

Aim:

~~Suspension~~
~~T/10²~~ Determination of bulk density, true density and porosity.

Reference:-

A Text book of pharmaceuticals -I, Dr. AA Hajare,
Nirali Prakashan, Page 19 to 21.

Requirements :-

- (a) Chemicals :-
- Any powder or granules for example.
 - CaCO_3
 - Starch
 - Kaolin
 - Calamine etc.

(b) Glasswares and apparatus:-

- Graduated measuring cylinder (100 ml)
- Bulk density apparatus
- Density bottle
- Beakers
- Pipette
- Electronic weighing balance etc.

Observations:

- 1.) Weight of powder (w_1) = 25 grams
- 2.) Volume of measuring cylinder occupied by powder (v_1) = ... mL
- 3.) Volume of measuring cylinder occupied by powder after tapping (v_2) = ... mL
- 4.) Volume of density bottle (v_3) = ... mL.
- 5.) Weight of powder in density bottle = 10 grams.
- 6.) Volume of solvent added to density bottle (v_4) = ... mL
- 7.) True volume of powder (v_t) = $v_3 - v_4 \approx 25.5$ mL

Theory:

Solid bodies retain their volume and shape under atmospheric pressure. An important factor in choosing a suitable sample for density determination is the question of whether the density is required as a characteristic of a material or whether density determination is performed to check for defects in a material. The choice of procedure for density determination will depend on this factor as well. The density of particles, powders, and compacts is an important property affecting the performance and function of many pharmaceutical materials. By definition, all density measurements involve the measurement of mass and volume.

Mass is determined with an analytical balance and the key to obtaining reliable density values is in the accuracy and precision of measuring volume.

Bulk density is a characteristic of a volume of divided material such as powders, grains, and granules. It includes the volume of the solid materials, open and closed pores, and the interparticle voids. The total volume of interparticle voids can change with packing, thus leading to the concept of tap density, which measures the volume of a mass of sample after inducing a closer packing of particles by tapping the container.

Calculations:

1.) Calculation of bulk density:

$$\text{Bulk density } (P_B) = \frac{\text{Mass}}{\text{Volume}} = \frac{W_1}{V_2}$$

2.) Calculation of true density:

$$\text{True density } (P_T) = \frac{\text{Weight of Powder}}{\text{True volume of powder}}$$

3.) Calculation of Porosity:

$$\text{Porosity} = \frac{\text{True density} - \text{Bulk density}}{\text{True density}}$$

$$= \frac{P_T - P_B}{P_T}$$

$$\text{Total Porosity} = \text{Porosity} \times 100$$

Taking this method to the extreme leads to the determination of void volume and compressed density after compressing the sample under extreme forces and measuring the volume changes as a function of applied pressure.

True density is the density of the solid material excluding the volume of any open and closed pores. Depending on the molecular arrangement of the material, the true density can equal the theoretical density of the material. True density measurements can be performed on APIs, excipients, blends and monolithic samples such as tablets. In micromeritic studies high-precision gas pycnometer which gives accurate to 0.02% of the sample volume are used to determine true density.

Porosity consists of volume of the pores relative to the envelope volume used to calculate envelope density. The porosity of pharmaceutical materials and medical devices can impact production, material movement, and pharmacokinetic behaviour. Tablet porosity determines the tensile strength (hardness) of tablets for a given composition. Tablet porosity may be regarded as a measure of the tabletting process. Variations in tablet porosity reflect various aspects of tablet press performance. Tablet porosity may relate to tablet disintegration and dissolution. In the case of coated tablets, coating quality may be

affected by tablet porosity.

Procedure:

(A) Determination of bulk density:

1. Take clean and dry measuring cylinder.
2. Weigh accurately 25g of powder (w_1),
3. Place it in dry graduated measuring cylinder and note the volume as V_1 mL.
4. Place the cylinder containing sample in bulk density apparatus. Adjust apparatus and operate it for 100 tapping. Record the volume occupied by the powder as V_2 mL. (Place sample in both cylinders of apparatus to balance the arms.)

(B) Determination of true density:

1. Take a clean and dry density bottle.
2. Accurately measure volume of density bottle (V_3) using solvent upto the mark.
3. Transfer accurately 10 grams of powder to density bottle.

4. Fill density bottle that contain powder with known amount of any solvent in which powder is insoluble. Note the volume of solvent added as (V_4) ml.
5. Repeat this procedure thrice and take average of all to obtain correct data.
6. Calculate true density of powder sample.

(C) Determination of porosity:

1. Calculate porosity and total porosity using the values obtained as bulk volume, true volume, bulk density and bulk density.

Precautions:

1. Record volumes correctly.
2. Perform weighing on calibrated balance.

Result:

1. Bulk density
2. True density
3. Porosity of sample
4. Total porosity of sample

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