

EFFECTIVE COMMINUTION OF DIATOMACEOUS EARTH IN A VERTICAL TYPE JET MILL

Suck Ho Yoon, Sang Do Kim, Yong Kang[†], Young Joo Kim and Seung Ho Cho

Department of Chemical Engineering, Chungnam National University, Taejon 305-764, Korea

(Received 19 January 1995 • accepted 20 May 1995)

Abstract—Comminution characteristics of diatomaceous earth have been investigated in a vertical type jet mill of pilot plant scale; the diameter of the chamber of mill is 26.3 cm. Fluidization of the particles in the chamber has been proposed to enhance the comminution efficiency of the jet mill by promoting the effective contacting of powders with compressed air as well as the chamber wall. Effects of the pressures or densities of pulverizing-air and fluidizing-air and comminution time on the mean size of the particles and the rate of comminution have been examined. The statistical analysis of pressure fluctuations in the chamber of the mill has been utilized to predict the characteristics of the vertical-type jet mill taking advantage of the fluidization technique. The particle size and comminution rate have been correlated with the operating variables, respectively.

Key words: *Comminution, Vertical Type Jet Mill, Fluidization, Statistical Analysis*

INTRODUCTION

Diatomaceous earth is essentially insoluble to the process liquors for which it can be used in the filtration processes. The diatomaceous earth has been successfully employed as a filter-aid in the various filtration processes to clarify the liquid including fine particles as well as particles with wide size distribution, because its micro-structure has known to be effective for the separation and filtration of the liquid-particle mixture [Alicatore and Neu, 1971; Culver, 1975; Basso, 1982; Yoon et al., 1983]. The filtration efficiency of the filter-aid, of course, has been appeared to be dependent upon the porosity and pore characteristics such as size, shape and distribution etc. [Yoon et al., 1993].

For the application of diatomaceous earth in the filtration processes, its coarse phase has to be comminuted to fine particles retaining micropores in it. However, it has been difficult to protect the micropores in the diatomaceous earth by grinding in the usual mechanical milling or grinding system. Thus, jet mill can be utilized to reduce the size of diatomaceous earth particles without breaking down the micropore existing interior of the particle. The jet mill has found a lot of applications in the industries where extremely fine particles are required, since it has several advantages compared with other conventional mills; it can produce fine powders with narrow size distribution, high level of purity and no attritional heat; it can be used as a reactor in which a gas-solid reaction would occur combined with comminution. It has been generally known that the jet mill can be suitable for comminution the thermally-weak powders such as nylon, resin etc., since it does not affect any thermal impact on the powders during comminution in the chamber. Thus, the characteristics of jet mill have been studied by several investigators [Mori, 1965; Hendry, 1967; Ramanujam and Venkateswarlu, 1969; Manyhart and Miskiewicz, 1976; Honma et al., 1982].

Recently, Ito [1987] examined the scale up theory of single track jet mill. To obtain the design criteria for the scale up to

whole-sized plant, a pilot plant of horizontal-type jet mill having two types of grinding chamber was studied by present authors [1990]. To improve the grinding effect for materials such as glass beads, Honma et al. [1984] used the grinding medium in the jet mill, and they suggested a method using grinding medium for materials with poor grindability. Present authors [1989] also tried to install a deflectors in the grinding chamber of the horizontal jet mill to increase the pulverizing efficiency of the mill. However, most of the investigators have used the circulation-type or horizontal-type jet mill.

In the present study, the vertical-type jet mill of pilot plant scale has been used to treat the larger amount of diatomaceous earth powder than that by the previous horizontal-type in a given time. And the fluidization technique has been employed to enhance the comminution efficiency of the vertical-type jet mill.

EXPERIMENT

Experiments were carried out in a vertical-type jet mill of pilot plant scale, which was composed of three parts; feeding comminution and collection as can be seen in Fig. 1. The diameter of comminution chamber of the mill was 26.3 cm. Compressed air was injected into the mill through the six nozzles located at the opposite sides of the mill wall. The air was dehumidified by means of dehumidifier and adjusted at a constant temperature of 25°C.

The pressure of pulverizing air was in the range of 5.0-7.0 kg/cm². The angle of the injection nozzle was either 35° or 45° downward from the horizontal. Air was also injected through a nozzle at the bottom of the mill to fluidize the particles during comminution, which was intended to improve the comminution efficiency of the mill. The pressure of fluidizing air was in the range of 0-3.0 kg/cm². The pressure of compressed air were controlled automatically by PID control system.

Comminuted fine particles were separated from the chamber by microseparator at the top of the mill. The separator was designed to separate comminuted fine particles by adjusting the revolu-

[†]To whom all correspondences should be addressed.

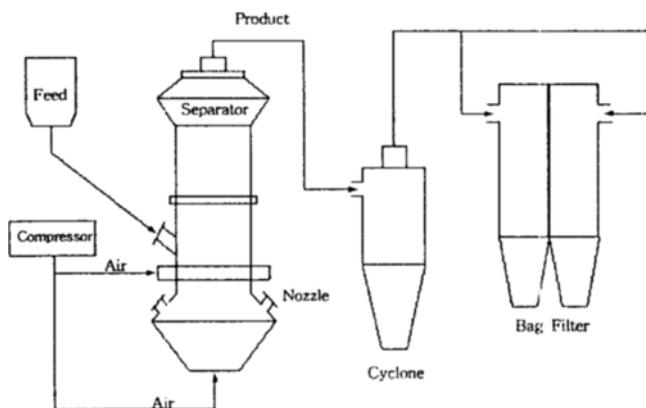


Fig. 1. Schematic diagram of experimental system.

tion, which varied in the range of 3000-6400 rpm depending on the particle size and density. The fine particles from the microseparator were collected by bag filter. The bag filter was designed to recover the fine particles effectively by impacting the air pulsation in a designated time interval. To analyze the product, the particles were sampled at each time interval from the product storage tank which was installed at the bottom of the bag filter. The comminution time was varied from 5 to 30 min for each experimental run. Diatomaceous earth whose size distribution can be seen in Fig. 2 was used as a feed material.

To predict the flow motion of the stream in the chamber, pressure fluctuations were also measured at each point as shown in

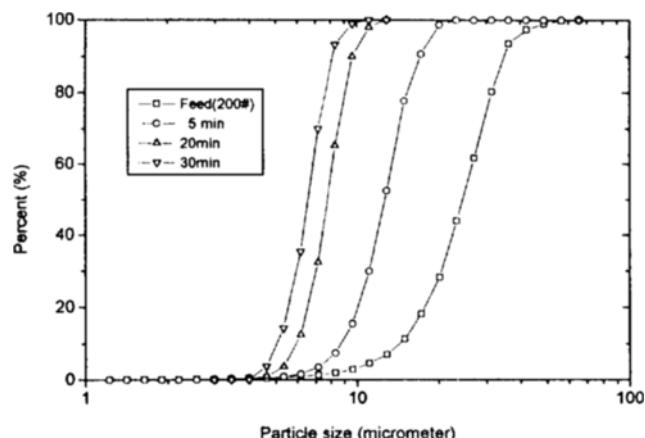


Fig. 2. Particle size distribution with the variation of comminution time.

(Feed material: Dialite # 200, $P_u = 7.0 \text{ kg/cm}^2$, $P_f = 1.5 \text{ kg/cm}^2$
Separator speed: 10,000 rpm, Feed amount: 4 L/batch)

Fig. 3, and analyzed by statistical method [Bendart and Piersol, 1980; Fan et al., 1993; Yoon et al., 1994]. The pressure was connected to the pressure transducer which produces an output voltage proportional to the pressure fluctuations. The sampling rate was 50 msec and a typical sample comprised 3000 points. The measured data were stored in the computer by means of data acquisition system (PCLS-805 SNAPSHOT) to analyze them off-line [Fan et al., 1993; Kang et al., 1994; Yoon et al., 1994].

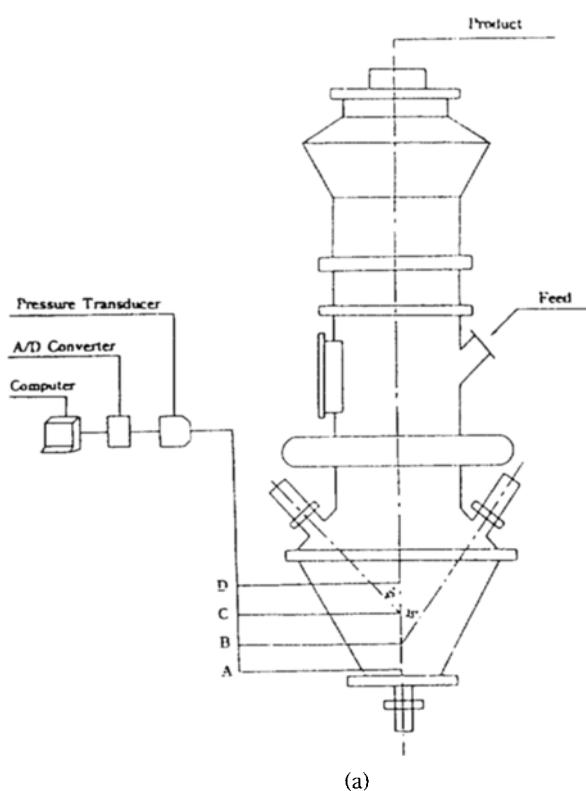
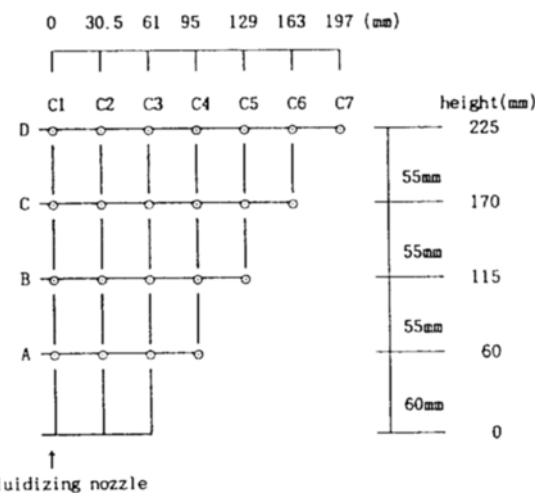


Fig. 3. Schematic diagram of vertical type jet mill.

(a) Schematic diagram of the pressure fluctuation measuring system. (b) Measuring points of the pressure fluctuation in the chamber



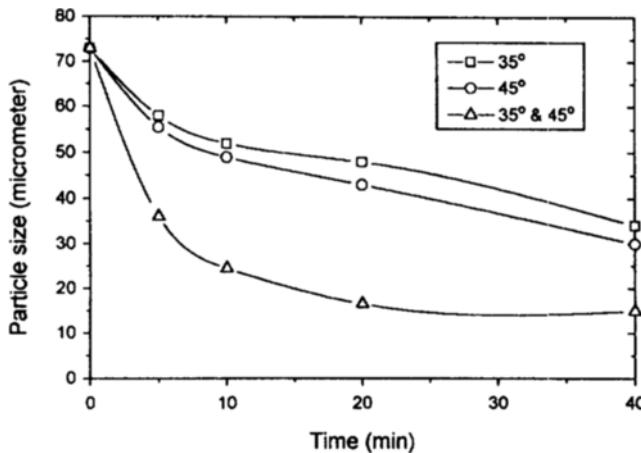


Fig. 4. Effect of nozzle angle on the particle size.

(Feed material: Dialite #200, $P_u = 7.0 \text{ kg/cm}^2$, $P_f = 1.0 \text{ kg/cm}^2$
Separator speed: 4,000 rpm, Feed amount: 4 L/batch)

ANALYSIS

The time series of pressure fluctuation $X(t)$ can be obtained by means of stochastic process to represent the historical random data. Thus, the mean value of the ensemble of the pressure fluctuations can be obtained as Eq. (1).

$$X_M(t) = \int_{-\infty}^{\infty} X(t) P(x:t) dx \quad (1)$$

where $P(x:t)$ is the first probability density function.

If the $P(x:t)$ is independent of time it can be written as $P(x)$, and the system is ergodic and stationary state. Then, the mean value, variance, skewness and kurtosis of the $X(t)$ can be calculated from the following equations, respectively [Bendart and Pierpol, 1980; Yoon et al., 1994].

$$X_M = \int_{-\infty}^{\infty} X(t) P(x) dx \quad (2)$$

$$\sigma^2 = \int_{-\infty}^{\infty} (X - X_M)^2 P(x) dx \quad (3)$$

$$S = \frac{1}{\sigma^3} \int_{-\infty}^{\infty} (X - X_M)^3 P(x) dx \quad (4)$$

$$K = \frac{1}{\sigma^4} \int_{-\infty}^{\infty} (X - X_M)^4 P(x) dx \quad (5)$$

RESULTS AND DISCUSSION

1. Comminution of Particles

Typical example of particle size distribution of the sample from the jet mill can be seen in Fig. 2 with the variation of comminution time in the chamber. In this figure, the mean size of the particle decreases and the size distribution becomes narrow with an increase in the comminution time.

Effects of the number and the angle of the nozzle through which the pulverizing-air has been injected into the chamber on the particle size comminuted can be seen in Fig. 4. It can be noted in this figure that the mean diameter of particle size in using the three nozzles installed at the wall of the chamber with an angle either 35° or 45° downward from the horizontal has been similar each other, however, the particle size produced from

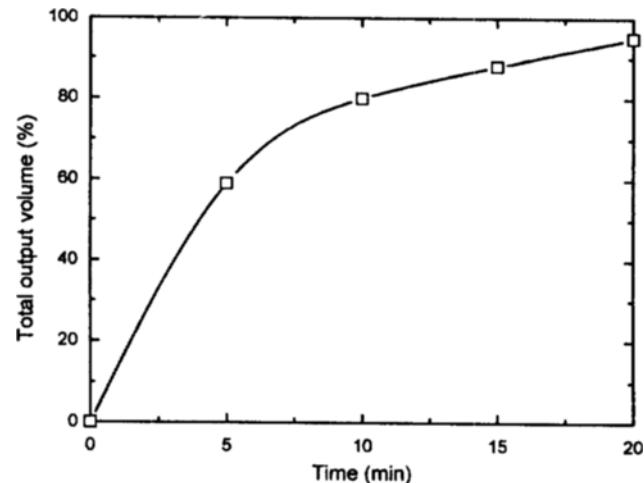


Fig. 5. Residence time of fine particles in the mill.

(Feed material: Dialite #200, $P_u = 7.0 \text{ kg/cm}^2$, $P_f = 1.5 \text{ kg/cm}^2$
Separator speed: 10,000 rpm)

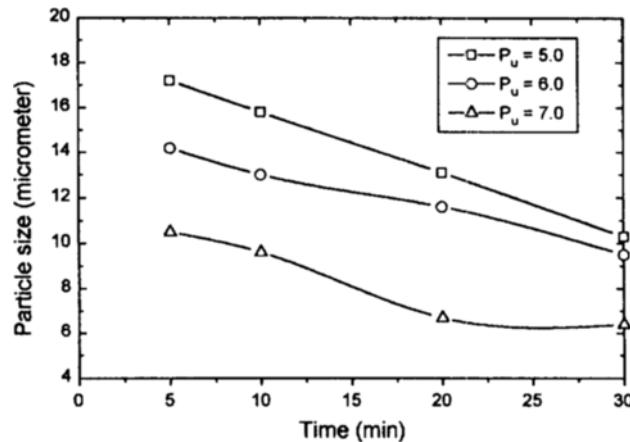


Fig. 6. Effect of pulverizing-air pressure on the particle size.

(Feed material: Dialite #200, Separator speed: 10,000 rpm
Feed amount: 4 L/batch, $P_f = 1.5 \text{ kg/cm}^2$)

the chamber with six nozzles; three of them inclined 35° downward and the others 45° downward from the horizontal, has been somewhat smaller than that from the chamber with three nozzles. Thus, the chamber with six nozzles has been employed in the present study. Typical relation between the output volume of the particles comminuted from the chamber and the comminution time can be detected from Fig. 5, where 60% of the particles has been produced within 5 minutes of operation and about 95% within 20 minutes.

Effects of density or pressure of pulverizing-air on the mean size of particle comminuted can be seen in Fig. 6. Note in this figure that the mean size of the particle decreases with an increase in the pulverizing air pressure injected at the wall of the chamber with designated angles.

Figure 7 shows the effects of density or pressure of fluidizing-air injected at the bottom of the chamber on the particle size comminuted. From this figure, the mean size of the particle comminuted decreases considerably when the fluidizing-air has been injected into the chamber, because the fluidization of particles

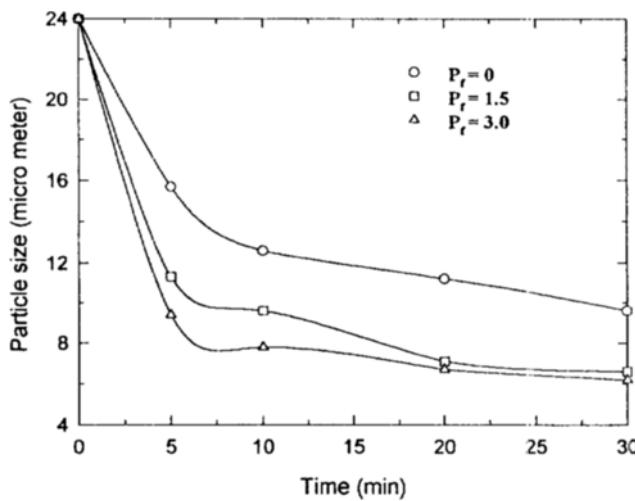


Fig. 7. Effect of fluidizing-air pressure on the particle size.
(Feed material: Dialite #200, Separator speed: 10,000 rpm
Feed amount: 4 L/batch, $P_f = 7.0 \text{ kg/cm}^2$)

in the chamber can enhance the efficiency of particle comminution. This can be due to the facts that the fluidization of particles can give rise to the formation of vertical ring-like flow of particles in the chamber where the particles are comminuted by jet streams of the compressed air [Yoon et al., 1989, 1994]. However, when the fluidizing-air pressure is greater than 1.5 kg/cm^2 , the ratio of particle size reduction is not profound, especially when the comminution time is longer than 20 minutes. The increase of fluidizing-air pressure greater than the suitable condition, furthermore, can result in the deterioration of the ring-like flow of particles at the comminuted region in the chamber, and thus it can not lead to the increase of the effects of effective fluidizing-air pressure on the wall as well as interior of the chamber. The pressure or density of the air in order to fluidize the particles in the chamber has to be controlled at an efficient condition in which the good flow stream of particles can be obtained in the chamber. At the higher level of fluidizing-air pressure, the jet stream of pulverizing-air injected at the wall of the chamber can not be focused but be distributed, thus, the efficiency of the jet stream to comminute the particles can be decreased. Therefore, the mean size of the particles does not decrease noticeably with an increase in the fluidizing-air pressure greater than 1.5 kg/cm^2 . The optimum combination of densities or pressures of pulverizing-air and fluidizing-air can be determined by considering the physical properties of particles such as size, density, shape and porosity etc.

The mean size of the particles comminuted in the vertical type jet mill can be correlated with the operating variables within operating conditions shown as Eq. (6), with a correlation coefficient of 0.928.

$$D_p = 6.93 P_u^{1.658} P_f^{-0.358} t^{-0.274} \quad (6)$$

where, $5.0 \leq P_u \leq 7.0 \text{ (kg/cm}^2\text{)}$
 $0 \leq P_f \leq 3.0 \text{ (kg/cm}^2\text{)}$
 $0 \leq t \leq 50 \text{ (min)}$

The comminution rate of particles, R_c , in the vertical type jet mill can be defined as the rate of change in particle size as Eq. (7).

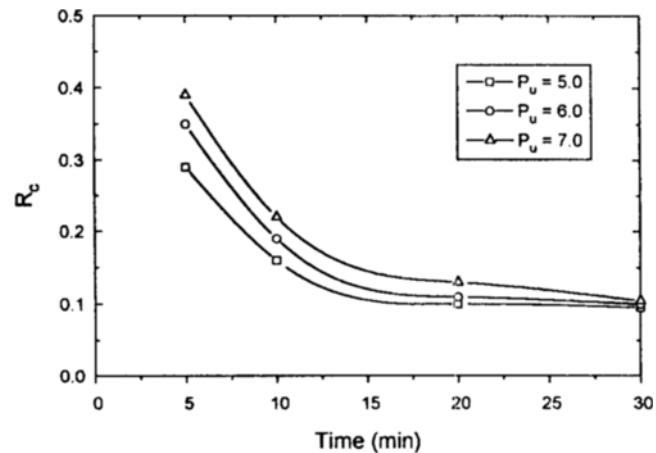


Fig. 8. Effect of comminution time on the comminution rate of particles.
(Feed material: Dialite #200, Separator speed: 10,000 rpm
Feed amount: 4 L/batch, $P_f = 1.5 \text{ kg/cm}^2$)

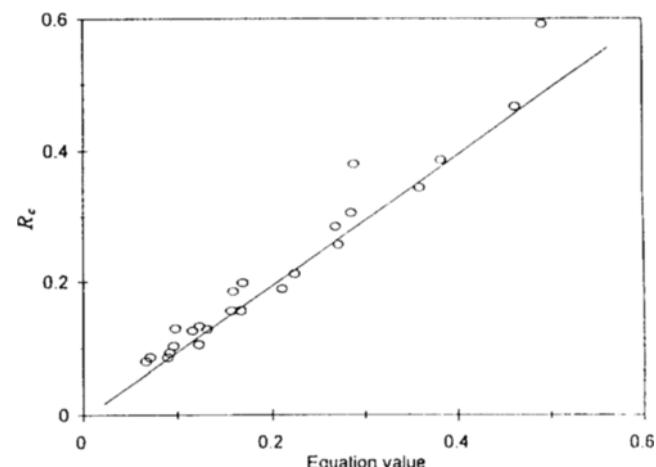


Fig. 9. Comparison between the calculated and the measured values of particle comminution rate.

$$R_c = \frac{d}{dt} \left(\frac{D_p}{D_p^0} \right) \quad (7)$$

Typical comminution rate of particles can be seen in Fig. 8 with pulverizing-air pressure and comminution time. The comminution rate decreases exponentially with an increase in the comminution time, but it increases with pulverizing-air and fluidizing-air pressures.

The comminution rate has been correlated with operating variables such as pulverizing-air pressure, fluidizing-air pressure and comminution time as Eq. (8) within experimental conditions.

$$R_c = 0.027 P_u^{1.907} P_f^{0.325} t^{-0.756} \quad (8)$$

where, $5.0 \leq P_u \leq 7.0 \text{ (kg/cm}^2\text{)}$
 $0 \leq P_f \leq 3.0 \text{ (kg/cm}^2\text{)}$
 $0 \leq t \leq 50 \text{ (min)}$

Eq. (8) has been well fitted to the experimental results with a correlation coefficient of 0.914 (Fig. 9).

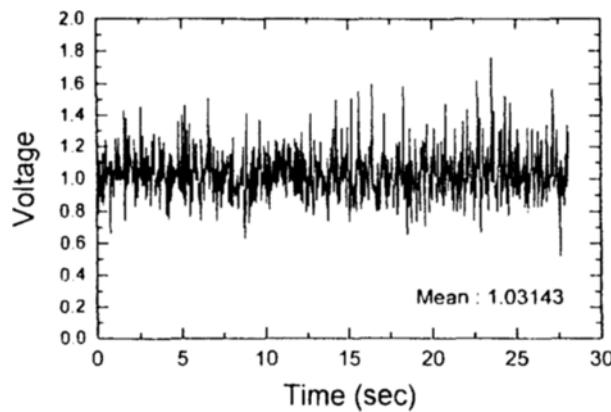


Fig. 10. Pressure fluctuation signals in the chamber of the mill.

($P_u = 7.0 \text{ kg/cm}^2$, $P_f = 1.5 \text{ kg/cm}^2$, Position: B)

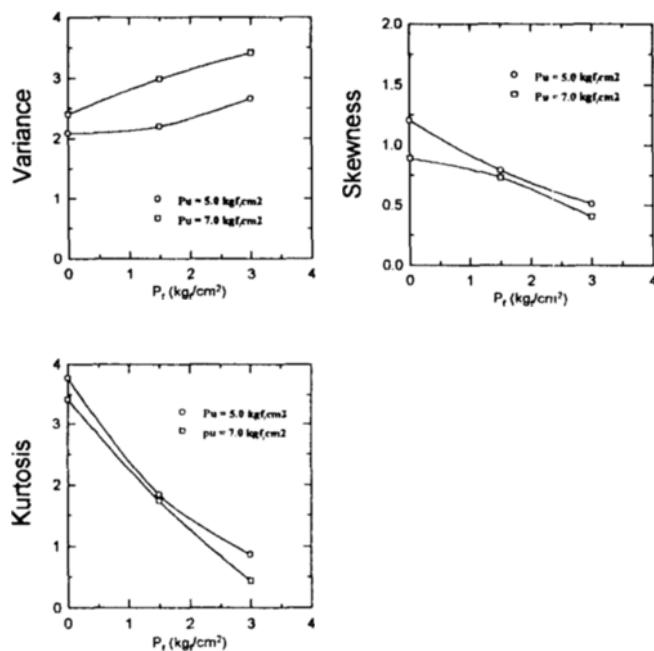
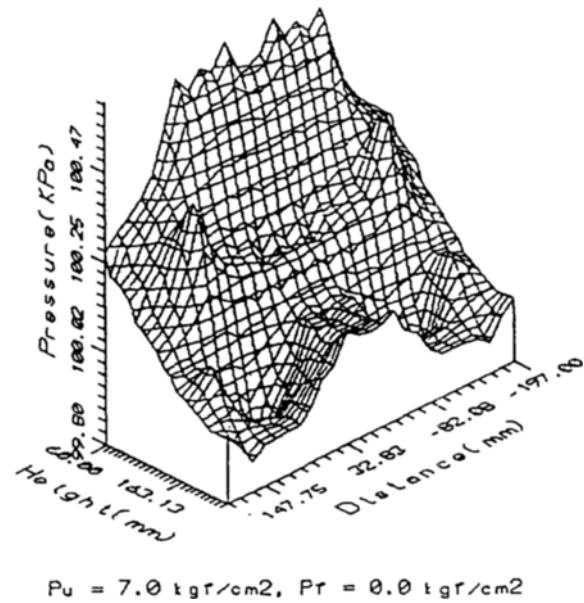


Fig. 11. Effect of fluidizing-air pressure on the variance, skewness, kurtosis of pressure fluctuation in the vertical type jet mill (Position: B).

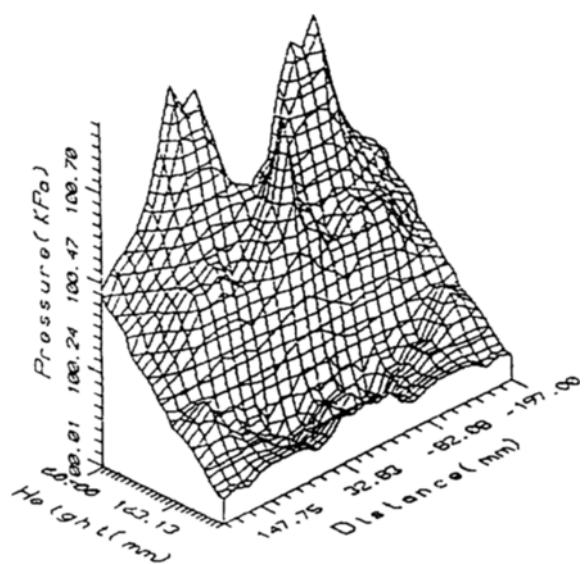
2. Pressure Fluctuations

Typical pressure fluctuation signals can be seen in Fig. 10. The data from these signals have been processed off line by means of statistical methods. The sample length comprises 3000 points for each operation, which is enough to analyze the characteristics of the pressure fluctuation signals.

Figure 11 shows the typical variance, skewness and kurtosis of the pressure fluctuation signals obtained at the position B in the chamber of the mill. From these figures, the variance increases but the skewness and kurtosis decrease with an increase in the fluidizing-air pressure. This can explain how the fluidization of particle can affect the flow in the chamber of the mill. That is to say, the increase of fluidizing-air pressure or density up to 1.5-3.0 kg/cm^2 can be resulted in the formation of vertical ring-like flow of particles in the chamber as can be seen in the three dimensional pressure distribution in the chamber (Fig. 12), how-



$P_u = 7.0 \text{ kgf/cm}^2$, $P_f = 0.0 \text{ kgf/cm}^2$



$P_u = 7.0 \text{ kgf/cm}^2$, $P_f = 1.5 \text{ kgf/cm}^2$

Fig. 12. The 3-D plot of the pressure fluctuation in the chamber of the mill.

ever, it can lead to the disturbance in the focusing of the jet stream of the pulverizing-air when the pressure level of fluidizing-air is relatively high. From Fig. 11, the variance of pressure fluctuation increases while the skewness and kurtosis decreases, when the pulverizing-air pressure has been changed from 5.0 to 7.0 kg/cm^2 . This can be related to the turbulent flow motion of gas stream in the chamber as follows; the increase of pulverizing-air pressure results in the increase in the turbulence and thus let the pressure fluctuations behave as described above. The turbulence generates the higher damage on the particles to be comminuted the smaller size.

CONCLUSION

The diatomaceous earth has been comminuted effectively in

the vertical type jet mill. The mean size of the particles decreases with increases in the pressure or densities of pulverizing-air and fluidizing-air and comminution time within this operating conditions. The comminution rate of particles increases with the pulverizing-air and fluidizing-air pressures, but it decreases with comminution time.

The statistical analysis of pressure fluctuation in the chamber of the mill can be utilized to predict the characteristics of the vertical type jet mill taking advantage of the fluidization technique.

NOMENCLATURE

D_p	: particle diameter [μm]
D_{p0}	: initial particle diameter [μm]
P_f	: fluidizing-air pressure [kg/cm^2]
P_p	: pulverizing-air pressure [kg/cm^2]
R_c	: the rate of comminution [1/min]
t	: comminution time [min]
$X(t)$: time series of pressure fluctuations [V]

REFERENCES

Alicatore, A. F. and Neu, E. L., "Comparison of Filteraids", *Chem. Eng. Progr.*, **67**, 49 (1971).

Basso, A. J., "Vacuum Filtration Using Filteraids", *Chem. Eng.*, **89**, 159 (1982).

Bendat, J. S. and Piersol, A. G., "Engineering Applications of Correlations and Spectral Analysis", Wiley, N. Y. (1980).

Culver, R. H., "Diatomaceous Earth Filtration", *Chem. Eng. Progr.*, **71**, 51 (1975).

Fan, L. T., Kang, Y., Neogi, D. and Yashima, M., "Fractal Analysis of Fluidized Particle Behavior in Liquid-Solid Fluidized Beds", *AIChE J.*, **39**, 513 (1993).

Hendry, R., "A Mathematical Model for Fluid Energy Mills", Dechema Monograph, Band 57, Verlag Chemie, 695 (1967).

Honma, T., Hasegawa, M. and Kanda, Y., "Jet Pulverizing Using Grinding Medium", *J. Chem. Eng. Japan*, **15**, 240 (1982).

Honma, T., Hasegawa, M. and Kanda, Y., "Effects of Feed Particle Shapes an Jet Pulverization", *J. Chem. Eng. Japan*, **17**, 221 (1984).

Ito, H., "Scale-up Theory of Single Track Jet Mill". Proc. of the 2nd Korea-Japan Powder Technol. Symp., 63 (1987).

Kang, Y., Min, B. T., Ko, M. H. and Kim, S. D., "Effects of Bubble Breakers the Pressure Fluctuations in the Continuous Bubble Columns", *Hwahak Konghak*, **32**, 58 (1994).

Menyhart, M. and Miskiewicz, L., "Comminution and Structural Changes in a Jet Mill", *Powder Technol.*, **15**, 261 (1976).

Mori, Y., "Studies an Jet Pulverizing", Dechema Zerkleinern Symp., 515 (1965).

Ramanujam, M. and Venkateswarlu, D., "Studies in Fluid Energy Grinding", *Powder Technol.*, **3**, 92 (1969).

Yoon, S. H., Kang, Y. and Cho, S. H., "Development of Fluidized Bed Energy Mill". Report to the Ministry of Commerce and Industry, Korea (1989).

Yoon, S. H., Kang, Y. and Cho, S. H., "Scale-up of Fluid Energy Mill in the University", Proc. 2nd World Congress of Particle Technol., Kyoto, Japan, 401 (1990).

Yoon, S. H., Kang, Y., Cho, S. H. and Kim, S. D., "Diatomite-filter-aid Manufacturing Process Improvement by Jet Grinding", Proc. 6th World Filtration Congress, Nagoya, Japan, 133 (1993).

Yoon, S. H., Kim, S. D., Kang, Y. and Cho, S. H., "Statistical Analysis of Pressure Fluctuations in a Fluidized Bed Energy Mill", Proc. of 4th Asian Conference on Fluidized Beds and Three-Phase Reactors, 37, Kyushu, Japan (1994).