Particle Size Measurement Based on Detection of Unbalance by Centrifugal Sedimentation†

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Abstract

For the purpose of determination of particle size distribution in the submicron range, a new apparatus has been developed on a basis of the principle of the balancing machine.

The change of balancing moment caused by the centrifugal sedimentation of the particles is detected with the sensors attached onto the rotor shaft and analyzed with personal computer, which gives particle size distributions according to the calculation proposed by one of the authors previously.

One of the advantages of this method is that the measurement by this method do not depend on the physical properties such as optical extinction coefficient, electric conductivity and so forth. It is also possible to determine the size distribution of the particles in a suspension of much higher concentration by this method than other ones. Furthermore, it is not necessary for the settling time of the particle to reach the detecting zone because the analysis by this method monitors the unbalance caused from the settling particles positioned in the whole suspension.

1. Introduction

The sedimentation method is one of the most popular ways to measure the particle size distribution of fine powders in the liquid phase. In order to determine the particle size in the submicron range, the sedimentation method is to be conducted in the field of the centrifugal force. Because of the simplicity in the measure-

ment, photosedimentmeter in which the particle concentration is determined from the transmission of the light through the suspension has been often used as one of the sedimentation methods. This method, however, requires the different coefficient for each material depending on the physical properties and the particle size of the sample powder.

One of our authors has previously proposed a sedimentation method based on detecting the magnitude of unbalance of a rotor caused by the centrifugal settling of the particles in a cell set in the rotor to obtain the particle size distribution¹⁾. Resting on the basis of this principle, a new apparatus for size analysis has been constructed recently equipped with a microcomputer to process the data of unbalance swiftly. In order to shorten the time required for the measurement, it was attempted to increase the rotation speed linearly to a constant

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value during the measurement and to determine the size distribution from the data during the period of the linear acceleration and the constant rotation^{2,3)}.

The measurement using this analyzer was carried out in the size range down to $0.03 \mu\text{m}$ with a sample having a good dispersibility to examine the reliability of the results and to find the lower limit of the size measurable by this method.

Some results of the size distribution obtained with this analyser were compared with those by other methods using the same sample.

2. Principle

The equation of motion of a single particle having a radius r in the field of the centrifugal force with an angular velocity ω is expressed as follows,

$$\frac{dx}{dt} = \frac{2}{9} \left(\frac{\rho_P - \rho_L}{\mu_L} \right) r^2 \omega^2 x \quad (1)$$

where ρ_P [kg/m^3] is the particle density,
 ρ_L [kg/m^3] is the liquid density,
 μ_L [$\text{Pa}\cdot\text{s}$] is the liquid viscosity,
 x [m] is the distance from the center in the radial direction.

Equation (1) is integrated under the condition of $x = x_i$ at $t = 0$, producing

$$\ln\left(\frac{x}{x_i}\right) = Kr^2 \omega^2 t \quad (2)$$

where

$$K = \left(\frac{2}{9} \right) \left(\frac{\rho_P - \rho_L}{\mu_L} \right) \quad (3)$$

When $Kr^2 \omega^2 t$ is replaced by T , Eq. (2) becomes

$$x = x_i e^T \quad (4)$$

The displacement of the center of gravity caused by the sedimentation of the particles in the cell is divided into the one due to the particles which have settled onto the bottom of the cell and the one due to the particles settling in the suspension as shown in Fig. 1.

Assuming that the particles located between x_i and x_m at $t = 0$ settle out of the suspension completely by $t = t$, the displacement of the center of gravity caused by these particles ϵ_a is given in the following equation.²⁾

$$\epsilon_a = \frac{m}{M} \frac{(1 - e^{-T})^2 x_m^2}{2(x_m - x_o)} \left(\frac{\rho_P - \rho_L}{\rho_P} \right) \quad (5)$$

Fig. 1 Schematic diagram of unbalance caused by centrifugal sedimentation

where

m [kg] is the weight of the particles in the cell,

M [kg] is the weight of the rotating system.

In the similar way, assuming that the particles located between x_o and x_i at $t = 0$ do not reach the bottom of the cell by $t = t$, the displacement of the center of gravity caused by these particles is given in the following equation.

$$\epsilon_b = \frac{m}{M} \frac{(e^T - 1)(x_m^2 e^{-2T} - x_o^2)}{2(x_m - x_o)} \left(\frac{\rho_P - \rho_L}{\rho_P} \right) \quad (6)$$

The total displacement of the center of gravity ϵ is obtained as the sum of ϵ_a and ϵ_b from Eqs. (5) and (6).

$$\epsilon = \epsilon_a + \epsilon_b = \frac{m}{M} \left(\frac{\rho_P - \rho_L}{\rho_P} \right) \frac{(e^T - 1)(x_m^2 e^{-T} - x_o^2)}{2(x_m - x_o)} \quad (7)$$

Therefore the magnitude of unbalance $U = \epsilon M$ is

$$U(T) = m \left(\frac{\rho_P - \rho_L}{\rho_P} \right) \frac{(e^T - 1)(x_m^2 e^{-T} - x_o^2)}{2(x_m - x_o)} \quad (8)$$

From Eq. (8), the magnitude of unbalance caused by the monodispersed particles with a radius R and a total weight m is expressed in the following form.

$$U(t) = A(r, t)m \quad (9)$$

On the linear acceleration of the rotor speed, the coefficient A is calculated as follows.

$$A(r, t) = \left(\frac{\rho_P - \rho_L}{\rho_P} \right) \frac{(e^\tau - 1)(x_m^2 e^{-\tau} - x_o^2)}{2(x_m - x_o)} \quad (10)$$

In case that the sample contains the particles of different sizes, Eq. (9) will change to a series of equations.

$$U(t) = \sum_{i=1}^n m_i \cdot A(r_i, t) \quad (11)$$

Equation (11) is expressed in the following matrix equation for the measuring time from t_1 to t_m .

$$\begin{pmatrix} A(r_1, t_1) & A(r_2, t_1) & \dots & A(r_n, t_1) \\ A(r_1, t_2) & A(r_2, t_2) & \dots & A(r_n, t_2) \\ \vdots & \vdots & \ddots & \vdots \\ A(r_1, t_m) & A(r_2, t_m) & \dots & A(r_n, t_m) \end{pmatrix} \begin{pmatrix} m_1 \\ m_2 \\ \vdots \\ m_n \end{pmatrix} = \begin{pmatrix} U(t_1) \\ U(t_2) \\ \vdots \\ U(t_m) \end{pmatrix} \quad (12)$$

In this way, when the magnitude of unbalance at each measuring time $t = t_1, t_2, \dots, t_m$ and each corresponding element of the coefficient matrix are known, the masses $m_1, m_2, \dots, m_n$ of the particles having each size are calculated, which gives the size distribution on the mass basis.

The calculation of the inverse matrix of Eq. (12) was carried out by the method of conjugate gradients which is suitable for the rapid computation with small capacity of memories.

This method has the following three advantages.

1) The measurement does not require the full time for the particles concerned to reach the detecting zone because the analysis of this measurement is carried out by monitoring the unbalance caused from the settling particles as well as completely settled ones in the whole suspension.

This analysis makes the time to obtain the

size distribution shorter than other centrifugal methods. However, to assure the precision of the calculation, there is a limitation in the shortening of the measuring time.

2) In many cases, size measurements such as the electric resistance, light scattering and photosedimentmeter give unreasonable results to evaluate the performance of the classifier or milling machines.

For instance, the size distribution of the raw material calculated from those of the classified coarse powder and the fine product does not always agree with that of direct measurement.

But the measurements such as a pipette method and sedimentation balance produce comparatively less difference between the direct measurement of the mixture and the calculation from the components.

Since this method detects the changes in the local concentration of the particles converted to the unbalance related to the weight, it tends to give the data coincident with those by the gravitational sedimentation methods mentioned above.

So, this method is well suited to evaluate the process handling the fine powders, especially submicron powders.

3) The photosedimentmeter is affected by the concentration of the powders even using dilute suspension and cannot be applied on the concentrated conditions.

The measurement of this method can be carried out at the higher concentration, which increases the S/N ratio.

We usually use the 3 wt% suspension for the measurements.

Even if the concentration is over 50 wt% as an extreme condition, the unbalance is still measurable but reliable size distribution cannot be obtained because of the interactions of particles in the suspension.

3. Apparatus

Photograph 1 shows the appearance of this apparatus. The rotor is made of aluminum casting with 300 mm diameter and equipped with two cells set symmetrically to the axis. The one is filled with the sample suspension and the other with the liquid of the same weight as the sample for balancing of the rotor. Each cell has 36 mm inner diameter with 50 mm depth and a maxi-

Photo. 1 Sedimenputer SPT-C

mum volume of 50 ml. A mechanical counter-balance with a screw to adjust its center of gravity is applicable in place of the cell filled with the liquid.

The measuring time required varies 5 to 45 minutes depending upon the particle diameter to be measured.

The apparatus is weighted with the rigid frames to avoid the resonance of the apparatus in the operation.

Figure 2 shows the schematic diagram of the measurement system. The details of the detecting part of the unbalance is shown in **Fig. 3**. The dynamic unbalance of the rotor produced by the centrifugal sedimentation of the particles exerts the centrifugal force F on the rotating shaft. The centrifugal force is converted to the amplitude χ of the vibration through the leaf springs with a spring constant k supporting the pillow block. The slight vibration of the

Fig. 3 Block diagram of the measuring unit

pillow block is detected with a pickup sensor of condenser type suitable to measure the minute amplitude and converted to the analog signals.

The rotor speed is counted with a non-contact magnetic proximity switch and converted to both the digital and the analog signals corresponding to the angular velocity ω in the measuring circuit.

In the measuring circuit, the magnitude of unbalance U is obtained from the relation de-

Fig. 2 Measurement system

scribed in the following equation with the data of the amplitude χ and the angular velocity ω and transferred to the micro computer.

$$U = \left(\frac{k}{\cos \omega t} \right) \left(\frac{\chi}{\omega^2} \right) \quad (13)$$

This apparatus which works at the rotor speed far below the natural frequency corresponds to the balancing machine of so-called "hard type" from the functional viewpoint.

The microcomputer connected with the measuring part stores the transferred data of unbalance and the measuring time counted with a built-in timer and works out the size distribution from the stored data after the measurement. It also sends the signals to the driving circuit to start and stop the rotor and to regulate the rotation.

The computer applied for the size analysis was an 8-bit microcomputer made by NEC. PASCAL compiler was used to increase the computing speed.

The driving circuit has the function to control the rotation speed of the AC motor so that the rotor acquires the preset rotation speed in a certain time.

The measuring range of the applied tachometer was 400 to 8000 r.p.m. and the vibration pickup had the minimum reading $0.5 \mu\text{m}$. The data of the magnitude of unbalance was processed with the computer through 12 bit A/D converter.

4. Results

Figure 4 shows the result of the measurement by this method at a constant speed of 2500 r.p.m. compared with those by other principles. Among the sedimentation methods applied for the liquid phase, the sedimentation balance, the X-ray extinction, the pipette and this unbalance method gave the similar results, while the photosedimentmeter presented the coarser size distribution. The results by the electric resistance method and the ultramicroscope were located between the former group of the sedimentation methods and the latter one.

This tendency of the size data obtained by various methods is not only for talc (Fig. 4) but also for CaCO_3 (Fig. 5). Therefore, it is supposed that the tendency was attributed to

Fig. 4 Size distributions measured by different methods

Fig. 5 Comparison with other measurements

the features of each method.

Figure 6 shows the size distributions of a finer and a coarser ground powder and a mixture of the same amount of them determined with the new apparatus. The dashed line was obtained by the calculation from the data of both ground powders.

If the measurement has no errors, the size of the mixed powder should be in complete agreement with the calculated result using the data of the raw materials before mixing.

However the actual measurement showed a slight difference in the finer range below $0.2 \mu\text{m}$ and the coarser part above $1 \mu\text{m}$ of the mixed powders.

This result implies some errors occur in the both sides, of the range. The cause of the errors has not been studied yet.

Fig. 6 Measured and calculated size distribution of a mixture of ground powders

An example of the variation of the rotor speed and the magnitude of unbalance in the case of the linear acceleration followed by the constant speed is shown in Fig. 7. Since the resolution of the sensor is critical during the initial period of low rotation speed as seen from the figure, the size analysis was conducted based on the data of unbalance above 1000 r.p.m. to minimize the disturbance resulted from the noises. In Fig. 7 the unbalances of the rotor in the axial and vertical directions of the cell shown in the x- and y-direction respectively. The constant unbalance in the y-direction except the initial period of rotation implies that the rotation of the rotor is very stable. The variation of the unbalance in the x-direction with time corresponds to the sedimentation curve to be obtained with the sedimentation balance and is used as the data for the calculation of the size distribution.

The comparison of the results obtained by this method of the linear acceleration followed by a constant rotation speed with those at a constant speed only is shown in Fig. 8. Both results agree well each other taking account of the experimental errors.

Figure 9 shows the results of the measurement with two samples including the particles finer than $0.1 \mu\text{m}$ which was carried out for the purpose of investigation of the lower limit size for this method. From the electron microscopic picture of BaSO_4 in Photo. 2 used as the sample, it is recognized that this new apparatus applying the unbalance method is capable of determining the particle size under $0.1 \mu\text{m}$.

Fig. 7 Variation of rotor revolution and unbalance with time

Fig. 8 Comparison of the measurement at a constant rotation with that of the linear acceleration

Fig. 9 Examples of the measurement in the submicron range

Photo. 2 SEM photograph of BaSO₄

5. Conclusion

A new size analyzer based on the unbalance method was constructed on the experimental basis. The apparatus applying the principle of the balancing machines detects the magnitude of unbalance caused by the displacement of the center of gravity owing to the centrifugal sedimentation of the particles which gives the size distribution theoretically.

It was confirmed that this apparatus presented the similar results of the size distribution to other sedimentation methods such as the X-ray extinction, the sedimentation balance and so forth.

The results by this method with the linear acceleration followed by a constant rotation coincides well with those at a constant speed only, taking the experimental errors into consideration.

Additionally, it was found in comparison with the microscopic picture that this analyzer is able to be used for the size analysis less than 0.1 μm down to nearly 0.03 μm.

Nomenclature

$A(r, t)$	: coefficient of the unbalance	[m]
D_p	: particle diameter	[m]
K	: constant for a given measurement condition	[s/m ²]
k	: spring constant	[N/m]
M	: weight of the rotating system	[kg]
m	: weight of the particles in the cell	[kg]
m_i	: weight of the particles with radius r_i	[kg]
r	: particle radius	[m]
r_i	: radius of the particles concerned	[m]
T	: non-dimensional time	[—]
$U(t)$	: magnitude of unbalance	[kg·m]
x	: position in the radial direction	[m]
x_m	: position of the cell bottom	[m]
x_o	: position of the surface of suspension at $t = 0$	[m]
α	: constant for the rotor acceleration	[1/s ²]
$\epsilon, \epsilon_a, \epsilon_b$	: displacement of the center of gravity	[m]
μ_L	: viscosity of dispersion liquid	[Pa·s]
ρ_L	: density of dispersion liquid	[kg/m ³]
ρ_P	: density of particles	[kg/m ³]
τ	: non-dimensional time	[—]
χ	: amplitude of vibration	[m]

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