CHAPTER 2 BACKGROUND

2.1 Rice

Rice (*Oryza sativa* L.) is one of the leading cereals worldwide and it is a staple food of over half of the world's population (Juliano, 1990). Rice can be consumed in different forms, namely brown rice, white rice and processed rice. The samples of processed rice are parboiled rice, instant rice, coated rice and germinated brown rice. The rice consumption depends on the region and life style of consumers. Rice not only is the sources of carbohydrate but also is the sources of dietary fibre, vitamins, minerals and phytochemicals. The nutrition values of rice depend on rice variety, growth location and rice forms. However, most consumers prefer the white rice than the brown rice since the palatability of cooked white rice is superior to cooked brown rice. But, in terms of the nutritional values the cooked brown rice. This is because the bran layers that are rich sources of minerals, vitamins and phytochemicals including antioxidants are removed from rice grain during milling process (Choi et al., 2007; Shen et al., 2009). Further, some vitamins and minerals are lost due to the washing and cooking of rice.

2.1.1 Effects of Milling, Washing and Cooking on Nutritional Values of Rice

Rice from harvesting is in the form of paddy or rough rice. Before consumption of rice, it needs to be processed to convert the rough rice to be the brown rice. The brown rice is then converted to white rice by polishing or milling. Polishing of brown rice leads to loss of vitamins and minerals as shown in Table 2.1. As can be seen in Table 2.1, for example, milling of brown rice removes 33.3% of thiamin (B1) and riboflavin (B2), 79.3% of niacin (B3), 53.1% of phosphorous, 53.4% of iron (Dexter, 1998). Dinesh-

Babu et al. (2009) reported that all of the dietary fiber and essential fatty acids are removed after milling. For the loss of vitamins and minerals due to washing and cooking of rice, Ranganathan et al. (1937) found that the losses resulting from washing and cooking were 75% for iron, 50% for calcium and phosphorus, 15% for calories and 10% for proteins. Kik and William (1945) reported that 43.1% thiamin, 25.9% riboflavin and 23% niacin were removed during washing but before cooking. They also revealed that 54% of thiamin, 48.1% of riboflavin and 41% of niacin were removed after cooking in excess water.

Vitamin and mineral content	Rice forms		
	Paddy	Brown rice	White rice
Calcium (mg/g)	0.3	0.1	0.1
Phosphorous (mg/g)	3.1	3.2	1.5
Zinc (ppm)	24	33	18
Iron (ppm)	38	8.8	4.1
Copper (ppm)	2.8	2.7	2.2
Thiamin (B1) ($\mu g/g$)	2.8	2.4	1.6
Riboflavin (B2) (µg/g)	0.5	0.3	0.2
Niacin (B3) (µg/g)	29.6	29	6
Folate ($\mu g/g$)	0.5	0.3	0.1
Biotin (ng/g)	91	48	43

 Table 2.1 Influence of milling on vitamins and minerals content of rice (Dexter, 1998)

For the phytochemicals of grains, Slavin et al. (1999) reported that phenolic acids, phytic acids, lignans and phytoestrogens are the main phytochemicals of grains. Phenolic acids including ferulic acid, p-coumaric acid and vanillic acids are prominent in the bran layer. Choi et al. (2007) studied the antioxidant activity of methanolic

extracts from some grains. They found that white rice had the lowest polyphenolics and antioxidant activity as compared to brown rice and black rice.

2.1.2 Rice Fortification and Coating

As mentioned above, it is seen that milling has significant effect on rice nutritional values. Thus, in the past the micronutrient deficiencies of the consumers who consume white rice as staple food are the main problem in many countries, particularly in the developing countries. For example, vitamin A deficiency is widespread in Bangladesh, India, Indonesia, Myanmar, Philippines, Sri Lanka, Nepal and Vietnam (Juliano, 1993). The deficiency of vitamin A causes Xerophthalmia which leads to night blindness and weakens the immune system, thereby increasing the risk of childhood morbidity and mortality (Sommer, 2008). The anemia that causes from the iron deficiency is one of the main problems in South Asia and Africa (Juliano, 1993). Before birth and during the first years of life, iron deficiency affects growth, neuro-development and cognitive performance (Lozoff et al., 2006). Beri-beri from thiamin deficiency is a common problem in the counties where white rice or polished rice is a staple food. In Thailand, for example, the new mothers restrict their diet, resulting in low thiamin content of breast milk predispose infants to beri-beri (Juliano, 1993). In addition to vitamin A, iron and thiamin deficiencies, the consumers who consume white rice also lack in riboflavin, calcium, zinc and folate. Thus, the preservation of rice nutrients or the fortification of white rice with vitamins or minerals is an alternative way to improve its nutritional values in order to reduce the micronutrient deficiencies in the population who are rice consumers, particularly the white rice consumers.

For the history of rice fortification, Dexter (1998) gave the information that in the United State the regulation was adopted in 1958 to establish as food standard for grain enrichment or fortification. A recent regulation requires that folic acid should be added to enriched rice. This is because the United State is concerned over low folate level in the diets of young women, which is a risk to have natural tube defects in infants born from folate deficiency mothers. In Canada, if the product is labeled enriched, the thiamin, niacin and iron must be added in those products. In Japan, the multinutrient enriched rice has been on the market since 1981. The raw rice was enriched with pantothenic acid, vitamin E and calcium, in addition to thiamin, niacin, riboflavin and iron. The enriched rice in Japan is well known as "Shingen". Moreover, the rice fortification has been followed in Philippines and Indonesia. In Philippines, the process of fortification of rice with thiamin, niacin and iron was developed by Hoffmann-La Roche Company. In Indonesia, rice is fortified with vitamin A. The fortified grains were made from rice flour extrusion to form a rice kernel shape. The vitamin D fortified grains are then blended with unfortified grains to reach target level of vitamin D in the final product.

Rice fortification can be performed by many methods such as direct mixing, coating, soaking and extrusion. For the direct mixing method, the solution which is the mixture of vitamins or minerals is mixed directly into rice kernels in the mixing container (Peil et al., 1981) or in rotating containers such as pan or drum. This method is easy and less expensive. However, it provides low coating uniformity and high nutrient losses if the fortified rice is rinsed before cooking (Dexter, 1998). The second method for rice fortification is coating, which is common and most popular. The example of coating methods are dipping, spraying and panning. For coating by spraying, the solution of

vitamin or mineral is sprayed onto the rice kernels surface (Lee et al., 1995; Shrestha et al., 2003; Kyritsi et al., 2011) in the rotating pan or rotating drum. The fortification of rice can also be performed by soaking method. With soaking method, the rice kernels are soaked in the solution of vitamin or mineral for a period of time and then they are dried to reduce the moisture content (Lee et al., 1995; Kyritsi et al., 2011; Prom-u-thai et al., 2010). The final method of rice fortification is performed via the extrusion process. The vitamins or minerals are added into rice flour and then it is extruded to form the rice kernel shape, which is known as artificial grains (Dexter, 1998).

2.2 Natural Antioxidants

Consumers are increasingly interested in foods that are rich in antioxidants because antioxidants play an important role in the low risk of cancers and cardiovascular (Gerber et al., 2002; Kris-therton et al., 2002), as well as neurodegenerative diseases including Parkinson's and Alzheimer's diseases (Di Matteo and Esposito, 2003). There are two main groups of antioxidants including natural antioxidants and synthetic antioxidants. However, consumers are more interested in natural antioxidants than synthetic antioxidants. The natural antioxidants are widely found in fruits, vegetables, spices and herbs. They are present in different forms such as flavonoids, phenolic acids and carotenoids (Brewer, 2011). Turmeric rhizome (*Curcuma longa* L.) is widely used as food ingredient, food additive and coloring agent in many food products (Jayaprakasha et at., 2005; Selvam et al., 1995). The major active chemical constituent of turmeric rhizome is curcuminoids which consist of curcumin, demethoxycurcumin and bisdemethoxycurcumin (Jayaprakasha et al., 2005). Curcuminoids are yelloworange color which has been found to be a rich source of phenolic compounds that possess high antioxidant activity. Previously, much research has reported about the biological, physiological and chemical properties of the curcuminoids, for example antiinflammatory, antimicrobial, antiparasitic, antitumor, antiviral and antimutagenic (Polasa et al., 1991; Selvam et al., 1995; Ruby et al., 1995; Ahsan et al., 1999; Masuda et al., 1999; Ramsewak et al., 2000; Jayaprakasha et al., 2006; Ak & Gülçin, 2008; Itokawa et al., 2008). The application of turmeric rhizome is interested in food products since curcuminoids are moderately stable to heat at the temperature below 100 °C (Khatun et al., 2006; Wang et al., 2009; Temitope et al., 2010; Paramera et al., 2011). Commandeur and Vermeulen (1996) suggest that daily intake of curcuminoids should not be higher than 100 mg/day and WHO (Series 52, 2004) recommends that the acceptable daily intake (ADI) for curcuminoids is 1-3 mg/kg body weight.

2.3 Coatings

Coatings are an important process in many industries such as pharmaceutical, food and agricultural industry. In food industries, coating has been applied to many objectives for an example to improve the shelf life, color, ingredient and functionality, including coating to protect coated foods from environment (Kester and Fennema, 1986; Bourtoom, 2008; Pavlath and Orts, 2009). Food coatings can be performed by various ways such as panning, dipping and spraying. Each coating method provides different advantages and disadvantages. Selection of the coating method depends on many factors such as coating quality, production cost, time as well as the characteristics of foods to be coated such as shape, surface property and size. Moreover, the coating method should be matched with the properties of coating solution, namely viscosity, density and surface tension. In this section, each method is discussed to highlight both the advantages and disadvantages.

2.3.1 Pan Coating

Pan coating has been known since the 9th century. Its origin comes from Greek-Arabian cultures where it was applied to coat the medicine tablets (Andrade et al., 2012). With pan coating, the core material is fed into the rotating pan or rotating drum. Then, the coating solution is poured or sprayed into the rotating pan. The tumbling of material due to the rotating pan helps to distribute the coating solution over the surface of material to be coated. During the rotating of pan, an air either at ambient temperature or elevated temperature is supplied to the rotating pan to reduce the moisture of coated material (Dangaran et al., 2009; Pandey et al., 2006). Pan coating is applied in many industries such as pharmaceutical, confectionery and chocolate industries. This is because it is simple to use, low capital cost and available for many material sizes. Pan coating can also be applied to coat the nuts and raisins (Lee et al., 2002; Geschwindner and Drouven, 2009). The disadvantages of pan coating are that the coating uniformity is not good and the thickness of coating layer is difficult to control.

2.3.2 Dip Coating

Dip coating is a very simple coating process. It can be performed by submerging the product directly into the container of coating solution. After dipping, the product is drained to remove the excess coating solution at the surface and then dried either at room temperature or with the dryer to the desired moisture content. The advantage of this coating method is that the coating solution can cover the entire surface of the product, which provides good uniformity around the complex and rough surface (Andrade et al., 2012). However, several problems may occur during coating by dipping, including the dilution of coating solution after dipping for a period of time and the contamination of coating solution with trash or dirt and microorganism. This coating

method is also difficult to control the thickness of coating layer, which may have the problems with some products that require an accurate amount of coating layer (Grant and Burns, 1994; Martin-Belloso et al., 2009). Another disadvantage of dip coating method is that the coating solution will be diluted and hence the layer of food surface is degraded leading to problems with its functionality. For example, the natural wax layer of fruits and vegetables has to be removed after coating by dipping method (Lin and Zhao, 2007).

2.3.3 Spray Coating

Spray coating has been used in many food processes since it provides the coating uniformity and can control the thickness of the coating layer (Grant and Burns, 1994). This coating method is performed by spraying the coating solution onto the surface of the product. Spraying can be applied to deposit various kinds of coating solution onto the foods surface (Debeaufort and Voilley, 2009; Antoniewski et al., 2007). Spray coating are also suitable for coating the food products when dual or more coating layers are required.

2.4 Fluidization

2.4.1 Principle of Fluidization

Fluidization can be used in many industries such pharmaceutical, chemical, agriculture and food. This is because this technique provides a high heat and mass transfer rate. Fluidization occurs when a fluid either gas or liquid is passed upwards through the bed of particles. At low fluid velocities the bed of particles remains stationary. At this state the bed of particles is called fixed bed or packed bed as shown in Figure 2.1(a). When increasing the fluid pressure, the particles in the bed start to separate and move away from another particle. On increasing the fluid pressure further, the state of particles will be reached at which the drag force exerted by the fluid on the particle is balanced by the net weight of the particle. The particles are now suspended in the upward moving stream of fluid. This is the point of minimum fluidization as presented in Figure 2.1(b). With increasing the fluid pressure beyond the minimum fluidization point the fluidized particles will be in the state of bubbling fluidized bed as shown in Figure 2.1(c). After this point, if fluid pressure is further increased the fluidization of particles will be slugging (Figure 2.1(d)) and turbulent (Figure 2.1(e)), respectively. Finally, at fluid pressure further beyond the turbulent fluidization point, the movement of particles acts as pneumatic transport as shown in Figure 2.1(f) (Kunii and Levenspiel, 1991; Smith, 2007; Szafran, 2013).

2.4.2 Minimum Fluidization Velocity

Normally in practice, fluidized bed is operated at superficial velocity of fluid. The superficial velocity of fluid relates to the minimum fluidization velocity. Superficial velocity has the greatest influence on the behavior of fluidized particles. Consequently, the knowledge of minimum fluidization velocity is important to the operation of fluidized bed. The minimum fluidization velocity can be calculated by Carman-Kozeny equation and Ergun equation. The Carman-Kozeny equation is suitable to calculate the minimum fluidization velocity of fine particles that have the diameter below 500 μ m. For the large particles that have the diameter higher than 600 or 700 μ m, the Ergun equation is more accurate. The Ergun relationship is a semi-empirical equation for the pressure drop per unit bed depth, which contains two terms.



Figure 2.1 Different state of fluidization occurring when a bed of particles is subjected to increasing gas flow rate (Kunii and Levenspiel, 1991)

The Ergun's equation can be expressed as:

$$\frac{\Delta P}{H} = \frac{150(1-\varepsilon)^2 \,\mu_{\rm f} u_{\rm f}}{\varepsilon^3 \phi_{\rm p}^2 d_{\rm p}^2} + \frac{1.75(1-\varepsilon) \rho_{\rm f} {u_{\rm f}}^2}{\varepsilon^3 \phi_{\rm p} d_{\rm p}}$$
(2.1)

The first term in equation (2.1) represents the pressure loss due to the viscous drag and the second term is the kinetic energy losses, which are significant at higher velocities. Equation (2.1) is valid in the range 1 < Re < 2000 of Reynolds number. The Reynolds number can be calculated from equation (2.2).

$$\operatorname{Re} = \frac{\rho_{\mathrm{f}} u_{\mathrm{f}} d_{\mathrm{p}}}{\mu_{\mathrm{f}}}$$
(2.2)

At the minimum fluidization velocity, the Ergun's equation can be expressed as:

$$\frac{\Delta P_{\rm mf}}{H_{\rm mf}} = \frac{150(1 - \varepsilon_{\rm mf})^2 \,\mu_{\rm f} u_{\rm mf}}{\varepsilon_{\rm mf}^3 \phi_{\rm p}^2 d_{\rm p}^2} + \frac{1.75(1 - \varepsilon_{\rm mf}) \rho_{\rm f} u_{\rm mf}^2}{\varepsilon_{\rm mf}^3 \phi_{\rm p} d_{\rm p}}$$
(2.3)

The pressure drop across the bed at minimum fluidization velocity can be calculated by the following equation.

$$\Delta P_{\rm mf} = (\rho_{\rm p} - \rho_{\rm f})(1 - \varepsilon_{\rm mf})gH_{\rm mf}$$
(2.4)

Substituting equation (2.4) into equation (2.3);

$$(\rho_{\rm p} - \rho_{\rm f})g = \frac{150(1 - \varepsilon_{\rm mf})\mu_{\rm f}u_{\rm mf}}{\varepsilon_{\rm mf}^{3}\phi_{\rm p}^{2}d_{\rm p}^{2}} + \frac{1.75\rho_{\rm f}u_{\rm mf}^{2}}{\varepsilon_{\rm mf}^{3}\phi_{\rm p}d_{\rm p}}$$
(2.5)

The equation (2.5) can be simplified by multiplying with $\frac{\rho_{\rm f} d_{\rm p}^{-3}}{\mu_{\rm f}^{-2}}$

$$\frac{\rho_{\rm f}(\rho_{\rm p}-\rho_{\rm f})d_{\rm p}^{3}g}{\mu_{\rm f}^{2}} = \frac{150(1-\varepsilon_{\rm mf})u_{\rm mf}d_{\rm p}\rho_{\rm f}}{\varepsilon_{\rm mf}^{3}\phi_{\rm p}^{2}\mu_{\rm f}} + \frac{1.75\rho_{\rm f}^{2}u_{\rm mf}^{2}d_{\rm p}^{2}}{\varepsilon_{\rm mf}^{3}\phi_{\rm p}\mu_{\rm f}^{2}}$$
(2.6)

The equation (2.6) can be put into form:

$$Ga = \frac{150(1 - \varepsilon_{mf})}{\varepsilon_{mf}^{3} \phi_{p}^{2}} Re_{mf} + \frac{1.75\rho}{\varepsilon_{mf}^{3} \phi_{p}} Re_{mf}^{2}$$
(2.7)

It is seen that the equation (2.7) is in the form of quadratic equation and Ga is Galileo number which defined by equation (2.8)

Ga =
$$\frac{\rho_{\rm f} (\rho_{\rm p} - \rho_{\rm f}) d_{\rm p}^{3} g}{{\mu_{\rm f}}^{2}}$$
 (2.8)

For the particle Reynolds number at minimum fluidization velocity can be written as equation (2.9)

$$\operatorname{Re}_{\mathrm{mf}} = \frac{\rho_{\mathrm{f}} u_{\mathrm{mf}} d_{\mathrm{p}}}{\mu_{\mathrm{f}}}$$
(2.9)

where,	ΔP	=	Bed pressure drop (Pa)
	Η	=	Bed height (m)
	З	=	Void fraction
	$\mu_{ m f}$	=	Fluid viscosity (kg/m.s)
	$\phi_{ m p}$	=	Particle sphericity
	$u_{\rm f}$	=	Fluid velocity (m/s)
	d_{p}	=	Particle diameter (m)
	$ ho_{ m f}$	=	Fluid density (kg/m ³)
	$\varepsilon_{ m mf}$	=	Void fraction at minimum fluidization
	U_{mf}	=	Minimum fluidization velocity (m/s)
	$ ho_{ m p}$	=	Particle density (kg/m ³)
	$H_{ m mf}$	=	Bed height at minimum fluidization (m)

From equation (2.7), the particle Reynolds number can be solved and then the minimum fluidization velocity will be obtained from equation (2.9). For more details about the minimum fluidization velocity calculation can be seen in Kunii and Levenspiel (1991) and Smith (2007).

2.5 Fluidized Bed Coating

Fluidized bed coating or air suspension particle coating was originally developed for pharmaceutical industries. It has been developed by Dale Wurster in the 1950's for coating the tablets (Jones, 1985; Jones, 1994). Thus, this coating technique is well known in the name of 'Wurster process' (Jones, 1985; Jone, 1994; Arshady, 1993). The objectives of coating in pharmaceutical industry are controlled release, taste making, enteric release, improved stability or aesthetics (Hall and Pondell, 1980; Mehta et al., 1986). The advantages of fluidized bed coating are that it is flexible to use and it can be applied for many particles with a wide range of particle sizes. For the application of fluidized bed coating in food industries, it is in an increasingly trend with a wide variety of coating objectives such as coating to improve the food ingredients, additives, color and so on. The fluidized bed coating can be classified into three configurations based on the nozzle position, namely top-spray, bottom-spray and tangential-spray fluidized bed coating (Jones, 1985; Jones, 1994; Teunou and Poncelet, 2002; Dewettinck and Huyghebaert, 1998).

2.5.1 Top-Spray Fluidized Bed Coating

The conventional top-spray fluidized bed coating is shown in Figure 2.2. It is evolved from the fluidized bed dryer and commercialized more than 30 years ago (Jones, 1994). The core particles are fed into the coating chamber which is typically a cylinder or conical cylinder. A gas distribution plate is equipped at the base of coating chamber. An air at sufficient velocity is drawn through the distributor plate to accelerate the particles to move as fluidization. The fluidized particles are moved from the bottom of bed past to the nozzle. During the movement of fluidized particles, the coating solution is sprayed countercurrently onto the surface of fluidized particles (Jones, 1994; Dewettinck and Huyghebaert, 1999; Szafran, 2013). The fluidized particles that are presented at the top of bed receive the droplet of coating solution. The region that particle receives the droplets is called coating zone. The size of coating zone depends on many factors such initial bed height, fluidizing air velocity, nozzle position and so on. After particles receive the droplet at the coating zone, they will fall back to the bottom of bed and continue cycling throughout the coating process (Jones, 1988). The batch top-spray fluidized bed coating has the capacity ranging from few hundred grams to more than 1500 kg (Jones, 1994). It can be used to coat a wide particle sizes. Jones (1988) was successful coating the particle that had the diameter as small as 100 μ m with top-spray fluidized bed. However, Dewettinck and Huyghebaert (1998), Guignon et al. (2002) and Chen et al. (2009) suggested that the size of particles coated with top-spray fluidized bed should be larger than 100 µm since the particles with small size do not have a stable fluidization state and moreover, they have the risk to agglomerate during coating. The main disadvantage of top-spray fluidized bed coating is that it is difficult to control the distance of droplet traveling before impacting on the particle surface.

Consequently, the premature droplet evaporation, droplet carried out and coating imperfection can occur.



Figure 2.2 Top-spray fluidized bed coater (Turton and Cheng, 2005)

2.5.2 Bottom-Spray Fluidized Bed Coating

In 1959, Dr. Dale Wurster at the University of Wisconsin developed the air suspension particle coating technique, namely 'Wurster Process'. Initially, it was designed to coat the medicine tablets. The process is now widely applied for many particles both small and intermediate size (Jones, 1994). The sample of batch bottom-spray fluidized bed coating is presented in Figure 2.3. Normally, the coating chamber of bottom-spray fluidized bed coating may be cylindrical or slightly conical. Inside the coating chamber, the draft tube with half diameter of the coating chamber is located. The draft tube is placed inside the coating chamber to control the particle movement. The distance between the draft tube and air distributor plate is adjustable to create ordered particle movement. The nozzle is placed axially upward at the bottom of bed (Jones, 1994; Dewettinck and Huyghebaert, 1999; Szafran, 2013). The air stream at sufficient velocity is supplied to the bed through the distributor plate. The open area of distributor plate which is under the draft tube is typically fully open, allowing the air at high volume and velocity to accelerate the particles passing through the draft tube. During the movement of particles in the draft tube, the coating solution is sprayed from the nozzle. Consequently, coating with bottom-spray fluidized bed provides the coating uniformity and coating efficiency better than top-spray fluidized bed coating. This is because the movement of particles is more controlled, which is opposed to the top-spray that the direction of particle movement is uncontrolled. Moreover, the droplets, fluidized particles and fluidizing air are moved in the same direction, therefore the distance between the droplets and fluidized particles are short. Hence, the premature droplet evaporation is reduced (Jones, 1994; Srivastava and Mishra, 2010; Dewettinck and Huyghebaert, 1999; Szafran, 2013). The capacities of batch bottom-spray fluidized bed coating are in the range from few hundreds grams to approximately 600 kg (Jones, 1994). The disadvantages of bottom-spray fluidized bed coating is that during coating process the nozzle has risk of clogging and sticking of particle to the nozzle.



Figure 2.3 Bottom-spray fluidized bed coater (Turton and Cheng, 2005)

2.5.3 Tangential Spray or Rotary Fluidized Bed Coating

The third and most recently developed fluidized bed coating configuration is the tangential spray or rotary fluidized bed coating as shown in Figure 2.4. This system is equipped with a rotating plate. The air is supplied to the bed via a gap between the rotating plate and the inner wall of the coating chamber. The centrifugal force by the rotating plate causes the particle movement as spiral pattern. The nozzle is positioned to spray the coating solution onto the particles surface tangentially. The droplet of coating solution moves concurrently in the direction of particles. Moreover, the spiral motion of the particles provides the high separation force that helps to prevent particle agglomeration. Thus, this coating system is intended for coating high density particles, grain or pellet to obtain a thick coating layer. However, the high kinetic energy of accelerated particles of this coating system makes it difficult to coat the large and non sphere particles. The batch capacities of tangential spray or rotary fluidized bed coating

range from approximately 0.5 kg up to 500 kg (Jones, 1985; Jones, 1994; Dewettinck and Huyghebaert, 1999; Srivastava and Mishra, 2010; Szafran, 2013).



Figure 2.4 Tangential or rotary fluidized bed coater (Turton and Cheng, 2005)

2.5.4 Continuous Fluidized Bed Coating

For some food industries, coating with batch fluidized bed may provide the high production cost and low production output. Thus, the design of continuous fluidized bed coating is more practical. The horizontal fluidized coating as shown in Figure 2.5 is one of the continuous coating system. The food material is continually fed at the one side while the coated product is discharged at the end of the bed. The difficulty of continuous coating system compared to the batch fluidized bed coating is that the coating uniformity and coating layer thickness depend on the residence time of particles that are present in the bed. Coating which requires a long residence time to obtain a high coating uniformity of coating layer needs a longer length horizontal continuous fluidized coater. The use of the continuous fluidized bed coating for food materials is widely available such as coating of nuts, dried fruits, corn flakes, puffed grain and so on (Teunou and Poncelet, 2002).



Figure 2.5 Horizontal fluid bed coater (top spray and continuous) (Teunou and Poncelet, 2002)

2.6 Atomization of Coating Solution

The atomization is an important component of spray coating process. Normally, the coating solution is sprayed via the nozzles. There are many kinds of spraying nozzles such as centrifuge, air pressure and ultrasonic nozzle (Guignon et al., 2002). These nozzles can be classified by the energy used to carry the coating solution (Guignon et al., 2002) or classified by the pattern of spraying droplets such as hollow cone, full cone and flat spray (Andrade et al., 2012). The nozzle typically used in the fluidized bed coating is the pressure nozzle (binary or two-fluid nozzle) (Dewettinck and Huyghebaert, 1999) as shown in Figure 2.6. The two-fluid nozzle is popularly used in

fluidized bed coating because the droplet size and the droplet distribution are more controllable than with a hydraulic and centrifugal nozzle (Hall and Pondell, 1980; Filkova and Mujumdar, 1995). With two-fluid nozzle, very strong shear forces are developed at the gas–liquid interface, generating waves which break the coating solution into the droplets. There are two types of two-fluid nozzle, namely internal and external mixing two-fluid nozzle. For the internal mixing two-fluid nozzle, the coating solution and atomizing air are supplied to the mixing part inside the nozzle and then the droplets are discharged through an exit orifice (Hede et al., 2008). For the external mixing two-fluid nozzle, the coating solution and atomizing air are impacted at the exit orifice.

The main key characteristics of two-fluid nozzle that should be considered are the spray rate, atomization air pressure, spray angle and droplet penetration depth, spatial distribution of droplets and droplet size. With two–fluid nozzle, the spray rate increases with the atomizing air pressure which usually varies between 0.5 and 3.5 bars. An increase in atomizing air pressure provides the smaller droplet and the spray angle, leading to high droplet penetration depth (Bayvel and Orzechowski, 1993; Dewettinck and Huyghebaert, 1998).

For the flow rate of coating solution supplied to two-fluid nozzle normally varies between 1 and 70 g/min. However, the spray rate should match with the drying capacity in the coating chamber to prevent the over spraying. With two-fluid nozzle where the coating solution is sprayed at an atomization air pressure ranges 0.5-3.5 bar and the feed rates of coating solution ranges 1-70 g/min; the droplet size is usually in the range of

10–40 μ m. In practice, the droplet size must be appropriate with the size of particle to be coated (Liu and Lister, 1993; Hall and Pondell, 1980; Kleinbach and Riede, 1995).



Figure 2.6 External mixing two-fluid nozzle (modified from Guignon et al., 2002).

2.7 Collision Mechanisms of Droplet on Particle Surface

During spraying the coating solution onto the fluidized particles, the collisions between the droplets of coating solution with fluidized particles usually occur in the coating zone. The collision probability in the coating zone depends on the characteristics of the droplet (size and velocity) and particle properties (size, velocity and shape), as well as the movement of fluidized particles and the nozzle position. There are three collision mechanisms that are possible during fluidized bed coating as shown in Figure 2.7. In the first case, if the droplet passes close to the particle surface, the collision will occurs by the interception mechanisms as shown in Figure 2.7(a). In the second case, if the particle is on the trajectory of the droplets, the collisions will be occurred by inertia force as shown in Figure 2.7(b). Finally, in the case of small droplet (inferior to 0.001 mm), the collisions may be due to the brownian diffusion with electrical interactions as shown in Figure 2.7(c). However, for the fluid bed coating where the sizes of droplet ranges from 0.01–0.04 mm and the size of particle ranges from 0.1–1 mm, the collisions mainly occur due to the interception or inertia. The quantity of droplets that impact particle depends on the density of droplets, size and their rate compared to those of particles. The probability of adhesion in the coating zone is defined as the ratio between the mass flow rate of droplets caught by the unit area of particle, and the mass flow density of droplets (Link and Schlünder, 1997; Guignon et al., 2002).



Figure 2.7 Collisions of coating solution droplet on particle surface

(Guignon et al., 2002)

2.8 Coating Layer Growth Mechanisms

For fluidized bed coating, there are many researches which revealed that particles are only coated in the small region which is near the nozzle (Smith and Nienow, 1982; Smith and Nienow, 1983; Maronga and Wnukowski, 1997; Maronga and Wnukowski, 1998). This region is called wetting zone or coating zone. The size of coating zone

depends on the droplet penetration depth of the spray and the position of the nozzle. In coating zone the droplet formation, droplet adhesion on the particle surface, spreading of droplet on the particle surface and solvent evaporation occur almost simultaneously (Guignon et al., 2002). The repeated cycle of coating and drying of particles in the coating chamber results in the creation of a uniform coating layer around the individual particle. When the coating solution is sprayed onto the fluidized particles, two different particle growth modes may occur, depending on the spray rate of coating solution and the evaporation capacity of fluidizing air (Hemati et al., 2003). Figure 2.8 shows two different growth modes that may occur in fluidized bed coating. If coating is operated at low evaporation capacity which is not enough to evaporate the liquid on the particles surface, the coated particles may be wetted and the wet bridge is then formed between them. The formation of liquid bridge depends on the bridge strength and the kinetic energy of collided particles. This liquid bridge may persist beyond the point of solidification or drying and consequently, the particles agglomeration is formed as shown in Figure 2.8(b) (Becher and Schiunder, 1997; Saleh et al., 1999). At this point, if the coating solution is still sprayed onto the fluidized particles the rate of liquid bridge formation could become too high, resulting in the large agglomeration with subsequent collapse of fluidized bed.

In the case where fluidized bed coating is operated at sufficient evaporation capacity, which means the spray rate of coating solution matches appropriately with the evaporation capacity of fluidizing air. The coating solution that adheres on the particle surface will sufficiently dries before coated particles collide with other coated particles. Consequently, the formation of liquid bridge does not occur (Link and Schlunder, 1997).

The coating solution can be formed as the coating layer on the particle surface as shown in Figure 2.8(a)



Figure 2.8 Coating layer growth (a) and agglomeration (b) during coating with fluidized bed (Ronsse, 2006)

2.9 Key Process Variables for Fluidized Bed Coating

Although fluidized bed coating has been developed for pharmaceutical industries for a long time, but in the food industries this coating technique is still a complex coating process. This is because this coating process consists of three major sub-processes, including fluidization, atomization and drying, which occur almost simultaneously during the coating process (Jones, 1985; Maa et al., 1996 and Dewettinck and Huyghebaert, 1999). All sub-processes involve with as many as 20 different variables as

presented in Table 2.2 (Werner et al., 2007). These variables can be classified as process variables, system design variables and properties of coating solution and the particles to be coated. Performance of coating such as coating quality, coating efficiency and energy efficiency depend on these variables. Some variables have direct effect on the performance and some variables have indirect effect on the coating performance. Therefore, in order to know the results from coating experiment, it is necessary to study these complexities by studying a single operation at a time (Werner et al., 2007).

 Table 2.2 Key variables for fluidized bed coating

Fluidization	Drying air flow rate ^a
	Equipment type and dimensions (top spray, bottom spray etc.) ^b
	Substrate size, size distribution, density ^d
	Substrate surface character (charge or chemical composite, i.e., fat-
	cintainning) ^d
	Batch size ^{a,b}
Atomization	Spray mode (top, bottom etc.) ^b
	Droplet size ^a
	Nozzle design (pneumatic, ultrasonic, rotary etc.) ^b
	Nozzle distance from bed ^a
	Atomizing air flow rate (pneumatic) ^a
	Coating spray rate ^a
	Coating solution viscosity, surface tension, density ^c
Drying	Inlet air temperature ^a
	Drying air flow rate ^a
	Outlet air relative humidity ^a
	Liquid coating concentration ^a
	Atomizing air flow rate ^a

The letters a, b, c and d that are superscript in Table 2.2 mean process variables, system design variables, physical properties of coating solution and physical properties of core particle, respectively.

2.10 Performance of Fluidized Bed Coating

Performance of fluidized bed coating can be classified as coating efficiency, coating quality, energy efficiency as well as productivity efficiency (Maa et al., 1996 and Teunou and Poncelet, 2002). Usually, performance of fluidized bed coating is defined in terms of coating efficiency (*CE*) as presented in equation (2.1) (Dewettinck and Huyghebaert, 1999).

$$CE = \frac{W_{\rm c}}{W_{\rm cs} \times D_{\rm m}} \times 100 \quad \% \tag{2.1}$$

where $W_c = W_p(w/(1-w))$ is the deposited mass of coating (kg)

- *w* is the coating content on particles surface (kg/kg)
- $W_{\rm p}$ is the mass of core particles (kg)
- W_{cs} is the mass of coating solution sprayed onto the fluidized particles (kg)
- $D_{\rm m}$ is the dried matter of coating solution on the coated particles (kg/kg)

There are several investigations studied the effects of operating parameters on the coating efficiency of top-spray fluidized bed coating. For example, Dewittinck and Huyghebaert (1998); Saleh et al. (2003); Ronsse et al. (2008) investigated the effect of particle size on the coating efficiency. They found that an increase in particle size provided the lower coating efficiency as shown in Figure 2.9. This can be explained by

the fact that the smaller particles have the larger specific area and thus they can capture more droplet than the larger particles. Ronsse et al. (2008) also gave more information that an increase in particle diameter without modifying the fluidizing air velocity provided a lower fluidized bed height. Thus, the distance between the fluidized particles and the nozzle was higher, resulting in a larger fraction of droplets that evaporate completely before impacting the surface of fluidized particles.



Figure 2.9 Effect of particle sizes of the coating efficiency of top-spray fluidized coating (Saleh et al., 2003)

The effect of atomization air pressure on the coating efficiency of top-spray fluidized bed coating was studied by Dewittinck and Huyghebaert (1998) and Ronsse et al. (2008). They found that coating efficiency of top-spray fluidized bed coating was increased with increase in atomization air pressure. Although, at higher atomization air pressure provides the smaller droplet, which is possible to completely evaporate before collision with fluidized particles. But, at a higher atomization air pressure the atomization air at ambient temperature is directly supplied to the two-fluid nozzle. Thus, an increase of atomization air pressure increases the volumetric flow rate of compressed air, resulting in the cooling effect in fluidized bed as shown in Figure 2.11. Consequently, the loss of droplet due to premature droplet evaporation was lower as shown in Figure 2.10 (Ronsse et al., 2008).



Figure 2.10 Effect of atomization air pressure on spray drying loss

(Ronsse et al., 2008)



Figure 2.11 Effect of atomization air pressure on bed temperature (Ronsse et al., 2008)

The effect of inlet fluidizing air temperature on the coating efficiency of top-spray fluidized bed coating, it was studied by Dewittinck and Huyghebaert (1998) and Kage et al. (1996). They found that inlet fluidizing air temperature had significant effect on the coating efficiency. An increase in inlet fluidizing air temperature led to the lower coating efficiency as shown in Figure 2.12. This is because the loss of coating solution droplets due to premature droplet evaporation increased at a higher inlet fluidizing air temperature. However, at low inlet fluidizing air temperature the evaporation capacity may not be enough to evaporate the solvent on particle surface. Hence, the particle may be wetted and the agglomeration of wetted particle will occur. This is a risk to the defluidization of fluidized particles and resulting in the collapse of fluidized bed.



Figure 2.12 Effect of bed temperature on coating efficiency (Kage et al., 1996)

The effects of spray rate of coating solution on the coating efficiency of top-spray fluidized bed coating was studied by Kage et al. (1996). They found that an increase in spray rate of coating solution provided a higher coating efficiency as depicted in Figure 2.13. This is because an increase in spray rate of coating solution increased the air humidity in the coating chamber particularly at the coating zone. When the air humidity in the coating zone is higher, the premature droplet evaporation will decrease. However, the spray rate of coating solution should match with the evaporation capacity of fluidizing air to prevent the agglomeration of coated particles.



Figure 2.13 Effect of spray rate on coating efficiency (Kage et al., 1996)

For the coating quality, it is classified as coating mass uniformity and coating morphology (Turton et al., 1999). Coating mass uniformity refers to the variation in the amount of coating layer on each coated particle. Figure 2.14 shows a variation in coating mass uniformity. In Figure 2.14, if particle (a) and (b) has the same size, density, surface characteristics and porosity but after coating they have different masses of coating layer, indicating that the coating mass uniformity is different (Turton et al., 1999). In pharmaceutical industries, the coating mass uniformity is very important when an active ingredient is coated onto the tablets. Cheng and Turton (2000) divided the variation in spray coating uniformity into two classes, including the variations due to the number of particle passing through the coating zone and the variations due to the amount of coating solution deposited per particle per pass through the coating zone.



Figure 2.14 Variation in coating mass uniformity (a) uniform, thin coating layer;(b) uniform, thick coating layer (Werner et al., 2007)

Coating morphology is one of the indicators that indicate the coating quality. Coating morphology refers to the variation of a given property between particles containing the same amount of coating layer (Turton et al., 1999). Figure 2.15 illustrates the variation in distribution of coating layer on the particle surface. All three particles have the same diameter and the same mass of coating layer; however, in Figure 2.15(b) it can be seen that the coating morphology also refers to the appearance or composition of the coating layer, which may contain amorphous, crystalline structures, porosity and fissures as shown in Figure 2.16. From Figure 2.16(a) and Figure 2.16(b), it is seen that the coating layer on particle surface appears as the fine crack. This crack result from the shrinkage during drying process (Link and Schlunder, 1997; Dewettinck et al., 1999). Guignon et al. (2002) suggested that it was difficult to predict the final structure of coating layer; it might be smooth film or non continuous film.



Figure 2.15 Variation in coating morphology (a) uniformly coated particle; (b) non uniformly coated particle; (c) coated particle with fissure (Werner et al., 2007)



Figure 2.16 Fracture in coating surface (a) PVP coated particle (Link and Schlunder, 1997); (b) hydrolysed gelatin coated particle (Dewettinck et al., 1999)