Comparative Data of Particle Size Distribution on Flake Like Particles by Various Methods†

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Abstract

The results of the particle size measurements by several methods based on different principles are almost equal to each other as far as these methods are applicable, when the powder is considered to be made of sphere shape particles. However, there are some questions about the particle size measurement of irregular shape particles such as plate or needle like powder. Using mica powders as samples of the thin plate like particles, we compared the results of particle size measurements based on six different principles; optical microscope, sieve, sedimentation balance, photo sedimentation, Coulter counter and Microtrac. The measurement of the particle thickness by the mono-particulate film method gave a relation between the thickness and the projected diameter of mica particles classified by microsieves with a high accuracy. From this relation, the volume of the plate like mica particle was obtained and then the sphere equivalent diameter was calculated. We compared the particle size distribution based on this sphere equivalent diameter with the results measured by the other methods.

1. Introduction

It is generally accepted that a knowledge of the particle size is the most essential in powder processing. For particle size measurements, there have been a lot of publications1), reviews, and reports, and various kinds of measuring methods and equipments have been successively developed in these days.

It is unfortunate, however, that the effect of sensing principles of the equipments on measured results has been little studied. This arises from the difficulty of obtaining a suitable expression of the particle size; for spherical or near-spherical shaped material, the size of it may be in good agreement, but for irregular material with lammellar, flaky, or acicular shape, comparison of data obtained by different equipments has been little carried out.

Computer-aided equipments which can output resultant data through printer have been widely used for particle size analysis. For these equipments, constants concerning some of the particle properties such as particle shape contained in the theoretical equations are relative values which are characterized by standard material; however there has been no suitable procedure developed for particles with irregular shape.

The aim of this study is to compare the particle size distributions which are obtained by several methods having different sensing principle by using a mica powder that is easy to estimate its shape irregularity.

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2. Experimental

2.1 Sample

The material used for this work was a ground product of refined Canadian phlogopite together with synthetic mica in part, which had been separated into less than 150 μm in advance. These particles were separated by standard sieves and precise sieves (Micro sieve) to be used as samples. Typical photomicrographs of them are shown in Fig. 1, in which the particle shape may be regarded as a hexagonal platelet similar to a crystal habit of mica.

In addition, ground calcium carbonate and colundum were used as standard particles for comparison since their shape might be considered to be almost spherical.

2.2 Particle size analysis

The methods for determining particle size used in this work are as follows:

Sieve

A set of JIS testing sieve with woven cloth was used to separate particles larger than 105 μm. For particles smaller than 105 μm, Micro sieve manufactured by electroforming method was employed with an opening of 63, 45, 32, 20, 10, and 5 μm.

Optical microscope

Since a flaky powder like mica tends to flatly spread over a slide glass, its two-dimensional image can be obtained by optical microscope. In this work, we determined Feret’s diameter which is one of the unidirectional particle size.

In addition, the individual dimensions such as length L and breadth W of 200 particles selected randomly from the separated fractions were measured to obtain the relation between elongation ratio L/W and particle size.

Sedimentation balance

For this method, a conventional procedure was employed with sodium hexametaphosphate solution of 0.2% in concentration. This method offers a diameter of sphere which falls in the medium with the same density and velocity as the particle in question. The diameter obtained in this way is well known as Stokes’ diameter and its distribution is based on particle weight.

Photosedimentation

This method also adopts the same medium as that for the sedimentation balance, providing Stokes’ diameter of particles. However, the particle size is dependent upon areas of the projected shadow because this method is based on the intersection of the light beam which passes perpendicularly to the settling direction of the particles in suspension.

Coulter counter

This method measures size distribution of particles suspended in an electrolyte by the presence of a fine particle in a little orifice placed between two electrodes. This size distribution is based on particle volumes because the particle size is given as a diameter of the sphere which has the same volume as that of the particle in question. The apparatus used in
this study was Coulter counter, Model TA II, which includes a microprocessor that outputs data of the particle size distribution through X-Y recorder.

**Microtrac**

As is well known, Fraunhofer diffraction patterns of particles take place when the monitoring zone in suspension is projected by a laser beam which is transmitted perpendicularly to the stream direction. The particle size distribution, which is based on volume of equivalent sphere, is obtainable by use of the principle that an angle distribution of its diffraction intensity is a function of particle sizes. This analyzer presents calculated results through a printer with the aid of microprocessor.

**Monoparticulate film method**

As reported in the literature, this method measures an average thickness of particles which are hydrophobicized and dispersed so that a monoparticulate film may be formed on the water. It provides a dimension which is dependent upon the particle shape; for near-

![Graph](https://example.com/graph.png)

Fig. 3 Relation between the particle size and the aspect ratio (Symbols show the types of crusher.)

spherical or near-cubic materials an average size is obtainable, and for flaky-shaped materials like mica an average thickness. The method of microscope or sieve offers a plane size of particles, so the degree of relative thickness can be given by use of the ratio of plane size to thickness, namely the aspect ratio.

The detail description of the individual equipments mentioned above is to be referred in the literatures.

3 Results and discussion

3.1 Particle shape

Figure 2 shows typical distributions of the elongation ratio \( L/W \) obtained from the photomicrographs of the sample powder which were separated into three fractions. It was found that smaller sizes of such particles might give slenderer figures with the mean value of \( L/W \) ranging from 1.3 to 1.8.

On the other hand, the mean thickness of the sample powder was determined by the monoparticulate film method. This sample powder consisted of a ground product given by three pulverizers with different mechanism and the fractions which were made by Micro sieve. By dividing the cumulative 50% diameter obtained by sieve by the thickness, one can get the aspect ratio. In this procedure, \( \sqrt{2} \) times the sieve opening must be used for the particle size determined by sieve, because a flaky parti-
Particle size distribution of classified mica by photomicrograph

As indicated in Fig. 3, plots of the aspect ratios against the cumulative 50% diameters show the linear relation expressed as

Aspect ratio = 0.36 \( D_{50} \) + 16 \( (1) \)

where \( D_{50} \) represents the cumulative 50% diameter.

These results state that a particle of mica powder may become more slender and relatively thicker in its figure as its particle size decreases.

### 3. 2 Accuracy of Micro sieve separation

The accuracy of Micro sieve separation was evaluated as follows; the mica powder was sieved into three fractions (Sample A of 20 to 32 \( \mu m \), Sample B of 32 to 45 \( \mu m \), and Sample C of 45 to 63 \( \mu m \)). Then we determined Feret's diameters of 1000 particle images selected randomly from the photomicrograph of each fraction.

The particle size distributions obtained in this way are shown in Fig. 4. It is found that these distributions were all regular, covering 20 to 80 percent of the distribution width within the size range of \( \sqrt{2} \) times the sieve opening used. This implies that a flaky particle like mica can pass diagonally through a square sieve opening as pointed out previously, despite having a considerably wide range of the elongation ratio. Thus Micro sieve was found to have sufficiently high accuracy for comparison in this work.

### 3. 3 Accuracy of the monoparticulate film method

To evaluate the accuracy of the monoparticulate film method, specific surface area was determined in two ways. The material used in this work was the ground mica powder having the particle size distribution shown in Fig. 5 and the mean thickness of 2.4 \( \mu m \) determined by the monoparticulate film method.

One of the ways for obtaining specific surface area is to make a particular solid model which may characterize a mica particle having a platelet figure. The cumulative 50% diameter of 40 \( \mu m \), as indicated in Fig. 5, was used as
a mean diameter of the particles, though it might be doubtful whether this assumption was suitable for such a broad particle size distribution or not. Using this diameter, the dimensions of the solid model were calculated as shown in Fig. 5. The breadth was considered to be $\sqrt{2}$ times the mean diameter; the length to be 1.5 times the breadth, judging from Fig. 1. With these values, the whole surface area of $2.96 \times 10^{-4}$ cm$^2$ and the weight of $9.58 \times 10^{-8}$ g of the model particle were calculated, and thus the specific surface area of 2964 cm$^2$/g could be obtained.

The other was given by calculating the volume of particles which were spread over the water to form a monoparticulate film. The area covered with the particles was 1650 cm$^2$/g, with the packing ratio of 0.9. Hence the specific surface area could be calculated to be $(1650 \times 0.9 \times 2) + 110 = 3080$ cm$^2$/g, where the value 110 represents the total of the side areas.

The specific surface areas obtained in these ways were found to have fairly good agreement, and this suggests that the monoparticulate film method could be reliable for estimation of particle shape characteristics.

3.4 Comparison of measured particle size distributions

By use of the procedures described in Section 2.2, particle size distributions of the same sample powder were determined to distinguish the difference in the expressions caused by the sensing principles. The results were compared using a sphere equivalent diameter as the same dimension and physical meaning since the particle size obtained in this work was based on the diameter of an equivalent sphere except the case of optical microscope and sieve.

Photomicrograph

As well known, a Feret diameter of projected two-dimensional micrographic images of particles has the tendency to be consistent with the arithmetic mean diameter of breadth and length when sufficiently large number of particles are counted. The mean value of the diameters may be considered to be little larger than the size which is determined by Micro sieve.

In this work, diameters of standard particles were calculated for the criterion of comparison
Fig. 8 Comparative data on the particle size distribution of mica C (45~63 \( \mu \text{m} \)) obtained by various methods

as follows; the particle volumes of each fraction of the sample A, B, and C were obtained by using the frequency distributions of the Feret diameters with an interval range of 2 to 5 \( \mu \text{m} \) and the thickness calculated from Eq.(1). By considering a sphere which had the same volume as the particle in question, the required diameter was obtained. The particle size distributions of the sample are indicated in Figs. 6 to 8, together with the distributions obtained by the methods mentioned above.

**Sedimentation**

The particle size distributions given by sedimentation balance and photosedimentation were found to have satisfactory agreement with those of the standard particles, but the ranges of the distributions covered were relatively wide. This reason may be considered as follows; small particles in suspension have tendency to fall down in various positions in the Stokes region of particle motion. It is supposed that the falling speed might be maximum when a particle was oriented perpendicularly to the streamline owing to the minimum flow resistance and, in turn, it might become minimum when the particle was oriented horizontally. Therefore the sedimentation method seemingly tends to provide a relatively wide distribution of particle sizes, even if the particles to be measured are actually monodispersed.

It should be noticed that an extraordinarily large number of particles apparently existed in the coarser side in the coarsest sample C. It is possibly because the orientation of the particles remained during the falling period since the settling depth was relatively short and the agitation flow was not negligible. This effect will be more noticeable when a particle was faced perpendicularly to the beam path.

**Coulter counter**

It is generally known that the values measured by Coulter counter may vary according to its aperture size. The result obtained in this work shows that a smaller diameter of the aperture might permit the particle size distribution to approach the standard distribution, as illustrated in Figs. 6 to 8, where the value in \( \mu \text{m} \) unit refers to a diameter of the apertures used.
The following problems should be noticed, though no sufficient discussion has been made in this work; no suitable standard material for calibration has been obtained and the value measured by this method is considerably dependent upon the magnitude of the electrical charge which does not seem to be distributed uniformly on the surface of a mica powder with two-dimensionally crystalline structure.

**Microtrac**

Microtrac permits liquid suspended with particles to flow down fast at the detecting zone which is placed perpendicularly to the path of a laser beam. In this case, flaky particles like mica tend to be oriented in parallel with the streamline or perpendicularly to the beam path, rotating arbitrarily around the streamline. Thus the particle size obtained in this way is larger than the Feret diameter. On the other hand, the size is smaller than the sedimentation diameter since the sedimentation methods allow the particles to settle with random attitude.

The results obtained in this work might support the tendency mentioned above, though it is not always possible because of the difference in sensing principle of the individual particle size measuring method.

As explained above, adequate comparison requires other information than geometrically equivalent diameter of a particle, unless the particle shape is fitted to the set-up condition of the equipment to be employed. This is understandable from the fact that for near-spherical particles like ground calcium carbonate or colundum, a relatively unitary particle size distribution independent of the measuring equipments could be obtained as shown in Fig. 9, except the case of Microtrac which would tend to provide a coarser distribution.

### 4. Conclusion

In this work, a mica powder with flaky shape was used to find the effect of irregular shape on its particle size distribution by use of 6 particle size measuring equipments with different sensing principles. For comparison based on the same dimension and physical meaning, the sample was sieved into several monodispersed particle fractions by precise sieve and the dimensions of the particles were determined by microscope and the monoparticulate film method. The accuracy of these methods was discussed before the particle size distribution of spheres which had the same volumes as those of the particles in question were obtained. By using these sphere equivalent diameters, the results were compared together with the distributions obtained by the other methods; for photosedimentation, the basis of a particle size was changed from area to volume.

The result of this work is that a particle size measuring equipment which is more sensitive to the orientation of irregular shape particle would give more deviation of particle size distribution from that of the standard material. This was ascertained from the comparison with the measured result of near-spherical materials.

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