
1 Introduction to spray drying

1.1 INTRODUCTION

“The process of simultaneously atomizing and desiccating fluid and solid substances, and its application to the purpose of the exhaustion of moisture from such substances, and for the prevention of destructive chemical change.”

Samuel R. Percy (1872)

The above words are excerpts from the first ever detailed description of a drying technique, which is now well-known and appreciated as “Spray Drying”. Spray drying is a 140 years young and flourishing drying technique. Throughout all these years, this perpetual process has exhibited an ebullient growth, imbibing innumerable innovations in terms of its operational design and widely varied applications.

Spray drying has its origin in the United States, since the first patented design was registered there in 1872. World War II was a significant chronological event in the history of spray drying, monopolizing the process in the dairy industries for continuous production of milk powder. Since then, the process has adapted itself to a number of design modifications, and today has evolved as an industry-friendly drying technique. Spray drying stands out from other processes involving liquid drying by its ability to handle feedstock of varying nature, producing flowing powders of specific particle size, high productivity and versatile applications.

By definition, *“Spray drying is the transformation of feed from a fluid state into a dried particulate form by spraying the feed into a hot drying medium.”* (Masters, 1991)

A spray dryer operates on convection mode. The principle of working is moisture removal by application of heat to the feed product and controlling the humidity of the drying medium. Here, the uniqueness is that the evaporation of moisture is promoted by spraying the feed into a heated atmosphere, resulting in improved drying rate. The mechanism can be better understood, when the spray drying process is divided into its constituent unit operations.

A liquid feed entering the spray dryer undergoes a series of transformations before it becomes powder. The changes are due to the influence of each of the four stages (Figure 1.1) involved in spray drying, namely:

1. Atomization of the feed solution.
2. Contact of spray with the hot gas.
3. Evaporation of moisture.
4. Particle separation.

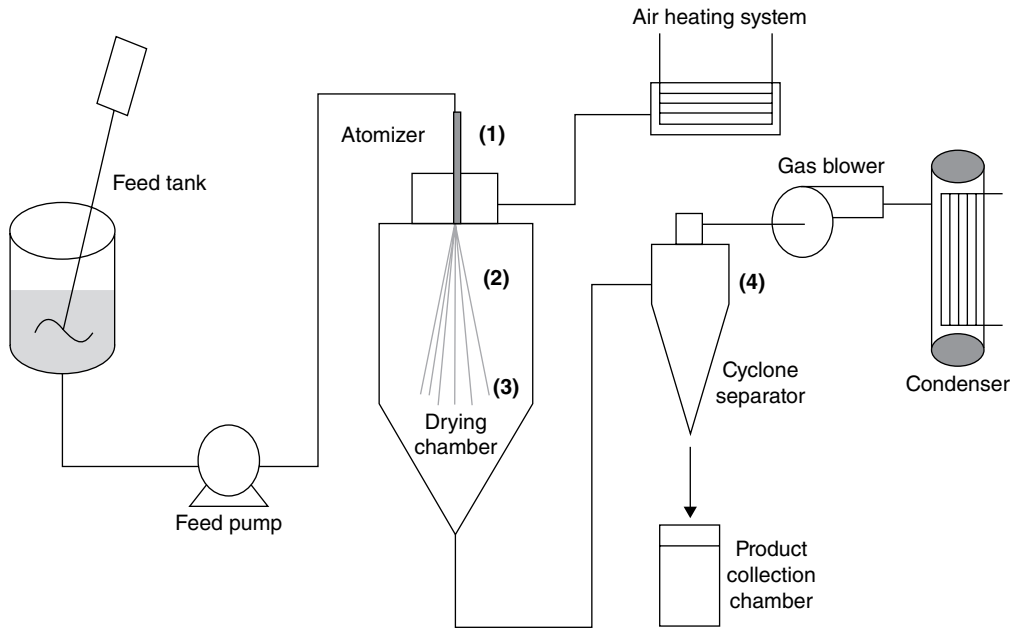


Figure 1.1 Process steps of spray drying. (1) Atomization. (2) Spray – hot air contact. (3) Evaporation of moisture. (4) Product separation.

Each of the above exerts influence on the final product quality. Understanding the process steps, along with the hardware systems involved in it, will enable visualization of the operation on a glimpse of a reading. Hence, subsequent sections will narrate in detail each of the above-mentioned unit operations, with a description of the associated hardware components.

1.2 STAGE 1: ATOMIZATION

Atomization is the heart of spray drying, and is the first transformation process that the feed undergoes during spray drying. Although several definitions of atomization exist, one of the initial definitions of the process, by Samuel Percy, is as interesting as it is precise: “*bringing fluid or solid substances into a state of minute division*”. The breakup of bulk liquid into a large number of droplets drives the rest of the spray drying process by reducing the internal resistances to moisture transfer from the droplet to the surrounding medium. This is because of the enormous increase in surface area of the bulk fluid as the droplet fission proceeds, with its instability increasing in accordance with the intensity of atomization.

Atomization is central to the spray drying process, owing to its influence on shape, structure, velocity and size distribution of the droplets and, in turn, the particle size and nature of the final product. A cubic meter of liquid forms approximately 2×10^{12} uniform 100 micron-sized droplets, offering a total surface area of over 60,000 m² (Masters, 2002). This greater surface-to-volume ratio enables spray drying to achieve a faster drying rate (as drying time is proportional to the square of the particle dimension). Consequently, there is minimal loss of heat sensitive compounds and, eventually, particles of the desired morphology and physical characteristics are obtained.

1.2.1 Principle of atomization

The working principle of the atomizers is governed by the liquid disintegration phenomenon explained by several researchers. It is worth understanding the progression in the concepts on atomization phenomenon across the years. This will also help in appreciating the science of droplet formation from an atomizer.

Joseph Plateau was the first to characterize liquid instability in 1873, through his experimental observations. A liquid jet, initially of constant radius, falls vertically under gravity. The liquid length increases and reaches a critical value. At this critical value, the jet loses its cylindrical shape and decomposes into a stream of droplets that occurs primarily due to decrease in surface tension (Figure 1.2).

Lord Rayleigh, in 1878, corroborated the above stated theory and gave an analytical explanation of the physical observation. He provided a mathematical insight to the break-up of non-viscous liquid under laminar flow conditions, now famously known as the “*Liquid jet theory*”. Rayleigh considered the simple situation of a laminar jet issuing from a circular orifice, and postulated the growth of small disturbances that produce breakup when the fastest growing disturbance attains a wavelength (i.e. λ_{opt} of $4.51d$, where d is the initial jet diameter). After breakup, the cylinder of length $4.51d$ becomes a spherical drop (Figure 1.3) and, hence, can be approximated to a sphere of equal volume (Equation 1.1).

$$4.51d \times \left(\frac{\pi}{4}\right) d^2 = \left(\frac{\pi}{6}\right) D^3 \quad (1.1)$$

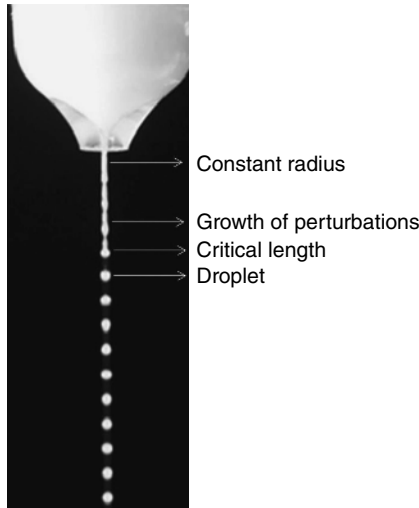


Figure 1.2 Schematic of liquid instability (Modified from Wu *et al.*, 2014).

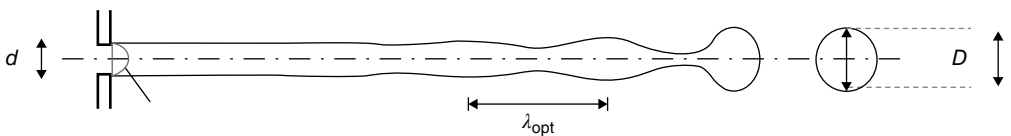


Figure 1.3 Mechanism of droplet formation (Adapted from Wu *et al.*, 2014).

Where D is the droplet diameter, which can be obtained as:

$$D = 1.89d \quad (1.2)$$

Although Rayleigh's analysis considered surface tension and inertial forces, the influence of viscosity, atomization gas and the surrounding air were neglected. The above gaps in knowledge were addressed by the work of Weber (1931) and Ohnesorge (1936). Weber revealed that the air friction shortens the optimum wavelength (λ_{opt}) for drop formation. He obtained a value of $\lambda_{opt} = 4.44d$ at zero relative velocity, which is close to the value of $4.51d$ predicted by Rayleigh for this case. Weber showed that as the relative velocity increases to 15 m/s, λ_{opt} becomes $2.8d$ and the droplet diameter is $1.6d$. Thus, the increase in relative velocity between the liquid jet and the surrounding air reduces the optimum wavelength for jet breakup and results in a smaller droplet size.

The explanation given by Ohnesorge in 1936 on the mechanism of atomization is credited for its clarity. The relationship proposed by him included all the significant factors responsible for atomization. He proposed the Reynolds number relationship, and expressed the tendency of the liquid jet to disintegrate in terms of its viscosity, density, surface tension and jet size. The relationship can be numerically expressed by the dimensionless Ohnesorge number (Oh) which is the ratio of Weber number to Reynolds number (Equation 1.3), as described by the equation below:

$$Oh = \frac{\sqrt{We}}{Re} = \frac{\mu}{\sqrt{\rho\sigma L}} = \frac{\text{Viscous forces}}{\sqrt{(\text{inertia} \times \text{surface tension})}} \quad (1.3)$$

where:

We is the Weber number;

Re is the Reynolds number;

μ , ρ and σ are the viscosity, density and surface tension of the feed droplet, respectively;

L is the characteristic dimension of the feed droplet (i.e. volume per unit area).

Disintegration of the liquid at the periphery or tip of the atomizer is by virtue of the turbulence in the emerging liquid jet and the action of air forces; the resistance to disintegration is offered by viscosity and surface tension forces in the liquid. The realignment of shear stresses within the liquid, once the droplet is airborne, contributes to the droplet fission during atomization.

1.2.2 Classification of atomizers

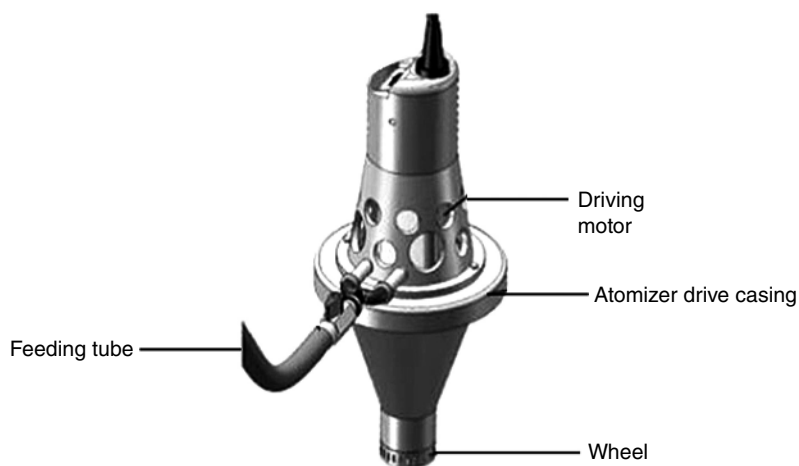
The atomization device is vital to this process, and its selection plays a major role in utilizing spray drying as an economical drying method. Prior to exploring the mechanism and working of the atomizers, understanding the rationale for their classification holds significance. The atomizers are differentiated on the basis of the criteria listed in Table 1.1. The major types include *rotary atomizers* and *nozzle atomizers*. The working principle of different types of atomizers is elaborated subsequently.

1.2.2.1 Rotary atomizers

Principle: Driven by high velocity discharge of liquid from the edge of a wheel or disc (Figure 1.4). Feed liquid is centrifugally accelerated at high velocity to the centre of a rotating wheel with a peripheral velocity of 200 m/s. The outward flowing feed with respect to the rotating wheel surface accelerates to the periphery and then disintegrates into a spray of droplets.

Table 1.1 Rationale for atomizer classification.

Basis of atomizer classification	Quantifying parameter
Energy utilized for atomization.	Centrifugal, pressure, kinetic, sonic or electrical energy.
Desired type of spray.	Fine, medium or coarse.
Required final size range of droplets.	Mean droplet size in μm or nm .
The relationship between mean size of droplets and atomization parameters.	Direct or inverse relationship of the mean droplet size with atomization pressure or atomizer speed of rotation, diameter or electric potential across the charged nozzle.
Physical properties of the feed that can be handled by the atomizer.	Viscosity and abrasiveness of the feed.
Capacity of feed that the atomizer can handle.	Feed rate in mL/min or L/h .
Need for duplication of atomizers.	The ratio of increase in the atomizer capacity to the number of atomizers linked in series.

**Figure 1.4** Rotary atomizer (Murali *et al.*, 2014).

Atomization energy: Centrifugal energy.

Atomization parameters: Wheel speed in rotation per minute (RPM).

Type of spray: Fine, coarse or medium.

Mean droplet size: 30–120 μm .

Relationship between mean droplet size (d) and atomization parameters: d is directly proportional to feed rate and feed viscosity, and inversely proportional to wheel speed and wheel diameter.

Physical property of feed: Demonstrates ability to handle abrasive feed-stocks by virtue of atomizer vanes and bushings.

Atomizer duplication: Rotary atomizers are known for their ability to handle high feed rates without atomizer duplication.

Advantages: The major advantages of rotary atomizers are that they do not clog, and they tend to produce more uniformly sized droplets. Since the necessary atomization energy is supplied by the rotating wheel, the feed supply unit can operate at low pressure than that required in hydraulic and pneumatic nozzle atomizers.

Limitations: Rotary atomizers present difficulties in handling viscous feed. The large amount of fine particles produced can potentially lead to environmental pollution. Furthermore, it is not possible to accommodate the spray produced by rotary atomizer in a horizontal spray dryer.

1.2.2.2 Pressure nozzle (or hydraulic) atomizer

Principle: Facilitated by discharge of liquid under pressure through an orifice (Figure 1.5). Pressure energy is converted to kinetic energy, and feed emerging from the nozzle orifice as a high speed film readily breaks into a spray of droplets.

Atomization energy: Pressure energy.

Atomization parameters: Nozzle pressure.

Operating pressure range: 250–10,000 PSI.

Type of spray: Coarse and less homogeneous.

Mean droplet size: 120–250 μm .



Figure 1.5 Pressure nozzle.

Relationship between mean droplet size (d) and atomization parameters: d is directly proportional to feed rate and viscosity, and inversely related to atomization pressure.

Physical property of feed: Low viscosity feed.

Atomizer duplication: Pressure nozzles can be integrated in multiple nozzle arrangements to obtain an increased amount of flow rate and particle size flexibility.

Advantages: Pressure nozzles result in particles with less occluded air when compared to twin fluid atomizers. Consequently, the powdered product is of higher density, with good flow characteristics. Depending on the specifications of the end product, it is also capable of producing particles with relatively greater size.

Limitations: At high feed rates, sprays are generally less homogeneous and coarser than rotary atomizers.

1.2.2.3 Two-fluid nozzle atomizer

Principle: The operational principle is based on Weber's findings, as explained in section 1.2.1. Two-fluid atomizers feature the break-up of liquid on impact with high-velocity air or other gaseous flow. Compressed air creates a shear field, which atomizes the liquid and produces a wide range of droplet sizes (Figure 1.6).

Atomization energy: Kinetic energy.

Atomization parameters: Nozzle pressure.

Operating pressure range: 250–10,000 PSI

Type of spray: Medium coarseness but poor homogeneity

Mean droplet size: 30–150 μm .



Figure 1.6 (a) Two-fluid nozzle; (b) Spray emerging from two-fluid nozzle.

Relationship between mean droplet size (d) and atomization parameters: d is directly proportional to feed rate and viscosity and inversely related to atomization pressure.

Physical property of feed: Can handle highly viscous feed.

Atomizer duplication: Rather than atomizer duplication, more than one atomization fluid is employed to transmit the kinetic energy to the feed. The use of four fluid nozzles is the latest advancement (Niwa *et al.*, 2010).

Advantages: Twin fluid nozzles are capable of handling highly viscous feed. These atomizers also produce much finer and more homogeneous spray when compared to pressure nozzles. These nozzles exert better control over the droplet size.

Limitation: The requirement of compressed air adds to the cost of operation. Twin fluid nozzles result in high occluded air content within the particles, resulting in low density. The use of these nozzles also introduces extra cold air into the spray chamber in the zone of atomization and, hence, reduces the temperature gradient that exists between the finely divided droplet and the surrounding drying medium. This impairs the effectiveness of heat transfer between the droplet and hot drying medium. Twin fluid nozzles exhibit a higher tendency to clog, especially when the liquid feed is of mucilaginous or fibrous nature. A further disadvantage of this type of liquid-gas nozzle is the “*downstream turbulence*” which causes the fine particles to be carried away to the atmosphere by the large gas flows used. This phenomenon has been termed as “*overspray*”; it tends to contaminate the atmosphere which is in close proximity to the nozzle, and demands expensive cleanup and tedious maintenance procedures (Sewell, 1987).

With the rotary and nozzle atomizers dominating in their spray applications and association with lab scale and commercial spray dryers, it is also important to understand the working principle of other types of atomizers: sonic and electrohydrodynamic atomizers. With the continuous advancements in this field, the future spray drying technology might be dominated by the use of these atomizers.

1.2.2.4 Ultrasonic atomizers

Ultrasonic atomization relies on an electromechanical device that vibrates at a very high frequency. Two piezoelectric disks, tightened between a mechanical amplifying element and a support element, constitute the electromechanical device of the ultrasonic atomizer. The fluid to be atomized initially passes over the surface of the vibrating piezoelectric disks, which sets ultrasonic vibrations within the liquid. The vibrations within the liquid cause molecules on the surface of the liquid to move about, disrupting the surface tension of the liquid. This creates areas on the surface of the liquid with reduced or no surface tension, which are very similar to holes in a sieve, and through which droplets of the liquid can escape (Loser, 2002; Pyo *et al.*, 2006; Fukumoto *et al.*, 2006). After bypassing the piezoelectric discs, the fluid passes through an amplifier, the tip of which is a resonant surface. On reaching the active resonant surface, a thin liquid film is formed. As the frequency of vibration approaches the resonance frequency, a square wave pattern forms onto the liquid surface. Further increase in the amplitude of vibration causes the droplet formation and its detachment from the liquid film (Figure 1.7).

As the pressure energy does for the pneumatic and hydraulic nozzle atomizers, it is the nozzle vibration frequency which aids the droplet fission in ultrasonic atomizers. In

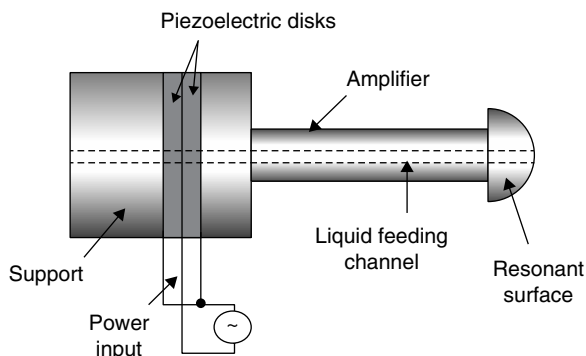


Figure 1.7 Ultrasonic atomizer [Dobre and Bolle, 2002].

addition to the vibration frequency, amplitude and the area of vibrating surface also play a role in ultrasonic atomization (Lixin *et al.*, 2004).

In contrast to conventional pressure nozzle atomizers, which impart a high initial velocity to the droplets, resulting in wider droplet distribution, the velocity of droplets emerging from the ultrasonic atomizer is one to two orders of magnitude smaller than the former. This is found to result in more uniform droplet size distribution (Lixin *et al.*, 2004). Consequently, the shorter residence time of the uniform droplets generated by the ultrasonic nozzle results in higher retention of the active components present in feed (Semyonov *et al.*, 2011). The low velocity spray also allows the spray drying chamber to be designed with shorter dimensions, thus enabling the ultrasonic atomizer system to be installed in a laminar flow cabinet or isolator (Freitas *et al.*, 2004). Furthermore, in an ultrasonic atomizer, the feed droplet outlet is larger, with no moving parts, and this arrangement serves to prevent clogging (Semyonov *et al.*, 2011) and facilitates easy maintenance and operation.

Because of the properties described above, ultrasonic atomizers have been effectively used for the drying of probiotic cells (Semyonov *et al.*, 2011), in order to obtain higher viability. However, ultrasonic atomization technology is effective only for low-viscosity Newtonian fluids. Since reduced pressure acts as the driving force for moisture evaporation from the atomized droplets, use of the ultrasonic spray head demands large quantities of hot air. Nevertheless, the use of sterile and hot drying medium would render this method appropriate for aseptic manufacturing of spray dried particles (Dalmoro *et al.*, 2012).

1.2.2.5 Electrohydrodynamic atomizers

A recent technique for atomizing the feed liquid is the use of electrospray or electrohydrodynamic sprays created by electrostatic charging. The mechanism has its roots in the Rayleigh's theory of instability and Taylor's theory. In the electrospray, electrical potential is applied to the needle to introduce free charge at the liquid surface. The high intensity of electric current applied between the two oppositely charged electrodes of an electrospray system enables the production of droplets of narrow particle size distribution. When the electrical potential rises to kilovolts, the liquid meniscus develops into a conical shape (Taylor cone), having a highly concentrated free charge. The free charge accelerates the droplets away from the needle due to the generated electric stress. Monodispersed particles will be formed when the jet breaks into fine particles due to varicose instabilities (Figures 1.8 and 1.9).

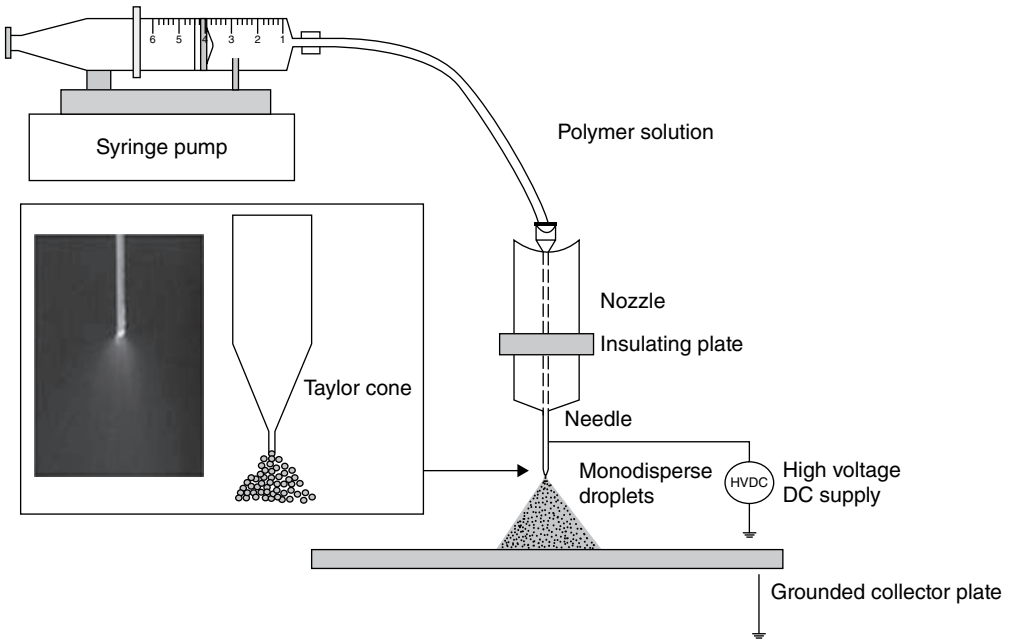


Figure 1.8 Mechanism of electrospaying. (Bhushani and Anandharamakrishnan, 2014. Reproduced with permission of Elsevier).

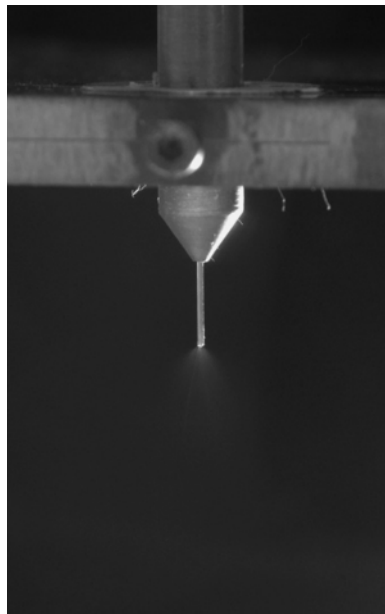


Figure 1.9 A visual of the spray emerging from an electrohydrodynamic atomizer.

The relationship between droplet size and conductivity is given by the Equation 1.4, after being confirmed by many experiments (Jaworek, 2007):

$$d_D = \alpha \left(\frac{Q^3 \varepsilon_0 \rho}{\pi^4 \sigma \gamma} \right)^{1/6} \quad (1.4)$$

where:

d_D is the droplet size;

Q is the flow rate;

ε_0 is the permittivity of vacuum;

ρ , σ and γ are the density, conductivity and surface tension of the feed liquid, respectively;

α is a constant which is generally equated to 2.9.

Requirement of solvents for feed preparation and extremely low flow rates limit the usage of electro-spray atomization for food applications and commercial exploitation respectively.

1.3 STAGE 2: SPRAY-AIR CONTACT

This stage, and the subsequent process steps of spray drying, constitute the particle formation phase. With the bulk feed atomized into tiny droplets, the next step is to bring the droplets into intimate contact with the hot gas. This enables rapid evaporation of moisture from the surface of all the droplets in a uniform manner. Here, the critical requirement is uniform gas flow to all parts of the drying chamber.

During spray-air contact, the droplets usually meet hot air in the spraying chamber, either in co-current flow or counter-current flow. In co-current flow (Figure 1.10(a)), the product and drying medium passes through the dryer in the same direction.

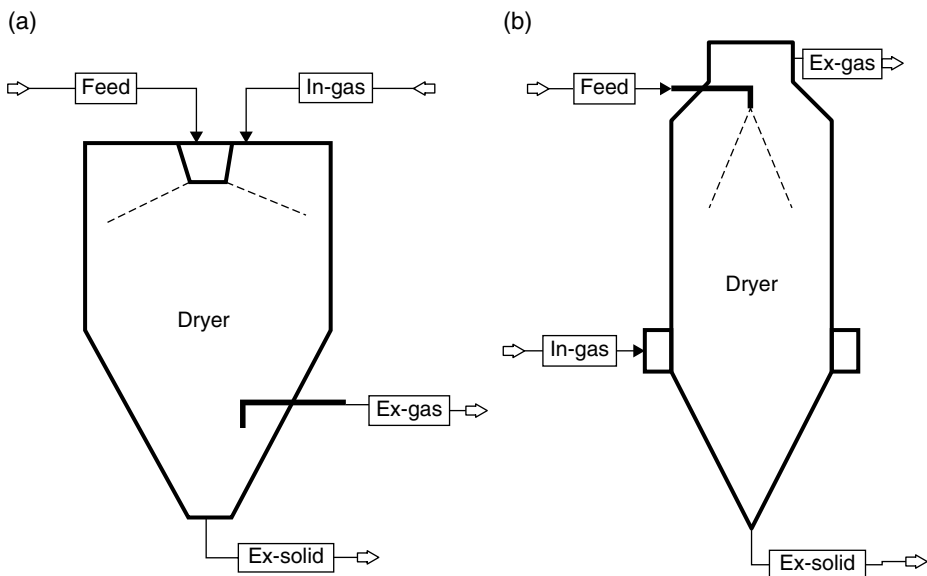


Figure 1.10 Spray dryer configurations: (a) co-current (left); (b) counter-current (right) (Oakley, 2004. Reproduced with permission of Elsevier).

In this arrangement, the atomized droplets entering the dryer are in contact with the hot inlet air, but their temperature is kept low due to a high rate of evaporation taking place and is approximately at the wet-bulb temperature. Wet-bulb temperature is the thermal energy of hot air used for evaporation (i.e., the removal of latent heat of vaporization from the air that cools it, and this is termed as “evaporative cooling”). This allows the particle to be maintained at a temperature below the outlet temperature of the drying air.) The cold air, in turn, pneumatically conveys the dried particles through the system. The contact time of the hot air with the spray droplets is only a few seconds, during which drying is achieved, and the air temperature drops instantaneously. This results in advantages of low temperature and low residence time of particles, with the added merit of less thermal degradation of heat sensitive products.

In contrast, in the counter-current configuration (Figure 1.10(b)), the product and drying medium enter at the opposite ends of the drying chamber. Here, the outlet product temperature is higher than the exhaust air temperature, and is almost at the feed air temperature, with which it is in contact. This type of arrangement is used only for heat-resistant products.

In another type, called mixed flow, the dryer design incorporates both co-current flow and counter-current flow. This type of arrangement is used for drying coarse free-flowing powder, but the drawback is the higher exit temperature of the product. The criteria for spray dryer design selection are summarized in Box 1.1.

Box 1.1 Ten guidelines on the choice of spray drying process parameters

1. The inlet temperature must be as high as possible in order to achieve a final product with low residual moisture and a higher thermal efficiency (choice of inlet temperature should take into account the heat sensitivity of the feed components to prevent thermal degradation).
2. Increasing the feed flow rate lowers the outlet temperature and thus increases the temperature difference between the inlet temperature and the outlet temperature. This results in product with higher residual moisture content.
3. High aspirator speed leads to higher degree of separation in the cyclone.
4. Lower aspirator speed leads to lower residual moisture content.
5. The higher the feed flow rate, the larger is the size of the particles in the final product.
6. The higher the feed concentration, the greater is the moisture content of the particles and, hence, the greater the possibility of agglomeration and the occurrence of irregular particle shapes.
7. The drying air temperature should be below the glass transition temperature in order to prevent product collapse and stickiness in the spray chamber.
8. The T_g of the feed material can be made higher for a convenient spray drying operation by the addition of high molecular weight components such as maltodextrin.
9. The percentage of water content in the feed is also a significant parameter in controlling the T_g , since water depresses T_g considerably.
10. A shorter residence time (RT) (10–15 sec) is recommended for fine particles containing an ample amount of free surface moisture content, enabling easy evaporation. A medium RT (25–35 sec) should be applied for fine to semi-coarse sprays that needs to be dried to low residual moisture content. A longer RT is needed for drying coarser sprays in order to achieve lower residual moisture content.

An air disperser to ensure uniform gas flow, and an appropriately designed drying chamber, are the important hardware elements associated with this step. The function of an air disperser is to create pressure drop by means of perforated plates or vaned channels, through which the gas is directed to facilitate equalized flow in all directions of the spray drying chamber. The air disperser is normally placed in the roof of the drying chamber, adjacent to the atomizer. The drying chamber usually has a conical bottom, with its height to diameter ratio (aspect ratio) determined by the end applications. The different types of drying chambers are discussed in later sections.

1.4 STAGE 3: EVAPORATION OF MOISTURE

The most critical step in particle formation, this process step is associated with the morphology of the final product. Evaporation of moisture during spray drying can be visualized as two stages:

- (i) constant rate period; and
- (ii) falling rate period.

Examining the drying kinetics of the spray drying process is critical in predicting the heat and mass transfer in the drying material. This can be best explained by a mathematical model for the evaporation of a single droplet which is subjected to convective drying in a spray dryer (Figure 1.8). Initially, when the droplet is exposed to hot gas, rapid evaporation takes place. During this exposure, the droplet is heated from its initial temperature (T_0) to the temperature of equilibrium evaporation temperature (T_{eq}) (Figure 1.11, AB). During this period, the removal of moisture follows the *constant rate period* of the drying rate curve as the moisture is removed constantly from the surface of the droplet keeping it sufficiently cool. The droplet surface remains saturated with moisture at this stage and its temperature is constant at the wet-bulb temperature (Figure 1.11, BC; Dolinsky, 2001).

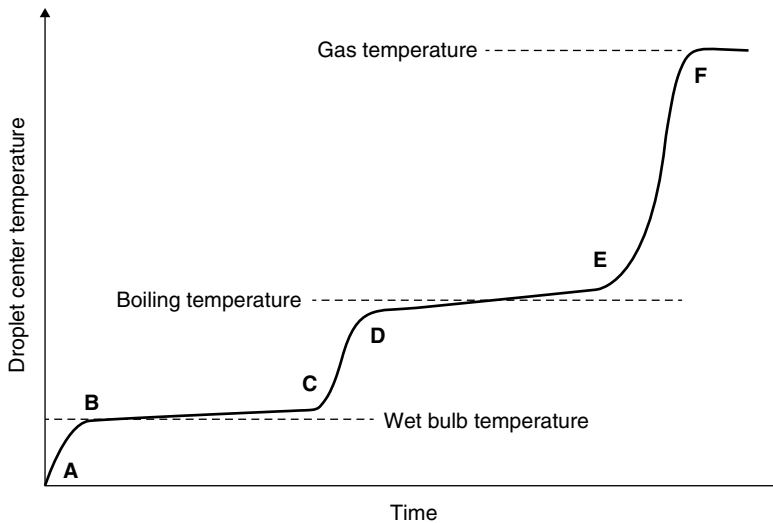


Figure 1.11 Temperature history during spray drying of a liquid droplet (Handscomb *et al.*, 2009. Reproduced with permission of Elsevier).

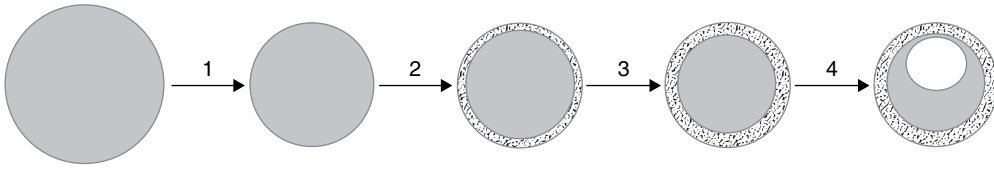


Figure 1.12 A diagrammatic representation of the droplet drying process (Modified from Charlesworth and Marshall, 1960; Walton and Mumford, 1999).

Wet-bulb temperature (T_{wb}) is the temperature that the drying gas reaches when it is saturated with vapor from the liquid (Seydel *et al.*, 2006). Also, the droplet shrinks due to the evaporation of the aqueous phase (Figure 1.12, step 1).

The quantification of evaporation rate at this stage can be understood by the “ d^2 law” (Law and Law, 1982). This is based on the fact that, during the constant rate period, the evaporation of a liquid droplet of diameter d is proportional to its surface area. Based on this law is the Peclet number (Pe) relationship given by the equation below (Equation 1.5). With this equation, Peclet number is depicted as the main controlling parameter of the droplet drying process and, hence, the particle formation (Huang, 2011):

$$\frac{\partial C}{\partial r} = Pe.C \quad (1.5)$$

where:

C is the concentration of the solute on weight by weight basis;

r is the droplet radius;

Pe is the Peclet number, which is the ratio of evaporation rate to diffusion rate (Equation 1.6).

$$Pe = \frac{\kappa}{D} \quad (1.6)$$

where:

κ is the evaporation rate;

D is the diffusion rate.

As the moisture removal from the droplet proceeds, the solute dissolved in the liquid reaches a concentration beyond its saturation concentration and tend to form a thin shell at the droplet surface described as “*crust formation*” (step 2, Figure 1.12).

The commencing of crust formation event is an important kinetic characteristic of the spray drying process as it transforms from low to high temperature drying. After the crust formation, the moisture removal turns into a *diffusion-controlled* process, and the evaporation rate is dependent upon the rate of water vapor diffusion through the dried surface shell (Figure 1.12, step 3; Farid, 2003). This constitutes the *falling rate period*. During the falling rate period, although the particle will begin to heat (Figure 1.11, CD), it is almost at the coolest part of the dryer, where the drying gas is at or near the outlet temperature of the dryer. Consequently, the particles are never heated above the outlet temperature of the dryer, despite the fact that the inlet temperature may be considerably higher. The final dried powder will be at a temperature approximately 20°C lower than the air outlet temperature (Gohel *et al.*, 2009).

An interesting phenomenon that happens during the falling rate period is “*bubble formation*” (Figure 1.12, step 4, and Figure 1.13). When the partial pressure of moisture vapor at the droplet centre exceeds ambient pressure, it results in bubble formation and a subsequent increase in temperature.

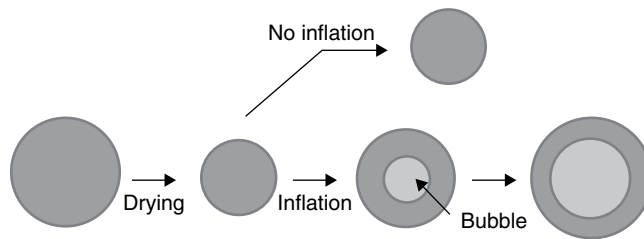


Figure 1.13 Schematic diagram of the bubble inflation phenomenon during spray drying (Etzel *et al.*, 1996).

A considerable amount of energy is required for this vaporization, which halts the sensible heating (Figure 1.11, DE). The droplet inflates to the outer radius and finally results in irregular randomly shaped particles (Figure 1.13; Etzel *et al.*, 1996). As mentioned above, a crust is formed as the moisture content decreases, and the droplet temperature ultimately rises towards the dry-bulb temperature of the air (Figure 1.11, EF). The varied morphologies of the spray dried particles resulting from the bubble inflation phenomenon are discussed in detail in the forthcoming section.

1.5 STAGE 4: PARTICLE SEPARATION

Two systems are employed in separating the product from the drying medium: the primary and secondary separation. Note that the spray drying chamber often has a conical bottom to facilitate the easy collection of the dried powder. During the primary separation, the dry powder is collected at the base of the dryer, followed by removal using a screw conveyor or a pneumatic system with a cyclone separator at the time of secondary separation. The gas stream loaded with the evaporated moisture is drawn from the centre of the cone above the conical bottom and is discharged through a side outlet. The relatively low efficiency of collection necessitates the use of an additional particle collection system, comprising dry collectors followed by wet scrubbers. The dry collectors include a cyclone separator, a bag filter and an electrostatic precipitator, depending on the size of the particles carried away by the exhaust gas and the final product specifications.

1.5.1 Cyclone separator

A cyclone separator, often integrated with a spray dryer, is a stationary mechanical device that utilizes centrifugal force to separate the solid particles from a carrier gas (Figure 1.14). It consists of an upper cylindrical part, referred to as the barrel, and a lower conical part, referred to as the cone. The gas stream, loaded with solid particles, leaving the spray dryer enters tangentially at the top of the barrel and travels downward into the cone, forming an outer vortex. The increasing air velocity in the outer vortex exerts a centrifugal force on the particles, separating them from the gas stream. When the gas stream reaches the bottom of the cone, an inner vortex is created, thus reversing its direction and exiting out at the top as clean gas. The particulates fall into the collection chamber attached to the bottom of the cyclone.

1.5.2 Bag filter

The bag filter (Figure 1.15) comprises a metallic housing designed for continuous operation and automatic cleaning. The particle-laden air enters under suction or pressure through the collector in the centre or bottom part (i.e. the hopper) of the bag filter. The air, with particles, travels through

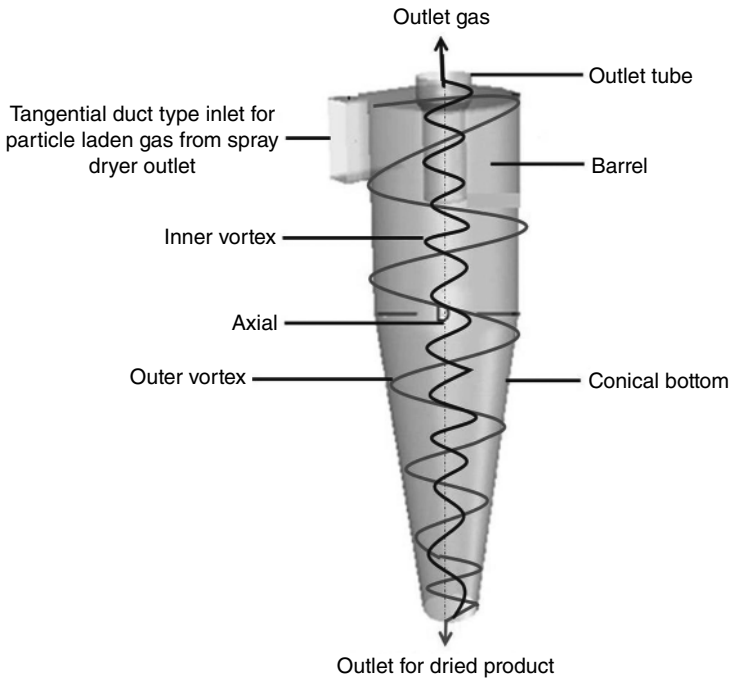


Figure 1.14 A typical cyclone separator (Utikar *et al.*, 2010. Reproduced with permission of InTech).

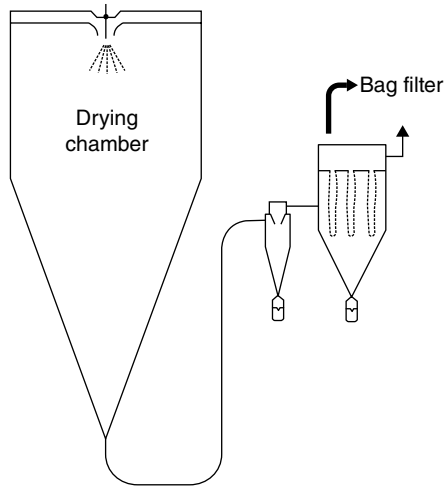


Figure 1.15 Schematic of spray dryer with bag filter (Lindeløv and Wahlberg, 2009. Reproduced with permission of Journal of Physics).

the filter bag, which retains the product particles on its surface. The clean air passes out through bags and plenum to the outlet of bag filter. Accumulation of dust on bags causes an increase in the differential pressure across the filter bags. Compressed air is pulsed by a timer-actuated series of normally closed pulse valves at preset intervals, causing the valves to open. The compressed air is stored in a reservoir located beside the higher filter chamber. Above each row of bags there is a tube with holes that are aligned with the central air passage gap, located on top of the bags,

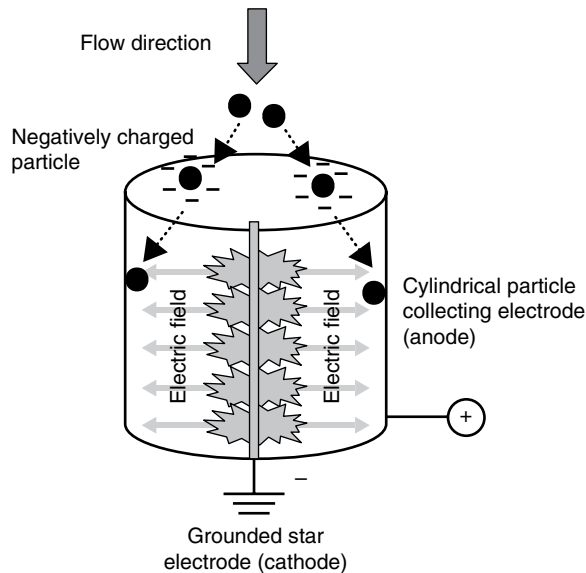


Figure 1.16 Schematic of the working principle of electrostatic precipitator (Lee *et al.*, 2011).

through which compressed air is injected to invert the gas flow momentarily. This causes the particulate material accumulated outside the bags to be removed.

1.5.3 Electrostatic precipitator

Electrostatic precipitation is a method of particle collection in spray drying that uses electrostatic force. An electrostatic precipitator (ESP) comprises of discharge wires and collecting plates (Figure 1.16). A high voltage is applied to the discharge wires to form an electrical field between the wires and the collecting plates. This high voltage ionizes the air around the discharge wires to supply ions. As the drying air that contains the product particles flows between the collecting plates and the discharge wires, the particles in the gas are charged by the ions. The Coulomb force caused by the electric field causes the charged particles to be collected on the collecting plates and the air is purified. The particles collected on the collecting plates are removed by rapping the collecting plates, scraping off with a brush or washing off with water, and removing from a hopper. Further discussion on ESP will be provided in Chapter 8.

The selection of particle separation equipment is governed by various factors such as collection efficiency, suitability for product handling, operational features, cost and space requirement.

1.6 MORPHOLOGY OF SPRAY DRIED PARTICLES

Particle morphology is an indicative signal which influences the decision on spray drying process parameters. Morphology affects the key quality characteristics of spray dried products such as particle size distribution, flowability, friability, moisture content and bulk and particle density. Hence, it is vital to understand the variables which decide the morphology, and the frequently occurring morphology patterns in the spray dried products.

Morphology is one delicate aspect of spray drying which makes it versatile as well as intricate. The literature shows it is possible to alter the morphology of spray dried particles by optimizing the process parameters. At the same time, quantifying and assessing the process variables influencing morphology is difficult, due to the complex interactions between the variables and unique drying patterns of different materials subjected to spray drying. In the case of spray drying, the interaction between variables such as inlet and outlet temperature, flow rate of drying gas, the feed properties constituting its concentration, solute diffusion coefficient and solvent latent heat, govern the final particle morphology.

Drying kinetics is central to the understanding of particle morphology (Vehring, 2008). The impact of different drying patterns on the product morphology is depicted in Figure 1.17. The crust formation stage is central to the particle formation during spray drying. Following the crust formation, the droplet may follow one of the two principal pathways, leading either to small, solid particles or large, hollow particles. The first is the “dry shell” route, which is similar to a shrinking core, producing particles which are susceptible to shattering when dried at high temperature. The second route is the “wet shell” type, which tends to form hollow particles which may inflate when subjected to higher drying temperature. Thus, the morphology of the spray dried particles also depends on the nature of the shell formed (Handscomb *et al.*, 2009). It is also apparent from the illustration that the drying temperature and solid content of the feed solution or suspension are the key factors in deciding the particle morphology.

The scope of further discussion is intended to provide an insight to the plausible morphological patterns of spray dried products, and the impact of major influential spray drying parameters on the below.

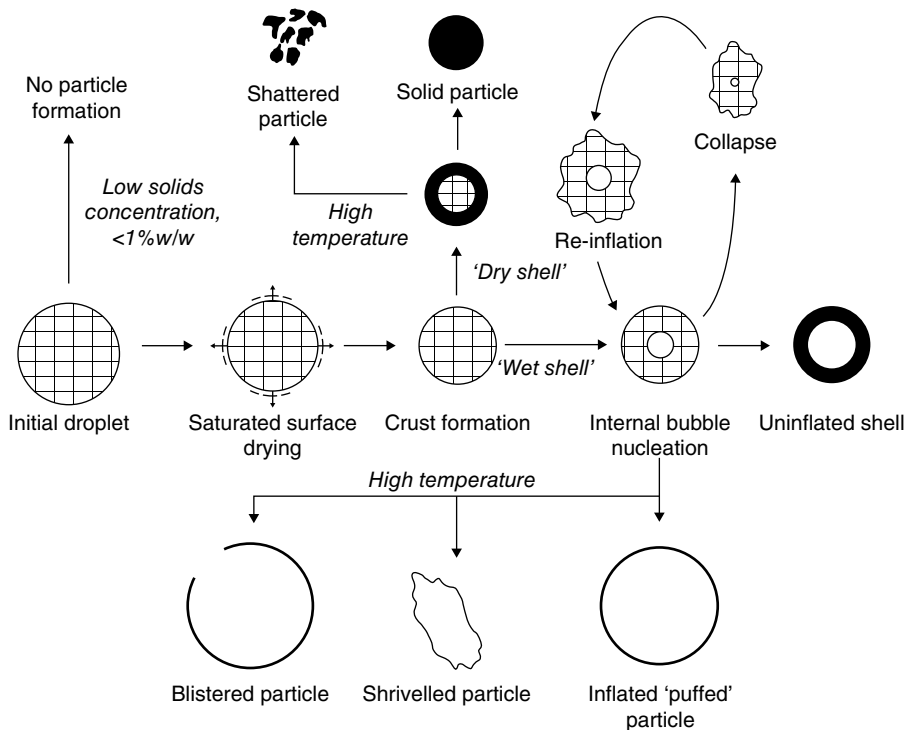


Figure 1.17 Different morphologies due to bubble inflation during spray drying (Handscomb *et al.*, 2009. Reproduced with permission of Elsevier).

1.6.1 Skin-forming morphology with hollow internal structure

A smooth skin-forming morphology (Figure 1.19(a)) can be described as a particle composed of a continuous non-liquid phase that is polymeric or sub-microcrystalline in nature. The skin-forming behavior is the result of solid precipitation which covers the droplet, entrapping the bulk of the droplet liquid inside. It is known that larger Peclet numbers result in an enlargement of the solute at the surface, which likely leads to shell or skin formation.

From Equation 1.6, at large Peclet numbers, the evaporation rate of the solute at the surface proceeds at a faster rate when compared to the diffusional motion of the dissolved molecules. The high evaporation rate, in turn, results in the rapid build-up of solute concentration at the surface. This leads to a local increase in viscosity, with subsequent skin or shell formation. In addition, the skin formation can also be promoted by the presence of surface active molecules that may accumulate at the surface and form a surface layer. The skin formation is important in maintaining the particle shape (Vehring *et al.*, 2007). It is also significant in the retention of volatile compounds during the spray drying process (Hassan *et al.*, 1996). The faster the rate of skin formation, the better the retention of sensitive compounds during spray drying.

The hollow internal structure of spray dried particles occurs as a result of substantially high evaporation rate experienced by small molecules with high solubility (ex. saccharides). Hollow particles also result from high drying gas temperatures of 200°C at the inlet and 90°C at the outlet, which corresponds to a Peclet number range of approximately 2–5 (Elversson *et al.*, 2003). The fate of the hollow particle depends on the thickness and mechanical properties of the skin surrounding it. Accordingly, it may eventually collapse or wrinkle.

The impact of drying air temperature on the formation of hollow internal structures can be best explained with reference to a study on the morphological differences of spray dried maltodextrin particles produced under two different conditions of inlet/outlet temperatures: 110°/74°C and 200°/173°C (Alamilla-Beltran, 2005). With an understanding of the d^2 law discussed in the previous section, the difference in morphology of the spray dried maltodextrin particles can be explained in terms of the Peclet number concept (Figure 1.18). At a low inlet-outlet temperature combination, Pe is low due to lower evaporation rate at a higher diffusion rate, resulting in smaller, solid particles. By contrast, at the higher inlet-outlet temperature combination, Pe is higher, resulting in larger, hollow particles. Furthermore, it is observed that there is no recognizable particle formation with feed of less solid content.

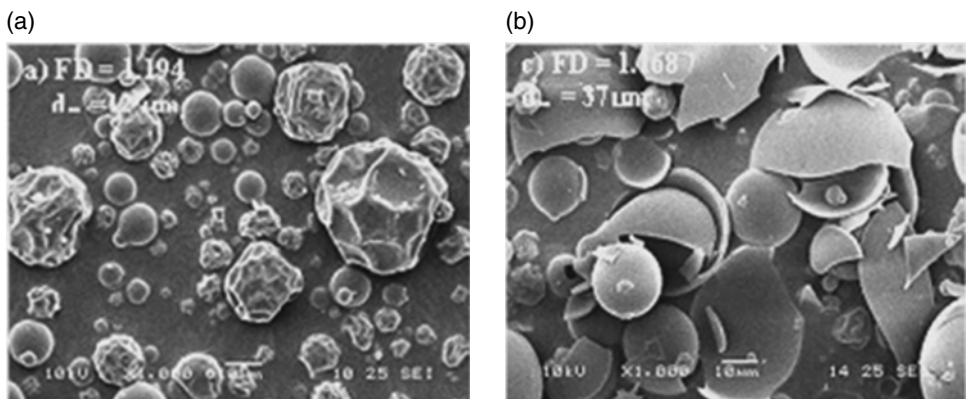


Figure 1.18 Difference in particle morphology due to different Peclet number as influenced by different inlet/outlet temperature combinations. (a) Peclet No. ≈ 0.7 . (b) Peclet No. ≈ 2.1 (Alamilla-Beltran *et al.*, 2005. Reproduced with permission of Elsevier).

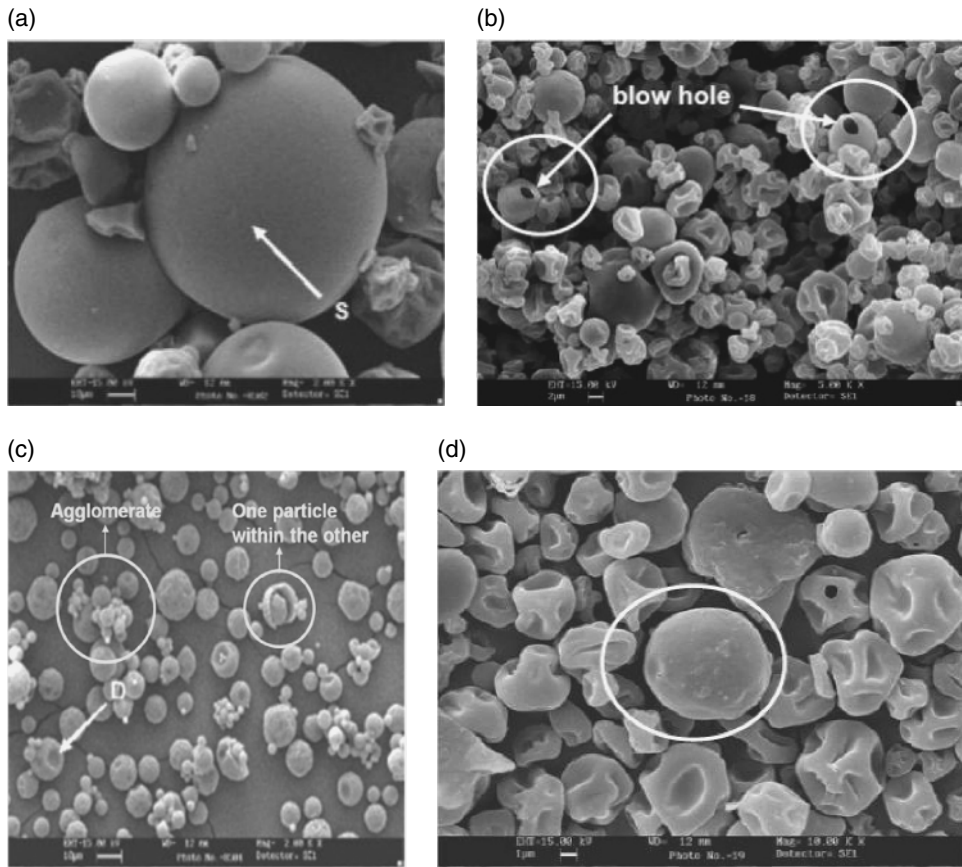


Figure 1.19 Different morphology patterns of spray dried particles. (a) Smooth surface (b) Blow-hole formation (c) Agglomerate (d) Dented surface. From: (b and d) Rajam *et al.*, 2012. Reproduced with permission of Elsevier; (a and c) Ezhilarasi *et al.*, 2013. Reproduced with permission of John Wiley & Sons.

It is difficult to predict at what solid concentration the particle formation becomes prominent during the spray drying process. However, an increase in feed concentration results in a particle which is resistant to disruption, bubble inflation and eventual collapse. Therefore, on an industrial scale, a pre-concentration step such as evaporation always precedes spray drying in order to feed an optimally concentrated solution to the spray dryer. This also enables efficient utilization of the limited thermal efficiency of a convective drying process such as spray drying.

Another commonly encountered feature in spray dried particle morphology is the formation of non-spherical, elongated particles. The shape deformation is mainly due to incomplete atomization, when the action of the viscous forces and surface tension on the bulk liquid to split it into droplets is not sufficient enough. Skin-forming materials are prone to this type of distortion, due to their pliability during drying.

1.6.2 Blow-hole formation

Blow-hole formation is a feature which is typical of spray dried particles (Figure 1.19(b)). Blow-holes are primarily due to the permeable nature of the particle, which allows the flow of water, water vapor and dissolved gases from the interior of the particle to its surface with minimal resistance.

Large particles dried at gas temperatures above the boiling point of the water may inflate after shell formation, due to build-up of internal gas pressure, potentially causing shell rupture or “blow-holes” in the shells (Walton and Mumford, 1999).

The formation of structurally rigid particle shell is favored by rapid drying and high particle mobility. This event occurs before the particles in the interior of the droplet come into contact with each other. Here, the drying rate is decreased, as the low permeability of the shell hinders the liquid flow to the surface. Eventually, the rigid shell structure leads to the formation of hollow granules that visually appears like craters or “blow- holes”, formed by an inward collapse of the shell. The inward collapse can be explained by the formation of a partial vacuum due to the capillarity-induced movement of particles from the interior to the surface shell (Oberacker, 2012).

1.6.3 Agglomerate

Structurally, an agglomerate can be defined as a particle composed of individual grains of material bound together by sub-micron dust or a binder (Figure 1.19(c)). Agglomeration between the particles inside the spray drying chamber occurs as a result of adhesion (solid link between the particles) and collision forces between them. The collision within the spray chamber can occur between two liquid droplets, or between a liquid drop and an already dried particle, or between two dry particles. The collision can also be between more than two dry particles. The adhesion depends upon the force of impact and contact time between the colliding particles. The relative speed of particles and the particle mass decide the impact of adhesion.

The agglomeration also depends upon the nature of particle surface, comprising of its composition, viscosity, temperature and water activity, all of which change during the spray drying process. As the droplets of the feed solution undergo drying, the water activity of the droplet surface decreases while its temperature increases. The viscosity of drop surface also continues to increase until it reaches a critical value (η_c), which is equivalent to 10^7 Pa.s. This critical viscosity is associated with stickiness, which eventually leads to the formation of viscous bridges between the particles to form agglomerate. η_c is also related to the glass transition temperature or stickiness temperature, which is explained in section 1.8.4.

In addition, the solubility of the material to be dried in water is also an influencing factor on spray dried particle agglomeration. Generally, insoluble and sparingly soluble solids in the feed result in agglomerates. To a certain extent, agglomeration is beneficial in ‘instant’, products, such as instant coffee, for achieving better wettability and dispersibility in water. However, excessive agglomeration may result in flow difficulties and severe caking problems (Barbosa-Cánovas *et al.*, 2005).

1.6.4 Formation of dented structure and presence of small particles within large particles

It is also interesting to learn about some unusual morphological patterns that are encountered during spray drying of some food components. One is cap-shaped, slightly dented structures (Figure 1.19(d)), and the other is the presence of small particles within large particles (Figure 1.19(c)), which are usually noticed in spray dried coffee. The phenomenon of smaller particles inside larger particles can be due to the entry of former into the latter through blowholes, or it may possibly occur where many smaller particles combine to form large particles during drying.

The formation of dented or cap-shaped structures is attributed to several causes by different works published in this context. Crosby and Marshall (1958) ascertain that the cap-shaped particles result from capillary action of the dried particle surfaces drawing the liquid and solids

outward and uniformly around the droplet. This creates sub-atmospheric internal pressures, which eventually result in the collapse of the particles. It may also be due to incorrect choice of inlet drying air temperature and insufficient solid content in the feed solution, resulting in deficient drying of the feed droplets. Consequently, the wet particles impact on the walls of the drying chamber which, in the course of the process, deforms on impact and collapses to result in the cap-shaped, dented structures. Yet another reason is that the higher drying rate leads to rapid wall solidification prior to expansion of the particles, which forms a strong crust on the particle surface and restricts the complete bubble inflation, thus resulted in caved-in or dented surfaces (Anandharamakrishnan *et al.*, 2007). However, surface dents result from a complex interaction between several factors, such as the drying rate, atomization mechanism and uneven shrinkage at early stages of drying, viscoelastic properties of the wall materials and rapid wall solidification.

Thus, an observation of the particle morphology, along with drying kinetics may enable the experts involved in product development to arrive at optimum process settings to achieve desired product properties and quality.

1.7 SPRAY-DRYING PROCESS PARAMETERS AND THEIR INFLUENCE ON PRODUCT QUALITY

It is obvious from the previous sections on the spray drying process steps that spray drying is a method which strongly and equally depends on material properties, equipment design and setting of operation parameters. The above listed factors influence the final product quality, mainly in terms of residual moisture, particle size and morphology. Although the optimization of these variables is usually achieved by a “trial and error” approach, an understanding of the basic guidelines of spray drying would result in intelligent operation of the equipment. The different spray drying process parameters, corresponding to each of the four steps of spray drying, are discussed in the following section. General guidelines for deciding the parameters for a spray drying operation and information related to its impact on product quality are also listed.

1.7.1 Atomization parameters

The performance of atomizers depends on three parameters: the atomization pressure, feed flow rate, viscosity and density. These parameters have an influence on the droplet size.

1.7.1.1 Atomization pressure

With the same type of nozzle and feed material, as the atomization pressure is increased, droplet size decreases according to the relationship below (Equation 1.7; Masters, 1991):

$$\frac{D_2}{D_1} = \left(\frac{P_2}{P_1} \right)^{-0.3} \quad (1.7)$$

Where D_1 and D_2 are the initial and final droplet sizes on changing the atomization pressure from P_1 to P_2 , respectively. This is due to the requirement of an efficient delivery of the atomization energy to the bulk liquid to drive droplet fission.

1.7.1.2 Feed flow rate

At constant atomization pressure, increasing the flow rate increases the droplet size, since the nozzle's hydraulic energy has to atomize more liquid. As the liquid interaction with the atomization energy is minimal, the droplet fission is insufficient to reduce its size. Feed rate is associated with the peristaltic pump speed which feeds to the spray solution to the rotary or nozzle atomizer.

As the atomization pressure is for the nozzle atomizers, wheel rotation speed and diameter are for the rotary atomizers. The wheel rotation speed and wheel diameter have an inverse relationship to the droplet size.

1.7.1.3 Feed viscosity

The feed viscosity has a direct relationship to droplet size. As the feed viscosity increases, the atomization energy supplied to the nozzle must overcome large viscous forces to achieve smaller droplet sizes. Viscous forces tend to reduce the energy available for breaking the droplets, thus resulting in larger droplets. The equation describing the relationship between droplet size and feed viscosity (Equation 1.8) is given below. The same principle also applies to that of feed density.

$$\frac{D_2}{D_1} = \left(\frac{\mu_2}{\mu_1} \right)^{-0.2} \quad (1.8)$$

Where D_1 and D_2 are the initial and final droplet sizes on changing the feed viscosity from μ_1 to μ_2 , respectively.

1.7.1.4 Feed surface tension

As described in the principle of atomization, surface tension of the liquid plays an important role in the extent of atomization. To achieve atomization, the atomizer should overcome the surface tension of the feed liquid. Hence, a liquid having higher surface tension is difficult to atomize. This necessitates the preparation of an emulsion with the aid of emulsifier addition, and a homogenization step prior to spray drying in certain cases, in order to combat the surface tension of a feed, especially that involving multiple components. In addition to the knowledge on atomization parameters, the rationale for atomization selection is provided in Table 1.2.

Table 1.2 Criteria for selection of spray dryer design (Modified from Masters, 1991).

Criteria	Selection
Fine-particle product (30–120 μm).	Co-current flow with a rotary atomizer.
Low product temperature.	
Coarse-particle product (120–250 μm).	Counter-current nozzle tower design with pressure nozzle atomizer.
High product temperature to be reached for porosity and bulk density.	
Coarse product (120–250 μm).	Mixed flow nozzle chamber
Product temperature to below, but the product is not so heat-sensitive and can tolerate a certain degree of heat treatment.	

1.8 PARAMETERS OF SPRAY-AIR CONTACT AND EVAPORATION

An important parameter involved in this process step is the spray angle, which is measured at the nozzle orifice and is related to the nozzle's liquid tangential velocity. The liquid tangential velocity is the speed at which the feed liquid spins inside the nozzle before it is divided into fine droplets and sprayed into the drying chamber. Widening the spray angle increases this velocity to reduce the droplet size. The choice of spray angle is related to the type of air flow, namely co-current or counter-current, with the wider angle being used with the former and the narrow angle with the latter. In the co-current operation, the downward-flowing air narrows down the spray angle. In the counter-current operation, the spray moving downward is widened by the upward-flowing air. The other important parameters in this step are the aspirator rate, air humidity, inlet and outlet air temperature, the glass transition temperature, and the residence time of particles in the spray dryer. Each of these is described below. A brief guideline on the selection of these parameters and their influence on final product is presented in Box 1.1.

1.8.1 Aspirator flow rate (or speed)

The aspirator in the spray dryer is associated with the supply of drying air to the spray chamber by the aspirator motor under pressure conditions. By altering the aspirator flow rate, the amount of heated drying air entering the spray chamber can be regulated.

1.8.2 Inlet temperature

Inlet temperature is the temperature of the heated drying air. The inlet temperature is significant in cooling the atomized feed droplets to their wet-bulb temperature. In addition, it has a direct relationship to the wet-bulb temperature of the surrounding hot air (Oldfield *et al.*, 2005; Figure 1.20). Inlet temperature is often associated with dryer evaporative capacity and thermal efficiency. Therefore, higher inlet temperature is favorable in terms of achieving higher throughput of spray dryers. However, a lower value of the inlet temperature reduces the wet-bulb temperature of the surrounding hot air, and prevents the degradative losses of the active compounds during the initial

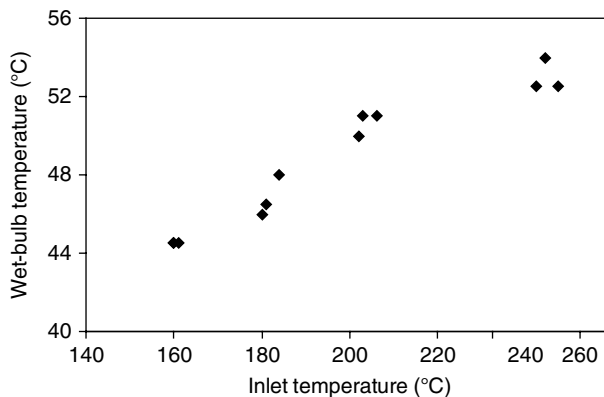


Figure 1.20 Influence of spray dryer inlet temperature on the wet-bulb temperature of the gas (Anandharamakrishnan, 2008).

stages of spray drying. The trade-off between the aforementioned circumstances is important in deciding an optimal inlet temperature for the spray drying process.

1.8.3 Outlet temperature

The temperature of the air laden with solid particles before entering the cyclone is defined as the outlet temperature. This temperature is a result of the heat and mass balance in the drying cylinder, and therefore cannot be regulated. Striking the optimum temperature difference between the inlet and outlet temperature is most critical in a spray drying process.

Outlet temperature correlates with the final moisture content and surface topography of the final product (Maas *et al.*, 2011). For instance, operation at high outlet temperature is carried out to achieve high moisture content to obtain agglomerated “instant” powdered products. This is because higher outlet air temperature promotes rapid crust formation while the drying of the inner core is still not complete. Furthermore, an increase in the outlet temperature also increases the particle temperature by shifting the wet-bulb lines on the psychrometric chart. When the inlet and outlet air temperatures are known, it is possible to calculate the particle temperature using the above relationship. Figure 1.21 illustrates an example of estimating the temperature of whey protein particles using a psychrometric chart.

The wet-bulb temperature is constant along the dotted lines. Curved lines of constant water activity, a_w , are also shown in Figure 1.21, in which a value of 5.2% corresponds to the relative humidity of the outlet gas and that of 16% is the equilibrium relative humidity corresponding to the whey product bulk moisture content, obtained from its moisture

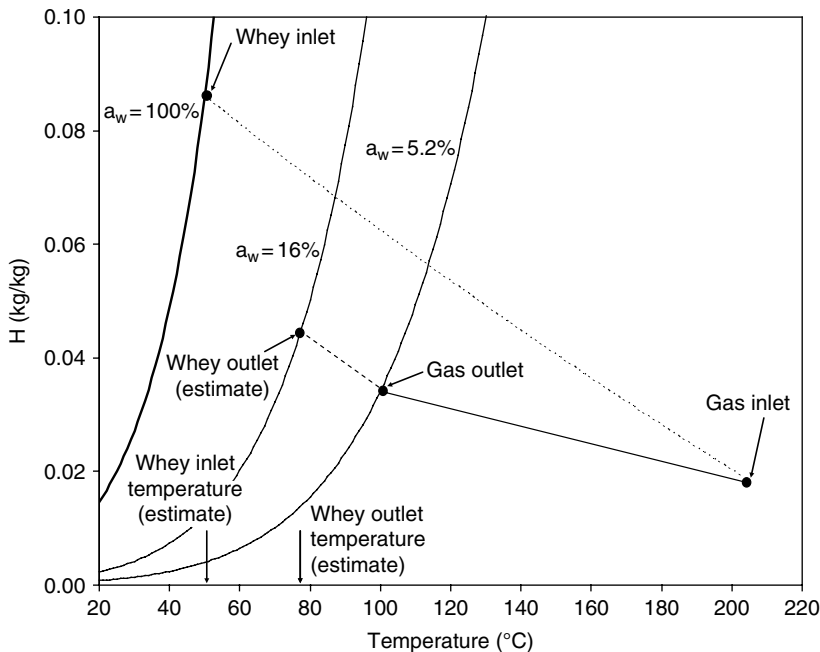


Figure 1.21 Psychrometric chart showing typical gas and particle inlet and outlet conditions (Anandharamakrishnan, 2008).

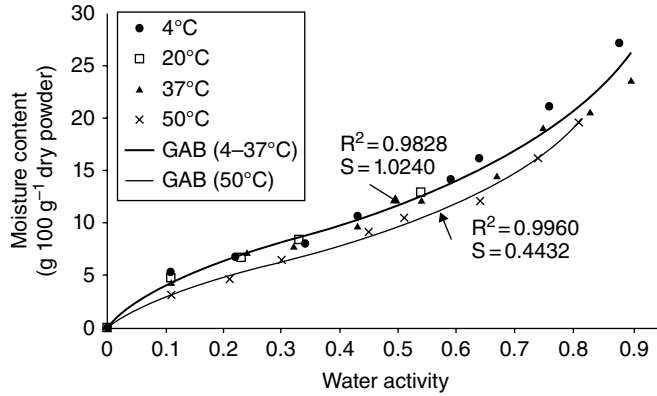


Figure 1.22 Moisture sorption isotherm of a WPI powder (Guggenheim-Anderson-De Boer (GAB) sorption isotherm model data) (Foster *et al.*, 2005. Reproduced with permission of Elsevier).

sorption isotherm (Figure 1.22). The outlet particle temperatures are then calculated from wet-bulb lines using the following relationships:

Mass transfer rate from particle:

$$-m^* = k_c A_p (Y_s^* - Y_{air}) \quad (1.9)$$

Heat transfer rate from particle:

$$-h_{fg} m^* = h A_p (T_{air} - T_p) \quad (1.10)$$

where:

k_c is the mass transfer coefficient (m/s);

A_p is the surface area of the particle (m²);

h is the heat transfer coefficient (W/m² K);

Y_s^* is the water activity at surface of the particles;

T_{air} and T_p are the temperatures of the surrounding air and particle respectively.

Combining the equations (1.9) and (1.10):

$$h A_p (T_{air} - T_p) = k_c A_p h_{fg} (Y_s^* - Y_{air}) \quad (1.11)$$

Hence, wet-bulb line is:

$$Y_s^* - Y_{air} = \frac{h}{h_{fg} k_c} (T_{air} - T_p) \quad (1.12)$$

The outlet particle temperature is then estimated by following the wet-bulb lines from the gas outlet conditions to the relative humidity line corresponding to the equilibrium relative humidity (ERH) on the surface of the whey powder. The ERH of the whey powder is obtained from its water activity ($a_w = \text{ERH}/100$), determined from the moisture sorption isotherm of whey protein isolate powder (Figure 1.22) at 50°C, reported by Foster *et al.*, (2005) using the measured product moisture content.

However, the above method of estimation for particle outlet temperature assumes that there is no significant effect of temperature, and the measured moisture content is representative of the surface moisture content as the particles exit the spray dryer. But, in reality, the surface moisture content will always be lower than the average moisture content and thus the estimated particle outlet temperatures would be lower than the actual value.

1.8.4 Glass transition temperature (T_g)

Glass transition is a feature of second-order time temperature dependent transition, which is characterized by a discontinuity in physical, mechanical, electrical, thermal, and other properties of a material (Rahman, 1995). T_g is the temperature above which the matrix shifts from the structurally rigid glassy state to a rubbery state, which is associated with product stickiness on the spray chamber wall in spray drying. Stickiness is considered to be the major process challenge in spray drying. It leads to product agglomeration and poses problems of caking and lumping of the product during packaging of the spray dried products. The T_g of feed depends on the constituent solutes present in the feed. It is given by the Gordon-Taylor equation (Equation 1.13), which applies to a multi-component system:

$$T_g = \frac{w_1 T_{g1} + cw_2 T_{g2}}{w_1 + cw_2} \quad (1.13)$$

where:

w_1 and w_2 are mass fraction of solute and water;

T_{g1} and T_{g2} are glass transition temperatures (K) of solute and water (138 K), respectively

c is the ratio of specific heat change of solute to water at the glass transition temperature.

Further discussions on the relevance of T_g in spray drying process are presented in detail in Chapter 4.

1.8.5 Residence time of particles in the spray chamber

This parameter is important from two perspectives – namely, with respect to complete drying of feed droplets to achieve optimum product specifications, and in the control of particle temperature to minimize aroma loss and thermal degradation of heat sensitive materials. The particle residence time (RT) also affects product quality indices such as solubility and bulk density. RT is divided into two parts: primary and secondary residence times. The primary RT is calculated from the time taken for droplets leaving the nozzle to impact on the wall or leave at the outlet. The secondary residence time can be defined as the time taken for a particle to slide along the wall from the impact position to the exit (Kuriakose and Anandharamakrishnan, 2010). While prediction of RT in a spray chamber is experimentally difficult, recent advancements in modeling and powerful computational simulation techniques aid in efficiently calculating the RT.

1.9 TYPES OF SPRAY DRYER

Spray dryers can be classified into different types, based on different criteria. They are categorized into co-current dryers, counter current dryers and mixed flow dryers, based on the direction of air and feed flow; as open cycle, closed cycle and semi-closed cycle dryers, based on whether

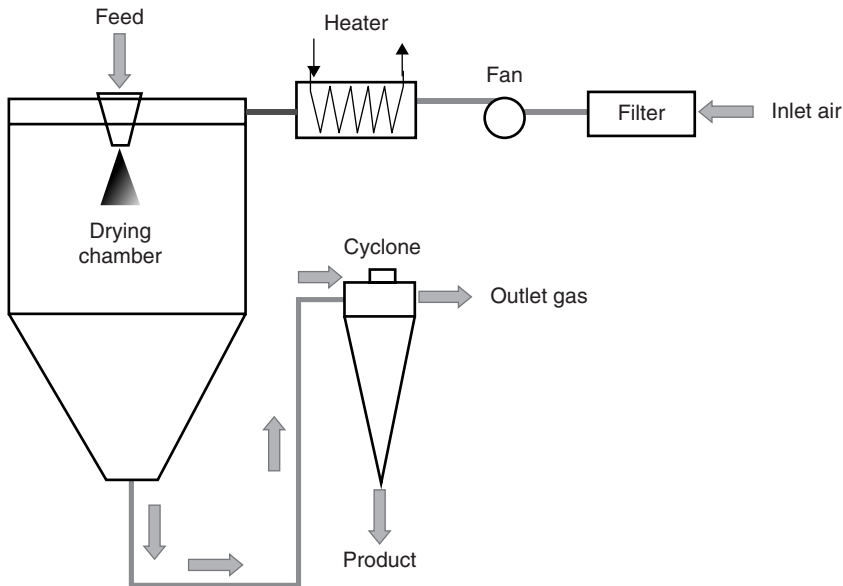


Figure 1.23 Schematic of an open cycle spray dryer.

the heating medium is recycled/reused; as single-stage and two-stage dryers, depending on the number of passes before the dried product is separated; and as short-form and tall-form, based on the aspect ratio. Each of the above categories of spray dryer is briefly described as below:

1.9.1 Open cycle spray dryer

This is the standard layout of spray dryer, with wide usage. It involves drawing the drying air from the atmosphere, heating and conveying it through the chamber once, and then exhausting it back to the atmosphere (Figure 1.23). The variations in this layout could be with respect to the type of separation equipment used (i.e. use of cyclone separator, bag filter or electrostatic precipitator).

1.9.2 Closed cycle spray dryer

Closed cycle dryers work on the principle of recycling and reusing the gaseous medium, which is usually a relatively inert gas such as nitrogen, or air in special cases (Figure 1.24). A closed cycle dryer is used when the feedstock is prepared by dissolving the solids in flammable solvents, in order to reduce the explosion risk and to obtain a complete recovery of the solvent. When the spray dried product is susceptible to oxidation, then a closed cycle dryer is the most appropriate choice (Gohel *et al.*, 2009). It is also advantageous to use this type of dryer when a toxic feed is subjected to drying, and the resultant pollution due to noxious vapors and undesirable particulate emissions or odor are not permitted by the regulatory norms. However, these conditions are normally not encountered in a food industry.

1.9.3 Semi-closed cycle spray dryer

This dryer design is a cross between open and closed cycle dryers. A direct-fired heater is used, and the air entering the system is limited to that required for combustion. An amount of air equal to the combustion air is bled from the system at the other end of the process. The gas (mainly

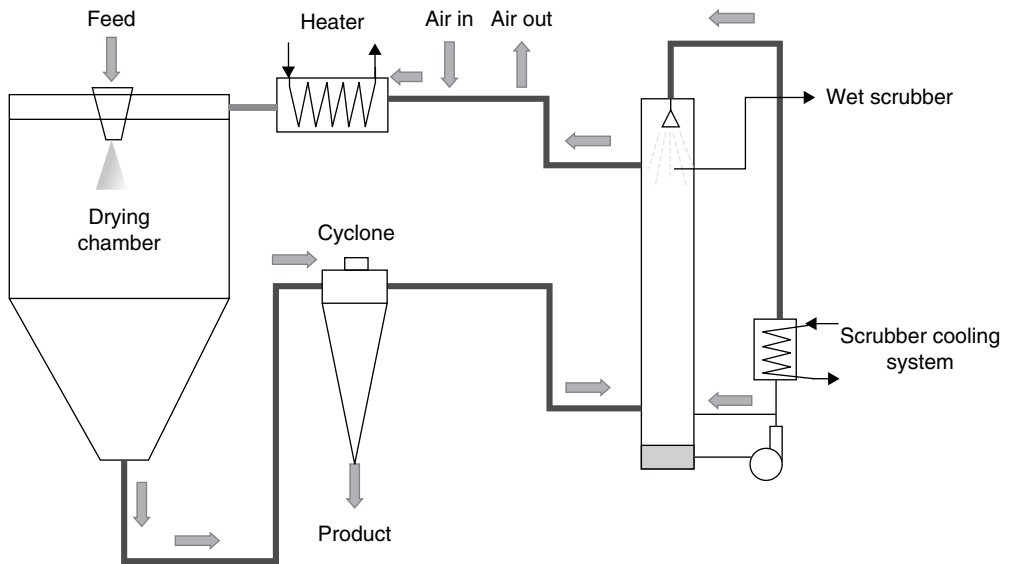


Figure 1.24 Schematic of a closed cycle spray dryer.

products of combustion) is recycled through the dryer. The recycled gas has very low oxygen content, making it suitable for materials that cannot be exposed to oxygen due to explosive hazard or product degradation (Anundhia *et al.*, 2011).

1.9.4 Single-stage spray dryer

The single-stage configuration is the most predominantly used spray dryer design. In a single-stage dryer, the moisture is reduced to the target level in a single pass through the spray chamber. A substantial reduction in weight (about 50%), volume (about 60%) and diameter (about 25%) of the initial particle has been reported during single stage-spray drying (Westergaard, 1994). The single-stage spray dryers operate at an inlet temperature of 150–200°C and an outlet temperature of around 95°C (Early, 1998). However, if lower residual moisture content is expected of the final product, then the relative humidity of the outlet air should be lower and, hence, the outlet temperature should be higher. The resultant particle temperature would therefore be higher, which is not suitable for heat-sensitive products. Furthermore, warmer temperatures would possibly cause the particles to stick together with very weak bindings to form large and loose agglomerates. At extremely high outlet air temperatures, blow-hole formation might occur, as discussed earlier (Westergaard, 2010).

1.9.5 Two-stage spray dryer

The two-stage spray dryer is considered to be a development over single-stage spray dryers. In single-stage spray dryers, the need for a higher outlet temperature to achieve lower residual moisture content might be detrimental to the final product quality. In order to overcome this limitation, in a two-stage spray dryer, the required moisture content is attained in two stages. After leaving the spray chamber, the moisture content of the particles from the first stage, which is typically at the level of 5–10%, is further reduced during a second stage, accomplished in a fluidized bed dryer (Figure 1.25). Two-stage spray dryers are therefore

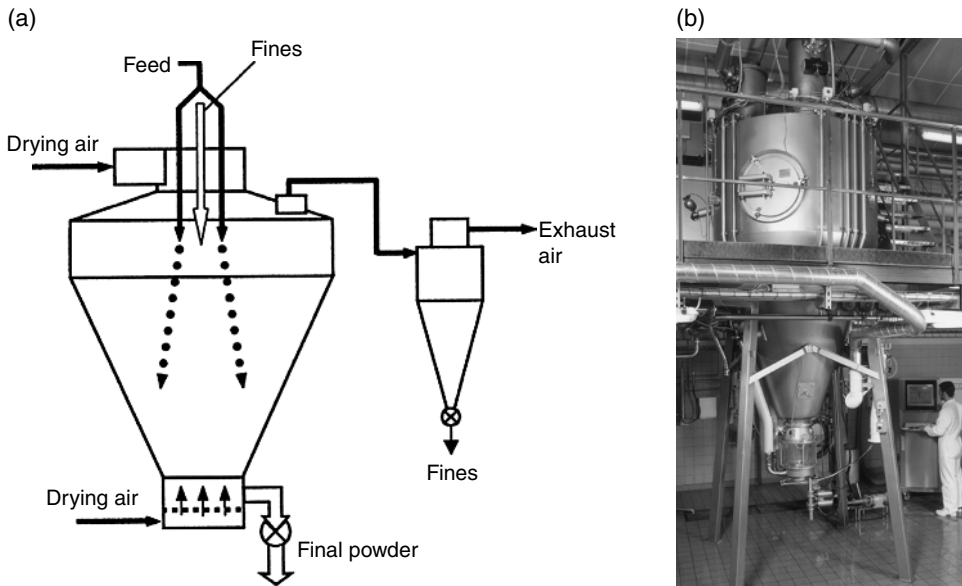


Figure 1.25 (a) Schematic of two stage spray dryer with its conical bottom attached to fluidized bed. (b) Pilot scale integrated fluidized bed spray dryer (Schwartzbach and Masters, 2001. Reproduced with permission of Taylor and Francis).

suitable for heat sensitive products because of their comparatively low temperature in each of the two stages relative to those used in a single-stage spray dryer.

However, in order to combat the lower diffusion coefficient for moisture removal during the second stage, the fluidized bed dryer should provide sufficient residence time for the particles to achieve the required final moisture content. The advantages of two-stage spray dryers include higher evaporation capacity per unit weight of drying air, lower operation cost, better quality products and lower powder emission (Westergaard, 2010).

1.9.6 Short-form

Short-form dryers have height-to-diameter ratios (aspect ratio) of around 2 : 1. The short-form dryers (Figure 1.26(a)) are the most widely used, as they accommodate the comparatively flat spray disk from a rotary atomizer (Masters, 1991). The flow patterns observed in short-form dryers are more complex than those in tall-form dryers, with many dryers having no plug-flow zone and a wide range of gas residence times (Langrish and Fletcher, 2001). A short-form dryer with a bottom outlet is more suitable for drying of heat-sensitive materials, such as proteins, due to the low amounts of recirculated gas and, hence, shorter residence time of the particles (Anandharamakrishnan *et al.*, 2008).

1.9.7 Tall-form

Tall-form designs are characterized by height-to-diameter aspect ratios of greater than 5 : 1 (Figure 1.26(b)). The spray angle is confined to the smaller diameter of the chamber, and nozzle atomizers are often used in combination with tall-form dryers. The greater aspect ratio also contributes to the longer residence time of the particles in the drying chamber. Computational fluid dynamics (CFD) simulation studies on tall-form spray dryers have indicated that more than

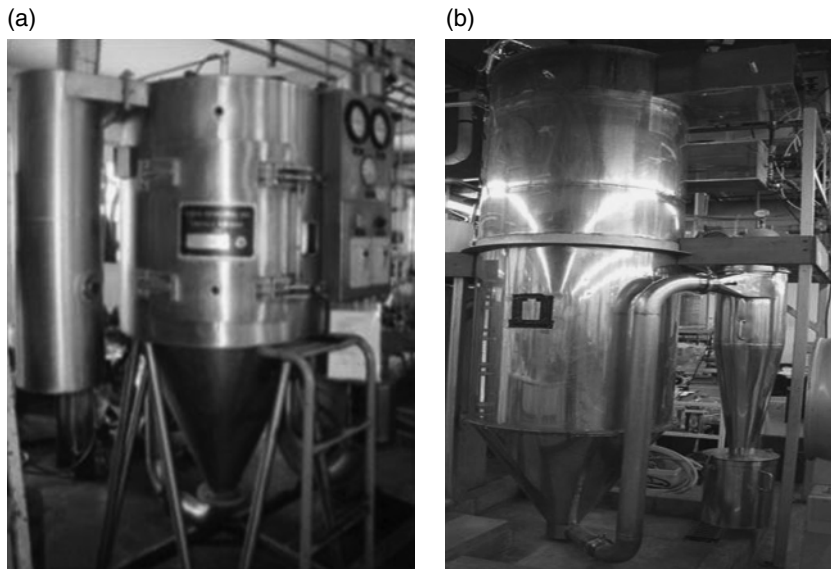


Figure 1.26 (a) Short-form; (b) Tall-form spray dryer.

Box 1.2 Advantages of spray drying

- The ability to be operated as fully automated control system, enabling continuous monitoring and recording of all the associated process variables simultaneously.
- A variety of spray dryer designs to meet different product specifications.
- Few moving parts and no moving parts are in direct contact with the product, minimizing corrosion problems.
- The ability to handle varied nature of feedstock, with the only mandate being that it has to be pumpable (solution, slurry, paste, gel, suspension or melt form).
- The possibility of obtaining tailor-made particle size and bulk density, depending on end product specifications, by optimization of nozzle design and operation parameters.
- Unique morphology of predominantly spherical particles, with uniform size and hollow structure, thus reducing the bulk density of the product.

60% of the particles impact on the cylindrical wall, which has an adverse effect on the product quality due to the solids adhering to the wall for appreciable times, drying out and losing their wet-bulb protection (Anandharamakrishnan *et al.*, 2008).

1.10 APPLICATIONS AND ADVANTAGES OF SPRAY DRYING

The major advantages and applications of spray drying are presented in Box 1.2 and Figure 1.27, respectively. The applications of spray drying have been categorized as food and non-food applications in Figure 1.27. The exclusive food applications of spray drying are presented in Table 1.3.

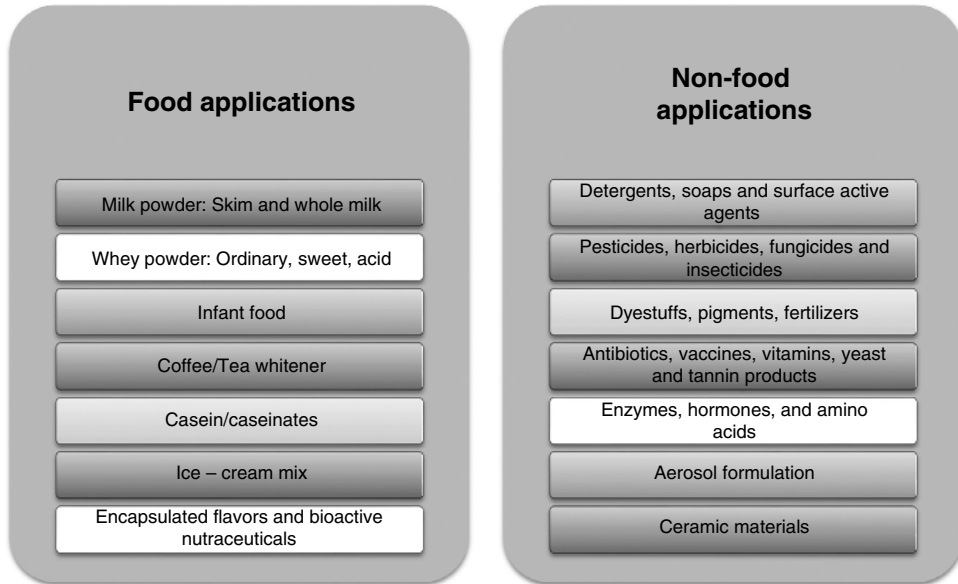


Figure 1.27 Applications of spray drying.

Table 1.3 Food applications of spray drying.

Application	Reference
Dairy products (whole milk, skim milk, whey protein)	Ye <i>et al.</i> (2007); Fang <i>et al.</i> (2012); Kim <i>et al.</i> (2009); Fu <i>et al.</i> (2012); Anandharamakrishnan <i>et al.</i> (2008)
Egg-based products	Kincal (1987); Rannou <i>et al.</i> (2013)
Instant coffee and tea	Yu <i>et al.</i> (2012)
Flavoring compounds	Bayram <i>et al.</i> (2005); Gharsallaoui <i>et al.</i> (2012); Yoshii <i>et al.</i> (2001); Soottitantawat <i>et al.</i> (2005).
Meat and fish products	Kurozawa <i>et al.</i> (2009); Zhang <i>et al.</i> (2013); Anwar and Kunz, (2011)
Fruit and vegetable products	Kha <i>et al.</i> (2010); Goula and Adamopoulos (2005); Fazaeli <i>et al.</i> (2012)
Edible protein products	Ortiz <i>et al.</i> (2009); Wang <i>et al.</i> (2013)
Corn products	Zhong and Jin (2009)
Wheat based products for bakery purpose	Liao <i>et al.</i> (2013)
Sugar products	Jayasundera <i>et al.</i> (2011); Truong <i>et al.</i> (2005); Adhikari <i>et al.</i> (2009); Cano-Chauca <i>et al.</i> (2005)

Thus, in a nutshell, the resourcefulness of spray drying lies in its capacity in terms of continuous operation, wide range of operating temperatures, short residence times, production of a powdered product, suitability for both heat-sensitive and heat-resistant materials, rapid drying and flexibility in capacity design (feed rates range from a few pounds per hour to over 100 tons per hour), to list just a few. In the following chapters, the spray drying technique is introduced to readers in its all-new dimension as an encapsulation process for food ingredients. The fundamental aspects of spray drying covered in this chapter are presented with an intention to assist readers to further relish the “*language of spray drying*” in the new accent of “*encapsulation*” in the forthcoming chapters.

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