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# 1

## Particle Size Analysis

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### 1.1 INTRODUCTION

In many powder handling and processing operations particle size and size distribution play a key role in determining the bulk properties of the powder. Describing the size distribution of the particles making up a powder is therefore central in characterizing the powder. In many industrial applications a single number will be required to characterize the particle size of the powder. This can only be done accurately and easily with a mono-sized distribution of spheres or cubes. Real particles with shapes that require more than one dimension to fully describe them and real powders with particles in a range of sizes, mean that in practice the identification of single number to adequately describe the size of the particles is far from straightforward. This chapter deals with how this is done.

### 1.2 DESCRIBING THE SIZE OF A SINGLE PARTICLE

Regular-shaped particles can be accurately described by giving the shape and a number of dimensions. Examples are given in Table 1.1.

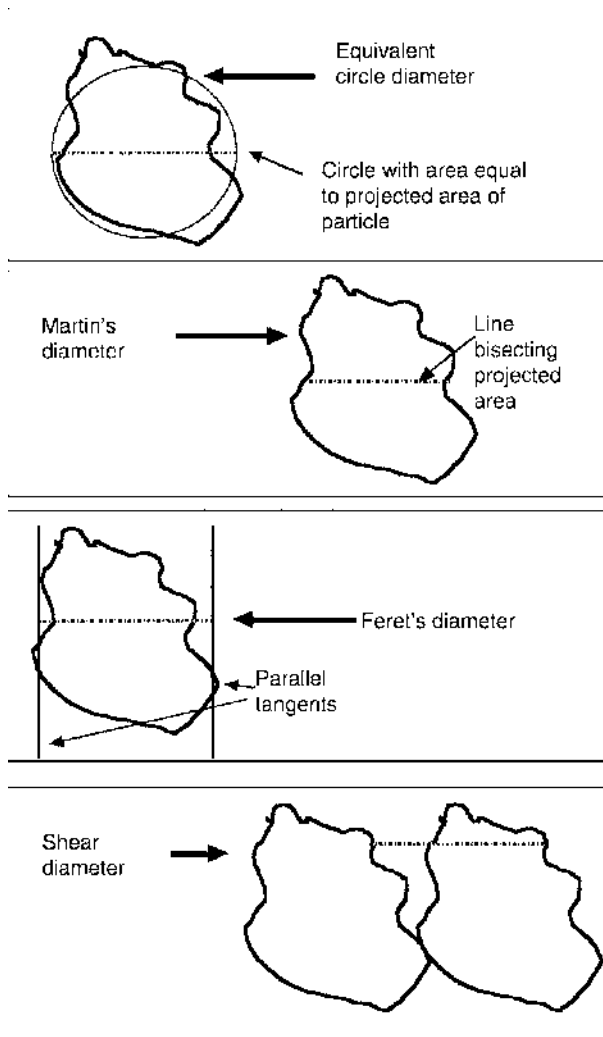
The description of the shapes of irregular-shaped particles is a branch of science in itself and will not be covered in detail here. Readers wishing to know more on this topic are referred to Hawkins (1993). However, it will be clear to the reader that no single physical dimension can adequately describe the size of an irregularly shaped particle, just as a single dimension cannot describe the shape of a cylinder, a cuboid or a cone. Which dimension we do use will in practice depend on (a) what property or dimension of the particle we are able to measure and (b) the use to which the dimension is to be put.

If we are using a microscope, perhaps coupled with an image analyser, to view the particles and measure their size, we are looking at a projection of the shape of the particles. Some common diameters used in microscope analysis are statistical diameters such as Martin's diameter (length of the line which bisects the particle

**Table 1.1** Regular-shaped particles

Shape	Sphere	Cube	Cylinder	Cuboid	Cone
Dimensions	Radius	Side length	Radius and height	Three side lengths	Radius and height

image), Feret's diameter (distance between two tangents on opposite sides of the particle) and shear diameter (particle width obtained using an image shearing device) and equivalent circle diameters such as the projected area diameter (area of circle with same area as the projected area of the particle resting in a stable position). Some of these diameters are described in Figure 1.1. We must

**Figure 1.1** Some diameters used in microscopy

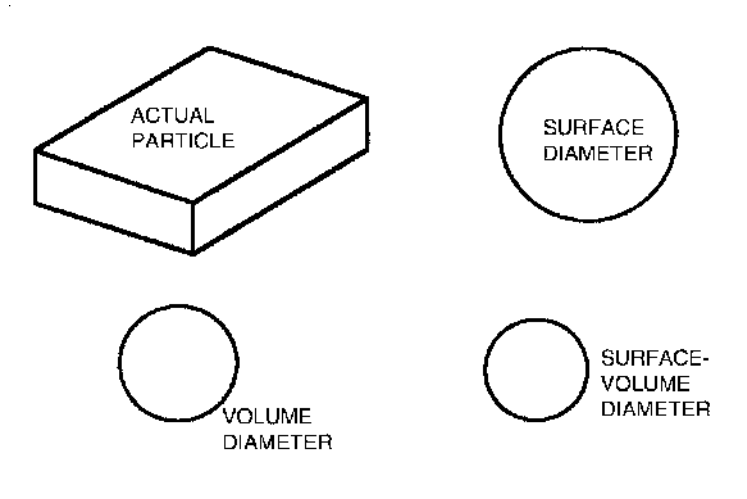


Figure 1.2 Comparison of equivalent sphere diameters

remember that the orientation of the particle on the microscope slide will affect the projected image and consequently the measured equivalent sphere diameter.

If we use a sieve to measure the particle size we come up with an equivalent sphere diameter, which is the diameter of a sphere passing through the same sieve aperture. If we use a sedimentation technique to measure particle size then it is expressed as the diameter of a sphere having the same sedimentation velocity under the same conditions. Other examples of the properties of particles measured and the resulting equivalent sphere diameters are given in Figure 1.2.

Table 1.2 compares values of these different equivalent sphere diameters used to describe a cuboid of side lengths 1, 3, 5 and a cylinder of diameter 3 and length 1.

The volume equivalent sphere diameter or equivalent volume sphere diameter is a commonly used equivalent sphere diameter. We will see later in the chapter that it is used in the Coulter counter size measurements technique. By definition, the equivalent volume sphere diameter is the diameter of a sphere having the same volume as the particle. The surface-volume diameter is the one measured when we use permeametry (see Section 1.8.4) to measure size. The surface-volume (equivalent sphere) diameter is the diameter of a sphere having the same surface to volume ratio as the particle. *In practice it is important to use the method of*

Table 1.2 Comparison of equivalent sphere diameters

Shape	Sphere passing the same sieve aperture, $x_p$	Sphere having the same volume, $x_v$	Sphere having the same surface area, $x_s$	Sphere having the same surface to volume ratio, $x_{sv}$
Cuboid	3	3.06	3.83	1.95
Cylinder	3	2.38	2.74	1.80

size measurement which directly gives the particle size which is relevant to the situation or process of interest. (See Worked Example 1.1.)

### 1.3 DESCRIPTION OF POPULATIONS OF PARTICLES

A population of particles is described by a particle size distribution. Particle size distributions may be expressed as frequency distribution curves or cumulative curves. These are illustrated in Figure 1.3. The two are related mathematically in that the cumulative distribution is the integral of the frequency distribution; i.e. if the cumulative distribution is denoted as  $F$ , then the frequency distribution  $dF/dx$ . For simplicity,  $dF/dx$  is often written as  $f(x)$ . The distributions can be by number, surface, mass or volume (where particle density does not vary with size, the mass distribution is the same as the volume distribution). Incorporating this information into the notation,  $f_N(x)$  is the frequency distribution by number,  $f_S(x)$  is the frequency distribution by surface,  $F_S$  is the cumulative distribution by

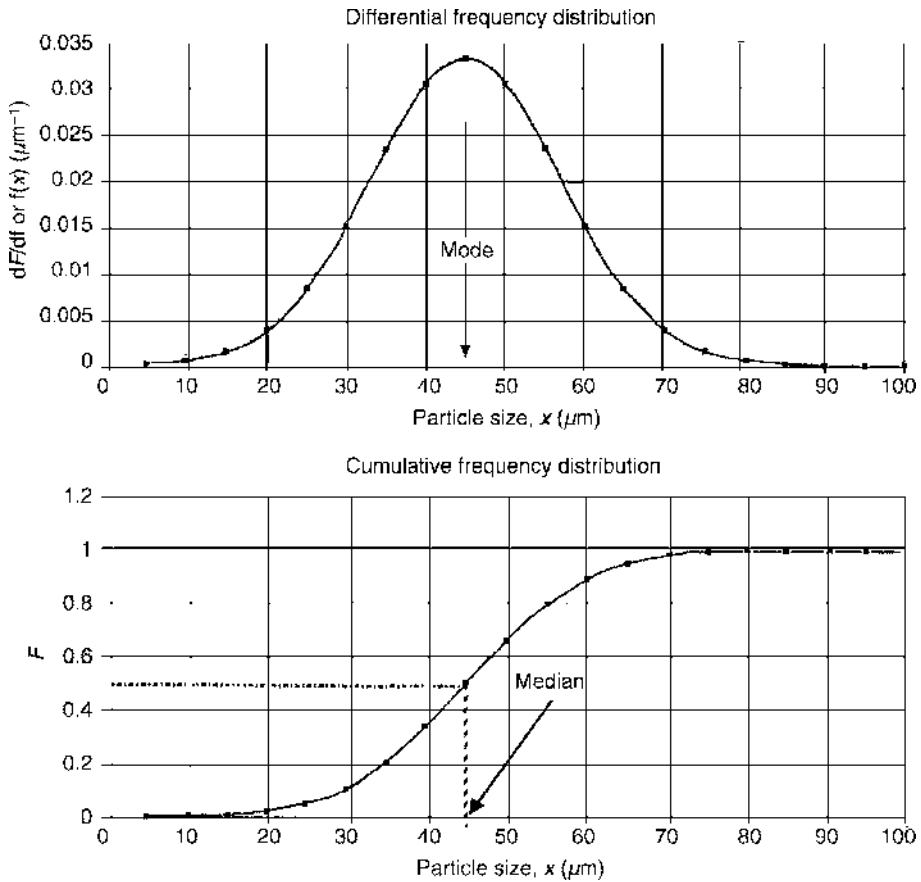


Figure 1.3 Typical differential and cumulative frequency distributions

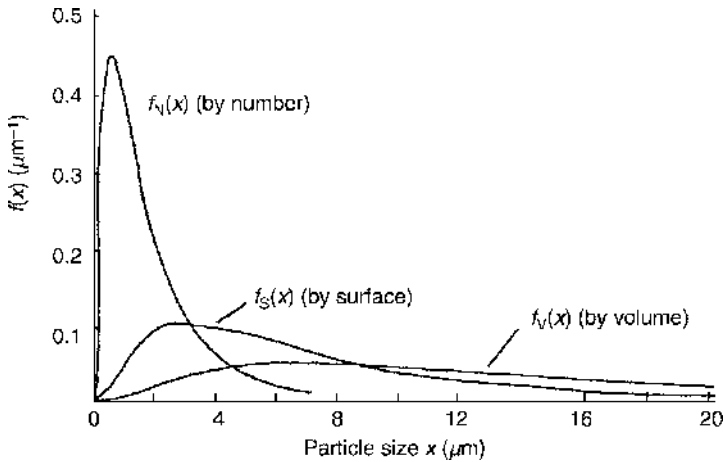


Figure 1.4 Comparison between distributions

surface and  $F_M$  is the cumulative distribution by mass. In reality these distributions are smooth continuous curves. However, size measurement methods often divide the size spectrum into size ranges or classes and the size distribution becomes a histogram.

For a given population of particles, the distributions by mass, number and surface can differ dramatically, as can be seen in Figure 1.4.

A further example of difference between distributions for the same population is given in Table 1.3 showing size distributions of man-made objects orbiting the earth (*New Scientist*, 13 October 1991).

The number distribution tells us that only 0.2% of the objects are greater than 10 cm. However, these larger objects make up 99.96% of the mass of the population, and the 99.3% of the objects which are less than 1.0 cm in size make up only 0.01% of the mass distribution. Which distribution we would use is dependent on the end use of the information.

### 1.4 CONVERSION BETWEEN DISTRIBUTIONS

Many modern size analysis instruments actually measure a number distribution, which is rarely needed in practice. These instruments include software to

Table 1.3 Mass and number distributions for man-made objects orbiting the earth

Size (cm)	Number of objects	% by number	% by mass
10–1000	7000	0.2	99.96
1–10	17 500	0.5	0.03
0.1–1.0	3 500 000	99.3	0.01
Total	3 524 500	100.00	100.00

convert the measured distribution into more practical distributions by mass, surface, etc.

Relating the size distributions by number,  $f_N(x)$ , and by surface,  $f_S(x)$  for a population of particles having the same geometric shape but different size:

Fraction of particles in the size range

$$x \text{ to } x + dx = f_N(x)dx$$

Fraction of the total surface of particles in the size range

$$x \text{ to } x + dx = f_S(x)dx$$

If  $N$  is the total number of particles in the population, the number of particles in the size range  $x$  to  $x + dx = Nf_N(x)dx$  and the surface area of these particles  $= (x^2\alpha_S)Nf_N(x)dx$ , where  $\alpha_S$  is the factor relating the linear dimension of the particle to its surface area.

Therefore, the fraction of the total surface area contained on these particles  $[f_S(x)dx]$  is:

$$\frac{(x^2\alpha_S)Nf_N(x)dx}{S}$$

where  $S$  is the total surface area of the population of particles.

For a given population of particles, the total number of particles,  $N$ , and the total surface area,  $S$  are constant. Also, assuming particle shape is independent of size,  $\alpha_S$  is constant, and so

$$f_S(x) \propto x^2f_N(x) \quad \text{or} \quad f_S(x) = k_Sx^2f_N(x) \quad (1.1)$$

where

$$k_S = \frac{\alpha_S N}{S}$$

Similarly, for the distribution by volume

$$f_V(x) = k_Vx^3f_N(x) \quad (1.2)$$

where

$$k_V = \frac{\alpha_V N}{V}$$

where  $V$  is the total volume of the population of particles and  $\alpha_V$  is the factor relating the linear dimension of the particle to its volume.

And for the distribution by mass

$$f_m(x) = k_m x^3 f_N(x) \quad (1.3)$$

where

$$k_m = \frac{\alpha_V \rho_p N}{V}$$

assuming particle density  $\rho_p$  is independent of size.

The constants  $k_S$ ,  $k_V$  and  $k_m$  may be found by using the fact that:

$$\int_0^{\infty} f(x) dx = 1 \quad (1.4)$$

Thus, when we convert between distributions it is necessary to make assumptions about the constancy of shape and density with size. Since these assumptions may not be valid, the conversions are likely to be in error. Also, calculation errors are introduced into the conversions. For example, imagine that we used an electron microscope to produce a number distribution of size with a measurement error of  $\pm 2\%$ . Converting the number distribution to a mass distribution we triple the error involved (i.e. the error becomes  $\pm 6\%$ ). For these reasons, conversions between distributions are to be avoided wherever possible. This can be done by choosing the measurement method which gives the required distribution directly.

## 1.5 DESCRIBING THE POPULATION BY A SINGLE NUMBER

In most practical applications, we require to describe the particle size of a population of particles (millions of them) by a single number. There are many options available; the mode, the median, and several different means including arithmetic, geometric, quadratic, harmonic, etc. Whichever expression of central tendency of the particle size of the population we use must reflect the property or properties of the population of importance to us. We are, in fact, modelling the real population with an artificial population of mono-sized particles. This section deals with calculation of the different expressions of central tendency and selection of the appropriate expression for a particular application.

The *mode* is the most frequently occurring size in the sample. We note, however, that for the same sample, different modes would be obtained for distributions by number, surface and volume. The mode has no practical significance as a measure of central tendency and so is rarely used in practice.

The *median* is easily read from the cumulative distribution as the 50% size; the size which splits the distribution into two equal parts. In a mass distribution, for example, half of the particles by mass are smaller than the median size. Since the

**Table 1.4** Definitions of means

$g(x)$	Mean and notation
$x$	arithmetic mean, $\bar{x}_a$
$x^2$	quadratic mean, $\bar{x}_q$
$x^3$	cubic mean, $\bar{x}_c$
$\log x$	geometric mean, $\bar{x}_g$
$1/x$	harmonic mean, $\bar{x}_h$

median is easily determined, it is often used. However, it has no special significance as a measure of central tendency of particle size.

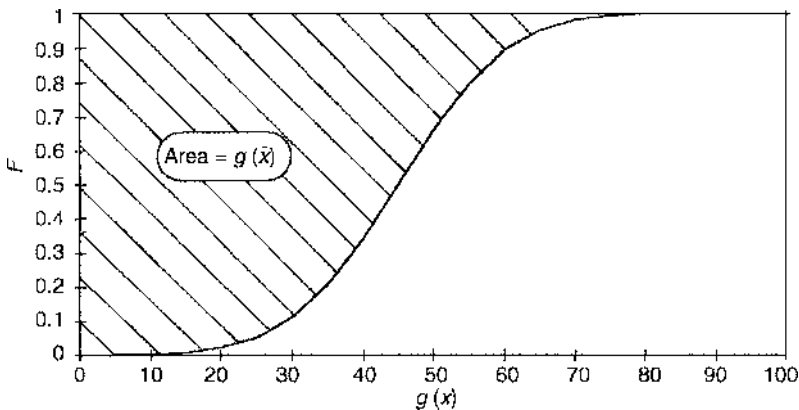
Many different *means* can be defined for a given size distribution; as pointed out by Svarovsky (1990). However, they can all be described by:

$$g(\bar{x}) = \frac{\int_0^1 g(x) dF}{\int_0^1 dF} \quad \text{but} \quad \int_0^1 dF = 1 \quad \text{and so} \quad g(\bar{x}) = \int_0^1 g(x) dF \quad (1.5)$$

where  $\bar{x}$  is the mean and  $g$  is the weighting function, which is different for each mean definition. Examples are given in Table 1.4.

Equation (1.5) tells us that the mean is the area between the curve and the  $F(x)$  axis in a plot of  $F(x)$  versus the weighting function  $g(x)$  (Figure 1.5). In fact, graphical determination of the mean is always recommended because the distribution is more accurately represented as a continuous curve.

Each mean can be shown to conserve two properties of the original population of particles. For example, the arithmetic mean of the surface distribution conserves the surface and volume of the original population. This is demonstrated in Worked Example 1.3. This mean is commonly referred to as the surface-volume mean or the Sauter mean. The arithmetic mean of the number



**Figure 1.5** Plot of cumulative frequency against weighting function  $g(x)$ . Shaded area is  $g(\bar{x}) = \int_0^1 g(x) dF$



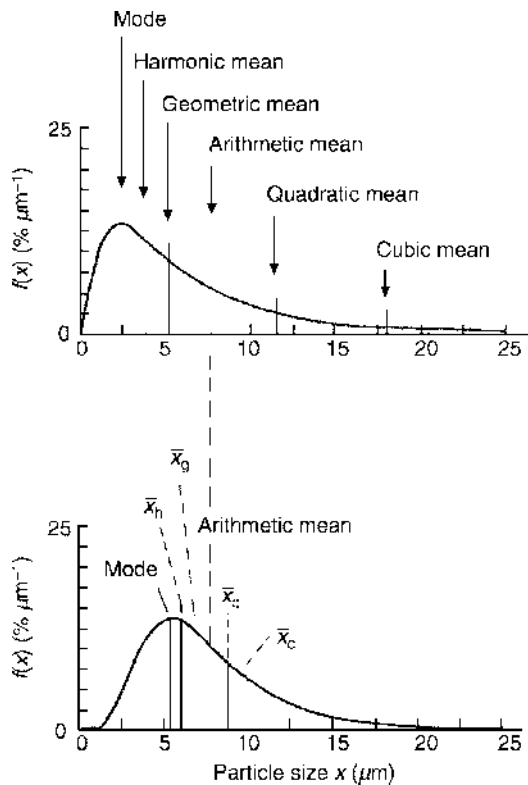
distribution  $\bar{x}_{aN}$  conserves the number and length of the original population and is known as the number-length mean  $\bar{x}_{NL}$ :

$$\text{number-length mean, } \bar{x}_{NL} = \bar{x}_{aN} = \frac{\int_0^1 x dF_N}{\int_0^1 dF_N} \tag{1.6}$$

As another example, the quadratic mean of the number distribution  $\bar{x}_{qN}$  conserves the number and surface of the original population and is known as the number-surface mean  $\bar{x}_{NS}$ :

$$\text{number-surface mean, } \bar{x}_{NS}^2 = \bar{x}_{qN}^2 = \frac{\int_0^1 x^2 dF_N}{\int_0^1 dF_N} \tag{1.7}$$

A comparison of the values of the different means and the mode and median for a given particle size distribution is given in Figure 1.6. This figure highlights two points: (a) that the values of the different expressions of central tendency can vary significantly; and (b) that two quite different distributions could have the same



**Figure 1.6** Comparison between measures of central tendency. Adapted from Rhodes (1990). Reproduced by permission

arithmetic mean or median, etc. If we select the wrong one for our design correlation or quality control we may be in serious error.

So how do we decide which mean particle size is the most appropriate one for a given application? Worked Examples 1.3 and 1.4 indicate how this is done.

For Equation (1.8), which defines the surface-volume mean, please see Worked Example 1.3.

## 1.6 EQUIVALENCE OF MEANS

Means of different distributions can be equivalent. For example, as is shown below, the arithmetic mean of a surface distribution is equivalent (numerically equal to) the harmonic mean of a volume (or mass) distribution:

$$\text{arithmetic mean of a surface distribution, } \bar{x}_{aS} = \frac{\int_0^1 x dF_S}{\int_0^1 dF_S} \quad (1.9)$$

The harmonic mean  $\bar{x}_{hV}$  of a volume distribution is defined as:

$$\frac{1}{\bar{x}_{hV}} = \frac{\int_0^1 \left(\frac{1}{x}\right) dF_V}{\int_0^1 dF_V} \quad (1.10)$$

From Equations (1.1) and (1.2), the relationship between surface and volume distributions is:

$$dF_V = x dF_S \frac{k_V}{k_S} \quad (1.11)$$

hence

$$\frac{1}{\bar{x}_{hV}} = \frac{\int_0^1 \left(\frac{1}{x}\right) x \frac{k_V}{k_S} dF_S}{\int_0^1 x \frac{k_V}{k_S} dF_S} = \frac{\int_0^1 dF_S}{\int_0^1 x dF_S} \quad (1.12)$$

(assuming  $k_S$  and  $k_V$  do not vary with size)

and so

$$\bar{x}_{hV} = \frac{\int_0^1 x dF_S}{\int_0^1 dF_S}$$

which, by inspection, can be seen to be equivalent to the arithmetic mean of the surface distribution  $\bar{x}_{aS}$  [Equation (1.9)].

Recalling that  $dF_S = x^2 k_S dF_N$ , we see from Equation (1.9) that

$$\bar{x}_{aS} = \frac{\int_0^1 x^3 dF_N}{\int_0^1 x^2 dF_N}$$

which is the surface-volume mean,  $\bar{x}_{SV}$  [Equation (1.8) - see Worked Example 1.3].

Summarizing, then, the surface-volume mean may be calculated as the arithmetic mean of the surface distribution or the harmonic mean of the volume distribution. The practical significance of the equivalence of means is that it permits useful means to be calculated easily from a single size analysis.

The reader is invited to investigate the equivalence of other means.

## 1.7 COMMON METHODS OF DISPLAYING SIZE DISTRIBUTIONS

### 1.7.1 Arithmetic-normal Distribution

In this distribution, shown in Figure 1.7, particle sizes with equal differences from the arithmetic mean occur with equal frequency. Mode, median and arithmetic mean coincide. The distribution can be expressed mathematically by:

$$\frac{dF}{dx} = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{(x - \bar{x})^2}{2\sigma^2}\right] \quad (1.13)$$

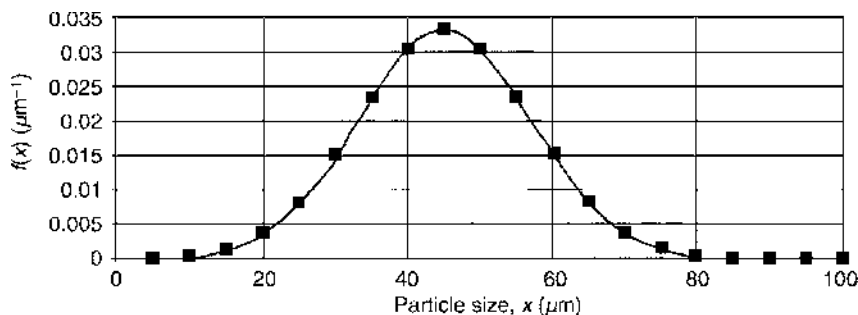
where  $\sigma$  is the standard deviation.

To check for a arithmetic-normal distribution, size analysis data is plotted on normal probability graph paper. On such graph paper a straight line will result if the data fits an arithmetic-normal distribution.

### 1.7.2 Log-normal Distribution

This distribution is more common for naturally occurring particle populations. An example is shown in Figure 1.8. If plotted as  $dF/d(\log x)$  versus  $x$ , rather than  $dF/dx$  versus  $x$ , an arithmetic-normal distribution in  $\log x$  results (Figure 1.9). The mathematical expression describing this distribution is:

$$\frac{dF}{dz} = \frac{1}{\sigma_z\sqrt{2\pi}} \exp\left[-\frac{(z - \bar{z})^2}{2\sigma_z^2}\right] \quad (1.14)$$



**Figure 1.7** Arithmetic-normal distribution with an arithmetic mean of 45 and standard deviation of 12

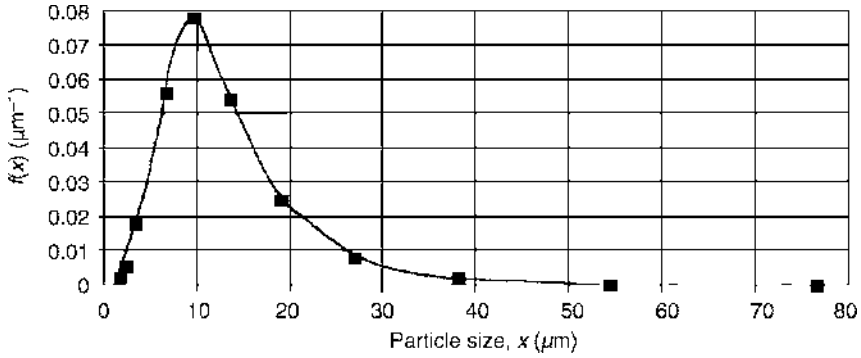


Figure 1.8 Log-normal distribution plotted on linear coordinates

where  $z = \log x$ ,  $\bar{z}$  is the arithmetic mean of  $\log x$  and  $\sigma_z$  is the standard deviation of  $\log x$ .

To check for a log-normal distribution, size analysis data are plotted on log-normal probability graph paper. Using such graph paper, a straight line will result if the data fit a log-normal distribution.

## 1.8 METHODS OF PARTICLE SIZE MEASUREMENT

### 1.8.1 Sieving

Dry sieving using woven wire sieves is a simple, cheap method of size analysis suitable for particle sizes greater than  $45 \mu\text{m}$ . Sieving gives a mass distribution and a size known as the sieve diameter. Since the length of the particle does not hinder its passage through the sieve apertures (unless the particle is extremely elongated), the sieve diameter is dependent on the maximum width and maximum thickness of the

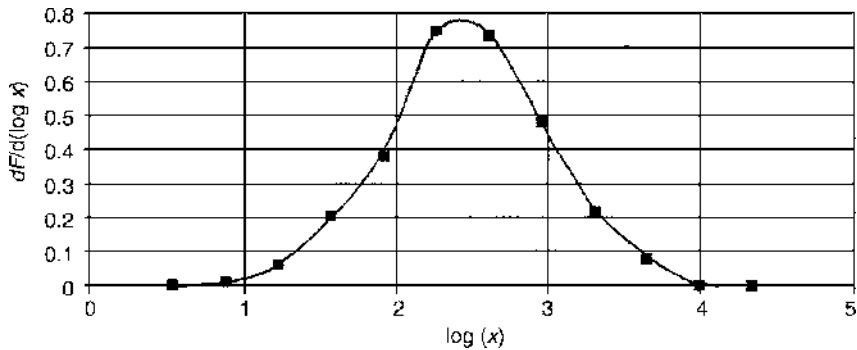


Figure 1.9 Log-normal distribution plotted on logarithmic coordinates

particle. The most common modern sieves are in sizes such that the ratio of adjacent sieve sizes is the fourth root of two (eg. 45, 53, 63, 75, 90, 107  $\mu\text{m}$ ). If standard procedures are followed and care is taken, sieving gives reliable and reproducible size analysis. Air jet sieving, in which the powder on the sieve is fluidized by a jet of air, can achieve analysis down to 20  $\mu\text{m}$ . Analysis down to 5  $\mu\text{m}$  can be achieved by wet sieving, in which the powder sample is suspended in a liquid.

### 1.8.2 Microscopy

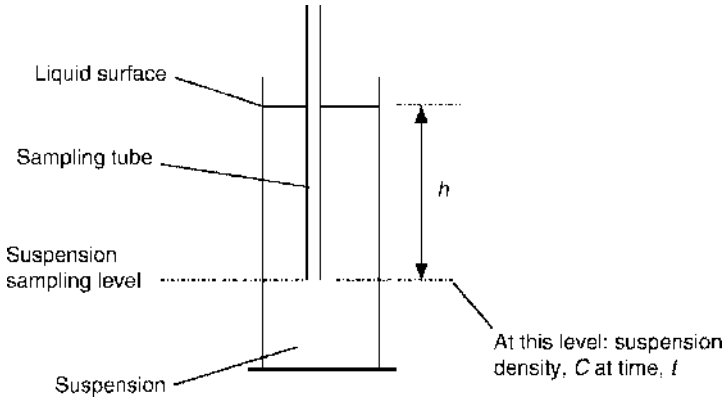
The optical microscope may be used to measure particle sizes down to 5  $\mu\text{m}$ . For particles smaller than this diffraction causes the edges of the particle to be blurred and this gives rise to an apparent size. The electron microscope may be used for size analysis below 5  $\mu\text{m}$ . Coupled with an image analysis system the optical microscope or electron microscope can readily give number distributions of size and shape. Such systems calculate various diameters from the projected image of the particles (e.g. Martin's, Feret's, shear, projected area diameters, etc.). Note that for irregular-shaped particles, the projected area offered to the viewer can vary significantly depending on the orientation of the particle. Techniques such as applying adhesive to the microscope slide may be used to ensure that the particles are randomly orientated.

### 1.8.3 Sedimentation

In this method, the rate of sedimentation of a sample of particles in a liquid is followed. The suspension is dilute and so the particles are assumed to fall at their single particle terminal velocity in the liquid (usually water). Stokes' law is assumed to apply ( $Re_p < 0.3$ ) and so the method using water is suitable only for particles typically less than 50  $\mu\text{m}$  in diameter. The rate of sedimentation of the particles is followed by plotting the suspension density at a certain vertical position against time. The suspension density is directly related to the cumulative undersize and the time is related to the particle diameter via the terminal velocity. This is demonstrated in the following:

Referring to Figure 1.10, the suspension density is sampled at a vertical distance,  $h$  below the surface of the suspension. The following assumptions are made:

- The suspension is sufficiently dilute for the particles to settle as individuals (i.e. not hindered settling – see Chapter 3).
- Motion of the particles in the liquid obeys Stokes' law (true for particles typically smaller than 50  $\mu\text{m}$ ).
- Particles are assumed to accelerate rapidly to their terminal free fall velocity  $U_T$  so that the time for acceleration is negligible.



**Figure 1.10** Size analysis by sedimentation

Let the original uniform suspension density be  $C_0$ . Let the suspension density at the sampling point be  $C$  at time  $t$  after the start of settling. At time  $t$  all those particles travelling faster than  $h/t$  will have fallen below the sampling point. The sample at time  $t$  will therefore consist only of particles travelling a velocity  $\leq h/t$ . Thus, if  $C_0$  is representative of the suspension density for the whole population, then  $C$  represents the suspension density for all particles which travel at a velocity  $\leq h/t$ , and so  $C/C_0$  is the mass fraction of the original particles which travel at a velocity  $\leq h/t$ . That is,

$$\text{cumulative mass fraction} = \frac{C}{C_0}$$

All particles travel at their terminal velocity given by Stokes' law [Chapter 2, Equation (2.13)]:

$$U_T = \frac{x^2(\rho_p - \rho_t)g}{18\mu}$$

Thus, equating  $U_T$  with  $h/t$ , we determine the diameter of the particle travelling at our cut-off velocity  $h/t$ . That is,

$$x = \left[ \frac{18\mu h}{t(\rho_p - \rho_t)g} \right]^{1/2} \quad (1.15)$$

Particles smaller than  $x$  will travel slower than  $h/t$  and will still be in suspension at the sampling point. Corresponding values of  $C/C_0$  and  $x$  therefore give us the cumulative mass distribution. The particle size measured is the Stokes' diameter, i. e. the diameter of a sphere having the same terminal settling velocity in the Stokes region as the actual particle.

A common form of this method is the Andreason pipette which is capable of measuring in the range 2–100  $\mu\text{m}$ . At size below 2  $\mu\text{m}$ , Brownian motion causes significant errors. Increasing the body force acting on the particles by centrifuging the suspension permits the effects of Brownian motion to be reduced so that particle sizes down to 0.01  $\mu\text{m}$  can be measured. Such a device is known as a pipette centrifuge.

The labour involved in this method may be reduced by using either light absorption or X-ray absorption to measure the suspension density. The light absorption method gives rise to a distribution by surface, whereas the X-ray absorption method gives a mass distribution.

### 1.8.4 Permeametry

This is a method of size analysis based on fluid flow through a packed bed (see Chapter 6). The Carman–Kozeny equation for laminar flow through a randomly packed bed of uniformly sized spheres of diameter  $x$  is [Equation 6.9]:

$$\frac{(-\Delta p)}{H} = 180 \frac{(1 - \varepsilon)^2 \mu U}{\varepsilon^3 x^2}$$

where  $(-\Delta p)$  is the pressure drop across the bed,  $\varepsilon$  is the packed bed void fraction,  $H$  is the depth of the bed,  $\mu$  is the fluid viscosity and  $U$  is the superficial fluid velocity. In Worked Example 1.3, we will see that, when we are dealing with non-spherical particles with a distribution of sizes, the appropriate mean diameter for this equation is the surface-volume diameter  $\bar{x}_{\text{SV}}$ , which may be calculated as the arithmetic mean of the surface distribution,  $\bar{x}_{\text{aS}}$ .

In this method, the pressure gradient across a packed bed of known voidage is measured as a function of flow rate. The diameter we calculate from the Carman–Kozeny equation is the arithmetic mean of the surface distribution (see Worked Example 6.1 in Chapter 6).

### 1.8.5 Electrozone Sensing

Particles are held in suspension in a dilute electrolyte which is drawn through a tiny orifice with a voltage applied across it (Figure 1.11). As particles flow through the orifice a voltage pulse is recorded.

The amplitude of the pulse can be related to the volume of the particle passing the orifice. Thus, by electronically counting and classifying the pulses according to amplitude this technique can give a number distribution of the equivalent volume sphere diameter. The lower size limit is dictated by the smallest practical orifice and the upper limit is governed by the need to maintain particles in suspension. Although liquids more viscous than water may be used to reduce sedimentation, the practical range of size for this method is 0.3–1000  $\mu\text{m}$ . Errors are introduced if more than one particle passes through the orifice at a time and so dilute suspensions are used to reduce the likelihood of this error.

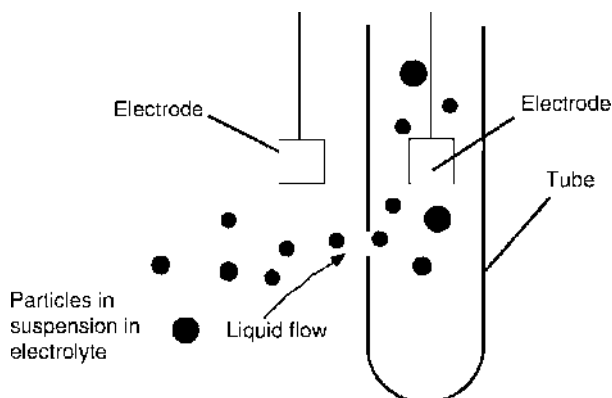


Figure 1.11 Schematic of electrozone sensing apparatus

### 1.8.6 Laser Diffraction

This method relies on the fact that for light passing through a suspension, the diffraction angle is inversely proportional to the particle size. An instrument would consist of a laser as a source of coherent light of known fixed wavelength (typically  $0.63\ \mu\text{m}$ ), a suitable detector (usually a slice of photo-sensitive silicon with a number of discrete detectors, and some means of passing the sample of particles through the laser light beam (techniques are available for suspending particles in both liquids and gases are drawing them through the beam).

To relate diffraction angle with particle size, early instruments used the Fraunhofer theory, which can give rise to large errors under some circumstances (e.g. when the refractive indices of the particle material and suspending medium approach each other). Modern instruments use the Mie theory for interaction of light with matter. This allows particle sizing in the range  $0.1\text{--}2000\ \mu\text{m}$ , provided that the refractive indices of the particle material and suspending medium are known.

This method gives a volume distribution and measures a diameter known as the laser diameter. Particle size analysis by laser diffraction is very common in industry today. The associated software permits display of a variety of size distributions and means derived from the original measured distribution.

## 1.9 SAMPLING

In practice, the size distribution of many tonnes of powder are often assumed from an analysis performed on just a few grams or milligrams of sample. The importance of that sample being representative of the bulk powder cannot be overstated. However, as pointed out in Chapter 11 on mixing and segregation, most powder handling and processing operations (pouring, belt conveying,



handling in bags or drums, motion of the sample bottle, etc.) cause particles to segregate according to size and to a lesser extent density and shape. This natural tendency to segregation means that extreme care must be taken in sampling.

There are two golden rules of sampling:

1. The powder should be in motion when sampled.
2. The whole of the moving stream should be taken for many short time increments.

Since the eventual sample size used in the analysis may be very small, it is often necessary to split the original sample in order to achieve the desired amount for analysis. These sampling rules must be applied at every step of sampling and sample splitting.

Detailed description of the many devices and techniques used for sampling in different process situations and sample dividing are outside the scope of this chapter. However, Allen (1990) gives an excellent account, to which the reader is referred.

## 1.10 WORKED EXAMPLES

### WORKED EXAMPLE 1.1

Calculate the equivalent volume sphere diameter  $x_v$  and the surface-volume equivalent sphere diameter  $x_{sv}$  of a cuboid particle of side length 1, 2, 4 mm.

#### *Solution*

The volume of cuboid =  $1 \times 2 \times 4 = 8 \text{ mm}^3$

The surface area of the particle =  $(1 \times 2) + (1 \times 2) + (1 + 2 + 1 + 2) \times 4 = 28 \text{ mm}^2$

The volume of sphere of diameter  $x_v$  is  $\pi x_v^3/6$

Hence, diameter of a sphere having a volume of  $8 \text{ mm}^3$ ,  $x_v = 2.481 \text{ mm}$

The *equivalent volume sphere diameter*  $x_v$  of the cuboid particle is therefore  $x_v = 2.481 \text{ mm}$

The surface to volume ratio of the cuboid particle =  $\frac{28}{8} = 3.5 \text{ mm}^2/\text{mm}^3$

The surface to volume ratio for a sphere of diameter  $x_{sv}$  is therefore  $6/x_{sv}$

Hence, the diameter of a sphere having the same surface to volume ratio as the particle =  $6/3.5 = 1.714 \text{ mm}$

The *surface-volume equivalent sphere diameter* of the cuboid,  $x_{sv} = 1.714 \text{ mm}$

**WORKED EXAMPLE 1.2**

Convert the surface distribution described by the following equation to a cumulative volume distribution:

$$F_S = (x/45)^2 \quad \text{for } x \leq 45 \mu\text{m}$$

$$F_S = 1 \quad \text{for } x > 45 \mu\text{m}$$

**Solution**

From Equations (1.1)–(1.3),

$$f_v(x) = \frac{k_v}{k_s} x f_s(x)$$

Integrating between sizes 0 and  $x$ :

$$F_v(x) = \int_0^x \left( \frac{k_v}{k_s} \right) x f_s(x) dx$$

Noting that  $f_s(x) = dF_s/dx$ , we see that

$$f_s(x) = \frac{d}{dx} \left( \frac{x}{45} \right)^2 = \frac{2x}{(45)^2}$$

and our integral becomes

$$F_v(x) = \int_0^x \left( \frac{k_v}{k_s} \right) \frac{2x^2}{(45)^2} dx$$

Assuming that  $k_v$  and  $k_s$  are independent of size,

$$F_v(x) = \left( \frac{k_v}{k_s} \right) \int_0^x \frac{2x^2}{(45)^2} dx$$

$$= \frac{2}{3} \left[ \frac{x^3}{(45)^2} \right] \frac{k_v}{k_s}$$

$k_v/k_s$  may be found by noting that  $F_v(45) = 1$ ; hence

$$\frac{90 k_v}{3 k_s} = 1 \quad \text{and so} \quad \frac{k_v}{k_s} = 0.0333$$

Thus, the formula for the volume distribution is

$$F_v = 1.096 \times 10^{-5} x^3 \quad \text{for } x \leq 45 \mu\text{m}$$

$$F_v = 1 \quad \text{for } x > 45 \mu\text{m}$$

**WORKED EXAMPLE 1.3**

What mean particle size do we use in calculating the pressure gradient for flow of a fluid through a packed bed of particles using the Carman–Kozeny equation (see Chapter 6)?

**Solution**

The Carman–Kozeny equation for laminar flow through a randomly packed bed of particles is:

$$\frac{(-\Delta p)}{L} = K \frac{(1 - \epsilon)^2}{\epsilon^3} S_v^2 \mu U$$

where  $S_v$  is the specific surface area of the bed of particles (particle surface area per unit particle volume) and the other terms are defined in Chapter 6. If we assume that the bed voidage is independent of particle size, then to write the equation in terms of a mean particle size, we must express the specific surface,  $S_v$ , in terms of that mean. The particle size we use must give the same value of  $S_v$  as the original population or particles. Thus the mean diameter  $\bar{x}$  must conserve the surface and volume of the population; that is, the mean must enable us to calculate the total volume from the total surface of the particles. This mean is the surface-volume mean  $\bar{x}_{sv}$

$$\bar{x}_{sv} \times (\text{total surface}) \times \frac{\alpha_v}{\alpha_s} = (\text{total volume}) \left( \text{eg. for spheres, } \frac{\alpha_v}{\alpha_s} = \frac{1}{6} \right)$$

$$\text{and therefore } \bar{x}_{sv} \int_0^\infty f_s(x) dx \cdot \frac{k_v}{k_s} = \int_0^\infty f_v(x) dx$$

$$\text{Total volume of particles, } V = \int_0^\infty x^3 \alpha_v N f_N(x) dx$$

$$\text{Total surface area of particles, } S = \int_0^\infty x^2 \alpha_s N f_N(x) dx$$

$$\text{Hence, } \bar{x}_{sv} = \frac{\alpha_s \int_0^\infty x^3 \alpha_v N f_N(x) dx}{\alpha_v \int_0^\infty x^2 \alpha_s N f_N(x) dx}$$

Then, since  $\alpha_v$ ,  $\alpha_s$  and  $N$  are independent of size,  $x$ ,

$$\bar{x}_{sv} = \frac{\int_0^\infty x^3 f_N(x) dx}{\int_0^\infty x^2 f_N(x) dx} = \frac{\int_0^1 x^3 dF_N}{\int_0^1 x^2 dF_N}$$

This is the definition of the mean which conserves surface and volume, known as the surface-volume mean,  $\bar{x}_{sv}$ .

So

$$\bar{x}_{sv} = \frac{\int_0^1 x^3 dF_N}{\int_0^1 x^2 dF_N} \tag{1.8}$$

The correct mean particle diameter is therefore the surface-volume mean as defined above. (We saw in Section 1.6 that this may be calculated as the arithmetic mean of the

surface distribution  $\bar{x}_{aS}$ , or the harmonic mean of the volume distribution.) Then in the Carman–Kozeny equation we make the following substitution for  $S_v$ :

$$S_v = \frac{1}{\bar{x}_{SV}} \frac{k_s}{k_v}$$

e.g. for spheres,  $S_v = 6/\bar{x}_{SV}$ .

### WORKED EXAMPLE 1.4 (AFTER SVAROVSKY, 1990)

A gravity settling device processing a feed with size distribution  $F(x)$  and operates with a grade efficiency  $G(x)$ . Its total efficiency is defined as:

$$E_T = \int_0^1 G(x) dF_M$$

How is the mean particle size to be determined?

#### Solution

Assuming plug flow (see Chapter 3),  $G(x) = U_T A/Q$  where,  $A$  is the settling area,  $Q$  is the volume flow rate of suspension and  $U_T$  is the single particle terminal velocity for particle size  $x$ , given by (in the Stokes region):

$$U_T = \frac{x^2(\rho_p - \rho_f)g}{18\mu} \quad (\text{Chapter 2})$$

hence

$$E_T = \frac{Ag(\rho_p - \rho_f)}{18\mu Q} \int_0^1 x^2 dF_M$$

where  $\int_0^1 x^2 dF_M$  is seen to be the definition of the quadratic mean of the distribution by mass  $\bar{x}_{qM}^0$  (see Table 1.4).

This approach may be used to determine the correct mean to use in many applications.

### WORKED EXAMPLE 1.5

A Coulter counter analysis of a cracking catalyst sample gives the following cumulative volume distribution:

Channel	1	2	3	4	5	6	7	8
% volume differential	0	0.5	1.0	1.6	2.6	3.8	5.7	8.7
Channel	9	10	11	12	13	14	15	16
% volume differential	14.3	22.2	33.8	51.3	72.0	90.9	99.3	100

- (a) Plot the cumulative volume distribution versus size and determine the median size.
- (b) Determine the surface distribution, giving assumptions. Compare with the volume distribution.
- (c) Determine the harmonic mean diameter of the volume distribution.
- (d) Determine the arithmetic mean diameter of the surface distribution.

**Solution**

With the Coulter counter the channel size range differs depending on the tube in use. We therefore need the additional information that in this case channel 1 covers the size range 3.17 μm to 4.0 μm, channel 2 covers the range 4.0 μm to 5.04 μm and so on up to channel 16, which covers the range 101.4 μm to 128 μm. The ratio of adjacent size range boundaries is always the cube root of 2. For example,

$$\sqrt[3]{2} = \frac{4.0}{3.17} = \frac{5.04}{4.0} = \frac{128}{101.4}, \text{ etc.}$$

The resulting lower and upper sizes for the channels are shown in columns 2 and 3 of Table 1W5.1.

**Table 1W5.1** Size distribution data associated with Worked Example 1.5

1	2	3	4	5	6	7	8	9
Channel number	Lower size of range μm	Upper size of range μm	Cumulative per cent undersize	$F_v$	$1/x$	Cumulative area under $F_v$ versus $1/x$	$F_s$	Cumulative area under $F_s$ versus $x$
1	3.17	4.00	0	0	0.2500	0.0000	0.0000	0.0000
2	4.00	5.04	0.5	0.005	0.1984	0.0011	0.0403	0.1823
3	5.04	6.35	1	0.01	0.1575	0.0020	0.0723	0.3646
4	6.35	8.00	1.6	0.016	0.1250	0.0029	0.1028	0.5834
5	8.00	10.08	2.6	0.026	0.0992	0.0040	0.1432	0.9480
6	10.08	12.70	3.8	0.038	0.0787	0.0050	0.1816	1.3855
7	12.70	16.00	5.7	0.057	0.0625	0.0064	0.2299	2.0782
8	16.00	20.16	8.7	0.087	0.0496	0.0081	0.2904	3.1720
9	20.16	25.40	14.3	0.143	0.0394	0.0106	0.3800	5.2138
10	25.40	32.00	22.2	0.222	0.0313	0.0134	0.4804	8.0942
11	32.00	40.32	33.8	0.338	0.0248	0.0166	0.5973	12.3236
12	40.32	50.80	51.3	0.513	0.0197	0.0205	0.7374	18.7041
13	50.80	64.00	72	0.72	0.0156	0.0242	0.8689	26.2514
14	64.00	80.63	90.9	0.909	0.0124	0.0268	0.9642	33.1424
15	80.63	101.59	99.3	0.993	0.0098	0.0277	0.9978	36.2051
16	101.59	128.00	100	1	0.0078	<b>0.0278</b>	1.0000	<b>36.4603</b>

Note: Based on arithmetic means of size ranges.

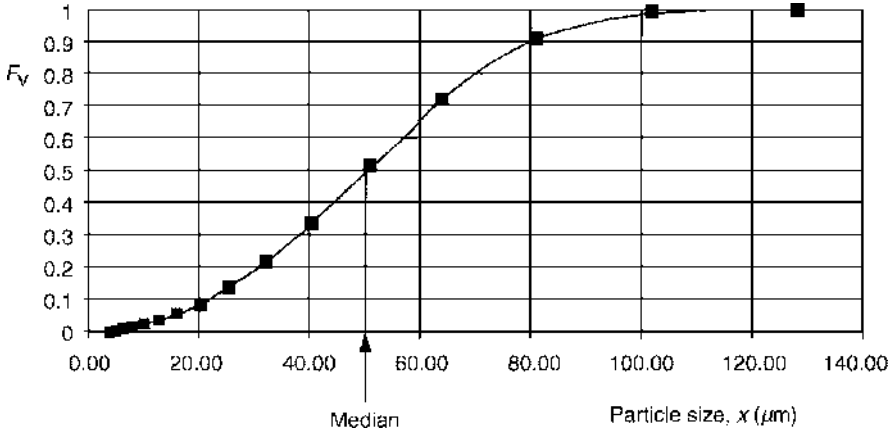


Figure 1W5.1 Cumulative volume distribution

(a) The cumulative undersize distribution is shown numerically in column 5 of Table 1W5.1 and graphically in Figure 1W5.1. By inspection, we see that the median size is  $50\ \mu\text{m}$  (b), i.e. 50% by volume of the particles is less than  $50\ \mu\text{m}$ .

(b) The surface distribution is related to the volume distribution by the expression:

$$f_s(x) = \frac{f_v(x)}{x} \times \frac{k_s}{k_v} \quad (\text{from [Equations(1.1) and (1.2)]})$$

Recalling that  $f(x) = dF/dx$  and integrating between 0 and  $x$ :

$$\frac{k_s}{k_v} \int_0^x \frac{1}{x} dF_v = \int_0^x \frac{dF_s}{dx} dx$$

or

$$\frac{k_s}{k_v} \int_0^x \frac{1}{x} dF_v = \int_0^x dF_s = F_s(x)$$

(assuming particle shape is invariant with size so that  $k_s/k_v$  is constant).

So the surface distribution can be found from the area under a plot of  $1/x$  versus  $F_v$  multiplied by the factor  $k_s/k_v$  (which is found by noting that  $\int_{x=0}^{x=\infty} dF_s = 1$ ).

Column 7 of Table 1W5.1 shows the area under  $1/x$  versus  $F_v$ . The factor  $k_s/k_v$  is therefore equal to 0.0278. Dividing the values of column 7 by 0.0278 gives the surface distribution  $F_s$  shown in column 8. The surface distribution is shown graphically in Figure 1W5.2. The shape of the surface distribution is quite different from that of the volume distribution; the smaller particles make up a high proportion of the total surface. The median of the surface distribution is around  $35\ \mu\text{m}$ , i.e. particles under  $35\ \mu\text{m}$  contribute 50% of the total surface area.

(c) The harmonic mean of the volume distribution is given by:

$$\frac{1}{\bar{x}_{\text{hV}}} = \int_0^1 \left( \frac{1}{x} \right) dF_v$$

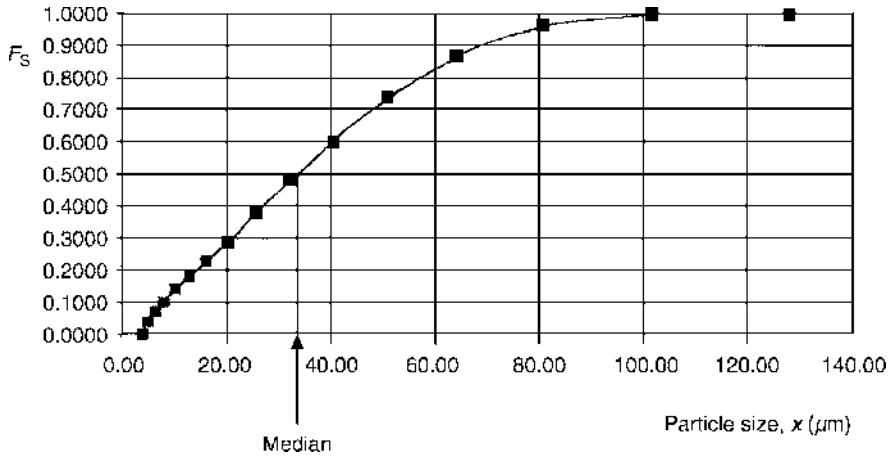


Figure 1W5.2 Cumulative surface distribution

This can be calculated graphically from a plot of  $F_v$  versus  $1/x$  or numerically from the tabulated data in column 7 of Table 1W5.1. Hence,

$$\frac{1}{\bar{x}_{hV}} = \int_0^1 \left(\frac{1}{x}\right) dF_v = 0.0278$$

and so,  $\bar{x}_{hV} = 36 \mu\text{m}$ .

We recall that the harmonic mean of the volume distribution is equivalent to the surface-volume mean of the population.

(d) The arithmetic mean of the surface distribution is given by:

$$\bar{x}_{aS} = \int_0^1 x dF_s$$

This may be calculated graphically from our plot of  $F_s$  versus  $x$  (Figure 1W5.2) or numerically using the data in Table 1W5.1. This area calculation as shown in Column 9 of the table shows the cumulative area under a plot of  $F_s$  versus  $x$  and so the last figure in this column is equivalent to the above integral.

Thus:

$$\bar{x}_{aS} = 36.4 \mu\text{m}$$

We may recall that the arithmetic mean of the surface distribution is also equivalent to the surface-volume mean of the population. This value compares well with the value obtained in (c) above.

**WORKED EXAMPLE 1.6**

Consider a cuboid particle  $5.00 \times 3.00 \times 1.00$  mm. Calculate for this particle the following diameters:

- (a) the volume diameter (the diameter of a sphere having the same volume as the particle);

- (b) the surface diameter (the diameter of a sphere having the same surface area as the particle);
- (c) the surface-volume diameter (the diameter of a sphere having the same external surface to volume ratio as the particle);
- (d) the sieve diameter (the width of the minimum aperture through which the particle will pass);
- (e) the projected area diameters (the diameter of a circle having the same area as the projected area of the particle resting in a stable position).

### Solution

(a) Volume of the particle =  $5 \times 3 \times 1 = 15 \text{ mm}^3$

$$\text{Volume of a sphere} = \frac{\pi x_v^3}{6}$$

$$\text{Thus volume diameter, } x_v = \sqrt[3]{\frac{15 \times 6}{\pi}} = 3.06 \text{ mm}$$

(b) Surface area of the particle =  $2 \times (5 \times 3) + 2 \times (1 \times 3) + 2 \times (1 \times 5) = 46 \text{ mm}^2$

$$\text{Surface area of sphere} = \pi x_s^2$$

$$\text{Therefore, surface diameter, } x_s = \sqrt{\frac{46}{\pi}} = 3.83 \text{ mm}$$

(c) Ratio of surface to volume of the particle =  $46/15 = 3.0667$

$$\text{For a sphere, surface to volume ratio} = \frac{6}{x_{sv}}$$

$$\text{Therefore, } x_{sv} = \frac{6}{3.0667} = 1.96 \text{ mm}$$

(d) The smallest square aperture through which this particle will pass is 3 mm. Hence, the sieve diameter,  $x_p = 3 \text{ mm}$

(e) This particle has three projected areas in stable positions:

$$\text{area 1} = 3 \text{ mm}^2; \text{ area 2} = 5 \text{ mm}^2; \text{ area 3} = 15 \text{ mm}^2$$

$$\text{area of circle} = \frac{\pi x^2}{4}$$

hence, projected area diameters:

$$\text{projected area diameter 1} = 1.95 \text{ mm};$$

$$\text{projected area diameter 2} = 2.52 \text{ mm};$$

$$\text{projected area diameter 3} = 4.37 \text{ mm}.$$



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**TEST YOURSELF**

- 1.1 Define the following equivalent sphere diameters: *equivalent volume diameter*, *equivalent surface diameter*, *equivalent surface-volume diameter*. Determine the values of each one for a cuboid of dimensions 2 mm × 3 mm × 6 mm.
- 1.2 List three types of distribution that might be used in expressing the range of particle sizes contained in a given sample.
- 1.3 If we measure a number distribution and wish to convert it to a surface distribution, what assumptions have to be made?
- 1.4 Write down the mathematical expression defining (a) the quadratic mean and (b) the harmonic mean.
- 1.5 For a give particle size distribution, the mode, the arithmetic mean, the harmonic mean and the quadratic mean all have quite different numerical values. How do we decide which mean is appropriate for describing the powder's behaviour in a given process?
- 1.6 What are the golden rules of sampling?
- 1.7 When using the sedimentation method for determination of particle size distribution, what assumptions are made?
- 1.8 In the electrozone sensing method of size analysis, (a) what equivalent sphere particle diameter is measured and (b) what type of distribution is reported?

**EXERCISES**

- 1.1 For a regular cuboid particle of dimensions 1.00 × 2.00 × 6.00 mm, calculate the following diameters:
  - (a) the equivalent volume sphere diameter;
  - (b) the equivalent surface sphere diameter;
  - (c) the surface-volume diameter (the diameter of a sphere having the same external surface to volume ratio as the particle);
  - (d) the sieve diameter (the width of the minimum aperture through which the particle will pass);
  - (e) the projected area diameters (the diameter of a circle having the same area as the projected area of the particle resting in a stable position).

[Answer: (a) 2.84 mm; (b) 3.57 mm; (c) 1.80 mm; (d) 2.00 mm; (e) 2.76 mm, 1.60 mm and 3.91 mm.]

1.2 Repeat Exercise 1.1 for a regular cylinder of diameter 0.100 mm and length 1.00 mm.

[Answer: (a) 0.247 mm; (b) 0.324 mm; (c) 0.142 mm; (d) 0.10 mm; (e) 0.10 mm (unlikely to be stable in this position) and 0.357 mm.]

1.3 Repeat Exercise 1.1 for a disc-shaped particle of diameter 2.00 mm and length 0.500 mm.

[Answer: (a) 1.44 mm; (b) 1.73 mm; (c) 1.00 mm; (d) 2.00 mm; (e) 2.00 mm and 1.13 mm (unlikely to be stable in this position).]

1.4 1.28 g of a powder of particle density  $2500 \text{ kg/m}^3$  are charged into the cell of an apparatus for measurement of particle size and specific surface area by permeametry. The cylindrical cell has a diameter of 1.14 cm and the powder forms a bed of depth 1 cm. Dry air of density  $1.2 \text{ kg/m}^3$  and viscosity  $18.4 \times 10^{-6} \text{ Pa s}$  flows at a rate of  $36 \text{ cm}^3/\text{min}$  through the powder (in a direction parallel to the axis of the cylindrical cell) and producing a pressure difference of 100 mm of water across the bed. Determine the surface-volume mean diameter and the specific surface of the powder sample.

(Answer:  $20 \mu\text{m}$ ;  $120 \text{ m}^2/\text{kg}$ .)

1.5 1.1 g of a powder of particle density  $1800 \text{ kg/m}^3$  are charged into the cell of an apparatus for measurement of particle size and specific surface area by permeametry. The cylindrical cell has a diameter of 1.14 cm and the powder forms a bed of depth 1 cm. Dry air of density  $1.2 \text{ kg/m}^3$  and viscosity  $18.4 \times 10^{-6} \text{ Pa s}$  flows through the powder (in a direction parallel to the axis of the cylindrical cell). The measured variation in pressure difference across the bed with changing air flow rate is given below:

Air flow ( $\text{cm}^3/\text{min}$ )	20	30	40	50	60
Pressure difference across the bed (mm of water)	56	82	112	136	167

Determine the surface-volume mean diameter and the specific surface of the powder sample.

(Answer:  $33 \mu\text{m}$ ;  $100 \text{ m}^2/\text{kg}$ .)

1.6 Estimate the (a) arithmetic mean, (b) quadratic mean, (c) cubic mean, (d) geometric mean and (e) harmonic mean of the following distribution.

Size	2	2.8	4	5.6	8	11.2	16	22.4	32	44.8	64	89.6
cumulative % undersize	0.1	0.5	2.7	9.6	23	47.9	73.8	89.8	97.1	99.2	99.8	100

[Answer: (a) 13.6; (b) 16.1; (c) 19.3; (d) 11.5; (e) 9.8.]

1.7 The following volume distribution was derived from a sieve analysis

Size ( $\mu\text{m}$ )	37–45	45–53	53–63	63–75	75–90	90–106	106–126	126–150	150–180	180–212
Volume % in range	0.4	3.1	11	21.8	27.3	22	10.1	3.9	0.4	0

(a) Estimate the arithmetic mean of the volume distribution.

From the volume distribution derive the number distribution and the surface distribution, giving assumptions made.

Estimate:

(b) the mode of the surface distribution;

(c) the harmonic mean of the surface distribution.

Show that the arithmetic mean of the surface distribution conserves the surface to volume ratio of the population of particles.

[Answer: (a)  $86 \mu\text{m}$ ; (b)  $70 \mu\text{m}$ ; (c)  $76 \mu\text{m}$ .]

