2 Particle characterisation

An obvious question to ask is, 'what is the particle diameter of my powder?' However, the answer is not so simple. Firstly, most materials are highly irregular in shape, as can be seen in Figure 2.1 – where should one make the measurement? Also, if we turn a particle on its side it is likely that the measurement would be different. When we have to verbally provide this information to someone, who cannot see the particle, it becomes almost impossible to describe the particle simply. In engineering, we wish to perform calculations using the diameter; so, we need some simple basis for describing the irregularly shaped particle that can be used in communication and calculations. This is the origin of the concept of the *equivalent spherical diameter*, in which some physical property of the particle is related to a sphere that would have the same property, e.g. the same volume. Volume is easily measured. If the particle is big enough, water displacement would work and the particle volume can be equated to the volume of a sphere. Note that we shall use diameter rather than radius and the symbol x rather than d. Also, it is common practice to talk about particle size, which really means particle diameter.
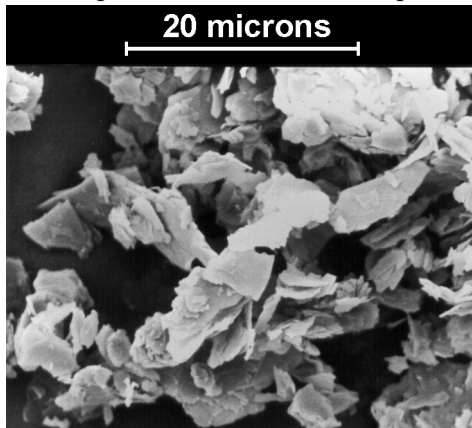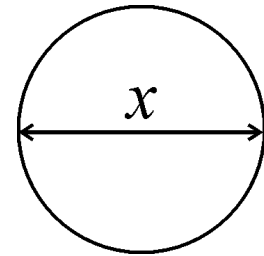
A sphere is a readily understood geometric shape and characterised by a single dimension: its diameter. If you have completed exercise 2.1 you will appreciate that, for the same particle, the equivalent spherical diameter depends upon the property selected for the equivalence. Unless, of course, the particle is spherical in shape. Hence, it is a sphere that all particles are related to and not some other simple geometric shape, e.g. a cube.

Even though we can relate a measured property of our particle to that of a sphere we should still consider particle shape, as it can have an important influence on processing requirements. One simple way to quantify shape is using Wadell's sphericity (Ψ) where:

$$\Psi = \frac{\text{surface area of sphere of equal volume to the particle}}{\text{surface area of the particle}} \qquad (2.1)$$

Fig. 2.1 Talc particles – as in talcum powder

Equations for spheres

circumference is πx

surface area is πx^2

projected[1] area is $\dfrac{\pi}{4} x^2$

volume is $\dfrac{\pi}{6} x^3$

specific surface is $\dfrac{6}{x}$

exercise 2.1
Calculate the equivalent spherical diameter of a 10 μm cube, using equivalence by:
perimeter,
projected area,
surface area,
volume,
specific surface, and
mesh size (i.e. sieve opening size)

e.g. for volume $10^3 = \dfrac{\pi}{6} x_v^{\,3}$

---

[1]projected area – what is observed when looking at a particle using a microscope

This uses the property that a sphere has the smallest surface area per unit volume of any shape. Hence, the value of sphericity will be fractional, or unity in the case of a sphere. There are a variety of accepted shape descriptors and some of these are provided in Table 2.1.

Table 2.1 Common particle shape descriptions

Descriptor	Wadell's sphericity	Example
spherical	1.000	glass beads, calibration latex
rounded	0.82	water worn solids, atomised drops
cubic	0.806	sugar, calcite
angular	0.66	crushed minerals
flaky	0.54	gypsum, talc
platelet	0.22	clays, kaolin, mica, graphite

Particle size analysis equipment is of fundamental importance in particle technology, as it provides the values used in the calculations. However, there are many different types of equipment and they typically provide equivalent spherical diameters based on: volume, projected area, chord length, area related to the light scattering properties of the particle, etc. A table of selected devices, the equivalent spherical diameter measured and links to appropriate images and descriptions are included in Table 2.2.

Table 2.2 Commonly used particle size analysis equipment

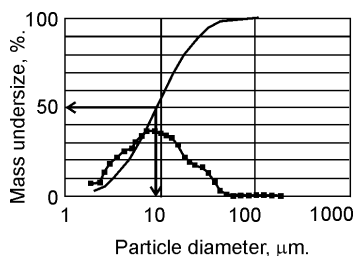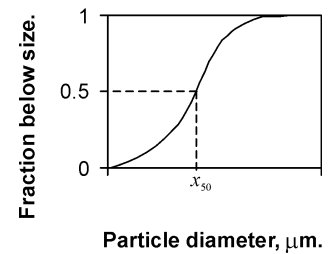| Name	Equivalent spherical diameter	Example URL (www)
microscope	projected area	
sieve	mesh size	www.atmcorporation.com
Lasentec™ (particle chord length FBRM)	length	www.lasentec.com
Malvern™ (Fraunhofer diffraction)	area – light scattering properties	www.malvern.co.uk
Coulter Counter™ (electric zone sensing)	volume	www.beckman.com see Multisizer
Sedigraph™ and Andreasen pipette	sedimentation	www.micromeritics.com see Sedigraph

Fig. 2.2 Example size distribution – Malvern

Note, for descriptions of principles of operation – see the recommended web sites.

In most cases instrumental equipment, such as the Lasentec, Malvern and Coulter Counter, are first calibrated against near monosized polystyrene latex particles of known diameter. Adjustments are made within the operating software so that the equipment provides the calibration material diameter. The equipment provides a full size analysis, an example is provided in Figure 2.2 and Section 2.7. In

order to simplify subsequent description and calculations it is usual to attempt to represent the full particle size distribution by a single diameter. Unfortunately, there are many possibilities and the median, or x_{50}, is commonly (and usually mistakenly) applied. This diameter is easily read off the cumulative distribution curve (see right).
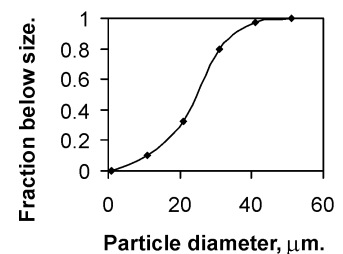
At this stage, the reader may well reflect on the best way to represent the particle size data. There are a number of options to consider: which equivalent spherical diameter, which size analytical equipment and which statistical diameter (mean, median, mode)? *The most appropriate technique for the end use of the data should be used*. For example, if the data is required to design a settling basin for effluent removal a sedimentation diameter is best. If the data is to be used in reaction engineering, or mass transfer calculations, then the equivalent spherical diameter by surface area per unit volume (or mass) is the most appropriate. In the latter case, it is the surface area that dictates the reactivity and is used in the equations for these subjects. However, it should be noted that the surface area exhibited to fluid flow is not, necessarily, the same as that exhibited to reactants in the gaseous phase. Catalyst particles often have a structure that provides internal surfaces for reaction. This will be discussed again in Chapter 7.

2.1 Particle size functions

One of the harder concepts to follow is that the particle size function of the same powder depends upon the basis on which it is reported. This is for similar reasons to the different values found in exercise 2.1. An illustration of this is given in Figures 2.3 and 2.4, and the data used to construct the curves is included. Firstly, it is a trivial matter to take the total number of particles and convert to a fractional amount in each size range (or grade). Hence, it is possible to plot the cumulative number of particles undersize against particle size. Clearly all the particles are less than 51 µm; hence, 100%, or 1.00 as a fraction, are less than this size. At the next size increment boundary, 41 µm, there are 0.975 particles less than this size; so, the cumulative number less than 41 µm is 97.5%, etc. Finally, there are no particles less than 1 µm; hence, the cumulative number undersize is zero at this point.

The above example covers a consideration of the number fraction of particles within size ranges, or grades (for example: 31 to 41 µm). It is quite common to want to consider the size distribution based upon mass rather than number. This can be achieved by conversion from the number distribution as follows. The mass of a single particle is

$$k_v x^3 \rho_s$$

where k_v is the *volume shape coefficient*. For spheres, the volume shape coefficient is $\pi/6$; i.e. it is the factor that the diameter cubed must be multiplied by in order to give the volume of the shape. Hence, the mass of many particles within a size range will be

$$k_v x^3 \rho_s f$$

Upper limit of grade (um)	Number particles in grade	Total number particles	Cumulative number undersize (-)
1	0	0	0
11	200	200	0.1
21	450	650	0.325
31	950	1600	0.8
41	350	1950	0.975
51	50	2000	1
	0	2000	1

Fig. 2.3 Example distribution

where f is the number of particles in that size range. The mass fraction, within a size range and compared to the total mass of the distribution (m_i), will be the mass in the size range in question divided by the mass of the entire distribution; i.e.

$$m_i = \frac{k_v x_i^3 \rho_s f_i}{\sum k_v x_i^3 \rho_s f_i} = \frac{x_i^3 f_i}{\sum x_i^3 f_i} \tag{2.2}$$

Equation (2.2) assumes that the volume shape coefficient and the solid density remain constant throughout all the size grades and, therefore, can be cancelled. Also, in order to apply the equation we need to define a size to use for x_i; this is usually the mid-point of the grade. Thus, in the size grade 31 to 41 μm, the mid-point is 36 μm, and it would be multiplied by the number of particles within the grade to give a value proportional to the mass in that grade. The constant of proportionality being the solid density multiplied by the volume shape factor. On consideration of equation (2.2) it will become apparent that a mass based distribution will provide greater emphasis on the coarser particles than a number based one. This follows from the simple fact that one large particle has much more mass than one small particle: both represent a number distribution of one, but the distribution of mass is much greater in the larger particle grade. Problem 2, page 18, covers the conversion of data from a number to a mass distribution. On consideration of equation (2.2) it should be apparent that a mass distribution is exactly the same as a volume distribution: to convert from volume to mass in each grade the true solids density is used, but to convert from mass in each grade to mass fraction the summation of the mass in each grade is divided into the component grade masses and the density will, in fact, cancel. Hence, the terms volume and mass distribution are used interchangeably.

Mass and number distributions are frequently met within industry, but distributions by length and area also exist. In the latter case it may be by projected area (like looking down a microscope) or surface area; see page 5 for the equations of these for a sphere. The analogue expression for projected area to equation (2.2) is

$$\frac{k_p x_i^2 f_i}{\sum k_p x_i^2 f_i} = \frac{x_i^2 f_i}{\sum x_i^2 f_i} \tag{2.3}$$

where k_p is the projected area shape factor, $\pi/4$ for spheres.

In the construction of Figure 2.3 the cumulative number undersize curve was considered. It is possible to convert the data to mass fractions, using equation (2.2), and to plot the cumulative mass undersize. Likewise, length and area undersize curves can be plotted and these are illustrated in Figure 2.4. So, *for the same powder* we have illustrated four different functions to represent the size data. Each function will have a different mean, median, etc. So, it should be becoming apparent why representing, or designing, systems with particles in them is difficult! In correspondence it is most important to specify whether the distribution is by number, area, mass, etc.

Upper limit of grade (μm)	Cumulative length undersize (-)	Cumulative area undersize (-)	Cumulative mass undersize (-)
1	0.000	0.000	0.000
11	0.025	0.005	0.001
21	0.175	0.092	0.047
31	0.690	0.577	0.467
41	0.952	0.920	0.878
51	1	1	1
	1	1	1

Fig. 2.4 Sizes for the same powder – different functions

Considering the data used to construct Figure 2.3, it would be possible to plot the number of particles within a size grade as a histogram, rather than the cumulative data considered so far. Further, if the mid-points of the size grades are used, it is possible to plot a smooth curve to represent the distribution of particle sizes on an actual number or number fraction basis. This is illustrated in Figure 2.5. In summary, we have two types of curves to represent the particle size data: the cumulative curve that starts at 0 and goes up to 1 and the distribution, or frequency, curve that starts at 0 goes through a maximum and then returns to zero. The latter curve is often illustrated as a bell shaped curve, whereas the cumulative curve is usually an 'S' shaped curve. Both of these curves are obtained from the same original set of data, so it is logical to deduce that there is a simple mathematical relation between them. It should be easy to spot that the number fraction distribution, or frequency, curve in Figure 2.5 can be used to construct the cumulative curve illustrated in Figure 2.3: for a given particle size the number fractions of particles below that size are simply added together. At the *top size* (i.e. x_{max}) all the number fractions added together must equal unity because all the distribution is less than that size.
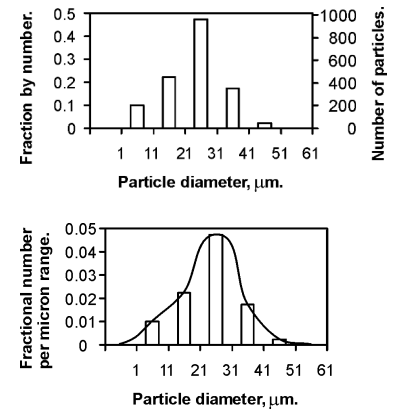
Distribution curves similar to Figure 2.5 are useful for comparison purposes and for modelling particle processes, as we will see in later chapters. In Figure 2.5 it is easy to see which grade contains the mode and it would be possible to compare this figure with a similar distribution curve to compare size distributions. The distribution curve in Figure 2.5 has been *normalised* to take into account the micron range; i.e. the total area under the curve is unity. There are two main reasons for this procedure: one reason is illustrated by Figure 2.6, the other is discussed later under Figure 2.7. The same overall data was used to plot Figures 2.6 and 2.5, but the interval sizes considered were changed: the lowest two intervals were added together and the 21 to 31 μm class was divided equally in two. Clearly, this is the same powder, but we can make the distributions look very different by our choice of interval, or class, sizes. The data in Figure 2.6 hasn't been divided by the micron range. Thus, one reason for normalisation is to produce a fair basis for comparison between the distributions as, after normalisation, the curves would be similar.

2.2 Algebraic representation of size functions

In the previous section the cumulative and distribution functions to represent particle size data were introduced. Also, conversion between distributions by number, length, area and volume were considered. For these conversions from a number distribution, we must raise the particle diameter to the power 0, 1, 2 and 3 in order to convert to a number, length, area and volume distribution respectively. This gives us a convenient shorthand by which to represent the distribution data and a starting point for algebraic manipulation; for the frequency curves:

Fig. 2.5 Histogram and distribution curve for data used in Figure 2.3

exercise 2.2
What is the approximate area under the curve in Figure 2.5?

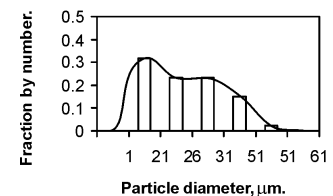| Upper limit of grade (μm)	Number particles in grade	Total number particles
1	0	0
21	650	650
26	475	1125
31	475	1600
51	350	1950
51	50	2000
61	0	2000

Fig. 2.6 Same data as Figure 2.5 but different class sizes

$n_0(x)$ represents a number distribution

$n_1(x)$ represents a length distribution

$n_2(x)$ represents an area distribution

$n_3(x)$ represents a volume or mass distribution

Similarly, the cumulative size functions are represented by:

$N_0(x)$ represents cumulative number

$N_1(x)$ represents cumulative length

$N_2(x)$ represents cumulative area

$N_3(x)$ represents cumulative volume or mass

In fact, the distribution functions $n_*(x)$ represents the fractional number/etc. of particles *per micron range*. Hence, integrating $n_0(x)$ with respect to particle diameter from x_{min} to x_{max} will provide the total fractional number of particles below size x_{max}; which is, of course, unity. Thus, the mathematical relation between $n_0(x)$ and $N_0(x)$ is formally written as

$$n_0(x) = \frac{dN_0(x)}{dx} \qquad (2.4)$$

i.e. the cumulative curve is the integral form of the distribution curve and the distribution curve is obtained by differentiating the cumulative curve. This has some logic, as the cumulative S shaped curve starts at low sizes with a small positive gradient, goes through a period of high rate of change with size, then ends with a period of a small positive gradient again. Hence, the distribution curve has the characteristic bell shape: low values – high values – low values.

The above size functions and distribution curves are generic: they do not imply a specific functional form for the equations. However, numerous functions have been applied to particle size distributions over the years. We will consider only the most common ones here, but before providing the equations it is worth mentioning that the functions have good practical use when trying to understand what is taking place within a process. For example, during crushing it may be possible to inspect the size distribution of the mill product and fit two, or more, well-known distribution functions, suitably weighted in terms of x% of one and y% of the other. This would probably give rise to a bi-modal distribution curve and this implies that more than one crushing mechanism is taking place in the mill. Alterations of the mill conditions could increase the bimodal nature, or remove it altogether. A careful track, and modelling, of the size distribution data enables the significance of the change in mill conditions to be evaluated. Hence, process decisions can be arrived at from the distribution data. This principle is illustrated in Chapter 11.
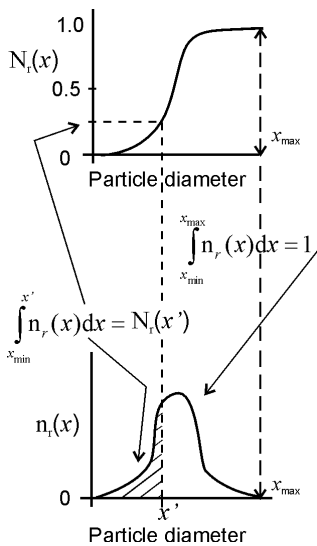

Fig. 2.7 Illustration of the relation between the cumulative and distribution curves – after normalisation

By *normalising* the distribution curve (dividing by the micron range) it is possible to easily relate the two types of curves: the cumulative is the integral of the distribution curve, up to any size *x*, and the distribution curve is the differential of the cumulative curve.

Rosin-Rammler Bennett – cumulative function

$$N_3(x) = 1 - \exp\left[-\left(\frac{x}{x_{63.2}} \right)^n \right] \qquad (2.5)$$

where $x_{63.2}$ represents the particle size at which 63.2% of the distribution lies below and *n* is a constant called the uniformity index.

Gaudin-Schuhmann – cumulative function

$$N_3(x) = \left(\frac{x}{x_{100}}\right)^n \qquad (2.6)$$

where x_{100} represents the top particle size of the distribution and n is again a uniformity constant.

Broadbent-Callcott – cumulative function

$$N_3(x) = \frac{1 - \exp(-x/x_{100})}{1 - \exp(-1)} \qquad (2.7)$$

This is a single parameter model: only the top-size is required. All previous models need two parameters.

Gaudin-Meloy – cumulative function

$$N_3(x) = 1 - \left(1 - \frac{x}{x_{100}}\right)^n \qquad (2.8)$$

Normal Distribution function (Figure 2.8)

$$n_3(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left(\frac{-x - x_{50}}{2\sigma^2}\right) \qquad (2.9)$$

In a normal distribution the median, mode and mean are all the same. This is still a two parameter model: x_{50} (median size) and x_{84} is used to determine the standard deviation from

$$\sigma = x_{84} - x_{50} \qquad (2.10)$$

this uses the property that 68% of the distribution lies one standard deviation away from the mean and the distribution is symmetrical.

Mean value

2.5%   2.5%

95% of distribution is ⁻2 times the standard deviation from the mean

Fig. 2.8 Illustration of the Normal distribution

Log-normal Distribution function (Figure 2.9)

$$\sigma_g = \frac{x_{84}}{x_{50}} \quad (2.11); \qquad x_m = cx_{50} \qquad (2.12)$$

This distribution is biased towards the coarser end, again x_{50} and x_{84} define the required parameters, but a geometric standard deviation and mean are calculated. In practice, the distribution has to be truncated at some maximum and minimum sizes.

$$b = \frac{1}{2\ln^2 \sigma_g} \quad c = \exp(-1/2b) \quad Z = (b/\pi)^{0.5} \exp\left(\frac{-1/4b}{x_m}\right)$$
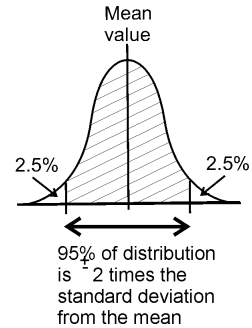
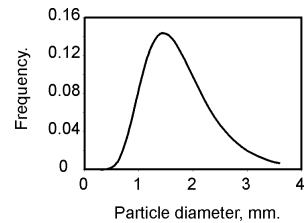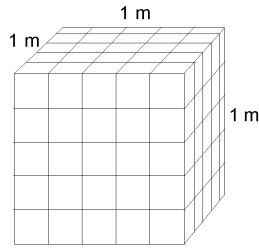$$n_3(x) = Z\exp[-b\ln^2(x/x_m)] \qquad (2.13)$$

Fig. 2.9 Illustration of the log-Normal distribution

Cube: volume   1 m³
     surface area   6 m²
     specific surface 6 m⁻¹

When separated into parts:

Cube: volume   1 m³
     surface area   30 m²
     specific surface 30 m⁻¹

2.3 Specific surface area per unit volume

The surface area of a single sphere is πx^2 and the volume of a sphere is $\pi x^3/6$; so, the specific surface area per unit volume (S_v) for a single spherical particle is the former divided by the latter, or

$$S_v = \frac{6}{x} \tag{2.14}$$

The specific surface area per unit mass (S_w) for a single spherical particle is: S_v/ρ_s. Hence, the SI units are m^{-1} and $m^2\,kg^{-1}$, respectively. It has become conventional to abbreviate the full name to simply *specific surface* and, throughout the rest of this book, when specific surface is referred to it implies by volume and not mass. It is a very important parameter because of its relevance to fluid flow problems and reactivity. In the former case it is the friction of fluid flowing over the surface of the particles that gives rise to an energy loss, or pressure drop. In the case of reactivity, a high specific surface is useful for the provision of many reaction sites in catalysts. For the same volume of solids small particles have a much higher surface area than larger ones – see the simple illustration on the left.

The specific surface can be extended to a full size distribution by considering the total surface area divided by the total volume of the distribution, for spheres

$$S_v = \frac{\pi \sum \bar{x}_i^{\,2} f_i}{\frac{\pi}{6} \sum \bar{x}_i^{\,3} f_i} = \frac{6 \sum \bar{x}_i^{\,2} f_i}{\sum \bar{x}_i^{\,3} f_i} \tag{2.15}$$

The fractional number of particles (n_i) could be used instead of the actual number within the increment because

$$f_i = n_i \sum f_i \tag{2.16}$$

and, after substitution, the total number of particles would appear on both the top and bottom of the equation and cancel. In many cases the distribution data is in mass, rather than number, terms. Equation (2.15) can be simply converted to mass terms by first considering the definition of mass fraction within an increment, equation (2.2), and rearranging to provide

$$\bar{x}_i^{\,2} f_i = \frac{m_i}{\bar{x}_i} \sum \bar{x}_i^{\,3} f_i$$

which can be substituted into equation (2.15). The summation of the entire distribution volume is now on both the top and bottom of the resulting equation and cancels, to leave the simple equation

$$S_v = 6 \sum \frac{m_i}{\bar{x}_i} \tag{2.17}$$

So, unexpectedly, it is a simpler calculation to determine the specific surface from a mass, rather than a number, distribution. A similar derivation can be written using the continuous functions introduced in Section 2.2. The analogues to equations (2.15), (2.2) and (2.17) are

$$S_v = \frac{6 \int x^2 \mathrm{n}_0(x)\mathrm{d}x}{\int x^3 \mathrm{n}_0(x)\mathrm{d}x} \tag{2.18}$$

$$n_3(x) = \frac{x^3 n_0(x)}{\int x^3 n_0(x)\mathrm{d}x} \tag{2.19}$$

$$S_v = 6\int \frac{n_3(x)}{x}\mathrm{d}x \tag{2.20}$$

We can now define another equivalent spherical diameter that may be used to represent the entire size distribution: the spherical particle diameter that has the same specific surface as that of the distribution. Rearranging equation (2.14) provides this value

$$x_{Sv} = \frac{6}{S_v} \tag{2.21}$$

This is called the *Sauter Mean Diameter*, and is sometimes represented as $x(3,2)$ in some distribution data. It is of great importance in particle technology because it is the most appropriate equivalent spherical diameter to represent the size distribution in fluid flow calculations. An example of this is given in Section 3.4 in the next chapter.

2.4 Distributions: moments and conversions

In statistical terms the moment (M_n) of a probability function $P(x)$ taken about a point a is defined by

$$M_n = \int(x-a)^n P(x)\mathrm{d}x \tag{2.22}$$

hence if a is equal to the mean and the first moment is considered (n=1) then the result of equation (2.22) will be zero. The second moment (n=2) will result in the variance (standard deviation squared).

In particle technology we often need to integrate distribution functions, see equation (2.19) for example. If, in equation (2.22), the point a is taken to be zero then the moment is

$$M_n = \int x^n P(x)\mathrm{d}x$$

but the probability function could be by number, mass, etc. Hence, we need to include this in our specification as follows

$$M_{k,r} = \int_{x_{min}}^{x_{max}} x^k n_r(x)\mathrm{d}x \tag{2.23}$$

In the case of k=0, then we have the simple result

$$\int_{x_{min}}^{x_{max}} n_r(x)\mathrm{d}x = 1 \tag{2.24}$$

which is valid regardless of the basis of the distribution data (number, mass, etc.) because the probability function is based on fractional number (mass, etc.) per micron range. Hence, an integration, or summation, of all the fractional numbers per micron range, from the minimum size to the maximum size, will result in the fractions adding up to unity. Graphically, this is the same as saying that the area under the distribution curve must be equal to unity, see Figure 2.7.

In the non-trivial case (k≠0), it is possible to encounter functions such as

> **Recommendation**
> Leave Sections 2.4 and 2.5 until after you have completed the Problems.

$$\frac{\pi}{6}\rho_s \int_{x_{min}}^{x_{max}} x^3 n_3(x) dx \tag{2.25}$$

which represents the *mass weighted mean particle mass* for a distribution, that is of some importance in solid/solid mixing. The weighting is towards the mass distribution because of the term $n_3(x)$ and it is the mean particle size of the distribution multiplied by the volume shape factor and solid density – thus giving the mean particle mass.

Conversion between distributions was first introduced in equation (2.2) and its analogue (2.19). In general, we have

$$n_r(x) = \frac{x^r n_0(x)}{\int_{x_{min}}^{x_{max}} x^r n_0(x) dx} = \frac{x^r n_0(x)}{M_{r,0}} \tag{2.26}$$

Substituting equation (2.26) into (2.23) results in

$$M_{k,r} = \frac{\int x^k x^r n_0(x) dx}{M_{r,0}} = \frac{M_{k+r,0}}{M_{r,0}} \tag{2.27}$$

Using equation (2.27) it is easily possible to show that

$$M_{-1,3} = \frac{M_{2,0}}{M_{3,0}}$$

which is consistent with equations (2.18) and (2.20) combined.

Another illustration of conversion between distributions is to change a mass distribution into a number one, considering both discrete data and continuous functions as follows. If we substitute equation (2.25) into (2.2) and use mid-points to represent the increment we arrive at the following equation for mass fraction

$$m_i = \frac{k_v x_i^3 \rho_s f_i}{\sum k_v x_i^3 \rho_s f_i} = \frac{\bar{x}_i^3 n_i}{\sum \bar{x}_i^3 n_i} \tag{2.28}$$

where n_i represents the fraction by number of the distribution within the increment considered. We must now rearrange for the number distribution

$$n_i = \frac{m_i}{\bar{x}_i^3} \sum \bar{x}_i^3 n_i \tag{2.29}$$

At first sight, equation (2.29) is without solution because the number fraction appears on both sides, but the summation will result in a constant which is multiplied by all the incremental values of mass fraction divided by mid-points. Hence, simply dividing the mass fraction in the increment by the mid-point cubed, summing all these values up and then dividing through by this summed value will provide the number fraction for each increment, as the constant value (i.e. the summation) will cancel. Rearranging equation (2.26) provides the analogue equation for the continuous functions

$$n_0(x) = \frac{n_3(x)}{x^3} M_{3,0}$$

2.5 Means of a distribution

The simplest way to calculate the mean of a distribution, based on discrete data, is to use the fractional amount by number, mass, etc. thus

$$\bar{x}_0 = \sum \bar{x}_i n_i \qquad (2.30)$$

$$\bar{x}_3 = \sum \bar{x}_i m_i \qquad (2.31)$$

where equation (2.30) is mean particle size by number and (2.31) mean by mass. Further consideration of the means by this approach are given in Problem 3, at the end of the chapter. However, a more complete approach considers that the distribution could be weighted by number, mass, etc. and that we require a single particle diameter that is representative of the entire distribution based on number, mass, etc.; i.e.

$$\bar{x}_{k,r}{}^k = \int_{x_{\min}}^{x_{\max}} x^k n_r(x) \mathrm{d}x = M_{k,r} \qquad (2.32)$$

Hence, the general equation for mean particle size is

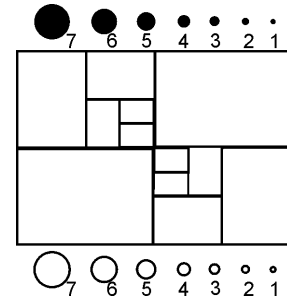$$\bar{x}_{k,r} = \sqrt[k]{M_{k,r}} \qquad (2.33)$$

Fig 2.10 Graticule used to compare particle sizes on a microscope – BS 3260

2.6 Image analysis and particle shape

Microscope counting of particles is an important characterisation technique (BS 3260), but when performed manually it is slow and prone to operator variation. The technique compares the size of the particle to circles of standard areas, see Figure 2.10, and to count the number of particles between each size range. Modern techniques have removed the tedium of assessing particle size against standard areas by eye and use computer image recognition for the size analysis. However, they still usually rely on there being no overlapping particles present. Other forms of electronic image analysis are possible, including shape assessment. Hence, the microscope (with computer) is an important tool in particle characterisation. A microscope is essential for the quick check of material to assess, for example, if the particles may be aggregating during a water based particle size analysis by one of the instrumental techniques mentioned in Table 2.2. An optical microscope is generally used for particles down to 1 micron in diameter, but the theoretical limit is slightly below this. Electron microscope images provide high resolution much this size, see Figure 2.1 for an example.

In automated microscope analysis three diameters are often used: Feret, Martin and Image Sheared. These diameters are illustrated in Figure 2.11, together with the equivalent spherical diameter by projected area. The Feret diameter is the perpendicular distance between two parallel tangents on opposite sides of the particle, where the tangents are drawn perpendicular to a fixed direction during the travel of the microscope stage. The Martin diameter is the length of a line which bisects the image of the particle. The line may be drawn in any direction but, once chosen, the direction must remain constant for all measurements of the distribution. The Image Shear diameter is
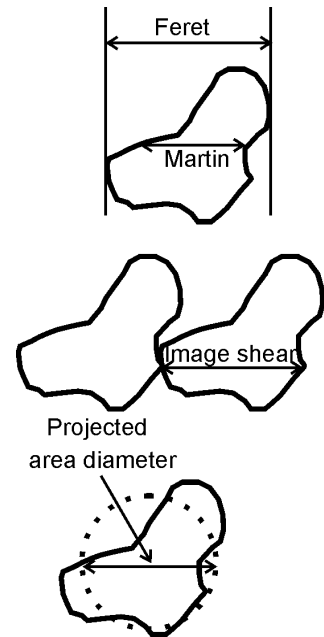

Fig. 2.11 Statistical diameters used in image analysis: Feret; Martin and Image Sheared

Fig. 2.12 Fractal analysis of a particle: perimeter increases with smaller step sizes (λ) –rate of increase in perimeter against decreasing step size gives the fractal dimension. Maximum particle dimension normalises step size and perimeter.

computer generated by taking the image of the particle and moving it until it is just touching the original image at an edge. The displacement distance to achieve this is the image sheared diameter.

Particle shape is a very complex property to measure. In practice, the shape descriptors and approach commonly used was covered in equation (2.1) and Table 2.1. Shape can considerably influence particle processing; an example is in the sedimentation of particles with a low sphericity – these tend to descend like a feather oscillating from side to side, whereas isotropic particles of the same size would fall in a single dimension. An alternative approach to the description of particle shape has considered using ratios based on *elongation*, particle length to width, and *flakiness*, particle width to thickness. Particle shape has also been described by fractal analysis, as illustrated in Figure 2.12. However, there has been only very limited success in quantitatively relating particle shape, by any of these means, with particle processing requirements or products.

2.7 Example interpretation of distribution data

Figure 2.13 shows the tabular results from a Malvern Mastersizer size analysis. The 'Results Statistics' are reproduced in Table 2.3 for further discussion. The full data can be seen as a jpg file and the associated spreadsheet calculation for this section can be found from http://www.midlandit.co.uk/particletechnology/chapter2.htm

Table 2.3   Illustration of Malvern Results Statistics

Density = 1.000 g/cub.cm	Specific S.A. = 0.7046 sq m / g
Mean diameters: D(v,0.1)=3.25 um	D(v,0.5)=29.8 um D(v,0.9)=107.1 um
D(4,3)=44.11 um D(3,2)=8.52 um	

Malvern use D to represent particle size, against the use of *x* in this book. The term D(v,0.1) represents the particle diameter at which 10% of the distribution is below; and similarly for the other percentiles on the same row of the table. Thus, the median size is 29.76 μm. The mean diameter by volume (i.e. same as mass) is 44.11 μm, calculated by equation (2.31), and the mean diameter by surface area to volume (i.e. Sauter mean) is 8.52 μm, calculated by equations (2.17) and (2.21). The specific surface area comes from equation (2.17), and the value of 0.7046 square metres per gram is reported. The equation provides a value in μm^{-1} but, for the Malvern calculation, the density has been given the value of 1 gram per cubic metre so that the reported specific surface is, in fact, in μm^{-1}. Using the size increment data from the analysis and the equations in a spreadsheet provides the values of 44.2 and 8.5 μm for the mean by mass and Sauter mean diameters, which is within rounding errors to that reported here by the Malvern software.

2.8 Summary

When using particle size distribution data the most important considerations to be aware of are: how the data was obtained, i.e. the basis of the analysis by number, mass, etc., and how the data will be used. For routine quality control, or assurance, the first principle is not so important as simply a change in results will be enough to signal a change in quality. However, these principles are important for equipment design, or understanding. For design, the equivalent spherical diameter, or distribution data, appropriate to the end use should be used; e.g. settling diameter for sedimentation basin design, Sauter mean for pressure drop calculations.

From a practical point, the repeated testing of samples of the same powder in a size analysis laboratory can result in significantly different particle size distribution data. This is a consequence of variations in both sampling as well as size analysis. Modern computer based electronics has resulted in size analysis equipment providing many channels, 256 and 1024, and the use of spreadsheets has made manipulation of the data relatively easy to accomplish. Hence, the previous practice of fitting mathematical functions, or undertaking graphical analysis of the data, in order to obtain values such as the specific surface, has become less important. The level of effort required to obtain a graphical solution is rarely justified when the distribution data changes from one analysis to another. Invariably, the requirement is for a single value to be used for design (such as an average particle diameter), or comparison purposes. This value can be adequately obtained from a spreadsheet analysis. However, given the possible lack of repeatability of the size analysis, and sampling, it may be wise to obtain a range of values for the average diameter; as a small change in its value might significantly influence a design variable, see Chapter 3 for an illustration of this in the context of fluid flow.

For a more thorough modelling of particle processes, the particle size distribution functions covered in Section 2.2 have considerable utility, as they can be used to investigate, or model, what happens within a process to the different sizes of particles.

Fig 2.13 Example tabular data output from Malvern – the original is available for viewing at: www.midlandit.co.uk/particletechnology The Results Statistics section is reproduced in Table 2.3

2.9 Problems

1. The following size distribution was obtained for glass spheres by microscope examination. Complete the table rows showing the relative number of particles, the cumulative number undersize and the relative (i.e. fractional) number per micron range.

Size range (µm):	<5	5-7	7-10	10-15	15-20	20-30	30-40	40-50	>50
Number in range:	0	50	150	200	55	45	20	5	0
Relative number:	0	0.0952							
Cumulative no. undersize:	0	0.0952						1.000	
Relative no. per µm:	0	0.0476							

The mass of a single particle is:

$$\frac{\pi}{6}x^3\rho_s$$

where x is particle diameter and ρ_s is solid density. Thus it is possible to convert a number distribution to a relative mass distribution by multiplying the number of particles by the mass of a single particle and dividing through by the total mass of the system. The range, or grade, mid-point is used for the particle diameter. Note that the volume shape factor and density cancel out during this operation. The relative number can also be used in preference to the actual number for a similar reason.

$$S_v = 6\sum\frac{m_i}{x_i}$$

where m_i is the relative mass - see Section 2.3

2.

Using the above number distribution data, complete the table:

Size range (µm):	<5	5-7	7-10	10-15	15-20	20-30	30-40	40-50	>50
(mid point)^3.frequency		10800							
Relative mass:	0	0.0039							
Cumulative mass undersize:	0	0.0039						1.000	

3.

The mean particle size by number is best calculated by determining the fractional contribution to the mean from each size range, or grade. This is the product of the relative number and the mid-point size in the grade. Summing all the contributions together gives the mean size. Note that this is mathematically equivalent to the more conventional:

$$\sum f_i x_i / \sum f_i$$

where f_i is the frequency (or number) of occasions that x_i occurs, e.g. you may have used this formula before to calculate an average mark.

select the correct answer from the following

Thus the mean size by *number* is (µm):

a: 12.4   b: 13.5   c: 25.0   d: 28.2

4.

The mean size by *mass*, calculated in a similar way, is (µm):

a: 12.4   b: 13.5   c: 25.0   d: 28.2

5.

The complete distribution specific surface area per unit volume (S_v) is the total surface area divided by the total volume. It may be calculated by several routes, one is shown left. For this distribution S_v is (µm^{-1}):

a: 0.213   b: 0.240   c: 0.264   d: 0.444

Now convert your answer into one with SI units.

6.

You will need the equations relating specific surface (S_v) to number frequency and also to mass fraction (both per micron). See Section 2.3 for the derivation of the last relation starting from the former. For spherical particles the two equations are given in the box on the right.

   These questions are based on an idealised cumulative size distribution that can be represented as a single straight line on the upper figure given to the right.

(i). The cumulative size distribution of a powdered material may be represented as a straight line on a % number undersize versus particle diameter (x) graph passing through the points 0% by number at 1 µm and 100% by number at 101 µm. The equation for $N_0(x)$ is: $N_0(x)$=...

a: x-1   b: x/100-1   c: x/100-0.01   d: x-100

Note, work in fractional terms and NOT percentages.

(ii). The equation for $n_0(x)$ is: $n_0(x)$=...

a: 1   b: 1/100   c: 100   d: x/100-1

(iii). What is the shape of the $n_0(x)$ graph given by your answer to part (ii)?

(iv). The best equation to use for calculation of specific surface is:

a: $S_v = 6 \dfrac{\int_1^{101} x^2 (1/100)\mathrm{d}x}{\int_1^{101} x^3 (1/100)\mathrm{d}x}$ b: $S_v = \dfrac{6}{\int_1^{101} x\mathrm{d}x}$ c: $S_v = 6 \dfrac{\int_1^{101} x^2 \mathrm{d}x}{\int_1^{101} x^3 \mathrm{d}x}$

(v). The specific surface area per unit volume is (µm^{-1}):

a: 0.013   b: 0.045   c: 0.059   d: 0.079

(vi). Now convert your answer into one with SI units:

(vii). If the sphericity of the material is really 0.9 the specific surface is (µm^{-1}):

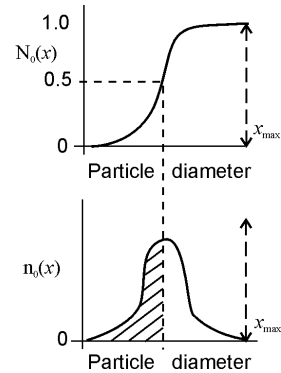a: 0.071   b: 0.088   c: 0.066   d: 0.053

7.

(i). The specific surface of another size distribution having the same limits as that given in question 6 but on a mass distribution $N_3(x)$ basis is (µm^{-1}):

a: 0.013   b: 0.138   c: 0.277   d: 0.554

(ii). The Sauter mean diameter of the distribution in part (i) is (µm):

a: 50.0   b: 43.5   c: 21.7   d: 10.8

Number distribution:

$$S_v = \frac{\pi}{\pi/6} \frac{\int x^2 n_0(x)\mathrm{d}x}{\int x^3 n_0(x)\mathrm{d}x}$$

Mass distribution:

$$S_v = \frac{\pi}{\pi/6} \int \frac{n_3(x)\mathrm{d}x}{x}$$

Relation between two types of distribution curves (given for a number distribution):

$$\frac{\mathrm{d}N_0(x)}{\mathrm{d}x} = n_0(x)$$

The relation between the mass distributions is similar.

8.

The attenuation of a parallel beam of light is used to monitor visibility on an airport approach during fog conditions. It is found, experimentally, that the droplet size distribution of the fog may be represented by the equations

for: $\quad 50 \geq x \geq 15$ μm $\qquad N_0(x) = 0.83 + 0.043\ln(x)$

$\quad 15 \geq x \geq 4$ μm $\qquad N_0(x) = 0.30 + 0.239\ln(x)$

$\quad 4 \geq x \geq 0.5$ μm $\qquad N_0(x) = 0.21 + 0.304\ln(x)$

where $N_0(x)$ is the cumulative fraction by number less than the particle size in μm.

(i). Write down below the equations for $n_0(x)$

for: $\quad 50 \geq x \geq 15$ μm $\quad n_0(x)=$

$\quad 15 \geq x \geq 4$ μm $\quad n_0(x)=$

$\quad 4 \geq x \geq 0.5$ μm $\quad n_0(x)=$

(ii). The specific surface area per unit volume (S_v) of the fog is the total surface area divided by the total volume, in number distribution terms (assuming spherical droplets):

$$S_v = 6 \frac{\int\limits_{0.5}^{4} x^2 n_0(x)\mathrm{d}x + \int\limits_{4}^{15} x^2 n_0(x)\mathrm{d}x + \int\limits_{15}^{50} x^2 n_0(x)\mathrm{d}x}{\int\limits_{0.5}^{4} x^3 n_0(x)\mathrm{d}x + \int\limits_{4}^{15} x^3 n_0(x)\mathrm{d}x + \int\limits_{15}^{50} x^3 n_0(x)\mathrm{d}x}$$

Just considering the size grade $50 \geq x \geq 15$ μm the area integral in the above equation (i.e $\int x^2 n_0(x)\mathrm{d}x$) is (μm²):

a: 48.91 $\qquad$ b: 12.35 $\qquad$ c: 293.5 $\qquad$ d: 1.286

(iii). The volume integral for the same grade is (μm³):

a: 5895 $\qquad$ b: 48.91 $\qquad$ c: 1743 $\qquad$ d: 102.1

(iv). The area and volume integrals for the other size ranges are as follows:

for: $\quad 15 \geq x \geq 4$ μm $\quad$ area is 24.98 μm² $\quad$ volume is 263.8 μm³

$\quad 4 \geq x \geq 0.5$ μm $\quad$ area is 2.39 μm² $\quad$ volume is 6.473 μm³

The specific surface area per unit volume of the fog is (μm⁻¹):

a: 0.0379 $\qquad$ b: 0.227 $\qquad$ c: 2.96 $\qquad$ d: 0.493

(v). Now convert your answer into one with SI units:

(vi). If the path length is 50 m, the concentration of fog that will reduce the intensity of the beam by a factor of 100 is (-):

a: 1.6x10⁻⁶ $\qquad$ b: 1.6x10⁻⁵ $\qquad$ c: 1.6x10⁻⁴ $\qquad$ d: 1.6x10⁻³

N.B. For Part (vi)

The attenuation of a light beam is given by the Beer-Lambert law:

$$\frac{I}{I_o} = \exp\left[-\frac{1}{4}S_v CL\right]$$

(for convex particles) where C is the volume concentration of the fog, L is the path length, and I and I_o are the intensity of the transmitted light and the intensity of the light with no fog present respectively.