UNIT 4

4.1 DETERMINATION OF PARTICLE CHARACTERISTICS.

4.1.1 Introduction.

In this unit some of the methods used to measure particle characteristics are reviewed. The particle characteristics considered are particle size and surface area. Particle size distribution functions are introduced to enable the extrapolation to sub sieve size to characterise the size distribution of particle where it is not possible measure the size using mathematical modelling.

4.1.2 Objectives.

By the end of this unit you should be able to;

1 Define the terms used in particle size characterisation for better liberation
2 Define various methods used in sub sieve size analysis

4.1.3 Reflection.

What do understand of particle size characteristics and the shapes analyses in mineral processing and in particular pharmaceutical applications? Particle characteristics and sizes are very important in such areas as making pellets, tablets in pharmacy and packing things like metal final products as in the case of bars, sheets or granules and in many forms. These properties are also very important in such as classification and in the monitoring various useful properties in quality maintenance and control.

4.2 MEASUREMENT OF SIZE DISTRIBUTION.

4.2.1 METHODS DEPENDING ON GEOMETRICAL SIMILARITY.

4.2.1.1 Sieving (d_A).

This is the simplest and most widely used sizing method. Woven wire sieves of square apertures are normally used down to about 45 um. Punched plates are used where larger apertures, of a few um upwards, are required whereas for smaller apertures where small tolerances (e.g. $\pm$ 2 um) are required electroformed (micromesh) sieves are utilised. However, the latter are expensive and fragile and are not normally used for routine laboratory work.

4.2.1.2 Standard Meshes.

A Wide variety of meshes is currently in use. The main ones are:-
U.S. sieve series
American Tyler series
British Standard series
German Standard and
French series.

The International Standards Organisation has recommended and international standard series corresponding to the U.S. series. This series is based on 1mm sieve with successive apertures following a $(2)^{1/4}$ progression. Apertures greater than 1mm are expressed in mm, those smaller than this are in um.

a) **Wet Sieving**: A common problem in sieving is the adherence of very fine particles to larger particles or to each other by electrostatic attraction or surface tension. These fine particles are more quickly eliminated by wet sieving. Wet sieving is also used for pigments and usually helps to disperse aggregates.

b) **"Endpoint" of a sieving test**: Main aim is to ensure that a "near mesh" particle which can only pass through if favourably presented is given sufficient time to pass through. This is normally defined by the condition at which the rate of passage of the particles is reduced to a specific weight. One way of ensuring this is by shaking for five minutes and noting the amount passing in subsequent two minutes. If this amount > 0.2% of sample weight the five minutes state is repeated followed by another retest of two minutes.

c) **Weight of sample**: Sieves must not be overloaded. Usually up to 100g is sufficient (for 8 inch screens) if more than 50% of sample is retained.

4.2.1.2 Microscopic Methods (d_a).

This is the most direct method of measuring particle size and has the advantage that qualitative information about shape can also be obtained. The main method used is usually the direct measurement technique in which the sample is directly examined under the microscope. A slide is usually prepared with the particles uniformly and randomly distributed in their stable orientation and are viewed in transmitted light.

Because it is impractical to measure the size of every particle on the slide representative areas are chosen in a predetermined fashion. The actual number of particles need to be measured. With a typical size distribution of ground material more than 600 particles are usually measured.

A conventional optical microscope with a standard graticule is normally adequate. The method is not suitable for particles < 0.8 um, and should preferably be restricted to particles less than 75 um.

A big problem of microscopic particle size measurement is deciding what diameter to measure. Several diameters can be defined.

a) **Martin's diameter:** M is the length of the line which bisects the image of the particle.
b) **Feret's diameter:** F is the distance between two tangents on opposite sizes of the particle, parallel to some fixed direction.
c) **Projected Area diameter:** d_a is generally found to be the most satisfactory although originally M or F was measured. Hence d_a is the most often measured using manual or semi-automatic techniques.

Measurement can easily be achieved by, for example, counting squares in a calibrated graticule.

In the semi-automatic methods settings of the microscope are made usually by the operator and the instrument records the results. Notable is the Zeiss-Ender microscope. They have the advantage that the operator can decide on the treatment of aggregates and distinguish foreign bodies e.g. human hair. The automatic methods on the other hand can count many more particles. They are usually based on a moving spot of light (or slit), recording the uninterrupted light falling onto a photocell.

Polished mounted and Thin sections are not usually used to determine particle size although they are frequently used to determine grain size and liberation sizes. The information obtained however has to be stereologically transformed in order to relate the apparent sizes measured to true sizes.

4.2.2 METHODS BASED ON HYDRODYNAMIC SIMILARITIES.

The methods are based on the settling rate of particles in a fluid. It is a characteristic of the methods therefore that they measure d_{st}.

In stoke's equation, which is written as

$$v = \left(\frac{\rho_s - \rho_l}{18\eta} \right) g d^2$$

η, ρ_s, ρ_l, p *and* g *are cons* tan *ts for a given system*

$$\therefore d_{st} = k(v)^{1/2} = k\left(h/t\right)^{1/2}$$

Where h is the distance (m or cm) fallen in time t (s). The assumptions made in the derivation of Stoke's law are:

a) laminar flow i.e. Re = $\frac{Vd}{\eta} < 0.2$

b) the particle is smooth and spherical

c) the particle is not influenced by both inter-particle or wall affects

d) the terminal settling velocity has been reached.

Condition (a) is easy to fulfil because the critical d_{st} diameter (i.e. largest diameter to which Stoke's law would apply) can be calculated.
Condition (c) is normally satisfied if the diameter of the vessel is 100d, and if the particle concentration is < 2% by volume.

The general method consists of measuring the terminal settling velocity of the particle and calculating d_{st} from the above relation. Particles must be properly dispersed. For particles > d_{st} critical, sieving is normally used. It is possible, however, to use hydrodynamic methods by employing a more viscous fluid or using non-laminar flow equations. These, however, are both difficult alternatives.

In practice hydrodynamic methods fall into two categories:

4.2.2.1 Sedimentation: fluid is relatively motionless and particles fall thorough it by gravity or centrifugal forces.

4.2.2.2 Elutriation: grading of particles effected by upward moving current of fluid.

i) **Sedimentation Methods.**

These methods are non-fractionating except for decantation technique. They can be subdivided into two groups:

a) **Cumulative Methods** in which the total mass fraction sedimented from a definite height is measured as a function of time.

b) **Incremental Methods** in which the concentration of solids at a given level is periodically determined.

i) Cumulative Methods.

The most popular of the methods is the sedimentation balance method. This consists of a pan suspended near the base of a sedimentation chamber with the pan connected to some device for recording mass gain.

A disadvantage of the method is that it is prone to errors arising from the immersion of the pan in the suspension which varies in concentration as sedimentation proceeds.

Sedimentation columns are another method. These are much simpler and are glass columns designed so that the sediment can be drawn off at the bottom at required time intervals. Although they are not susceptible to disturbance during sample withdrawal, errors may arise from the taper at the bottom of the tube (e.g. hang-ups).

In the interpretation of the data, it is necessary to realise that the total weight of the particles which settle during time t includes those particles not large enough to fall the full distance h, but which started part of the way down. If d_t is the particle size of those particles whose settling rate is just sufficient to fall through h in time t, the basic equation relating to cumulative methods is:

$$W = P - t\frac{dp}{dt}$$

Where

W = fractional weight of particles of size greater than d_t deposited in time t.

P = fractional weight deposited in time t.

In principle (p) and (t) are measured and (w) calculated from the equation. The cumulative % oversize can also be obtained graphically from a plot of p against t.

Graphical approach: Avoids problems associated with integration of the basic cumulative equation.

Let x be the size of particles which could have theoretically fallen the whole distance h in time t. At time t, all particles > x have settled but at this time particles < x are settling at the rate $\frac{dp}{dt}$. In time t the fractional amount of particles < x that settled is given by $t\frac{dp}{dt}$

$\therefore$ Total weight of particles settled during t is

$$P = W + t\frac{dp}{dt}$$

A sedimentation curve of P against t is plotted as shown in figure 12.

For a specific fall time t_x, through h, we can find the corresponding theoretical particle size from the relation

$$d_{st},x = k\left(h/t_x\right)^{1/2}$$

In order to obtain W_x, the fraction of size > $d_{st,x}$ a tangent is drawn to the sedimentation curve at tx and extrapolated to the ordinate. W_x is given by the intercept on the ordinate as shown in the figure.

The proportion of oversize particles (> x) in the original sample is given by: W/P_{max}

Where P_{max} is the settled weight if the smallest particle in the suspension has been given sufficient time to settle. The cumulative oversize (weight %) is given by:

$$Y = W/P_{max} \times 100$$

P_{max} is approximately the original sample weight.

The cumulative weight % undersize is given by (100-V)

Thus a conventional cumulative weight % finer (or greater) that particle size can be plotted. Furthermore, the weight frequency curve can be obtained from the two cumulative curves.

Repeated Decantation is another cumulative method which is widely used because it does not require special equipment other than a set of beakers. The sediment in beaker A is freed of particles > d_{st} by decanting into a fresh beaker. The re dispersed suspension is allowed to settle again prior to further decantation. The procedure is repeated several times with different settling times producing different size fractions. The method is however often used to prepare close size fractions (e.g. for chemical analysis) than as an analytical method.

For cumulative methods, the concentration of solids required is lower than for incremental methods with balance techniques being the most attractive.

a) Incremental Methods.

The principle is that at any given depth in the sedimentation vessel the maximum possible particle size instantaneously present at that depth can easily be calculated from a knowledge of the fall height and the time of fall, using stoke's equation. If the suspension concentration is therefore measured at that depth and after different time intervals the different mass fractions collected will be devoid of particle sizes greater than the calculated d_{st} values. Hence a plot of C/C_o against d_{st} is effectively a cumulative undersize mass fraction. C is the suspension concentration at time t and C_o is the initial particle concentration.

- The different incremental methods available differ only in the way the concentration is measured. One of the most widely used is the **Andreasen pipette** shown below:

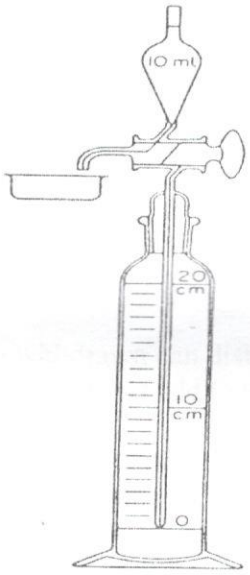

FIG. 4.9. Andreasen pipette.

This is a fixed depth pipette in which a known volume of suspension is withdrawn at known times after stirring has ceased. The sample is evaporated to dryness and the solids weighed.

Calculation of results.

The results obtained are directly cumulative weight % undersize which are plotted as such.

The basic calculation is:

$$p_n = \frac{w_n}{w_s} \times \frac{V}{V_f} \times 100$$

Where p_n = cumulative weight % of particles smaller than each selected d_{st} value

W_n = weight of solids in sample (g)