Flavor Encapsulation Technologies: An Overview Including Recent Developments

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The present overview is intended for the flavor practitioner who seeks an understanding of economically feasible and commercially available flavor encapsulation technologies and the issues involved in using these technologies and related products. Most reviews in the past, with a few notable exceptions, either dealt with encapsulation technologies for the entire food field, or had a strong focus on spray drying.[1,3,4,6,7,8,11,18,19,20,21,22]

It is our view that in spite of the continuing importance of spray drying for flavor encapsulation, various flavor encapsulation technologies are now emerging that will be available for practical food developments in the future. Building on the well-established method of spray drying, an entire bundle of technologies will satisfy the increasingly specialized needs of the market in a toolbox approach.

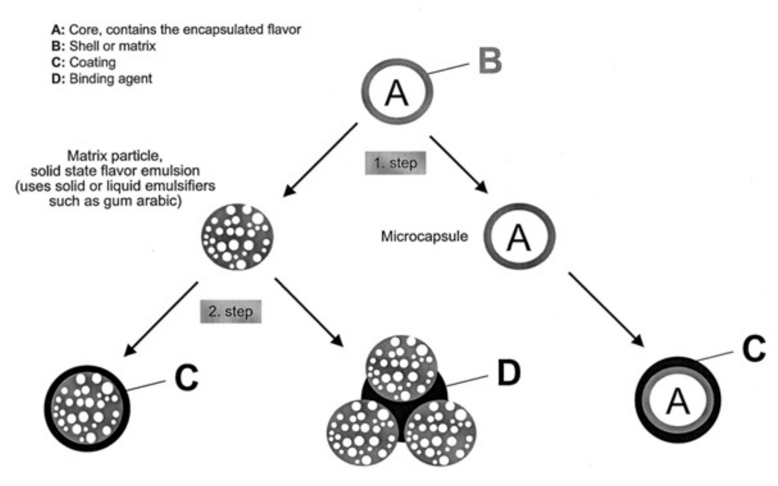
We will initially provide some definitions and point out some important considerations in selecting a flavor encapsulation technology for a given application. Then, we will cover some fundamentals of flavor spray drying because, on the one hand, they provide a conceptual framework for understanding other technologies such as spray granulation, and because on the other hand, improved spray drying technology (e.g. two-stage drying) is now becoming available to satisfy increased product requirements.

The second part of the article covers other encapsulation technologies of increasing importance for food and flavor applications such as extrusion, granulation, submerged nozzle technology, coacervation, molecular inclusion in cyclodextrin and spray chilling.

Modern Encapsulation Technologies Satisfy Numerous Requirements

Today, various encapsulation methods are available to cover different needs and applications. Generally, an encapsulated flavor will consist of a core containing the encapsulated flavor (A) and a shell or matrix (B) (See Figure 1). A fundamental distinction must be made between two categories of products: true microcapsules and matrix particles.

Figure 1. Morphologies of encapsulated flavors



True microcapsules possess a liquid flavor core surrounded by a shell — such as a gelatin capsule. Because microencapsulation is fairly costly, it is only an option in a relatively limited number of applications.

Matrix particles are comprised of ultra-fine flavor droplets (droplet size typically 1 µ to 5 µ) enclosed in a matrix consisting of a wide variety of carriers. Depending on their hydrophilicity, certain flavor ingredients will dissolve in the matrix material, while others form the ultra-fine flavor droplets.

Both microcapsules and matrix particles can be coated with appropriate materials (C) (e.g. to modify release characteristics). Matrix particles can be agglomerated by binding agents (D) (e.g. for instant solubility) (Figure 1).

The reasons for using flavor encapsulation can be manifold:

- Stabilization of labile ingredients
- Liquid flavor in a solid state
- Controlled release
- Improved technological properties
- Improved handling (e.g. dust-free)
- Improved safety (e.g. reduced flammability)
- Creation of visual and textural effects

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Encapsulated flavors possess greater stability and are better protected against external influences, such as oxidation. In addition, they also provide a dry version of liquid flavors. This means they can be easily used in dry products. In addition, specific properties — a controlled water solubility, for example — can be designed into them through selection of the encapsulation technology. Additionally, size, shape and morphology of encapsulated flavor particles can support the perception of aroma, taste, and appearance of food products. And, finally, encapsulated flavors can be designed in a way that there is no dust or odor formed when they are processed. However a flavor's release properties continue to be the key issue in selecting a particular encapsulation technology.

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Appropriate design of flavor release is crucial to ensure full product performance. We speak of solubility-driven release when a flavor capsule is dissolved by water, thus releasing the flavor. The speed at which the capsule dissolves, and thus the speed at which the flavor is released, can be governed through selection of the carrier. On the other hand, it is also possible to design water-insoluble encapsulation systems that keep the flavor encapsulated in aqueous products (e.g. sorbets) until the product is consumed. Temperature-driven flavor release can be achieved, for example, by coating an encapsulated flavor with a specific fat that melts at a specific temperature. A typical application for this would be cake mixes. In other products, such as chewing gum, it is possible to use small gelatin capsules that are mechanically crushed when the consumer chews the product, thus releasing the flavor immediately. In these examples, encapsulated flavors are released in entirely different ways — either during the production process itself, when the food is prepared, or not until the product is eaten.

Hence, encapsulated flavors used in baked goods develop in the oven at temperatures above 70°C, while flavors that employed in teas, soup mixes or candies are not released until the product is consumed. Soup mixes develop some of their flavor — especially the highly volatile elements — after hot water is poured over the mix to produce the soup's characteristic aroma. The situation is similar in tea applications; the flavors develop while the tea is steeping. In the case of chewing gum, the flavor should be released as soon as the gum is chewed, yet it should still be clearly perceivable even after being chewed for 10 to 20 min, meaning that flavor impact and long-lasting effect should be well-balanced.

The application economics are another important concern in selecting an encapsulation technology and can be measured by cost-in-use or, more specifically, by the flavoring cost.[16] Of course, flavor encapsulation always implies added costs compared to the original liquid flavor and thus increased flavoring cost. However, certain applications, such as a flavor burst from chewing gum, only become feasible by using gelatine capsules. Also, cost-in-use has to be considered for the entire product life cycle. Hence, some improvements, such as shelf stability, reduced flavor loss and reduced off-flavors, justify increased flavoring costs.

As in all industrial applications, intellectual property considerations are becoming increasingly important. Depending on the technology, certain encapsulated products are only available from certain suppliers. Also, certain applications of encapsulated flavors may be protected. In order to avoid any confusion, we have not referenced any patents in this review.

This article will take a closer look at several encapsulation processes. The classical encapsulation processes still consist of spray drying, compacting, gelatin encapsulation and agglomeration. Even though these methods continue to represent basic technologies, the limits of their capabilities are reached in connection with certain requirements. This prompted the development of further new encapsulation processes that would satisfy these kinds of more demanding requirements. These encapsulation technologies are frequently adapted from the pharmaceutical industry, which is a very prolific source of ideas.

However, processes from related fields of application first have to be adapted to the special food legislation and technology requirements for flavor encapsulation, which primarily restricts the selection of available shell and carrier materials. These materials have to be taste-neutral and safe for foods. Consequently, a significant part of today's research work focuses on materials selection. A proper selection of the encapsulation material is an important driver for a proper control of flavor release. Depending upon the encapsulation material, the aroma compounds might only be released at a certain temperature, pH or moisture content.

With regard to process technologies, methods based on fluid-bed processes, gelatin encapsulation extrusion, molecular inclusion and spray chilling have gained significance. Technologies, such as liposome encapsulation, alginate encapsulation, co-crystallization and interfacial polymerization, are presently still in the trial stages and will play only a limited role in actual practice for some time to come.

Liposomes are capsules with one or more layers of phospholipids or certain other lipid amphiphiles, with particles in the range of 25 nm to several µm.[13,27] Alginate beads can contain flavor in their gelled alginate matrix. However, their poor diffusion barrier properties severely limit their application in flavor encapsulation.[10] Co-crystallization involves the inclusion of flavor in carbohydrate crystals.[28] Interfacial polymerization is based on a polymerization at the interface between a flavor oil droplet and an aqueuos continuous phase.[17] The lack of food-permissible polymers severely limits applications of this technology in foods, a statement that may have to be revised for neighboring fields, such as cosmetics, household and drugs.

Spray Drying

It is by no means obvious that spray drying is also suitable for encapsulating flavors, because in principle the volatile flavor compounds evaporate faster than water. Hence, it is a matter of finding suitable carriers that will prevent the volatile flavor compounds from being lost during the drying process, yet simultaneously allow water to evaporate unhindered. With increasing knowledge of how these processes occur, suitable materials have been identified that afford these properties yet still assure sufficient encapsulation. Such substances, which also have to have good emulsifying properties and be taste-neutral, include gelatin, modified dairy proteins, modified and/or hydrolyzed starches, maltose or gum arabic.

Essentially, there are two steps involved in the spray drying process. After a suitable carrier has been selected, it is first dissolved in water. Then the liquid flavor is added, homogeneously emulsified and finely dispersed in the spray drier. Various techniques are used to atomize this emulsion in the spray drier, where it is shock dried through contact with hot air at temperatures of 180° to 200°C (Figure 2). As a result of the shock evaporation of the water, the carrier substances tend to form a fine membrane around the flavor droplets. This membrane assures that the remaining water in the enclosed droplet is still able to permeate and evaporate from the drying particle. The large flavor compound molecules, on the other hand, are retained and enriched. After remaining in the drier for a period of up to 30 s, various techniques are used to remove the relatively small particles of carrier and enclosed flavor. The spray-dried finished product has a flavor load of approximately 20 percent.

Spray drying is an extremely cost-effective and widely applicable process; the equipment is mature and has been available for years. Its advantages are: 1. it can largely be employed with existing equipment and at low cost; 2. it assures good results with a wide selection of encapsulation materials; 3. it can be employed for the vast majority of substances that have to be encapsulated.

Compacting and Agglomeration

The processes of compacting and agglomeration are common ways of complementing spray drying (Figure 2).[23] In

Figure 2. Spray drying and related secondary processing technologies (compacting, agglomeration)

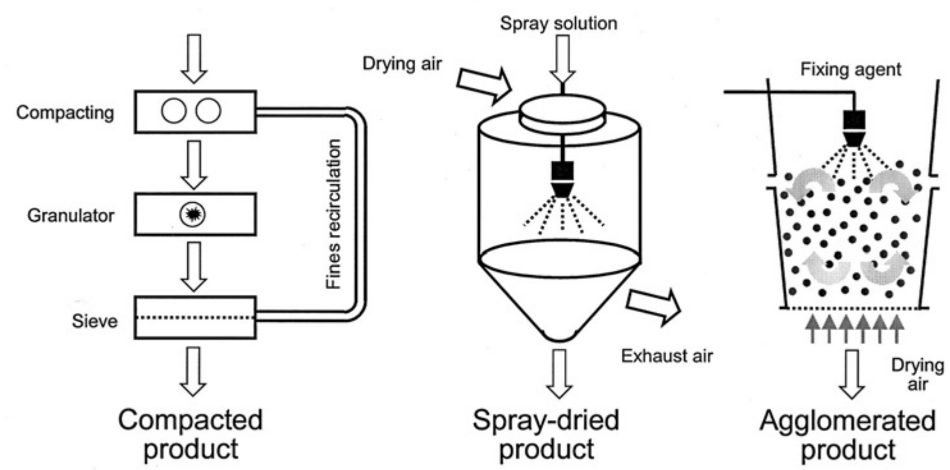


Figure 3. Fluidized spray drying

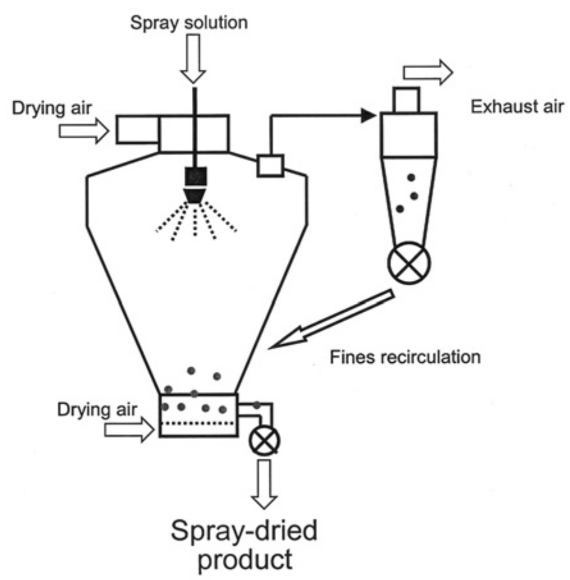
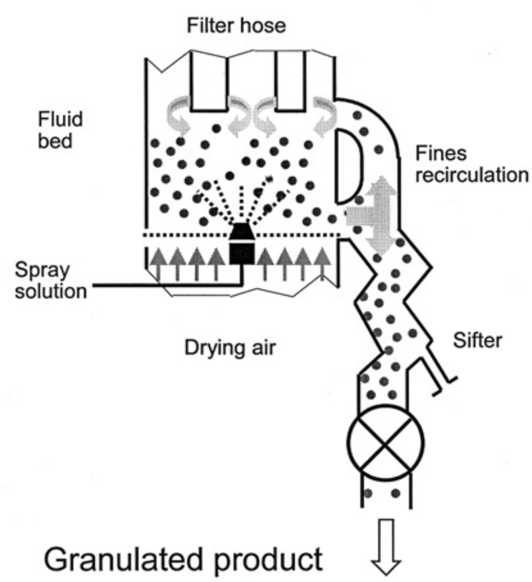


Figure 4. Continuous fluidized bed spray granulation



both processes, the objective is to obtain larger particles. Compacting produces a compressed product with lower porosity ("strength"), while agglomeration produces a loose product with high porosity ("instant properties").

The compacting process: The spray-dried flavors are compressed under high pressure into lumps and then crushed into small pieces ranging in size from 0.7 to 3.0 mm. This process is useful for applications where a grainy structure is required to assure that flavors will not separate (e.g. in tea bags).

The agglomeration process: The preparation of agglomerated products typically starts with spray-dried products. The spray-dried product is fluidized in hot air. The fluidization singles out powder particles and allows them to be sprayed from all sides. By spraying on a binder — such as water — the powder particles gradually stick to one another to form larger particles.

Fluidized Spray Drying

An important characteristic of fluidized spray dryers (i.e. spray dryers with fluid beds integrated into the base of drying chambers) is the ability to produce a free-flowing powder in agglomerated form (Figure 3).[15,24]

In a fluidized spray dryer, drying air enters the drying chamber through a roof-mounted air disperser around the atomizer and leaves through the ceiling of the drying chamber. The spray droplets travel downwards towards to

Figure 5. Rotor granulation

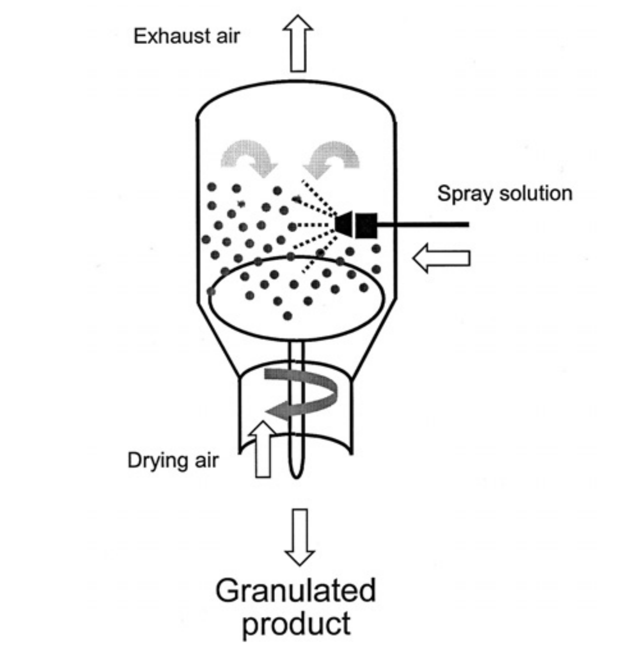
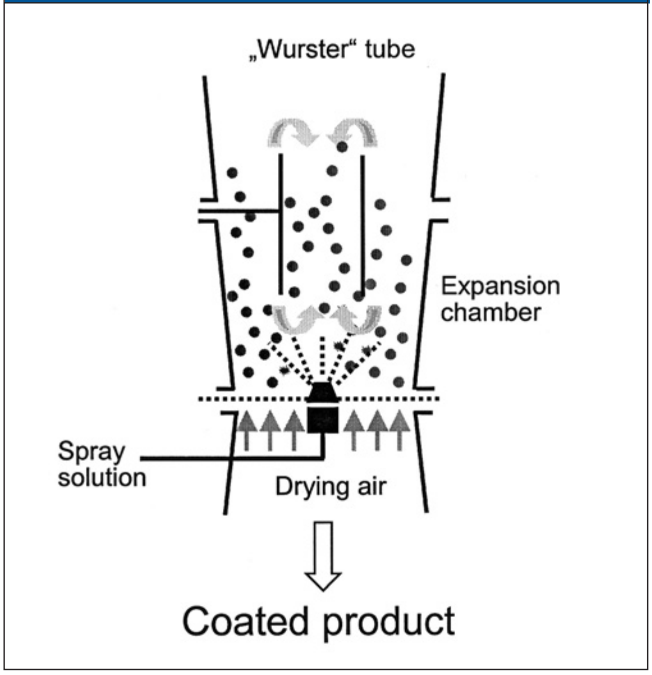


Figure 6. Wurster coating



the fluid bed while the exhaust drying air is led to either cyclones or bag filters. Product recovered from the dry particulate collectors is introduced back into the process.

Fluidized Bed Methods (Spray Granulation and Coating)

As in the case of agglomeration and fluidized bed spray drying, fluidized bed spray granulation occurs in a fluidized bed. However, this technology allows specific particle size distributions (0.2 mm to 1.2 mm) and low porosities to be designed into the product.[26]

Continuous spray granulation — as spray drying — starts off identically, with an aqueous emulsion (Figure 4). The process is primarily characterized by the fact that extremely precise particle size distributions can be achieved. Repeatedly spraying, applying and drying drops in a fluidized bed forms granules with an onion-like structure. A particular advantage offered by granulation technology is the possibility of producing large flavored particles of uniform particle size and shape without the need for any additional production steps.

An alternative batch spray granulation process utilizes the fluidized bed rotor-granulator for the manufacture of spherical flavor granules (Figure 5).[9] In this process, a flavor emulsion is sprayed into a fluidized bed of core material.

Moreover, these technologies permit the coating of particles.[2] By selecting a specific coating material, it is possible to design specific properties of the encapsulated flavors (Figure 6). In addition to coating with sugar-based, i.e. water-soluble, encapsulation materials, it is also possible to employ the same fluidized bed method to provide fatty coatings.

Extrusion

Extrusion processes have gained importance in recent years (Figure 7). Highly viscous carriers can be processed into glassy systems that are characterized by high stability and long shelf life. Water or other plasticizers are added to carbohydrates, which are melted before the liquid flavor is added. The flavored melt is forced through the extruder die hole plate under high pressure; when quickly solidified, the extrudate forms an amorphous, glassy, yet firm mass that completely encloses the flavor droplets, forming small, needle-shaped pellets. This process is especially well suited for encapsulating highly sensitive citrus flavors, and offers the advantage that the products have a long shelf life and especially good protection against oxidation. Typical applications are flavorings for tea, instant drinks and various confectionery products, such as compressed tablets.

Older versions of this technology are based on two stirred tanks, one to form the melt and one to cool and wash the extruded products with an appropriate solvent such as isopropanol.[18] Newer versions of this technology are based on continuous twin-screw extrusion, which allows more processing flexibility.

Coacervation and Submerged Nozzle Process

In the case of coacervation and the submerged nozzle process, defined gelatin capsules enclose a flavor droplet. In coacervation, the materials that will be used to produce the capsules — usually gelatin and gum Arabic — are first dissolved in water (Figure 8).[12] The water-insoluble flavor is then added. By altering temperature or pH, the interfacial surface between the water and flavor droplet forms a thin skin that envelops the flavor droplet. To stabilize the

Figure 7. Extrusion process

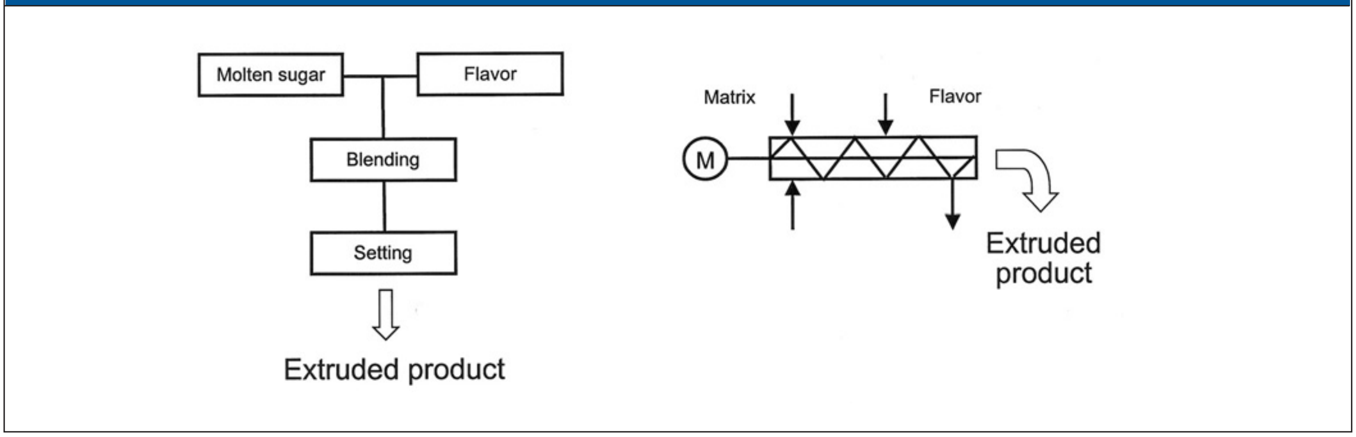


Figure 8. Complex coacervation

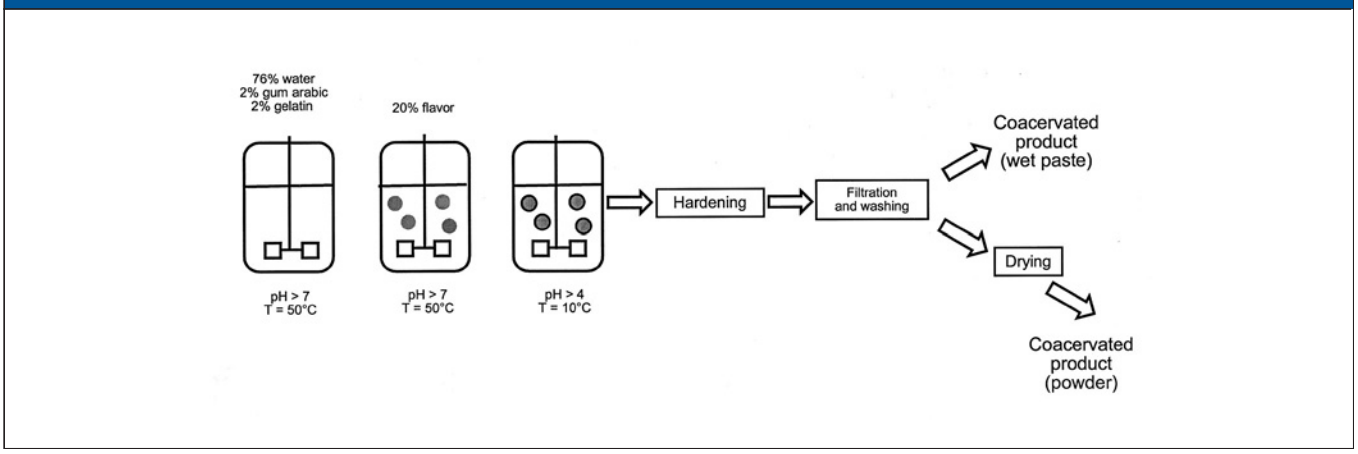


Figure 9. Submerged nozzle encapsulation

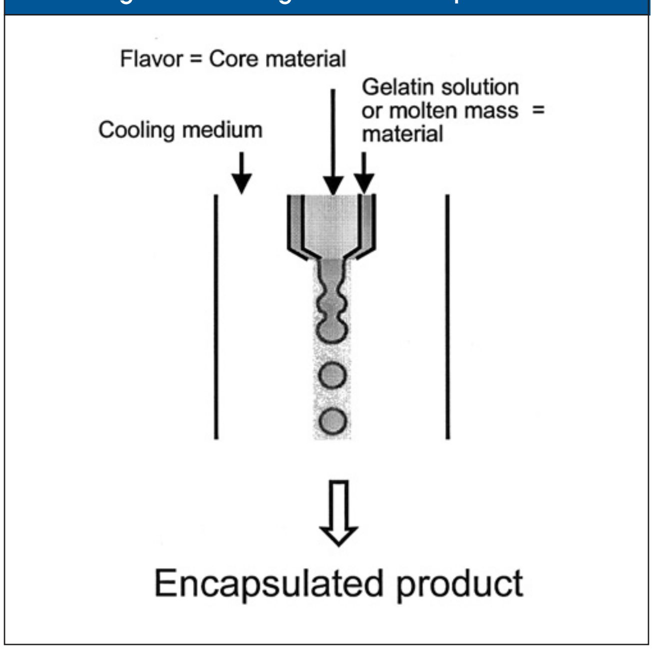


Figure 10. Spray chilling

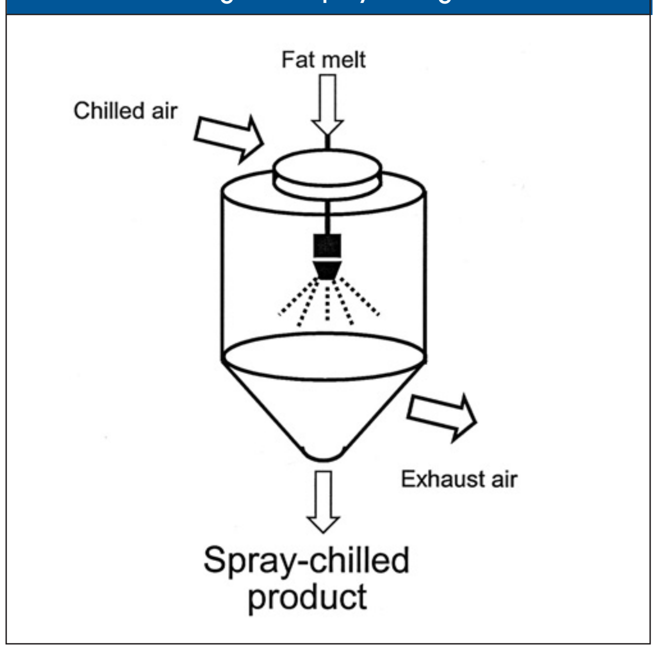


Table I. Overview of flavor encapsulation technologies (typical product characteristics and applications)

Technology	Min. particle size (µm)	Max. particle size (µm)	Typ. flavor load (%)	Max. flavor load (%)	Shell	Morphology	Flavor release	Application
Spray drying	20	100	20%	50%	Maltodextrin, gum arabic, modified starch	Matrix particle	Water soluble	Pharmaceuticals, instant beverages, confectionery, instant desserts.
Compacting	1000	3000	5%	20%	Maltodextrin, gum arabic, modified starch	Matrix particle	Water soluble	Tea, chocolate fillings, pretzels
Agglomeration	500	3000	5%	20%	Maltodextrin, gum arabic, modified starch	Matrix particle	Water soluble	Savory, instant beverages
Fluidized spray drying	200	400	20%	50%	Maltodextrin, gum arabic, modified starch	Matrix particle	Water soluble	Pharmaceuticals, instant beverages, confectionery, instant desserts
Continuous fluidized bed granulation	200	2000	20%	50%	Maltodextrin, gum arabic, modified starch	Matrix particle	Water soluble	Chocolate, instant soups and sauces, instant beverages, pharmaceuticals, tea
Rotor granulation	200	2000	5%	20%	Maltodextrin, gum arabic, modified starch	Matrix particle	Water soluble	Chocolate, instant soups and sauces, instant beverages, pharmaceuticals, tea
Extrusion	200	2000	6%	20%	Maltodextrin, mono- and disaccharides	Matrix particle	Water soluble	Tea, instant beverages, compressed tablets, instant soups and sauces, pharmaceuticals
Coacervation	20	800	40%	50%	Gum arabic, Gelatin, Crosslinking agent	Microcapsule	Water insoluble, mechanical	Chewing gum, toothpaste, instant soups and sauces, spreads, baked goods, cereal extrusion
Submerged nozzle	800	5000	70%	90%	Gelatin, plasticizer	Microcapsule	Water soluble, mechanical	Chewing gum, confectionery, instant soups and sauces, spreads, breathfresheners
Spray chilling	20	200	10%	20%	Hydrogenated or fractionated vegetable oil	Matrix particle	Temperature	Any moist food with heat treatment
Molecular inclusion	5	50	5%	10%	Cyclodextrin	Matrix particle	Moisture	Top note protection

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skin, the gelatin has to be chemically treated to further cross-link and cure it after it has been separated from the surrounding water. For various applications, the resulting pasty capsules then have to be gently finished-dried in a further step.

In the case of the submerged nozzle process, flavors are also enclosed in gelatin capsules — although these capsules are significantly larger compared to coacervation (Figure 9).[14] In this technology, the flavor to be encapsulated and the gelatin are simultaneously forced through a special coextrusion nozzle into a suitable medium, such as vegetable oil, with the gelatin capsule curing and fully surrounding the flavor droplet. This kind of equipment demands the utmost precision and requires constant monitoring of the process steps.

Spray Chilling

In spray chilling, the flavor to be encapsulated is mixed with the carrier and atomized into cooled or chilled air as opposed to heated air as in spray drying (Figure 10).[25] The outer material is usually vegetable oil in the case of spray cooling (45° to 122 °C), or a hydrogenated or fractionated vegetable oil in the case of spray chilling (32° to 42 °C). The flavors are released when the carrier material is molten. Typically applications are foods that undergo a heat treatment such as soups and sauces and deep fried products.

Molecular Inclusion (β-Cyclodextrin)

Molecular inclusion compounds are another possible technique for the encapsulation of flavor substances.[5] β-Cyclodextrin is particularly well suited for this technique (Figure 11). It is a cyclic glucose oligomer and forms inclusion compounds with substances which, in terms of molecular structure, fit into the active center, and which are less polar than water. A typical application is the protection of instable and high added value speciality flavor chemicals. Molecular inclusion can also be used to achieve a long-lasting effect in chewing gum.

Summary

A toolbox approach to flavor encapsulation: Today, the comprehensive technology portfolio of encapsulation technologies enables flavorists to satisfy all relevant product requirements, such as designable properties, easy product handling, improved shelf life and controlled release. Table 1 and Figure 12 provide a summary of these technologies and show typical morphologies. Building on well-established methods of spray drying and compacting, an entire bundle of complementing technologies is now becoming available to satisfy the increasingly specialized needs of the market.

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