DRYING OF MICROPARTICLE SLURRY AND SALT-WATER SOLUTION BY A POWDER-PARTICLE SPOUTED BED

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Key Words: Drying, Microparticles, Slurry, Fluidized Bed, Spouted Bed

Adhesion and agglomeration between fine particles and coarse particles are very strong for fine particles under several microns in diameter. The powder-particle spouted bed (P-PSB) was developed as a new type of dryer for continuous drying of microparticles under several microns in diameter. The powder-particle spouted bed dryer is a cone-bottomed fluidized bed without a gas distributor. Coarse particles were loaded into the dryer and the hot gas was fed to it from the cone-bottom so that coarse particles were fluidized violently, and a microparticle slurry and a salt-water solution were continuously fed to it.

Continuous drying of a microparticle slurry and a salt-water solution were carried out by using the powder-particle spouted bed, and the drying characteristics were investigated. The moisture content of the microparticles dried by this process from the submicroparticle (under $1\mu\text{m}$ in diameter) slurry and the salt-water solution were nearly equal to their equilibrium moisture content over the range of the experiment. When the drying efficiency was above 60%, agglomerates were observed in the bed. The relative humidity of the outlet gas and the drying efficiency can be calculated by mass and heat balances across the bed.

Introduction

Drying of microparticles is of importance in industry because of their attractive properties. Usually, microparticles are produced by liquid phase reactions and wet grinding processes, in such production processes require drying of microparticles, so it is important to develop an efficient dryer of microparticles.

The fluidized bed has been widely used in gas-solid contact operations and has been applied to the drying of solid particles, because of its greater specific heat capacity and higher drying rate (Toei and Matsuno, 1965). However, it is rather difficult to fluidize fine particles such as group C of Geldart classification (Geldart, 1973) in a normal fluidized bed, because fine particles agglomerate easily.

Recently, a type of fluidized bed dryer that prevents microparticles from agglomeration was developed (Kato *et al.*, 1992). This type of fluidized bed is called a powder-particle fluidized bed (P-PFB) and the principle of it is as follows. A fine particle powder is fed to a bed in which coarse particles are fluidized. Agglomerates of the particles are crushed by collisions with coarse particles, so the fine particles are uniformly dispersed in the bed and fluidized. Only the fine particles are elutriated from the column, based on the difference in the terminal velocity of the particles. The fine particles seem to move through the bed while adhering to the coarse particles. The average residence time of the fine particles in the bed was considerably

longer than that of the gas (Kato *et al.*, 1991), so that they have enough time to contact with hot gas and can be perfectly dried. Since the rates of mass and heat transfer between the fluids and the particles in the fluidized bed are very fast in general, the powder-particle fluidized bed has been used as an efficient dryer of fine particles (Nakagawa *et al.*, 1992).

In our present work, drying of a microparticle slurry and a salt-water solution were conducted by using the powder-particle fluidized bed without a gas distributor. **Figure 1** shows a scheme of the bed. If coarse particles are poured into a cone-bottomed column having a small diameter gas inlet at the base of the cone and subjected to an upward hot gas flow and a downward slurry, the following steps will occur. At some gas velocities, a stream of coarse particles will rise rapidly as a central core, or spout, and contact with falling slurry within the bed. The particles in the spout, having reached a height somewhat above the bed level, fall back onto the annular space around the spout and travel downward uniformly. A considerable intensive cross flow of particles from the annulus into the spout takes place all along the bed height. During this period, the slurry experiences a constant rate drying phase and a deceleration drying phase, and finally, the slurry of microparticles is dried and the dried microparticles elutriate. We call this type of fluidized bed a powder-particle spouted bed.

In this study, drying of a microparticle slurry and a salt-water solution were conducted in a powder-particle spouted bed. The slurry or solution of salt was continuously

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Fig. 1 Mechanism of spouted bed and its drying principle

fed to a bed in which coarse particles were fluidized by dried hot air. The effects of operational conditions on the relative humidity of the outlet gas and on the drying efficiency were examined.

1. Experimental Apparatus and Procedure

A schematic diagram of the experimental apparatus is shown in Fig. 2. The experimental system consisted of six major sections: (1) gas heating system, (2) flow control and measurement system, (3) powder-particle spouted bed, (4) slurry injection system, (5) microparticles collection system, and (6) temperature and humidity control and measurement system.

The spouted bed consisted of a cylindrical column of polycarbonate 10.4 cm in I.D. and 100cm high. Air from a compressor was passed through an oil filter and dehydration towers filled with silica gel. The dried air was heated to 403 K by an electric heater and was fed to the bottom of the column. The flow rate of the air was measured with orifice meters. The column and the pipes were covered with glass wool to minimize heat loss. Silica sand particles of average diameter 460 μm [32⁺- 35⁻ mesh] or 650 μm [24⁺- 28⁻ mesh] and alumina particles of average diameter 1000 μm [14⁺-20⁻ mesh] were used as coarse particles and were fluidized by dried hot air.

Alumina particles and converter dust of 0.6 μm , 1.8 μm , 2.5 μm , 5.0 μm , 10.0 μm and 25.0 μm in average diameter were used as microparticles. These microparticles were not porous and were mixed with water in a stirred tank. The microparticle slurry or salt-water solution was fed to the spouted bed of coarse particles by a micro-tube pump, as shown in Fig. 2. The wet microparticles and salt-

Fig. 2 Schematic diagram of experimental apparatus

water solution were dried by contacting with the dried hot air, and only the dried microparticles were elutriated from the column, based on the difference in terminal velocity between the microparticles and coarse particles as shown in Table 1 and Table 2. The microparticles were collected by two bagfilters, which could be changed to collect at regular intervals of time.

Temperatures of the inlet gas of bed T_{d1} , the outlet gas of bed T_{d2} , and the fluidized bed T_b were measured at 5cm below, 60cm above and 10cm above the cone-bottom with chromel-alumel thermocouples, respectively as shown in Fig. 2. The humidity of the outlet gas was also measured. The relative humidity at the outlet was defined as follows.

$$H_2^* = H_2 / H_{w2} \quad (1)$$

where H_2 is the absolute humidity of the outlet gas and H_{w2} is that of the saturated gas. At the gas exit of the bagfilter, both wet- and dry-bulb temperatures were also measured. Microparticles collected in the bagfilters were dried at 403 K for 12 hours in an electric dryer. The moisture content of the microparticles in the bagfilter, ω_b , was obtained from the change in weight by this procedure.

The properties of coarse particles and the characteristics of the bed are shown in Table 1 and the properties of microparticles is shown in Table 2, and the operational conditions used in this study is shown in Table 3.

Here we define drying efficiency η_d as follows.

$$\eta_d = \frac{\bar{G}_{i12} \bar{C}_{H12} (T_{d1} - T_{d2})}{\bar{G}_{i1w} \bar{C}_{H1w} (T_{d1} - T_{w1})} \quad (2)$$

where $\bar{G}_{ij}$ and $\bar{C}_{Hij}$ are the average mass flow rate of gas and average humid heat of humid gas, respectively between i and j , and T_{w1} is the wet-bulb temperature at the inlet. The drying efficiency is the ratio of the heat of hot air used for drying to the theoretical (maximum) available heat of hot

Table 1 Properties of coarse particles and characteristics of bed

Material	Average diameter d_p [μm]	Density ρ_p [kg/m^3]	Min. fluidization velocity U_{mf} [m/s]	Terminal velocity U_t [m/s]	Mass M_c [g]	Static bed height L_c [mm]	Min. spouting* velocity U_{ms} [m/s]	Max. spoutable** bed depth H_m [mm]
Silica sand	460	2700	0.184	3.83	450	86.4	0.35	315
Silica sand	650	2700	0.368	5.41	450	86.4	0.43	284
Alumina	1000	3970	1.28	9.861	350	64.9	0.58	321
Silica sand	460	2700	0.184	3.83	1100	138.0	0.40	315

* O. Uemaki, *et al.*, 1983

** Ayse Cecen, 1994

Table 2 Properties of microparticles

Material	Average diameter d_p [μm]	Density ρ_p [kg/m^3]	Min. fluidization velocity U_{mf} [m/s]	Terminal velocity U_t [m/s]
Activated alumina	0.6	3970	4.67×10^{-7}	4.28×10^{-5}
Activated alumina 154	1.8	3970	3.40×10^{-6}	3.10×10^{-4}
Converter dust	2.5	2000	3.79×10^{-6}	3.55×10^{-4}
Converter dust	5.0	2000	1.52×10^{-5}	1.42×10^{-3}
Converter dust	10	2000	6.06×10^{-5}	5.68×10^{-3}
Converter dust	25	2000	3.79×10^{-4}	3.55×10^{-2}
Salt	Water soluble	Commercially available	$< 1.5 \times 10^{-5}$	$< 1.42 \times 10^{-3}$

air for drying.

The following equations are obtained from the mass balance of water and the heat balance across the spouted bed.

$$W_f(\omega_1 - \omega_2) = G(H_2 - H_1) \quad (3)$$

$$W_f(C_{pf} + C_{pw} \cdot \omega_1)(T_{d2} - T_f) + W_f(\omega_1 - \omega_2) \cdot Y_w = \bar{G}_{12} \cdot \bar{C}_{H12}(T_{d1} - T_{d2}) \quad (4)$$

where W_f is the feed rate of microparticles, and ω is the moisture content of microparticles. Subscripts 1 and 2 show inlet and outlet, respectively. From Eqs. (3) and (4), the absolute humidity at the outlet, H_2 , and the temperature of the outlet gas, T_{d2} , respectively, can be calculated without knowing the rates of heat and mass transfer between the fluid and the particles. Therefore, the relative humidity defined by Eq. (1) can be calculated from Eq. (3), and the drying efficiency of Eq. (2) can be calculated from Eq. (4).

The effects of feed condition of the inlet gas, feed condition of slurry and other operational conditions on the moisture content of outlet microparticles and relative humidity of outlet gas were examined.

Table 3 Operational conditions used in this study

U [m/s]	0.57-0.83
L_c [cm]	64.9, 86.4, 138.0
d_{pc} [μm]	460, 650, 1000
d_{pf} [μm]	0.6, 1.8, 2.5, 5.0, 10.0, 25.0
T_{d1} [K]	403
W_f [mg/s]	17.0-91.5

Fig. 3 Relationship between time elapsed and gas temperature

2. Results and Discussions

2.1 The characteristics of collected microparticles

Fig. 3 shows the relationship between time elapsed after start-up and the temperature of bed T_b and the outlet gas of bed T_{d2} , at gas velocities of 0.66 m/s and 0.74 m/s, respectively. T_b and T_{d2} gradually decreased with time, and became almost constant after 10 - 12 minutes. T_{d2} was almost equal to the bed temperature T_b . On the other hand, the gas temperature measured at the bagfilter was almost 10 K-12 K below that of the bed, T_b , suggesting that the heat loss of the total apparatus was not negligible. The higher the superficial gas velocity was, the faster the steady state was reached. After the bed temperature and the elutriation rate of microparticles became constant, the steady state was considered to be established, and the drying efficiency η_d and the relative residual ratio of microparticles

Fig. 4 Effect of superficial gas velocity on drying efficiency of activated alumina

Fig. 5 Effect of feeding rate of microparticles on drying efficiency of alumina

in the bed could be obtained.

The moisture content and equilibrium moisture content of outlet microparticles were measured by a differential thermo-gravimetric analyzer. The moisture content of the microparticles dried by this process from the submicroparticle (under $1\mu\text{m}$ in diameter) slurry and the salt-water solution were nearly equal to their equilibrium moisture contents over the range of the experiment. Therefore, perfect drying of these microparticles in the slurry could be conducted in the powder-particle spouted bed, because the equilibrium moisture content of these two kinds of microparticles were almost zero over the range of experimental conditions. The moisture content of microparticles at the outlet is thus negligible in Eq.(3) and Eq.(4), $\omega_2 = 0$.

Through observation of SEM photographs of the microparticles before and after the drying procedure, more scattered distribution of microparticles was observed after drying, and the shape of most microparticles was the same as that before drying. This demonstrated that powder-particle spouted bed fluidization prevented the

Fig. 6 Effect of superficial gas velocity on drying efficiency of salt solution

Fig. 7 Effect of feeding rate of microparticles on drying efficiency of salt solution

microparticles from agglomerating.

2.2 Drying characteristics

a) **Drying efficiency** Figure 4 shows the effect of superficial gas velocity on drying efficiency as defined by Eq.(2). Drying efficiency decreased with increasing gas velocity, and with decreasing feeding rate of microparticles. The solid lines show the drying efficiency as calculated by Eqs. (2) and (4). Very good agreement between experimental and calculated values was obtained. The drying efficiency was also evaluated without knowing the rates of heat and mass transfer. From Fig. 4, we know that there are two ways to get higher drying efficiency, one is increasing the feeding rate of the microparticles, the other is reducing the gas velocity. However, if the feeding rate of microparticles were too large, agglomerates between microparticles were formed in the bed, so continuous operation would not be achieved, and if the gas velocity were too slow, for example, less than U_{ms} , it would be difficult to cause particles to fluidize in the bed. Therefore, continuous drying would not be achieved. In this study, when drying efficiency was above 60% under certain operational conditions, we found that residual microparticles increased in the bed and continuous drying was not achieved.

Fig. 8 Relationship between feeding rate of moisture and drying efficiency

Figure 5 shows that drying efficiency increased with increasing feeding rate of microparticles, and that it was not affected by the diameter of coarse particles or static bed height. However, it was supposed that there was a minimum static bed height to maintain continuous operation.

Drying of salt-water solution was carried out, and Fig. 6 and Fig. 7 show the experimental results. Fig. 6 shows that drying efficiency decreased with increasing gas velocity, and Fig. 7 shows that drying efficiency increased with increasing feeding rate of microparticles. The solid lines show the drying efficiency as calculated by Eqs. (2) and (4). The moisture content of the outlet salt particles was less than 0.003[kg-water/kg-solid], as measured by differential thermo-gravimetric analyzer. Therefore, perfect drying of salt-water solution was achieved.

Figure 8 shows the relationship between the feeding rate of moisture, $W_f \omega_1$, and the drying efficiency. The solid line shows the drying efficiency as calculated by Eqs. (2) and (4) for alumina 0.6 μm in average diameter, and $\omega_1 = 1.5$. The drying efficiency increased proportionally with the feeding rate of moisture, and the drying efficiency was hardly affected by the type of slurry, the diameter of particles, and the moisture content of microparticles, ω_1 .

b) Relative humidity Figure 9 shows the relationship between superficial gas velocity and relative humidity of gas at the exit of the spouted bed. The relative humidity decreased with increasing gas velocity. The solid line shows the relative humidity calculated by Eqs. (1) and (3), and it is in agreement with experimental values.

Fig. 10 shows the relationship between the feeding rate of moisture, $W_f \omega_1$, and the relative humidity of gas at the exit of the spouted bed. The relative humidity increased with increasing feeding rate of moisture, and the experimental results agreed with the values calculated from Eqs. (1) and (3). The relative humidity was also evaluated without knowing the rates of heat and mass transfer.

c) Collection of microparticles As shown in Fig. 3, it took some time for the collection rate of microparticles to reach

Fig. 9 Relationship between superficial gas velocity and relative humidity

Fig. 10 Relationship between feeding rate of water in slurry and relative humidity

a steady state. This could be considered to be because of the fact that only microparticles in the bed reaching their saturated concentration can be elutriated at constant flow.

In this work, at the steady state, the saturated attachment capacity of microparticles on the surface of coarse particles was expressed by the mass ratio of microparticles to coarse particles in the bed. C^* defined as Eq. (5), was called relative residual ratio of microparticles in the bed.

$$C^* = M_f / M_c \quad (5)$$

where M_f is the mass of microparticles in the bed and M_c is the mass of coarse particles in the bed.

Figure 11 shows the relationship between C^* and the gas superficial velocity. C^* decreased with increasing gas velocity, and under the same operating conditions, C^* is smaller in the spouted bed than that in the powder-particle fluidized bed with gas distributor.

Fig. 12 shows the relationship between C^* and the feeding rate of microparticles. C^* increases with increas-

Fig. 11 Effect of superficial gas velocity on relative residual microparticles in bed

Fig. 12 Effect of feeding rate of microparticles on relative residual microparticles in bed

ing feeding rate of microparticles. Therefore, the saturated attachment capacity of microparticles on the surface of coarse particles in the spouted bed was a dynamic equilibrium which was related to operating conditions, and it was also noticed that C^* was hardly affected by the diameters of coarse particles and microparticles. This can be explained as follows.

Assuming that only when the surface of a coarse particle was closely adhered with a layer of the microparticles under dry conditions, the buoyancy of microparticles could balance with the electrostatic force and the Van der Waals force of coarse particles. The dynamic equilibrium between microparticles adhering to the surface of coarse particle and departing could be established. We could call this coarse particle at saturated state and the relative residual ratio of microparticles in the bed, C^*_{min} . This state was defined as follow.

$$C^*_{min} = 1.155 \phi \left(1 + \frac{d_{pc}}{d_{pf}} \right)^2 \cdot \left(\frac{d_{pf}}{d_{pc}} \right)^3 \frac{\rho_f}{\rho_c} \quad (6)$$

Certainly, when $C^* < C^*_{min}$, the relative residual ratio of

Fig. 13 Correlation between the relative microparticles residual ratio calculated and observed

microparticles in the bed was affected by the diameters of particles. However, when $C^* > C^*_{min}$, this effect would be reduced. In ideal cases, C^* would not be larger than C^*_{min} , but in real cases, e.g. in our present experiment, there were cases when $C^* > C^*_{min}$. This could be due to liquid bridge action between the wet coarse particles and wet microparticles, so that the adhesion force from the liquid bridge action was larger than the electrostatic and Van der Waals forces. Although the liquid bridge action was related to the diameters of particles, as soon as the particles were dried, the liquid bridge adhesion force would lose its dominant position. Therefore, when $C^* > C^*_{min}$, the residual microparticles in the bed were chiefly related to the drying characteristics, but not the diameters of particles. An empirical equation for estimating C^* was obtained as follows.

$$C^* = 0.00464 W_f^{1.735} U^{-1.643} \quad (7)$$

Figure 13 shows the difference between experimental values and calculated ones based on equation (7).

According to Eq. (7) the coarse particles can be estimated as a reference to design the spouted bed drier, and the average residence time of microparticles in the bed can be determined by Eq. (8)

$$\bar{\mu} = \frac{M_c \cdot C^*}{W_f} \quad (8)$$

In this experiment, the experimental results showed that the moisture content of microparticles (slurry) hardly affected C^* . One reason is that the heat capacity coefficient in the bed is greater than $5.815 \text{ kW/m}^3 \text{ } ^\circ\text{C}$ (Nakagawa and Kato, 1992). Another reason is that the slurry could uniformly adhere to the surface of coarse particles due to violent fluidization in the bed, so that the slurry could be quickly dried to the critical moisture content of the microparticles. Then the effect of moisture content of microparticles on C^* could be neglected.

Conclusions

Drying of a microparticle slurry and a salt-water solution were carried out in a powder-particle spouted bed, and the drying characteristics were examined. The following conclusions were obtained.

- (1) Continuous drying of the microparticle slurry and the salt-water solution could be achieved by the powder-particle spouted bed.
- (2) The powder-particle spouted bed without a gas distributor is more efficient to dry much smaller microparticles compared with the powder-particle fluidized bed with a gas distributor.
- (3) The moisture content of microparticles dried from the submicroparticle (under 1 μm in diameter) slurry and the salt-water solution was nearly equal to their equilibrium moisture content, and the drying efficiency was about 60% over the range of the experiment.
- (4) The powder-particle spouted bed can be designed according to the calculation of mass and heat balances across the bed.
- (5) The conception of saturated residual microparticles in the bed was presented.
- (6) An empirical equation for estimating the relative residual ratio of microparticles in the bed was obtained.
- (7) Under steady state conditions, the relative residual ratio of microparticles in the bed depended on operating conditions, and is an important parameter for a designing powder-particle spouted bed dryer.
- (8) It is necessary to establish a drying model to demonstrate the drying mechanism of the spouted bed as shown in Fig. 1.

Nomenclature

C_H	= humid heat	[kJ/kg-dry gas K]
C_{pf}	= specific heat of microparticles	[kJ/kg K]
C_{pw}	= specific heat of water	[kJ/kg K]
C^*	= relative residual ratio of microparticles in bed	[-]
D	= bed diameter	[m]
d_{pc}	= average diameter of coarse particles	[m]
d_{pf}	= average diameter of microparticles	[m]
D_s	= spout diameter	[m]
G	= total mass flow rate of gas	[kg/s]
H	= humidity of gas	[kg-water/kg-dry gas]
H^*	= relative humidity	[-]

H_m	= max. spoutable bed depth	[m]
L_c	= static height of spouted bed	[m]
M_c	= mass of coarse particles in bed	[kg]
M_f	= mass of microparticles in bed	[kg]
T_b	= temperature of spouted bed	[K]
T_d	= dry-bulb temperature of gas	[K]
T_f	= temperature of inlet slurry	[K]
T_w	= wet-bulb temperature	[K]
U	= superficial gas velocity	[m/s]
U_{mf}	= minimum fluidization velocity	[m/s]
U_{ms}	= min. spouting velocity	[m/s]
U_t	= terminal velocity	[m/s]
W_f	= feed rate of microparticles	[mg/s]
ϕ	= shape factor of coarse particle	[-]
γ_w	= latent heat of water	[kJ/kg]
η_d	= drying efficiency defined by Eq. (2)	[-]
$\bar{\mu}$	= average residence time of microparticles in bed	[s]
ρ_c	= density of coarse particles	[kg/m ³]
ρ_f	= density of microparticles	[kg/m ³]
ω	= moisture content of microparticles	[kg-water/kg-dry solid]

<Subscript>

1	= inlet
2	= outlet
b	= bagfilter
w	= saturated

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