Presentation on,

"MICROMERITICS"

Presented by-

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Defination-

"The science and technology of small particles is known as micromeritics."

Particle size importance in pharmacy

- *Physical property of powder*

- *Flow property of powder*

- *Rate of dissolution*

- *Chemical property of powder*

- *Rate of absorption*

- *Elegance of pharmaceutical preparation*

- *Stability of system.*

- *Extraction and drying process*

- *Adsorption capacity*

Properties of powder

Fundamental properties

⬇

Individual particle.

Derived properties

Derived

From

⬇

Fundamental properties

Fundamental properties:-

1. Particle size and size distribution
2. Particle shape
3. Particle surface area
4. Particle weight
5. Particle number

Derived properties:-

1. Density of powders
2. Flow properties of powders
3. Porosity
4. Bulkiness

1.Particle size

Denoted in **micrometers**

One micrometer is equal to **10^{-3} mm or 10^{-6} m**

One millimicrometer is called one **nanometer (nm)**

One nanometer = **10^{-9} m or 10^{-6} mm or 10^{-3} μm**

1 m   = 1000 mm

1 mm = 1000 μm

1 μm = 1000 nm

Particle size determination (PSD) Methods-

1. Optical Microscopy
2. Sieving Method
3. Sedimentation Method
4. Conductivity Method

1. OPTICAL MICROSCOPY

➢Particle size in the range of 0.2 – 100 μm can be measured.

➢This method gives number distribution which can be converted to weight distribution

➢Optical microscope lens has limited resolving power

➢Advanced microscopes have better resolving power and can measure size in nano range: Ultramicroscope, Electron microscope-Scanning Electron microscope (SEM), Transmission Electron microscope (TEM).

Application:

 Particle size analysis in suspensions, aerosols, globule size analysis in emulsion.

Procedure:

- Eye piece of the microscope is fitted with a micrometer.
- This eye-piece micrometer is calibrated using a standard stage micrometer.
- The powder sample is dispersed in a suitable vehicle in which it does not dissolve and its properties are not altered.
- This sample is mounted on a slide and placed on the stage under the objective of microscope.
- Around 300-500 particles are visualized. Their diameter is noted and mean is computed.

Calibrated Using A Standard Stage Micrometer.
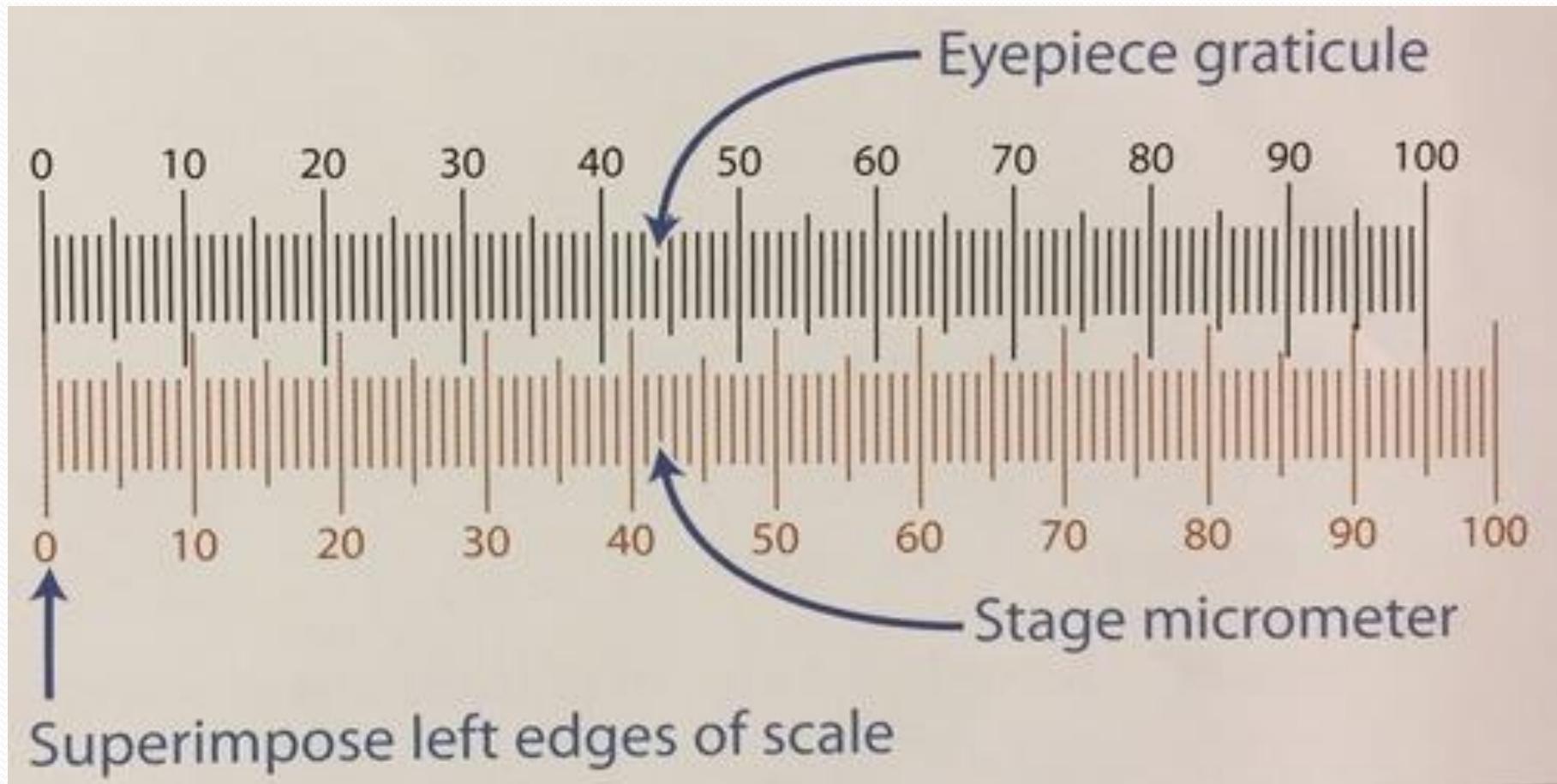
Advantages-

➢One can view particles

➢Any aggregates detected

➢Contamination of particles detected

➢Use of cover slip for arresting motion of particles

➢Easy and simple

Disadvantages-

➢Length and breadth can be detected but depth or thickness of particles cannot be measured

➢Slow- time consuming , tedious, inaccurate Number of particles to be measured is more

➢Large sample required

2. <u>Sieving Method-</u>

➤ Particle size in the range of 30–5000 µm can be measured.

➤Sieving method is an ordinary and simple method.

➤It is widely used as a method for the particle size analysis

➤Sieve analysis is usually carried out using dry powders.

➤Although, for powders in liquid suspension or which agglomerate during dry sieving, a process of wet sieving can be used.

➤Sieving method directly gives weight distribution.

➤It find application in dosage form development of tablets and capsules.

➢Normally, 15% of fine powder should be present in granulated material to get proper flow of material and achieve good compaction.

➢Thus percent of coarse, moderate, fine powder is estimated by this method.
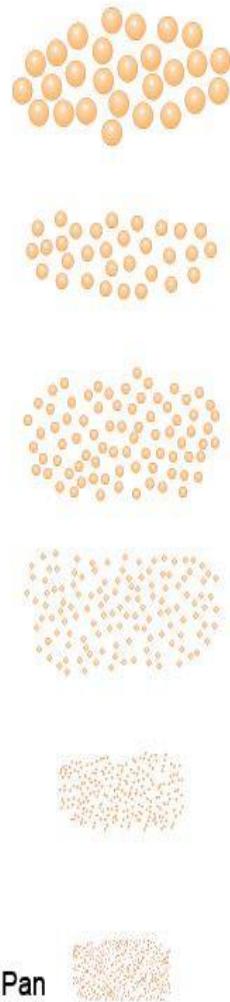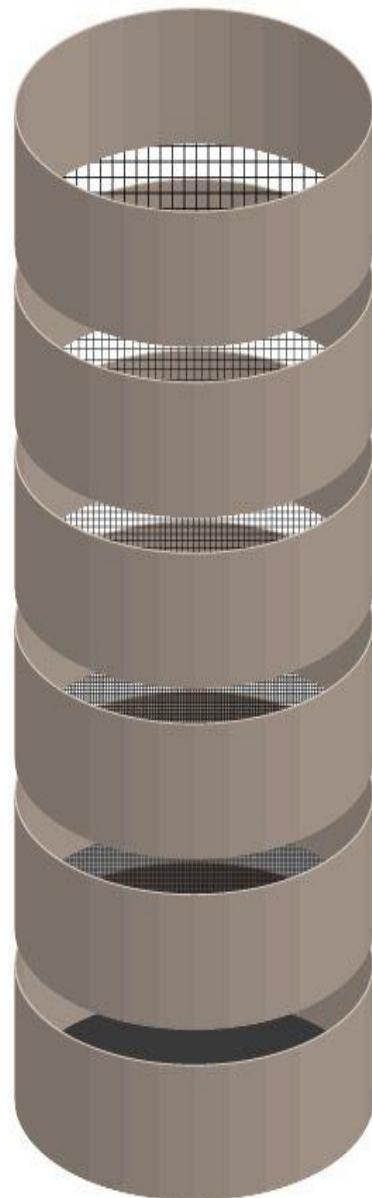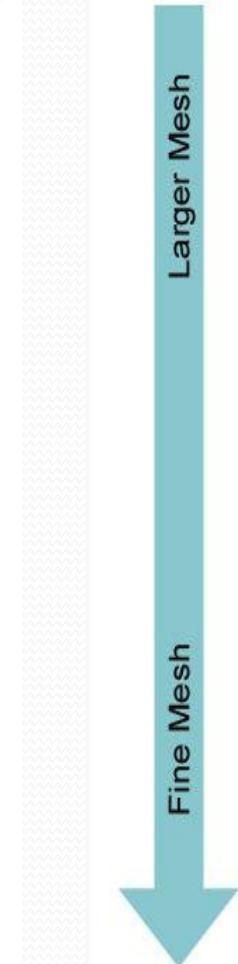
➢Sieve analysis utilizes a wire mesh made of brass, bronze or stainless steel with known aperture (hole) diameters which form a physical barrier to particles.
➢The standard sieve sizes are as per the pharmacopoeia
Most sieve analyses utilize a series, stack (layer) of sieves **which have the coarser mesh at the top of the series and smallest mesh at the bottom above a collector tray.**

(The mesh size goes on decreasing from top to bottom)

METHOD OF SIEVING

1. Mechanical dry sieving method

A-Agitation method (Oscillation,Vibration,Gyration)
B-Brushing method
C-Centrifugal method

2. Wet sieving method

Method:

A sieve stack usually comprises 6-8 sieves.

Powder is loaded on to the coarsest sieve of the stack and then it is subjected to mechanical vibration for specified time.

After this time, the powder retained on each sieve is weighed.

The particles are considered to be retained on the sieve mesh with an aperture corresponding to the sieve diameter.

The size is estimated as per the standards given in pharmacopoeia.

TABLE 6-5

Designations and Dimensions of I.P. Specification Sieves

Sieve number	Aperture size micrometer	Sieve number	Aperture size micrometer
10	1700	44	325
12	1400	60	250
16	1000	85	35
22	710	100	36
25	600	120	34
30	500	150	36
36	425	170	35

Care should be taken to get reproducible results.

➢The type of motion, time of operation, speed, weight of powder should be fixed and standardized.

Advantages-

Inexpensive, Simple, Rapid, Reproducible results.
(if parameters are standardized)

Disadvantages-

lower limit is 30 microns
Powder if moist, can cause clogging of apertures
Attrition between particles during the process may cause size reduction giving inaccurate results.

3. Sedimentation Method

➤ In this method particle size can be determined by examining the powder as it sediments out.

➤ **Sample preparation: Powder is dispersed in** a suitable solvent

➤ If the powder is hydrophobic, it may be necessary to add dispersing agent to aid wetting of the powder.

➤ In case where the powder is soluble in water it will be necessary to use non- aqueous liquids or carry out the analysis in a gas.

Principle of Measurement

➢Particle size analysis by sedimentation method can be divided into two main categories according to the method of measurement used.

➢*One of the type is based on measurement of particle in a **retention zone.***

➢*Another type uses a **non-retention measurement zone.***

➢An example of a non-retention zone measurement is known as the pipette method.

Andreasen pipette method:

➤One of the most popular of the pipette methods was that developed by Andreasen and Lundberg and commonly called the Andreasen pipette.

➤*In this method , known volumes of suspension are drawn off and the concentration differences are measured with respect to time.*

➤It involves measuring the % of solids that settle with time in a graduated vessel.

Construction:

➢The Andreasen fixed-position pipette consists of a 200 mm graduated cylinder which can hold about 500 ml of suspension fluid.

➢A pipette is located centrally in the cylinder and is held in position by a ground glass stopper so that its tip coincides with the zero level.

➢A three way tap allows fluid to be drawn into a 10 ml reservoir which can then be emptied into a beaker or centrifuge tube.

Method:

➢A 1% suspension of the powder in a suitable liquid medium is placed in the pipette.

➢At a given intervals of time, samples are withdrawn from a specified depth without disturbing the suspension.

➢The amount of powder can be determined by **weight following drying or centrifuging;** alternatively, chemical analysis of the particles can be carried out.

The particle size is determined in terms of **stokes' diameter** *(**the diameter of a particle measured during** sedimentation at constant rate) using modified* Stokes' equation.

$$dst = 18nh / (p_s - p_f)gt$$
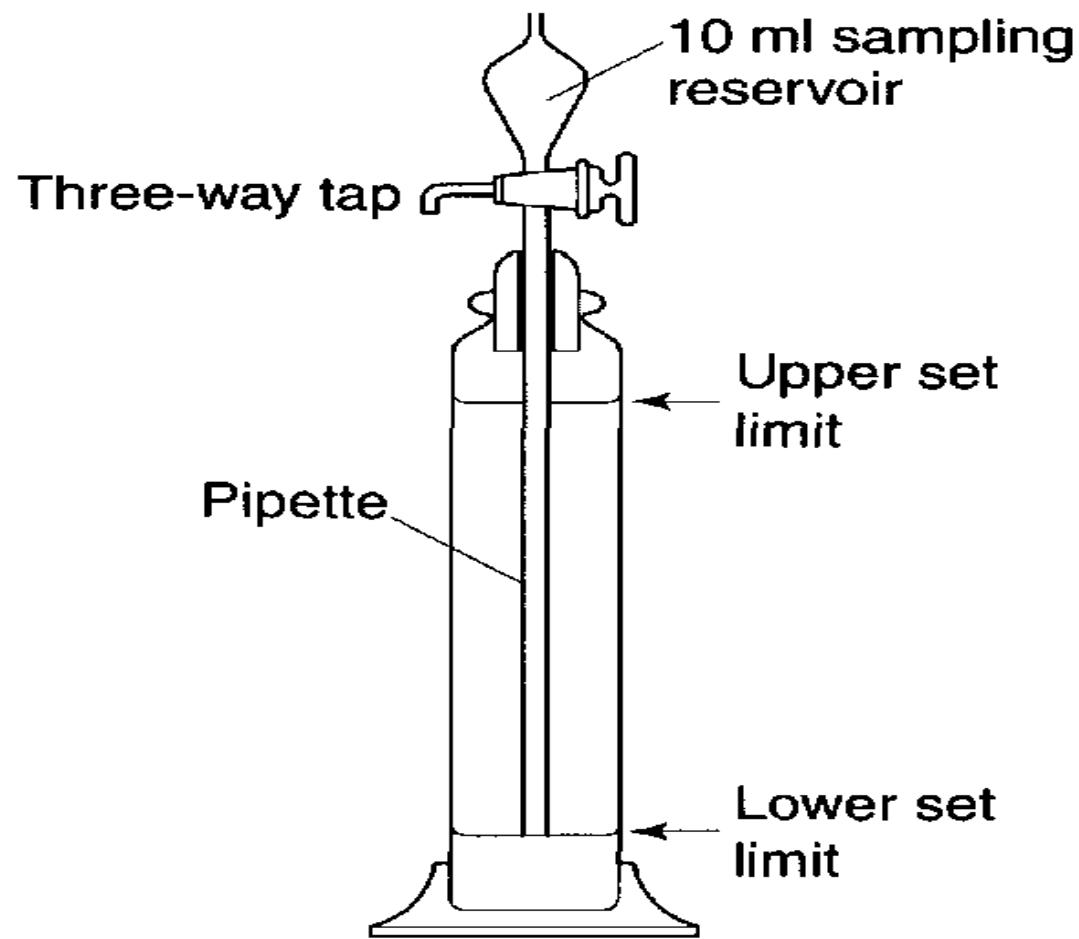
Fig. 10.19   Diagram of Andreasen pipette.

4. Conductivity methods

There are various subtypes. Two popular methods are-
Electrical stream sensing zone method
(Coulter counter)
Laser light scattering methods
*It is based on the principle of **change in light intensity**.*
***The measurement of this change in** light intensity gives estimate of particle size.*
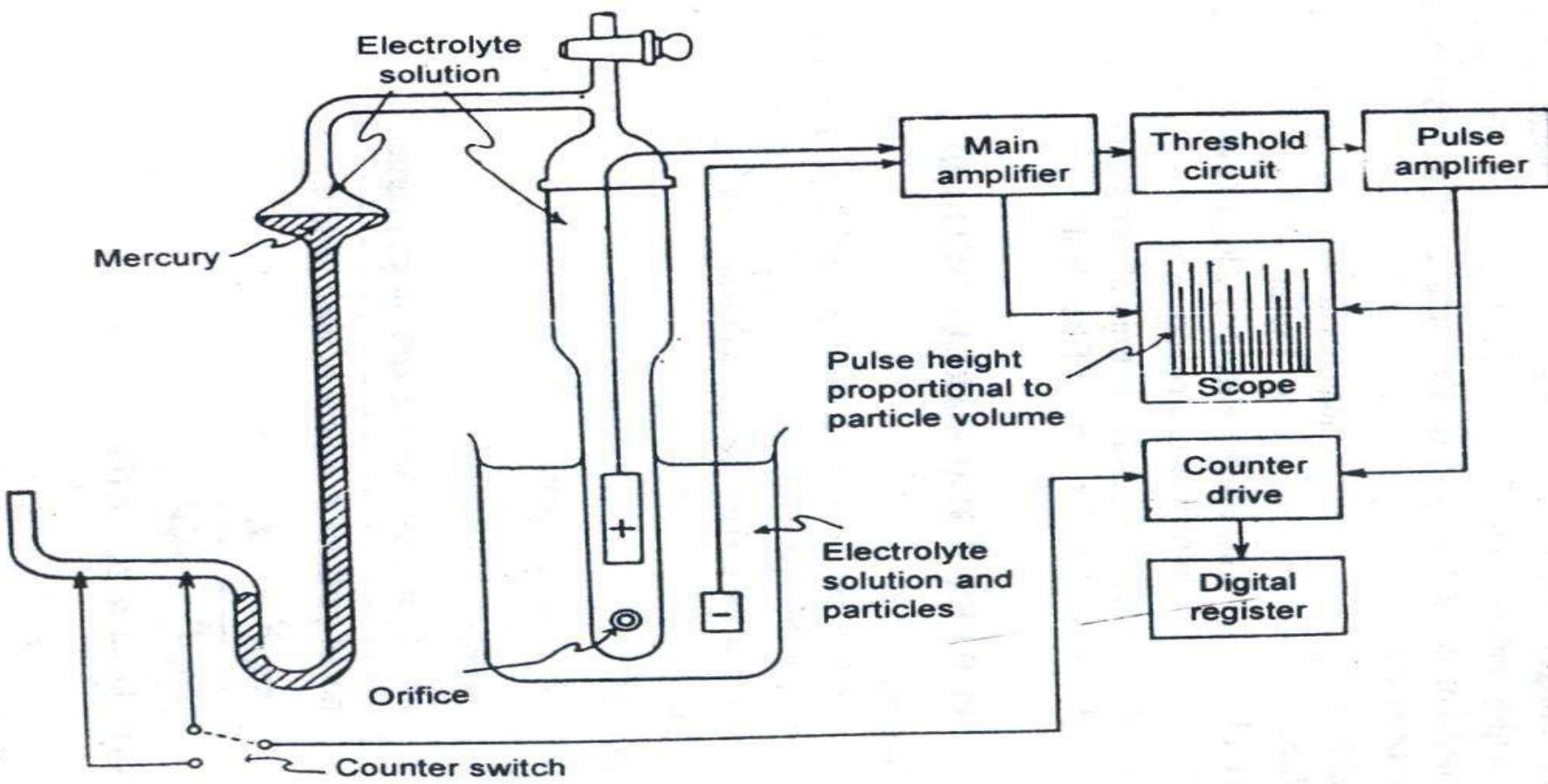
Figure 6-9. Schematic diagram of Coulter counter used to determine particle volume.

Advantage:

➢Particle size ranging from 0.5 – 500 microns

➢Gives number distribution, Particle volume measured and can be converted to diameter.

➢Accurate, sensitive, fast technique.

➢4000 particles per second can be counted.

Disadvantage

Expensive

THANK YOU