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Characterization of Powder Flowability Using FT4 – Powder Rheometer

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Abstract
The understanding of powder flow is also important for mixing, packaging and transportation. And thus, it becomes essential to measure the flow properties of these materials prior to tabletting or capsule filling. There are various methods available to measure the powder flow. The compendial methods include measurement of angle of repose, bulk density, tapped density, carrs compressibility index or hausners ratio [9] and another method like FT4 powder rheometer. The FT4 powder rheometer is designed to characterize powders under various conditions in ways that resemble large – scale production environment. The methods include stability and variable flow rate, shear cell, compressibility, aeration, permeability, wall friction and consolidation. The FT4 has proven application in all powders processing industries, including pharmaceuticals, Fine chemicals, Food, Cosmetics, Powder coating and Additive manufacturing. The FT4 Application extend to Filling, Tablet compression, Hopper flow, Wet granulation end point, Flow additive selection and optimisation, Humidity effects, Electrostatic change, Mixing/ Blending, Feeding, Segregation, Attrition, Dry powder inhalers, Caking, Milling, Wall friction and adhesion, Hopper design, Compact hardness.

INTRODUCTION:

Knowledge of the powder behaviour is complex. It's influenced by a combination of physical properties and the characteristics of the processing equipment and powder flowability cannot be expressed adequately as a single value or index [1]. During processing, powders are subjected to several physical environments requiring different behavioural properties [2]. Multiple approach should be applied in which powders are tested by several methods each evaluating different powder properties relevant to manufacturing [3]. The behaviour of powders in preparation, die filling and powder transfer is important, since their packing structure and density distribution may influence later stages and affect the integrity of the final components [4]. Powder and granular systems must be considered as a multiphase continuum whose behaviour is a function of both the fundamental properties of the powder and individual particles (for example size, shape, surface texture and moisture content) and external factors such as humidity temperature and consolidation history. As such powders and granular materials exhibit diverse and complex behaviour [5]. Powder behaviour is difficult to both predict and control. In order to compare and optimize powders or a bulk solid is necessary to design silos and other bulk solid handling equipment so that no flow problems (flow obstructions, segregation, irregular flow, flooding, etc.). Powder flow is a key requirement for pharmaceutical manufacturing process [6]. Tablets are often manufactured on a rotary multi – station tablet press by filling the tablet die with powders or granules based on volume. The flow of powder from the hopper into the dies often determines weight, hardness and content uniformity of tablets [7]. In case of capsule manufacturing, similar volume filling of powders or granules is widely used. The understanding of powder flow is also important for mixing, packaging and transportation. And thus, it becomes essential to measure the flow properties of these materials prior to tabletting or capsule filling [8]. There are various methods available to measure the powder flow. The compendial methods include measurement of angle of repose, bulk density, tapped density, carrs compressibility index or hausners ratio [9] and another method like FT4 powder rheometer. The FT4 powder rheometer is designed to characterize powders under various conditions in ways that resemble large – scale production environment. The method include stability and variable flow rate, shear cell, compressibility, aeration, permeability, wall friction and consolidation [10].

FACTORS INFLUENCING POWDER FLOW PROPERTIES

The particles alone or complex and rarely defined by an adequate set of descriptors. Particle size distribution has traditionally been considered, and it remains important, but in fact there are many particle properties that will influence the overall behaviour of the powder. Powders normally flow under influence of gravity, dense particles are generally less cohesive than less dense particles of the same size and shape. poor flow may result from the presence of moisture, in which case drying the particles will reduce the cohesiveness with the various apparatus for the measurement of the properties of cohesive powders and the effects on cohesive powders of particle size, moisture, glidants, caking, and temperature [11].

Particles with a high density and a low internal porosity tend to possess free-flowing properties. This can be offset by surface roughness, which leads to poor flow characteristics due to friction and cohesiveness.

Adhesion and cohesion:

The presence of molecular forces produces a tendency for solid particles to stick to themselves and to other surfaces [12].

Cohesion occurs between like surfaces, such as component particles of a bulk solid. Adhesion occurs between two unlike surfaces, for example between a particle and a hopper wall.

Effect of particle size:

Cohesion and adhesion are phenomena that occur at surfaces, particle size will influence the flowability of a powder.Fine particles with very high surface to mass ratios are more cohesive than coarser particles which are influenced more by gravitational forces. Particles larger than 250μm are usually relatively free flowing, but as the size falls below 100 μm are powders become cohesive and flow problems are likely to occur [13]. Powders having a particle size less than 10μm are usually extremely cohesive and resist flow under gravity, except possibly as larger agglomerates.

Effect of particle shape:

Powders with similar particle sizes but dissimilar shapes can have markedly different flow properties owing to differences in inter-particles contact areas [14].

For example: A group spheres has a minimum inter-particle contact and generally optimal flow properties, whereas a group of particle flakes or dendritic particles has a very high surface to volume ratio and poor flow properties.

Density of the particle:

Powders normally flow under influence of gravity, dense particles are generally less cohesive than less dense particles of the same size and shape.

Potential for electrostatic charge:
Some powders become electro statically charged as a result of handling and processing, resulting in a change in their behaviour of the powder.

MEASURING POWDER FLOWABILITY:
Measuring powder flowability using two different methods like
1. Conventional Method
2. FT4 Powder Rheometer (Freeman Technology – USA)

CONVENTIONAL METHOD:
- Particle size distribution
- Angle of repose (AOR)
- Flow meters (critical orifice diameter)
- Tapped density tests (carrs index/hausner ratio)

Particle size distribution – sieving method
The most commonly used method for determining the particle size distribution and simplest method. A powder is placed on the mechanical shaker that is made of a series of screens with sequentially smaller apertures. The most widely used screens are woven wire screens ranging in size starting from 400 opening per inch [15]. In the united states, Tyler standard and US standards (ASTM E11 – 70) are commonly used.

Angle of repose (AOR) – Fixed Funnel Method
The material is poured through a funnel to form a cone. The tip of the funnel should be held close to the growing cone and slowly raised as the pile grows, to minimize the impact of falling particles. Stop pouring the material when the pile reaches a predetermined width. Rather than attempt to measure the angle of the resulting cone directly, divide the height by half the width of the base of the cone [16]. The inverse tangent of this ratio is the angle of repose. The AOR is used in the design of equipment for the processing of particulate solids.

Bulk Density
In a 100 ml graduated measuring cylinder approximately 50% of the cylinder was filled with the granular blend. The apparent weight and volume of the granular blend was noted down.
Bulk density is calculated using following formula
Bulk density (ρ_b) = M/V_0
where M- mass of granules blend (gm),
V_0- apparent volume of powder (mL).

Tapped Density
Approximately 50% of the 100 ml graduated measuring cylinder was filled with granular blend and the volume of mass without compacting i.e. unsettled apparent volume level was noted (V_0) was noted. The cylinder was secured in the holder of tapped density tester apparatus and tapped for 10, 500, 1250 taps and corresponding volumes V10, V500, V1250 were read to the nearest graduated unit (until the difference between succeeding measurements is less than or equal to 2 ml). The tapped density was calculated using the formula:
Tapped density (ρ_t) = M/V_t
Where M- mass of granules blend (gm),
V_t-final tapped volume of powder blend (ml).

Compressibility Index and Hausners Ratio
Mathematically they are calculated using the following formula [17].
Compressibility index (CI) = V_0 - V_t÷ V_0 × 100 or ρ_t - ρ_b÷ ρ_t × 100
Hausner's Ratio (H) = V_0/ V_t
Where V_0 - unsettled apparent volume,
V_t - final tapped volume
ρ_t- tapped density, ρ_b- bulk density

DISADVANTAGES:
- These methods cannot characterize the full range of powder flowability/ cohesion
- Flow meters require the powder or granular material to flow through a funnel and are therefore unsuitable for cohesive powders.
- Tap density testers are also unsuitable for highly cohesive powders as the tapping force is insufficient to overcome the strong interparticular cohesive bonds meaning that the powder bed will not necessarily consolidate when tapped.
- Other issue with this test method when dealing with granular materials as the heavier / denser particles are more likely to pack efficiently under their own mass, resulting in falsely low results.
- Due to particle size limitations, commercially available instruments are not readily available for large granular materials [5].
- There is no standard test for measuring powder flow in all four flow regimes (plastic, inertial, fluidised and entrained flow)

FT4 POWDER RHEOMETER (FREEMEN TECHNOLOGY)
FT4 technology was primarily developed to measure powders under dynamic conditions. This technique is capable of evaluating both free flowing and highly cohesive materials even at larger particle sizes.

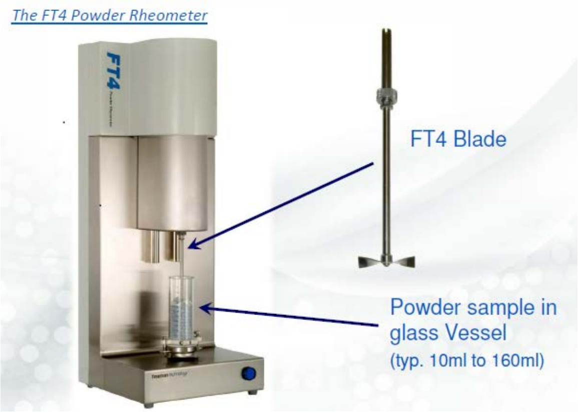

Fig 1. FT4 Powder Rheometer

FT4 is used to measure the static and dynamic testing methods for characterizing powder flow. Designed by freeman technology was used to measure the materials flow properties. Single instrument can be used to monitor the process from raw material to the finished product [18]. The sample is placed in a spilt vessel as shown in fig which allows a precise volume to be attained and the density of the sample can be measured with unprecedented levels of accuracy.

FT4 – METHODOLOGIES

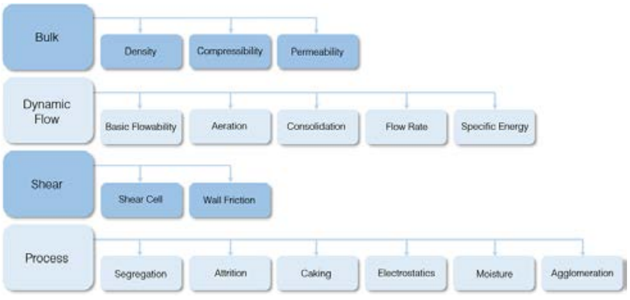

Fig 2. Four Categories of Methodologies

Measuring the flow properties include include compressibility,permeability,shear cell and stability and variable flow rate tests and compared the static and dynamic testing methods for characterizing powder flow and used FT4 as the static method to better predict agglomeration with cohesion. A blade or impeller is needed for the tests before each test cycle, a preparation step called conditioning process performs to prepare the sample for the following measurement and remove any effects remained in the sample by rotating and moving the impeller downwards and upwards through the powder three times.

COMPRESSIBILITY:

Compressibility is used to measure of how density changes as a function of applied normal stress.

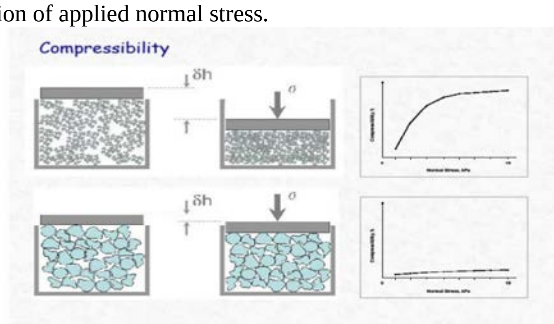

Fig 3. Compressibility

The bulk property of the powder is influenced by many factors such as particle size distribution, cohesivity, particle stiffness, particle stiffness, particle shape and particle surface texture. It is related to many process environments, such as storage in hoppers or behaviour during roller compaction [19].

BULK DENSITY

= SPILT MASS

VOLUME AFTER COMPRESSSION

COMPRESSIBILITY INDEX

= DENSITY AFTER COMPRESSION

CONDITIONED BULK DENSITY

A vented piston is used to apply a normal force from 0.5 to 15kpa onto the sample. Conditioned bulk density (CBD) and percentage change in volume after compression (CPS) were obtained.

PERMEABILITY:

Permeability is a measure of how easily material can transmit a fluid through its bulk. Consolidation stresses are also likely to influence permeability by changing the porosity and particle contact surface areas, making it more difficult for the air to pass through the bulk.

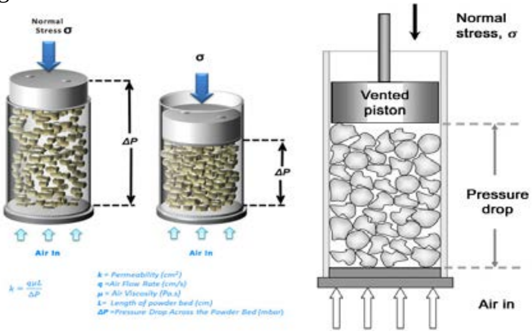

Fig 4. Permeability

It is influenced by many physical properties such as particle size and distribution, cohesivity, particle stiffness,shape, surface texture and bulk density [20]. It is related to many process storage in and flow out of hoppers, pneumatic transfer, vaccum transfer and specific processes such as vial filling or dry dosage inhalation.

STABILITY AND VARIABLE FLOW RATE:

The stability and variable flow rate by combining the two methods into one program, REP+VFR. There are benefit of running the two methods in one combined program as less powder is required and less time [21] is needed to caarry out the analysis.REP+VFR is used to describe the stability of a powder in a system and the sensitivity of a powder to flow rate.

The seperate stability study used if it was known that materialmdeagglomerated or segregated.

The seperate variable flow rate study used if the powder being analysed was known to be stable and did not require the stabiliyty program to be run.

Basic flowability energy (BFE), Stability Index (SI), Specific Energy (SE), Flow Rate Index (FRI), Conditioned Bulk Density (CBD) were obtained.

BFE – The energy required to establish a particular flow pattern in a conditioned, precise volume of powder (The blade moving downward)

SI – Stability Index is used to measure the stability of a powder.

FRI – Used to charactrize the sensitivity of a powder to flow rate change.

SE – It is a dynamic measurement, similar to BFE (The blade moving upward)

SHEAR CELL:

Shear properties are important for understanding how easily a previously at rest, consolidated powder will begin to flow. Measuring the shear properties will provide important information as to whether the powder will flow through the process or wheather bridging, blockages and stoppages.

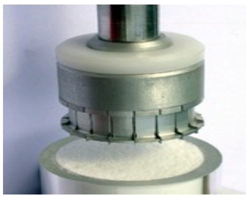

Fig 5. Shear Cell Head

Shear cell determine simple flow indices which are measures of the forces required to initiate flow based on measurement of bulk density, cohesive strength, powder friction and wall friction with this information we can estimate powder arching or ratholing, mass discharge or feed rate, blend segregation, powder caking, stress transmission in tabletting and encapsulation. The shear head in Fig moves downwards inserting the blades into yhe powder and induces a normal stress as the shear head face contacts the top of the powder.

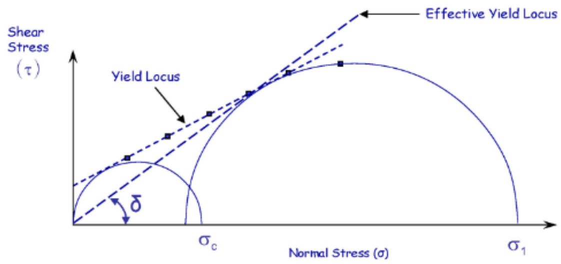

Fig 6. Mohr circle – Yield locus for cohesive solid and related parameters

After the required normal stress σ is established, the shear head begins rotations slowly to induce a shear stress τ. It will increase the shear stress until the bed fails or shears, at which time a maximum shear stress is observed [22]. The maximum shear stress is the yield point. The sample will be pre – sheared at the maximum normal stress until the shear stress reaches steady state.

FLOW FUNCTION (FF) = MPS/UYS

Flowability	Hardened	Very cohesive	Cohesive	Easy Flowing	Free flowing
Flow Index(FFc)	**<1**	**<2**	**<4**	**<10**	**>10**

Table 1. The classification of powder flowability by flow index [23]

Then the sample will be sheared to obtain five yield points with different normal stress and shear stress. Cohesion, Uncofined Yield Strength (UYS), Major Principle Stress (MPS), Flow Function (FF) were obtained.

Cohesion – Point of intersection of the yield locus (Intercept y – axis) i.e.Normal stress=0

UYS – Energy required to break or drform a material and it is greater of the two values at which the smaller mohr circle intercepts the x axis.

MPS – A maximum stress a particle can have at its same time (Maximum normal stress). The greater of the two values at which larger mohr circle intercept the x axis.

AERATION:

The bulk powder of all powders are affected by air to some extent since the space between the particles is filled with air. The amount of air present influences how the particles interact with each other and this impacts directly upon the flow properties[24].

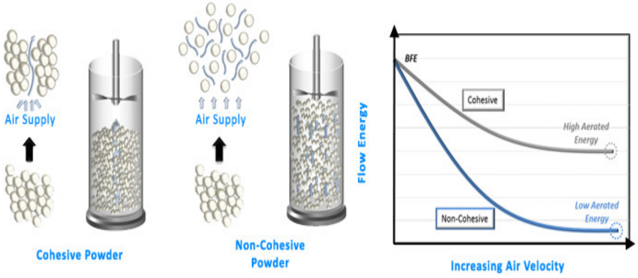

Fig 7. Aeration

The Aeration program introduces air into the base of the powder column and quantifies how this changes the flow properties by measuring the reduction in flow energy. The energy is reduced is dependant on many physical properties such as cohesion, particle shape, texture and density [25].

Cohesive powder are generally not readily aerated although they may still have thier flow properties enhanced by increasing the level of entrained air. Non – cohesive powder are readily aeratable and only require a small amount of air to transform the powder bulk into a fluidised bed, in which the powder behaves as a fluid and requires only a small amount of energy to produce flow.

WALL FRICTION

Wall friction properties important for understanding how easily a previously at rest, consolidated powder will begon to flow in relation to the wall material of its container. Measuring the wall friction properties will provide important information as to whether the powder will flow against the material with it is in contact. In every process and storage environment, powders will be subjected to consolidation stresses, causing changes in density and mechanical interparticulate forces.

Fig 8. Wall Friction Impellers

Whether it is transport in a keg, storage in a hopper or processing through an IBC on the t Op of a tablet press or roller compactor, powders will be subjected to some lwvel of consolidation stress during their handling. For flow to occur, it is necessary that not only is the yield point of the powder overcome, but that frictional forces between the powder and the container surface are also overcome [26].

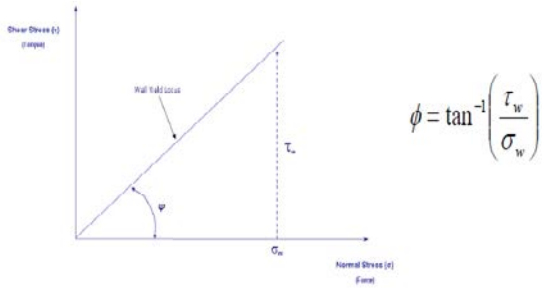

Fig 9. Calculating Wall Friction Angle.

The over consolidation is acheived by pre – shearing at a maximum normal stresses, until the shear stress reaches steady state. The pre – shear normal stress must be greater than the maximum normal stress value applied during the wall friction test. Wall friction angle is measure of resistance. Higher WFA, powder less likely to slide against surface. Required for hopper design and useful for understanding adhesion of powder to surfaces.

CONCLUSION

Due to more micronization powders results in formation of agglomerates. Agglomerates are formed when subjected to vibration in sieve shaker and as a result it cannot pass through the sieves, resulting in settling of agglomerates. Tap density testers are also unsuitable for highly cohesive powders resulting in falsely low results. Freeman Technology has over 15 years experience in the design of powder processing applications was used to measure the materials flow properties including compressibility, permeability, shear cell and stability and variable flow rate tests. To compared the static and dynamic method to better predict agglomeration with cohesion. The FT4 has proven application in all powders processing industries, including pharmaceuticals, Fine chemicals, Food, Cosmetics, Powder coating and Additive manufacturing. The FT4 Application extend to Filling, Tablet compression, Hopper flow, Wet granulation end point, Flow additive selection and optimisation, Humidity effects, Electrostatic change, Mixing/ Blending, Feeding, Segregation, Attrition, Dry powder inhalers, Caking, Milling, Wall friction and adhesion, Hopper design, Compact hardness.

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