Determination of End-Point with a Complex Granulation Applying Infrared Moisture Sensor

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A granulation experiment was conducted in a complex granulator, which was fluidized bed equipped with an agitator blade. An infrared moisture sensor was used to feed back control the moisture content of the granule particles.

Granule properties (mean particle diameter, geometric standard deviation, yield, apparent density and appearance shape) were examined to know the mechanism and the process of moisture feed back control of the complex granulation.

To clear up the factors which determine the mean particle diameter of the granules, the influence of the liquid (binder solution) flow rate, inlet temperature and moisture content were examined to get an experimental equation which expressed the mean particle diameter of the granules. The influence of the moisture content and the rotation speed of the agitator blade on the apparent density was also investigated to get an experimental equation. With the basis of both equations, we established a system which can control the granule mean particle diameter and apparent density using a personal computer, and in addition, a self control system from granulation to drying using an infrared moisture sensor.

Keywords complex granulation; infrared moisture sensor; moisture feed back control; experimental equation; granulation end point

Introduction

Recently, factory automation has been developed in the pharmaceutical and other industries which are dealing with powdered materials.

In keeping abreast of the times, however, demands for automation have gradually changed from mass production to multivariate production with multiitems, thus, there is a great need for a multi system of individual unit operations.

In this paper, we focused our attention on granulation which is carried out with widespread use in many fields. The purpose of granulation had been to control basic components (size, size distribution and yield of granules), however, needs for granules have changed to produce high quality materials with various characteristics like appearance shape, density, hardness and so on.

Many authors have carried out a great deal of investigation to understand the granulation mechanism^{1–9)} and to detect the process applying different devices.^{10–18)} These researches, however, were insufficient from the view of factory automation. Therefore, we tried to set up a self system which can be carried out automatically from mixing to drying, and can produce the desired particles with various characteristics. To carry out several operations in one piece of equipment, we adopted a complex granulator which was a fluidized bed equipped with an agitator blade and an infrared moisture self control system. Owing to this blade, this granulator can produce spherical and compact granules which could not be produced by an ordinary fluidized bed, and the moisture content can be continuously measured and feed back controlled by applying the infrared moisture sensor.

To manipulate the granule properties at will, factors which influence the granule properties must be made clear, thus we first investigated the factors which determine the mean particle diameter of the granules by examining the influence of the liquid (binder solution) flow rate, inlet temperature and moisture content to get an experimental equation which expresses the mean particle diameter of the granules. The influence of the moisture content and the rotation speed of the agitator blade on the apparent density was also investigated to get an experimental equation.

Moisture feed back control was conducted by applying an infrared moisture sensor. During the experiment, granule properties (mean particle diameter, geometric standard deviation, yield, apparent density and appearance shape) were examined to know the mechanism and process of moisture feed back control (PID control) of the complex granulation.

As a result, we established a system which can control granule mean particle diameter and apparent density using a personal computer on the basis of the experimental equation, in addition to a self control system from granulation to drying using an infrared moisture sensor.

Experimental

Equipment The measurement theory of the infrared moisture auto-measuring system with optical fiber is the same as previously reported.^{16–18)}

The unit system of complex granulator and infrared moisture sensor is schematically illustrated in Fig. 1. A complex granulator (NQ-LABO, Fuji Paudal, Co., Ltd.) equipped with a binary spray nozzle was used for the granulation experiment. On the bottom of the beds, an agitator blade was equipped for tumbling and compacting the granule particles with fluidization by heated air. This agitator blade is possibly changeable to various types. Powder accompanied with fluidized air was entrapped by a bag filter, and exchanged by pulse jet air. The pulse jet interval selected was 20 s.

The output signals from the transducer of the moisture sensor are both electric current (4–20 mA) and voltage (0–10 mV), and the former is transported to a digital program controller (D/D converter) to be used as a roller pump control signal, the latter is digitalized in a 12 bit A/D converter to be calculated in a personal computer. During the experiment, the moisture content was continuously monitored in a personal computer, and feed back controlled (PID controlled) by a digital program controller (D/D converter).

The sampling interval of this system was 70 ms (14 times/s).

Materials Starting material and its mixing weight are listed in Table I. Starting materials were 0.3 kg of a mixture of lactose and corn starch (mixing weight ratio is 7:3, respectively). 0.015 kg of hydroxypropylcellulose was adopted as a binder, which was mixed as a form of dry powder into the starting materials before granulation. Purified water was used as a binder solution.

Method The experiment was conducted as follows. Powder samples penetrated through a 50-mesh (300 μ m) sieve were fed into a preheated container, then mixed with fluidized air for 600 s. After the operational conditions were instituted as described in Table II, the binder solution (purified water) was top sprayed by a binary nozzle as a form of mist. After granulation was over, the adding of binder solutions was stopped to shift to the drying process.

Fig. 3. Apparent Density ρ_a as a Function of Agitator Rotation Speed N

Moisture content: ●, 15%; □, 12.5%; △, 10.0%.

to 15%, binder solutions were partly used for liquid bridges, therefore, gradual growth of granules could be found. At a moisture content was above 15%, the rapid increase of D_{50} was found. It was because that the surface of the particles were saturated with solutions, thus the binding force became large. Therefore $W=15\%$ was the turning point of granule growth. The following experimental equations were obtained using the least squares method;

$$\begin{aligned} 0 \leq W \leq 15\%: & \quad D_{50} = 16.27W(\%) - 32.11 \\ 15 \leq W \leq 20\%: & \quad D_{50} = 42.92W(\%) - 433.79 \end{aligned} \quad (1)$$

Therefore, we can predict approximately the particle diameter by means of monitoring the moisture content of the granules.

Effect of Moisture Content and Agitator Rotation Speed on Apparent Density of Granules To examine the influence of the moisture content and agitator rotation speed on the apparent density of granules, experiments were carried out at varying moisture contents (setting values of constant moisture content were 10.0, 12.5, 15.0%) and rotation speeds.

Figure 3 shows the relation between the apparent density, ρ_a , and rotation speed, N . It was found from Fig. 3 that an increase of rotation speed resulted in an increase of apparent density, which was attributed to the tumbling and shearing effect of the agitator blade. No remarkable influence of moisture content could be found on the apparent density, thus, the apparent density depended on the effect of the agitator blade rather than on the moisture content. D_{50} of the granules here were approximately constant in spite of the variation of rotation speed. From these results, we can predict approximately the apparent density of the granules with a correlation between the apparent density and the agitator rotation speed. The experimental equation which expresses apparent density is as follows:

$$\begin{aligned} 0 \leq N \leq 25 \text{ (s}^{-1}\text{):} \\ \rho_a \text{ (kg/m}^3\text{)} = 0.016N^3 - 1.10N^2 + 25.45N + 418.58 \end{aligned} \quad (2)$$

Granulation Process under Moisture Self-Control Moisture self control of this complex granulator was carried out by means of feed back control (PID control) of the flow rate with the application of an infrared moisture sensor. The binder solution was programmed to spray to the granules at a moisture content less than 15%. After the

Fig. 4. Relation between Moisture Content W and Granulation Time t

Fig. 5. Relation between 50% Mean Particle Diameter D_{50} , Geometric Standard Deviation σ_g and Granulation Time t

○, 50% mean particle diameter, D_{50} ; □, geometric standard deviation, σ_g .

moisture content had reached 15%, PID control of the flow rate was carried out to keep the moisture content constant for 900 s. At $t=1800$ s, the binder solution was programmed to stop so as to shift to the drying process. The drying process was ended when $W=0\%$.

Figure 4 shows the relation between moisture content, W and granulation time, t while PID control is conducted. Overshoot of the moisture content was only 0.5% and the fluctuation was $\pm 0.3\%$ which is within the permitted limit, thus, it was found that the moisture content was favorably controlled and kept at the preset value.

To examine the change of the granule properties, mean particle diameter, and particle size distribution, the apparent density and shape index were examined. Here, the shape index was indicated the mean ratio of the short/long diameter of 50 granules. Figure 5 shows the relation between mean particle diameter, D_{50} , geometric standard deviation, σ_g and granulation time, t . Figure 6 also shows the relation between the yield of granules and granulation time. An increase of mean particle diameter could not be found from $t=900$ to 1800 s, which attributed to liquid bridges originating from moisture content being kept constant. After $t=900$ s, a slight increase in the yield of large granules (Y_g) and fine granules (Y_s), and a slight decrease of ungranulated fine particles (under 200 mesh ($75 \mu\text{m}$)) were found. Although the number of liquid bridges were constant, the abrasion of particles was prevented and fine ungranulated particles under $75 \mu\text{m}$ were made to adhere to the granules, which was resulted in an increase of Y_s , Y_g .

Fig. 6. Relation between Yield of Granule Particle and Granulation Time t

○, yield of fine particles, Y_s ($75 < d_p < 500 \mu\text{m}$); □, yield of granules, Y_g ($297 < d_p < 1410 \mu\text{m}$); △, yield of fine particles, Y_f ($d_p < 75 \mu\text{m}$).

Fig. 7. Relation between Apparent Density ρ_a , Shape Index ψ_e and Granulation Time t

○, apparent density, ρ_a ; ●, shape index, ψ_e .

Fig. 8. Flow Sheet Illustrated Determination of Granulation End-Point

and a decrease of Y_f .

Particle size distribution tended to become sharp as granulation time passed.

Figure 7 shows the relation between apparent density ρ_a ,

shape index ψ_e and granulation time t . Apparent density slightly increased with granulation time passed because the frequency number needed to get a tumbling effect and shearing stress became longer. The shape index ψ_e showed a tendency to increase (becoming spherical) as the granulation time passed. This was also attributed to the tumbling effect of the blade.

It was found that a desired granule diameter corresponding to its moisture content with sharp particle size distribution, a high yield of granules, high density and spherical granules could easily be obtained with the application of moisture control.

Determination of Granulation End-Point A flow sheet which indicating the determination end-point in the complex granulation is illustrated in Fig. 8. By inputting the mean particle diameter and apparent density of the desired granules to Eq. 1 and Eq. 2, a preset value of moisture content, W and agitator rotation speed, N are calculated by the personal computer. By programming the value and granulation time into the controller, control of the process of mixing–granulation–drying is conducted applying feed back control of the moisture content.

By this means, determination of the end-point which selects mean particle diameter and apparent density of the desired granules at one's will is made possible.

Conclusions

Complex granulation was conducted under moisture feed back control and we obtained the following results;

- 1) If granulation is conducted under a constant value of moisture content, it is found that spherical and compact granules with sharp particle size distribution and high yield can easily be produced.
- 2) Mean particle diameter was mainly determined by the moisture content, and the correlation between mean particle diameter and moisture content could be expressed by the linear equation.
- 3) Apparent density of the granules depended on the rotation speed of the agitator blade rather than on the moisture content, thus, we set up the experimental equation which expressed the apparent density with agitator rotation speed.
- 4) With the basis of both experimental equations, we established a system which can control the granule mean particle diameter and apparent density using a personal computer, and in addition, a self control system from granulation to drying using infrared moisture sensor.

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