Development and Application of Infrared Moisture Sensor to Complex Granulation

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Development and Application of Infrared Moisture Sensor to Complex Granulation

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Infrared moisture auto-measuring system with optical fiber was developed to measure and control the moisture content in a complex granulator. Moisture content was continuously monitored in personal computer and its self control was conducted by means of feed back control (PID control) of rotation speed of roller pump used for adding binder solution with the application of digital program controller (D/D converter).

Granulation was carried out in the complex granulator which is a type of fluidized bed combined an agitator blade, which could be used for multipurpose.

In this paper, influence of the agitator blade on granule properties was examined at the variety of the type and rotation speed of the blade, thus we determined the optimum operational conditions for the production of high yield of compacted fine granules.

1. Introduction

Recently, "Good Manufacturing Practice" has become a serious subject of discussion in the pharmaceutical and other industries which are dealing with powder materials. To clear up this difficulty as well as to produce uniform quality reproducible, introduction of factory automation has been indispensable.

In this paper, we focused our attention on granulation which is carried out with a widespread use in many fields. Purpose of granulation had been for controlling basic component (size, size distribution and yield of granules), however, needs for granules have changed to produce high quality materials with a 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[10-16]. These researches were, however, insufficient from the view of factory automation, therefore, knowledge which is needed for factory automation as well as for the production of high quality materials is to be necessary obtained. By this reason, we

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adopted a complex granulator[17], which is a type of fludized bed combined an agitation blade and an infrared moisture sensor.

Moisture content was measured by the infrared moisture auto measuring system which we have developed, and its self control was conducted by means of feed back control (PID control) of rotation speed of roller pump used for adding binder solution with the application of digital program controller (D/D converter).

As we have already reported the granulation mechanism under moisture control[18,19], in this paper we investigated the influence of agitator blade on granule properties (mean particle diameter, apparent density and yield of fine granules), thus we determined the optimum condition for complex granulation.

2. Experimental

2.1 Equipment

Measurement theory of the infrared moisture sensor[20] is as follows. Absorption spectrum of water is indicated in Fig. 1. Water has a absorption spectrum at the wave length of 1.43, 1.94, 3.0 μm in the near-infrared range, energy of the spectrum is absorbed in proportion to its moisture content when irradiated these spectrum to wetting substance. Moisture content of the substance can be detected by measuring the difference between the energy of flood and reflected spectrum. As the measuring method of only absorption spectrum is easy to accept a disturbance owing to a surface condition and a change of measuring length, we adopted a method calculating a ratio of reflected energy of the spectrum from the water absorbed spectrum and its both side unabsorbed spectrum. It can be cancelled the disturbance because disturbance is influenced same proportion to a absorbed and unabsorbed spectrum.

Details of detector is illustrated in Fig. 2. Spectrum from light source is condensed

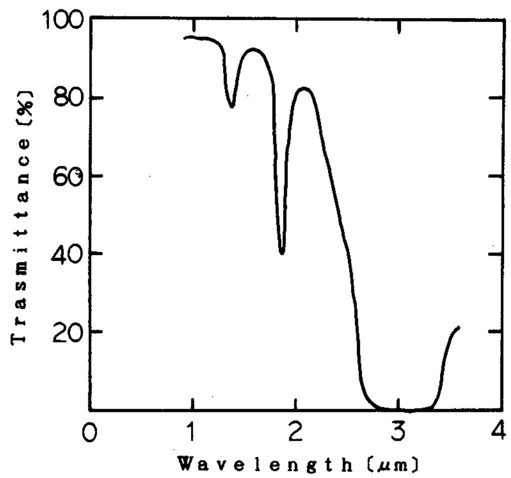

Fig. 1 Absorption spectrum of water.

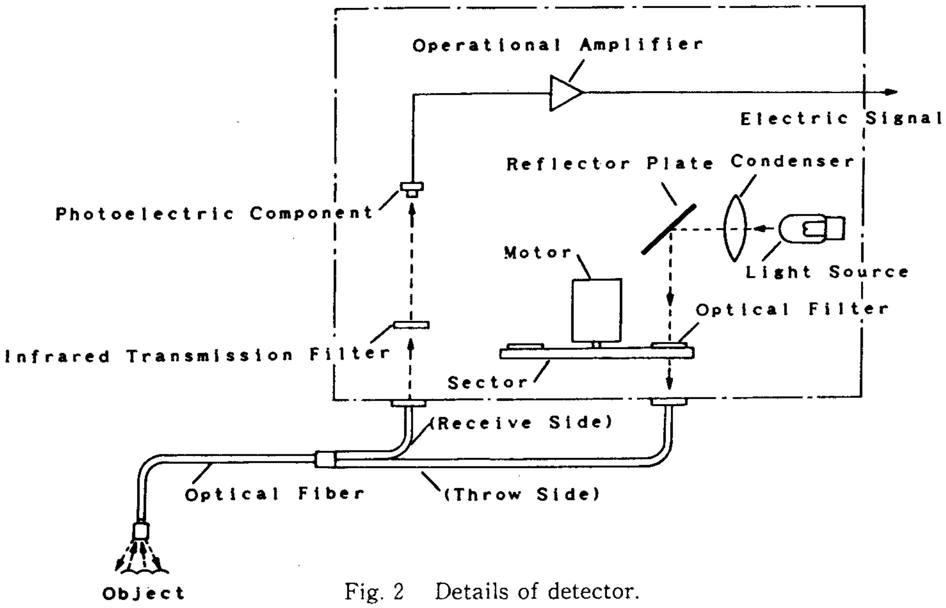

Fig. 2 Details of detector.

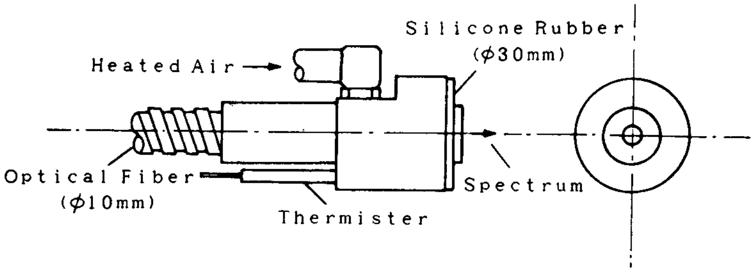

Fig. 3 The extremity of the optical fiber.

by a condenser, reflected by reflector plate, then changed to an alternating-current spectrum by going through a sector which has four optical filter. This filter can go through only spectrum of absorption wave length of water (1.94 μm), two side wave length of it, and visible radiation. Spectrum of absorption wave length of water is partly absorbed and the rest of it is irregularly reflected in proportion to the moisture content of the object, while comparison spectrum of unabsorbable wave length is fully reflected. These spectrum are transmitted to electric signal at a photoelectric component after carried by a optical fiber (10 mm in diameter and 2 m in length) and go through far-infrared transmission filter. Direct-current of this electric signal is cut off in a condenser, which meaning that direct-current disturbance spectrum like solar rays can be cut off to prevent from disturbance. This cut signal is digitalized by a 12-bit A/D converter and calculated in personal computer. Sampling interval of this system is about 70 ms (14 times/s), which is smoothing treated to deprive the scattering of data.

The extremity of the optical fiber is schematically described in Fig. 3. The extremity of the optical fiber is 30 mm in diameter, which is very compact useful to laboratory size apparatus without a large scale reconstruction. This sensor has a device to prevent powder adhesion at the extremity of the optical fiber by blowing a heated air at the feed speed of 5×10^{-4} m^3/s. Temperature of this heated air is measured by a thermister at the extremity of fiber, which is controlled to set up at the same temperature as an inlet air temperature so as not to disturb the flow pattern. Silicone rubber is also equipped to protect the extremity of fiber.

Unit system of complex granulator and the infrared moisture sensor is schematically illustrated in Fig. 4. Complex granulator (NQ-LABO, Fuji Paudal, Co., Ltd.) equipped with a binary spray nozzle was used for granulation experiment. Container of the complex granulator is made of acrylic resin in order to observe the particle flow. On the bottom of this container, agitator blade was equipped for tumbling and compacting the granule particle with fluidized by heated air. This agitator blade is possibly changeable to various types for any purposes. Powder accompained with fluidized air was entrapped by a bag filter, and exchanged by a pulse jet air. Pulse jet interval selected was 20 s.

Output signal from transducer is electric current (4-20 mA) and voltage (0-10 mV), and the former is transported to digital program controller (D/D converter) to be used for a control signal of roller pump, the latter is digitalized in a 12 bit A/D converter to be calculated in a personal computer. During the experiment, moisture content was continuously monitored in a personal computer.

2.2 Powder samples

Starting materials and its mixing ratio are listed in Table 1. Starting materials

Table 1 Materials and Composition

Materials	Mean particle size [μm]	mixing weight [kg]
Lactose[a]	104	0.21
Corn Starch[b]	42	0.09
Hydroxypropylcellulose[c]	65	0.015
(Total)		0.315

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

Table 2 Experimental Conditions

Air Volume	0.01	m^3/sec
Spray Nozzle Height	0.15	m
Spray Pressure	2.0×10^5	Pa
Inlet Temperature	333	K
Liquid Flow Rate	2.5×10^{-4}	kg/sec

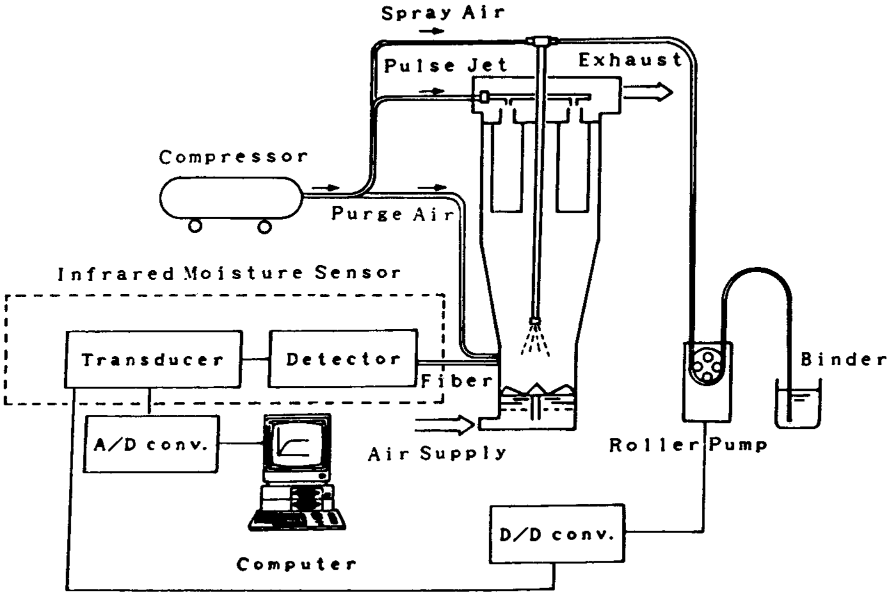

Fig. 4 Unit system of complex granulation.

were 0.3 kg of 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 starting materials before granulation. Purified water was used as a binder solution.

2.3 Experimental method

Experiment was conducted as follows. Powder samples penetrated through a 50 mesh (300 μm) sieve were fed into a preheated container, then mixed with fluidized air for 600 s. After instituted the operational conditions as described in Table 2, binder solution (purified water) was top sprayed by a binary nozzle as a form of mist. After granulation was over, adding of binder solution was stopped to shift to drying process.

Granule size distribution was determined by sieve analysis with rotating sieve shaker. The screen opening of the sieves were 75, 106, 150, 177, 250, 297, 500, 840, 1410 and 2180 μm, respectively. About 10 g of the granules were shaken for 180 s. After measuring the weight of the granules on each sieves, particle size distribution was calculated by the log-normal distribution with computer.

3. Results and Discussion

3.1 Correlation between moisture content and output of detector

Output characteristic of the infrared moisture sensor is differed from measuring

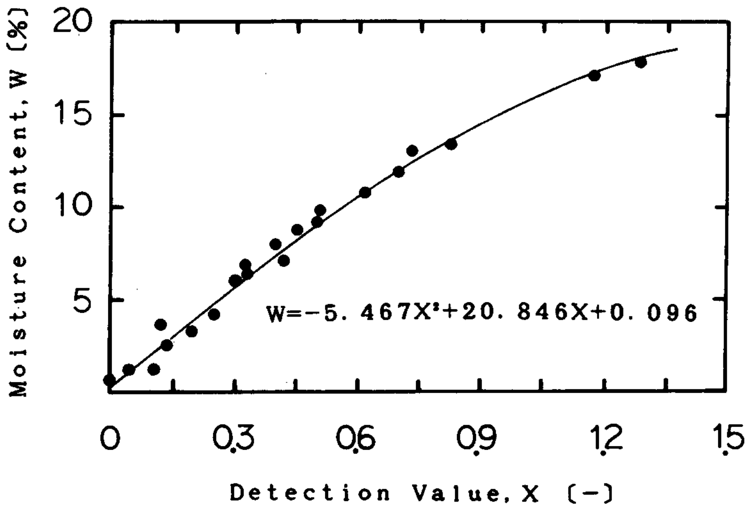

Fig. 5 Moisture content as a function of detection value.

object. To measure the moisture content correctly correlation between moisture content of each object and output of moisture sensor must be needed. Samples of various moisture content of the object were taken to measure the moisture content by substituting the wet mass and its dry mass to following equation (Eq. (1)). Here, dry mass was obtained after drying for 24 hours at 318 K in a shelf dryer.

$$\text{moisture content} = \frac{\text{dry mass (g)} - \text{wet mass (g)}}{\text{wet mass (g)}} \times 100\ (\%) \qquad (1)$$

Correlation between moisture content obtained by the drying method and output of detector was indicated in Fig. 5.

Correlation between the moisture content and the output of detector was corrected by the least squares method to obtain quadratic curve. The quadratic equation was as follows.

$$W = -5.46X^2 + 20.846X + 0.096 \qquad (2)$$

Substituted this equation into the transducer of moisture sensor, moisture content of this formulation is all transmitted from the output of detector with the basis of Eq. (2).

3.2. Effect of the agitator blade on granule particles

To investigate the effect of agitator blade on the granule particles experiment was conducted with changing the types and rotation speed of agitator blade. Here, moisture content of granulation was controlled to be 15% in order to ignore the factor of moisture content. Figure 6 shows the change of moisture content during the experiment. Binder solution was programmed to spray to the granules at the moisture content before 15%. After the moisture content had reached 15%, PID

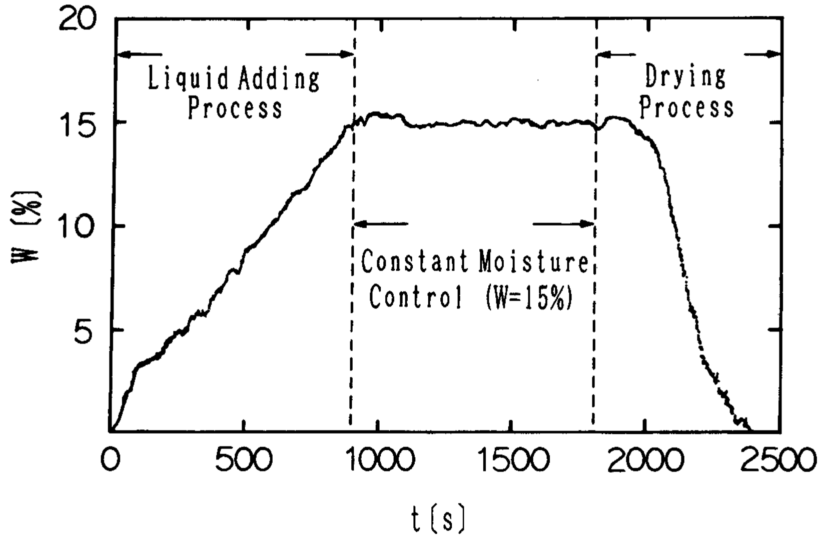

Fig. 6 Change of moisture content during the experiment.

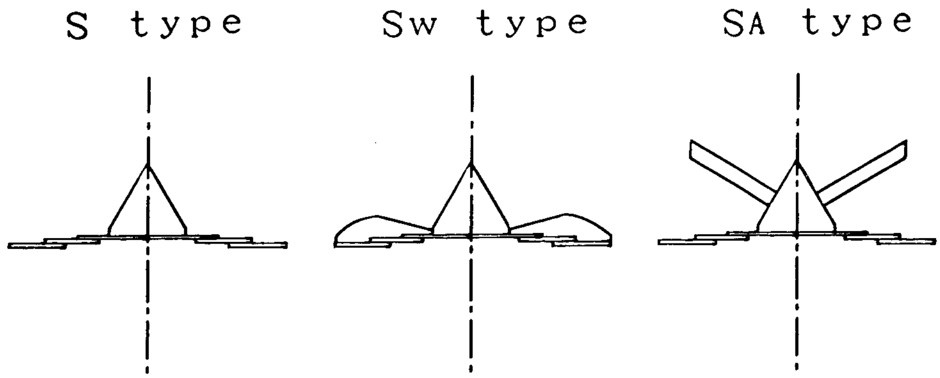

Fig. 7 Types of agitator blade

control of flow rate was carried out to keep the moisture content constant for 900 *s*. At $t=1800$ *s*, binder solution was programmed to stop so as to shift to drying process. Drying process was ended when $W=0\%$. Overshoot of the moisture content was only 0.5% and the fluctuation from the set up moisture content ($W=15\%$) is also $\pm 0.3\%$, which is in the permitted limit, thus it is found that control to keep the moisture content at the preset value was favorably conducted.

Granulation mechanism under moisture control has been already reported[17], thus in this paper we omitted.

Three types of agitator blade used are illustrated in Fig. 7. S type blade has no agitator blade equipped only distribution plate, however, S_w and S_A have different shape of agitator blade, respectively.

Figures 8, 9 and 10 show the mean particle diameter, apparent density and yield

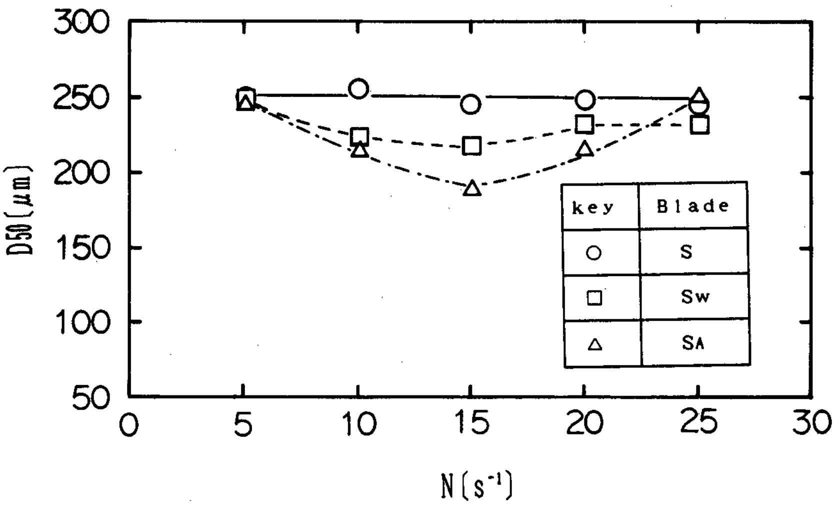

Fig. 8 Relation between mean particle diameter, D_{50} and rotation speed, N.

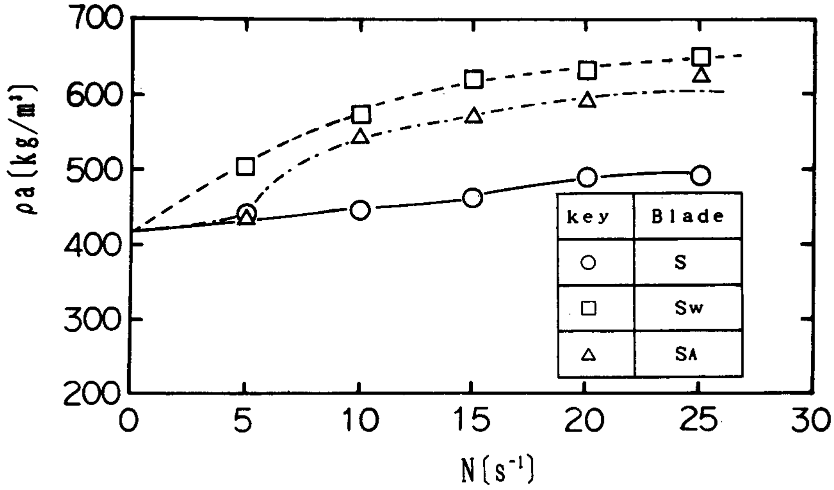

Fig. 9 Relation between apparent density, ρ_a and rotation speed, N.

of fine granules as the function of rotation speed at the variety of the types of blade. To discuss these phenomena, particle flow pattern is illustrated in Fig. 11. Flow pattern of the particle was mainly divided into three phase.

Phase $A(0 \leqq N \leqq 5\text{rps})$: the flow of particles were random circulation, with only vertical motion by the fluidized air because the agitation faculty was small. Owing to this low shearing stress and tumbling effect, particles in this phase show the low apparent density ($\rho_a = 420 \sim 500$ kg/m^3).

Phase $B(5 \leqq N \leqq 15\text{rps})$: circulating laminar agitation flow was observed. Vertical motion of particles by the fluidized air was valanced to the rotational motion by the agitation flow. Granules in this pahse were caught appropriate shearing stress, which was resulted in the high apparent density. Reason that the D_{50} indicated the tendency

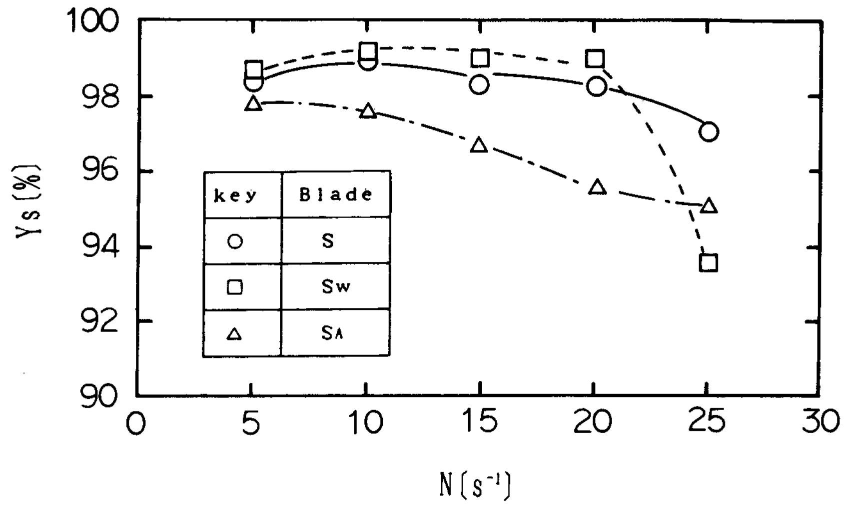

Fig.10 Relation between yield of fine granules, Y_s and rotation speed, N.

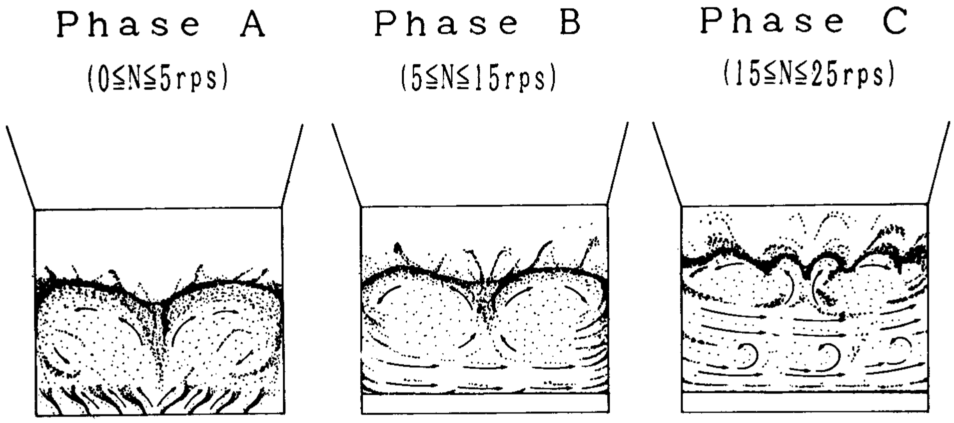

Fig.11 Particle flow pattern.

of decrease in this phase in the Fig. 7 was that the particles were compactly packed by the agitator blade. As the yield of fine granules were indicating the maximum value in this phase, we determined the optimum rotation speed was in this phase.

Phase $C(15 \leqq N \leqq 25\text{rps})$: rotational motion was dominated the particle flow because of the high rotation speed. High shearing stress and tumbling effect was caught to the particles, which was resulted that particles were irregularly agglomerated, thus increase of mean particle diameter and decrease of yield of fine granules were observed.

Over the range of this rotation speed $(0 \leqq N \leqq 25\text{rps})$, S_w blade indicated the high apparent density and high yield of fine granules, thus this blade was suggested to be preferable to agitator blade.

These results were finally lead to the conclusion that it was the optimum condition for the production of high yield of compacted fine granules when we use the S_w blade at the rotation speed of $N=10$ rps.

4. Conclusions

Complex granulation was conducted under moisture feed back control, we got following results;

1) By means of infrared moisture auto measuring system we developed, self control system of complex granulation from mixing to drying was fully established. These knowledge suggests that they will have a beneficial effect on the construction of factory automation.
2) Effect of agitator blade on the granule particles was examined at the variety of the types and rotation speed of agitator blade, thus we acquired the optimum condition for the production of high yield of compacted (large value of ρ_a) granules.

Nomenclature

D_{50} : 50% mean particle diameter, μm
N : rotation speed of agitator blade, s^{-1}
t : granulation time, s
W : moisture content, %
X : detection value, −
Y_s : yield of fine granules (−500/+74 μm), %
ρ_a : apparent density, kg/m^3

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