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MICROCAPSULES

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Background

Microencapsulation is the coating of small solid particles, liquid droplets, or gas bubbles with a thin film of coating or shell material. Although no officially approved definition of a microcapsule exists, most workers use the term microcapsule to describe particles with diameters between 1 and 1000 μm that contain a desired ingredient of some sort. Particles smaller than 1 μm are called nanoparticles; particles greater than 1000 μm can be called microgranules or macrocapsules.

Many terms have been used to describe the contents of a microcapsule: active agent, actives, core material, fill, internal phase, nucleus, and payload. Many terms have also been used to describe the material from which capsules are formed: carrier, coating, membrane, shell, or wall. For the sake of consistency, in this article, the material being encapsulated is called the core material, and the material from which the capsule is formed is called the shell material.

Several reviews discuss the general features of microencapsulation technology, including encapsulation techniques and applications not discussed here. A number of authors have reviewed the preparation of microcapsules that contain food components and the application of microcapsules to food products.

An understanding of microencapsulation technology and the potential contribution that microcapsules can make to food products rests on a knowledge of mass transport phenomena, properties of coating materials, and an understanding of processes by which small particles are produced. Since the primary purpose of microencapsulation is to control in some manner mass transport behavior, the shell of a microcapsule must control diffusion of material either from a microcapsule or into a microcapsule (Figure 1). The

shell can provide protection of sensitive food components such as flavors, vitamins, or salts from oxygen, water, light and heat, convert difficult-to-handle liquids into free-flowing powders readily incorporated into various foods or isolate specific food components during storage.

In most cases, food components concentrated inside microcapsules are released as the food is consumed or during a food preparation step. Release is achieved by destroying the integrity of the microcapsule shell. This is done by dissolving it in water, melting it, or mechanically rupturing it. There are cases where no release of core material is desired until after the food has been ingested and is present in the digestive system. Perhaps, the intent is for the core material carried by a microcapsule never to be released. In these latter situations, the capsule shell must remain intact throughout the food preparation and ingestion steps.

In order to develop microcapsules with shells that function as intended, it is essential to understand the fundamentals of mass transport through shell materials from which microcapsules are formed, especially under use conditions. In many cases, the shell is thin, perhaps having a thickness of a few micrometers or less. Thus, the morphology or structure of a capsule shell has a significant impact on microcapsule performance. Overall, capsule morphology also affects capsule performance. The geometry of a microcapsule sample can vary significantly and often is a function of the process by which the microcapsule was formed. Figure 2 illustrates two typical capsule structures, with Figure 2a representing a continuous core/shell capsule in which a single continuous shell of uniform thickness surrounds a continuous spherical region of core material, and Figure 2b representing a multinuclear capsule in which a number of small domains of core material are distributed uniformly throughout a matrix of shell material.

During the development of microcapsules for a specific food application, it is tacitly assumed that a food-grade microcapsule shell material with suitable

barrier and fabrication properties is available at an economically viable price. Proper selection of such materials requires an appreciation of the properties of candidate coating materials and requires an understanding of materials science. Significantly, few, if any, candidate capsule shell materials are able to provide all desired functions economically, so compromises must consistently be made when selecting specific shell materials for a given application.

Table 1 lists representative examples of capsule shell materials currently used to produce microcapsules for commercial food products, and the chemical

class to which the shell material belongs, the encapsulation process typically used to produce microcapsules with each shell material and frequent food applications. Although gelatin–gum arabic complex coacervate capsules treated with glutaraldehyde are approved for limited consumption of selected food flavors, they are not approved for general food use (see **Figure 3**). Shell material costs vary greatly. As expected, the food industry favors the cheapest acceptable shell materials that are capable of providing desired performance, are available in commercial quantities, and are approved by the FDA.

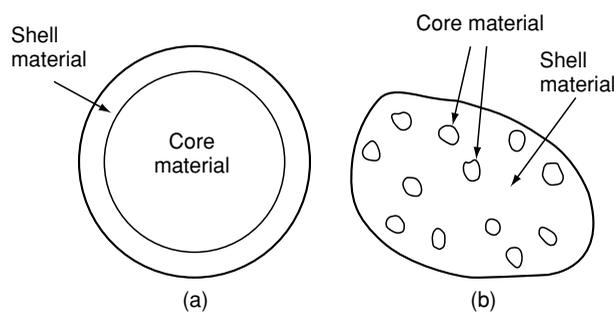


Figure 1 Two structures characteristic of many commercial microcapsules: (a) continuous core/shell structure; (b) multi-nuclear.

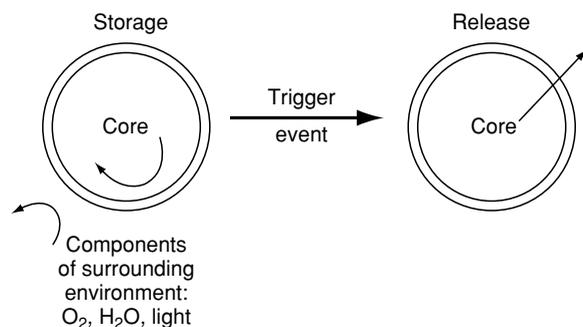


Figure 2 Ideal storage and release behavior properties desired for many capsules used by the food industry. Courtesy Thies Technology.

Microencapsulation Processes

General Comments

Many microencapsulation processes exist. Some are based exclusively on physical phenomena. Some utilize polymerization reactions to produce a capsule shell. Others combine physical and chemical phenomena. Because there are so many encapsulation techniques, it is logical to make an effort to attempt to categorize or classify them in some manner, thereby providing a means of identifying the concepts on which various encapsulation technologies are based. Many authors do this by identifying encapsulation processes as either chemical or mechanical processes. This author prefers to classify them as Type A or Type B processes, since so-called mechanical or physical processes actually may involve a chemical reaction, and so-called chemical processes may rely exclusively on physical phenomena. **Table 2** lists representative examples of Type A and B processes. Type A processes are processes in which microcapsules are immersed in a liquid-filled stirred tank or tubular reactor throughout the encapsulation procedure. In a Type B process, a gas phase is involved at some stage of the encapsulation process. Microcapsules are formed by spraying droplets of coating material on a core material being encapsulated, solidifying liquid droplets sprayed or ejected into a gas phase, gelling droplets sprayed or ejected into a liquid bath, or

Table 1 Shell materials used to produce commercially significant microcapsules

Shell material	Chemical class	Encapsulation process	Applications
Gum arabic	Polysaccharide	Spray drying	Food flavors
Derivatized starch	Polysaccharide	Spray drying	Food flavors
Gelatin	Protein	Spray drying	Vitamins
Whey protein	Protein	Spray drying	Fats
Maltodextrins	Low-molecular-weight carbohydrates	Spray drying and desolvation	Food flavors
Hydrogenated vegetable oils	Glycerides	Fluidized bed	Assorted food ingredients
Complex coacervation	Protein–polysaccharide complex	Complex coacervation	Assorted flavors

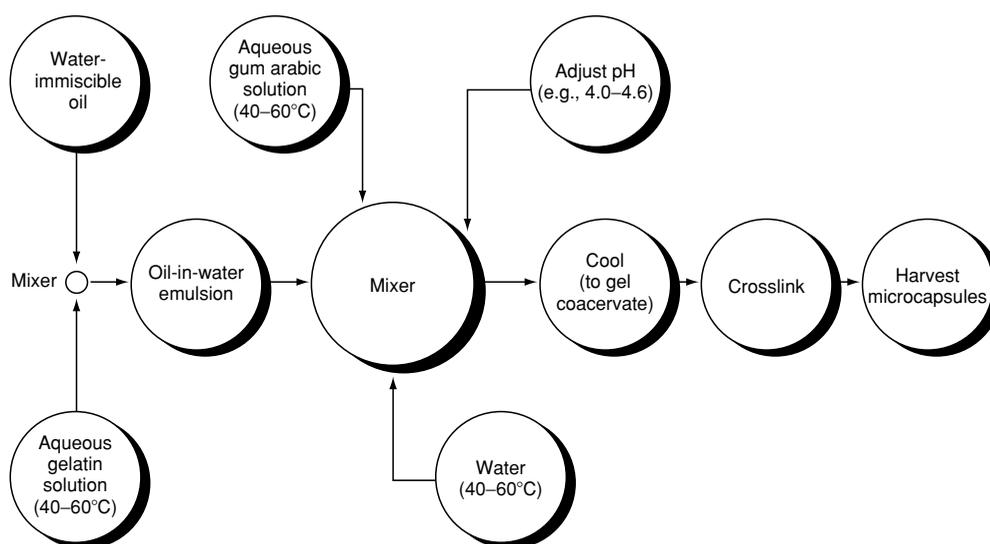


Figure 3 Sequence of steps of a complex coacervation encapsulation process. Courtesy Thies Technology.

Table 2 Representative examples of two broad classes of encapsulation processes

Type A processes	Type B processes
Complex coacervation	Spray drying and spray chilling
Polymer/polymer incompatibility	Fluidized bed
Interfacial polymerization at liquid/liquid and solid/liquid interfaces	Interfacial polymerization at solid/gas or liquid/gas interfaces
<i>In-situ</i> polymerization	Centrifugal extrusion
Solvent evaporation or in-liquid drying	Extrusion or spraying into a desolvation bath
Submerged nozzle extrusion	Rotational suspension separation (spinning disk)
	Melt spinning

by carrying out a polymerization reaction at solid/gas or liquid/gas interfaces.

No encapsulation process developed to date is able to produce the full range of capsules desired by potential capsule users. Some processes readily produce small, liquid-filled capsules, whereas others produce relatively large capsules with a solid core material, capsules with water-soluble shells, or capsules with water-insoluble shells. In order to provide insight into which process is most appropriate for a specific food application, features of Type A and B encapsulation processes used by the food industry are summarized in the following sections. Processes that produce capsules not approved for food applications are discussed elsewhere.

Type A Encapsulation Processes

Complex coacervation Complex coacervation is a phenomenon in which cationic and anionic water-soluble polymers interact in water to form a liquid, polymer-rich phase called a complex coacervate. This coacervate is used to form a microcapsule shell (see Figure 3). Gelatin is normally the cationic polymer used. A variety of natural and synthetic anionic water-

soluble polymers interact with gelatin to form a complex coacervate, but the food industry appears to use gum arabic exclusively. When the complex coacervate forms, it is in equilibrium with a dilute solution called the supernatant. The supernatant acts as the continuous phase in which the complex coacervate is dispersed. If a water-insoluble core material such as a flavor or fat is dispersed in the system, each droplet or particle of dispersed core material is spontaneously coated with a thin film of coacervate provided that the coacervate adsorbs on the surface of the dispersed oil droplets. When this liquid film is gelled, capsules are formed. The 'wet' complex coacervate gel is a very rubbery shell that deforms extensively without rupturing. In order to increase the strength of the water-swollen shell and create a gel structure that is not thermally reversible, complex coacervate capsules shells are typically cross-linked with glutaraldehyde.

Complex coacervation encapsulation processes can encapsulate many water-immiscible oils such as vitamins, plant oils and flavors as well as water-insoluble solids. Such processes routinely produce single capsules 20–800 μm in diameter that contain 80–90 wt.% core material. Capsules outside these size and core

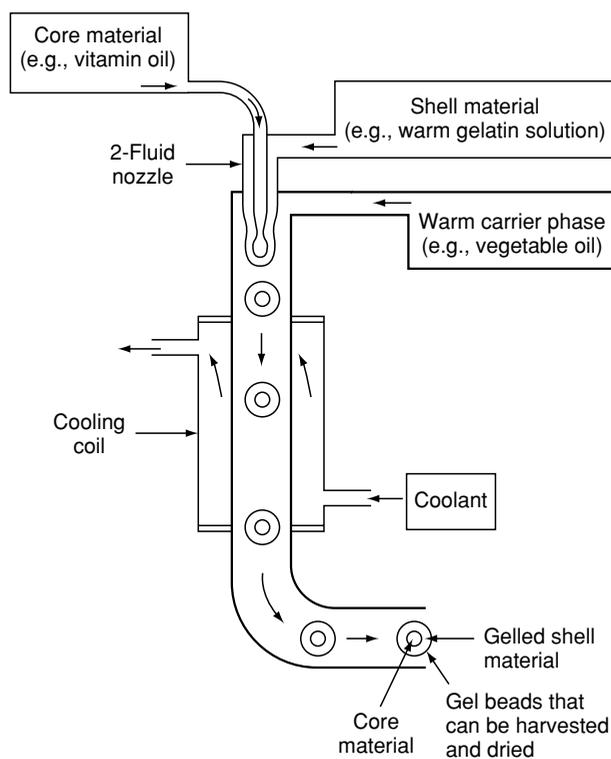


Figure 4 Stationary two-fluid nozzle used to produce capsules.

content ranges can be produced, although considerable experience may be required. Most coacervate capsules approach the continuous core/shell structure shown in Figure 2a, but the shell may not have a uniform thickness. Complex coacervation processes are adversely affected by active agents that have a finite water solubility, are surface active, or are detrimentally affected by pH conditions used to produce the microcapsules. The shell of dry complex coacervate capsules is sensitive to variations in atmospheric moisture content and becomes plasticized at a relative humidity of 70% or higher. Thus, the release properties of a complex coacervate capsule can be affected significantly by environmental conditions to which the capsules are exposed during storage.

Submerged centrifugal force and nozzle processes

Several Type A processes use submerged centrifugal force or nozzles to form microcapsules (see Figure 4). The nozzles can be rotating, stationary, or vibrating. They may be single or multifluid nozzles. An example of the former is the spinning cup encapsulation apparatus developed by the Atlantic Coast Fisheries Company. This consists of a spinning cup that contains a row of small holes. When the cup is immersed in a container filled with mineral oil or vegetable oil, an oil-in-water (o/w) emulsion fed into the interior of this spinning cup is extruded dropwise by centrifugal

force through the openings into the oil phase that surrounds it. This creates a suspension of o/w emulsion droplets suspended in an oil phase. The continuous phase of these droplets is an aqueous solution of a polymer that gels on cooling (e.g., gelatin), whereas the dispersed phase is a water-immiscible liquid like a vitamin or fish oil. As the extruded droplets sink in the oil phase, the aqueous phase of the emulsion gels thereby creating gel beads throughout which an oil such as a vitamin or fish oil is dispersed. The beads are harvested from the oil phase by a solvent wash and dried. The dried beads have the structure shown in Figure 2b (see Figure 5).

Various submerged stationary and vibrating nozzles are used to produce microcapsules. A number of different nozzle configurations exist, and these produce capsules with a variety of structures. Sophisticated multifluid vibrating nozzles have been incorporated into capsule production units. Such units typically produce microcapsules by coextruding an aqueous polymer solution that gels on cooling and an oil to be encapsulated into a third or suspending fluid. The suspending fluid is a cooled oil phase in which the aqueous polymer solution gels, thereby forming gel beads with a continuous core/shell structure, as shown in Figure 2a. Such beads are isolated from the suspending fluid, washed in order to remove residual oil from the suspending medium, and subsequently dried.

To date, submerged Type A centrifugal force and various nozzle encapsulation processes are used primarily for the encapsulation of a variety of water-immiscible oils. The capsules tend to be larger than 250–500 μm but are typically formed from food-grade shell materials that can be gelled thermally.

Type B Encapsulation Processes

General comments Type B encapsulation processes utilize centrifugal force, extrusion, coextrusion, and spray technology in order to produce microcapsules. They predate Type A encapsulation processes, since spray drying encapsulation was developed in the 1930s. This class of encapsulation processes generally utilizes self-contained encapsulation units marketed by equipment manufacturers. In contrast, equipment for Type A encapsulation processes is typically custom-designed and manufactured for a specific capsule producer. It is the author's opinion that Type B processes often cannot produce microcapsules < 100 μm economically, whereas many Type A processes can. A notable exception is spray drying. Other exceptions undoubtedly exist, but there is clearly a question of how well many current Type B processes function when an effort is made to use them to produce economically viable amounts of small (e.g., < 100 μm) microcapsules.

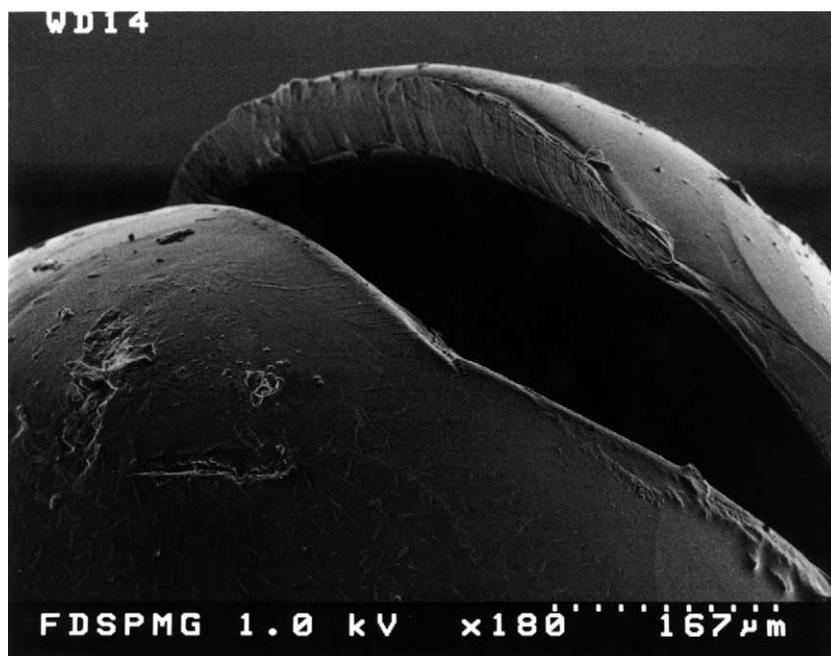


Figure 5 Scanning electron photomicrograph of a cut flavor capsule produced by a submerged nozzle encapsulation apparatus. Magnification: 180 \times . Courtesy Thies Technology.

Spray drying Spray drying is used to produce a variety of encapsulated food components. The first step is to emulsify the core material, typically a water-immiscible flavor, vitamin, fish or plant oil, in a concentrated (35–60 wt.%) aqueous solution of shell material until 1–3 μm oil droplets are obtained. The shell material normally is a water-soluble material like gum arabic, maltodextrin (hydrolyzed starch), modified starch, hydrolyzed gelatin, sugars, or various mixtures of these materials. All are approved for food use. They are water-soluble and not chemically cross-linked. Capsules prepared from them dissolve in water and release core material without leaving any residual capsule shell debris. **Figure 6** illustrates the steps involved in the process. (See **Drying: Spray Drying**.)

Once a suitable core material/shell material dispersion has been prepared, the resulting emulsion is fed as droplets into the heated chamber of a spray drier. The droplets are rapidly dehydrated, thereby producing dry capsules that fall to the bottom of the drying chamber where they are harvested. Capsules produced in this manner are typically 10–300 μm in diameter. They may have an irregular geometry and be aggregates of a number of small particles. Spray-dried capsules contain a number of small droplets of core material dispersed throughout it, as illustrated in **Figure 2b**.

Spray-dry encapsulation has a number of advantages:

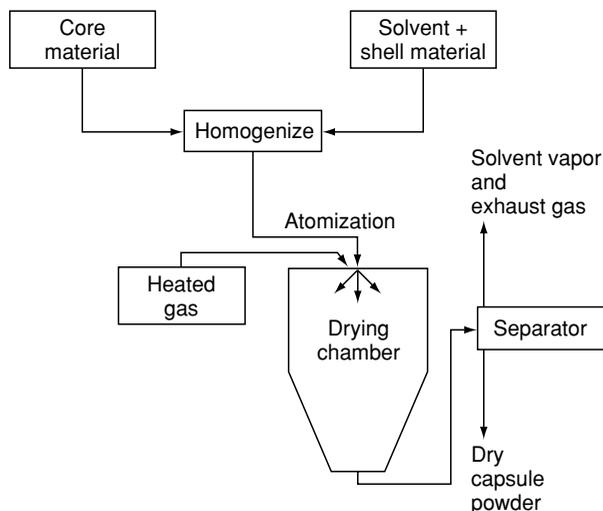


Figure 6 Steps involved in a spray-drying encapsulation process.

1. It is a well-established technology.
2. It utilizes readily available equipment.
3. It can produce large amounts of capsules economically.

It also has several limitations. For example, the list of candidate water-soluble shell materials is limited to candidate materials that form concentrated (30–60 wt.% solids) aqueous solutions of sufficient low viscosity that they can be pumped. Another limitation

is the 20–30% core loading carried by most spray-dried capsules. Spray-drying protocols that allow core loading up to 50–60% have been reported, but current spray-dried capsules carry a lower loading. A persistent problem with spray-dried capsules is free or surface core material. Because water evaporation from a capsule in the chamber of a spray drier occurs rapidly, it is not uncommon to harvest spray-dried capsules that have a significant amount of free or unencapsulated oil. This free oil exists because of discontinuities in the capsule shell, some of which form as a result rapid evaporation of water from the droplets being dried, thereby forming microcapsules. The higher the core loading, the more pronounced this problem can become. Free or surface oil is undesirable, since it is susceptible to oxidation and development of an off-odor or off-taste when the encapsulated core material is a fragrance or a flavor, respectively. It has been found that maximizing core loading and minimizing free core content involve a judicious choice of coating material, emulsifying agent, and spray-dryer operating conditions. Finally, it is important to stress that low-boiling-point compounds with a finite degree of water solubility have posed a persistent problem to spray-drying encapsulation. Such compounds volatilize from the capsules in the spray-drying chamber.

In summary, spray drying is a viable commercial method of forming microcapsules for the food industry. It is an established, comparatively low-cost encapsulation technology that continues to develop. To date, spray drying for food products has primarily been used to encapsulate flavors, various fish and plant oils as well as liquid vitamins.

Spray chilling Spray chilling, cooling, or congealing are variations of conventional spray-drying. In these cases, chilled air is used to solidify molten capsule shell material formulations rather than volatilize a solvent. Various fats, waxes, fatty alcohols, fatty acids, or combinations of these materials are the shell materials used. In such encapsulation procedures, the active agent is dispersed in a molten shell material with the aid of an emulsifier, if necessary. The resulting dispersion is atomized through heated nozzles into a cooling chamber analogous to that used in a spray drier. The shell material is solidified by cooling, thereby producing solid particles.

Particles produced by this method have water-insoluble shells. The processing temperature has an influence on shell material polymorphism, a phenomenon that is characteristic of many fats. If polymorphic changes in a fat coating occur on storage, such changes can have a significant negative effect on capsule storage and release properties.

Fluidized-bed coaters Fluidized-bed coaters function by suspending a bed or column of solid particles in a moving gas stream, usually air. Three types of fluidized beds are available: top-spray, tangential-spray, and bottom-spray (see Figure 7). These units differ in location of the nozzle or nozzles used to apply the coating formulation. Nevertheless, in each case, a liquid coating formulation is sprayed on to the individual suspended particles, and the freshly coated particles are cycled into a zone where the coating formulation is solidified either by solvent evaporation or by cooling. This coating and drying sequence may be repeated until a desired coating thickness has been applied. (See **Drying: Fluidized-bed Drying.**)

In a bottom- or tangential-spray unit, droplets of liquid coating material leaving a spray nozzle move

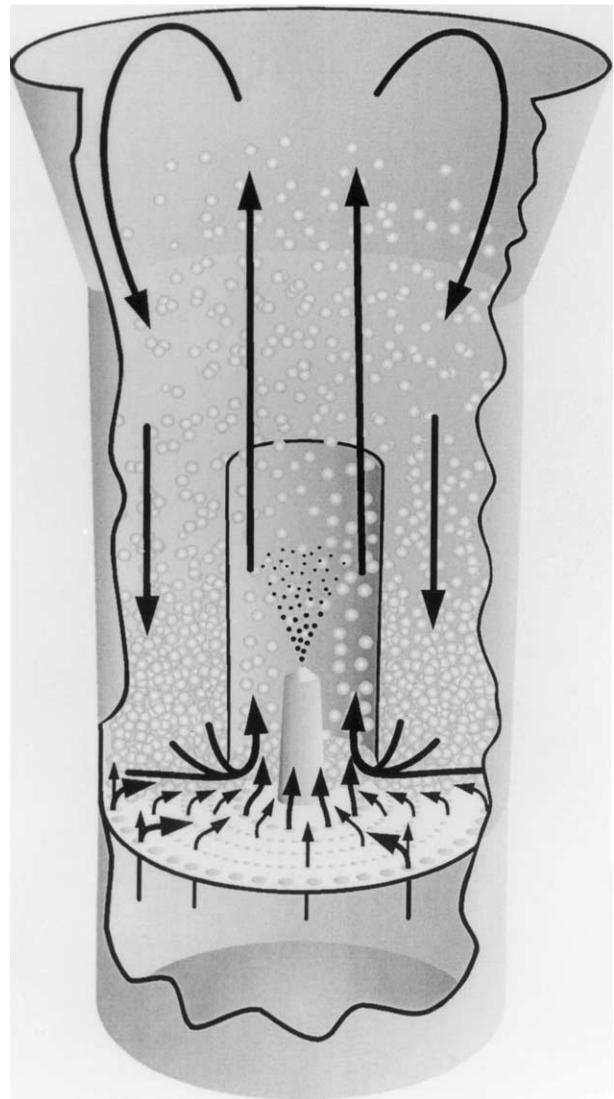


Figure 7 Bottom spray or Wurster fluidized-bed coating apparatus. Courtesy Coating Place, Verona, WI.

concurrent with the gas stream used to suspend the bed of solid particles being coated. In a top-spray unit, the coating formulation is sprayed down on and into the top of the fluidized bed. The droplets of spray leaving the nozzle move countercurrent to the gas stream until they impact the particles being coated. In the former case, a uniform film of coating material can be deposited when the coating formulation contains a volatile solvent. In the latter case, volatile solvent(s) in the coating formulation can evaporate from the spray droplets, thereby increasing the solids content, perhaps to such a degree that they cannot spread on to the particles being coated. This spray-drying effect has been used to explain why solvent-based coating formulations applied in top-spray fluidized bed coaters often yield coated particles with a degree of internal void volume and porous coatings. Enteric coatings applied as aqueous latex dispersions are an exception. Such coatings applied in a top-spray fluidized-bed coater form a continuous coating analogous to that obtained with tangential- and bottom-spray units. Molten fats also can be applied by top-spray units, since, in such cases, there is no solvent evaporation. Although top-spray units have limitations, they are claimed to be simpler to operate and have a higher production capacity than the other types of units.

Application of the capsule shell material in a series of steps, as is typically done in a fluidized-bed coating process, is both an advantage and a disadvantage. The advantage is that sequential deposition of the shell formulation in a number of different cycles covers up or heals defects found in any single application, thereby yielding relatively defect-free coatings. It is also possible to coat layers of different shell materials on a particle surface. The disadvantage is that it takes time to apply the capsule shell in a series of cyclical steps, and this increases the operating costs.

Fluidized-bed coaters can encapsulate only solid particles or perhaps porous particles into which a liquid has been absorbed. However, no other encapsulation technology can apply as broad a range of shell materials: hot melts, aqueous latex dispersions, organic solvent solutions, or aqueous solutions. Several commercial organizations use fluidized-bed coaters to coat various solid food ingredients with fats (see [Figure 8](#)). This appears to be the primary use of fluidized-bed coaters by the food industry.

Desolvation or liquid extraction encapsulation technology Desolvation encapsulation processes consist of dissolving a shell formulation in a finite amount of water, thereby forming a concentrated solution (e.g., 40 wt.%) of shell material. Core material is dispersed

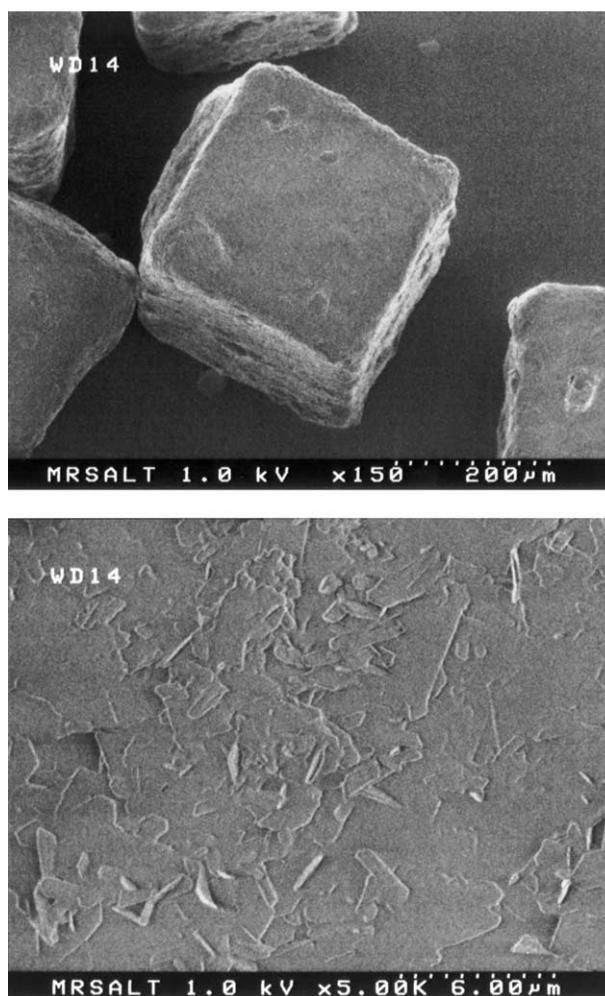


Figure 8 Scanning electron photomicrographs of a single NaCl crystal coated with a fat by a fluidized-bed coating process: top, 150 \times magnification; bottom, 5000 \times magnification. Courtesy Thies Technology.

in this solution with the aid of a surfactant. The dispersion produced can be extruded or atomized directly into a desolvation bath ([Figure 9](#)) that solidifies the shell formulation by extracting the solvent used to dissolve the shell material from the spray droplets. Alternately, the core material/shell dispersion can be extruded or atomized into a vessel that contains excess spinning solvent. The spinning bath produces droplets of core/shell formulation that are subsequently solidified in a desolvation bath. Capsules with a relatively low core loading (e.g., 15–20%) are produced, although more recent patents indicate that a core loading up to 40% is feasible.

The core material is generally a water-immiscible liquid such as a flavor. The shell material can be various combinations of carbohydrate(s) or protein(s). Spinning solvents (e.g., mineral or vegetable oil) are immiscible with the shell material solvent (water).

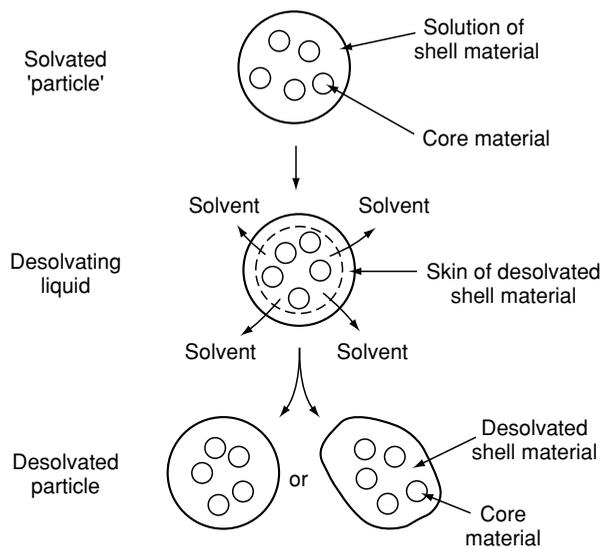


Figure 9 Sequence of steps that occur in a desolvation bath.

Water-miscible desolvation solvents (e.g., isopropanol, ethanol) are nonsolvents for the shell material (s). The desolvation solvent, often cooled, extracts water from the shell formulation, thereby producing solid particles or rods. Residual desolvation solvent is removed from solidified particles by drying. Rods or strands of solidified extrudate can be broken up into small particles by agitation in the extraction vessel or after isolation.

For this encapsulation technology to be successful, the desolvation solvent must be able to extract solvent (e.g., water) from the dispersed core/shell formulation with minimal simultaneous leaching of core material from the formulation. This occurs if the desolvation solvent rapidly solidifies the outer surface region of core/shell formulation droplets or rods to form an outer barrier that prevents diffusion of core material into the extraction solvent, but allows diffusion of water from the interior of the droplets to the extraction medium.

Desolvation encapsulation technology can produce water-soluble food-grade capsules loaded with a range of flavors. Typical shell materials include edible water-soluble materials like gum arabic, maltodextrins, or modified starches (i.e., the same materials used in spray drying). This technology can be carried out in an inert atmosphere and at reduced temperatures, thereby minimizing loss or degradation of sensitive components. It can incorporate water soluble materials in water-soluble shells and produce stable capsules free of unencapsulated core material, since the desolvating liquid extracts actives from defective capsules.

Problems with this technology include the use of solvents other than water that are more costly than

heated air, are flammable, must be recycled, and must be essentially eliminated from the final product. Desolvating liquids also can break core/shell material emulsions, thereby preventing suitable particle formation. The formation of stable emulsions in concentrated aqueous shell solutions can pose serious problems, especially if they are heated.

Melt extrusion A melt extrusion encapsulation process (Figure 10) involves dispersing a core material in a molten shell formulation at 85–125 °C with or without the aid of a surfactant. Once formed, the dispersion is extruded as filaments into a relatively cool environment that solidifies the extruded mass. The receiving environment can be a gas phase or a tank that contains a suitable solvent. If extruded into a gas phase, the cooled mass is simply broken up into particles and used. If extruded into a solvent (e.g., –20 °C isopropanol), the solvent simultaneously cools and removes unencapsulated or free core material from the filaments. The solidified product is subsequently dried and broken up to yield particles with the multinuclear structure shown in Figure 11. The particles are glass matrices loaded with dispersed core material. Flavor-loaded particles produced in this manner have an excellent resistance to oxidation during storage. An anticaking agent like pyrogenic silica can be added to enhance particle flow properties. (See **Extrusion Cooking: Principles and Practice.**)

The patent literature discloses a number of melt extrusion methods and combinations of shell materials used to produce particles loaded with a core material. Claimed core loading in the final product ranges from 5 to 40% of final particle weight, although a loading of 5–30% is most common. Preferred core materials are flavoring oils. Both oil- and water-miscible flavors have been entrapped. The core/shell formulation typically has a temperature of ≤125 °C at the time of extrusion. An antioxidant

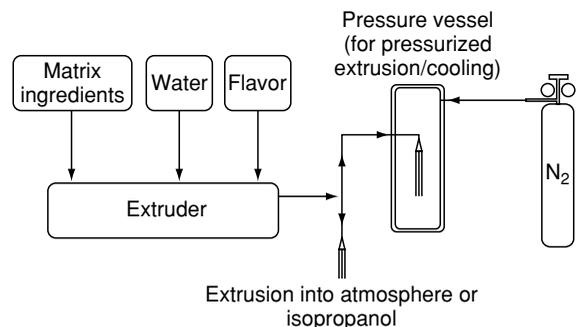


Figure 10 Melt extrusion encapsulation process. Courtesy Thies Technology.

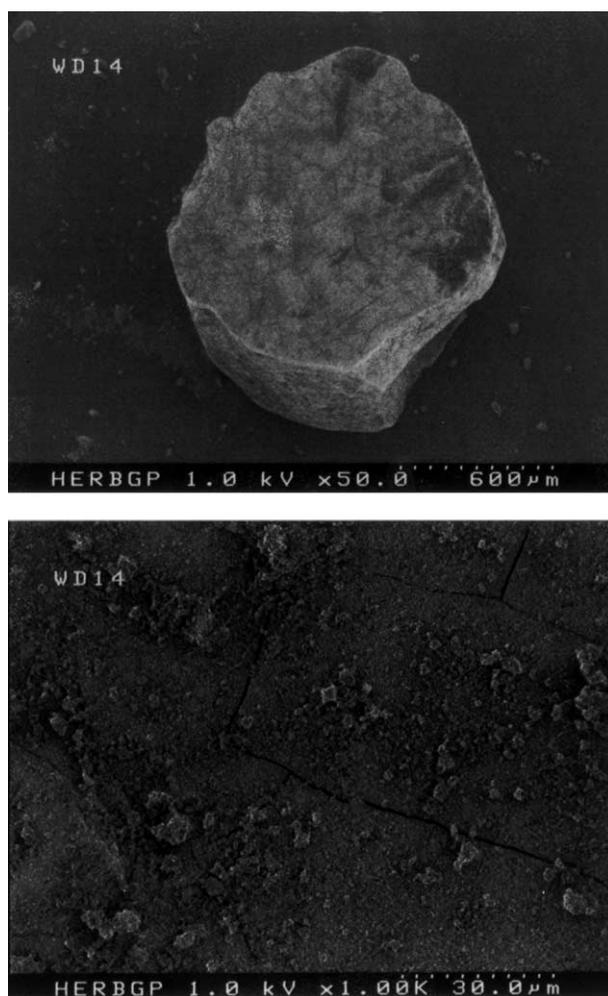


Figure 11 Scanning electron photomicrographs of the cross-sectional surface of a commercial flavor capsule produced by melt extrusion. Top, 50 × magnification; bottom, 1000 × magnification. Courtesy Thies Technology.

can be added to heat-sensitive oils. Preferred shell materials are various combinations of sugars, corn-syrup solids, maltodextrins, modified starches, and gum arabic, although other shell materials such as proteins like gluten have been used. In all cases, the water present in the system at the time of extrusion is limited to that required to reduce melt viscosity of the core/shell formulation to a point where extrusion occurs freely. This is usually no more than 10% of the mass of material extruded. Extrusion is carried out under pressure through spinnerets or screw extruders (single and double) with zoned heating. That is, different parts of the extruder screw and die-head are at different temperatures.

The shell formulation in the original extrusion process was corn-syrup solids containing 3–8.5% water. Improved properties were obtained by using a corn-syrup solids shell formulation plasticized with

glycerol, and a further improvement in the process was achieved by using a mixture of sucrose and maltodextrins with a dextrose equivalent of less than 20 (preferably 10–15). This mixture produced particles with an improved stability towards oxygen and humidity variations.

Since the processing temperatures involved in melt extrusion are relatively high, loss of volatile components and oxidative degradation of heat-sensitive materials are problems that must be addressed. Accordingly, particle formation and solidification can be carried out in an extrusion system pressurized with an inert gas like nitrogen or carbon dioxide. The final particles are isolated under pressure in order to avoid loss of volatiles resulting from venting or the formation of so-called puffed particles with flaws that cause loss of volatile core material.

Suspended nozzles A number of workers have produced microcapsules by ejecting droplets that contain a core and shell material from suspended nozzles into a gas phase, usually air. The droplets pass through a gas phase until the capsule shell is solidified by cooling, or they fall into a curing bath where they are gelled and subsequently harvested. The particles produced can have a structure like that shown in [Figure 2a or b](#). In all cases, the rate of droplet production and, hence, rate of capsule production is increased by increasing the number of nozzles, using centrifugal force, or a combination of these approaches.

Many types of nozzles have been described: single-fluid, two-fluid, or even three-fluid nozzles. Some are stationary, whereas others are vibrating or spinning. They can be single-fluid tubes through which an emulsion of core material in an aqueous shell solution flows by gravity extrusion or under an applied external pressure. Improved control of droplet, and ultimately capsule, size distribution is accomplished by vibrating the extrusion tubes or orifices. Single-fluid tubes usually produce capsules with a structure like that shown in [Figure 2b](#), since the extruded droplets are dispersions of core material in a solution of shell material.

When droplets are formed by using multifluid nozzles, each droplet formed contains a continuous core region surrounded by a liquid shell or by placing the nozzles in a spinning head through which the droplets are ejected with the aid of centrifugal force. The nature of the shell material determines how ejected droplets are converted into capsules (See [Figure 2b](#)). If the shell material is a relatively low-viscosity hot melt that crystallizes rapidly on cooling (e.g., a wax or wax toughened with a polymer), the droplets are converted into solid particles as they fall

away from the nozzle and are cooled. Suitable core materials are typically polar liquids like water or aqueous solutions, since they are immiscible with a range of hot-melt shell materials like waxes.

Alternatively, droplets emerging from the spinning nozzle may have a shell that is an aqueous solution of a gelable polymer. In this case, the compound droplets fall into a gelling bath where they are converted into gel beads. A specific example is the gelation of an aqueous sodium alginate shell by an aqueous CaCl_2 gelling bath. The calcium alginate gel beads produced can be dried to give free-flow capsules. In one previously documented encapsulation apparatus, compound droplets produced by a vibrating two-fluid nozzle pass through an aqueous CaCl_2 (20%) mist, thereby causing a degree of prehardening before the droplets fall into the actual CaCl_2 hardening bath. Prehardening minimizes droplet breakage on impact with the surface of the curing bath.

An understanding of the physical phenomena involved in the formation of microcapsules by nozzle devices requires an understanding of the principles of fluid mechanics. Capsules prepared by centrifugal extrusion tend to be large, with diameters typically ranging from over $250\ \mu\text{m}$ up to several millimeters. Capsules produced by stationary vibrating nozzles can be smaller (e.g., $100\ \mu\text{m}$ in diameter) and very uniform in size. Atomization can produce much smaller capsules, although the particle size distribution can be large, and the capsules may not have a perfectly spherical geometry.

Spinning or rotating disc In spinning disc encapsulation processes, core material dispersed or emulsified in a liquid shell formulation is fed onto the center of a

rotating disc. Centrifugal force generated by the rotating disc forces the dispersed core material and liquid coating formulation across the surface of the disc to the outer edge. There, the dispersion is thrown off the disc into a gas phase (usually air) as discrete droplets. If the core material is in particle form, the droplets are discrete particles covered with a thin film of liquid shell formulation. If the core material is an emulsion or a dispersion of particles in a shell formulation, droplets filled with dispersed core material are produced. If the shell is a hot-melt formulation, it cools and solidifies as the particles fall through the gas phase, thereby producing capsules that can be harvested and used. If the shell formulation is an aqueous polymer solution that can be gelled by ions or a combination of ions and cooling, the coated particles fall into a liquid-filled curing bath that gels the shell formulation. The particles with a gelled coating can be used as gel particles or, in principle, can be dried to a free-flowing powder.

Disc geometry, diameter, and speed of rotation, as well as volume flow rate of liquid across the disc are parameters that affect the size of the capsules produced. In the case of gel beads, surface tension of the aqueous phase being dispersed in droplet form affects gel bead geometry. In addition to capsules, one patented spinning disc process produces particles of shell material free of core material. In this process, the disc is operated in such a manner that the particles of shell material collect in a zone or region distinctly separated from the capsules and can be recycled. A variety of hot-melt shell materials can be applied to dispersed solid particles, but a melt viscosity below 5000 centipoises is favored. Capsule shell formulations that do not solidify rapidly pose problems.

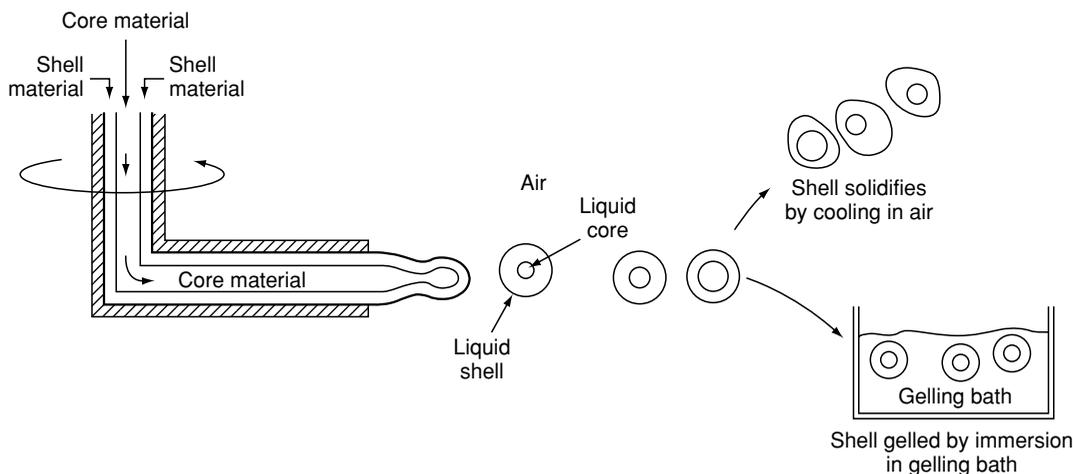


Figure 12 Spinning two-fluid drop-forming device used to produce microcapsules.

Spinning disc processes are claimed to be a fast and efficient way of producing large amounts of capsules economically. Claimed sizes of capsules produced in this manner range from below 100 μm to over 2 mm. Both solid and liquid core formulations have been encapsulated. In the case of solid core formulations, the core material must approach spherical geometry.

Microencapsulated Food Ingredients

A number of food ingredients and additives have been encapsulated and are available commercially. Microencapsulation holds much hidden potential and promise for the food industry in the future.

Solid ingredients encapsulated are typically water-soluble compounds. They are encapsulated with a hydrophobic or hydrophilic coating material usually applied by the Wurster process. Preferred hydrophobic coating materials are partially hydrogenated vegetable oils of varying melting points, monoglycerides, and diglycerides. Hydrogenated vegetable oils used include cottonseed, soybean, and palm. Hydrophilic coating materials tend to be maltodextrins and occasionally gum arabic. Both types of coating materials are well-accepted food-grade products.

Acidulants like citric and lactic acid encapsulated in partially hydrogenated vegetable oil are used in meat processing, where they provide direct acidification and shortened processing times. Sodium acid pyrophosphate encapsulated in hydrogenated vegetable oil is used in frozen cake batters in order to aid mixing and reduce gas release during batter make-up. In both types of applications, release of core material occurs during a heating cycle that melts the shell formulation and releases the core material.

Acidulants like citric, lactic, and fumaric acids encapsulated in a water-soluble maltodextrin shell formulation are used in dry-mix beverages and desserts as well as prepared premixes for the baking and dairy industries. The maltodextrin coating is designed to minimize hygroscopicity, reduce dusting, and minimize reactions with incompatible ingredients. It dissolves in the presence of liquid water to rapidly release the contents of the capsules during a mixing cycle.

Ferrous sulfate, and vitamin C (ascorbic acid) encapsulated in hydrogenated vegetable oil or maltodextrin are used to fortify a nutritional product of some sort. The capsules provide taste-masking, possibly a degree of prolonged release, stabilization of the core material against oxidation, and minimization of reaction with other ingredients in the final product.

Calcium propionate, and sodium bicarbonate encapsulated in hydrogenated vegetable oil are used in chemically leavened products. Release typically

occurs during the baking cycle as a result of melting of the hydrogenated shell material. Sodium bicarbonate encapsulated with maltodextrin is used in dry mix baking and other chemically leavened products. In this case, release occurs during the mixing step. Sodium chloride encapsulated in a hydrogenated vegetable shell is used in various meat products, yeast-containing mixes, and assorted types of dough. The capsules are designed to minimize inhibition of yeast activity, rancidity, and excessive salt binding during product storage.

Liquid food ingredients encapsulated are typically oil-soluble flavors, spices, vitamins, food oils, and fats. These core materials are often encapsulated with a water-soluble shell material applied by spray drying from water, but fat shell formulations are used occasionally. Preferred water-soluble shell materials are gum arabic, modified starch, or blends of these polymers with maltodextrins and sugars. Vitamins are encapsulated with zero bloom strength gelatin by spray drying.

A range of spray-dried flavor-filled capsules primarily with water-soluble shell formulations are used in various dry beverage mixes and other dry food products. Flavors containing ethyl acetate and other low boiling point components have been found to pose problems for successful spray-dry encapsulation. Such components are lost either during the initial emulsification process or during the actual de-watering step as a result of azeotrope formation. Another problem with spray-dry encapsulation is the formation of free surface oil. The rapid desolvation that occurs in the drying chamber can produce blow holes in the capsules, thereby essentially leaving a small amount of flavor oil of free or surface oil that oxidizes on storage and reduces product quality.

Melt extrusion under pressure is an alternate approach to food flavor encapsulation. Rod-like particles loaded with a flavor are often produced in this manner. An advantage of this technology is that it yields essentially defect-free particles with superb shelf-life storage stability. A commercial product (FlavorCell™) has been produced in this manner. Release data have revealed that the FlavorCell™ particles, when placed in ambient-temperature water, release their flavor contents as rapidly as spray-dried capsules that had the same shell formulation. Other approaches to the encapsulation of food flavors include complex coacervation and spray chilling.

Summary

The food industry is currently incorporating a number of different microcapsules into a range of food products. Nevertheless, the volume of capsules

being sold remains small relative to the huge potential that exists. Many groups continue to develop encapsulation technology and microcapsule-based products for the food industry. For this reason, it is anticipated that the volume of microcapsules sold for food use will experience a steady growth for the foreseeable future.

See also: **Chilled Storage:** Principles; **Drying:** Spray Drying

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