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Spray Drying as an Appropriate Technology for the Food and Pharmaceutical Industries - A Review

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Abstract: Spray drying is a method used to produce dry powder from a liquid or slurry by rapidly drying with a hot gas and it is mostly used in the food and pharmaceutical industries. This paper covers the parameters of spray dryer, principles of spray dryer, basics of spray drying, dryer configuration, collection of dried powder, and flow of drying gas, process design and control. Spray dryers can dry a product very quickly compared to other methods of drying. They also turn a solution or slurry into a dried powder in a single step, which can be advantageous for profit maximization and process simplification. Spray dryers employ atomizer or spray nozzle to disperse the liquid or slurry into a controlled drop size spray. The commonest of these are rotary disks and single-fluid high pressure swirl nozzles. Spray drying offers multiple opportunities that no other single drying technology can claim. Besides spray drying offers unique opportunities in particle size engineering.

Key words: Spray drying, pharmaceutical, dryer configuration, solution, nozzles

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INTRODUCTION

Spray drying is widely used in the industry for conversion of a suspension or solution into a dry powder product. In spray drying the suspension or solution feed is atomized and the droplet formed comes into contact with a hot gas. When the droplets and the heated gas come into contact, the solvent in the droplets evaporate, leaving a dry powdered product. Spray drying is presently one of the most exciting technologies for the pharmaceutical industry. It is an ideal process where the end products meet the precise quality standards regarding particle size distribution, residual moisture/solvent content, bulk density and morphology¹.

The development of spray drying equipment and techniques started in the 1870s. Spray drying has come of age ever since the technique was used during the World War II, because the idea to reduce the transport weight of foods and other materials became important². The dried product from spray dryers can be in the form of powders, granules or agglomerates depending on the physical and chemical properties of the feed, the dryer design and final powder properties desired².

Spray dryers can dry a product very quickly compared to other methods of drying. They can also turn a solution or slurry into a dried powder in a single step, which can be advantageous for profit maximization and process simplification³. All spray dryers employ atomizer or spray nozzle to disperse the liquid or slurry into a controlled drop size spray. The commonest of these are rotary disks and single-fluid high pressure swirl nozzles. Alternatively, for some applications two-fluid or ultrasonic nozzles are used. Depending on the process needs, drop sizes from 10 to $500 \,\mu\text{m}$ can be achieved with the appropriate choices. The most common applications are in the 100 to $200 \,\mu\text{m}$ diameter range³. The most common spray dryers are called single effect because there is only one drying air on the top of the drying chamber. In most cases the air is blown in co-current with the sprayed liquid. The powders obtained with such type of dryers are fine with a lot of dusts and poor flow ability⁴

In order to reduce the dusts and increase the flow ability of the powders, multiple effect spray dryers are used instead of drying the liquid in one stage. The drying is done through two steps: one at the top (as per single effect) and an integrated static bed at the bottom of the chamber. The integration of this fluidized bed allows the fluidizing powder inside a humid atmosphere to agglomerate to fine particles or granules with medium particle size within a range of 100 to $300 \,\mu\text{m}^{5}$. The fines generated by the first stage drying can be recycled in continuous flow either at the top of the chamber (around the sprayed liquid) or at the bottom inside the integrated fluidized bed. The drying of the powder can be finalized on an external vibrating fluidized bed. The hot drying gas can be passed as a co-current or counter-current flow to the atomizer direction. The co-current flow enables the particles to have a lower residence time within the system and the particle separator (typically a cyclone device) operates more efficiently. The counter-current flow method enables a greater residence time of the particles in the chamber and usually is paired with a fluidized bed system ⁵. The aim of this paper is it to bring to fore the modern advances in the usage of a spry dryer in food and drug production

PARAMETERS OF SPRAY DRYER

Every spray dryer consists of feed pump, atomizer, air heater, air disperser, drying chamber, and systems for exhaust air cleaning and powder recovery (**Fig 1**).

- **Inlet temperature of air**: The higher the temperature of inlet air, the faster is the moisture evaporation but the powder is subjected to higher temperatures, which may distort the chemical/physical properties of heat- sensitive products ².
- **Outlet temperature of air:** This governs the sizing of powder recovery equipment, and the higher the outlet air temperature, the larger will be the size of powder recovery equipment and conveying ducts and plenums⁶. The outlet air temperature controls the final moisture content of the powder.

- **Viscosity:** High viscosity hinders correct drop formation. As the viscosity is lowered, less energy or pressure is required to form a particular spray patterns.
- **Solid content:** Care must be taken with high solid loadings (above 30%) to maintain proper atomization to ensure correct droplet formation.
- **Surface tension:** Addition of a small amount of surfactant can significantly lower the surface tension. This can result in a wider spray pattern, smaller droplet size, and higher drop velocity.
- **Feed temperature:** As the temperature of a solution to be sprayed is increased, the solution may easily dry as it brings more energy to the system.
- Volatility of solvent: A high volatility is desirable in any drying process. Unfortunately, choices are limited today. In many cases, these restrict the solvent choice to water.
- **Nozzle material:** Most pharmaceutical applications use stainless steel inserts. However, tungsten carbide nozzles are often available and have excellent resistance to abrasion and good corrosion resistance for most feedstock.

PRINCIPLES OF SPRAY DRYER

Atomization: The formation of sprays having the required droplet size distribution is vital to any successful spray dryer operation so that powder specifications can be met. Atomization is created by either a rotary atomizer or spray nozzle atomizer as shown in **Fig. 1**. The location of the fluid bed within the drying chamber achieves drying at lower temperature levels. It results in higher thermal efficiencies and cooler conditions for powder handling.



Fig.1: The Spray dryerhttp://www.indjst.org (2012)

Co-current: Drying air and particles move through the drying chamber in the same direction. Product temperatures on discharge from the dryer are lower than the exhaust air temperature, and hence this is an ideal mode for drying heat sensitive products. When operating with rotary atomizer, the air disperser creates a high degree of air rotation, giving uniform temperatures throughout the drying chamber.

However, an alternative non-rotating airflow is often used in tower or FILTERMAT®-type spray dryers using nozzle atomizers with equal success.

Counter-current: Drying air and particles move through the drying chamber in opposite directions. This mode is suitable for products which require a degree of heat treatment during drying. The temperature of the powder leaving the dryer is usually higher than the exhaust air temperature¹.

Mixed flow: Particle movement through the drying chamber experiences both co-current and countercurrent phases. This mode is suitable for heat stable products where coarse powder requirements necessitate the use of nozzle atomizers, spraying upwards into an incoming airflow, or for heat sensitive products where the atomizer sprays droplets downwards towards an integrated fluid bed. The air inlet and outlet are located at the top of the drying chamber¹.

Atomization: Several types of atomization are available. They are centrifugal, nozzle, pneumatic and sonic atomization.

Centrifugal atomization: This uses a rotating disc or wheel to break the liquid stream into droplets. These devices normally operate in the range of 5,000 to 25,000 rpm with wheel diameters of 5 to 50 cm. The size of the droplets produced is nearly inversely proportional to the peripheral speed of the wheel¹. The distribution of particle sizes about the mean is fairly constant for a given method of atomization. The mass flow of the liquid, its viscosity, solids content and surface tension influence particle size directly, but none to the degree of peripheral wheel velocity. Consequently, an increase in feed rate may slightly increase the particle size but use of a variable-speed drive on the centrifugal atomizer facilitates correction to the specified size¹. One advantage of centrifugal atomization is that atomizing machines are available in many sizes. A small air-driven laboratory unit handles from 1 to 10 L/h of liquid feed, while the largest commercial units driven by 850-kW motors can handle in excess of 200,000 kg/h.¹⁰

Nozzle atomization: The second most common form of atomization is hydraulic pressure-nozzle atomization. Here the liquid is pressurized by a pump and forced through an orifice to break the liquid into fine droplets. Orifice sizes are usually in the range of 0.5 to 3.0 mm. As a result, a single nozzle is limited to somewhere in the order of 750 kg/h of feed, depending on pressure, viscosity, solids content and orifice size¹. Greater pressure drop across the orifice produces smaller droplets. Therefore, to reduce the particle size for a given feed rate, the nozzle must be removed and a smaller orifice substituted. This in turn requires a higher pump pressure to achieve the same mass flow through the nozzle. Very large systems may have as many as 40 nozzles, making control of particle size difficult. Precise control, however, is not always required, and large, multiple-nozzle dryers are often used when the only requirement is that the mean particle size be quite large. Although nozzles are considerably less complicated than centrifugal atomizers, a high-pressure pump is required. During the drying of abrasive materials, the nozzles can pose special problems. The potential for plugging the relatively small orifices is another drawback for nozzle-based atomization systems¹.

Pneumatic atomization: A third method used primarily in smaller drying systems is two-fluid pneumatic atomization. Here atomization is accomplished by the interaction of the liquid with a second fluid, usually compressed air. Neither the liquid nor the air requires very high pressure, with 200 kPa to 350 kPa being typical. Particle size is controlled by varying the ratio of the compressed air flow to that of the liquid. The main advantage of this form of atomization is that the liquid has a relatively low velocity as it exits the nozzle, and therefore, the droplets require a shorter flight path for drying. This makes two-fluid nozzles ideal for use in pilot- or laboratory-scale equipment 1 .

Sonic atomization: this type of atomization employs the use of ultrasonic energy where the passing of liquid over the surface is vibrated at ultrasonic frequencies. These systems are suitable for producing very fine droplets at low flow rates. However, more development is needed if these atomizers are to find wider acceptance in industrial drying both in capacities handled and the range of different products to be atomized¹.

Dryer configuration: The atomized droplets that are formed from the atomizing device have a velocity and direction initially established by the atomizer (**Fig. 2**). It is necessary for the heated gas to mix with the cloud of droplets, then begin evaporation, and influence the movement of the droplets inside the dryer, so that they can dry sufficiently and do not stick on contact with the dryer walls. This is accomplished by placing the atomizer in, or adjacent to, a properly designed air-disperser.



Fig.2: The atomized droplets that are formed from the atomizing device established by the atomizer

A concurrent configuration with nozzle atomizer is suited for commodity chemicals; a counter-current design with a nozzle atomizer is best suited for products requiring heat treatment; a mixed-flow unit with a nozzle atomizer is ideal for coarse powders of heat-stable products⁷.

The atomizer, disperser and drying chamber must all be properly configured to allow complete drying of all the droplets without deposits of wet material on the interior surfaces of the dryer. In addition, the total volume of the drying chamber and the flow patterns of the droplets and the air through the dryer must provide for sufficient contact time to allow evaporation of essentially all of the liquid. As a result, centrifugal atomizers are usually installed at the center of the roof of a relatively large-diameter spray dryer. The heated air is introduced through a roof-mounted air disperser around the atomizer, creating a concurrent flow of air and product. By coming in contact with the droplets as soon as they are formed, the heated air causes rapid surface evaporation, and keeps the solids relatively cool⁷.

By the time evaporation slows down and becomes limited by diffusion of liquid from the center of the droplet to the surface, the particles have passed to a cooler region of the dryer. Therefore, heat-sensitive products can often be spray-dried using elevated temperatures in the inlet gas, even though those temperatures would damage the product in an oven or other processes that are not concurrent, or as fast as spray drying. The larger the particle size desired in the final powder, the larger must be 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, is often found to be a lot more practical. Here 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 big drawback in fountain-nozzle dryers is that the process is not concurrent. Rather, it is mixed flow, and 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 greater. Sometimes lower inlet temperatures solve this problem, but also reduce total evaporation capacity⁷.

The third most commonly used configuration has pressure nozzles at the top of a dryer, spraying concurrently with the heated air. This takes advantage of evaporative cooling, but often requires the dryer to have a cylinder height of about 20 m. These "nozzle towers" are often used for foodstuffs, dyes, pesticides and other heat-sensitive products that must also be in a coarse, free-flowing powder form⁷.

Collecting the dried powder: Once the product is dried to a free-flowing powder, it must be separated from the drying gas, which is now cooled and contains the evaporated liquid. Coarser powders are most easily collected directly from the bottom of the drying chamber cone. 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 greatest fraction of the powder to settle in the cone, and slide to the bottom outlet often equipped with an air-lock ⁵. Because the spent drying gas has some entrained powder, cyclones or fabric filters are often used to clean the gas. In some cases, the combination of cyclones followed by a wet scrubber proves more effective. If the powder is very fine, little is collected in the drying chamber. In this case, the cyclones or even the bag collector can become the primary collection point. Chamber collection is eliminated by using a U- bend at the outlet for both gas and powder from the chamber to the other collectors ⁸.

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 turns out to be the most efficient. Fuel oil or propane backup is often provided when gas curtailment is possible. If indirect heating is required, shell-and-tube or finned-tube exchangers are used with steam or a heat-transfer fluid as a heat source. Electric heaters are used on small-scale dryers. In some instances, however, waste heat from another process is recovered either by direct injection into the drying gas stream or by heat exchanger. 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, it may be filtered by coarse filters to remove leaves, dirt and so on. If a very clean process is required, high-efficiency particulate air filters can be used. Ductwork with appropriate dampers, expansion joints, vibration isolators and noise-abatement devices is supplied with most dryers. All equipment is usually insulated and clad to minimize heat loss condensation, and personnel hazards ⁹.

Process design and control: 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, actual values are usually determined experimentally⁹. 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 particle size desired and the product's known drying characteristics. This permits direct calculation of a chamber volume. At this point, the method of atomization must be selected and matched with chamber dimensions to obtain the desired volume and configuration with respect to the atomized cloud. If nothing is known about the product, one needs to conduct pilot-scale experiments. Once designed and built, the drying system needs fairly simple controls. Although one should have a rough estimate of the actual gas flow through the dryer, it is usually best to fix the flow at the design rate. 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. In the simplest case, the outlet temperature controls the heat input to the feed and thereby the inlet temperature, while holding the feed rate constant. In fact, dryers with a small nozzle atomizer do just that, using a single feedback control loop. One slightly more advanced approach is to use a "cascade control configuration" in which the outlet temperature controller can change the inlet controller's set point to achieve correct final moisture level in the product ⁹. Pressure drops across filters and cyclones, and the pressure in the drying chamber are usually

monitored, not controlled, to assure that the system is operating properly. Centrifugal atomizers require monitors for lube-oil flow, temperature, and vibration. On the other hand, nozzle systems require feed-pressure monitoring ⁹. Although a spray dryer can be operated with simple controllers, it is becoming normal practice to use programmable logic controllers (PLCs), which offer greater capability in monitoring alarming functions. In addition, these PLCs can initiate programmed start-ups and shutdowns. Inclusion of a personal computer offers data logging, trend analysis and other features used in statistical process control and other quality-assurance programs ⁹.

BENEFITS OF SPRAY DRYING

It has a high precision control over:

- Particle size, bulk density, degree of crystalline and residual solvents
- Typical application in pre-formulated products
- Microencapsulation, solid solutions
- Improved bioavailability, improved product stability
- Products with unusual or difficult characteristics
- Sticky or hygroscopic products
- Slowly crystallizing products
- Difficult to isolate products
- Rapid drying for temperature sensitive materials

ADVANTAGES OF SPRAY DRYER TECHNOLOGY

Pharmaceuticals: Some pharmaceuticals occur in crystal form, making them difficult to use. Crystalline products do not dissolve easily in water and are absorbed slowly, so they are currently unused because of bioavailability. Spray dryers dry the compound once it has been dissolved in water for easier absorption. Drugs that are in crystal form are harder for the body to use, therefore sprays dryers make them more readily available and usable to the body. Spray drying can offer commercial and medical advantages with encapsulation because it helps give particles the ability to be controlled in a time-release pattern (such as a 6 or 12-hour allergy, headache, or cold medicine). The substance is spray-dried and then compressed into a capsule form. Prolonged release of antibiotics allows a reduction in the dosage or concentration, and can be effective when treating chronic illnesses. Because of the process used to develop spray-dried products, vitamin and mineral content loss is kept to a minimum.

Food Products: The quick process of spray drying keeps flavor loss to a minimum. Dairy products, such as milk, whey, cheese, buttermilk, butter and dry creamer are common items made using the spray drying technique. Instant coffee, dry creamer and instant soups can also be spray-dried, and previously spray-dried foods often serve as baby foods. Spray drying keeps the retail price of such foods low, because the process extends the product's shelf life.¹⁰

Industrial Products: Spray dryers offer an advantage in many commercial and industrial industries. Reconstituted dyes are used on fabrics and clothing, and spray-dried pigments appear in many wall paints. Spray dryers reduce the size of particles found in dyes to allow more consistent and convenient dispersion into paints. This same process also allows the dyes to be dissolved in liquids that can then dye clothes and fabrics. Many ceramic and clay tiles consist of particles that have been spray-dried. The density and level of moisture in ceramic and clay tiles are controlled more readily when produced with a spray dryer.¹⁰

The production of milk powder using spray drying technique: Milk powder production plant is an example of spray drying plant. During the process of milk powder production, after cooling, pasteurization and homogenization stages, the milk emulsion is concentrated to 48–52 wt% of total solids in a multiple-effect evaporator system (typically of the falling-film type or plate type)¹¹ and after this the

concentrated emulsion becomes ready for spray drying. The concentrated emulsion is atomized into droplets of 200 μ m by a centrifugal wheel atomizer or a high pressure spray nozzle, located at the top of the spray chamber. The droplets fall into the spray chamber in a concurrent flow with a hot filtered air, the moisture in the emulsion droplets is removed by hot air. Milk droplets shrink in size as water is evaporated from its surface.

Finally, the droplets lose most of their moisture and become particles with a solid crust formed at their surfaces. In the single-stage spray drying process, a pneumatic conveying system is always needed to remove the final fraction of moisture from the nominally dried powder and cool it prior to storage. In the single-stage spray drying system, energy consumption is high, and the fine powder product is not readily soluble in water. Due to these reasons, a multi-stage drying system was developed in the 1970s. In this system, a vibro-fluidized bed drying (VFBD) and cooling system are found below the spray drying chamber. It consists of a rectangular chamber, inside which is an inclined or wrinkled perforated plate. Hot air at 80–120 °C is first used to reduce the particle moisture from 8–9 wt% to 3–4 wt%, as the particles move along the perforated plate. Downstream of the VFBD, dehumidified air is always used to cool the dried particle before safe packaging. In the 1980s, a three-stage drying system was developed for milk drying purposes. In this system, a conventional fluidized bed is inserted into the conical bottom of the spray drying chamber. Hot air at 90–100 °C is used to fluidize the semi-dried milk powder. Drying and cooling operations can be better controlled in the second and third stages. The fine powders from the spray drying chamber and VFBD are recycled into the spray drying chamber, and then agglomerated powders are finally obtained¹².

Recycling the fine particles that have been classified out of the fluidized bed(s) and/or cyclone(s) may be done at different locations in the primary chamber of the spray dryer for agglomeration purpose. The Niro method is called "straight-through method" that returns the fines and feeds them into the liquid atomization zone to induce agglomeration^{13,14}. This enhances powder quality by controlling the particle size distribution, and by reducing dustiness. It is reported that a three-stage drying process can save 20% of energy consumption compared to the single-stage spray drying system¹⁵. Multi-stage drying process is preferred in dairy product processing due to energy efficiency and agglomeration of the particles obtained which makes the product instantly soluble in water. Partially wet particles can be dried on the belt at a lower temperature. Combined drying system is suitable for heat-sensitive products, such as dairy foods.

Multi-staging reduces the size of the spray drying chamber considerably. Pulse combustion spray drying has been reported as a relatively new but unpopular. However, it can be used to dry milk, Wu and Liu¹⁶ used CFD method to model such a process. They found that the drying rates are much faster than in normal spray drying. Xiao et al.¹⁷ have investigated the effects of atomizing parameters on droplet characteristics in a pulse combustion spray dryer. Generally, air is used as the drying medium for spray drying. Ducept et. al.¹⁸ and Frydman et al.¹⁹ used a commercial code to simulate a spray dryer using superheated steam as the drying medium. However, in their models, the elevation of the boiling point for suspension was not considered. Although superheated steam can provide a number of advantages, e.g., excellent energy efficiency if the exhaust steam can be utilized elsewhere in the plant and also advantages resulting from the absence of oxygen, the equipment and operation are more costly and complex.

Pilot tests are being conducted commercially to examine the product quality of steam-dried milk powders. Freeze-drying is a good way to obtain high-quality heat-sensitive products. Sonner ²⁰ used liquid nitrogen to obtain frozen powder. The frozen powder was transferred to a vacuum freeze-drying chamber and it was realized that the drying time reduced significantly compared to normal freeze-drying. Leuenberger et al.²¹ reported spray freeze-drying of pharmaceuticals using cooled and dehumidified air obtained by dry ice. Rogers et. al.²² investigated milk powder characteristics in laboratory-scale spray freeze-dryer using liquid nitrogen as the cryogen. However, this process still needs to be developed to the industrial scale ²³.

Granulation: Spray dried granulation has improved flow, better distribution of drug, colors, etc. and requires less lubricant than wet massed products². Spray drying results in a shell of concentrated binder at the surface of the granular material, providing strong tablets and maximum use of binder.

Bioavailability: With spray drying one can co-precipitate a polymer in a stable amorphous solid dispersion, thereby greatly improving the dissolution rate of many drug substances, including tolbutamide, in domethacin and ibuprofen ²⁴. Complexes of paracetamol and diazepam have been prepared with pcyclodextrin.

Encapsulation: With a concurrent drier, heat exposure is minimized. The product is usually recovered about 15° C below the outlet temperature ²⁵. This has been applied to microencapsulation of products such as antibiotics, vaccines, peptides and proteins.

Inhalation: Highly specialized spray drying nozzles that give increased particle engineering capabilities, even on large scale- making it possible to accurately manipulate aerodynamic particle size and properties. Spray drying technologies make it easier than ever to efficiently produce therapies in the form of free-flowing particles that are ideally suited for inhalation ²⁶.

CONCLUSION

Spray drying is presently one of the most exciting technologies for the pharmaceutical and the food industries. It is an ideal process where the end product complies with precise quality standards regarding particle size distribution, residual moisture/solvent content, bulk density and morphology. One advantage of spray drying is the remarkable versatility of the technology, evident when analyzing the multiple applications and the wide range of products that can be obtained. From very fine particles for pulmonary delivery to big agglomerated powders for oral dosages, from amorphous to crystalline products and the potential for one-step formulations, spray drying offers multiple opportunities that no other single drying technology can claim. Besides the aforementioned opportunities, spray drying offers unique opportunities in size particle engineering.

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