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Analysis of Granulation Process and Determination of Operational End Point in a Tumbling Fluidized Bed Granulation

Satoru WATANO*, Keijiro TERASHITA* and Kei MIYANAMI*

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In this paper, measurement and control of process variables needed for an automated operation of a tumbling fluidized bed granulation was described. Power consumption required for a rotation of an agitator blade was used to analyze the granulation process and the progress of the particles. Properties of the granulated particles were examined, and the factors which affected the fluctuation of the power consumption were investigated experimentally. From these findings, a practical method for the determination of an optimum operational end point in the tumbling fluidized bed granulation was developed.

1. Introduction

Powder handling processes are given much weight among many manufacturing processes in chemical and pharmaceutical industries. A high reproducibility of the product quality, improvement of the operational safety and the labor-saving are actualized by the automated powder handling processes. This automation needs measurement and control of the individual unit operation, however, only a few works have been made to the powder handling processes.

Wet granulation, being widely used for many purposes, is one of the most important operation in the powder handing processes. In the past years, many improvements have been made on the granulator. A tumbling fluidized bed of complex type granulator was designed and developed in the reflection of the demand that a wide range of different products and several operations were available in one equipment, and a contamination was reduced to a minimum.

Itoh and Kamata[1)], Takei and Myo[2)] conducted fundamental studies of this granulation method. By way of an attempt to control the granulation process, we[3)] have already developed an IR (infrared) sensor to measure the moisture content without touching the powder bed even in the powder and dust, furthermore, we have controlled the damping, granulation and drying process by use of the system developed.

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* Department of Chemical Engineering, College of Engineering.

In this paper, measurement and control of the process variables which were necessary for the whole automated operating system of the granulation were demonstrated first. In the next stage, power consumption required for a rotation of a blade was picked out to investigate a quantitative relation to the granule properties. By applying this method to the wet granulation under moisture control in the tumbling fluidized bed, analysis of the granulation process and determination of the optimum operational end point which had been obscure in the ordinary method were conducted.

2. Experimental

2.1 Equipment

The experimental set-up used is schematically illustrated in Fig. 1. A tumbling fluidized bed of complex type granulator (NQ-LABO, Fuji Paudal, Co., Ltd.,) was used for a wet granulation. This granulator consisted of two parts; bottom cylindrical vessel (0.125 m in diameter, 0.10 m in depth), and upper cone vessel tapered 15 degrees (0.20 m in height), which were both made of acrylic resin. On the bottom of the cylindrical vessel, an agitator blade was equipped for the purpose of giving the tumbling and compacting motion to the granule particles, and under it, three circular plates of different diameter were superimposed at a

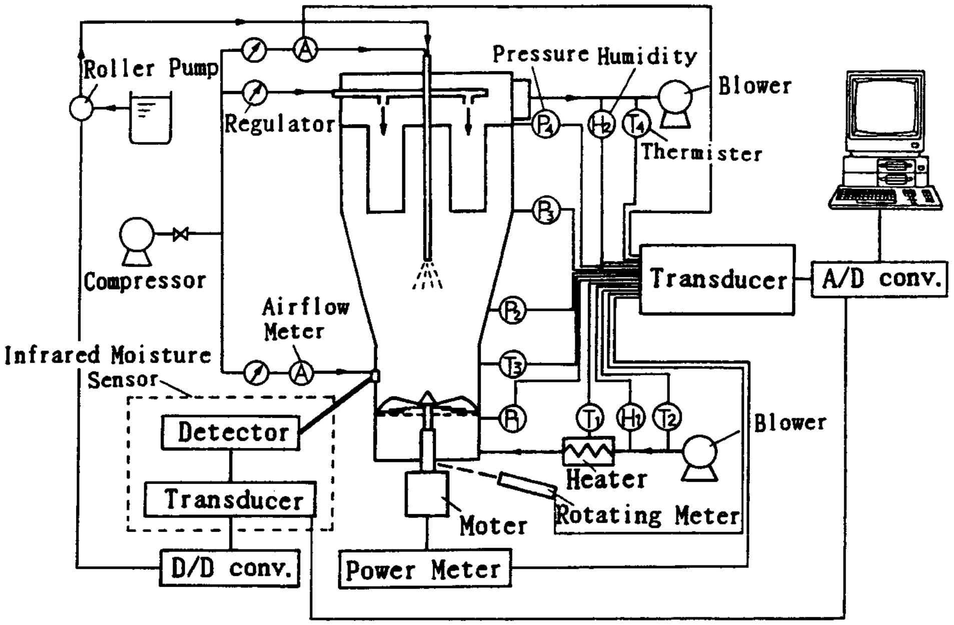

Fig. 1 Experimental set-up

distance of 3 mm. A heated air needed for the fluidization of the particles was blown from the slit between the three plates so as to make a circulating flow. Fine powders accompanied with the fluidization air were entrapped by bag filters, and brushed down by a pulse jet air.

The sensors equipped in this system are listed in Table 1. The principle of measurement of the infrared moisture sensor and the details of the control system were the same as previously reported[3]. Output signal from the moisture sensor was transported to a digital program controller (D/D converter) to control the rotation speed of a roller pump used for a supply of a binder liquid. This output signal from the sensor was also simultaneously digitalized in a 12 bit A/D converter to be monitored in a desk-top computer. In this system, inlet air temperature, air volume, inside pressure of the vessel and the agitator rotation speed were also feed back controlled to maintain a stable operation. Humidity, pressure drop, power consumption etc. were measured to detect the state of the granulation. These 16 data from sensors were all converted to signals by transduceres of a range of 0-10V, which were then amplified and A/D converted to be monitored and memorized in the computer.

Table 1 Sensors equipped in the system

	Measuring Object	Detecting Element	Final Controlling Element
Measurment and Control	Moisture content	Infrared Moisture Sensor	Roller Pump
	Rotation Speed	Tacho Meter	Motor (Inverter)
	Air Volume	Orifice	Blower(Inverter)
	Inside Pressure	Pressure Transducer	Blower(Inverter)
	Heater Temperature	Thermister (Pt100Ω)	Heater
Measurment	Power Consumption	Power Meter	
	Inlet Temperature	Thermister ($RuO_2$100Ω)	
	Outlet Temperature	Thermister ($RuO_2$100Ω)	
	Object Temperature	Thermister (Pt100Ω)	
	Inlet Humidity	Ceramic Sensor (Alumina System)	
	Outlet Humidity	Ceramic Sensor (Alumina System)	
	Pressure of Slit	Pressure Transducer	
	Pressure of Bagfiter	Pressure Transducer	
	Inside Pressure	Pressure Transducer	
	Air Volume of Spray	Thermal Flow Mass Meter	
	Amount of Binder Liquid Sprayed	Electronic Balance	

The power supply of the motor drive of the agitator blade used for the analysis of the granulation process was interfaced with the power consumption measurement device. The principle of an actual efficiency of the power consumption, P was defined by a general equation:

$$P=E\times I\times\cos\theta \tag{1}$$

Here, E was a circuit voltage, I was a current, and $\cos\theta$ was a constant factor according to the specification of the motor drive. The actual efficiency of power consumption of the motor drive was converted for a range of 0-1999 J/s by the power consumption measurement device according to a range of 0-10V.

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

2.2 Powder Samples

Starting materials and their mixing ratio are listed in Table 2. Starting materials were 0.300 kg, which consisted of lactose and corn starch (mixing ratio was 7:3 by weight, respectively). 0.015 kg of hydroxypropylcellulose was adopted as a binder, which was mixed as a form of dry powder into starting materials before granulation. Purified water was used as a binder solution.

Table 2 Powder samples

Materials	Mean Particle Size [μm]	Mixing Weight [kg]
Lactose[a]	104	0.21
Corn Starch[b]	42	0.09
Hydorxypropylcellulose[c]	65	0.015
(Total)		0.315

a) Pharmatose 200M, DMV (Crystallized lactose)
b) Corn Starch W, Nippon Shokuhin Kakou Co., Ltd.,
c) HPC-EFP, Shin-Etsu Chemical Co., Ltd.,

2.3 Method

Experiment was conducted as follows. The powder samples having penetrated through a 50-mesh (300 μm) sieve were fed into the preheated vessel, then mixed by the fluidization air for 600 s. After setting the operational conditions as described in Table 3, the binder solution (purified water) was top sprayed by a binary nozzle as a form of mist with controlling the feeding speed by the digital

Table 3 Experimental conditions

Rotational Speed	10	rps
Air Volume	0.01	m^3/sec
Spray Nozzle Height	0.10	m
Nozzle Insert	ϕ1.0	mm
Spray Pressure	2.0×10^5	Pa
Inlet Air Temperature	333	K

program controller. After granulation was over, drying process was started.

Granule size distribution was determined by a sieve analysis with rotating sieve shaker. About 10 g of the granules were shaken for 180 s. After measuring the weight of the granules on each sieve, the particle size distribution was calculated by the log-normal distribution with the computer.

3. Results and Discussion

Figure 2 shows the temporal change of the moisture content and the power consumption measured. In the figure, $0<t\leqq 900$ s was the process of damping (the damping speed, $dW/dt=1.6\times 10^{-2} s^{-1}$), $900<t\leqq 2800$ s was the process of moisture control (set point was $W=15\%$), and $2800<t\leqq 3300$ s was indicating the drying process. Seen from Fig.2, the power consumption indicated almost steady state, and the fluctuation of each point seemed to be small. This was because the impacting forces that the blade received from the particles were small due to the fluidization of the particles. Therefore, we thought that discussion about the granulation mechanism under moisture control by means of this measured power consumption data was impossible.

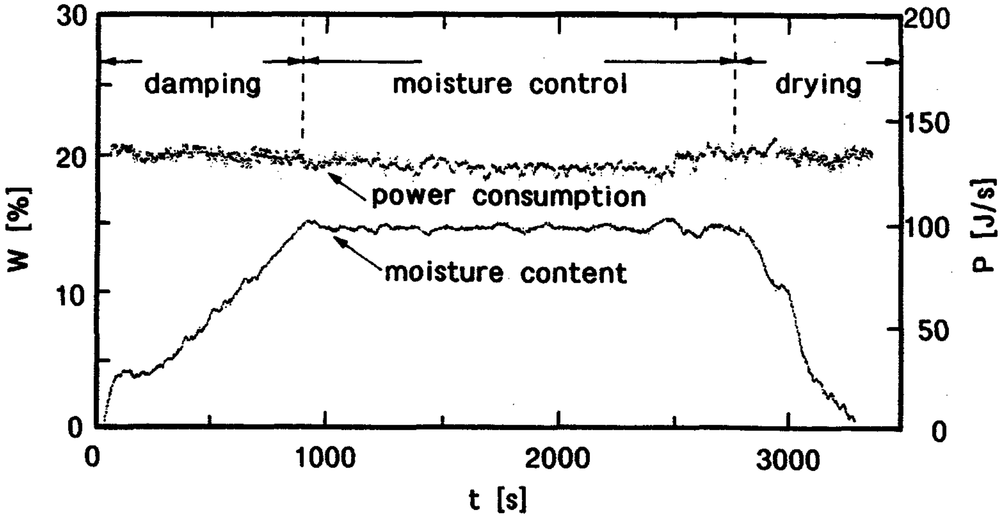

Fig. 2 Temporal change of the moisture content and the power comsumption

In this paper, for the analysis of granulation process, we applied coefficient of variation to the power consumption data as a new trial. The method used was as follows.

The mean difference of the power consumption was calculated as

$$\bar{P}=\sum_{i=t}^{t+T-1} P_i/T \tag{2}$$

Here, P_t was the power consumption data at time t, and T was the calculation interval (selected 30 s).

The mean deviation of the fluctuations σ was calculated by the following equation.

$$\sigma = \{\sum_{i=t}^{t+T-1} (P_i - \bar{P})^2/T\}^{0.5} \qquad (3)$$

The coefficient of variation δ was defined as

$$\delta = \sigma/\bar{P} \qquad (4)$$

Figure 3 illustrates the temporal change of the coefficient of variation. The coefficient of variation δ indicated a large value at the damping process, then decreased at the moisture control phase. After taking the minimum value at t=1700 s, the δ increased again.

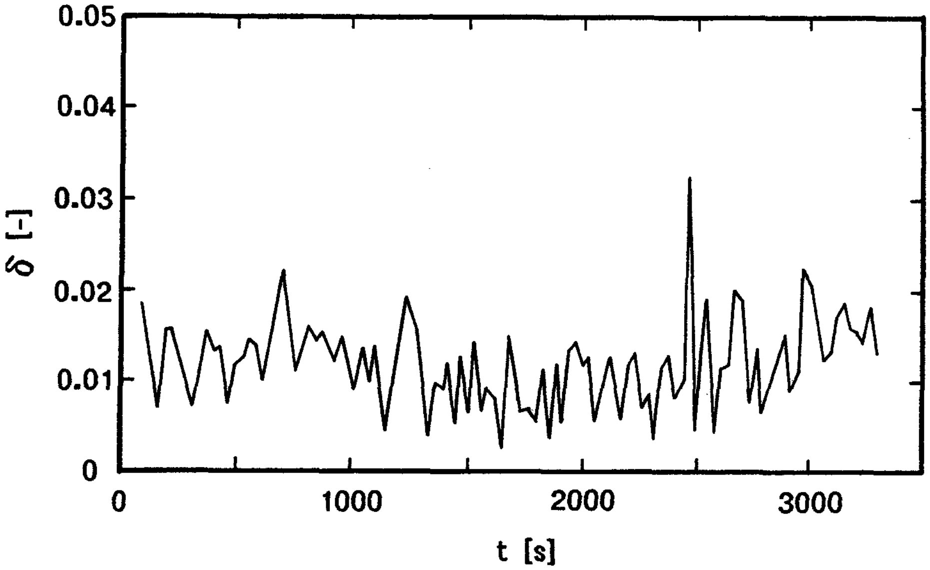

Fig. 3 Temporal change of the coefficient of variation of the power consumption

The factors affecting the fluctuation of the power consumption could be as follows; particle size distribution, appearance shape and the surface condition of the granulated particles. Therefore, we investigated the relation between the fluctuation of the power consumption and these granule properties.

Figure 4 illustrated the plots of the mean particle diameter and the geometric standard deviation as a function of time. In the initial stage of the moisture control, the mean particle size was a constant value. As we have already reported[3], that the mean particle diameter was determined by the moisture content, thus as far as moisture content was controlled to be constant value, D_{50} could not be changed. As the surface melted with the passage of time, however, cohesive force due to the liquid bridge increased. This phenomena gave rise to the cohesion of particle together, and the worsening of the fluidization condition in the phase t>1700 s. Judging from the geometric standard deviation plotted in the Fig.4, the granulated particles at t=1700 s have a most sharp particle size distribution, and from that time the particles indicated secondary agglomeration.

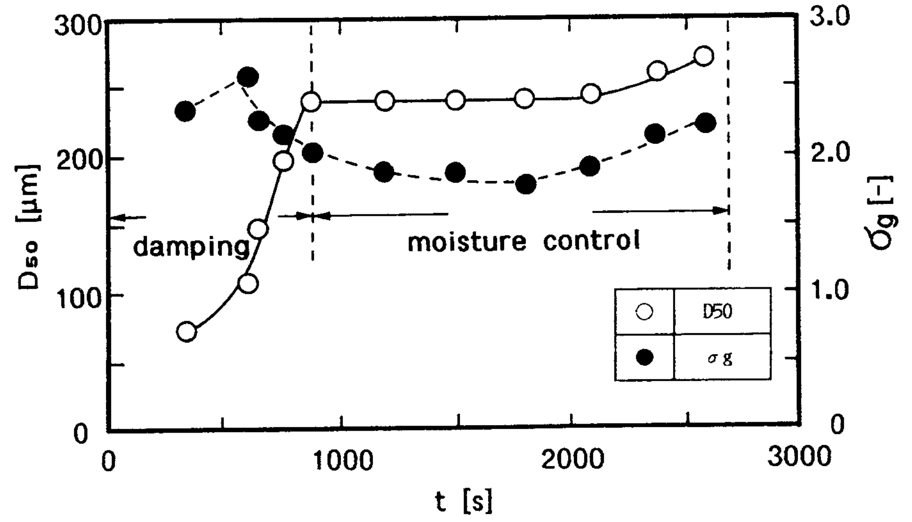

Fig. 4 Plots of the mean particle diameter and the geometric standard deviation

Figure 5 shows the plots of the angle of repose and the shape index. Here, shape index ϕ was the mean ratio of the short diameter/long diameter of the 50 granules obtained. The shape index of the particles in the moisture control phase was gradually approaching 1.0, meaning that they were gradually brought closer to the sphere. The angle of repose plotted in this figure indicated a rapid decrease in the moisture control phase. Since the number of times that the particles received the tumbling motion increased as the increase of operating time, the surface of the particles smoothed and the flowability was progressed. The angle of repose took the smallest value at t=2100 s and the shape index took the biggest value at this point.

Checking the correlation between each granule property and the temporal change of the fluctuation, geometric standard deviation of the particles was thought to be the main factor which affected the fluctuation of the power consumption. Thus, we investigated the relation between the geometric standard deviation and the fluctuation of the power consumption in the following.

Figure 6 denotes the plots of the coefficient of variation as a function of the geometric standard deviation. Here, the coefficient of variation plotted here was the value which was given a smoothing treatment from the original value because the data showed the periodical change. These plots gave a straight line, thus in case of the similarities of the size and the shape under moisture control, it was clear that the particle size distribution caused the fluctuation of the power consumption.

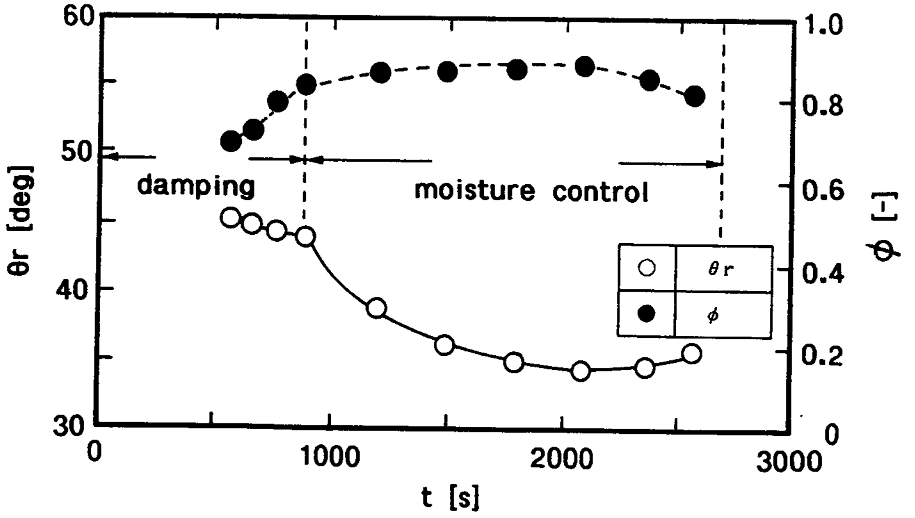

Fig. 5 Plots of the shape index and the angle of repose

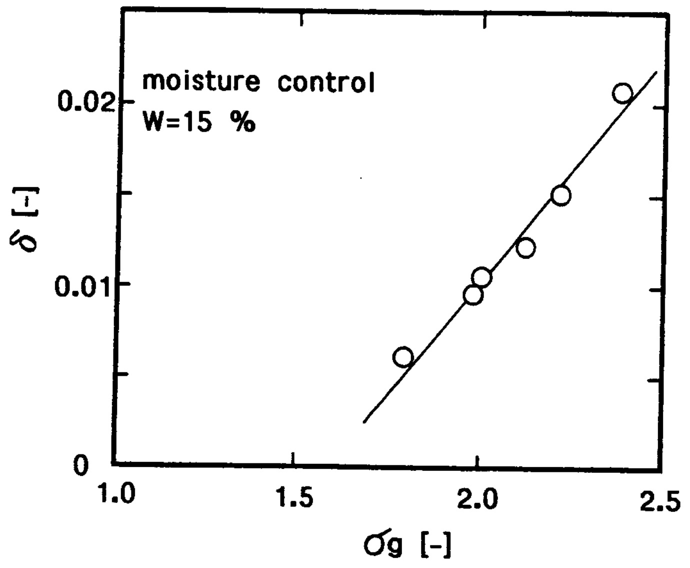

Fig. 6 Effect of the geometric standard deviation on the coefficient of variation of the power consumption

From these findings, it was concluded that in the phase where the particle size distribution was wide, the impacting force closely connected with the mass of the particles could be distributed widely. These wide distribution of impacting force caused the large value of the fluctuation of the power consumption. As a results of this, it was found that by calculating the fluctuation of the power consumption we could determine the optimum operating condition where the particle size was indicating the most narrow distribution.

In this way, the method which could detect the particle growth and determine the operational end-point was demonstrated in this work, and a practical technique which realize these analysis in real time will be presented in the next paper.

4. Conclusions

Fluctuation analysis of the power consumption was conducted to detect the granule growth and to determine the operational end point under moisture control. From the experimental findings, we obtained the following conclusions;

1) Measurement and control of the process variables needed for the automatic operation of the tumbling fluidized bed were discussed.
2) Since the relation between the coefficient of variation of the power consumption and the geometric standard deviation was linear, it was clear that the fluctuation of the power consumption was mainly originated from the unevenness of the impacting force caused by the wide particle size distribution.
3) A practical method for the determination of the optimum operational end point by use of the fluctuation of the power consumption in the tumbling fluidized bed granulation was developed.

Nomenclature

D_{50}: mean particle diameter, μm
E : voltage, V
I : current, A
P : power consumption, J/s
W : moisture content, %
δ : coefficient of variation of power consumption
θ : phase difference, deg
θ_r : angle of repose, deg
σ_g : geometric standard deviation
ϕ : shape index

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