Date

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Aim :-

Determination of particle size and particle size distribution using sieving method.

Reference :-

A Text book of Pharmaceutics - I Dr. A.A Hajare, Nirali Prakashan Page 11 to 14.

Requirements :-

Chemicals :- A sample of any granules or powder.

Glassware and apparatus :- Sieve set, Mechanical Sieve shaker, Weighing balance.

Theory :-

The powder contains particles of uniform size whereas granules are non-uniform in size. When sample of powder or granules is poly disperse, For application purpose they must be characterized for terms size, shape and particle size distribution. The particle size is expressed in terms of diameter. The particle size distribution (i.e. the number of particles of different sizes), is responsible for

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important physical and chemical properties such as : Mechanical bulk behaviour, Surface reaction, taste miscibility, Filtration properties and conductivity. The examples clearly show important. It is to have knowledge of the particles or distribution particularly within the context of quality assurance in the production of bulk goods. If the particle distribution changes during the manufacturing process then the quality of the finished product will also change. Only continuous monitoring of the particle size distribution can guarantee a constant product quality. Since all particles or granules in sample are never being uniformly spherical their size is expressed in terms of equivalent spherical diameter. The equivalent spherical diameters includes Surface diameter, Volume diameter, Projected diameter, Stokes diameter, Volume surface diameter, Volume spherical diameter, Projected diameter, Stokes diameter, Volume surface diameter and surface-surface diameter. For sample with non-spherical particles or powder, their size is measured horizontally by arbitrarly fixing line across the centre of the particle. These measurements are expressed as Feret diameter. Mathindia meter and the projected area diameter.

There is no universal method available for determination of particle size of a polydisperse powder. It is either derived from the arithmetic mean or harmonic mean or geometric mean. Arithmetic mean of a powder is defined as the geometrical sum of the various particle size divided by the number of particles. It is mathematically written as

$$\text{Arithmetic mean} = \frac{\text{Sum of particle size}}{\text{Number of particle size}} \quad ---(1.1)$$

Edmonodson's general equation for the average particle size is.

$$d_{mean} = \frac{\left(\Sigma nd^{P+F}\right)^{1/P}}{\left(\Sigma nd^{1/P}\right)}$$

Where n is number of particles, d is diameter or the equivalent diameter and p is an index related to size of an individual particle. The value of P can be 1, 2 or 3. If P = 1 it means QF geometoric means surface area and P = 3 means the term F is a Frequency factor which can be 0, 1, 2 and 3.

More useful information is obtained when sample segregation method are used, for example sieving method where in number of particles of weights within a certain range is plotted as function of mean particle size method that gives reproduce results in short time but unfortunately it is suitable for fine particles samples.

Procedure :-

1. Arrange the set of sieves (IP or USP standard) in descending order. (Place sieve number 10 at top, below which place sieve numbers 20, 40, 60, 80, 100 respectively and 120 at the bottom).
2. Weigh accurately, the given sample (100g) and place in the top sieve. Cover sieve with lid to avoid loss during shaking.
3. Operate the sieve-shaking machine for 5 min.
4. Collect fractions of samples retained on each sieve and on receiver at the bottom of set.
5. Weigh samples using weighing balance.
6. Calculate percent frequency of each size of particles.

Table 1.1

Data of powder analysis obtained from sieving method:

Sieve size in mm (Passed/retained)	Weight (g) retained on sieve	Cumulative weight % weight retained
Size class (mm)	B 3 [%]	Q 3 [%]
(A)	(B)	(C)
< 0.045	6.0	6.0
0.045 - 0.063	8.0	14.0
0.063 - 0.125	15.0	29.0
0.125 - 0.250	19.0	48.0
0.250 - 0.500	25.0	73.0
0.500 - 1.000	13.0	86.0
1.000 - 2.000	12.0	98.0
2.000 - 4.000	2.0	100.0
> 4.000	0.0	100.0

7. Plot histogram (bar graph) of fractions (B) and % weight retained (C) as given in table 1.1.

8. Draw the % cumulative frequency curve (Q3) from the cumulative % weight retained against the nominal sieve mesh (x).

For Example, the data of powder analysis is given in table 1.1. below

The result of the evalution are presented in tubular and graphical forms, As can be seen from fig 1.1 the fractions (P3) are normally shown as a histogram (bar graph). The cumulative frequency curve (Q3) from the percentage mass fractions is plotted against the nominal sieve mesh (x).

Calculations:

1.) Calculations of cumulative %.weight retained:

% weight retained on sieve	$= \dfrac{\text{weight retained on sieve}}{\text{Total weight of powder}} \times 100$

2.) Calculation of % cumulative frequency:

% Cumulative frequency	= % weight retained on previous sieve + % weight retained on sieve under consideration

In this example 25% of the sieved material has a particle size between 0.250 mm and 0.500mm form the cumulative frequency curve. for example: It can be seen that 73% of the material is smaller than 0.500 mm. The median, particle diameter X 50 value of the sieved particle fraction, in the example shown above indicates that 50% of the sample is larger or smaller than X = 0.750 mm.

Observations:-

(i) Weight of sample 100g
Time of shaking 5 min.

Note:-

The sample retained on sieve number 10 is oversize and therefore not considered. The sample collected at receiver is passed through sieve number 120 and therefore it is written against sieve number 120. The size is reported as undersize.

Observation Table :
(For any set of IP standard sieves)

S.No.	Sieve no.	Sieve Size (μm)	Sieve no. passed/ retained	Sieve size in μm Passed/ retained	Weight (g) retained on sieve	Cumulative % weight retained	Cumulative frequency of under size
(1)	(2)	(3)	(4)	(5)	(6)	(7).	(8)
1	10	1700	10 - 22	1700 - 710	2	2 %	2 %
2	22	710	22 - 36	710 - 425	12	12 %	14 %
3	36	425	36 - 44	425 - 355	13	13 %	27 %
4	44	355	44 - 60	355 - 250	25	25 %	52 %
5	60	250	60 - 85	250 - 180	19	19 %	71 %
6	85	180	85 - 100	180 - 150	15	15 %	86 %
7	100	150	100 - 120	150 - 125	8	8 %	94 %
8	120	125	120 - Reciever	125 - Reciever	6	6 %	100 %

Precautions:-

1. Shake powders or granules for short period.

2. Arrange sieves in perfect descending order of their size from top to receive.

3. If the difference between the original sample weight and the sum of the individual fraction is greater than 1% then according to Deutsches Institut für Normung (German Institute for Normalization) DIN 66 165, the sieving process must be repeated.

4. Avoid loss of sample during shaking and weighing

Result:-

The average diameter of the sample was found to be ...... µm. In this example ..... % of the sieved material has a particle size between ...... µm and ...... µm. From the cumulative frequency curve. It can be seen that ..... % of the material is smaller than ..... µm.

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