A New Particle Size Distribution Apparatus based on Unbalance by Centrifugal Sedimentation

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Abstract

A new apparatus for the measurement of particle size distribution based on the unbalance caused by centrifugal sedimentation was made on an experimental bases. The size distributions obtained by this method with a constant revolution of the rotor were compared with those measured by other methods using the same sample.

Furthermore, in order to shorten the measuring time and expand the measuring size range, some additional attempts were conducted with a linear acceleration followed by a constant revolution of the rotor. They gave similar results of size distribution to the measurements at a constant revolution.

1. Introduction

The principle of sedimentation of particulate materials in a liquid phase is used most widely and reliably among various principles for the measurement of particle size distribution. One of the most popular methods based on this principle adapts the opacity with the particles settling by a centrifugal force. This method has two disadvantages. One of them is the difficulties caused by the fact that the size of particles in the submicron range becomes the same order to the wave length of light. The other is that the optical extinction coefficient varies with the particle size and the material.

A new method based on the displacement of the center of gravity caused by particle settling was proposed to overcome the disadvantages of light extinction methods1).

In this report, we first introduce a test apparatus that was made to measure size distribution applying this method and combining a personal computer for immediate analysis. Then we show some examples of the measurement using this instrument. In order to expand the range of measuring size it was examined to increase rotor speed of the centrifuge with the measurement time.

2. Measuring principle and procedures

Pouring homogeneous suspension including a sample powder into a cell and setting it in a rotor, the balance of the rotor is arranged before measuring. The rotor being set in motion, the particles settle in the centrifugal field and the center of gravity of the cell is shifted from its initial position.

A sedimentation curve in terms of the unbalance can be obtained by the instrument which detects the deviation of the center of gravity caused by particle settling.
gravity making use of a balancing machine.

The equation of motion for centrifugal sedimentation is written with an angular velocity \( \omega \) and a particle radius \( r \) as follows.

\[
\frac{dx}{dt} = \frac{2}{9} \left( \frac{\rho_p - \rho_L}{\mu_L} \right) r^2 \omega^2 x
\]

where \( \rho_p \): density of particles \([\text{g/cm}^3]\)
\( \rho_L \): density of dispersion medium \([\text{g/cm}^3]\)
\( \mu_L \): viscosity of dispersion medium \([\text{g/cm-sec}]\)

\( x \): distance in the radial direction \([\text{cm}]\)

Defining \( K \) as the following,

\[
K = \frac{2}{9} \left( \frac{\rho_p - \rho_L}{\mu_L} \right)
\]

Substituting Eq. (2) for (1), and applying the boundary condition \( x = x_i \) at \( t = 0 \), we obtain

\[
ln \frac{x}{x_i} = Kr^2 \omega^2 t
\]

or

\[
x = x_i e^{Kr^2 \omega^2 t}
\]

For the displacement of the center of gravity caused by the particles which are located between \( x_i \) and \( x_m \) at \( t=0 \) and settle out completely by \( t=t \).

The displacements defined in above a) and b) are shown with \( E_a \) and \( E_b \), respectively.

Mass of particles \( m_a \) positioned between \( x_i \) and \( x_m \) is given:

\[
m_a = \frac{x_m - x_i}{x_m - x_0} m
\]

where \( m \) is the mass of particles in the cell. Letting the initial eccentric distance be \( e_0 \) and the one after the displacement of the center of gravity be \( e_1 \), the balancing moment takes the following forms in each case,

\[
e_0 M = m_a \left( \frac{x_i + x_m}{2} \right) \left( \frac{\rho_p - \rho_L}{\rho_p} \right)
\]

\[
e_1 M = m_a x_m \left( \frac{\rho_p - \rho_L}{\rho_p} \right)
\]

where \( M \) is the total mass of the rotating system. Combining Eq. (6) and Eq. (7),

\[
e_a = e_1 - e_0 = \frac{m_a}{M} \left( \frac{x_m - x_i}{2} \right) \left( \frac{\rho_p - \rho_L}{\rho_p} \right)
\]

For particles settling from \( x_i \) to \( x_m \) with time \( t \)

\[
x_m = x_i e^{Kr^2 \omega^2 t}
\]

Reforming the above,

\[
x_m - x_i = (1 - e^{-Kr^2 \omega^2 t}) x_m
\]

Substituting Eq. (10) for Eq. (5),

\[
m_a = \frac{\left(1 - e^{-Kr^2 \omega^2 t}\right) x_m}{x_m - x_0} m
\]

From Eqs. (8) and (11), we obtain

\[
e_a = \frac{m}{M} \left(1 - e^{-Kr^2 \omega^2 t}\right) x_m \frac{2}{x_m - x_0} \left( \frac{\rho_p - \rho_L}{\rho_p} \right)
\]

On the other hand, the balance moment for the mass of particles \( m_b \) positioned between \( x_0 \) and \( x_i \) at \( t=0 \) gives:

\[
e_0 M = m_b \left( \frac{x_i + x_0}{2} \right) \left( \frac{\rho_p - \rho_L}{\rho_p} \right)
\]
where \( e_0', e_1' \) are the initial eccentric distance and the one after the displacement of the center of gravity by sedimentation respectively and \( x_0' \) is the interface position between the particle suspension and the supernatant liquid containing no particles.

Similar to Eq. (4), we obtain the following form for \( x_0' \)

\[
x_0' = x_0 e^{Kr^2\omega^2 t}
\]  

(15)

And from this relation

\[
e_b = e_1 - e_0 = \frac{m_b \left( x_m + x_0 e^{Kr^2\omega^2 t} - x_i - x_0 \right)}{M} \times \left( \frac{\rho_p - \rho_L}{\rho_p} \right)
\]  

(16)

where \( m_b \) is given by the proportional allotment of \( m \),

\[
m_b = \frac{x_i - x_0}{x_m - x_0} m
\]  

(17)

Substituting Eqs. (9) and (17) for Eq. (16), simplification gives

\[
e_b = \frac{m \left( \frac{\rho_p - \rho_L}{\rho_p} \right)}{M} \times \left( e^{Kr^2\omega^2 t} - 1 \right) \left( x_m^2 e^{-Kr^2\omega^2 t} - x_0^2 \right)
\]  

(18)

Total eccentric displacement \( e \) for the rotor is the sum of \( e_a \) and \( e_b \) and given from Eq. (12) and Eq. (18),

\[
e = m \left( \frac{\rho_p - \rho_L}{\rho_p} \right) \times \left( e^{Kr^2\omega^2 t} - 1 \right) \left( x_m^2 e^{-Kr^2\omega^2 t} - x_0^2 \right)
\]  

(19)

In the balancing test, centrifugal force \( P \) located on the rotor is expressed in general

\[
P = \omega^2 \cdot Me
\]  

(20)

where \( e \) is eccentric distance. The value of \( Me \) is determined by the centrifugal force \( P \) with a known \( \omega \) and thus we defined an unbalance \( U \) as follows,

\[
U = Me
\]  

(21)

Now assuming that the unbalanced state of the actual rotor can be realized being replaced by a completely balanced rotor with attaching a weight having mass of \( m \) at a radius \( R \) as illustrated in Fig. 2.

\[
e = \frac{mR}{M}
\]  

(22)

The unbalance of the measuring rotor is obtained from Eq. (19) with setting \( e = e \),

\[
U(t) = m \left( \frac{\rho_p - \rho_L}{\rho_p} \right) \times \left( e^{Kr^2\omega^2 t} - 1 \right) \left( x_m^2 e^{-Kr^2\omega^2 t} - x_0^2 \right)
\]  

(23)

Letting \( \tau_c = Kr^2 \omega^2 t \), Eq. (23) is reformed

\[
U(t) = m \left( \frac{\rho_p - \rho_L}{\rho_p} \right) \left( e^{\tau_c} - 1 \right) \left( x_m^2 e^{-\tau_c} - x_0^2 \right)
\]  

(23)'
In the period of constant rotation after $t = T_c$, the total measuring time is the sum of $T_c$ and additional time $t_e$ with constant rotation.

$$t = T_c + t_e$$

(29)

The unbalance $U$ is given

$$U(t) = U(T_c) + U(t_e)$$

(30)

The first time on the right hand side $U(T_c)$ is derived from Eq. (28)

$$U(T_c) = m \left( \frac{\rho_P - \rho_L}{\rho_P} \right) \times \frac{(e^{\tau(T_c)} - 1)(x_m^2 e^{-\tau(T_c)} - x_0^2)}{2(x_m - x_0)}$$

(31)

where $\tau(T_c) = K r^2 \left( \frac{a^2}{3} T_c^3 + a \omega_0 T_c^2 + \omega_0^2 T_c \right)$

(32)

The second term $U(t_e)$ is calculated in the same way,

$$U(t_e) = m_c \left( \frac{\rho_P - \rho_L}{\rho_P} \right) \times \frac{(e^{\tau(t_e)} - 1)(x_m^2 e^{-\tau(t_e)} - x_0^2)}{2(x_m - x_0)}$$

(33)

where $x_0'$ is the position of the interface between the supernatant part containing no-particles and the turbid part with suspended particles in the cell at $t = T_c$ and $m_c$ is the mass of particles suspended.

When the rotor revolution reaches a constant condition, $x_0'$ and $m_c$ are given by the following equations,

$$x_0' = x_0 e^{\tau(T_c)}$$

(34)

$$m_c = \frac{x_1' - x_0}{x_m - x_0} m$$

(35)

where $x_1'$ is the position of particles at $t = 0$ which will sediment completely at $t = T_c$, and so it can be shown that

$$x_1' = x_m e^{-\tau(T_c)}$$

(36)

Substituting Eq. (36) for Eq. (35), $m_c$ is given

$$m_c = \frac{x_m e^{-\tau(T_c)} - x_0}{x_m - x_0} m$$

(37)

$U(t_e)$ is derived from Eqs. (33) and (37).

$$U(t_e) = \frac{x_m e^{-\tau(T_c)} - x_0}{x_m - x_0} m \left( \frac{\rho_P - \rho_L}{\rho_P} \right) \times \frac{(e^{\tau(t_e)} - 1)(x_m^2 e^{-\tau(t_e)} - x_0^2)}{2(x_m - x_0 e^{\tau(T_c)})}$$

(38)

where $\tau_e(t_e)$ is given as

$$\tau_e(t_e) = K r^2 \omega_c^2 t_c$$

(39)

and $\omega_c$ is given as

$$\omega_c = \omega_0 + a T_c$$

(39')

From Eqs. (23), (28) and (38), we obtain the following simple equation as a general form showing the relation of the unbalance $U(t)$, coefficient $A$ and mass of particles $m$ for particles having uniform diameter,

$$U(t) = mA (r, t)$$

(40)

The unbalance $U(t_i)$ is therefore calculated as a sum of products of the mass $m_i$ and the coefficient $A(r_j, t_i)$ of the particles having radius $r_j$ at $t = t_i$.

$$U(t_i) = \Sigma m_j \cdot A(r_j, t_i)$$

(41)

The variation of unbalance with time is expressed by the following matrix based on Eq. (41).

$$\begin{bmatrix} A(r_1, t_1), A(r_2, t_1), \ldots, A(r_n, t_1) \\ A(r_1, t_2), A(r_2, t_2), \ldots, A(r_n, t_2) \\ \vdots \\ A(r_1, t_m), A(r_2, t_m), \ldots, A(r_n, t_m) \end{bmatrix} \begin{bmatrix} m_1 \\ m_2 \\ \vdots \\ m_n \end{bmatrix} = \begin{bmatrix} U(t_1) \\ U(t_2) \\ \vdots \\ U(t_m) \end{bmatrix}$$

(42)

From the above equation, one can get the mass
3. Measurement apparatus

The system developed to determine particle size distributions is shown in Fig. 4. We use a dynamic balancing machine type FH-214G prepared by Akashi Seisakusho, Ltd. reformed for the attachment of the rotor and the cell shown in Fig. 5.

The block diagram of the unbalance detecting unit is shown in Fig. 6. The balancing machine like this "hard" type has condenser pick-ups which detects the amplitude of vibration of the bearing supported by springs having constant $k$. The vibration is produced by the rotor unbalance caused by the centrifugal sedimentation of particles. The tachometer detects the rotor speed and the signal of the rotation is transferred to the measuring circuit, which makes analog processing of the unbalance with the vibration amplitude $\chi$, spring constant $k$ and angular velocity $\omega$ and convert it in the forms of voltage.

The personal computer receives the real-time signal converted to BCD coded data by the digital voltmeter through the isolated parallel interface. The computer saves the data inputs of the unbalance and corresponding time to a flexible disk unit.

After the measurement, the computer processes the data saved in the disk with the execution of the analyzing program and transmit the size distribution to an X-Y plotter or a printer. The computer we used is NEC PC-9801 personal computer with 16 bit CPU and numerical co-processing LSI. On the measurement, the computer is taking the data and simultaneously sends the control signal through the D/A converter to regulate the rotor revolution. The frequency converter is Toshiba TOSVERT-130 and controls the rotation according to the computer instruction. The resolution of the balancing machine used for this work is $0.5 \mu m$ and $8 mg$ converted in terms of weight.

Concentration of the suspension was set at $3 \text{ wt}\%$ as usually applied for gravitational sedimentation method. The suspension of about $30 ml$ was weighed accurately and used for the measurement. After adjustment of the initial balance of the rotor is performed, the computer conducts the measurement automatically.

4. Results

The measurements were performed on the following three conditions.
- Constant rotation of $1300 \text{ r.p.m.}$
- Constant rotation of $2500 \text{ r.p.m.}$
Linear acceleration and constant rotation of 2500 r.p.m. shown in Fig. 3. The last condition in the above-mentioned was planned to shorten the measuring time and to expand the measuring size range.

Figure 7 shows an example of sedimentation curve of the unbalance with a constant rotation. The horizontal axis presents the measuring time and the vertical axis indicates the relative unbalance from the base in the figure. The unbalance has a unit [g-mm] as seen from Eq. (21).

Figure 8 indicates the results of four measurements for the same sample on the condition of constant rotation to examine the reappearance. The measurements give small fluctuations of the results in the finer size range. The results are shown in Fig. 9, compared with other methods, such as sedimentation balance, electric resistance and photo extinction. The figure indicates that sedimentation balance gave the finest, having good agreement with this method, while photo extinction tended to produce a coarser result and electric resistance method had the intermediate tendency. The comparison of the methods in case of CaCO₃ gave the same feature. From these results, it is assumed that the tendency in size distribution depends on the measuring method itself.

The examples of the results in the cases of linear acceleration and constant revolution are shown in Figs. 10 and 11. The sample shown in Fig. 10 had smaller number of the coarser particles than other samples and was measured on the condition of constant rotation of 2500 r.p.m. and linear acceleration from 1300 to 2500 r.p.m. for the comparison.

Figure 11 shows the results of Kanto loam powder with mean diameter of about 2 μm, the maximum diameter of 8 μm and no finer fraction of the particle less than 0.2 μm. Since there was no suitable talc samples available to
measure in the coarser particle size range, Kanto loam was employed as substitute material to compare with. The rotation speed was set at 1300 r.p.m. in this case, because the Kanto loam was relatively coarse and would have settled out immediately at 2500 r.p.m. Decreasing the rotation produces smaller vibration amplitude and so reduces the resolution of pickup as seen from Fig. 6. To avoid decreasing the resolution and rapid settling of particles, the measuring rotation was set at 1300 r.p.m. There was a little difference in the results between the constant rotation and the linear acceleration. It may come from the error in the determination of a correcting values. The signal band detected by the pickup varied with rotor revolution and therefore would require blank tests and correction of the directly obtained values. We will examine the correction more exactly.

5. Conclusion

A new method for particle size distribution measurement making use of a dynamic balancing machine was introduced and a test apparatus was made based on this method. It has advantages of the direct relation with the displacement of the center of gravity caused by the particle settling and no connection with properties depending on the materials such as the extinction coefficient in the case of photo extinction. The other advantage of the measurement is comparatively short time required to determine the size distribution. In this instrument, as the particle settling in any part of the suspension causes the deviation of the center of gravity, the unbalance data can be obtained without reaching specified distance for measuring.

The results of measurement by the method were similar to those by other methods and showed specially good agreement with sedimentation balance. Additionally, in order to shorten the measuring time and to expand the measuring size range, a new approach to the measurement was examined combining a linear acceleration with a following constant rotation using the same instrument. The results of the approach gave a little finer but approximately the same as those of constant rotation.

We are planning to make further developments on the measurement and improve the apparatus in order to complete a simple and precise instrument for the measurement of particle size distribution in the future.

Nomenclature

\[ A(r, t) : \text{coefficient of the matrix} \quad [\text{mm}] \]
\[ a : \text{constant acceleration of the rotor speed} \quad [1/\text{sec}^2] \]
\[ D_p : \text{particle diameter} \quad [\text{cm}] \]
\[ e : \text{eccentric distance} \quad [\text{cm}] \]
\[ e_0, e_0' : \text{initial eccentric distance} \quad [\text{cm}] \]
\[ e_1, e_1' : \text{eccentric distance after sedimentation} \quad [\text{cm}] \]
\[ K : \text{constant for a given measurement} \quad [\text{sec/cm}^2] \]
\[ k : \text{spring constant} \quad [\text{dyne/cm}] \]
\[ P : \text{centrifugal force} \quad [\text{dyne}] \]
\[ R : \text{equivalent radius of the rotor} \quad [\text{cm}] \]
\[ r : \text{particle radius} \quad [\text{cm}] \]
\[ r_i : \text{radius of a concerned particle} \quad [\text{cm}] \]
\[ T_c : \text{interval of constant acceleration} \quad [\text{sec}] \]
\[ t, \tilde{t} : \text{time} \quad [\text{sec}] \]
\[ t_c : \text{time of constant revolution} \quad [\text{sec}] \]
\[ U(t) : \text{unbalance} \quad [\text{g-mm}] \]
\[ M : \text{total mass of the rotating system} \quad [\text{g}] \]
\[ m : \text{mass of particles in the cell} \quad [\text{g}] \]
\[ m_a : \text{mass of particles located between} \]
\[ x_1 \text{ and } x_m \text{ at } t = 0 \quad [\text{g}] \]
\[ m_b : \text{mass of particles located between} \]
\[ x_o \text{ and } x_1 \text{ at } t = 0 \quad [\text{g}] \]
\[ m_c : \text{mass of particles suspended at } t = T_c \quad [\text{g}] \]
\[ x : \text{position in the radial direction} \quad [\text{cm}] \]
\[ x_i : \text{position of the particles to reach} \]
\[ \text{the cell bottom at } t = \tilde{t} \quad [\text{cm}] \]
\[ x_m : \text{position of the cell bottom} \quad [\text{cm}] \]
\[ x_o : \text{position of the surface of suspension} \quad [\text{cm}] \]
\[ x_c : \text{position of the boundary between} \]
\[ \text{the supernatant and the suspension} \quad [\text{cm}] \]
\[ e_a, e_b : \text{displacement of the center of gravity} \quad [\text{cm}] \]
| Symbol | Description | Unit |
|-------|-------------|------|
| \( \mu_c \) | viscosity of dispersion medium | [g/cm sec] |
| \( \rho_L \) | density of dispersion medium | [g/cm³] |
| \( \rho_P \) | density of particles | [g/cm³] |
| \( \omega \) | angular speed | [1/sec] |
| \( \omega_0 \) | initial angular speed | [1/sec] |
| \( \omega_c \) | constant angular speed | [1/sec] |
| \( \tau_c, \tau_l \) | non-dimensional variables | [−] |
| \( x \) | amplitude of vibration | [cm] |

**References**

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