Bulk Powder Behavior

Handling of powders and granular materials is of great importance to industry. In 80% of the industry these materials are involved. In the chemical industry alone, half of the products and at least 75% of the raw materials are in the form of powders and granular solids.

In any particulate process, the physical characteristics of powder and granular material e.g. cohesiveness, density, compressibility, particle size distribution, particle shape and humidity have an effect on the flowability and floodability of the bulk material.
Also, solid particles and granules wear down during production, handling and transport. The forces particles undergo are broadly classified into impact forces, compressive forces and abrasive forces. Compressive forces tend to occur in hoppers, conveying systems, silos and on truck or rail where the particles are compressed by their own weight.
Compressive forces can cause fragmentation of particles, a process whereby a particle splits into smaller parts, usually large in number and including a range of sizes of daughter particles this sometimes results in the release of enclosed material. Abrasion causes dust and segregation has an impact on the quality of the powder material.

A range of different techniques is available for characterizing bulk powder behavior and testing. Single pellet crush strength for radial and axial crush of pellets and tablets, Delft robotic compression technique for particles and granules, shear testing according to Jenike, Peschl or Schulze, Delft compact strength test after uniaxial compaction, powder characteristics using Hosokawa for Carr indices, attrition and abrasion resistance in a rotating drum or repeated impact tester and dustiness by a rotating drum according to Heubach or EN 15051 methodologies.

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The tablet hardness tester measures the crush strength axial or radial of a single pellet and tablet, directly in Newtons (N), Strong Cobbs (Sc), Kiloponds (Kp), or Pounds Apothecary (Lb), in a hardness range of 0 – 400 N. A motorized test jaw drives continuous forward increasing the pressure applied to the pellet, tablet, capsule, etc. The pressure is monitored and at the moment of breakage the reduction in resistance recognized with the highest value just before as breakage strength.

Robotic Compression Tester according to Pitchumani

The Robotic Compression Tester (RCT) measures the maximum crushing force of individual particles or granules. The crushing force of particles with size ranging from 500 µm to ≈ 4000 µm can be measured. About 40 up to 200 particles are placed on a smooth finely polishes stainless steel plate and the coordinates of each particle and size determined by a CCD camera. Consecutively all particles at the known coordinates are crushed and the required force measured with a quartz force transducer. The highly sensitive transducer is suitable for measuring quasi-static and dynamic tensile and compressive forces ranging from a few mN to 80 N.

Jenike, Peschl Shear or Ring Shear (Brookfield) Testing

After storage of powder the intention is usually to make the powder flow or yield again. With free flowing material the shearing of the material is easily performed. With cohesiveness materials the initiation of flow is more difficult. Jenike's method is the most common method to measure powder flow properties after steady-state flowing. The methods apply the history to the powder by consolidation after which the resulting strength is measured. In this way the situation in a silo or hopper is simulated, in which the powder undergoes a certain history of shear and consolidation in the top giving it strength when it reaches the bottom of the silo or hopper.
With the Jenike tester internal friction and wall friction on various wall surfaces can be measured. A sample is placed in a shear cell of specific dimensions. The specimen is pre-consolidated by twisting the cell cover while applying a compressive load normal to the cover; subsequently with a normal load to the cover the specimen is presheared until a steady state and finally by shearing in horizontal direction under a reduced load until the shear force goes through a maximum value.
The rotational split level shear tester designed by Peschl rotates the powder over itself producing a torque from which the shear stress can be calculated.
Consolidation pressures up to 16, 30 and 50 kPa, respectively are applicable by Jenike, up to 2.5 kPa by Peschl and for the Brookfield 0.6 - 4.85 kPa with the large ring cell and 1.6 - 13.3 kPa with the small ring cell.
The data points, (maximum shear vs load) result in a yield locus with two Mohr circles. Several sets of Mohr circles result in a flow function which visualizes the powder flow behavior and as such can be used to compare

The flow characteristics of various powders upon to a certain stress. The flow function is categorized into 5 different categories: free flowing, easy flowing, cohesive, very cohesive and non-flowing. Also the effective angle of internal friction, the exit opening and angle of a hopper or silo can be calculated.

Compact Strength Tester according to Van der Kraan

The technique measures the strength of consolidated powder after uniaxial compaction. The force at failure is measured as a function of the surface ratio and is suitable for processes which don't involve steady state shearing as for storage of powders in silos but for problems in powder technology for which the construction of the flow function is not representative like caking behavior, tabletting at low stresses, storage in big bags, trains, mixers or storage in any container in which the compaction of the powder is governed mainly by its own weight.

Hosokawa Powder Characteristics Tester (Carr Indices)

Bulk powders are characterised by seven mechanical measurements and three supporting measurements. This provides a numerical evaluation of the characteristics of a powder by determining the flowability and floodability at the transition points when transferred from a static to a dynamic state. The measured values are assigned to indices based on standardised analysis of about 3000 different bulk materials by R.L. Carr.
The 'Angle of repose' measurement characterises the flowability of a material.
'Compressibility' testing indicates the difference between the aerated and the packed density of material, with low compression indicating good flowability.
'Angle of spatula' indicates the relative angle of internal friction of the bulk powder.
The 'Angle of fall' alters the angle of gradient of the cone or heap of material by applying vibration.
'Dispersibility' indicates the fugacity, dusting and flushing characteristics of the powder.
'Angle of difference' is the difference between the angle of repose and the angle of fall, a large value indicates that the material has the characteristics of flushing.
Supporting measurements are 'Aerated bulk density', 'Packed bulk density' and 'Uniformity'.

Attrition and Abrasion

Rotating drum

The test method covers the attrition and abrasion resistance of tablets, extrudates, spheres and irregularly shaped particles of 1.6 mm up to 19 mm. A sample is rotated for a set period of time in a cylindrical drum having a single baffle. The fines produced by attrition and abrasion are determined by sieving through a standard sieve. The values obtained are significant principally in relation to values for other materials.

Repeated Impact Tester according to Pithumani

The Repeated Impact Tester (RIT) basically consists of a box filled with an amount of particles or granules.

A motor is driving a shaft which is connected with a plate confined between two steel rods. On the plate a box with a sample is mounted. The plate is allowed to slide over the steel rods by two linear ball splines, only in vertical direction. The confined motion prevents acceleration in the horizontal direction and only forces in the normal direction are transferred. After the test only the mass of half the original particle size is determined as function of the number of collisions in the RIT.

Dustiness according to Heubach, Stauber-Heubach and EN 15051-2 and -3

Heubach according to DIN 55992 is the European standardized method for the determination of dust generated by the handling of powders, granules, tablets, etc. These emissions tend to generate human diseases or cause other environmental effects..In the Heubach assessment of the dustiness potential, handling and conveying is simulated by the rotation of the drum in which the sample has been placed. The dust released from dropping materials is conducted by an air flow to a sampling section where it is preseparated aerodynamically and the airborne particles are deposited onto a filter.

The particulates are quantified or can be used for further analyses.
Stauber-Heubach is an adaptation of Heubach and is focused on the dusting potential of feed premixes and additives.
EN 15051 is the general standard specifying apparatuses and reference methods for the production of dust from bulk materials and the measurement of the inhalable, thoracic and respirable fractions of dust.

Segregation

Segregation can affect final product quality e.g. tablet-to tablet variation of the active ingredient in pharmaceuticals. Tendencies of powders and dry bulk solids for segregation are simulated by means of the fluidization mechanism or the sifting segregation mechanism. In fluidization segregation (ASTM D 6941), controlled fluidization is used to potentially cause vertical segregation, fine particles will accumulate near the top and large and or dense particles will settle to the bottom. Sifting segregation is investigated by a controlled emptying of a first hopper into a second hopper (ASTM D 6940). Subsequently, the second hopper with the potentially segregated material is emptied thereby collecting different fractions. The difference in properties of the variously collected fractions is an indication for segregation and can be evaluated by particle size distribution and skeletal density, but also by other possible differences e.g. color, reactivity or concentration of a certain component.