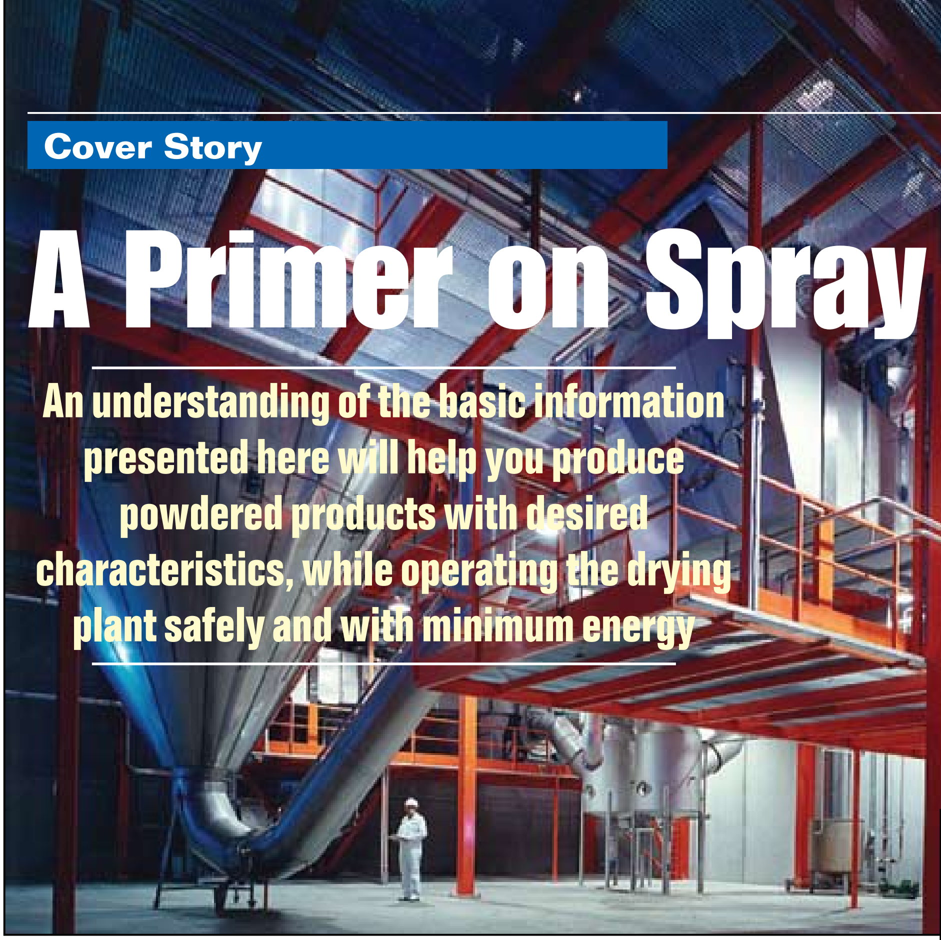
Cover Story

A Primer on Spray Drying

An understanding of the basic information presented here will help you produce powdered products with desired characteristics, while operating the drying plant safely and with minimum energy



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This photo shows a spray dryer with a heat recuperator. The plant has an evaporative capacity of 1,825 kg/h

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Spray drying is one of the major industrial drying technologies. It is applied by many industries because of its ability to convert a liquid product into a dried powder in a lenient single step and because it allows you to control temperature and the particle formation process very accurately. Altering the process parameters allows you to produce complex powders that meet exact powder properties in terms of particle size and shape, bulk density, dispersibility, polymorphism, flow properties and so on, in a very efficient manner. Spray drying is applied in the production of an endless number of products in the chemical process industries (CPI) ranging from advanced chemical compounds to bulk chemicals. Spray drying plants can be designed for almost any capacity from very small quantities up to several metric tons (m.t.) per hour.

Historical development

Spray drying of liquid products commenced at the end of 19th century with the first patents issued for drying of egg products. In the 1920s, the commercial use of spray drying increased with the major breakthrough being for production of milk powder and detergents. The milk powder production was a major step forward in a period when refrigerators were not that widespread and the shelf life of milk consequently was very low.

In the pioneer years of spray drying, the emphasis was simply put on removing the water without too much heat distortion and thereby obtaining a dry powder with good keeping properties. Spray drying proved to be an outstanding technology for this as the drying process is almost instantaneous. With the spray of liquid having a very large surface, heat transfer and mass transport are very rapid, and the solid product is protected against thermal overload by the evaporation of the water.

Since the early years, spray drying technology has developed tremendously and some of the major achievements have been to divide the drying process into several stages — reflecting that the conversion of the liquid product into the final dry powder usually takes place in two steps (Figure 1). In the first step — referred to as the constant rate period — drying is controlled at the surface of the liquid droplets, that is, heat transfer through the gas phase to the droplet surface and mass transport of water vapor from the droplet surface into the gas phase. In the second step, a solid particle has been formed and the evaporation rate is then controlled by diffusion of moisture inside the particle towards the particle surface. Multistage drying takes advantage of the above knowledge by adding one or more fluidized-bed drying stages where the residence time is higher and the applied drying media temperatures are lower. The overall drying process is thus divided into a very rapid evaporation of surface moisture in the spray chamber part and an accurately controlled drying of the internal particle moisture in the fluidized bed.

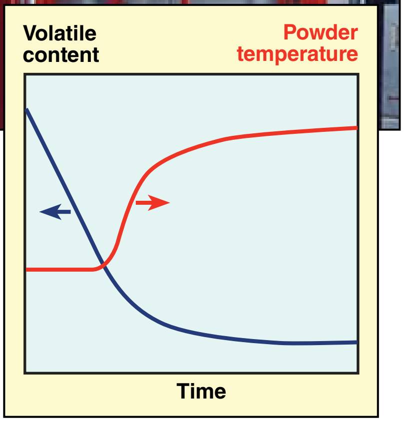

FIGURE 1. This product drying curve shows that two stages typically occur: a constant rate period followed by diffusion-controlled period

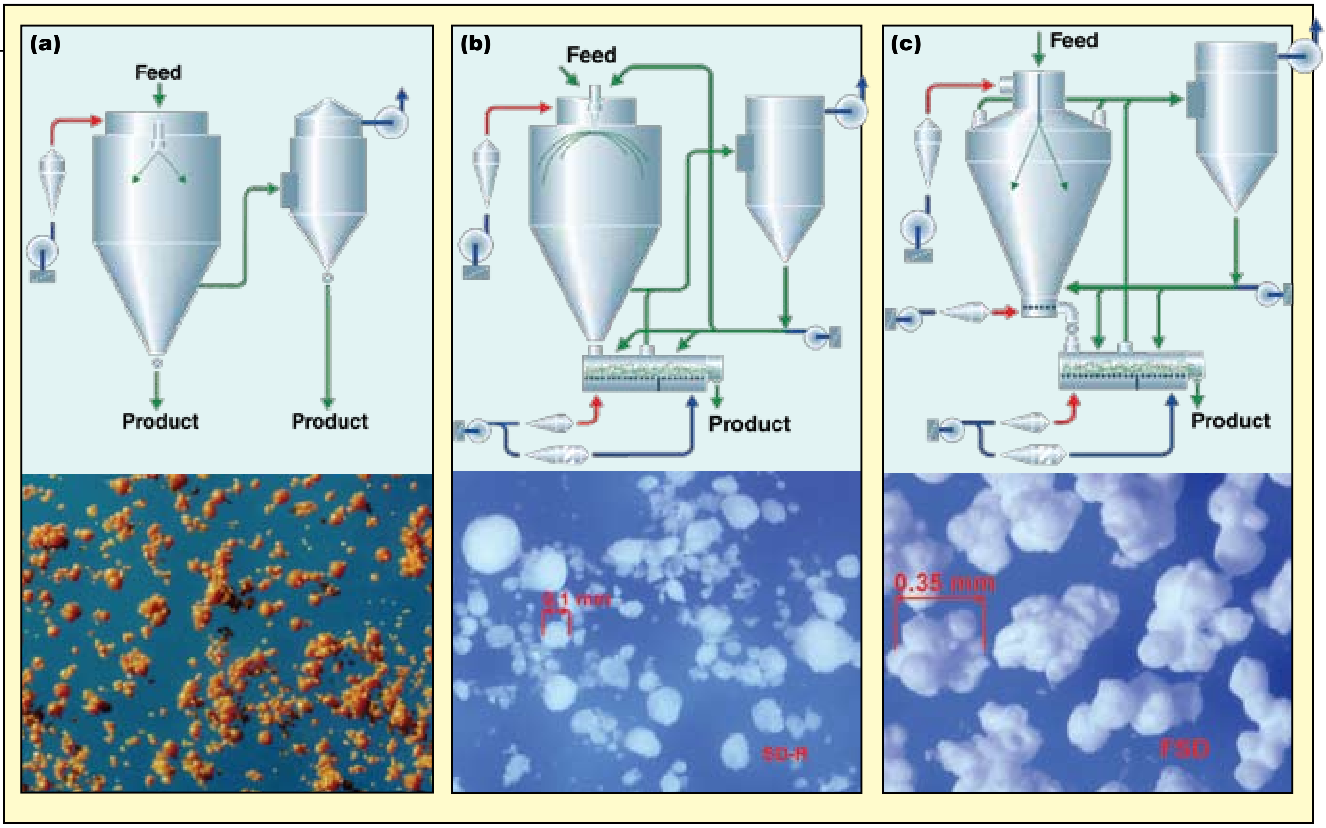

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FIGURE 2. In the simplest configuration (a), a spray drying system consists of the dryer and a cyclone for product recovery. Accounting for the multi-stage drying process, systems can also incorporate an external vibrating fluidized bed (b) and an integrated fluidized bed (c). Typical products from these configurations are shown below

The first spray dryers built according to the multistage principle were made with a separate, vibrating fluidized bed of rectangular shape (Figure 2b), which sometimes caused problems when the moist solid was difficult to fluidize at the entrance. This problem was overcome with the introduction of an integrated fluidized bed mounted directly at the conical bottom of the spray dryer (Figure 2c). The integrated fluidized bed is — contrary to the external fluidized bed — working in back-mix mode (mixing finished and moist powders) in order that the average powder moisture in the integrated fluidized bed is sufficiently low to ensure a satisfactory fluidization.

With the use of integrated fluidized beds, further developments were made to improve the quality of the dried powders. By reintroducing the fine powder fraction to the atomization zone and by using the fluidized bed to classify the powder, powders with less dust, improved dispersibility and a narrower particle-size distribution can be produced.

Applications of spray drying

The principle of drying a spray of liquids has found many uses beyond the mere removal of water. Spray drying is also applied in formulating products with unique properties. In the aroma industry, water-insoluble liquid aromas are encapsulated in a solid matrix of water-soluble carrier material and surface active ingredients. After spray drying, the result is a powdery flavor with excellent shelf life and good redispersibility in water. The same is the case for oil-soluble vitamin powders.

Very fine powders, such as ceramics or hard metals, can — by the addition of binding agents — be formulated into larger compact particles of spherical shape with good flowability. Being very uniform and with a consistent density, they can be used directly in pressing dies for forming ceramic products, cutting and mining tools and other products. Within dyestuffs and pesticides, the non-soluble active material can be formulated with binding and dispersing agents to produce a non-dusting and water-dispersible powder.

Coating of suspended solids by spray drying the suspension is used for taste masking and controlled release of active materials in the pharmaceutical industry.

The spray drying process can also be applied for congealing. In this case, a melted feedstock is atomized and turned into a free-flowing powder by cooling it in a stream of cold air or gas. It finds use for several types of products — from palm oil derivatives to special waxes, fats, glycerides, hydrates and other inorganic or organic melts. Spray congealing is also applied for encapsulation. If a potent or otherwise harmful chemical is suspended in a molten wax, it can be encapsulated and the user is protected from the malicious effects. Many enzymes for the detergent industry are congealed this way.

Spray drying is also applied for production of e-PVC (emulsion polyvinyl chloride) and PVAc (polyvinyl acetate), where formulations have been developed so the liquid feed can be spray dried to produce high quality powders.

The spray drying process can be applied for carrying out chemical reactions. Dry absorption of SO_2 from fluegases from coal-fired power plants, and HCl and HF from waste incineration plants are some examples. The reaction takes place when the atomized liquid is suspended in the drying air/gas stream.

Spray drying is used for bioactive products. In this case, the gentle process dries the product without destroying the bioactive elements. It is also applied in solid dosage pharma-

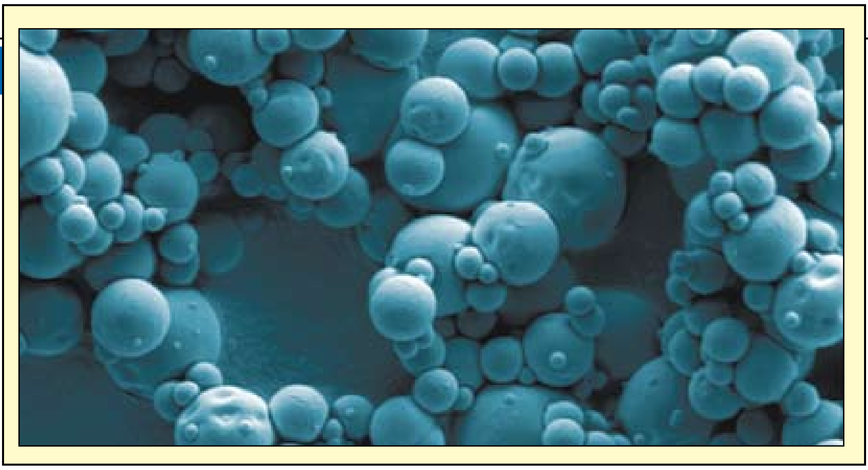

FIGURE 3. APIs in an amorphous structure often have better bioavailability. Stable structures can be made by spray drying the API with an excipient

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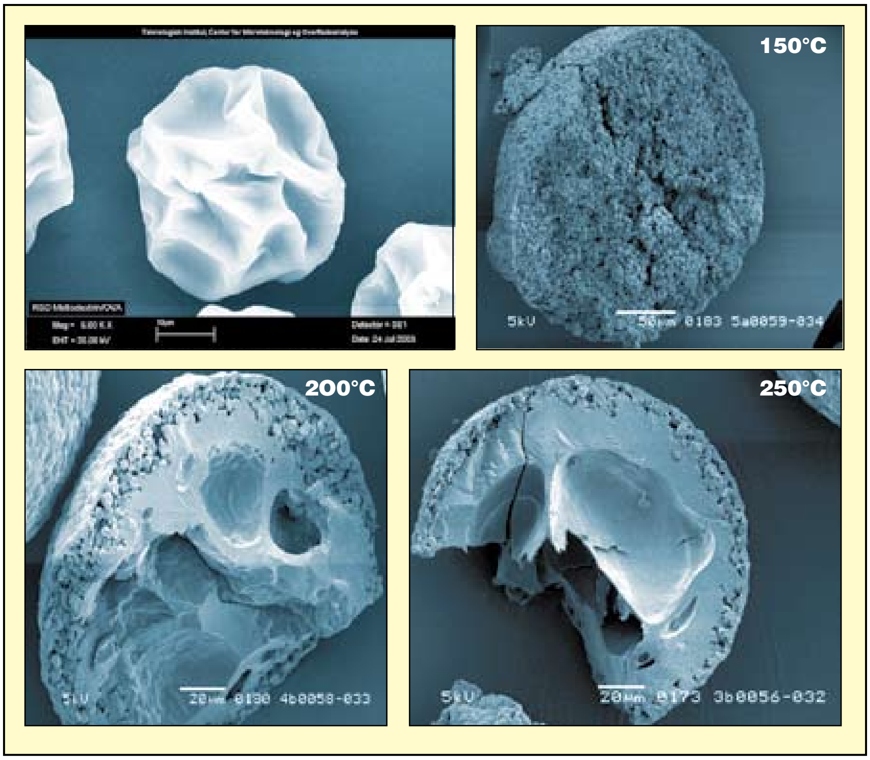

FIGURE 4. Shrinking can be observed (upper left) due to water evaporation from a particle. The other three images show the effect of increasing air drying temperature, which influences the rate of water evaporation, for the same product

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ceuticals where spray drying can be applied to increase the bioavailability of the drug. Active pharmaceutical ingredients (APIs) in an amorphous structure often have a better bioavailability (Figure 3). Stable structures containing amorphous materials can be made by spray drying the API with an excipient.

Structure and morphology

One of the major benefits of spray drying is that it allows for production of precisely defined powders. The basis can be almost any pumpable solution, suspension or emulsion with a wide range of rheological properties. Depending on the characteristic of the liquid feed, the atomization technology, plant geometry and process parameters, particles of different sizes, shapes and porosities can be produced.

The size of the particle produced from the liquid droplet depends on the solids content in the liquid feed, inlet air temperature and the plasticity of the moist solid phase.

Often, the particles' shrinking due to water evaporation can be seen directly on the surface; other products form a rigid shell at the droplet surface and leave a hollow interior part when the remaining water evaporates. As the inlet air temperature determines the rate of water evaporation after atomization, it will often influence the ability of the particles to shrink and thereby the porosity of the particles. In extreme cases, the particles may break down due to very high internal vapor pressures (Figure 4).

Powder flowability and dispersibility can be greatly improved by agglomerating several fine particles into larger clusters of porous structure. Due to the large quantity of capillaries, the particles will have improved wettability. Agglomerated powders will furthermore be less dusty and therefore more environmentally friendly.

The basics of spray drying

The essential in spray drying is the atomization of the liquid feed and the distribution of the drying media allowing the liquid to evaporate and particles to form. The dried particles are continuously discharged from the drying chamber and recovered from the drying media using a cyclone or a bag filter. The spent drying media is often treated in a scrubber to meet environmental requirements before being exhausted to the atmosphere. It can also be recirculated. The whole process generally takes no more than a few seconds.

Atomization. Several types of atomization can be employed in a spray drying system, including centrifugal, nozzle, pneumatic and sonic atomization. The average droplet size and distribution is fairly constant for a given method of atomization, but the average particle size can be in the range of 10–300 microns. Atomizing 1 L of feed generates a total surface area of 20–600 m^2.

The droplet size from a given type of atomization device depends on the energy spent for breaking down the liquid into fragments, that is, increasing the overall surface of the liquid. For most atomization systems, the liquid does not leave the atomizing head as a droplet, but as a fragment of a thin liquid film. The droplet formation takes place immediately after the liquid has left the atomizing head due to the surface tension of the liquid. The formation of a perfect droplet is therefore very dependent on the rheological properties of the liquid and the inter-

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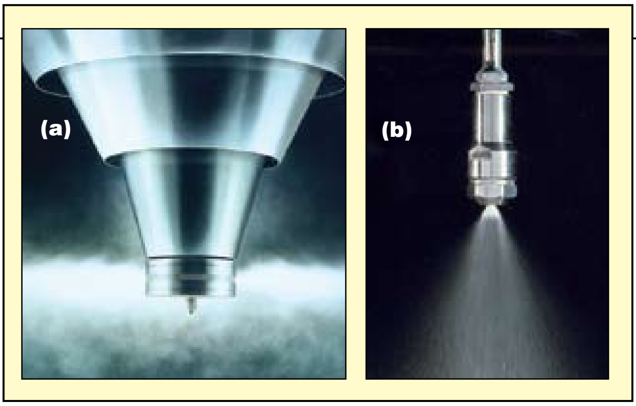

FIGURE 5. Rotary atomizers (a) produce a liquid mist horizontally from the atomizer wheel. Atomization by nozzle (b) often leads to a narrower particle-size distribution

action with the hot drying medium just outside the atomizing device.

Centrifugal (or rotary) atomization is the most common form of atomization. Here, a rotating disc or wheel breaks the liquid stream into droplets (Figure 5a). The devices normally operate in the range of 5,000 to 25,000 rpm. Discs or wheels typically have a diameter of 5 to 50 cm. The size of the droplets produced is nearly inversely proportional to the peripheral speed of the wheel.

Rotary atomization produces a liquid mist horizontally from the atomizer wheel. The spray cloud leaving the atomizer wheel will be distributed over an angle of 180 deg., and therefore the drying chamber is often designed with a height-to-diameter ratio close to 1:1. Due to the limited impact of the liquid flow on the particle size, it is possible to operate the rotary atomizer with a large turndown in feed capacity keeping the particle size within the specifications. The use of variable speed drives makes the control of droplet size — and therefore particle size — very easy.

Rotary atomizers are available in many sizes. A small air-driven laboratory unit handles from 1–10 kg/h of liquid feed, while the largest commercial units driven by 1,000 kW motors can handle in excess of 200 m.t./h.

With pressure-nozzle atomization (Figure 5b), the liquid is pressurized by a pump and forced through the orifice of a nozzle to break it into fine droplets. The orifice size is usually in the range of 0.5 to 3.0 mm. This limits the capacity of a nozzle to approximately 750–1,000 kg/h of feed, depending also on pressure, viscosity and the solids content of the feed. The size of the droplet depends on the size of the orifice and the pressure drop. A larger pressure drop across the orifice produces smaller droplets. Therefore, to reduce the particle size for a given feedrate (capacity), a smaller orifice and a higher pump pressure must be provided to achieve the same mass flow through the nozzle. Large systems may have as many as 40 nozzles, making control of particle size difficult. Although the pressure nozzle is very simple, maintenance — especially of multiple nozzle systems — can become troublesome as wear of the insert changes the characteristics of a given nozzle. The potential for plugging the relatively small orifices is another drawback for nozzle-based atomization systems.

Pressure nozzles usually give a narrower particle-size distribution, and the spray angle, and pattern can be adjusted by varying nozzle inserts and position in the drying chamber.

Two-fluid pneumatic atomization is primarily used in smaller drying systems. The atomization is accomplished by the interaction of the feed with a second fluid — usually compressed air. Neither the feed nor the air requires very high pressure (typically in the range of 200 to 350 kPa). Particle size is controlled by varying the ratio of the compressed-air flow to that of the feed. As the two-fluid nozzles have rather large openings for the feed, the risk of clogging is reduced, which makes this nozzle ideal for use in pilot- or laboratory-scale equipment. Both nozzle types' spray patterns (angle and flight paths of the droplets) can be altered by different nozzle types and internals.

Sonic atomization has been tested in small capacity dryers, but has not been applied for larger production units thus far. Ultrasonic energy is used by passing the liquid over a surface vibrated at ultrasonic frequencies. These systems are suitable for producing very fine droplets at low flowrates. A very uniform particle-size distribution is furthermore achievable.

Dryer configuration. Proper size and geometry of the spray drying chamber and the gas disperser are essential for the optimum particle formation as the flow patterns of the droplets and the gas through the dryer must provide for sufficient contact time to allow evaporation of essentially all of the liquid. As a result, atomizers are usually installed at the center of the roof of a relatively large diameter spray dryer. The heated gas is introduced through a roof-mounted air/gas disperser around the atomizer, creating a co-current flow of gas and droplets/particles. This takes advantage of evaporative cooling and decreasing temperatures downwards.

With atomization by pressure nozzles, a spray drying chamber with extended height — for some products up to 20 m or more — is required for the particles to obtain sufficient retention time in the chamber. These types of spray dryers — also referred to as "nozzle towers" — are often used in production of coarse powders like foodstuffs, dyes, pesticides and other heat-sensitive products.

The larger the particle size desired in the final powder, the larger the diameter of the drying chamber, regardless of the unit's total throughput. When coarse powders are needed in small production rates, a pressure nozzle spray in fountain configuration (for example, spraying upwards from the bottom part of the chamber) is often found to be more practical. The spray travels upward until overcome by gravity and the downward flow of air. It then reverses direction and falls, finally landing in the bottom cone of the drying chamber. The major drawback in fountain nozzle drying can be that drying actually begins in a cooler part of the dryer and continues into the hottest zone. Since each droplet is already partly dried, the evaporative cooling effect is lessened and the chance of thermal degradation becomes larger. Lower inlet temperatures can solve this problem, but also reduces the total evaporation capacity.

Collecting dried powder. After drying, the particles must be separated from the drying media, which is cooled due to the evaporation of the liquid from the droplets. This colder and humid gas is discharged from the dryer after separation of the now dry particles. Due to the fact that the gas has some entrained powder, cyclones or fabric filters are used to clean the gas. In some cases, the combination of cyclones followed by a wet scrubber proves more effective.

Coarse powders are most easily collected directly from the bottom of the drying chamber. In this arrangement, the spent drying gas exits through an outlet duct in the center of the cone. The reversing of the gas flow allows the majority of the powder to settle in the cone and slide to the bottom outlet, which is often equipped with an airlock for discharge. If the powder is very fine, a small amount is collected from the drying chamber. In this case, the cyclones or the bag filter become the primary collection point. To eliminate chamber collection, a U-bend is used at the outlet for both gas and powder from the chamber to the downstream collectors.

Process gas flow. The flow of drying gas through the system is much the same as for any gas-suspension drying system. Heating by direct combustion of natural gas is the most efficient — backed up by fuel oil or propane combustion when gas curtailment is possible. If indirect heating is required, shell-and-tube or finned-tube heat exchangers are used with steam or a heat transfer fluid as heating source. Electric heaters are used in smaller spray dryers.

The design of the gas disperser is of ultimate importance for the proper function of a spray dryer. Today, gas dispersers are often configured by means of computational fluid dynamics (CFD) analysis to define air flow pattern and temperature distribution within the drying chamber. Different types of gas dispersers are often used with different atomization technologies and chamber geometries. For example, with a rotary atomizer a gas disperser with air rotation is often preferred, whereas a more streamlined air distribution is applied in nozzle towers.

For most applications, the gas disperser is constructed with adjustable guide vanes allowing for fine tuning during plant commissioning.

Industrial radial fans are used to move the gas through the system, employing a combination of forced and induced draft or induced draft only. If ambient air is the drying gas and a very clean process is required, high-efficiency, particulate-matter air filters are applied. In some cases, additional measures are required in order to protect the environment and eliminate emissions completely. This can be achieved by working in a closed loop system or by adding HEPA filters to clean the exhaust air. Some products may contain powerful odor components that have to be removed. This can be done either by thermal or catalytic incineration, carbon black absorption, chemical scrubbing or bio filtration.

Ductwork with appropriate dampers, expansion joints, vibration isolators and noise abatement devices is supplied with most dryers. All equipment is usually insulated and cladded to minimize heat loss and condensation, and personnel hazards.

Process design and control. The evaporation rate in a spray dryer is directly proportional to the product of the temperature difference from inlet to outlet and the mass flow of gas through the system. Outlet temperature is established by the desired moisture content in the product according to that product's equilibrium isotherm. Since true equilibrium is never reached, the actual values are usually determined experimentally. Inlet temperature is also determined by experience and should be as high as possible without product degradation. Then, for a given evaporation rate, the required process gas flow can be determined from the temperature difference. All system components can be sized based on gas flow. A gas residence time must be selected from experience based on the particle size desired and the product's known drying characteristics. This permits direct calculation of a chamber volume.


Flamex Inc.

FIGURE 6. By the very nature of the spray drying process — fine dust suspended in air — there is a risk of fire and dust explosions. Over-pressure venting, explosion suppression and inerting the complete plant are ways to prevent accidents and to make any incident proceed in a controlled manner to minimize damages

As mentioned above, spray drying is still largely based on empirical data, and industrial-scale drying tests are required for determining the process parameters and plant design that will result in the desired product — unless, of course, experience and data is already available from dryers in production. Optimization of the performance of existing installations can also be carried out by testing in smaller units.

Once designed and built, a spray drying system needs fairly simple controls. As the performance of a spray dryer is very dependent on the air velocities and flow pattern inside the drying chamber, it is common practice to operate the dryer at a fixed air flowrate. Since outlet temperature determines the moisture content in the final product, the temperature must be controlled and modulated with respect to other changes in the system. Depending on the mode of operation, the outlet air temperature is either controlled via the amount of feed conveyed to the dryer or by adjusting the temperature of the inlet gas.

Pressure drops across filters and cyclones are usually monitored to assure that the system is operating properly. The pressure in the drying chamber is usually controlled by the suction fan and kept at slight vacuum in order to avoid dust escaping the equipment. Rotary atomizers require monitoring lube-oil flow, temperature and vibration, whereas nozzle atomization systems require monitoring feed pressure or flow.

The level of automatic control of the

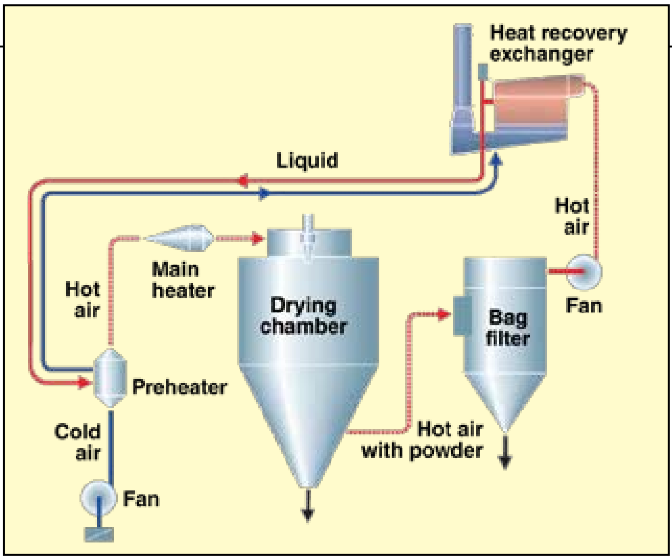

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FIGURE 7. This spray drying system is equipped with a heat recovery unit

plant can be varied from a start/stop command, and afterwards the programmable logic controller (PLC) is programmed to undertake all startup and shutdown routines and all operation parameters set via a predefined recipe to almost manual control. Trend analysis of the plant's operation facilitates troubleshooting and quality control.

Safety & environmental issues

By the nature of the spray drying process — a fine dust suspended in air — there is a risk of fire and dust explosion (Figure 6). This risk needs to be considered very carefully and for this, characteristics of the powder need to be established. The most important parameters to be determined are the following:

- Dust explosion pressure rise, *Kst*
- Maximum dust explosion pressure, P_{max}
- Minimum ignition energy, MIE
- Minimum ignition temperature, MIT
- Minimum auto-ignition temperature, MAIT

MAIT is of particular interest as most fires and dust explosions in spray dryers are initiated by product deposits starting an exothermic reaction. Based on the product data, a risk analysis of the entire spray drying plant has to be carried out identifying all possible ignition sources and drying parameters, and possible protection of the plant must be defined. (For more on preventing dust explosions, see *CE*, October, pp. 49–51.)

In Europe, this risk analysis should be carried out according to the ATEX directives, whereas NFPA (National Fire Protection Assn.) will provide guidance for plants in the U.S. With the safety regulations in place, explosions are very rare. For protection of the spray dryers, overpressure venting is widespread, and guidance is established both in Europe and the U.S. for sizing in relation to the chamber volume and the powder characteristics.

Explosion suppression is also used. This system is often the only realistic way of protection if the powder is harmful and an escape of the product in the event of an explosion could be critical for the environment. Containment, that is, designing the plant to resist the maximum explosion pressure, is only an option for small scale plants.

For products where the minimum ignition energy is very low, the likelihood of an explosion can become so large that it is preferable to "inert" the entire plant. In this case, the plant is operating in a closed loop with a condenser for removal of evaporated liquid, and the inerting gas can either be taken from an external source or be produced by a direct, gas-fired air heater for the dryer (the self-inerting principle). In cases where organic solvents are evaporated, the drying gas will always have to be inert and supplied from an external source.

Energy conservation

The spray drying process is rather energy intensive, and consequently, an effort must be made to optimize the plant in order to reduce the energy consumption per kilogram dry material. The first parameter to consider in this context is the concentration of the feed. Increasing the solids content in the feed with just a few percentages can reduce the specific energy consumption per kilogram dry material by 10–20%.

The drying process efficiency, η, is often defined as:

$$\eta = (T_{in} - T_{out})/(T_{in} - T_{amb}) \qquad (1)$$

Where T_{in} is the inlet air temperature, T_{out} the outlet air temperature and T_{amb} the ambient temperature.

From Equation (1), it appears that the higher the inlet temperature and the lower the outlet air temperature, the better the efficiency. In practice, this means that one should strive to operate at the highest possible inlet air temperature without deteriorating the product and the lowest possible outlet air temperature that can result in acceptable powder moisture. By performing the drying process in multiple stages, the outlet air temperature from the spray dryer can be reduced significantly whereby the overall energy consumption will be lowered.

Heat recovery (Figure 7) by preheating the incoming fresh air by means of the outgoing hot air — or excess heat from another process — is a viable way of saving energy. Efficient heat recovery planning and design has proved to save as much as 20% of the energy for heating the drying media. Heat recovery can be either by direct injection into the drying gas stream or using a heat exchanger. Most systems include heat exchangers with a heat transfer fluid (water) in order to avoid complicated large air ducting within the plant. Finned-tube or plate-type heat exchangers are used depending on the dust content in the hot drying media.

Generally, it is not possible to exploit the latent heat from the dryer because the dew point of the outgoing air is rather low (40–50°C). For products that are not very heat sensitive, a partial recirculation of the warm drying air offers a cost effective and simple way of heat recovery. The dew point of the outlet air will, in this case, increase significantly and hot water in large quantities may be produced utilizing the latent heat in this instance.

Novelties in spray drying

A major development in spray drying technology has been the ability to make feasibility tests on just a few droplets of feed material (Figure 8).

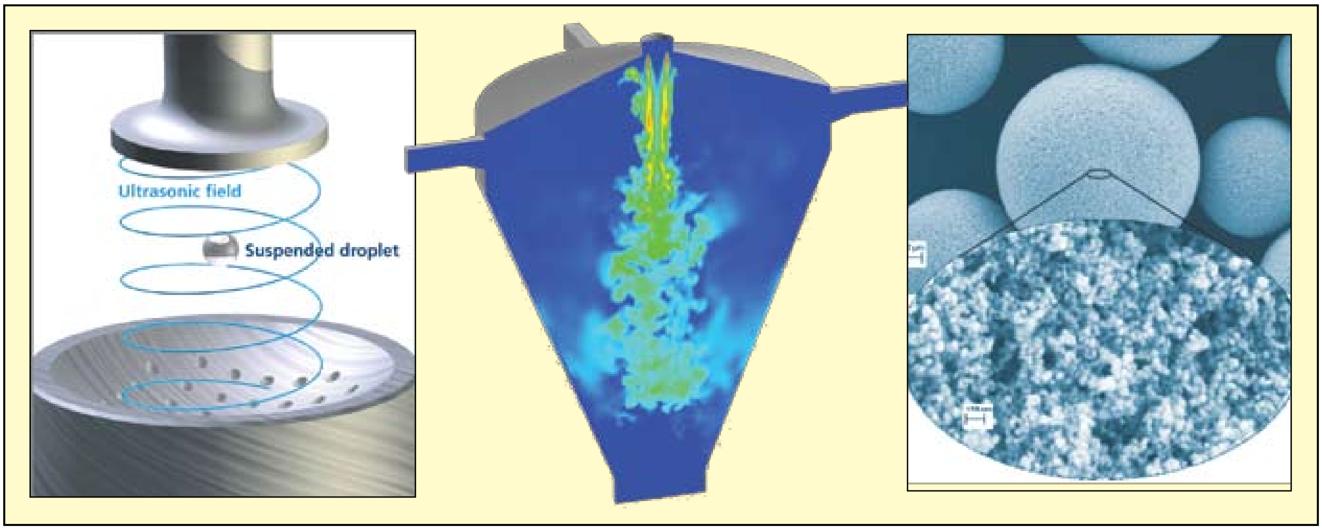

FIGURE 8. Ultrasonic levitation (left) is used to suspend a single droplet of feed being tested, making it ideal for observing and measuring the drying process. CFD simulation results (center) show the instantaneous fraction of water vapor in a spray dryer. The combination of levitation and CFD simulation has enabled better designed spray dryers. Agglomerating nanoparticles into larger, spray-dried particles (right) allows for safer processing

This makes it possible to determine the applicability of spray drying and to optimize product formulations at a very early stage in development when only a small amount of the product is available. The results also allow for more precise CFD simulations and thereby better designed spray dryers.

The method is based on an ultrasonic levitator equipped with a climate chamber to control air humidity, temperature and velocity. The levitator keeps the droplet to be studied suspended in the air, allowing for precise studies and measures of the drying kinetics. A mathematical description of the drying kinetics is established, and very accurate spray-drying simulations using CFD software are performed. It is now possible to calculate the time-temperature history during drying, which enables the design of minimum-thermal-degradation spray dryers for temperature sensitive products. Knowing the state of drying when the particles hit the dryer walls will — together with a stickiness criterion — give accurate information on what areas will be prone to develop deposits. Utilizing high performance computing clusters makes it possible to design optimal spray dryers with unprecedented accuracy.

Spray drying can be a vital link in the application of nanotechnology to achieve products with superior performance (for instance, fuel cell elements, automobile light covers and so on). It is still uncertain to what extent nano-based materials will be implemented in the future. Safe processing technology is of paramount importance and one safe route will be spray drying of nano-suspensions into powders or granules sized 10–100 microns. Developing process technology to exploit nanotechnology has international attention. An example is the EU-funded Saphir project (www.saphir-project.eu), which aims at demonstrating an environmentally safe production process — from the synthesis of nanoparticles, particles processing to the making of the final products.

Within the pharmaceutical industry, particle engineering is very important, and the use of spray drying is being explored widely. Spray drying can be applied to produce encapsulated powders for controlled release of API or taste masking, just as it can maintain the API in its amorphous form to enhance bioavailability. Since solid dosage forms in general are preferred to liquid based systems, research is driven toward delivery forms based on powders, for which spray drying is an ideal process. Research in spray drying and, for instance, controlled release is conducted by several groups worldwide, including the international Swedish-based research consortium Codirect (www.codirect.se) and others.

The field of spray drying is constantly developing. Increasing and more precise knowledge about the spray drying process and its dynamics opens avenues for using the technology in new fields just as new products and standards set new demands. ■

Edited by Gerald Ondrey

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