Spray Drying

An in-depth look at the steps in spray drying and the different options available to flavorists

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A good percentage of flavors formulated by a flavorist will be encapsulated. While a variety of encapsulation technologies have been developed and are commercially employed, the majority of encapsulated flavors are spray dried.[1–4] **T-1** presents some commercial flavor encapsulation options available to the flavorist along with an estimated degree of industry usage and preferred state for the encapsulated flavor-carrier material. Spray drying remains the predominant encapsulation process because it handles both water-soluble and oil-soluble flavor systems equally well, is cost-effective and easily scaled from pilot plant to commercial production. Over the past 70 years encapsulation of flavors by spray drying has become so routine and standardized that some of the nuances and options available to flavorists may no longer be obvious and thus are worth reviewing.

Factors in Spray Drying

Flavor encapsulation employing the spray drying process involves a large number of interactive factors. These include: the flavor, the flavor cosolvent, the carrier composition, the emulsion solids-to-water ratio, emulsion preparation and lipid particle size distribution, as well as the dryer system and the operational drying conditions. The encapsulation process starts with the formulated flavor (i.e., a compounded flavor, flavor extract, flavor oil or reaction flavor) designed to meet the organoleptic character of the flavor brief. As the flavorist compounds the flavor, the choice of cosolvent will also influence the ultimate quality of the encapsulated flavor. For water-soluble flavors, use of ethanol or propylene glycol will not have as big an effect on the final product relative to the cosolvent selected for oil-soluble flavor systems. Since flavor chemicals vary greatly in water and oil solubility (their partitioning character), volatility and chemical reactivity, the flavor cosolvent and carrier will affect the final sensory character.

The Spray Dry Carrier

The carrier composition is the most critical component in the spray dry encapsulation system. The preferred carrier material should dissolve easily, be water-soluble, bland, inexpensive, have excellent oil-emulsifying and droplet-stabilizing properties, exhibit low solution viscosities at higher dissolved solids levels, have excellent film-forming properties, dry easily and form a glassy amorphous powder upon drying. No single material meets all of these criteria.

Carrier systems are usually formulated by combining a number of water-soluble components. The first of these components are the water-soluble polymers. Two classes of food polymers used almost exclusively are gum arabic and the OSAN-starches. Gum arabics are hydrocolloid gums, obtained from *Acacia* trees of the Leguminosae family, alternately referred to as gum arabic, gum acacia, gum Senegal or Turkey gum. These plant exudates have the advantage of being natural, providing excellent emulsifying and film-forming properties while exhibiting relatively low solution viscosities even at 35–40% dissolved solids in solution. On the negative side, they suffer from large swings in ingredient cost, as well as quality and availability issues. The gum arabic sourced from the sub-Saharan region is also subject to climate change variability and political instability in the harvesting areas. To avoid some of these issues, acacia tree farms have been established and supply significant quantities of gum arabic to the trade.

The alternate food polymers used in spray drying are the octenylsuccinnic acid anhydride (OSAN) modified starches. The precursor starches are derivatized by reaction with octenylsuccinnic anhydride. This process adds octenylsuccinate side groups to the starch polymer chain and the modification generates the surface active properties needed to emulsify and stabilize lipid-flavor droplets. Upon homogenization this polymer will generate and stabilize 0.5–2 micron-sized oil droplets in an emulsion. The advantages of the OSAN-starches include low cost, uninterruptible availability and reduced viscosities at higher concentrations, in addition to good emulsification and lipid-droplet-stabilization properties. All the OSAN carriers are acidic with solution pHs ranging from 3.0 to 5.6, depending upon OSAN-starch type and source. A number of second generation hydrolyzed OSAN-starches are becoming available and are blander and less acidic than the initial generation of dextrinized OSAN-starches.

Other food polymers that have been suggested for use in spray drying include larch gum, modified celluloses, corn fiber gum, milk proteins and, most recently, an OSAN-modified gum arabic.[5] **T-2** lists a number of food polymers used in spray drying, their trade names, suppliers and the pHs of 10% (w/w) solutions.

Commercial encapsulation systems		T-1
Encapsulation system	**Relative contribution (%)[a]**	**Preferred physical state**
Spray drying	~80–85	Glassy solid—fine powder
Lipid–spray chilling	5–10	β-Polymorph—fine particles
Melt extrusion	2–3	Glassy solid—particulates
Lipid–flakes	1	β-Polymorph—flakes
Melt injection/solvent cooling	2	Glassy solid—threads
β-Cyclodextrin molecular complex	1	Crystalline complex
Complex coacervation	< 1	Cross-linked polymer membrane surrounding flavor droplet
Co-crystallization	< 1	Crystalline sucrose with occluded flavor

[a]personal communication–F. Paulicka

Polymer solution pH's		T-2
Trade name/source	**Polymer type**	**pH of 10% solution**
Emulgum [a]	gum arabic	4.88
Capsule [b]	OSAN-starch	3.10
Capsul TA [b]	OSAN-starch	3.26
Miracap [c]	OSAN-starch	3.07
Emcap 12634 [d]	OSAN-starch	4.80
Hicap 100 [b]	OSAN-starch	4.00
Emcap 12639 [d]	OSAN-starch	5.64
Emser 555 [e]	sodium caseinate	6.51

[a]Colloides Naturels; [b]National Starch; [c]Tate & Lyle; [d]Cargill; [e]Main Street Ingredients

Food polymers are not generally used as a single carrier component but are blended with maltodextrins, corn syrup solids and sugars. This approach reduces cost-in-use while improving carrier functionality. Maltodextrin provides a low-cost, bland-food polymer component, and corn syrup solids, or mono and disaccharides, improve the glassy state character by the mechanism of molecular packing in the dried matrix (see below).

Flavor Emulsion

Efficient formation of the flavor emulsion starts with dispersion of the carrier solids in water. The water source should be devoid of contaminates, such as pro-oxidant ions (Fe^{+++} or Cu^{++}) and chemical odors. A deionized water source would be ideal. Alternately, a metal-complexing agent such as disodium EDTA may be added to the water preceding emulsion preparation. The carrier solids must be fully hydrated and dissolved before the flavor component is added and dispersed. High shear mixing is generally employed to disperse flavor oil droplets followed by homogenization using a two-stage homogenizer. With nozzle dryers the homogenization step can also be performed by passage through the dryer nozzle. In either case, a uniform emulsion is prepared and the feed stream metered into the dryer chamber. The ratio of carrier and flavor to aqueous component is balanced to reach a compromise between a maximum practical emulsion viscosity and the reduced water levels leading to improved drying efficiencies.

As noted in T-2, several of the OSAN-starches have pHs of approximately 3 in solution. This pH range can cause acid-catalyzed reactions with specific acid-sensitive flavor compounds such as citral or acetal. Also, this low pH becomes more of a concern with commercial production where emulsions must be prepared and held for extended periods of time or held at high temperatures in the plant feed tank.

The Dryer and the Drying Process

A general schematic of a spray dryer is shown in **F-1**. While the flavorist generally does not get involved in the spray dryer systems used in the preparation of flavor samples, those interested in the number and types of drying systems can consult the text by Masters.[6] The spray drying process can be broken down into a number of operational steps. These include preparation of aqueous carrier phase; emulsification or solubilization of the flavor; delivery of the emulsion/flavor as a fine particle stream into the dryer; and drying under controlled rates and drying temperatures to obtain the product—a dried encapsulated flavor particulate.

Referring to F-1, the flavor emulsion is prepared by first dissolving the carrier in the mixing tank [1]. The flavor is added and the emulsion generated by passage of the flavor through a homogenizing unit [2], which is then fed into a surge tank [3]. This flavor emulsion is metered into the drying chamber [10] that has a preheated hot air stream, set at a predetermined temperature, within the 160–220°C range. The liquid flavor-carrier emulsion droplets are dispersed by the centrifugal head atomizer (or nozzle atomizer) into the heated air within the chamber plenum at a specific rate. The droplets are almost instantaneously heated to 100°C. Each liquid droplet rapidly gives up moisture at the air-liquid interface forming a film on the outer droplet surface. Water molecules rapidly diffuse out of the droplet, increasing the solids content of the particle as it dries. Evaporative cooling protects the flavor and the increasing solids retain the entrapped aromatic flavor molecules. By balancing the air-outlet temperature with air-inlet temperature by the liquid feed rates, the particles can be held at 100°C—the wet bulb temperature—during the full drying process. Ultimately, the fully dried particles are swept out of the chamber into the cyclone [11] and collected in the receiving chamber [12]. The recovered encapsulated flavor-carrier matrix will be an amorphous solid and based upon the carrier composition and drying dynamics; the solid should be in the desired glassy state. These particulates are usually 10–120 microns in size and resemble collapsed soccer balls when viewed under a scanning electron microscope (SEM).

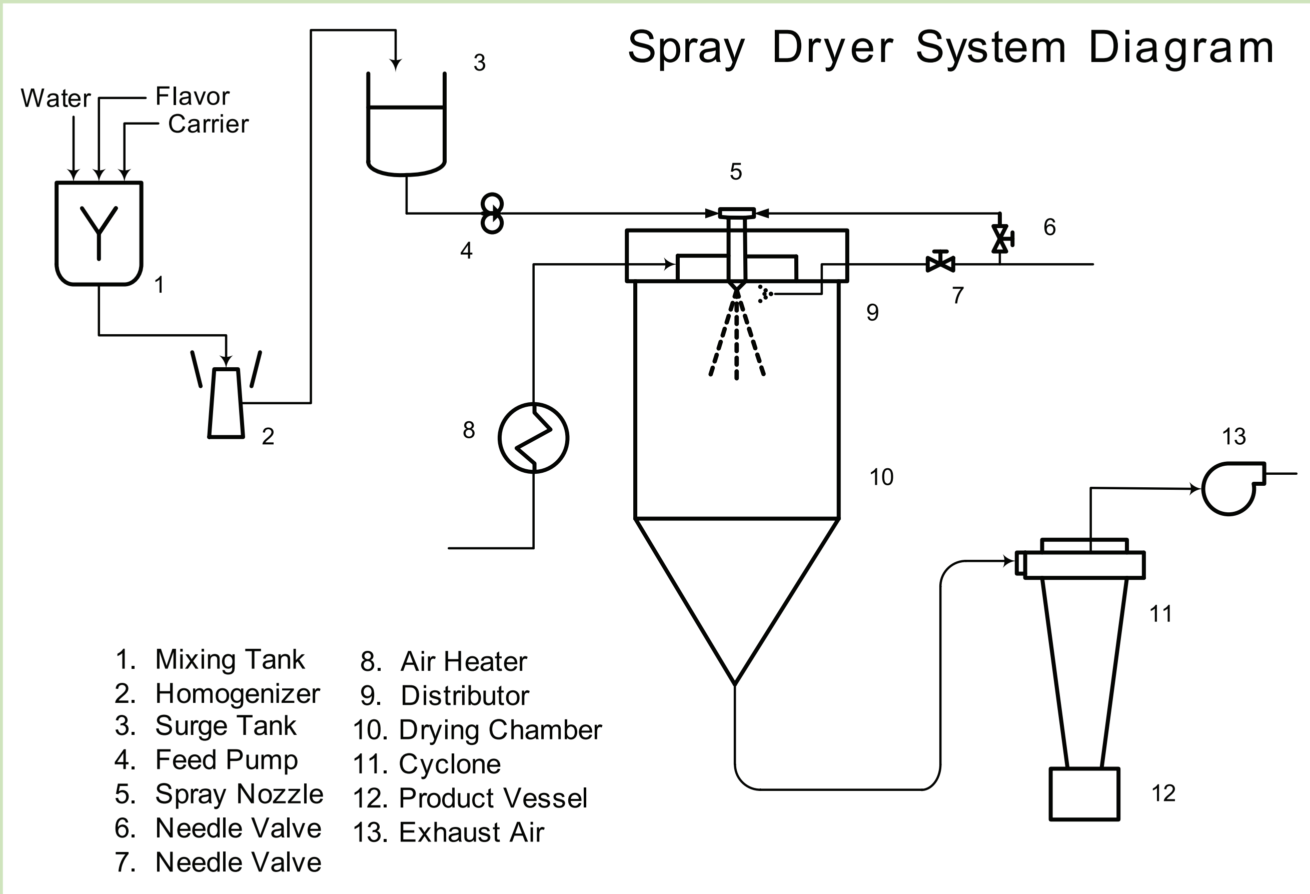
In some flavor houses, the flavorist will compound a flavor, choosing the appropriate cosolvent and specifying the carrier composition in the formula system. In larger flavor houses, the flavorist may only be required to compound the flavor and supply the formula to the process staff. The choice of the carrier will be made from a carrier library developed and optimized for specific flavor systems. Still in some flavor houses, multiple forms of the spray dried encapsulated flavor utilizing a number of carriers may be prepared and returned to the flavorist for sensory evaluation and the resulting best formula identified.

The Glassy State and Encapsulated Flavor

Whenever a water-soluble flavor carrier system is spray dried, the result is an amorphous powder. The amorphous powder will be in either the glassy state or the rubbery state, the former being preferred (T-1). The glassy state is more effective in preventing diffusion and loss of water-soluble flavor actives than the rubbery amorphous state. More details regarding the glassy state in food systems can be found in a number of articles.[7–9]

As a general rule the mixture of a food polymer—an intermediate weight carbohydrate, such as maltodextrin or corn syrup solids, and the mono and disaccharides—gives an optimum glass transition character (ΔCp) and a higher glass transition temperature (Tg) at lower final moisture levels. It is worth noting that when an oil-soluble flavor is encapsulated within a glassy matrix, the lipid droplets are dispersed within the solid matrix. These lipid (solvent-flavor) droplets containing the flavor actives are still able to undergo normal liquid phase chemistries (i.e., cyclizations, inter-esterifications, isomerizations, ketal or acetal formation in the presence of alcohols or polyols, condensation reactions, etc.).[10] In essence, the droplets can act as microreactors. The significant advantage of a glassy matrix is in prevention of oxygen permeation into the lipid, which could initiate terpene oxidation or other oxidative flavor-degrading reactions.

A general schematic of a spray dryer **F-1**



Flavor Carrier Systems

A number of patents have been issued describing spray dry flavor encapsulation systems. In US Patent 3,971,852, Brenner describes a polymer—low molecular weight plasticizer carrier composition for encapsulation of large ratios of lipid (flavor) relative to the carrier.[11] With a sophisticated understanding and control of the dynamics of drying, Brenner designed a carrier matrix that remained fully plasticized during the full drying range. In this way, the partially dried particle does not crack or collapse during drying while retaining the increased load of lipid (flavor) phase. Saleeb and Pickup disclosed in US Patent 4,532,145 a spray drying carrier for the encapsulation of acetaldehyde.[12] This carrier is composed of a high molecular weight component (molecular weight range of 1000–6000 Daltons) and a lower molecular weight component (molecular weight range of 90–500 Daltons) with the specification that the carrier be composed of 70–90% of higher molecular weight component and 10–30% of lower molecular weight agent. However, no disclosure on the extended retention stability of the encapsulated acetaldehyde was supplied.

In US Patent 5,124,162, Boskovic et al. disclosed a carrier system designed for citrus based oils.[13] Here, the dried particle was characterized as a dense product with at least 0.5 g/cc bulk density, exhibiting < 20% voids and existing in the glassy state. His carrier was based upon a composition of 10–35% food polymer (gum arabic, gum acacia, OSAN-starch), 25–50% film-forming carbohydrate (maltodextrins) and 22–45% mono- and disaccharides where the mono/disaccharide is primarily maltose. The glassy state particle's physical character was designed to protect citrus oils from oxygen perfusing into the matrix and initiating oxidative reactions. Levine and Slade employed similar carrier formulas to improve stability of spray dried flavors in a double encapsulation process as disclosed in US Patent 5,087,461.[14] The flavor was spray dried using a carrier composition consisting of 40–80% OSAN-starch, 10–40% maltodextrin, 5–20% corn syrup solids or polydextrose and 5–20 % mono- or disaccharide. The spray dried encapsulated flavor was then re-encapsulated in a carbohydrate carrier by melt extrusion.

Alternate Systems

For water-soluble flavor extracts the spray dry carrier does not require a surface active polymer. A simpler (and less expensive) carrier based upon maltodextrin, corn syrup solids and mono/disaccharides can be formulated. When employed with simple water-alcohol-based extracts, such as vanilla extracts, this carrier yields a clean-tasting material. Even with such a simple system the physical chemistry of the flavor components should be considered. For example, it is well-known that crystalline vanillin has significant vapor pressure as a solid at ambient conditions. Sublimation of vanillin is one process that can affect the impact of the vanillin character over time. The encapsulated vanillin present in a folded vanilla extract may still sublime over time in its final amorphous state. A practical approach when spray drying a folded vanilla extract would be to assay the initial vanillin level of the extract, the resulting level in the spray dried material and the stability level of the vanillin in the stored material.

Volatile aromatic chemicals such as acetaldehyde, ethanol, diacetyl and dimethylsulfide are very sensitive to the spray drying process. Compounded fruit and allium flavors containing significant levels of acetaldehyde as a "fresh" top note will lose significant levels of this key compound upon drying. One approach in compounding with these volatiles employs the use of increased levels of the agent to act as overages to account for drying losses. This approach is in essence "formulating to the dryer." Using an iterative process, the unbalanced flavor becomes more balanced with the proper formula adjustments until the volatile loss results in a final level to supply the desired organoleptic character in the spray dried flavor.

A more efficient approach might be considered. This approach is based on the concept of encapsulating a single flavor chemical and then post-blending the encapsulated active into the remaining spray dried flavor. The carrier for the single volatile agent would be selected for optimized load retention and stability. For example, a top-noted fruit flavor needs to be designed and encapsulated for a dry drink mix. If the flavor was originally formulated at 10% by weight acetaldehyde in the compounded flavor actives, and the flavor fraction is set at 20% flavor in the spray dry product, then the acetaldehyde component would be at a 2% level assuming no loss in the encapsulation process. If the resulting spray dried flavor was to be formulated (arbitrarily) at 5% in the drink mix, then a final acetaldehyde level would be estimated at 0.1%. Knowing this, the flavorist or the encapsulation specialist, could prepare the drink mix by first having the acetaldehyde at much lower concentrations spray dried using a re-optimized carrier and process conditions. The initial acetaldehyde level would be lowered to approximately 2% for safety issues. After drying and recovery of the acetaldehyde, addition of 5 parts by weight encapsulated acetaldehyde with approximately 4.9 parts final spray dried flavor is formulated to yield the drink mix blend. These parallel spray drying steps will add additional process costs but should result in a balanced and more stable flavored drink mix.

Other strategies may be used in spray drying flavor systems to enhance flavor quality and delivery. Dairy-based flavors could be encapsulated utilizing sodium caseinate, nonfat dry milk, whey protein or blends of these ingredients with lactose. This system would yield a flavor-compatible carrier system, which would encapsulate the dairy oils/flavors as well as generate a glassy matrix. See Rothenberg in US Patent 5,601,760 for a specific teaching on the use of milk protein carriers and spray drying.[15]

The constraints involved in the spray dry formularies preclude a simple route to controlled release functionality in the encapsulated flavor. This limitation derives from the fact that water-soluble carriers used in spray dried encapsulates easily release the flavor upon rehydration. There are alternate methods to overcome these restraints. However, combining control-release functionality into a spray dry carrier elevates the technology into the field of control release. For this objective, alternate routes may be designed such as a double encapsulation process or a phase irreversible process. For the former, the spray-dried flavor is the starting point for additional processes, such as agglomeration followed by lipid coating or melt encapsulation, as noted in US Patent 5,087,461.[14] A reverse encapsulation spray dry process is another option. Here, a complex coacervate of an oil-based flavor might initially be prepared. Following cross-linking of the protein-hydrocolloid membrane, additional carbohydrate carriers would be added to the original reaction solution and the total polydispersed mixture spray dried.

Another proposed approach for thermal protection of a spray dried flavor would utilize a phase irreversible process. This system employs a protein that denatures at or below the approximate 100°C droplet temperature reached during the drying process. A lipid-stabilizing, water-soluble protein with thermo-gelling or thermal-denaturing properties solubilizes in the original aqueous carrier, then the emulsion formed and the system spray dried. Assuming the protein denatures and still effectively encapsulates the flavor droplets during denaturing transitions, the protein would remain in the dried product as a barrier to provide functionality.

These and a number of potential encapsulation options can be explored if the flavorist and encapsulation specialist leverage their understanding of the physical chemistry of flavor-carrier-drying interactions and dynamics. However, for routine spray drying of most flavors, understanding the principals of the spray drying encapsulation process and utilizing selected carriers and process conditions will ensure a flavor can be delivered in a quality manner.

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