

# Particle Coating in Fluidized Beds

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## Nomenclature

$C_0$	Initial moisture content, kg water/kg dry solid	$M_{CP}$	Weight of the coated particles, g
$C_t$	Moisture content at any time $t$ , kg water/kg dry solid	$M_{UP}$	Weight of the uncoated particles, g
$C_e$	Moisture content at equilibrium, kg water/kg dry solid	$M_{CM}$	Weight of the coat material sprayed into the coater, g
$d_0$	Initial droplet diameter, m	$P_{at}$	Atomization pressure, bar
$d_t$	Droplet diameter at any time $t$ , m	$R$	Droplet radius, m
$d_d$	The mean diameter of droplets, $\mu\text{m}$	$t_{eva}$	The evaporation time, s
$d_p$	The particle diameter, m	$T_{bed}$	Bed temperature, $^{\circ}\text{C}$
$d_{VM, solution}$	Volume mean droplet diameters of solution, m	$T_{inlet}$	Inlet air temperature, $^{\circ}\text{C}$
$d_{VM, solvent}$	Volume mean droplet diameters of solvent, m	$\Delta T$	Difference between the air temperature and droplet surface temperature, K
$D_{eff}$	Effective moisture diffusivity, $\text{m}^2\text{s}^{-1}$	$U_{sol}$	Fluid volumetric flow rate, $\text{m}^3\text{s}^{-1}$
$g$	Gravity acceleration, $\text{m s}^{-2}$	$U_{at}$	Air volumetric flow rate, $\text{m}^3\text{s}^{-1}$
$k$	Thermal conductivity of fluidized air, $\text{W m}^{-1}\text{K}^{-1}$	$v_{REL}$	Outlet air velocity (the velocity of air relative to liquid at the atomizer nozzle exit), $\text{m s}^{-1}$
		$w_m$	Necessary work of wetting per unit area, $\text{J m}^{-2}$
		$Y$	Coating yield

**Symbols**

$\mu_f$	Fluid viscosity, $\text{m Pa s}^{-1}$	$\rho_f$	Fluid density, $\text{kg m}^{-3}$
$\mu_g$	Fluidizing gas viscosity, $\text{kg m}^{-1} \text{s}^{-1}$	$\rho_g$	Fluidizing gas density, $\text{kg m}^{-3}$
$\mu_{\text{solution}}$	Viscosity of the solution, $\text{kg m}^{-1} \text{s}^{-1}$	$\rho_p$	Particle density, $\text{kg m}^{-3}$
$\mu_{\text{solvent}}$	Viscosity of the solvent, $\text{kg m}^{-1} \text{s}^{-1}$	$\rho_{\text{solution}}$	Density of the solution, $\text{kg m}^{-3}$
$\gamma_{\text{LV}}$	Liquid–vapor interfacial tension, $\text{N m}^{-1}$	$\rho_{\text{solvent}}$	Density of the solvent, $\text{kg m}^{-3}$
$\gamma_{\text{SL}}$	Solid–liquid interfacial tension, $\text{N m}^{-1}$	$\sigma_{\text{solution}}$	Surface tension of the solution, $\text{N m}^{-1}$
$\gamma_{\text{SV}}$	Solid–vapor interfacial tension, $\text{N m}^{-1}$	$\sigma_{\text{solvent}}$	Surface tension of the solvent, $\text{N m}^{-1}$
$\varepsilon_{\text{mf}}$	The bed void fraction	$\sigma_d$	Droplet surface tension, $\text{N m}^{-1}$
$\theta$	Contact angle	$\sigma_f$	Fluid surface tension, $\text{N m}^{-1}$
$\lambda$	Latent heat of vaporization, $\text{kJ kg}^{-1}$	$\varphi$	The particle sphericity
$\rho_d$	Droplet density, $\text{kg m}^{-3}$		

**Introduction**

In a coating process, the surface of a material or a mixture of materials is covered with another material (coat or shell) for a variety of purposes. This process is known as encapsulation and the entrapped material is called the core material, which may be solid particles or a droplet of a liquid or gas.<sup>1</sup> The structure of an encapsulated particle is illustrated in Fig. 1. The Birds' egg shells, red blood cells, bacterial spores, seashells, and plant seeds are natural examples reminding us of the encapsulation.<sup>1,2</sup> Encapsulation technique is widely used in pharmaceutical, food, agricultural, chemical, cosmetic, and printing industries.<sup>3,4</sup>

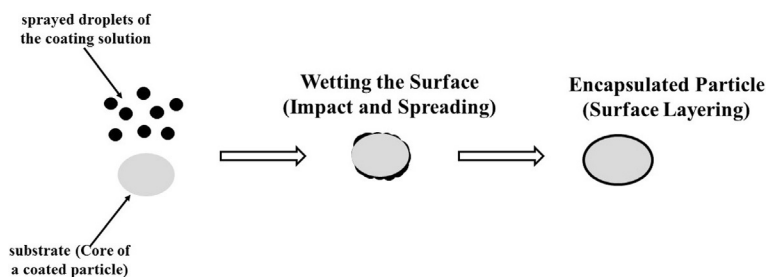
Coating of pharmaceutical products has evolved over the years. In the traditional coating, sugar was applied to the tablets to improve the drug's taste and its appearance. Sugar coating required a long process duration (it could last for 5 days) and the process should have been carefully manipulated by high skilled operators. However, introduction of film coating technique has brought several advantages over the traditional sugar coating. The development of film coating owes much to the availability of a wide range of materials (e.g., cellulose derivatives) as well as the improvement of the coating equipment, particularly the Wurster coating system. The first reference to film coating of tablets was made in 1930, while the first film-coated tablets were commercially presented in 1954 by Abbott Laboratories. They produced the first film-coated product utilizing the Wurster coater.<sup>5,6</sup>

The coating technique was originally introduced in the pharmaceutical industries whose processes are relatively expensive. Accordingly, the application of coating devices in food industries was limited due to the problems related to production cost and the necessity of suitable adoption of product-specific devices. However, employing coating in the food industries has become more practical and feasible after the following modifications: (1) increase of the production capacities; (2) development of more economically coating techniques; and (3) development of less expensive coating materials.<sup>1,7</sup>

In this article, solvents and their compositions are explained and four main approaches for film coating are discussed. These techniques are spray drying coating, hot-melt coating, supercritical fluid coating, and compression coating. In addition, various fluidized bed coating equipment are introduced and the coating life cycle is more elaborated.

**Fundamentals of the Coating Process**

Coating in fluidized beds is carried out in order to obtain particles with uniform quality as well as uniform morphology. This is a complex process, in which layering of the coating material around the particles depends on several parameters, including atomization, collision, adherence, and spreading. Proper coating cannot be achieved unless the particles become involved in the process of layering by following circular trajectories in a controlled way. Such a circulation can be easily obtained in a fluidized bed. Coating in fluidized beds also involves atomizing a liquid through the spray nozzle onto the fluidized particles. Each liquid droplet contains a solvent, acting as the carrier, and a solute which is the desired coating material. Deposition of the sprayed liquid on the



**Fig. 1** Schematic of a multilayer-coated particle.

fluidized particles follows a life cycle with different steps in order to attain a successful coating. Atomized by the nozzle, the droplets come into contact with the particles. After the impact and coherence of the droplets onto the particles, the droplets spread on the surface. Solvent is then evaporated, which leads to the formation of a thin, uniform layer of solute around the particles. Complete coverage of the surface is achieved during several passes of particles through the coating zone.<sup>8,9</sup>

### Objective of Coating

The objective of the coating process is to obtain coated particles with an appropriate and standard quality as well as reproducible products. The coating quality may be characterized based on both uniformity of the coating mass and coating morphology. The coating mass uniformity describes the variation in the amount of coating material deposited on each product particle, while the coating morphology can be ascribed to the variation of a given property of the finished particles with the same amount of coating material.<sup>10</sup> Quality of the coated product may be assessed based on two approaches, namely, macroscopic and microscopic.

The microscopic quality evaluation of the coating is related to the quality of the coating materials deposited on each particle. This coating quality depends on the product application. For example, if the retarding potential of the final product is important, a constant coating thickness across the surface of the particles would be required. Also, no holes should be found on the coated surface. However, in the case of coloring the food, it may be credible to have an uneven coated product with tiny holes on its surface.

The *kinetic release curve* of the product demonstrates the quality of coating. For this purpose, the experiments should be conducted using a specific amount of distilled water (e.g., 100 or 200 mL). The water is poured in a beaker and the desired amount of coated product (e.g., 2 or 3 g) is added to it. The beaker is then shaken mildly and the concentration of solution is measured at preset time intervals. In this way, the release curve of the coated product can be obtained. Examples of such curve are shown in Fig. 2.<sup>4,11</sup> Considering this figure, different coating qualities are classified in Table 1.

The macroscopic approach of evaluating the coating quality is focused on the coater performance based on product uniformity, production yield, degree of agglomeration, and production time. The coating uniformity is evaluated by means of relative standard deviation of thickness of the coat among individual particles.

The coating yield (%Y) is defined by the following equation:

$$\%Y = (M_{CP} - M_{UP}) \times 100 / M_{CM} \quad (1)$$

where  $M_{CP}$  and  $M_{UP}$  stand for the weight of coated and uncoated particles (g), respectively, and  $M_{CM}$  is the weight of the coat material sprayed into the coater (g). Particle agglomerates are formed due to establishing liquid bridges between particles. Generally, particle agglomeration is undesirable in the coating process and should be avoided by optimizing the process parameters. The degree of agglomeration is defined as the ratio of the mass of particle agglomerates to the total mass of particles. The mass of the particle agglomerates can be obtained using a sieve with desired nominal opening size larger than the maximum limit value of the coated particle. The optimum operation in the coating process can be achieved when more uniform coated particle, higher production yield, and lower degree of agglomeration are achieved. Accordingly, lower amounts of coating material are applied for a specific coating thickness and shorter production time is required.<sup>4,12,13</sup>

### Coating Applications

Particle coating can be found in several industries, such as pharmaceutical, food, and agricultural. The coating process is conducted for several reasons, depending on the usage of the final product. The main reasons are:

- To mask unpleasant taste of the substances<sup>14,15</sup>
- To increase the shelf life<sup>16,17</sup>

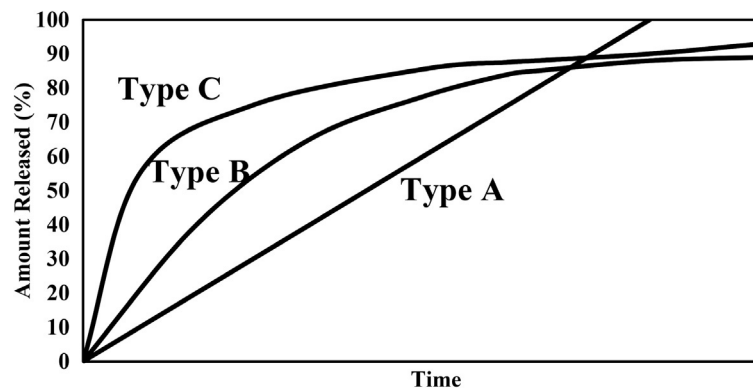


Fig. 2 Release behavior of active ingredients.

**Table 1** Classification of the coating quality<sup>11</sup>

<i>Types</i>	<i>Description</i>
Type A	<ul style="list-style-type: none"> <li>● The coatings on all the products are intact.</li> <li>● The local coating thicknesses are the same.</li> <li>● The relationship between the release of the active substance and time is linear.</li> </ul>
Type B	<ul style="list-style-type: none"> <li>● The coatings on all the products are intact.</li> <li>● The local coating thicknesses vary.</li> <li>● The relationship between the release of the active substance and time is nonlinear.</li> </ul>
Type C	<ul style="list-style-type: none"> <li>● Some of the particles have holes in their coating. Therefore, these particles release their active ingredient almost immediately at the beginning.</li> <li>● After sudden release of the active ingredient at the beginning, the release rate shows similar behavior to that of the type B coating.</li> </ul>

- To provide sustained and prolonged release<sup>18,19</sup>
- To improve flowability of the product particles<sup>20,21</sup>

The main benefit of coating in pharmaceutical industries is to modify or control the drug release. By introducing film coating to the pharmaceutical industries, reproducibility of the process has become more satisfactory and batch-to-batch uniformity of the products is assured.<sup>22,23</sup> Coating can improve the stability of drugs which are sensitive to oxygen, moisture and light. In addition, it can prevent volatile drugs (e.g., methyl salicylate and peppermint oil) from vaporization. Toxicity and gastrointestinal disorders of some drugs (e.g., ferrous sulfate and KCl) are reduced by coating.<sup>24</sup>

In agricultural industries, the fertilizers are used to provide sufficient concentration of the nutrients nearby the plant root for a desired period. Commercial fertilizers should be immune to degradation, volatilization, leaching, adsorption, and land immobilization. Therefore, applying the coating on granules or pellets of fertilizers will improve their releasing feature.<sup>4,25</sup>

If coating is applied to the food ingredients, masking of the taste and odors would be the most desirable effect. Besides, edible films and coatings protect the food ingredients from physical, chemical, and biological deterioration. For example, coating protects the food against moisture migration, microbial growth on the surface, oxidation of nutrients, etc. Introducing edible films and coating leads to the enhancement of physical strength of the food, reduction of particle clustering, and improvement of visual and tactile features of food products.<sup>26–28</sup> It can also tune the effect of functional ingredients and additives, including processing aids (i.e., enzymes), preservatives (i.e., acids and salts), and fortifiers (i.e., vitamins and minerals).<sup>7,29</sup>

## Coating Solution

Coating materials are also called coat, shell, wall, or membrane materials and are usually polymeric based. In the pharmaceutical coating processes, the size of the core (i.e., the drug particle to be coated) is in the range of 0.7–2 mm, while the thickness of the coat varies in the range of 5–50  $\mu\text{m}$ .<sup>30</sup>

There are a number of coating materials being applied in the controlled release coating. However, the use of the aforementioned coating materials in pharmaceutical and food industries is limited due to the strict regulations from regulatory agencies. Selection of the suitable coating material is related to some considerations: (1) the coating material must be compatible with the drug core and dissolution of the drug media, (2) the coating material must provide desired characteristics (e.g., appropriate release pattern) for the core within the acceptable limits.<sup>6,31–33</sup> Formulation of the coat is usually consisted of solvent, polymer, plasticizer, colorant/opacifier, and antitack agent.

## Solvent

The solvent plays the role of carrier and transports other coating materials onto the surface of particles. The most commonly used solvents are water, alcohols, ketones, esters, and chlorinated hydrocarbons. Accordingly, the solvent can be either organic or aqueous. In the past, organic solvents were widely used. However, its usage has become limited due to food and environmental regulations and danger of explosion. On the other hand, using water as the liquid solvent has caused various challenges. Water has a greater heat capacity compared with organic solvents. Therefore, longer drying time and higher drying temperatures are needed for its evaporation. This may be harmful to drugs which are sensitive to heat and moisture.<sup>34–39</sup>

## Polymer

There are a vast majority of polymers which can be classified based on their origins, as given in [Table 2](#). It is reported in literature that natural polymers are the most commonly used polymers in the coating formulation due to their low toxicity. Synthetic polymers may suffer from the impurities, such as residual monomers, plasticizers, softeners, and fillers. Thus, it is necessary to

**Table 2** The polymers used in the coating formulation

<i>Types</i>	<i>Examples</i>
Natural	Zein, <sup>40</sup> alginate, <sup>41</sup> poliglusam (CHitosan), <sup>42</sup> pectin, <sup>43</sup> Shellac, <sup>44</sup> and Rosin <sup>45</sup>
Semisynthetic	Ethylcellulose, <sup>46</sup> cellulose acetate, <sup>46</sup> and hypromellose (HPMC) <sup>47</sup>
Synthetic	Methacrylic acid copolymers <sup>48</sup>

**Table 3** Classification of the plasticizers<sup>50</sup>

<i>Types</i>	<i>Plasticizer</i>
Polyols	Glycerol (glycerin), propylene glycol, polyethylene glycols (PEG)
Organic esters	Phthalate esters (diethyl, dibutyl), dibutyl sebacate, citrate esters (triethyl, acetyl triethyl, acetyl tributyl), triacetin
Oils/glycerides	Castor oil, acetylated monoglycerides, fractionated coconut oil

evaluate the concentration of these residues for their potential toxicity. The most preferred polymers for sustained release particulates are ethylcellulose, methacrylic acid, copolymers, and cellulose acetate.<sup>19,49</sup>

### Plasticizer

Plasticization refers to the modification in thermal and mechanical properties of a given polymer. Plasticizers are low molecular weight substances added to a polymer solution to promote its plasticity and flexibility. Therefore, the plasticizers make the polymer solution more suitable for the application of film coating. There should be chemical similarities between the polymers and its plasticizers. It is supposed that the function of the plasticizer is that its molecules are embedded between the individual polymer chains and disintegrate the polymer–polymer interactions.<sup>50,51</sup>

One of the most important properties of the polymers is known as the glass transition temperature ( $T_g$ ), which measures chain mobility. This is a temperature at which a polymer transforms from a hard, glassy material to a soft, rubbery material. Plasticizers lower the glass transition temperature of the polymers. Plasticizers can be categorized as shown in [Table 3](#).<sup>50,52</sup>

### Colorant/Opacifier

Edible colors or pigments are sometimes added to the coating formulations. Colorants can be categorized into three main groups: (1) synthetic water-soluble organic dyes (e.g., tartrazine, sunset yellow, erythrosine); (2) insoluble aluminum lakes (consisted of water-soluble dyes adsorbed onto small, insoluble particles of alumina); (3) inorganic pigments (e.g., titanium dioxide, talc, calcium carbonate, and the iron oxides).<sup>5</sup> Colorants and opaquant extenders are usually used in the coating materials in order to improve the esthetic appearance. Colorants may reduce the film tackiness. Consequently, the possibility of agglomeration decreases.<sup>53,54</sup> In addition, color can be an identification of the final product uniformity. Color measurement can be used for the evaluation of product uniformity. Inconsistencies in the color of the final coated product can be attributed to its poor quality of production or product instability.<sup>55</sup> The surface color of tablets, tablet colorants, and color stability of the tablets can be measured using Tristimulus colorimeters or fiber optic instrument.<sup>56,57</sup> Colorants have more or less pacifying properties and may be added to the coating formulation to enhance the ability of the coat to protect core particles against light exposure.<sup>5</sup>

### Antitack Agent

The tackiness of coat is crucially important. Excess tack may cause adhesion of particles to each other which leads to their agglomeration. The tack problem especially becomes more important at higher temperatures and at greater composition of plasticizers. There are a number of antitack additives added to the coating formulation, which modifies the tackiness and reduces the particle agglomeration.<sup>58,59</sup> Talk is one of the most commonly used antitack agent in pharmaceutical industries. It exists in the coating solution at 25%–100% based on the dry polymer weight. Glyceryl monostearate (GMS), colloidal silicon dioxide, and magnesium stearate have also been used as antitack agents in coating formulation.<sup>5,60</sup>

## Different Methods of Coating of Particles

There are several techniques for coating of materials. These techniques can basically be categorized as melt coating, supercritical fluid coating, compression coating, and spray drying coating. These coating techniques are further described as follows.

### Melt Coating

If melting point of the coating material is low, the coating material could be used in the molten state and the melt coating technique can be applied. It is recommended to use a product bed temperature of up to 140–150°C in order to have physically and chemically stable materials. Moreover, the melting point of the coating material should be less than 80°C. The coating materials used in this method are usually derived from plants or animals, including hydrogenated vegetable oil (e.g., cotton seed oil, palm oil, and soy bean oil), carnauba wax, and glyceride. Fluidized bed coaters are the commonly used equipment in this technique.<sup>61–65</sup>

### Supercritical Fluid Coating

Application of the supercritical fluid coating method in the coating of particles is limited. In this technique, the solid coating material is suspended in a supercritical fluid. Carbon dioxide is the most used supercritical solvent in this case. In fact, supercritical carbon dioxide is the medium used for reducing the viscosity in order to spray the coating materials onto the particles. The coating material should have adequate solubility in the supercritical carbon dioxide. Such materials include lipids, fatty alcohols, fatty acids, fats, waxes, Eudragit polymers, triglycerides of myristic acid, glycerides and glyceride ester of polyethylene glycol, trimyristin, Myvacct, octadecanol, cholesterol, and lecithin.<sup>66–69</sup>

### Compression Coating

In this method, the coating material is compressed around a core (i.e., particle) using a special tablet press. This method is mostly used for larger particles. In the compression coating method, first, the drug materials are compressed into the shape of a tablet (i.e., the drug is in the core). Afterward, the core is placed in a die with the half of the coating material. Subsequently, the core is surrounded with the remainder coating material which forms the outer coating layer. Finally, it is compressed by punch to become a coated particle. The compression coating is not a commonly used process because of some deficiencies. For example, if the core is not placed in the center of the die, this may result in an incomplete coating. The coating materials in this technique include polyethylene oxide, polyethylene glycol, hydrogenated castor oil, cellulose derivatives, complex of alginate-chitosan, sodium alginate, and behenic acid.<sup>6,70–74</sup>

### Spray Drying Coating

In the spray drying method, a solution is sprayed by a nozzle into a bed of particles. Layers of the coating material are formed after evaporation of the solution. Different polymers are used in this method, including Eudragits, shellac, and ethyl cellulose.<sup>75–77</sup> The equipment used for this purpose are pan coater and air suspension coaters, including top spray fluidized bed, tangential spray fluidized bed, and bottom spray fluidized bed. These air suspension coaters are the most common equipment for coating applications in food and pharmaceutical industries.<sup>76,78–81</sup>

### Pan coating

The first pan coaters were employed for sugar coating of tablets which was borrowed from the confectionary industry. The open, copper, bowl-shaped, pan coaters were widely used in the pharmaceutical industries which are now substituted with stainless steel ones. Conventional pan coaters rotate at an angle of about 45° to the horizontal at a speed range of 20–50 rpm. The conventional pan coater suffers from the inconvenient heat transfer between the drying air and the particle bed. In addition, it is a lengthy process and requires skilled operators. However, the basic pan coaters have been improved by a number of modifications including: (1) application of side air exhaustion (improving the drying capacity), (2) installation of the perforated distribution plate, and (3) implementation of different shaped pans (cylindrical, hexagonal, and pear shaped).<sup>6,35,36</sup>

### Air suspension coating

Applying modifications to the pan coating system led to the introduction of a new coating technique, the air suspension coating technique. The basic fundamental characteristic corresponding to air suspension coaters is the fluidization of particles upwardly. Injecting the fluidization gas causes the particle circulation throughout the chamber. Accordingly, formation of agglomerates may be controlled more properly and a product with more superior quality can be obtained.<sup>6,10,82</sup>

This technique has great advantages over other conventional coating systems due to the high heat and mass transfer rates. Accordingly, there exist uniform temperature distribution and relatively short coating time in fluidized beds. This technique is one of the most preferred methods of particle coating due to producing high quality product, possibility of multilayer coating, and its

capability of large-scale production. Moreover, fluidized beds are used for granulation processes and are the most preferable equipment for this purpose because of short processing time and easiness of operation.<sup>6,64,83,84</sup> The coating material is atomized from a spray nozzle. The spray nozzle may be located at different positions, such as at top of the particle container in the top spray fluidized bed coater, at the bottom of the particle container in the bottom spray fluidized bed coater, or on the side of the particle container in the tangential spray fluidized bed coater. The air suspension coating technique is explained more thoroughly in the proceeding sections.

### Top-Spray Fluidized Bed

The top-spray fluidized bed is known to be the simplest one among the spray fluidized beds. It has evolved from commercial fluidized bed dryers and provides a high capacity and low capital cost. The spray nozzle is placed at the top of the coating chamber. Coating materials are countercurrently sprayed at a low pressure and sheared onto the fluidized particles. The spray nozzle is typically a binary or pneumatic nozzle. The volume capacity of the apparatus is in the range of 2 L (lab scale) to more than 2000 L.<sup>85–87</sup> Schematic of the top spray fluidized bed is shown in Fig. 3.

The fluidizing gas is blown from the bottom of the chamber through the gas distributor plate and fluidizes the substrates. At lower fluid velocities, the bed remains static. As the air velocity is increased, an abrupt change of the substrates is observed which is called minimum or incipient fluidization. From the fixed bed conditions, the minimum fluidization velocity can be theoretically calculated using Carman–Kozeny equation<sup>88</sup>:

$$u_{mf} = \frac{(\varphi d_p)^2 (\rho_p - \rho_g) g}{180 \mu_g} \left( \frac{\varepsilon_{mf}^3}{1 - \varepsilon_{mf}} \right) \quad (2)$$

where  $\varphi$ ,  $d_p$ , and  $\rho_p$  denote the particle sphericity, the particle diameter (m), and the particle density ( $\text{kg m}^{-3}$ ), respectively;  $\rho_g$  and  $\mu_g$  are the fluidizing gas density ( $\text{kg m}^{-3}$ ) and fluidizing gas viscosity ( $\text{kg m}^{-1} \text{s}^{-1}$ );  $g$  is gravity acceleration ( $\text{m s}^{-2}$ ); and  $\varepsilon_{mf}$  represents the bed void fraction at this condition.

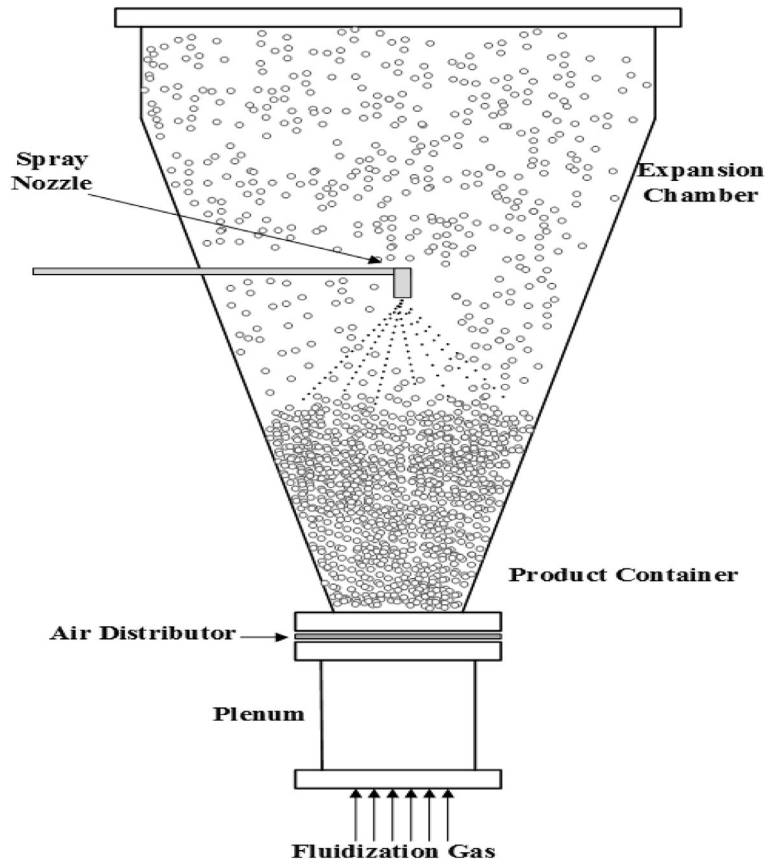


Fig. 3 Schematic of the top-spray fluidized bed coater.

At the minimum fluidization, the velocity of the substrates is very low, which may not be economically practical and promote agglomeration.<sup>82,89,90</sup> For this purpose, the top-spray coating fluidized beds operate in the bubbling fluidization regime. According to Fig. 3 the substrates are randomly exposed to the sprayed coating material, after which the coated substrates pass the coating zone into the expansion zone. Evaporating the coating solvent from the surface in the expansion zone, the coated substrates fall back into the product container.<sup>82,90</sup>

Moving randomly and unrestrictedly, the particles circulate in a less ordered pattern in the top-spray fluidized bed. Accordingly, the travel distance of droplets cannot be controlled prior to its contact with the substrate surface. As a result, the droplets may become dried prematurely which is considered as the main drawback of the top-spray fluidized beds. As a result, the top-spray fluidized bed is improper equipment for the film coating and controlled release coating of particulates. However, it is more common to utilize the top-spray fluidized bed for the aim of taste masking, barrier coating, cosmetic coating, and functional coating.<sup>10,86</sup>

This process has the most possibility of being used in food industries as compared with the other air suspension coating methods in order to further enhance the shelf life (i.e., allowing a longer period of storage against moisture, oxygen, and light). Top-spray fluidized bed is considered to be more feasible for food industry mainly because of its high versatility, relatively large batch size, and simplicity.<sup>7,31</sup> It should be noted that the top-spray fluidized beds offer the best results in the case of hot-melting coating when compared with the tangential-spray and bottom-spray coaters. This is due to the fact that the product container of the top spray fluidized bed is designed for an unrestricted particle flow, the principle which is one of the most important features of the hot-melt coating.<sup>91-94</sup>

### Tangential-Spray Fluidized Bed

The tangential-spray fluidized bed is a relatively new fluidization technique for coating of particles. This approach takes advantage of a rotating disk to provide a circular pattern for movement of particles. The disk is adjusted at a height to give a slit between the edge of the disk and the wall of the coater chamber. The fluidizing gas passes upward through the annular space between the periphery of the rotating disk and the wall. Exploiting the rotating disk and fluidizing gas, the tangential-spray fluidized bed can process a large volume of solids in relatively small equipment. The spray nozzle is mounted at the side of the coating chamber, spraying the coating material concurrently onto the substrates. Batch sizes are in the range of 1–400 kg.<sup>10,82</sup> Schematic of the tangential spray fluidized bed is illustrated in Fig. 4.

The particle movement is achieved by three different forces: (1) the rotating disk creates a centrifugal force which brings forth the movement of the particle toward the wall; (2) the air passing through the disk causes a lifting force which accelerates the particles upward in the chamber; (3) the gravity force which causes particles to fall down toward the disk. Combining these forces, the tangential-spray coater offers a homogenous spiral motion of particles.<sup>82,95-97</sup>

The tangential-spray coater has the ability to carry out both pelletization and coating, which is known as one of the most significant features of this type of coaters. The tangential-spray coaters can be used properly for application of controlled release coating of particulates, and most particularly, it is suitable for multilayer coating of particulates as well as formation of thicker coats.<sup>91,98,99</sup> The most significant drawback of this device is that the particles experience a high level of kinetic energy caused by the intensive mechanical forces. This exposure makes it difficult for the coater to be used for coating of brittle particles. Moreover, this approach is not suitable for large and nonspherical particles due to the possibility of attrition and breakage of particles. However, the granulation processes can take advantage of these high mechanical forces.<sup>86,96,98</sup>

### Bottom-Spray Fluidized Bed (Wurster)

The bottom-spray fluidized bed, also known as the Wurster system, was first developed by Dale Wurster in late 1950s.<sup>100,101</sup> Schematic of a Wurster fluidized bed is shown in Fig. 5. This system is well designed to accomplish suitable coating for small particles, owing to the insertion of a draft tube located at the center of the chamber (see Fig. 5). The draft tube is also known as the Wurster partition, named after its inventor. The size of this tube is roughly half the diameter of the base of the chamber. Particle circulation and their contact efficiency are much more controlled in this approach by means of application of the draft tube in the bed.<sup>23,82,102,103</sup> It is illustrated in Fig. 5 that the partition column has divided the Wurster coater into four separate zones, called upbed region, expansion region, bed downbed region (annular region), and horizontal transfer region.

Particles pursue a predetermined circulation pattern in the Wurster fluidized bed. This unique characteristic enables the Wurster fluidized bed to excellently provide homogeneous and dense films of the coat on the particles. In the Wurster coating system, the spray nozzle is placed at the bottom of the chamber, beneath the draft tube, spraying concurrently the coating material upward into the spray zone to the particles. The traveling path of the droplets is short enough to prevent the premature evaporation. Hence, the Wurster process can be applied in a wide range of coating liquid viscosity, spray rate, fluidizing air flow rate, and process air temperature.<sup>7,103-105</sup>

The system mainly consists of the wetting section (using the spray nozzle) and the drying section (with injecting the hot air). This results in a continuous layering of the coating material and particles become involved in the process of layering by following circular trajectories in a controlled way. These trajectories pass through two different zones with different superficial gas velocities.



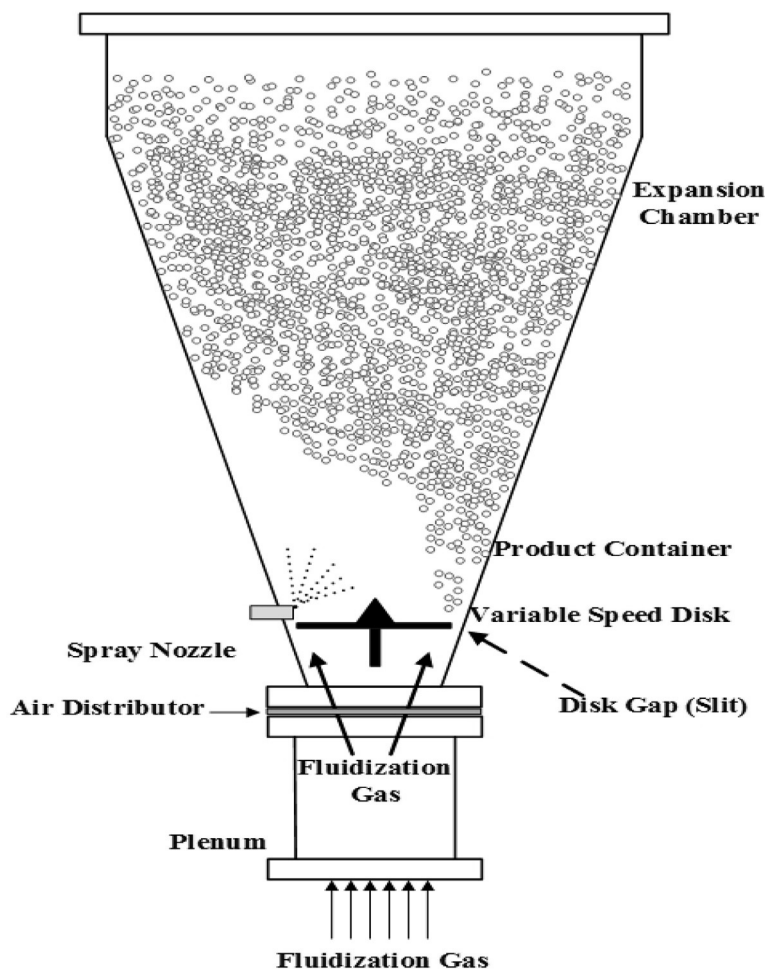


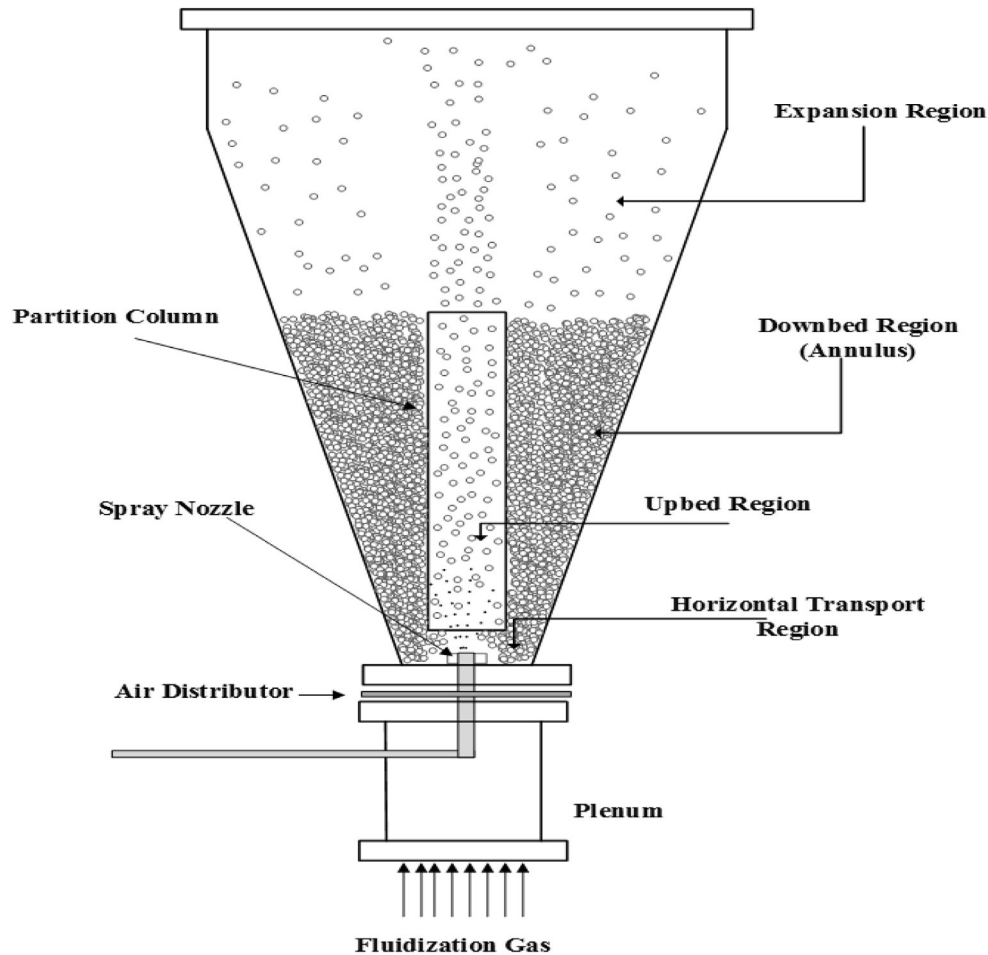
Fig. 4 Schematic of the tangential-spray fluidized bed coater.

The high-velocity zone is located below the draft tube where a jet of gas is employed to convey the particles upward through the draft tube until a certain height where the particles fall into the low velocity zone.<sup>86,93,106–109</sup>

The Wurster coating process is the most preferred method for the application of controlled release coating of the small particulates, particularly in pharmaceutical industries. It is reported that the Wurster process provides the most uniform and smoothest final product among different forms of fluidized bed coaters.<sup>10,23,102</sup> In comparison, Teunou and Poncellet<sup>110</sup> studied the coating of particles in different fluidized bed systems. They stated that the Wurster coater provided a higher coating efficiency. Bertelsen et al.<sup>111</sup> reported that the bottom-spray fluidized bed resulted in a more efficient retardation compared with the top-spray fluidized bed.

### Upbed Region

In the Wurster system the distributor plate supports the particles and has more perforation at the upbed region (beneath the partition column) than the downbed region. Accordingly, the air velocity in the upbed region is higher than that in the annulus region. The particles are entrained with the gas in the upbed region. In other words, the fluidizing gas pneumatically conveys particles upward in the partition column. The air flow is considered to be turbulent in the upbed region. The flow of air from the spray nozzle also adds to this turbulence. The air velocity in the partition column cannot be considered as plug flow since the air velocity is higher at the center of the tube and lower in the region near the walls. Therefore, velocity of the particles is lower near the walls of the partition column and the particles fall down along the wall. This may impose the problem of adhesion of particles to the wall of the partition column. The maximum permitted velocity in the upbed region is restricted by the height of the expansion zone in order to prevent the collision of the particle with the ceiling of the chamber. The amount of particles in the upbed region should be adjusted properly via the partition gap in order to have sufficient amount of particle exposure to the spray of the coating material.<sup>103,112–115</sup>



**Fig. 5** Schematic of the bottom-spray fluidized bed coater.

### Expansion Region

Leaving the upbed region (out of the partition column), the partially coated particles enter the expansion region. The height of the expansion region should be high enough to avoid the collision of the particles to the top ceiling of the chamber. In this region, the gas velocity decreases and particles decelerate as a result of the expanded cross section. Hence, the particles fall down in the annular bed region. In order to avoid particle–particle agglomeration, the solvent should adequately become evaporated and the particles should become dried prior to reaching the annular bed region.<sup>103,115</sup>

### Downbed Region

Having decelerated in the expansion region, the particles fall back down in the downbed region (annular region). In the annulus, the particles are fluidized around the minimum fluidization velocity. Some researchers have recommended that the particles should be fluidized above the minimum fluidization velocity<sup>115</sup> and some suggested that the particles should move below the minimum fluidization velocity in the annular region.<sup>103</sup> In any case, it can be conceived from the particle circulating pattern that the velocity of particles in the downbed is at least lower than in the other three regions. If the particles do not become completely dried in the upbed and the expansion regions, they would land in the annular section surrounding the partition column (downbed region) in a sticky manner. Accordingly, the particles are prone to form particle–particle agglomeration.<sup>103,115</sup>

### Horizontal Transport Region

The particles advance steadily through the downbed region and enter the horizontal transport region, which consists of the bottom of the downbed region as well as the partition gap (the opening area under the partition column). The partition gap is responsible for the pneumatic transport in the horizontal transport region. The higher the partition gap, the more the particle transport can be

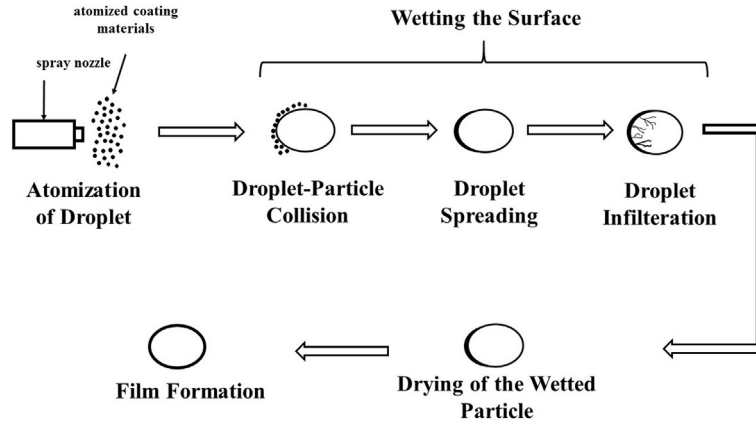


Fig. 6 The coating life cycle in a fluidized bed coater.

achieved. As was discussed above, the air velocity is higher in the central part of the distributor plate (below the partition column) compared with the air velocity at beneath the downbed region. Consequently, there exists a region of lower pressure below the partition column, which enables the particles to be conveyed in the horizontal transport region. Afterward, the particles enter the partition gap into the upbed region and the particles follow the aforementioned circulation pattern once again. This cycle is repeated several times until the desired coating layer is deposited on the surface of particles.<sup>10,11,103,112,114,116</sup>

### Coating Life cycle in a Spray Fluidized Bed

According to the discussions in the previous section, the particles that move in the fluidized bed chamber experience different phenomena during their journey in a single coating cycle. A number of physical and transport phenomena occur during the coating life cycle of the particles which are called<sup>117</sup>: (1) particle movement, (2) atomization, (3) wetting the particle surfaces, and (4) drying and film formation. The life cycle of coating in the fluidized bed coaters is discussed in detail in this section. To better understand the coating life cycle, different phenomena occurring during a coating life cycle are illustrated in Fig. 6.

#### Particle Movement

The fluidizing gas causes a rising movement of gas and particles in the coater chamber. Different regimes of fluidization can be observed in fluidized bed coaters, from fixed bed to pneumatic transport. In the top-spray fluidized bed, the particles circulate in the chamber at the bubbling regime of fluidization. In the tangential-spray fluidized bed, the particles rotate in a spiral motion caused by fluidization air, rotating disk, and their gravity force. In the Wurster coater, particles experience various moving patterns in different regions (upbed region, expansion region, downbed region, and horizontal transport region). In the upbed region, the fluidizing gas propels the particles at the turbulent regime.<sup>115</sup> In the expansion region, the particles slow down until falling back into the downbed region. In the downbed region, the velocity of particles may be either greater or lesser than the minimum fluidization velocity. In the horizontal transport region, the particles are pneumatically conveyed toward the upbed region. The movement of particles is discussed in detail in the preceding sections (sections "Top-Spray Fluidized Bed", "Tangential-Spray Fluidized Bed", and "Bottom-Spray Fluidized Bed (Wurster)").

#### Atomization

During the coating process, the coating solution is usually sprayed by means of a bifluid nozzle in which the coating solution is atomized by air under the pressure in the range of 0.5–3.5 bar. Average size of the droplets can be adjusted using the air pressure in the bifluid nozzle. The higher the atomization pressure, the smaller become the droplet sizes. It is recommended that the droplets should be smaller than 100  $\mu\text{m}$  and the particle to droplet ratio must be at least 10. On the other hand, small droplets are in favor of more homogenous final coating.<sup>10,90,117</sup> Effect of physical properties of the coating solution on average droplet diameter atomized by the nozzle can be obtained from<sup>5</sup>:

$$\frac{d_{VM, \text{solution}}}{d_{VM, \text{solvent}}} = \left[ \frac{\sigma_{\text{solution}}}{\sigma_{\text{solvent}}} \right]^{0.5} \times \left[ \frac{\mu_{\text{solution}}}{\mu_{\text{solvent}}} \right]^{0.2} \times \left[ \frac{\rho_{\text{solvent}}}{\rho_{\text{solution}}} \right]^{0.3} \quad (3)$$

where  $d_{VM, \text{solution}}$  and  $d_{VM, \text{solvent}}$  are the volume mean droplet diameters of solution and solvent (m), respectively;  $\sigma_{\text{solution}}$ ,  $\mu_{\text{solution}}$ , and  $\rho_{\text{solution}}$  denote the surface tension ( $\text{N m}^{-1}$ ), viscosity ( $\text{kg m}^{-1} \text{s}^{-1}$ ), and density of the solution ( $\text{kg m}^{-3}$ ), respectively;  $\sigma_{\text{solvent}}$ ,  $\mu_{\text{solvent}}$ , and  $\rho_{\text{solvent}}$  denote the surface tension ( $\text{N m}^{-1}$ ), viscosity ( $\text{kg m}^{-1} \text{s}^{-1}$ ), and density of the solvent ( $\text{kg m}^{-3}$ ), respectively. The droplet size atomized from the nozzle may be calculated using the following correlation<sup>5,90</sup>:

$$d_d = \frac{585 \times 10^3 \sqrt[3]{\sigma_f}}{v_{REL} \sqrt{\rho}} + 597 \left( \frac{\mu}{\sqrt{\sigma_f \rho}} \right)^{0.45} \left( \frac{1000 U_{sol}}{U_{at}} \right)^{1.5} \quad (4)$$

where  $d_d$  is the mean diameter of droplets ( $\mu\text{m}$ ) and  $\sigma_f$ ,  $\rho_f$ ,  $\mu_f$ ,  $U_{sol}$ ,  $U_{at}$ , and  $v_{REL}$  denote the fluid surface tension ( $\text{N m}^{-1}$ ), fluid density ( $\text{kg m}^{-3}$ ), fluid viscosity ( $\text{m Pa s}^{-1}$ ), fluid volumetric flow rate ( $\text{m}^3 \text{s}^{-1}$ ), air volumetric flow rate ( $\text{m}^3 \text{s}^{-1}$ ), outlet air velocity (the velocity of air relative to liquid at the atomizer nozzle exit) ( $\text{m s}^{-1}$ ), respectively.

The air pressure influences the droplet temperature. The bed temperature may be calculated as a function of the atomization pressure and inlet air temperature as follows<sup>90</sup>:

$$T_{bed} = -1.07 - 1.1552P_{at} + 0.6221T_{inlet}, P < 0.001, R^2 = 0.835 \quad (5)$$

where  $T_{bed}$  is the bed temperature ( $^{\circ}\text{C}$ ),  $P_{at}$  denotes the atomization pressure (bar), and  $T_{inlet}$  is the inlet air temperature ( $^{\circ}\text{C}$ ). This equation has been derived for the sodium caseinate coating solution at the atomization pressure of 1.5, 2.5, and 3.5 bar.

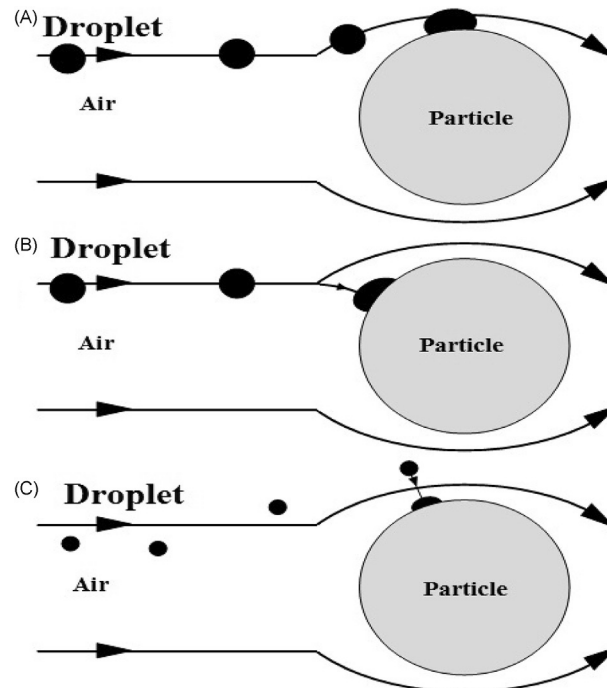
The atomization pressure dictates the droplet velocity which also influences the velocity of impact of droplet and particle. In the Wurster coaters, the particle makes its way from the horizontal transport region through the partition gap into the upbed zone where the coating material is sprayed via the spray nozzle. The particle velocity in the upbed region varies in the range of 3–5 m/s. Ejecting from the spray nozzle, the coating droplets have relatively high velocity (approximately 100 m/s). However, the droplets quickly decelerate, approaching the superficial air velocity. Meanwhile, its solvent evaporates.<sup>90,115</sup>

### Wetting the Particle Surfaces

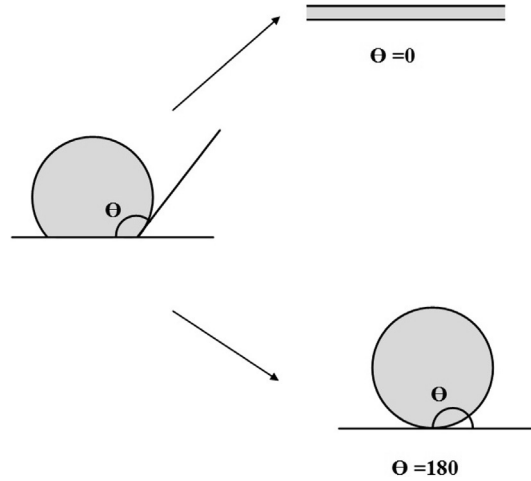
After injection of the coating solution, there exist a number of micro-level phenomena which should properly take place in order to have fine coating film around the particle.

#### Droplet–particle impact

Relative velocity of droplets and particles can result in collision. However, not all the collisions lead to adherence. It is illustrated in Fig. 7 that the droplet–particle collision can be classified into three different mechanisms, including interception, inertia, and diffusion. According to this classification, the collision by interception is due to the overlap of the droplet and the particle surface which is the most probable mechanism for larger droplets. If the droplet deviates from its streamline and encounters the surface of the particle, the inertia mechanism would take place. The inertia mechanism is the favorable mechanism in fluidized bed coating. If the droplet size is less than 0.001 mm, the droplet–particle collision occurs mainly due to the diffusion mechanism. In this mechanism, the droplets diffuse across their streamline via the Brownian motion.<sup>115,117</sup> According to Guignon et al.,<sup>117</sup> a successful



**Fig. 7** Droplet–particle collision: (A) interception, (B) inertia, and (C) diffusion. Adopted from Guignon, B.; Duquenoy, A.; Dumoulin, E. D. Fluid Bed Encapsulation of Particles: Principles and Practice. *Dry. Technol.* **2002**, *20*, 419–447).



**Fig. 8** Droplet contact angle.

coating can be achieved due to interception or inertia in the fluidized bed coaters. Link and Schlünder<sup>118</sup> stated that the inertia mechanism is the most important mechanism for a successful coating in fluidized beds. Therefore, nozzle positioning, spray angle, number of droplets, and droplet velocity relative to the circulating particles are the most important factors affecting a successful coating.<sup>115</sup>

#### ***Droplet spreading and adherence on the particle surface***

Having impinged on the surface of the particles, the droplet should properly spread on the surface of the particle. This microlevel phenomenon of spreading is of particular importance, especially in thin coating, pesticide application, spray painting, spray combustion, and spray cooling of hot surfaces.<sup>115,117</sup> After the impingement of droplet on the surface of particle, the droplet can sit as a discrete droplet on the surface of the particle or it can spread out and cover the surface completely (see Fig. 8). The angle which the liquid–vapor interface meets the solid surface is called the contact angle ( $\theta$ ).<sup>5</sup>

Contact angle can be used to evaluate the wettability of particle by the coating solution. The contact angle may be calculated by Young's equation as<sup>5</sup>:

$$\cos\theta = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} \quad (6)$$

where  $\gamma_{SV}$  is the solid–vapor interfacial tension ( $\text{N m}^{-1}$ ),  $\gamma_{SL}$  is the solid–liquid interfacial tension ( $\text{N m}^{-1}$ ), and  $\gamma_{LV}$  is the liquid–vapor interfacial tension ( $\text{N m}^{-1}$ ). Accordingly, in order to improve the spreading of the droplet over the surface of the particle, the coating formulation must be adjusted in order to wet the particle surface. If the contact angle is less than  $90^\circ$ , wetting of the surface can take place and the wetting energy is negative.<sup>115,117</sup>

Infiltration occurs simultaneously to spreading. This is an unfavorable phenomenon since it decreases the coating yield. Accordingly, the rate of spreading should be faster than that of infiltration. The infiltration phenomena depend on the property of the coating material, the interaction between the coating material and the particle, porosity, and pore size distribution.<sup>115</sup> Turton et al.<sup>10</sup> reported that decreasing the binder viscosity leads to increasing both droplet spreading rate and infiltration rate.

The adhesion efficiency depends on various parameters, including momentum of droplet and particles at collision, their angle of incidence, liquid properties, interfacial properties, and the surface structure of particle. Moreover, the drying properties of the coating material are crucially important since the collision of premature dried droplets may not bring about adhesion of the droplet to the surface of particle.<sup>115</sup> The necessary work of wetting per unit area can be calculated as a function of surface tension of the solution and contact angle as follows<sup>119</sup>:

$$w_m = -\sigma_d \cdot \cos\theta \quad (7)$$

where  $\sigma_d$  denotes the droplet surface tension ( $\text{N m}^{-1}$ ). The work of wetting per unit area ( $\text{J m}^{-2}$ ) may also be determined as a function of liquid–vapor interfacial tension and contact angle as:

$$w_m = -\gamma_{LV} \cdot (\cos\theta + 1) \quad (8)$$

It is necessary to note that a negative value for  $w_m$  is considered as the work of adhesion per unit area ( $\text{J m}^{-2}$ ) which is the required work to restore initial conditions.<sup>5</sup> Sobolev et al.<sup>120</sup> concluded that the main mechanisms of droplet–particle adhesion are as follows:

- Mechanical mechanisms of adhesion, including coating–substrate mechanical interaction and interlocking (keying) as well as deformation of the substrate surface and rebounding of particles.
- Thermal mechanisms of adhesion, mostly including coating–substrate thermal interactions.
- Diffusive and chemical mechanisms of adhesion.

### Drying and Film Formation

Drying of coated particles can be conducted by means of hot fluidizing air during which the solvent is evaporated to advance the coating deposition on particles. This stage plays an influential role in a successful coating process. A suitable drying condition is in favor of droplet–particle adherence in the atomization zone. In addition, partially coated particle may satisfactorily be dried in the air and the possibility of particle agglomeration can be decreased. The drying condition should be suitable for: (1) drying of partially coated particles and (2) drying of atomized droplets prior to the collision. Therefore, there should be a compromise between the two above-mentioned concepts.<sup>115,117</sup>

Becher and Schlünder<sup>121</sup> suggested that the heat required for drying of the coated particle (i.e., evaporation of the excess solvent) may originate from two sources: (a) heat convection with the hot fluidizing gas, (b) heat conduction with the particle.

For a single small and pure droplet (low terminal velocity), the evaporation time ( $t_{\text{eva}}$ ) can be determined from the following equation<sup>5,115</sup>:

$$t_{\text{eva}} = \frac{\lambda \rho_d (d_0^2 - d_t^2)}{8k\Delta T} \quad (9)$$

where  $\lambda$  is the latent heat of vaporization,  $\rho_d$  denotes the droplet density ( $\text{kg m}^{-3}$ ),  $d_0$  and  $d_t$  are the initial droplet diameter and the droplet diameter at any time  $t$  (m), respectively,  $k$  is thermal conductivity of fluidized air ( $\text{W m}^{-1} \text{K}^{-1}$ ), and  $\Delta T$  is the difference between the air temperature and droplet surface temperature (K). Werner et al.<sup>115</sup> stated that Eq. (9) is not very practical, for the drying rate is not constant. Therefore, the evaporation time may be calculated in terms of diffusion using Crank's equation<sup>115,122</sup>:

$$\frac{C_t - C_e}{C_0 - C_e} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left(-\frac{n^2 \pi^2 D_{\text{eff}} t}{R_0^2}\right) \quad (10)$$

where  $C_t$ ,  $C_e$ , and  $C_0$  are the moisture content at any time  $t$ , the moisture content at equilibrium  $e$ , and the initial moisture content at 0, respectively ( $\text{kg water/kg dry solid}$ ).  $R$  is the droplet radius (m) and  $D_{\text{eff}}$  is the effective moisture diffusivity ( $\text{m}^2 \text{s}^{-1}$ ).

The film formation can be achieved after the solvent is gradually evaporated and the solubility limits are exceeded. All previously mentioned phenomena interact with the formation of a uniform and coherent film.<sup>115,117</sup>

### Conclusion

Although the coating technology existed for years, it has significantly evolved from 1930. It has been applied in various industries, especially pharmaceutical, agricultural, and food. The coating solution mainly consists of solvent, polymer, plasticizer, colorant/opacifier, and antitack agents. Water is the most commonly used solvent. There are many polymers used in coating, as listed in Table 2. However, the natural polymers are the most preferred ones. Plasticizer, colorant/opacifier, and antitack agent are usually added to the coating formulation in order to improve the coating properties. In this article, four coating approaches were discussed: melt coating, supercritical fluid coating, compression coating, and spray drying coating. Spray drying coating (pan coating and air suspension coating) has offered considerable advantages, which practically enable film coating and multilayer coating of particles. Air suspension coating is classified into three groups: top-spray fluidized bed, tangential-spray fluidized bed, and bottom-spray fluidized bed. Top-spray fluidized bed coating is used in food industry because of its simplicity and low cost. Tangential-spray fluidized bed coating can be used in multilayer coating, although it suffers from some deficiencies. Bottom-spray fluidized bed coating (Wurster coater) is the most preferred coating equipment due to its excellent characteristics, which provide more homogenous coating of particles. Finally, the coating mechanism in fluidized bed coaters is discussed in detail. The coating process is divided into four major stages with different micro scale phenomena including particle movement, atomization, wetting the particle surfaces (droplet impact, spreading, and adherence), drying, and film formation.

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