Spray Drying of Food Flavors I. Theory of Flavor Retention

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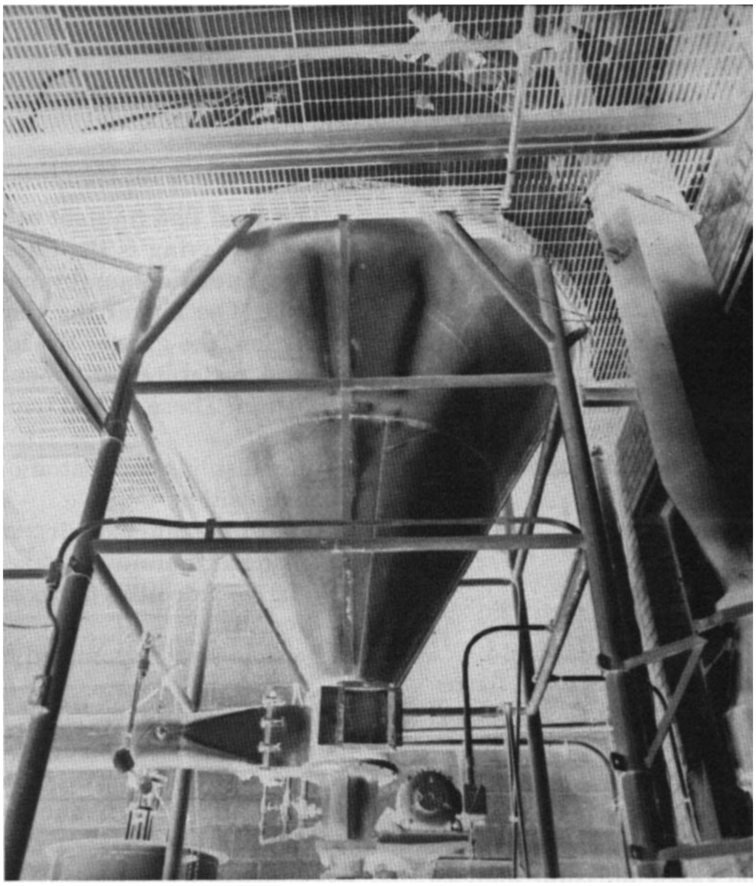
The retention of substantial quantities of food flavors during spray drying is rather unexpected. Flavors are composed of a very broad group of organic volatiles with a boiling point range of at least −80°C (H_2S) to 280°C (vanillin). While one may expect to retain volatiles with boiling points in excess of that of water, one would not expect to retain the low boilers during the drying process. One may also expect to lose a substantial proportion of the higher boilers since these high boilers may actually become more volatile than water when placed in an aqueous system for spray drying. The water tends to force the hydrophobic flavor constituents to the air/water interface and out of solu-



Spray dryer photo courtesy of Fritzsche Dodge & Olcott

tion. Therefore, flavors which have a high boiling point when pure may become relatively more volatile than water when in aqueous solution.

A question then arises why more volatile flavors are retained during spray drying but the less volatile water is nearly completely evaporated. This phenomenon is explained by the fact that during the drying process a film of high solids material forms on the surface of the drying droplet. This partially dried or high solids film is quite permeable to water molecules which are a solvent for the solids being dried and are quite small in size compared to most flavor molecules. Since the flavor molecules have little solubility in the typical flavor carrier and are substantially larger than water molecules, they will not readily diffuse through this high solids surface film and are trapped within the drying droplet. Therefore, water molecules continue to be lost from the drying droplet while flavor components are retained.

The critical moisture content of this surface film necessary for it to become selectively permeable has been considered in terms of percent moisture, water activity (% relative humidity ÷ 100) and diffusion coefficient ratios (aroma to water). In terms of diffusion coefficient ratios, the critical moisture content is that point in the drying process where Da ÷ Dw (Diffusion coefficient of aroma ÷ Diffusion coefficient of water) is less than 0.01.[1] Below Da/Dw = 0.01, the particle surface is considered to be permeable to water only. This selective diffusity is a property common to all noncrystalline hydrophilic organic systems that include nearly all foods and encapsulating agents used in spray drying. Selective loss from the drying droplet may also be considered to occur at a water activity of <0.90[2] or a moisture content in the range of 7-20%.[1] Between 40-100% moisture, diffusion coefficients (water vs. aroma) vary by less than a factor of 10. However, below 40% moisture, diffusion coefficients for volatile organics decrease very rapidly while water diffusion decreases only slowly.

The factors which determine flavor retention during spray drying are those which determine flavor loss until a semipermeable membrane forms around the dryir,' droplet. This includes the time necessary to form a semipermeable membrane and the molecular diameter and vapor pressure of the flavor compounds.

Flavor Compound Vapor Pressure

Compound vapor pressure would have an influence on the amount of a flavor component lost until a semipermeable membrane was formed. The driving force for evaporation is the difference in vapor pressure of a component in solution versus vapor pressure in the air above the solution. Compounds which exhibit high vapor pressure in solution would be lost to a greater degree than those which exhibit lower vapor pressures. Vapor pressure of a flavor compound being spray dried would depend upon several factors. These factors include

- absolute vapor pressure
- solubility in the aqueous phase
- presence of a nonaqueous phase giving higher solubility of the flavor compound
- possible interactions or binding to the flavor carrier material

It has been previously discussed that compounds insoluble in water may exhibit vapor pressures in excess of their pure vapor pressure. The magnitude of this vapor pressure increase, when in aqueous solution, depends upon compound structure and functional groups. One would expect greater hydrophobicity to result in greater vapor pressure increases in aqueous solution. Solubility in the aqueous phase or a second phase such as lipid or water insoluble flavor solvents would be expected to decrease volatility. Binding to the flavor carrier would greatly decrease volatility.

There is ample data in the literature to show binding of organic volatiles to carbohydrates (e.g., starches) and proteins (e.g., soy flour). A concern would be whether this binding was too strong to preclude sensory detection. Flavor retention via irreversible binding to the flavor carrier would be of no benefit. However, weak bonds would be desirable since a decrease in vapor pressure would occur and promote retention during the spray drying process.

The Role of Molecular Diameter

The role of flavor compound molecular diameter in determining flavor retention is apparent. The larger the flavor compound, the more slowly it diffuses to the drying interface. Larger organic molecules would also be affected earlier by the semipermeable effects of the drying particle surface than small molecular weight molecules. Therefore, it is expected that molecular diameter is an important parameter determining flavor retention during spray drying.

Time Variables for Membrane Formation

The final determinant of flavor retention during spray drying is the time necessary to form a semipermeable membrane around the drying droplet. The longer the time necessary for film formation, the greater the loss of volatile flavors. Several variables influence time for membrane formation. These would include

- infeed solids concentration, temperature and composition
- dryer inlet and exit air temperatures
- drying air humidity
- relative velocities of drying air and drying droplets
- particle size of atomized material

The operating variable shown to have the greatest effect on flavor retention during spray drying is **infeed solids concentration.** Several studies have shown minimal flavor loss when infeed solids are 50% or greater.[3-5] This is as expected, since a high starting solids content requires minimal drying to provide a semipermeable surface film. High solids contents also result in more viscous infeed materials. High viscosities tend to limit convection currents within the drying droplet and would slow volatile diffusion to the drying surface. One would ordinarily use the highest infeed solids content that is still possible to pump and atomize.

The influence of **infeed temperature** on flavor retention has opposing effects. A low infeed temperature has been shown to produce better flavored coffee via spray drying. The lower temperature increased infeed viscosity which improved flavor retention by reducing circulation currents within the drying droplets and producing a larger particle size distribution.[5] Assuming constant solids infeed, flavor may be improved by using a low temperature infeed.

Increasing infeed temperature permits one to use higher infeed solids. Since infeed solids are a primary determinant of flavor retention, the higher solids levels made possible by higher infeed temperatures results in improved flavor retention. Therefore, one should use as high an infeed temperature as permitted (and, therefore, higher solids) without volatizing the flavor or causing heat damage to the product.

Infeed composition can also strongly influence flavor retention during spray drying. The flavor encapsulant* must be an adequate film former to produce good flavor retention. Materials which form crystals rather than amorophous structures are not suitable. The encapsulating material should also be soluble in water, promote emulsion stability and permit the use of high infeed solids levels (viscosity considerations).[6,7] Flavor carriers typically used in the flavor industry are either gum arabic or a modified starch.

One would expect an optimum **inlet air temperature** for flavor retention. As inlet air temperature increases, the rate of film formation increases (water loss rate increases). The faster this film forms, the more flavors are retained. This relationship would hold true until inlet air temperature was sufficiently high to cause "ballooning" of the drying droplet. At some inlet air temperature, the drying droplet will attain a temperature sufficiently high to cause internal steam formation. The steam will expand the droplet resulting in a hollow particle with reduced flavor retention. Flavor retention is less in ballooned powder, since the ballooning results in a large surface area per unit weight of powder.

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*Since flavor encapsulant choice is the subject of a later paper in this series, discussion will be postponed until then.

The critical temperature for ballooning to occur is dependent upon several parameters. Infeed composition is one of these parameters. Infeed materials which quickly transport water to the drying surface or remain relatively porous during drying are less subject to ballooning. Flavor composition is an additional factor. Flavors composed of large proportions of volatile solvents or components will also promote ballooning. Artificial flavors using alcohol as the major flavor solvent, for example, promote ballooning. The optimum inlet temperature must be determined for each given infeed material. As a starting point, Reineccius and Coulter found ballooning occurring at an inlet air temperature of 450°F for a skim milk infeed (30% T.S.).[3]

Flavor retention increases as **exit air temperature** increases. Exit air temperature is a function of both inlet air temperature and rate of water evaporation. Achieving a high exit air temperature via reduced water evaporation results in more flavorable flavor retention. This is the result of rapid film formation because of low humidity in the drying air. Exit air temperature choice is again a compromise. While higher exit air temperatures improve flavor retention, this comes at a cost in production capacity. High exit air temperature may also contribute to flavor problems via thermally induced chemical changes. Therefore, one has to consider production capacity, flavor retention, and off-flavor generation when exit air temperature is chosen.

The operating parameters of **drying air humidity and relative particle/air velocities** are seldom varied during spray drying. While inlet air humidity may be controlled via dehumidification devices on the air intake, operating costs generally prohibit their use in the spray drying of food flavors. Relative air/particle velocity is typically optimized via dryer design and not changed to a significant extent. Increased relative velocity and decreased inlet air humidity both result in more rapid film formation and, therefore, improved flavor retention.

The role of **particle size** in determining flavor retention is unclear. The majority of research indicates that larger particles retain more flavor.[5,8] However, Reineccius and Coulter found no relationship between particle size and flavor retention.[3] This conclusion was based on evaluating flavor retention in a model system by analyzing different sieve fractions. That is, a dryer run was made and the powder was sieved to obtain powder fractions of different particle sizes.

No differences in flavor concentration could be found between the small particles and large particles. They postulated that while larger particles have less surface area/unit volume, which would result in better flavor retention, it also takes longer for a selective membrane to form around the larger drying droplets. The longer drying time apparently offsets the benefits of reduced surface to volume ratio.

Conclusion

Research to date suggests that optimum flavor retention may be obtained by using as high infeed solids as possible, raising infeed temperature if this permits higher infeed solids levels, choosing a carrier which favors flavor retention and operating the dryer at optimum inlet and exit air temperatures. These suggestions are based on research which has generally dealt with a study of a limited number of volatiles and flavor carriers seldom or never used in the flavor industry.

A host of unique problems occur when one considers the encapsulation of artificial flavors and citrus oils. These problems include a question of the effect of emulsion size going into the dryer, carrier choice (gum arabic vs. modified starches), choice of atomization technique (pressure spray vs. centrifugal wheel), optimum inlet and exit air temperatures for each carrier, flavor solvent (alcohol, benzyl alcohol, triacetin, triethyl citrate, vegetable oil, etc.), infeed temperature and concentration. The effect of these parameters on overall flavor retention, retention of low vs. high molecular weight flavor compounds (flavor balance) and flavor stability during storage (citrus oils) all need study. These problems will be addressed in a continuing series of research articles to be published in this journal.

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