

Microgranulation of fine powders by a novel rotating fluidized bed granulator

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Abstract

A novel rotating fluidized bed system has been developed for fluidizing, granulating and coating cohesive fine powders to tailor their properties and functionalities. The system basically consists of a plenum chamber and a horizontal porous cylindrical air distributor, which rotates around its axis of symmetry inside the chamber. The pressure drop and minimum fluidization velocity of cohesive fine cornstarch powder (mass median diameter of 15 μm , Geldart Group C powder) were measured under various rotating conditions and the fluidization behavior was studied. The system was then used for wet granulation of the cornstarch powder and the effects of operating parameters on the granule properties such as granule size, size distribution, density and flowability were investigated. The experimental results indicated that the flowability of cohesive fine cornstarch was dramatically improved by the microgranulation resulting in spherical granules with a narrow size distribution while inhibiting the size enlargement.

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1. Introduction

Fine powders have become of major interest lately. Many industrial sectors such as pharmaceutical, agriculture, foods, chemicals, ceramics and electronics are expected to find applications and take advantage of the new functionalities and many desirable properties attributed to ultrafine powders.

Fluidization is one of the major techniques for fine powder handling and its applications has been extended to a wide variety of processes such as cracking of hydrocarbons, combustion of solid fuels/wastes and roasting of ores by chemical processing and filtration, drying, wet granulation and coating by physical processing. This is because fluidization exhibits excellent advantages of high heat and mass-transfer rates, temperature homogeneity and high flowability of particulate materials. However, as pointed out by Geldart [1] in his classification map, powders in Group C (fine size and low density) fluidize poorly,

exhibiting channeling, lifting as a plug and forming rat holes when aerated. Therefore, the development of reliable techniques to improve the fluidization of cohesive fine powders is required.

So far, several mechanisms such as vibration [2], mechanical agitation [3], sound [4] and magnetic force [5] have been used to improve the flowability of cohesive powders. The application of these methods will help in the handling of fine powders and fluidization can often be obtained. However, unit operations before or after fluidized bed processing such as conveying, weighing, packing and tableting still present problems originating from the cohesive properties of fine powders, which remain the same regardless of any external forces applied. Therefore, an improvement in the flowability of fine powders is needed before they are further processed since their poor flowability is due to their small size.

In this study, a novel rotating fluidized bed has been developed for improving the flowability of cohesive fine powders. Microgranulation, where the flowability of cohesive fine powders is improved, while inhibiting size enlargement as much as possible, has been conducted. The physical

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properties of the granules were evaluated experimentally and the performance of the system was investigated.

2. Experimental

2.1. Equipment

A schematic diagram of the experimental apparatus is shown in Fig. 1. The rotating fluidized bed composes of a plenum chamber and a porous cylindrical air distributor (ID 400 × D 100 mm) made of stainless sintered mesh with 20 μm openings. The horizontal cylinder (air distributor) rotates around its axis of symmetry inside the plenum chamber. There is a stationary cylindrical metal filter (ID 140 × D 100 mm, 10 μm openings) inside the air distributor to retain elutriated fine powder. A binary spray nozzle mounted on the metal filter sprays binder mist (mist size is around 7–10 μm) into the powder bed. A pulse air-jet nozzle is also placed inside the metal filter, which cleans up the surface of the metal filter in order to prevent clogging. An air knocker is installed outside the plenum chamber to prevent powder adhesion onto the air distributor mesh and front cover. Pressure taps are mounted on the inlet and exhaust air pipes and on the metal filter, so that the manometer (1) measures the pressure drop across the powder bed and air distributor, while manometer (2) measures pressure drop across the metal filter. In this study, the pressure drop across the powder bed, ΔP , is defined by Eq. (1)

$$\Delta P = P_1 - P_2 \quad (1)$$

where P_1 is the pressure drop across the powder bed and air distributor measured during the experiments and P_2 is

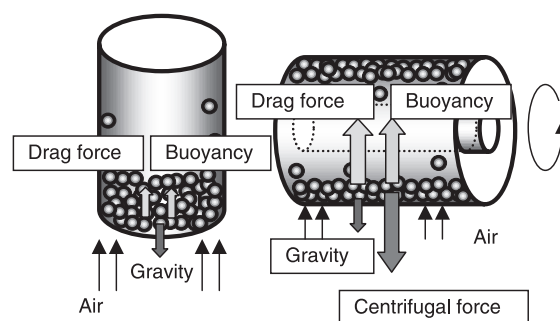


Fig. 2. Powder fluidization mechanisms.

the pressure drop across the air distributor measured during idling without powder, respectively.

Fig. 2 illustrates the powder flow mechanism in the rotating fluidized bed. In a conventional fluidized bed, the air distributor is mounted horizontally and powder samples are introduced onto the distributor. Powders are lifted up by a vertical airflow (drag force and buoyancy against the gravity force). In a rotating fluidized bed, powder samples are introduced inside the air distributor and are forced to the wall by a centrifugal force due to the rotation of the distributor. Air flows radially inward through the air distributor and the forces on the powder are balanced by the airflow (drag force and buoyancy) and the centrifugal force. Unlike conventional fluidized beds, a rotating fluidized bed can impart a high centrifugal force, which enables fine particles to behave as Geldart Group A powder [6]. The centrifugal force can be made much larger when compared to other mechanical forces given by vibration [2], mechanical agitation [3], sound [4] and magnetic force [5]. This implies that the rotating fluidized bed can uniformly fluidize much finer powders than conventional fluidized beds even when equipped with these mechanical devices. Theoretically, if the distributor rotates at a high enough speed and the airflow increases correspondingly to keep a uniform fluidization, the adhesion force between powders can be neglected as compared to the centrifugal and drag forces.

It is noteworthy that the energy consumption of a rotating fluidized bed is not very large because the rotating fluidized bed can operate near the minimum fluidization velocity (usually less than 3 times the minimum fluidization velocity), whereas a conventional fluidized bed always operates at a velocity more than 5–10 times the minimum fluidization velocity, especially in wet granulation and coating, to avoid blocking. Also, the contact efficiency between the particles and air is very good, so that the operating time can be shortened. In addition, due to an air sealing between the inner metal filter and the rotating air distributor, mechanical abrasion is negligible.

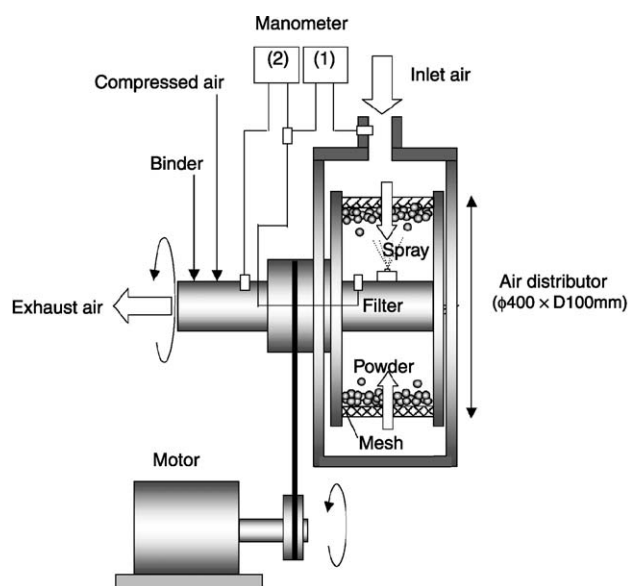


Fig. 1. Schematic diagram of a novel rotating fluidized bed.

Table 1

Operating conditions	
Rotational speed	5.57 rps (25G) ^a
Airflow rate	0.596 m/s ($u/u_{mf}=3.0$)
Air temperature	333 K
Spray air pressure	0.3 MPa
Binder feed rate	10 g/min

^a 1G = 9.8 m/s² (gravity acceleration).

2.2. Powder sample

Cornstarch with a mass median diameter of 15 μm was used in this work. This powder belongs to Geldart Group C (cohesive powder). A mass of 0.66-kg cornstarch powder was charged into the equipment, which gave a bed height of approximately 0.02 m. For wet granulation, an aqueous solution of 10% hydroxypropylcellulose (HPC-L) was used as a binder.

2.3. Experimental procedure and operating conditions

Table 1 lists the operating conditions. Granulation experiments were conducted as follows:

- (1) The powder sample (cornstarch) was fed into the cylindrical air distributor (vessel).
- (2) The air distributor was rotated and fluidization air was supplied.
- (3) Binder liquid was sprayed onto the powder bed.
- (4) After a predetermined amount of binder was sprayed, drying of granules was conducted until the moisture content decreased to less than 1%.

2.4. Evaluation of granules

The size and size distribution of the granules were measured by a laser diffraction particle size analyzer (SALD-2100, Shimadzu). The degree of compression was measured based on the following equation

$$C = \frac{P - A}{P} \times 100 \quad (\%) \quad (2)$$

where P and A indicate the tapped and bulk densities, respectively. Both densities were measured by using a Powder Tester (Hosokawa Micron).

The flowability of the granules was measured by using a discharge tester. This involved feeding 0.02 kg of granules into a glass funnel (largest ID of the top part is 50 mm, ID of the sleeve pipe is 5 mm) and subjecting the powder to mechanical vibration. The time required to completely discharge the powder was measured and used as the indicator of flowability. In the literature, the angle of repose measurement has been widely used; however, this method was impossible to apply to cornstarch powder. The main reasons were that the cornstarch powder

adhered to the surface of the funnel sleeve pipe and also formed bridges inside the funnel, so that powders could not be discharged smoothly [7] and instead were discharged in a pulsatory manner. This caused poor reproducibility of the measured angle of repose. Therefore, we decided to use the discharge test instead of using the angle of repose method to determine flowability.

3. Results and discussions

3.1. Fluidization behavior of cornstarch

Fig. 3 shows the pressure drop plotted against airflow velocity under different rotating conditions. The pressure drop increased to a peak at minimum fluidization velocity (u_{mf}) and then showed a constant pressure drop regardless of a further increase in air velocity. Due to the radial acceleration, the pressure drop increased with an increase in the centrifugal force. At all rotational speeds, the pressure drop did not show a linear increase before reaching the minimum fluidization velocity. This was because the initial cornstarch powder formed some agglomerates and was also compressed due to the high centrifugal force while idling. Thus, a higher-pressure drop through the powder bed was recorded until each powder uniformly fluidized at the minimum fluidization velocity.

From Fig. 3, it can be seen that fine cohesive cornstarch powder (Geldart Group C) can be uniformly fluidized without any channeling. This figure also confirms the possibility of handling fine powder in a rotating fluidized bed.

Fig. 4 shows the relationship between the minimum fluidization velocity and the rotational speed. The minimum fluidization velocity increased approximately linearly with rotational speed. This agrees well with the equation

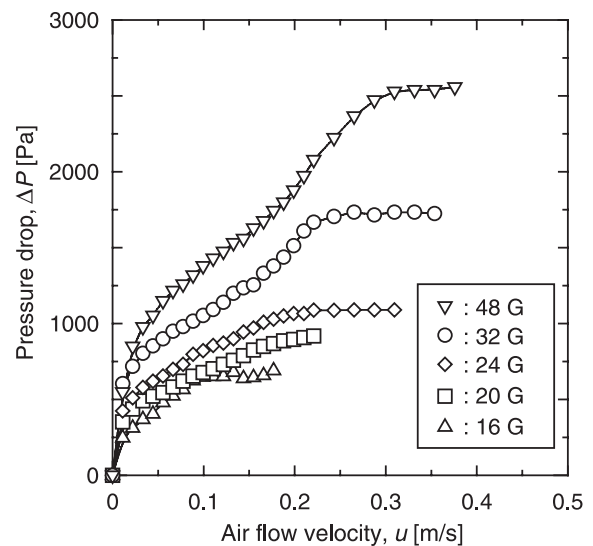


Fig. 3. Pressure drop against airflow velocity.

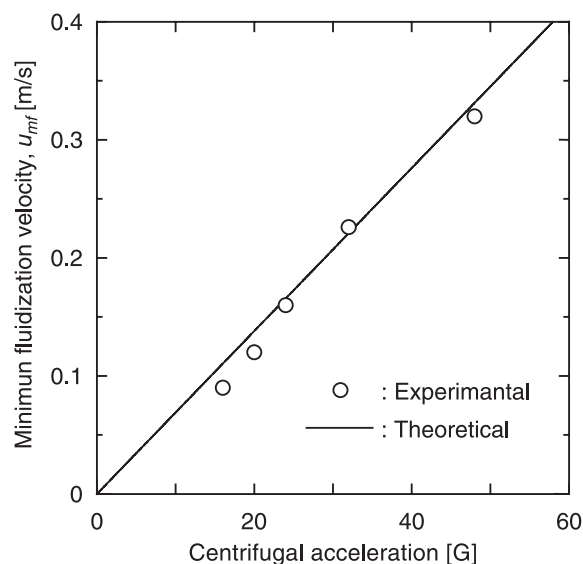


Fig. 4. Relationship between minimum fluidization velocity and centrifugal acceleration.

based on a theoretical model proposed by Kao et al. [8] given by

$$\frac{u_{mf}\rho_f d_g}{\mu} = \left[\left(33.7 \frac{C_2}{C_1} \right)^2 + 0.0408 \frac{\rho_f(\rho_g - \rho_f) d_g^3 \omega^2}{\mu^2} \frac{C_3}{C_1} \right]^{1/2} - 33.7 \frac{C_2}{C_1} \quad (3)$$

where

$$C_1 = r_0^2(1/r_i - 1/r_0), \quad C_2 = r_0 \ln(r_0/r_i), \\ C_3 = (r_0^2 - r_i^2)/2$$

It is believed that particles contacting the cylinder wall experience a direct centrifugal force, whereas other particles away from the wall also transmit the centrifugal force due to particle collisions and slip. Details will be reported in a subsequent paper, which analyzes the particle movement and force transmission by using a discrete element method (DEM).

The results in Fig. 4 imply that the behavior of cohesive cornstarch powder in the rotating fluidized bed is almost the same as the behavior of Geldart Group A powders [6]. Also, this result suggests that the adhesion force between fine powders is negligible under a high centrifugal force field as shown in Fig. 2.

3.2. Microgranulation

Fig. 5 shows evolution of the mass median diameter and geometric standard deviation of granules. The mass median diameter increased gradually with time, implying that the adhesion between individual cornstarch particles was occurring due to liquid bridges generated by the binder mist. By

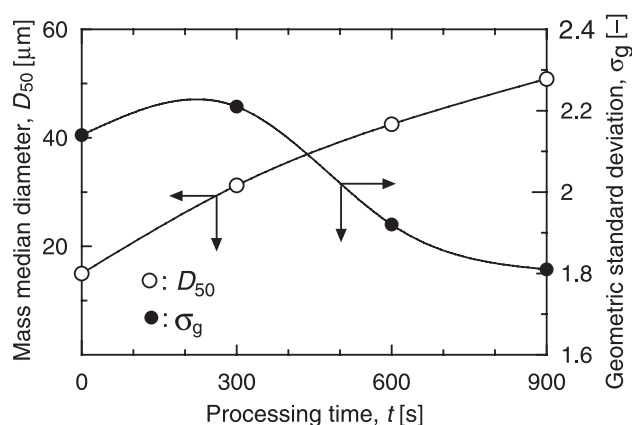


Fig. 5. Temporal change in mass median diameter and geometric standard deviation.

contrast, the geometric standard deviation increased initially, followed by a decrease (size distribution became narrow) with operational time. At the initial stage of granulation, granulated and original cornstarch particles co-existed, leading to a wide size distribution. As time elapsed, the powder was gradually and uniformly granulated to reduce the size distribution. In practice, coarse granules were never produced and ungranulated fine powders were never seen after 600 s of operation. These results occurred due to the movement of individual cornstarch powder particles in the high centrifugal force field.

Fig. 6 shows the temporal change in the compressive degree of the granules. The compressive degree decreased with time, indicating an increase in the tapped density of the granules. Due to granulation, an increase in granule density and improvement of flowability were achieved (as shown in the next figure).

Fig. 7 shows the change in flowability by measuring the time required for powder discharge. The required time was decreased rapidly with operational time, showing that the flowability was greatly improved. When the operation time was 600 s, the discharge time was only 7 s. It is noteworthy that this is almost the same time required for 100 μm

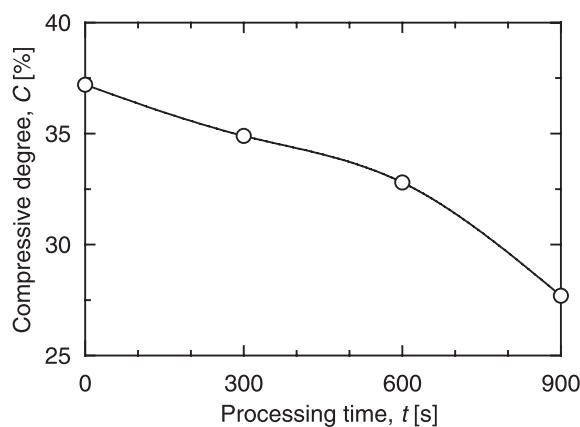


Fig. 6. Temporal change in compressive degree.

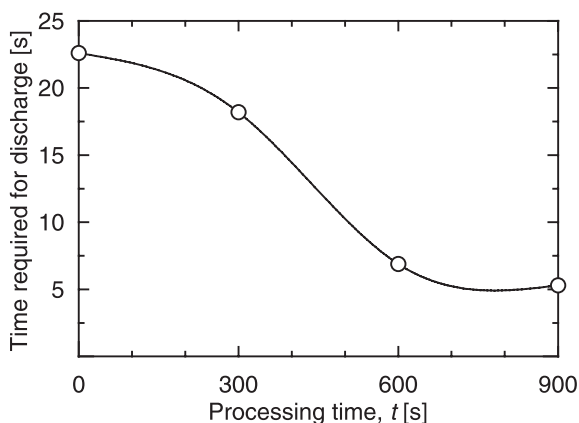


Fig. 7. Temporal change in time required for powder discharge (flowability).

diameter spherical glass beads to discharge through the same funnel. From this result, it is easy to see that the flowability of cohesive cornstarch was greatly improved by the microgranulation process.

Fig. 8 shows scanning electron microscopy (SEM) pictures of granules. Since cornstarch does not dissolve into water, each cornstarch particle maintained its original shape even when forming granules. In addition, each cornstarch particle regularly adhered to another particle, forming spherical granules.

Fig. 9 confirms the effects of centrifugal acceleration on granule bulk (apparent) density after 600 s of processing. With an increase in centrifugal acceleration, the granule bulk density increased gradually. Due to fluidization, granules experience a high impaction force, resulting into the formation of well-compacted granules.

The granule size and density are determined by a balance of the adhesion force and separation force experienced by the granules. When the centrifugal force is increased, the separation force becomes large, leading to a decrease in the granule size and an increase in the granule density. Also, a high centrifugal force results in a high fluidization air volume to maintain uniform fluidization. This leads to an increase in drying efficiency and granule growth rate.

These results indicate that we have succeeded in developing a method for microgranulation of cohesive fine

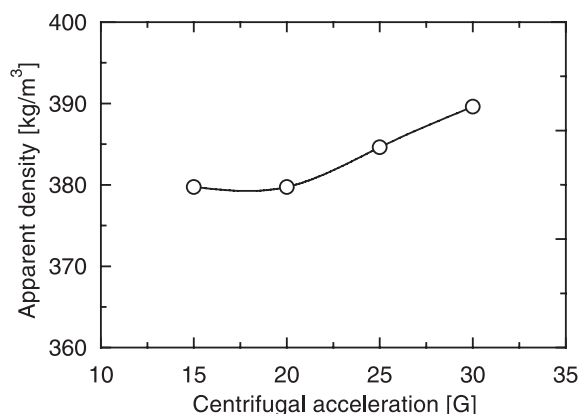


Fig. 9. Effects of centrifugal acceleration on the granule bulk density.

powders, with a dramatic improvement in flowability, an increase in density and a sharpening of the particle size distribution. In the manufacturing processes of particulate materials, problems due to the cohesiveness of fine powder are well known. Our system can overcome these problems by granulating the cohesive fine powders just enough to meet the requirement for smooth transportation, accurate weighing, better handling and the further work, we will investigate the powder flow and the granule growth mechanism in the rotating fluidized bed.

4. Conclusions

A novel rotating fluidized bed has been developed and applied to fluidization of cohesive fine powder (cornstarch powder, mass median diameter of 15 μm), which is normally categorized as a Geldart Group C powder. However, uniform fluidization was obtained in the rotating fluidized bed and the minimum fluidization velocity was well-predicted by using a model for a Geldart Group A powder. By spraying a binder into the fluidized powder, microgranulation was achieved. The flowability of cohesive fine cornstarch powder was dramatically improved and a narrower size distribution of particles was observed. This method can be extended to improve the handling and processing of other fine cohesive powder, which are used in transportation,

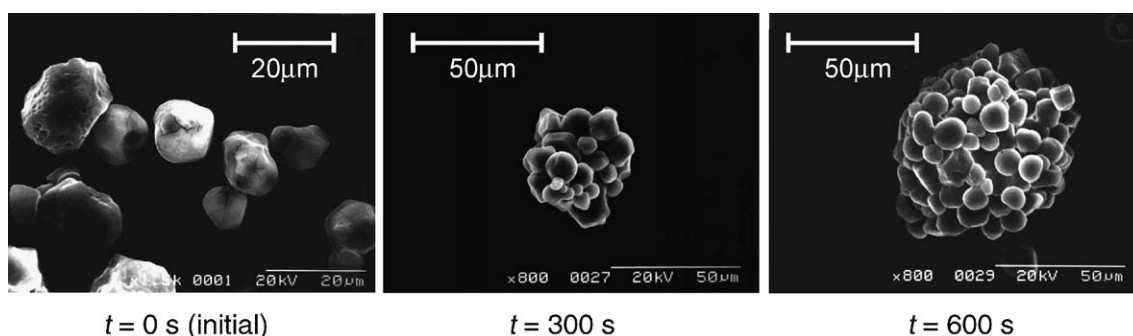


Fig. 8. SEM photographs of cornstarch granules.

dispersion and synthesis of composite materials in many different industries.

List of symbols

A	Bulk density in Eq. (2), kg/m^3
d_g	Granule diameter, m
C	Compressive degree defined by Eq. (2)
D_{50}	Mass median diameter, μm
N	Rotational speed, s^{-1}
P	Tap density in Eq. (2), kg/m^3
P_1	Pressure drop across powder bed and air distributor, Pa
P_2	Pressure drop across air distributor, Pa
r_i	Radius of inner surface of granule bed, m
r_0	Radius of rotating fluidized bed, m
t	Processing time, s
u	Air flow velocity, m/s
u_{mf}	Average minimum fluidization velocity, m/s
μ	Air viscosity, $\text{kg/m}\cdot\text{s}$
ΔP	Pressure drop, Pa
ρ_f	Air density, kg/m^3
ρ_g	Granule density, kg/m^3
σ_g	geometric standard deviation
ω	Angular velocity, rad/s

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