



Technical Overview: Microencapsulation

Definition

Microencapsulation may be defined as the process of surrounding or enveloping one substance within another substance on a very small scale, yielding capsules ranging from less than one micron to several hundred microns in size. Microcapsules may be spherically shaped, with a continuous wall surrounding the core, while others are asymmetrically and variably shaped, with a quantity of smaller droplets of core material embedded throughout the microcapsule. All three states of matter (solids, liquids, and gases) may be microencapsulated. This allows liquid and gas phase materials to be handled more easily as solids, and can afford some measure of protection to those handling hazardous materials.

Microencapsulation may be achieved by a myriad of techniques, with several purposes in mind. Substances may be microencapsulated with the intention that the core material be confined within capsule walls for a specific period of time. Alternatively, core materials may be encapsulated so that the core material will be released either gradually through the capsule walls, known as controlled release or diffusion, or when external conditions trigger the capsule walls to rupture, melt, or dissolve.

The substance that is encapsulated may be called the core material, the active ingredient or agent, fill, payload, nucleus, or internal phase. The material encapsulating the core is referred to as the coating, membrane, shell, or wall material. Microcapsules may have one wall or multiple shells arranged in strata of varying thicknesses around the core.

Applications

There are almost limitless applications for microencapsulated material. Microencapsulated materials are utilized in agriculture, pharmaceuticals, foods, cosmetics and fragrances, textiles, paper, paints, coatings and adhesives, printing applications, and many other industries.

Historically, carbonless copy paper was the first marketable product to employ microcapsules. A coating of microencapsulated colorless ink is applied to the top sheet of paper, and a developer is applied to the subsequent sheet. When pressure is applied by writing, the capsules break and the ink reacts with the developer to produce the dark color of the copy.

Today's textile industry makes use of microencapsulated materials to enhance the properties of finished goods. One application increasingly utilized is the incorporation of microencapsulated phase change materials (PCMs). Phase change materials absorb and release heat in response to changes in environmental temperatures. When temperatures rise, the phase change material melts, absorbing

excess heat, and feels cool. Conversely, as temperatures fall, the PCM releases heat as it solidifies, and feels warm. This property of microencapsulated phase change materials can be harnessed to increase the comfort level for users of sports equipment, military gear, bedding, clothing, building materials, and many other consumer products. Microencapsulated PCMs have even been used in NASA-patented thermal protection systems for spacecraft.

Pesticides are encapsulated to be released over time, allowing farmers to apply the pesticides less often rather than requiring very highly concentrated and perhaps toxic initial applications followed by repeated applications to combat the loss of efficacy due to leaching, evaporation, and degradation. Protecting the pesticides from full exposure to the elements lessens the risk to the environment and those that might be exposed to the chemicals and provides a more efficient strategy to pest control.

Ingredients in foods are encapsulated for several reasons. Most flavorings are volatile; therefore encapsulation of these components extends the shelf-life of products by retaining within the food flavors that would otherwise evaporate out and be lost. Some ingredients are encapsulated to mask taste, such as nutrients added to fortify a product without compromising the product's intended taste. Alternatively, flavors are sometimes encapsulated to last longer, as in chewing gum. The amount of encapsulated flavoring required is substantially less than liquid flavoring, as liquid flavoring is lost and not recovered during chewing. Flavorings that are comprised of two reactive components that, when encapsulated individually, may be added to the finished product separately so that they do not react and lose flavor potential prematurely. Some flavorings must also be protected from oxidation or other reactions caused by exposure to light.

Many varieties of both oral and injected pharmaceutical formulations are microencapsulated to release over longer periods of time or at certain locations in the body. Aspirin, for example, can cause peptic ulcers and bleeding if doses are introduced all at once. Therefore aspirin tablets are often produced by compressing quantities of microcapsules that will gradually release the aspirin through their shells, decreasing risk of stomach damage.

Techniques

Microencapsulation processes are usually categorized into two groupings: chemical processes and mechanical or physical processes. These labels can, however, be somewhat misleading, as some processes classified as mechanical might involve or even rely upon a chemical reaction, and some chemical techniques rely solely on physical events. A clearer indication as to which category an encapsulation method belongs is whether or not the capsules are produced in a tank or reactor containing liquid, as in chemical processes, as opposed to mechanical or physical processes, which employ a gas phase as part of the encapsulation and rely chiefly on commercially available devices and equipment to generate microcapsules.

Chemical Methods

Capsules for carbonless paper and for many other applications are produced by a chemical technique called complex coacervation. This method of encapsulation takes advantage of the reaction of aqueous solutions of cationic and anionic polymers such as gelatin and gum arabic. The polymers form a concentrated phase called the complex coacervate. The coacervate exists in equilibrium with a dilute supernatant phase. As water-immiscible core material is introduced into the system, thin films of the

polymer coacervate coat the dispersed droplets of core material. The thin films are then solidified to make the capsules harvestable.

Interfacial polymerization (IFP) is another chemical method of microencapsulation. This technique is characterized by wall formation via the rapid polymerization of monomers at the surface of the droplets or particles of dispersed core material. A multifunctional monomer is dissolved in the core material, and this solution is dispersed in an aqueous phase. A reactant to the monomer is added to the aqueous phase, and polymerization quickly ensues at the surfaces of the core droplets, forming the capsule walls. IFP can be used to prepare bigger microcapsules, but most commercial IFP processes produce smaller capsules in the 20-30 micron diameter range for herbicides and pesticide uses, or even smaller 3-6 micron diameter range for carbonless paper ink.

Polymer-polymer incompatibility, also called phase separation, is generally grouped with other chemical encapsulation techniques, despite the fact that usually no actual chemical reaction is involved in the process. This method utilizes two polymers that are soluble in a common solvent, yet do not mix with one another in the solution. The polymers form two separate phases, one rich in the polymer intended to form the capsule walls, the other rich in the incompatible polymer meant to induce the separation of the two phases. The second polymer is not intended to be part of the finished microcapsule wall, although some may be caught inside the capsule shell and remain as an impurity.

In situ polymerization is a chemical encapsulation technique very similar to interfacial polymerization. The distinguishing characteristic of in situ polymerization is that no reactants are included in the core material. All polymerization occurs in the continuous phase, rather than on both sides of the interface between the continuous phase and the core material, as in IFP. Examples of this method include urea-formaldehyde (UF) and melamine formaldehyde (MF) encapsulation systems.

Centrifugal force processes were developed in the 1940s to encapsulate fish oils and vitamins, protecting them from oxidation. In this method an oil and water emulsion is extruded through small holes in a cup rotating within an oil bath. The aqueous portion of the emulsion is rich in a water-soluble polymer, such as gelatin, that gels when cooled. The resulting droplets are cooled to form gelled polymer-matrix beads containing dispersed droplets of oil that are dried to isolate.

Similar in concept to centrifugal force processes, submerged nozzle processes produce microcapsules when the oil core material is extruded with gelatin through a two-fluid nozzle. The oil droplets are enveloped in gelatin as they are extruded through the nozzle. Then the capsules are cooled to gel the walls, before being collected and dried.

Physical Methods

Spray drying is a mechanical microencapsulation method developed in the 1930s. An emulsion is prepared by dispersing the core material, usually an oil or active ingredient immiscible with water, into a concentrated solution of wall material until the desired size of oil droplets are attained. The resultant emulsion is atomized into a spray of droplets by pumping the slurry through a rotating disc into the heated compartment of a spray drier. There the water portion of the emulsion is evaporated, yielding dried capsules of variable shape containing scattered drops of core material. The capsules are collected through continuous discharge from the spray drying chamber. This method can also be used to dry small microencapsulated materials from an aqueous slurry that are produced by chemical methods.

Fluid bed coating, another mechanical encapsulation method, is restricted to encapsulation of solid core materials, including liquids absorbed into porous solids. This technique is used extensively to encapsulate pharmaceuticals. Solid particles to be encapsulated are suspended on a jet of air and then covered by a spray of liquid coating material. The capsules are then moved to an area where their shells are solidified by cooling or solvent vaporization. The process of suspending, spraying, and cooling is repeated until the capsules' walls are of the desired thickness. This process is known as the Wurster process when the spray nozzle is located at the bottom of the fluidized bed of particles. Both fluidized bed coating and the Wurster process are variations of the pan coating method. In pan coating, solid particles are mixed with a dry coating material and the temperature is raised so that the coating material melts and encloses the core particles, and then is solidified by cooling; or, the coating material can be gradually applied to core particles tumbling in a vessel rather than being wholly mixed with the core particles from the start of encapsulation.

Centrifugal extrusion processes generally produce capsules of a larger size, from 250 microns up to a few millimeters in diameter. The core and the shell materials, which should be immiscible with one another, are pushed through a spinning two-fluid nozzle. This movement forms an unbroken rope which naturally splits into round droplets directly after clearing the nozzle. The continuous walls of these droplets are solidified either by cooling or by a gelling bath, depending on the composition and properties of the coating material.

Another mechanical encapsulation process is rotational suspension separation, or the spinning disk method. The internal phase is dispersed into the liquid wall material and the mixture is advanced onto a turning disk. Droplets of pure shell material are thrown off of the rim of the disk along with discrete particles of core material enclosed in a skin of shell material. After having been solidified by cooling, the microcapsules are collected separately from the particles of shell material.