (Kenyon, 1995; Shiga *et al.*, 2001) and its availability and cost are subject to fluctuations; hence, there is a need to evaluate alternatives.

Mixtures of gum arabic and maltodextrin have shown promise as high solid carriers, giving acceptable viscosity in studies on microencapsulation of cardamom oil by spray drying (Sankarikutty *et al.*, 1988). When a mixture of ethyl propionate, ethyl butyrate, orange oil, cinnamic aldehyde and benzaldehyde was encapsulated in a blend of gum arabic and maltodextrins, a general trend towards an increase in retention was observed when the gum arabic fraction increased (Reineccius, 1991). Spray-dried particles formed by mixtures of maltodextrins with gum arabic are typically 10–200 μm in size and the retention of the volatile material, which is normally >80%, depends on a number of variables including the inlet temperature of the spray drier, the emulsion concentration and viscosity and the proportion of gum arabic to maltodextrins (Williams & Phillips, 2000).

Yoshii *et al.* (2001) studied flavour release from spray-dried maltodextrin/gum arabic or soy matrices as a function of storage relative humidity and showed that the release of ethyl butyrate decreased as the concentration of maltodextrin in the feed liquid increased. Apintanapong & Noomhorm (2003) used different ratios of gum arabic and maltodextrins to investigate the appropriate wall materials for encapsulation, by spray drying, of 2-acetyl-1-pyrroline. These authors showed that the 70:30 combinations of gum arabic and maltodextrin gave the best quality capsules.

Proteins

Although food hydrocolloids are widely used as flavour microencapsulants, food proteins, such as sodium caseinate, whey protein and soy protein isolates, have, apparently, not been used extensively for this purpose (Kim *et al.*, 1996). Because of their different chemical groups, amphiphilic properties, ability to self-associate and interact with a variety of different types of substances, large molecular weight, and molecular chain flexibility, these proteins have excellent functional properties such as solubility, viscosity, emulsification, and film-forming properties and would be capable of being used in encapsulation. During emulsion formation, the protein molecules become rapidly adsorbed at the newly formed oil–water interface. The resulting steric-stabilizing layer immediately protects the oil droplets against recoalescence and thereafter provides physical stability to the emulsion during processing and storage (Dagleish, 1997; Dickinson, 2001).

Whey proteins

Whey proteins manifest the functional properties that are desired for wall material (Amrita *et al.*, 1999). Applications using whey proteins as microencapsulating agents have been often reported (Moreau & Rosenberg, 1993, 1996, 1998, 1999; Rosenberg & Young, 1993; Young *et al.*, 1993a,b; Sheu & Rosenberg, 1995; Rosenberg & Sheu, 1996; Rosenberg, 1997). In the international market, whey proteins are available as whey protein isolates (95–96% protein) or whey protein concentrate (WPC-50, WPC-70) powders.

Whey protein isolates have been shown to provide a good barrier against oxidation for microencapsulated orange oil (Kim & Morr, 1996) and they provide an effective basis for microencapsulation of volatiles by spray drying. WPC 70 offers those surface properties required to stabilize emulsions. However, Fäldt & Bergenstahl (1996) showed that the encapsulating ability of WPC is low in comparison with sodium caseinate as an encapsulating material for soybean oil. Heelan & Corrigan (1998) investigated encapsulation of different water-soluble core materials in whey protein-based wall system, cross-linked with aqueous glutaraldehyde. Preparation and some

properties of microcapsules made by using water-insoluble whey protein and containing some model-lipids have been reported (Lee & Rosenberg, 2000a,b).

Whey proteins in combination with carbohydrate have been used as carrier material in encapsulation of volatile components (Young *et al.*, 1993b; Sheu & Rosenberg, 1995). In such systems, whey proteins served as an emulsifying and film-forming agent while the carbohydrates (maltodextrins or corn syrup solids) acted as the matrix-forming material (Sheu & Rosenberg, 1998). β -Lactoglobulin is the most important whey protein and possesses interesting emulsifying and foaming properties and is widely used in the food industry (Jouenne & Cruzet, 2000).

Other proteins

Protein-based materials such as polypeptone, soy protein, or gelatin derivatives are able to form stable emulsions with volatiles compounds. However, their solubilities in cold water, the potential to react with carbonyls and their high cost limit potential applications (Bangs & Reineccius, 1988).

Gelatin, a collagen hydrolysis product, is widely used in complex coacervation (Ducel *et al.*, 2004). It is a water-soluble material having an ability to form walls when a mixture of volatile aroma compounds, water and wall material is spray dried (Lee *et al.*, 1999). These flavoured gelatin capsules may be used to contain a wide range of materials, including seasonings and other flavourings for use in foods (Gourdel & Tronel, 2001). Microcapsules for volatile compound delivery have been made from coacervates of gelatin and carboxymethylcellulose (Bakker *et al.*, 1999). Addition of gelatin (1%) into maltodextrins and gum arabic carrier mixture increased the retention of ethyl butyrate in spray drying and provided better controlled release ability. This suggests that gelatin would promote the formation of crust on the surface of the droplet (Yoshii *et al.*, 2001).

The amphiphilic character and emulsification properties of sodium caseinate would appear to offer the physical and functional characteristics required to encapsulate oil materials (Hogan *et al.*, 2001). Previous work has shown that sodium caseinate provides an effective wall material for the retention of orange oil (Kim & Morr,

1996). Edris & Bergmahl (2001) have encapsulated orange oil by first preparing a triple emulsion o/w/o/w and then evaporating the outer continuous aqueous phase, which contained sodium caseinate and lactose as wall material, by spray drying. It was shown that, to encapsulate liquid oils, sodium caseinates were more efficient encapsulants than whey proteins (Keogh *et al.*, 1999; Millqvist-Fureby *et al.*, 1999). A mixture of casein and carbohydrates, such as maltodextrin and corn syrup solid, may offer potential as a cost-effective, functional, core encapsulating material (Hogan *et al.*, 2001).

Encapsulation techniques

New techniques of encapsulation continue to emerge and many companies market trademarked products from their patented technologies (Fig. 2). Flavour encapsulation is accomplished by a variety of methods. The two major industrial processes are spray drying and extrusion (Beristain *et al.*, 1996; Goubet *et al.*, 1998). Freeze drying, coacervation and adsorption techniques are also used in the industry. Major applications of the different encapsulation techniques are reported in Table 3.

Chemical processes

Coacervation

Coacervation, which is a phenomenon occurring in colloidal solutions, is often regarded as the original method of encapsulation (Risch, 1995). This technique was the first encapsulation process studied and was initially employed by Green & Scheicher (1955) to produce pressure-sensitive dye microcapsules for the manufacturing of carbonless copying paper. Coacervation consists of the separation from solution of colloid particles which then agglomerate into separate, liquid phase called coacervate (Korus, 2001). Generally, the core material used in the coacervation must be compatible with the recipient polymer and be insoluble (or scarcely soluble) in the coacervation medium. Tolstuguzov & Rivier (1997) described a process for encapsulating solid particles within a protein. In this process, the additive in a protein solution was mixed with a polysaccharide and maintained

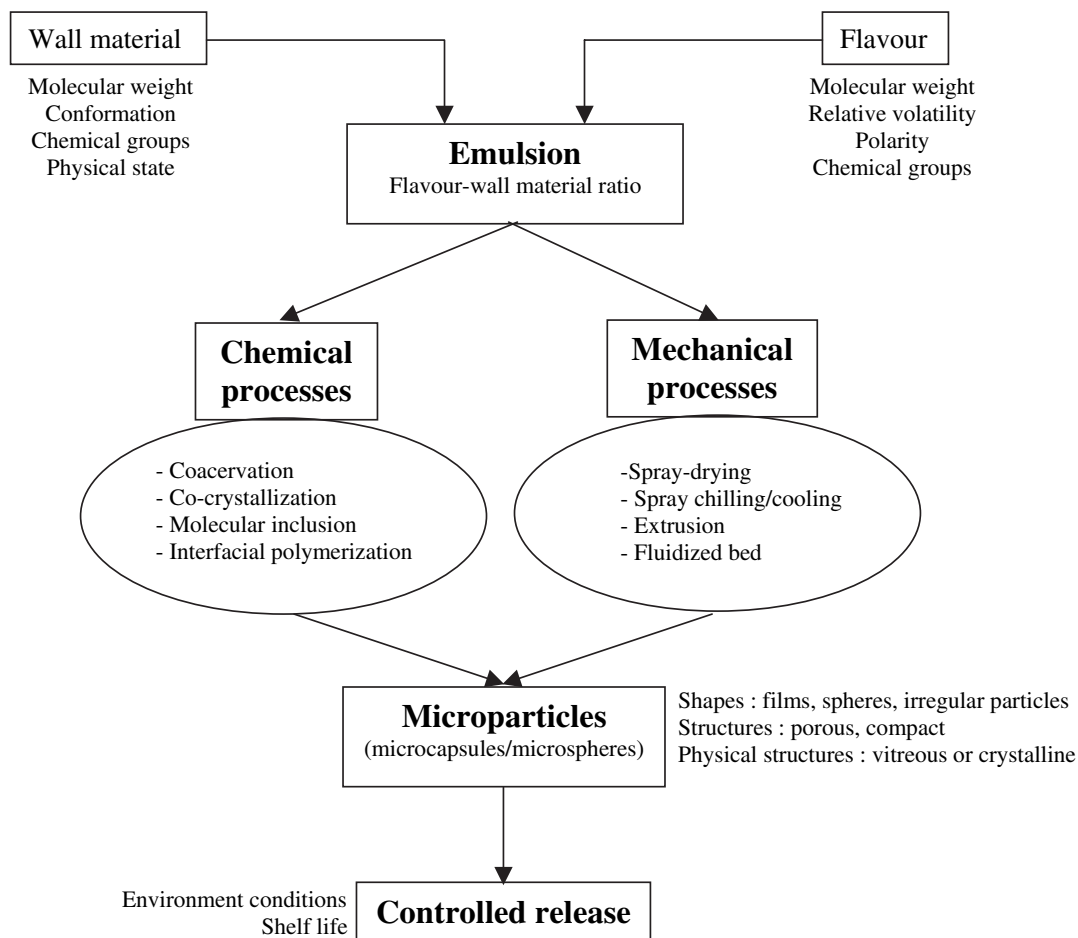


Figure 2 A schematic illustration of different processes of encapsulation of flavour compounds.

Table 3 Applications of different encapsulation method in food industry

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

at a pH greater than the isoelectric point of the protein. A mixture with two phases was formed, one of which was the heavier phase containing the encapsulated material.

Coacervation can be simple or complex. Simple coacervation involves only one type of polymer with the addition of strongly hydrophilic agents to the colloidal solution. For complex coacervation,

two or more types of polymers are used. The flavour material should be present in the mixture as the coacervation proceeds and the coacervate nuclei are then adsorbed onto the surface of the volatile compounds (Fig. 3). However, the flavour may also be added during or after phase separation. In either case, the coacervation mixture must be continually stirred. Addition of a suitable droplet stabilizer may also be necessary to avoid coagulation of the resulting microcapsules (Arshady, 1999).

In its simplest form, coacervation can be understood by describing a typical experiment. An aqueous solution of gelatin (1–10%) is prepared and maintained at 40–60 °C. Dropwise addition of ethanol to this solution leads to the formation of two phases, one of which has a higher gelatin concentration. This procedure of phase separation, as employed for preparation of microcapsules, is basically the same as the one routinely practised in polymer fractionation. The amphoteric hydrocolloidal gelatin forms complex coacervates with anionic polysaccharides like gellan gum. As the pH is gradually lowered to 4.5, microcapsules form from coacervate material (positively charged gelatin and negatively charged gellan gum) depositing around the oil droplets (King, 1995).

Until recently, this technology has not been commonly used in the food industry, this is because it is complicated and expensive (Soper,

1995; Tuley, 1996). Optimization of wall material concentration in the emulsification and coacervation process is problematic because the concentration required to obtain a fine emulsion may be different to that needed to increase the yield of microcapsules (Nakagawa *et al.*, 2004). Other limitations of flavour encapsulation by coacervation are: evaporation of volatiles, dissolution of active compound into the processing solvent and oxidation of product, because residual core materials sometimes cling to the exterior of capsule (Miles *et al.*, 1971; Flores *et al.*, 1992). The complex coacervates are highly unstable and that toxic chemical agents, such as glutaraldehyde, are necessary to stabilize them (Sanchez & Renard, 2002).

Co-crystallization

Compared with various flavour encapsulation processes, co-crystallization offers an economical and flexible alternative as the procedure is relatively simple (Jackson & Lee, 1991; Chen, 1994). Small numbers of studies have been published that report on the use of co-crystallization to drive the encapsulation process (Chen *et al.*, 1988; Beristain *et al.*, 1994, 1996). Chen *et al.* (1988), described numerous products that can be encapsulated by co-crystallization process. These included fruit juices, essential oils, flavours, brown sugar, etc.

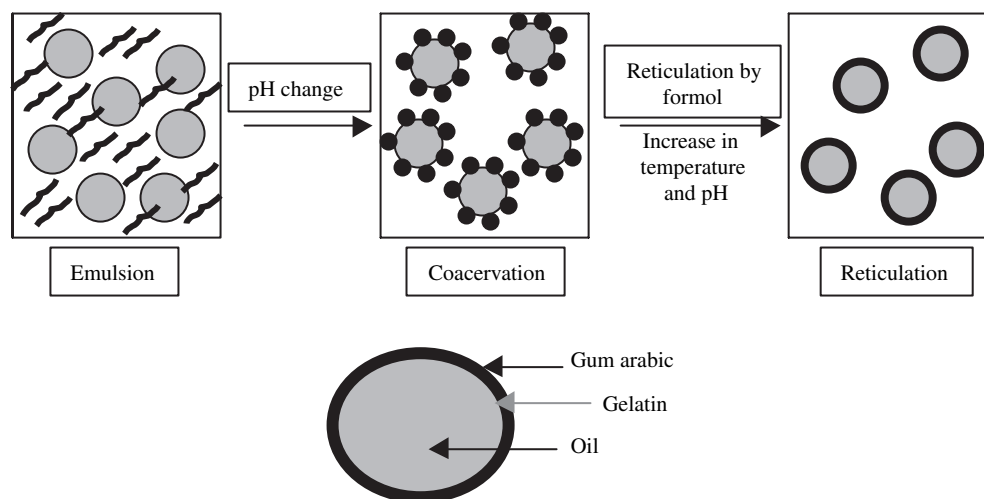


Figure 3 Principle of the complex coacervation method.

For instance, spontaneous crystallization of supersaturated sucrose syrup is achieved at high temperature (above 120 °C) and low moisture (95–97 ° Brix) and aroma compounds can be added at the time of spontaneous crystallization (Bhandari *et al.*, 1998). The crystal structure of sucrose can be modified to form aggregates of very small crystals that incorporate the flavours; either by inclusion within the crystals or by entrapment. This serves to enhance flavour stability (Mullin, 1972; Chen *et al.*, 1988). The granular product has a low hygroscopicity and good flowability and dispersion properties (LaBell, 1991; Quellet *et al.*, 2001). But, during the process, the liquid flavour was transformed into dry granules and some heat-sensitive compounds may have degraded (Bhandari *et al.*, 1998). Beristain *et al.* (1996) encapsulated orange peel oil using a co-crystallization process and stored the resultant product. The authors showed that co-crystallization products retained as much volatile oil as did spray-dried and extruded products. Although the product had a free-flowing property, the addition of a strong anti-oxidant was necessary to retard development of oxidized flavours during storage.

Molecular inclusion

Cyclodextrins are enzymatically modified starch molecules, which can be made by the action of cyclodextrin glucosyltransferase upon starch. After cleavage of starch by the enzyme, the ends are joined to form a circular molecule with $\alpha(1-4)$ linkage. The characteristics of cyclodextrin and their use as encapsulating material have been extensively described (Hedges & McBride, 1999). The inclusion complexes are defined as the result of interactions between compounds in which a smaller guest molecule fits into and is surrounded by the lattice of the other (Godshall, 1997).

A typical application is the protection of unstable and high added value speciality flavour chemicals (Uhlemann *et al.*, 2002). In the food industry, flavours have been encapsulated within cyclodextrins (Reineccius & Risch, 1986; Loftsson & Kristmundsdottir, 1993; Reineccius *et al.*, 2002).

The inner hydrophobic cavity of β -cyclodextrin is torus shaped, and its molecular dimensions allow total or partial inclusion of a wide range of

aroma compounds. The central cavity of the molecule creates a relatively hydrophobic environment, whereas its external surface has a hydrophilic character. This unique conformation is largely responsible for the characteristic physico-chemical properties of cyclodextrins (Shieh & Hedges, 1996; Steinbock *et al.*, 2001).

According to Goubet *et al.* (1998), the retention of aroma compounds can be influenced to a greater or lesser extent by the molecular weight and shape, steric hindrance, chemical functionality, polarity and volatility of the core material. For instance, if the volatile compound is small compared with the cavity, only some of the surface of the guest molecule is in contact with the walls of the cavity. Consequently, the full potential of the sensitive molecule to interact with the cyclodextrin is not realized (Fig. 4) (Hedges *et al.*, 1995). The presence of water or high temperature is required to liberate guest molecules once complexed (Reineccius *et al.*, 2002). Pajington (1986) and Bhandari *et al.* (1999) listed several methods for complexing β -cyclodextrin with flavour compounds. The most used are as follow:

- 1 stirring or shaking a cyclodextrin with flavours in aqueous solution and filtering off the precipitated complex;
- 2 blending solid cyclodextrin with guest molecules in a powerful mixer, and bubbling the flavours, as vapours, through a solution of cyclodextrin;

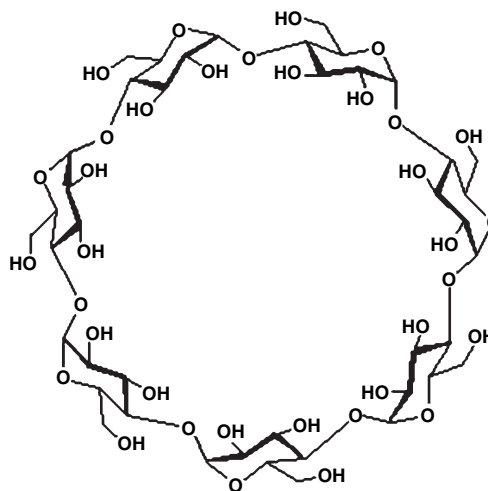


Figure 4 Structure of β -cyclodextrin molecule.

3 Kneading the flavour substance with the cyclodextrin-water paste.

The same authors compared methods of encapsulating lemon oil in β -cyclodextrin and showed that the spray-drying system was much simpler and more quickly achieved a reasonable powder (very fine with an excellent flowability) than drying in a vacuum oven. Qi & Hedges (1995) provided experimental details of a co-precipitation method deemed to be very suitable for laboratory evaluation. These authors claim that slurry or paste method was more convenient for large-scale production, as less water would, subsequently, have to be removed during drying. The addition of maltodextrins and β -cyclodextrin as stabilizing or thickening agents could retain some aroma compounds in food matrices during thermal processes (cooking, pasteurization) (Jouquand *et al.*, 2004). Cyclodextrins are relatively expensive: a recent report suggests that the cost of cyclodextrins would never be <\$6 per kg (Gouin, 2004).

Mechanical processes

Spray drying

Spray drying is a commercial processes which is widely used in large-scale production of encapsulated flavours and volatiles (Deis, 1997). The merits of the process have ensured its dominance, these include availability of equipment, low process cost, wide choice of carrier solids, good retention of volatiles, good stability of the finished product, and large-scale production in continuous mode (Reineccius, 1989). According to Teixeira *et al.* (2004), this technique provides a high retention of aroma compounds during drying. Spray drying can be used for many heat-labile (low-boiling point) materials because of the lower temperatures that the core material reaches (Dziezak, 1988). Sharma & Tiwari (2001) and Re-MI (1998) presented a review on micro-encapsulation using spray drying. The process involves the dispersion of the substance to be encapsulated in a carrier material, followed by atomization and spraying of the mixture into a hot chamber (Dziezak, 1988; Watanabe *et al.*, 2002). The resulting microcapsules are then transported to a cyclone separator for recovery. The formation of a stable emulsion, in which the

wall material acts as a stabilizer for the flavour, is considerable.

Retention of volatile core material during encapsulation by spray drying is achieved by chemical and physical properties of the wall and core materials (Reineccius & Coulter, 1969; Menting *et al.*, 1970; Bomben *et al.*, 1973; Leahy *et al.*, 1983; Rosenberg, 1985; Reineccius, 1988; Rosenberg *et al.*, 1990; Desobry *et al.*, 1997), solid content of the dryer, processing temperature and also by the nature and the performance of the encapsulating support, i.e. emulsion-stabilizing capabilities, film-forming ability and low viscosity at a high concentration (Rosenberg *et al.*, 1990; Goubet *et al.*, 1998). The functionality profile of wall materials that are optimal for spray drying includes a high solubility in water, a low viscosity at high concentration, effective emulsification and film-forming characteristics and efficient drying properties (Reineccius, 1988; Re-MI, 1998). When core materials of limited water solubility are encapsulated by spray drying, the resulting capsules are of a matrix-type structure. In such, the core is organized into small droplets coated with wall materials that are embedded in the wall matrix. Microstructures of spray-dried capsules have been shown to be affected by wall composition and properties, flavour-to-wall ratio, atomization and drying parameters, uneven shrinkage at early stages of drying, the effect of a surface tension-driven viscous flow and storage conditions (Buma & Henstra, 1971a,b; Kalab, 1979; Greenwald, 1980; Greenwald & King, 1982; Keith, 1983; Mistry *et al.*, 1992; Rosenberg & Young, 1993; Young *et al.*, 1993a,b).

One disadvantage of spray drying is that some low-boiling point aromatics can be lost during spray drying and the core material may also be on the surface of the capsule, this would encourage oxidation and possible flavour changes of the encapsulated product (Dziezak, 1988; Desobry *et al.*, 1997). Kerkhof (1994) has reviewed the difficulties in quantitatively understanding drying processes, identifying the non-linearity of the processes, the complex transfer processes and the tendency for the dominating phenomena to change during drying. The degree of aroma retention is strongly dependent on the moisture content of the final microcapsules and on the humidity of the exhaust air.

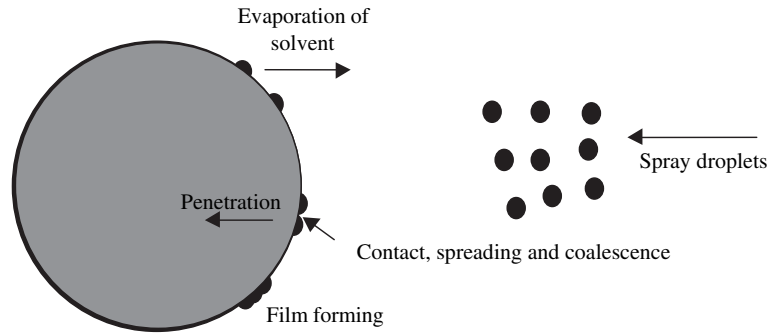


Figure 5 Schematic approach of film formation with the fluid bed coating device (Richard & Benoît, 2000).

Other problems with spray drying for flavour or microencapsulation are that this technology produces a very fine powder, typically in the range 10–100 µm in diameter, which needs further processing, such as agglomeration, to make the dried material instantly or make it more soluble if it is a liquid application. The ability of agglomeration to influence the properties of spray-dried encapsulated flavourings is limited by the processing characteristics of the carrier materials (Risch, 1995; Buffo *et al.*, 2002). Table 4 outlines the advantages and disadvantages of the spray-drying technique. To avoid these problems, spray-drying powders can be agglomerated by using the fluidized bed process.

Fluid bed spray coating is a three-step process. First, the particles to be coated are fluidized in the hot atmosphere of the coating chamber. Then, the coating material is sprayed through a nozzle onto the particles and film formation is initiated, there follows a succession of wetting and drying stages. The small droplets of the sprayed liquid spread

onto the particle surface and coalesce. The solvent or the mixtures is then evaporated by the hot air and the coating material adheres on the particles (Fig. 5) (Jacquot & Perneti, 2003). This technique relies upon a nozzle spraying the coating material into a fluidized bed of aroma particles in a hot environment (Fig. 6). The size of the product varies from 0.3 to 10 mm (Panda *et al.*, 2001).

The fluid bed process is already widely employed in the pharmaceutical and cosmetic industry, both of which have a greater budget for processing than the food industry, but its use in the food industry to encapsulate flavours has also been studied (Dezarn, 1998; Lee & Krochta, 2002). It is the most suitable method for

Table 4 Advantages and disadvantages of the using of spray-drying

Advantages	
Low operating cost	
High quality of capsules in good yield	
Rapid solubility of the capsules	
Small size	
High stability capsules	
Disadvantages	
Produce no uniform microcapsules	
Limitation in the choice of wall material (low viscosity at relatively high concentrations)	
Produce very fine powder which needs further processing	
Not good for heat-sensitive material	

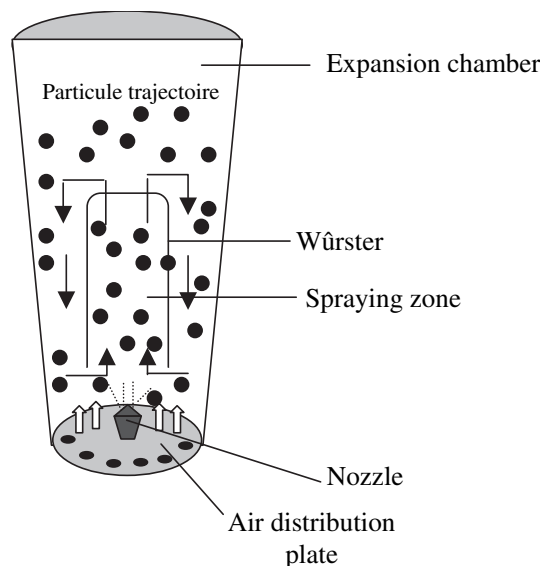


Figure 6 Fluid bed drying with the Würster device.

encapsulating spray-dried flavours because the wall materials used in flavour systems are readily dissolved and form strong interparticle bridges on re-drying (Buffo *et al.*, 2002). This technology allows specific particle size distribution and low porosities to be designed into the product (Uhlmann & Mörl, 2000). Other advantages of fluidized bed (Mujumdar & Devahastin, 2000) are:

- 1 high drying rates because of good gas-particle contact, leading to optimal heat and mass transfer rates;
- 2 smaller flow area;
- 3 high thermal efficiency; lower capital and maintenance costs;
- 4 ease of control.

According Chua & Chou (2003), the fluidized bed dryer is a low-cost drying technology for developing countries compared with some state-of-the-art drying equipment, such as spray dryers.

Freeze drying

The freeze-drying technique, which is lyophilization, is one of the most useful processes for drying thermosensitive substances that are unstable in aqueous solutions. In this process, upon water crystallization, the non-frozen solution is viscous and the diffusion of flavours is retarded. Upon starting freeze drying, the surface of the solution becomes an amorphous solid in which selective diffusion is possible (Karel & Langer, 1988). Buffo & Reineccius (2001) compared spray drying, tray drying, drum drying and freeze drying to encapsulate cold-pressed orange oil Valencia with gum acacia and modified food starch. They concluded that freeze drying is the process that gives the most desirable properties to spray-dried powder. Minemoto *et al.* (1997) compared oxidation of menthyl linoleate when encapsulated with either gum arabic by hot air drying and freeze drying. These authors showed that freeze drying was better than hot air drying. Indeed, the menthyl linoleate encapsulated by freeze drying was more slowly oxidized at any relative humidity and this did not change during storage. Heinzlmann & Franke (1999) showed that the production of dried microencapsulated fish oil by freezing and subsequent freeze drying offered an opportunity to achieve a product with

good resistance to oxidation. It was shown that the freeze drying process maintained the shape of the microcapsules because of fixation by freezing (Nagata, 1996).

However this drying technique is less attractive than others because the costs of freeze drying are up to 50 times higher than spray drying (Desobry *et al.*, 1997) and the storage and transport of particles produced is extremely expensive (Jacquot & Perneti, 2003), the commercial applicability is also severely restricted by the long processing time (Barbosa-Canovas & Vega-Mercado, 1996).

Spray cooling/spray chilling

Spray cooling and spray chilling are the least expensive encapsulation process and are routinely used for encapsulation of aroma compounds to improve heat stability, delay release in wet environments and/or convert liquid flavour into free-flowing powders (Gouin, 2004). These technologies are similar to spray drying where the core material is dispersed in a liquefied coating or wall material and atomized. There is generally no water to be evaporated. There is emulsification of the flavour compounds into molten wall materials, followed by atomization to disperse droplets from the feedstock. After that the droplets are immediately mixed with a cooling medium and subsequently solidify into powder form (Lamb, 1987; Risch, 1995).

Spray chilling

In the spray chilling technique, the coating material is melted and atomized through a pneumatic nozzle into a vessel generally containing a carbon dioxide ice bath (temperature $-50\text{ }^{\circ}\text{C}$) as in a hot-melt fluidized bed. Thus droplets adhere on particles and solidify forming a coat film. The process is suitable for protecting many water-soluble materials that may otherwise be volatilized or damaged during thermal processing (Augustin *et al.*, 2001). The encapsulating material is typically a fractionated or hydrogenated vegetable oil with a melting point in the range of $32\text{--}42\text{ }^{\circ}\text{C}$. Spray chilled products have applications in bakery products, dry soup mixes and foods containing a high level of fat.

Spray cooling

This method is similar to spray chilling, the only difference is the temperature of the reactor in which the coating material is sprayed. A molten matrix material containing minute droplets of the core materials may be spray cooled. Also, vegetable oil can be used and the normal melting point is 45–122 °C (Risch, 1995). The disadvantage of spray chilling and spray cooling is that special handling and storage conditions can be required (Taylor, 1983).

Extrusion

Extrusion was first patented in 1957 and further developed by the group that originally patented the technique (Swisher, 1957). Encapsulation of flavours via extrusion has been used for volatile and unstable flavours in glassy carbohydrate matrices (Reineccius, 1991; Blake, 1994; Benczedi & Blake, 1999; Gunning *et al.*, 1999; Qi & Xu, 1999; Saleeb, 1999; Benczedi & Bouquerand, 2001). The principal advantage of the extrusion method is the stability of flavours against oxidation. Carbohydrate matrices in the glassy state have very good barrier properties and extrusion is a convenient process enabling the encapsulation of flavours in such matrices (Gouin, 2004). However, process parameters and diffusion of flavour from extruded carbohydrates is enhanced by structural defects such as cracks, thin wall, or pores formed during or after processing (Wampler, 1992; Villota & Hawkes, 1994). Extrusion of polymer solutions through nozzles to produce either beads or capsules is mainly used on a laboratory scale (Heinzen, 2002).

Simple extrusion

A volatile compound is dispersed in a matrix polymer at 110 °C. This mixture is then forced through a die and the filaments obtained are plunged into a desiccant liquid that, by hardening the extruded mass, traps the active substances (Rizvi *et al.*, 1995; Crouzet, 1998). The most common liquid used for the dehydration and hardening process is isopropyl alcohol. The strands or filaments of hardened material are broken into small pieces, separated and dried

(Risch, 1995). Crocker & Pritchett (1978) used the extrusion method for encapsulation of citrus oils with corn syrup solids and glycerine. These authors report that several factors improve the quality of capsules and these included the DE of the corn syrup, emulsifier and flavour oil content and emulsification pressure.

Double-capilarity extrusion devices

Coaxial double capillary device

The core substance and the carrier material are fed through, respectively, the inner and outer opening of a coaxial double capillary. The core is usually a liquid and the polymer may be applied as a solution or as a melt (the core and the coat fluids must be immiscible). At the tip of the coaxial nozzle the two fluids form a unified jet flow, which breaks up to form the corresponding microdroplets.

Centrifugal extrusion device

This process utilizes nozzles located on the outer circumference of a rotating cylinder. The liquid flavour is pumped through the inner orifice and the liquid shell material through the outer orifice forming a coextruded rod of flavour components surrounded by wall material. As the device rotates, the extruded rod breaks into droplets which form capsules (Schalmeus, 1995).

Recycling centrifugal extrusion

The technology of rotating disc extrusion is combined with a facility for recycling of the excess coating fluid. The core material is dispersed in the carrier material. The suspension is extruded through the rotating disc in such a way that the excess coating fluid is atomized and separated from the coated particles. Excess coating fluid is then recycled, while the resulting microcapsules are hardened by cooling or solvent extraction. Optimizing the cook temperature, pressure, emulsifier level, residence time and extrusion vessel pasteurization permits the production of encapsulated flavouring with a high flavour load. But, as stated previously, the diffusion of flavours out of extruded carbohydrates is enhanced by structural defects such as cracks, thin walls, or pores formed during or after processing (Miller & Mutka, 1986;

Wampler, 1992). However, the major problem still facing this process is related to emulsion stability, this is difficult to obtain in extremely viscous carbohydrate melts (Risch, 1988).

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). Many researchers have sought a better understanding of the effects that govern the flavour release from complex matrices as this represents an important target in many fields, including the food industries (Guichard, 2000). An overview of physical chemistry relevant to flavour release has been presented previously (Taylor, 1998). 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 5).

Table 5 The mechanistic of flavour-controlled release (Richard & Benoît, 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

Flavour retention in the matrix is greatly dependent on the type of food ingredient and the physico-chemical properties of the flavour compound. Retention will clearly induce a noticeable decrease in flavour perception. Generally, flavour release decreases with increasing lipid level in the food matrix, with the exception of hydrophilic compounds possessing log P values [P , volatile permeability ($\text{mol m}^{-1} \text{s}^{-1}$ per bar)] near or below zero (Guichard, 2002). The presence of salts increases the volatility of an aroma compound; this is in contrast with the effects of salts on other small molecules, such as caffeine or naringin, where they induce a solubilization effect (Druaux & Voilley, 1997). Boland *et al.* (2004) investigated the release of eleven flavour compounds from gelatine, starch and pectin gels. These authors showed that flavour release was significantly affected by the texture of the gels. Thus, the gelatin gel showed large increases in flavour release in the presence of saliva, while the starch and pectin gels showed a reduction in flavour release under these conditions. Interactions between proteins and aromas have been the subject of numerous studies (Lubbers *et al.*, 1998), showing that covalent binding, hydrogen binding and hydrophobic interactions are all detectable.

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).

Release of flavour by diffusion

Diffusion is controlled by the solubility of a compound in the matrix (this establishes a concentration in the matrix which drives diffusion) and the permeability of the compound through the matrix. Diffusion is important in food because it is the dominant mechanism in controlled release from encapsulation matrices (Crank, 1975; Cussler, 1997).

The vapour pressure of a volatile substance on each side of the matrix is the major driving force influencing diffusion (Gibbs *et al.*, 1999a). The principal steps in the release of a flavour

compound from matrix system are: diffusion of the active agent to the surface of the matrix; partition of the volatile component between the matrix and the surrounding food and transport away from the matrix surface (Fan & Singh, 1989). It should be obvious that if the food component is not soluble in the matrix, then it will not enter the matrix and so diffusion will not take place irrespective of the pore size of the matrix (Reineccius, 1995).

Two distinct mechanisms of diffusion may apply. One mechanism is molecular or static diffusion, which is caused by the random movement of the molecules in the stagnant fluid. The rate of molecular diffusion varies only slightly with flavour type. The second mechanism is eddy or convective diffusion, which transports elements of the fluid from one location to another, carrying with them the dissolved solute. The rate of eddy diffusion is usually much higher than the rate of molecular diffusion and is independent of the flavour type (Roos, 2003).

Release of flavour by degradation

The release of an active compound from a matrix-type delivery system may be controlled by diffusion, erosion or a combination of both. Homogeneous and heterogeneous erosion are both detectable. Heterogeneous erosion occurs when degradation is confined to a thin layer at the surface of the delivery system, whereas homogeneous erosion is a result of degradation occurring at a uniform rate throughout the polymer matrix (Pothakamury & Barbosa-Canovas, 1995).

Release of flavour by swelling

In systems controlled by swelling the flavour dissolved or dispersed in a polymeric matrix is unable to diffuse to any significant extent within the matrix. When the matrix polymer is placed in a thermodynamically compatible medium, the polymer swells because of absorption of fluid from the medium. The aroma in the swollen part of matrix then diffuses out (Fan & Singh, 1989). The degree of swelling is controlled by water absorption or presence of solvents such as glycerine or propylene glycerol (Gibbs *et al.*, 1999b).

Release of flavour by melting

This mechanism of release involves the melting of the capsule wall to release the active material. This is readily accomplished in the food industry as there are numerous materials that can be melted and that are approved for food use (lipids, modified lipids or waxes). In such applications, the coated particles are stored at temperatures well below the melting point of the coating, then heated above this temperature during preparation or cooking (Sparks *et al.*, 1995).

Conclusion

The stakes in the functional food and pharmaceutical industry is the efficient encapsulation of high added value ingredients, such as polyunsaturated fatty acids, flavours, vitamins and ingredients, the volatile permeability of which is used to improve functionality. Numerous developments have been made in the field of encapsulated food flavours. This is because of several favourable properties of the encapsulated flavours. The choice of an appropriate technique of encapsulation depends on the properties of the flavour compounds, the degree of stability required during storage and processing, the properties of the food components, the specific release properties required, the maximum obtainable flavour load in the powder and the production cost. However, each encapsulation process, usually developed to solve a particular problem encountered during a products development, presents advantages and disadvantages. The relationships among problems, capabilities, and encapsulation methods are discussed in this review. Microencapsulation by spray drying is the most economical and flexible way that the food industry can encapsulate flavour ingredients. Thus, this technology is now becoming available to satisfy the increasingly specialized needs of the market. In addition, fluid-bed process is also becoming a promising encapsulation technique for large-scale production of flavour powders to be applied in food industry. Today, the comprehensive technology of encapsulation enables it to satisfy all relevant product requirements, including tailoring food properties, easy product handling, improved shelf-life and controlled release. However, the most important aspect of research and

development is an understanding of how industrial constraints and requirements to make a microencapsulation technology viable can be modelled at the laboratory scale so as to make it possible to undertake the transition to full-scale production and marketing of the final product.

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