Introduction to Powder Rheology

Relevant for: Characterization of Powders and Granular Media

Understanding the behavior of powders or granular media can be a complex undertaking. The Anton Paar powder cells offer a toolbox of measurement methods for a wide range of powder properties. This toolbox is useful for both quality control (QC) and scientific purposes. The powder flow cell includes fast and easy-to-perform methods for analyzing powder cohesion and compressibility, as well as other properties such as flowability, fluidizability and air retention capacity. More elaborate measurements allow the study of powder viscosity. This application report first describes the powder flow cell methods, and then the powder shear cell methods, as well as how to analyze and understand the measured properties.

1 Introduction

Powder and granular media can be found in almost any industry. They are used and processed as raw materials, intermediate or final products. Working with powder can be difficult, therefore, efficient quality control and smooth powder processing is important. Powder behavior can change during a manufacturing process, especially when the conditions or environment change, e.g. a powder is fluidized during pneumatic conveying and consolidated during storage. When the powder characteristics are known, the process can be modified - at best - so no problems occur during processing (e.g. segregation).

The two powder cells (powder flow cell and powder shear cell) from Anton Paar offer a toolbox of measurements, which allow determination of a variety of powder properties and handling parameters. This toolbox helps to characterize and describe a powder, as well as to predict its behavior during processing, handling and storage. A wide range of dedicated powder measurement methods are implemented in the software, and most only take a few minutes.

While both cells overlap in their applications and techniques to a certain extent, their areas of expertise can be divided by the cohesiveness of the powders involved: cohesive powders work better in the powder shear cell, while free-flowing generally work better in the powder flow cell. An overview is displayed in Figure 1.

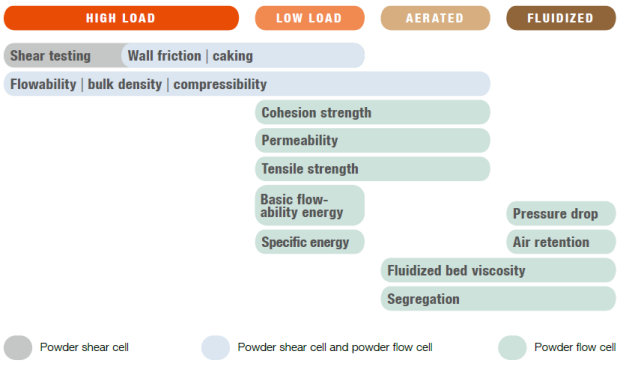

Figure 1: Areas of expertise and methods of the two powder accessories for MCR Rheometers.

In this application report, various methods and the corresponding parameters for characterization of powders and granular media are shown and discussed. An overview of the methods, which can be carried out in the Anton Paar powder flow cell, is listed in Table 1. The overview of the powder shear cell methods are displayed in Table 2. Further methods are constantly being developed at Anton Paar, universities and research laboratories and can be found in scientific publications and other application reports on our website.

Method	Parameters and Possible Applications
Pressure Drop	Volumetric flow for incipient/full fluidization for: pneumatic conveying/transport; flow rate for sample preparation (erase “powder memory”); filling, oscillation measurements
Deaeration	Deaeration behavior (air retention capacity) for: pneumatic conveying/transport (dense phase flow, diluted phase flow), filling, tableting
Cohesion Strength	Cohesion in deaerated state for: detection of differences in a powder formulation
Warren-Spring Cohesion	Cohesion in consolidated state for: flowability, behavior during storage (caking), detection of differences in powder formulation
Wall Friction	Friction between powder and any material for: wall friction angle for hopper design
Compressibility	Volume change by consolidation for: bulk density, maximum packing density, Carr index, Hausner ratio; compaction, tableting
Powder Shear Rate Sweep	Fluidized bed viscosity in dependence on the shear rate and air flow for: powder behavior in a nozzle (powder coating, spray drying)
Segregation	Particle segregation or demixing for: formulation stability, storage, tableting
Tensile Strength	Interparticle adhesive forces between powder particles for: simulations, additive manufacturing
Permeability *	Air flow resistance for: aerosolization processes, tableting and filling
Dynamic Flow	Determination of the dynamic flow characteristics of a powder. Investigation of flow stability and flow rate sensitivity under variable shear rates. Calculation of the total flowability energy under confined flow.

Table 1: Methods for powder characterization using the Anton Paar Powder flow cell

** not described in this application report*

Method	Parameters and Possible Applications
Yield Locus Analysis	Basis for all shear cell measurements; yields a wide range of coefficients and values for multiple processing steps; can be done for multiple pre-compaction values temperatures and moisture levels
Flow Function Analysis	Easy to interpret coefficient; useful from quality control to silo design
Bulk solid density *	Measuring both the compaction as well as the final density of bulk solids under different pre-compactions; storage, handling and transport-essential for silo design
Time consolidation measurements*	Measuring the values above but including the influence of prolonged storage under different ambient conditions
Wall friction measurements	Friction between powder and any material for: wall friction angle for hopper design

Table 2: Methods for powder characterization using the Anton Paar powder shear cell

** not described in this application report*

2 Methods Powder Flow Cell

2.1 Pressure Drop

Granular media can be fluidized through introduction of air flow through the medium. Fluidized particulate substances show behavior strikingly similar to fluids.

The pressure drop method is used for two reasons:

(i) Determination of the volumetric air flow rate needed for incipient or full fluidization of a powder sample.

(ii) Sample preparation – full fluidization at a corresponding volumetric air flow prior to any measurements erases the “powder memory”. Full fluidization results in erasure of residual tensions and agglomerates between the particles. This leads to a homogenous powder bed with removed non-intrinsic and operator influences. (1)

Attention: not all powders can be fluidized.

More information can be found in the application reports “Silicon Dioxide: Powder Flow Measurements of Fumed Silica” and “Rheological Powder Measurements on Coffee Creamer”.

2.1.1 Possible Applications

Knowing the flow rate for incipient and full fluidization for transport can be interesting for pneumatic conveying of cement, food powders, fly ash, washing powders, paint powders, plastic and metal powders.

The flow rate for sample preparation can be useful when measuring cohesion strength (2.3), permeability and flow curves (2.7).

2.1.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Fluidization set scientific
- Two-blade stirrer

The powder sample is filled into the powder flow cell, which is sealed with the dust protection hood for clean handling. Air flow is introduced into the sample through a glass frit on the bottom of the powder flow cell, and the pressure is recorded. Mechanical stirring or oscillation can improve fluidization for powders that are difficult to fluidize.

Normally the measurement consists of two steps. First, the air flow is continuously decreased from the maximum to minimum value, which allows for studying the full fluidization rate. In the second step, the air rate is continuously increased. This shows the air rate for the powder’s incipient and full fluidization, as well as the hysteretic behavior of the powder.

For simplicity, only the part with increasing air flow is displayed in Figure 2 (in red). It is crucial to take into account the influence of the system (glass frit, filters, etc.) by performing the same measurement on the empty cell. This baseline (grey line in Figure 2) then has to be subtracted from the values of the sample measurement. The resulting graph is displayed in Figure 3.

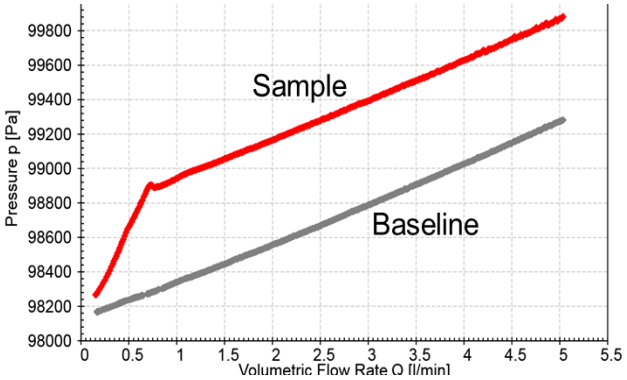

Figure 2: Pressure signal curves of a powder sample (red) and a baseline (grey), the latter is deducted from the sample

2.1.3 Analyzing and Understanding the Data

Figure 3 shows a typical pressure drop curve after subtraction of the baseline. The sample consisted of glass beads with a diameter of 40 to 80 µm. The pressure inside the cell grows with increasing volumetric flow, because the particles create a rising counter-pressure against the fluidization air. Once a certain volumetric flow is reached (depending on the particle characteristics), the powder fluidizes and an overshoot can be detected. In this case, the overshoot of the incipient fluidization can be seen at a flow rate of 0.75 L/min. At full fluidization a constant pressure signal is observed, meaning the powder is fully fluidized at 1 L/min. At this point the residual tensions between the particles are removed. The fluidization points can also be determined by oscillation and mechanical stirring with powders that are difficult to fluidize.

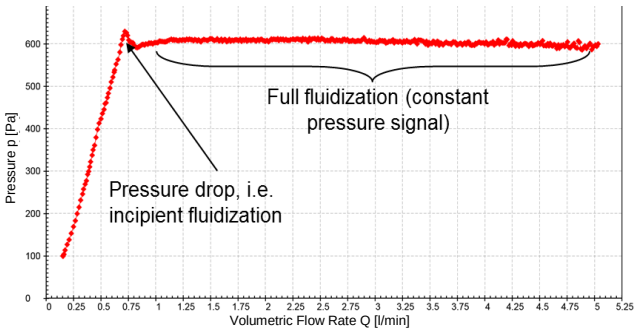

Figure 3: Pressure signal curve showing a pressure drop (incipient fluidization) at 0.75 L/min and full fluidization at 1 L/min

2.2 Deaeration

The deaeration behavior of a powder describes how the powder holds air after fluidization. It is a characteristic property of a powder and depends, amongst others, on the particle size as well as the particle morphology. Often also called air retention capacity, it is associated with bulk density and the median aerodynamic radius of the powder, similarly to a powder's capacity for dust generation. While not an absolute measurement, it can serve as a rough guideline.

If a powder has a high air-holding capacity, it will often show a strong increase in powder bed height by fluidization. After turning off the airflow, the air will escape the powder slowly. In contrast, a powder with low air-holding capacity will only show limited increase in powder bed height. After switching off the airflow, the initial powder bed height is quickly reached.

More information can be found in the application reports "A Multi-Method Approach to Quality Control for Industrial Powder Coating" and "Silicon Dioxide: Powder Flow Measurements of Fumed Silica".

2.2.1 Possible Applications

- Pneumatic conveying:
  Air-holding capacity data provides information about whether the bulk solid can be transported via a dilute or dense phase fluidized flow.
- Packing/filling processes:
  The powder needs a certain time to deaerate after discharge into a container (canister, bag, silo etc.) in order to fill the container without voids. This needs to be observed in order to deliver the goods to the customer in a uniform manner.

2.2.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Fluidization set scientific
- Two-blade stirrer

A powder sample is fluidized, if necessary, while being rotated with a 2-blade stirrer. The volumetric flow can be varied in order to simulate different conditions. Afterwards, the volumetric flow is stopped and the pressure inside the cell is measured. The time needed to detect a constant pressure signal inside the powder flow cell is defined as the deaeration behavior (in seconds). An example of a deaeration measurement is displayed in Figure 4.

The deaeration measurement is also included in the standard template of the pressure drop method.

2.2.3 Analyzing and Understanding the Data

In general, there are three different pressure falloff phases that can be observed:

1. A fast logarithmic release of surplus air. (usually 0 to 0.3 s)
2. A constant slower falloff. This part is usually the most interesting because it allows the analysis of air retention and dusting properties. The major influences here are aerodynamic radius, density of the material and particle size distribution.
   The metal powder (red curve) is fully deaerated after 2.5 s, while the fire extinguisher powder (grey curve) is fully deaerated after 14 s.
3. Depending on the powder, sometimes a third and very slow falloff similar to time consolidation of powders can be observed. The grey curve (fire extinguisher powder) continues to decrease very slowly even after 14 s.

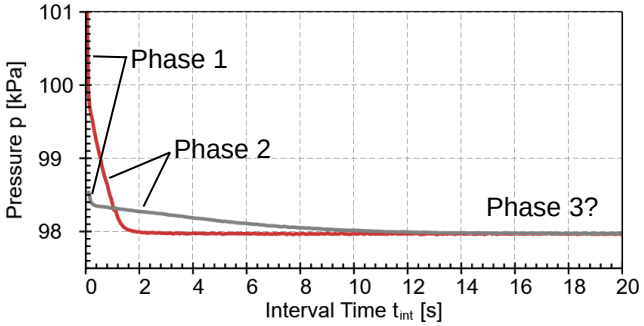

Figure 4: Pressure signal curves of deaeration of a metal and a fire extinguisher powder, in red and grey, respectively

2.3 Cohesion Strength

Cohesion strength describes the internal resistance of the powder to flow, and thereby a measure of powder flowability. It is defined as a measure for the strength of the bonding forces between powder particles. Cohesion strength measurements are fast with a high reproducibility and help as a quality control tool for predicting powder behavior. The theoretical background can be found in the Mohr-Coulomb theory.

More information can be found in the application reports "Characterization of Rice Grains" and "Rheological Powder Measurements on Coffee Creamer".

2.3.1 Possible Applications

This measurement can be used as a quick and simple QC tool, since it usually has high repeatability and helps differentiate even very similar powders. It also gives information on the air-retention capacity and the aeration behavior through mechanical stirring.

This method can be used with all fluidizable powders, like metal powders, many food powders, some cement mixes, some polymer powders etc.

2.3.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Fluidization set scientific / Fluidization set QC
- Two-blade stirrer

The powder flow cell is filled with sample and sealed with the dust protection hood for clean handling.

The measurement then consists of two steps:

- Sample preparation: the sample is fully fluidized in order to reset the powder and remove residual tensions and agglomerates. The necessary volumetric flow should be determined beforehand using the pressure drop method (see section 2.1).
- Sample measurement: the air flow is turned off and the torque of a rotating 2-blade stirrer is measured, as displayed in Figure 5. The measurement ends by default after 100 s.

Comparability of different powders is given by identical or very similar stress states in the powder bed, which are given by measuring at the same position in the powder bed (i.e. using the same volume/mass).

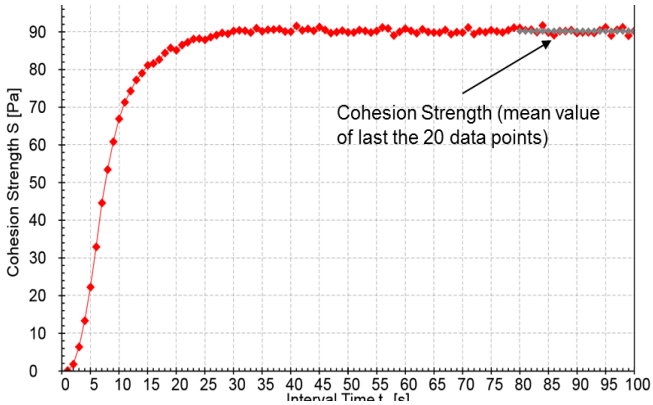

Figure 5: Typical cohesion strength curve

2.3.3 Analyzing and Understanding the Data

The slope at the beginning of the measurement (interval time 0 to 20 seconds in Figure 5) is influenced, among others, by the powder's aeration behavior, air holding capacity and packing behavior. A lower starting point indicates a higher air-holding capacity. A flatter increase can stem from a higher air-holding capacity, but also from the powder's aeration behavior from stirring. After a certain measurement time, easy- and free-flowing powders should display a constant torque (and thereby constant cohesion strength). In contrast, cohesive and very cohesive powders do not necessarily show the same behavior and the torque might not become constant during the standard measurement time. In this case, an increase in the measurement time will help.

Cohesion strength S is calculated with the measured torque value and a geometry-specific factor (the CSS factor), and the retrieved results are, therefore, relative values. The calculation is displayed in Equation 1. The torque value is found using a linear regression over the last 20 data points (see Figure 5). For the CSS factor, a calibration measurement with calcium carbonate (CRM 116, Community Bureau of Reference) was carried out. The calibration was carried out with a normal stress of 3 kPa and no applied normal strain during measurement. This gives the intercept of the yield locus function with the y-axis.

$$S = M \cdot CSS$$

S: Cohesion strength in Pa

M: Torque in Nm

CSS: Controlled shear stress standard value in Pa/Nm (calibration factor)

Equation 1: Calculation of cohesion strength with calibrated CSS factor

2.4 Warren-Spring Cohesion

This method is applied to measure powder cohesion strength, especially of very cohesive powders (e.g. flour or cement). It is based on the work of Geldart, by using a torsional device called the Warren-Spring-Bradford tester. The powder is studied in a consolidated state, which also homogenizes the powder. (2) The results can be used to analyze flowability and flow function of cohesive powders. This method can also be used to study caking of powders.

More information can be found in the application reports „Quality control of Cohesive Bulk Solids with the Powder Cell“ and “Taking the Cake”.

2.4.1 Possible Applications

This method can be used for quality control, powder characterization (elasticity, cohesion in a consolidated state), flowability analysis (ff_c) and to study caking behavior.

It works best with cohesive powders such as flour, titania or calcium carbonate, but is generally applicable to all but the most free-flowing powders.

2.4.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Fluidization set scientific
- Powder preparation set (permeable disc)
- Warren-Spring geometry

The measurement consists of two steps:

- Sample preparation:
  The powder is consolidated with an air-permeable piston in the powder flow cell. This creates a homogenous powder bed by removing residual tension and aggregates between the particles.
- Sample measurement:
  The Warren-Spring geometry is completely submerged in the powder sample, which can provide information on cohesion. Then the powder is sheared at 0.1 rpm, while the torque is recorded, resulting in the Warren-Spring cohesion.

If the Warren-Spring cannot fully penetrate the sample, it is recommended to either consolidate the sample less, or to only insert the geometry to half the normal depth. This is also a convenient indication of arching behavior, since the powder is therefore very prone to build force chains that could lead to blockage in processes.

A typical measurement is displayed in Figure 6.

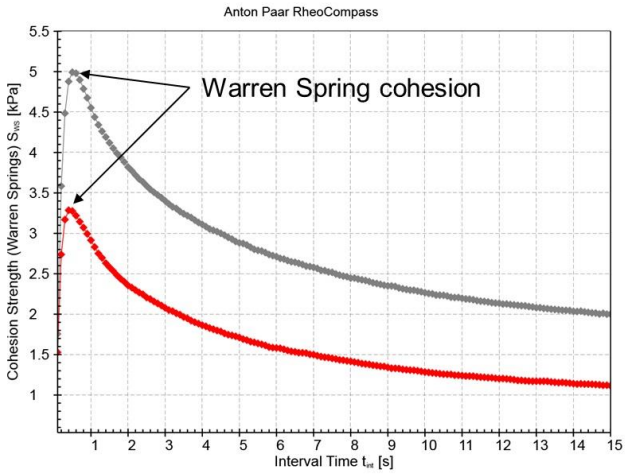

Figure 6: Typical cohesion curves using the Warren-Spring geometry (curve peak: Warren-Spring cohesion)

2.4.3 Analyzing and Understanding the Data

Torque is measured while shearing. It reaches a maximum at the beginning of each measurement when the rotation overcomes the powder’s internal friction and initiates movement of the particles. This movement (or flow) indicates a final equilibrium value (constant torque signal). The recorded maximum torque is multiplied by a specific factor as presented:

$$S_{WS} = \frac{3 \cdot M}{2\pi \, (R_O^3 - R_I^3)}$$

S_{WS}: Warren-Spring cohesion in Pa

M: Torque in Nm

R_O: Outer diameter of the Warren-Spring geometry in m

R_I: Inner diameter of the Warren-Spring geometry in m

Equation 2: Calculation of Warren-Spring cohesion

Cohesive powders will show a higher Warren-Spring cohesion than less cohesive powders. If a sharp maximum is observed, the sample breaks fast and strong (e.g. Tospearls). A long maximum, on the other hand, corresponds to a slow-breaking sample (e.g. 62K). A late maximum indicates that the sample has elastic properties (e.g. Blush) or might have not been consolidated enough.

2.5 Wall Friction

The wall friction describes the friction between granular media and a solid body. It is measured by compressing a sample at a defined normal stress and rotating a disc while recording the torque and, thereby, the shear stress. The resulting wall friction angle is an important parameter in hopper design (3) with the aim to prevent core flow and achieve mass flow. The disc used for the measurement can be replaced easily, allowing analysis of friction between any wall material and bulk material.

More information can be found in the application report "Rheology of Powdered Milk".

2.5.1 Possible Applications

- Hopper and silo design, conveying powders…
- Measuring friction between a powder and nearly any material (food powders, pharmaceutical, building industry powders, metal powders, chemicals, polymers, etc.).

2.5.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Powder preparation set (steel/PTFE disc)

A disc manufactured from the wall material is attached to the powder preparation set. This allows the use of individual materials for friction measurements between the bulk material and wall material. The powder is consolidated with this disc at a defined normal stress σ_W and sheared with 0.05 rpm, while the torque is recorded. This measuring step is performed at different normal stresses (usually at 3, 6 and 9 kPa). The torque is converted into shear stress τ_W and the resulting value pairs (shear stress τ_W / normal stress σ_W) are plotted in a graph (Figure 7).

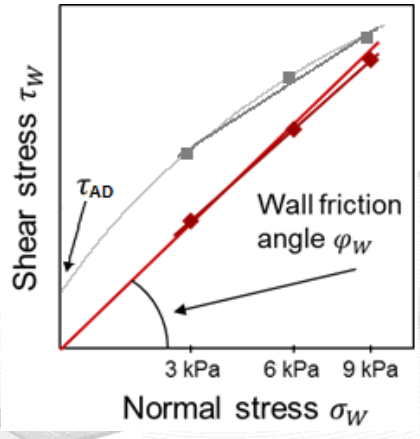

Figure 7: Schematic representation of a standard wall friction measurement (red) and a sample with high adhesion (grey).

2.5.3 Analyzing and Understanding the Data

The red curve in Figure 7 shows a standard wall friction angle measurement. In this case, the regression of the data points (wall yield locus) is linear and passes through the origin. The wall friction angle is the angle of this trend line, and is the same for all wall normal stresses (independent of normal stress).

The grey curve in Figure 7 displays a wall friction angle measurement of a very adhesive powder. The trend line is now no longer linear and does not pass through the origin. In this case, each wall normal stress corresponds to a different wall friction angle. This results in the necessity of estimating the normal stresses of the real-life application and performing measurements at these values in order to retrieve the correct wall friction angle. The intercept of the trend line with the τ-axis gives the adhesion value τ_{AD}, which is relevant for bulk solids with high enough adhesive forces to stick to vertical walls.

The calculated wall friction angle can then be used with the diagram in Figure 8 to get a hopper angle, which will allow mass flow. This can help avoid problematic silo discharge like core flow bridging, arching and ratholing.

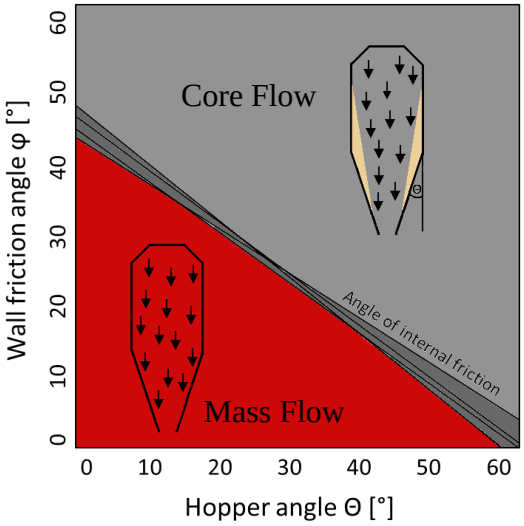

Figure 8: Graphic relation between the wall friction angle and the hopper angle for mass flow / core flow

2.6 Compressibility

Compressibility is a measure of the relative volume change a sample undergoes when pressure is applied or changed. It describes the relationship of bulk density to applied pressure. The compressibility can be influenced by many particle parameters such as size and form, elasticity, water content and temperature. Despite being a simple test, it can be used to identify the nature of powder flow. Bulk density is used, for example, to avoid ratholing and arching in silos and hoppers. Combined with the wall friction angle (see section 2.5), silos can be optimized. It is also used to study loads on walls and feeders. Other parameters, which can be analyzed, are the Carr compressibility index and the Hausner ratio.

More information can be found in the application report "Compressibility of Powders and Granular Media with the Powder Cell: Bulk Density and Flowability Indices".

2.6.1 Possible Applications

- Hopper and silo design, empirical studies on the flowability of powders.
- Food industry, pharmaceutical industry, building industry, etc.

2.6.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Powder preparation set (permeable disc)

The powder preparation set (or compression stamp) is lowered until it is in contact with the sample. That position is recorded and used to calculate the unconsolidated bulk density. Then it is lowered further until a certain normal stress (usually 3 kPa) is reached. The normal stress is further increased to two higher normal values (e.g. 6 and 9 kPa). This allows the calculation of the consolidated bulk densities, as well as the Hausner ratio and the Carr index.

2.6.3 Analyzing and Understanding the Data

The Carr index (see red curve in Figure 9) is a characteristic of a powder's compressibility. It is often used as an indication of a powder's flowability and is defined by:

$$C = 100 \cdot \frac{V_B - V_T}{V_B}$$

C: Carr index

V_B: Volume when let settle freely

V_T: Volume after tapping or consolidation

Equation 3: Calculation of the Carr index

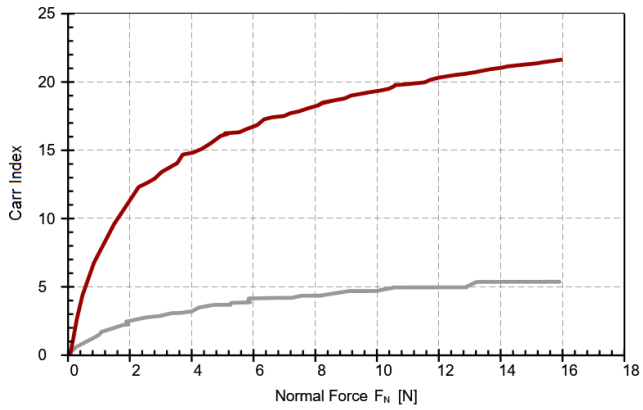

Figure 9: Carr index of coffee (red) and a metal powder (grey)

Free-flowing powders often show similar values for bulk density and consolidated bulk density (see Figure 10) and, therefore, a small Carr index. In contrast, poor-flowing powders often show a high change of density (and volume), therefore having a higher Carr index. A Carr index under 15 is an indicator for good flowability, while a Carr index over 25 indicates poor flow properties.

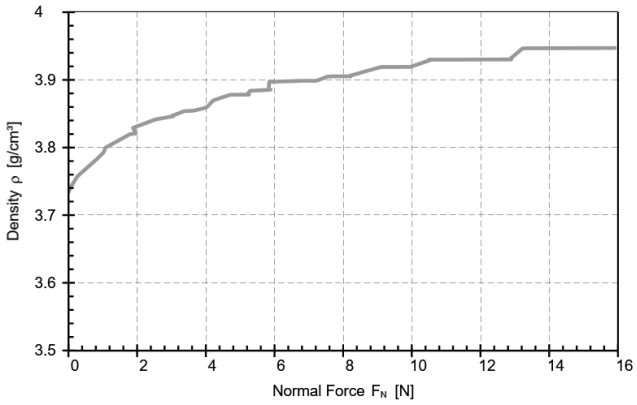

Figure 10: Bulk density of a metal powder in dependence on compression

The Hausner ratio is also an indicator for flowability (not displayed). Similar to before, larger values indicate poor flowability. Powders with a Hausner ratio above 1.25 are considered to be poor-flowing. It is calculated by:

$$H = \frac{\rho_T}{\rho_B}$$

H: Hausner ratio

ρ_B: Density when let settle freely

ρ_T: Density after tapping or consolidation

Equation 4: Calculation of the Hausner ratio

However, Hausner and Carr ratios are compound values of many particle properties and, therefore, usually not sufficient for understanding sample behavior.

2.7 Powder Shear Rate Sweep

Powders can be studied and treated in a way similar to conventional liquids. When shearing powders, it is possible to measure the apparent viscosity. Apparent shear viscosities stem from microscopic forces due to collisions between particles and from interparticle friction.

Using the powder flow cell, the shear-rate-dependent viscosity can be measured in relation to the powder's state of fluidization (non-, sub- and fully fluidized state). For example, this can indicate what difficulties can be expected during the transport process. A powder with a high shear viscosity will have difficulties passing through narrow gaps or kinks, where there is a dramatic increase in shear rate. The apparent viscosity can also be interesting for powders that undergo processing steps with different shear rates (e.g. pneumatic transport followed by spraying through a nozzle).

A fluidized powder's apparent viscosity can be calculated in a manner entirely analogous to that of a complex fluid. The dimensions of the measuring chamber and the profiled cylinder can be introduced into the Bingham fluid model, as detailed in DIN 53019 and ISO 3219.

Estimation of such rheological properties is important for e.g. modeling the hydrodynamics of fluidized beds.

More information can be found in the application reports "Silicon Dioxide: Powder Flow Measurements of Fumed Silica" and "A Multi-Method Approach to Quality Control for Industrial Powder Coating".

2.7.1 Possible Applications

This can be interesting for coating powders, reactor design, pneumatic conveying, form-filling processes. Since any powder in freefall is fluidized, it is also useful for characterization of a variety of discharge processes.

2.7.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Fluidization set scientific
- Profiled cylinder

Powder sample viscosity can be detected at various shear rates and introduced volumetric flow rates. The shear rate is increased during a measurement and every measurement is conducted at a different volumetric flow rate. Between every measurement, the sample is fully fluidized (flow rate determined using the pressure drop method) in order to erase residual tension between the particles.

Figure 11 shows the viscosity curves of both unmodified and modified (added fumed silica) coating powders at different air flow rates. In the unfluidized state (upper curves), the flow is aided by addition of fumed silica, as demonstrated by the reduced apparent viscosity of the modified powder.

This is also in accordance with cohesion strength measurements (not shown here). However, in the case of the fully fluidized powder (Figure 11, lower curves), the fumed-silica-supplemented powder displays a slightly higher apparent viscosity than the unmodified system.

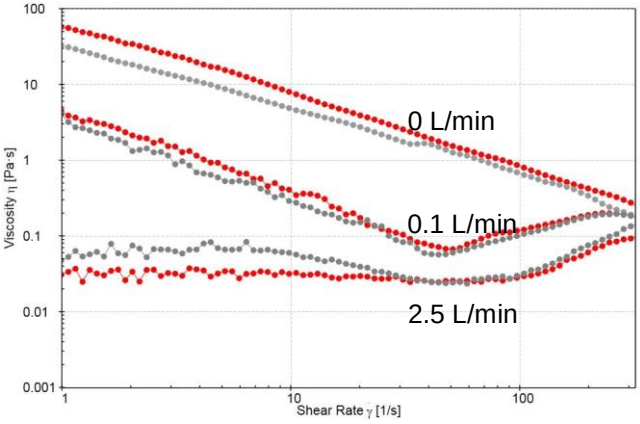

Figure 11: Shear rate ramps of unmodified (red) and fumed-silica-supplemented (grey) powder coatings at air flows of 0, 0.1 and 2.5 L/min (top to bottom, respectively)

2.7.3 Analyzing and Understanding the Data

Analysis of flow curves is very similar to analysis of conventional liquid rheology, with air flow as an additional dimension.

The results of the shear-rate-dependent measurement are presented in Figure 11. In an unfluidized state, regular shear thinning behavior can be observed. In a sub-fluidized state, shear-thinning behavior is also observed at low shear rates, but is then replaced by a shear thickening behavior at shear rates over 50 s^{-1}. In a fully fluidized state, an almost Newtonian behavior can be observed at low shear rates. At higher shear rates, a shear-thickening effect takes place. Increasing fluidization and rotational speed causes increased collisions between the particles and, at the same time, the friction between the particles is decreased. This effect is known as "jamming transition". (4)

2.8 Segregation

Particle segregation, or demixing, is the segregation of solids according to their physical properties such as size, volume, density, shape, etc. This is a constant problem in industrial processes that handle bulk solids. Segregation is generally either of mechanical nature or a product of fluidization or pneumatic transport.

The mechanical effect is called granular convection, or, more commonly, the Brazil nut effect. This effect describes the segregation of granular media by mechanical agitation, such that the largest particles end up on top. (5)

The opposite effect is observed in fluidized systems like pneumatic conveyors. The larger particles are unable to be carried into the airstream, resulting in their segregation on the bottom of the system. (6)

More information can be found in the application report "A Qualitative Fluidization Segregation Test for Pneumatic Transport".

2.8.1 Possible Applications

Segregation is a known issue in pneumatic transport/conveying, as well as during mechanical transport (e.g. in a truck).

The issue can occur in any non-homogeneous powders, and can manifest as segregation by size, by density, by composition etc. For example, milk permeates can show segregation by particle size. A very problematic example for segregation by composition can be found in pharmaceutical products, where demixing of the active ingredient from the excipient can lead to serious dosage problems and, thereby, health risks.

2.8.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Fluidization set scientific / Fluidization set QC
- Profiled cylinder / Two-blade stirrer

The measurement itself is a variation of the cohesion strength measurement. Cohesion strength is measured at different heights in the powder bed before and after segregation. To ensure a nicely mixed sample it is both fluidized and vigorously stirred for brief periods of time. In contrast, segregation is induced by a long fluidization step of several hours.

An example of segregation induced by a long fluidization step is shown in Figure 12.
A measurement of a control sample, which underwent the same treatment, where no segregation took place is displayed in Figure 13.

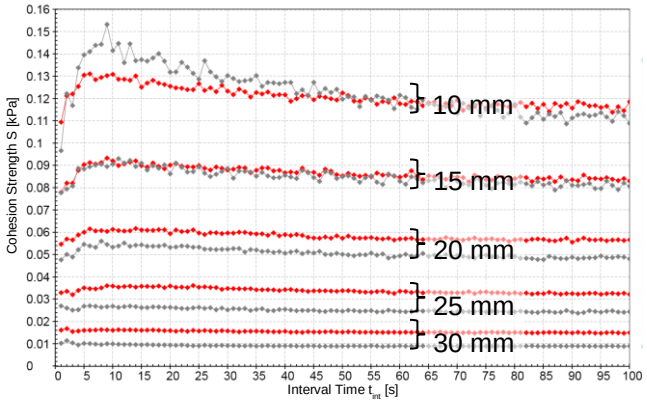

Figure 12: Overnight segregation test. Red data is from a fresh sample, while the grey data is measured after segregation by long fluidization. From top to bottom: 10, 15, 20, 25 and 30 mm measurement height in the powder bed

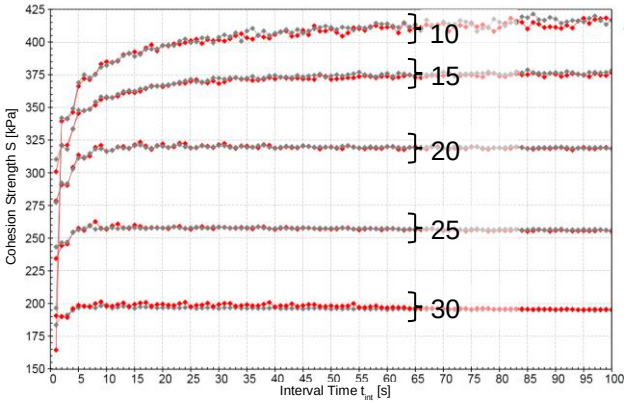

Figure 13: Overnight segregation test on a control sample. Red data is from a fresh sample, while the grey data is measured after segregation by long fluidization. From top to bottom: 10, 15, 20, 25 and 30 mm measurement height in the powder bed

2.8.3 Analyzing and Understanding the Data

In Figure 12, segregation via fluidization can be observed as cohesion strength changes due to segregation (red curves before segregation, grey curves after segregation). It is also possible to observe that the change of cohesion strength depends on the measurement position in the powder bed.

In contrast, Figure 13 displays the cohesion strength measurement of a sample where no segregation was induced by the long fluidization step. This becomes evident when comparing the unchanged values for the cohesion strength before and after the long fluidization step, which should have induced segregation.

2.9 Tensile Strength

Tensile strength describes the interparticle adhesive forces between the powder particles. The measurement is based on the fact that powder particles are connected via electrostatic bonding, van der Waal's forces and hydrogen bridge bonding. Van der Waal`s forces have the greatest influence on the tensile strength of powders. (3)

2.9.1 Possible applications

This measurement yields an intrinsic powder parameter which can be used to analyze the flowability, elasticity and the cohesive behavior of a powder. Tensile strength can be of interest in additive manufacturing in quality control or as an input parameter for simulations.

Typical powders include metal or polymer powders for additive manufacturing, powder coatings and building materials like gypsum.

2.9.2 Experimental Setup

Necessary equipment:

- Powder flow cell
- Powder preparation set (steel disc)
- Optional: fluidization set scientific
- Optional: two-blade stirrer

The measurement consists of the following steps:

- Sample preparation part 1 (optional): The sample is fully fluidized to reset the powder and reduce agglomerations. The required air flow for full fluidization can be found via the pressure drop method.
- Sample preparation part 2: The lower side of the steel disk is coated with a double-sided tape or alternatively a thin layer of petroleum jelly. The steel disc is moved down in order to pre-compact the powder. This measurement step can be performed at different normal stresses (e.g. 3, 6 and 9 kPa) in order to retrieve the tensile strength in dependence of an applied normal stress. When the required normal force is reached the measuring system sustains the stress for 60 seconds.
- Sample measurement: After compaction, the drive motor moves upward at a defined speed. A layer of powder is removed by the adhesive tape. The normal force required to overcome the intramolecular bonding forces is recorded.

2.9.3 Analyzing and Understanding the Data

The normal force is recorded while moving the disc upwards (see Figure 14 where the normal force is plotted over measurement time). In the beginning the normal force starts from a high positive value, passes through a negative minimum and then returns to a higher and constant value. The positive value in the beginning stems from the force which was used to consolidate the sample. Then the minimum of the normal force is reached - at this point the thin powder layer on the sample preparation disc disconnects from the bulk powder. The normal force returns nearly to zero, but not entirely, as the mass of the powder sticking to the sample preparation disk also creates a measureable normal force.

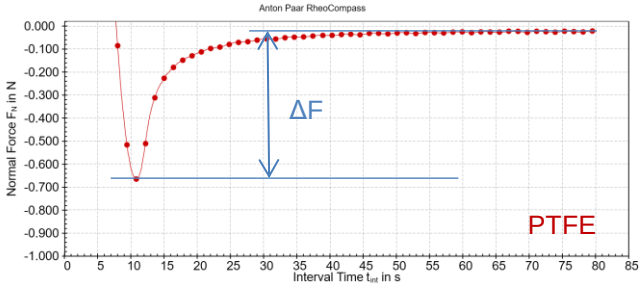

Figure 14: Normal force curve of PTFE after 9 kPa pre-compaction.

The difference in normal force between the minimum value and the constant value (displayed as ΔF in Figure 14) is then used to calculate the tensile strength by dividing it by the area of the sample preparation disc (Equation 5). For the displayed example of PTFE this results in a tensile strength of 354 Pa.

$$T = \frac{\Delta F}{A}$$

T: Tensile strength

ΔF: Separation force

A: Area of sample preparation disk

Equation 5: Calculation of the tensile strength

Further information can be retrieved by studying the form of the curve– for example the time passed until the minimum is reached will point out the elasticity of a sample.

2.10 Dynamic Flow

Dynamic flow measurements enable the analysis of the powder's flowability under motion. The helical two-blade stirrer moves through the powder bed at a pre-defined radial and vertical speed. From the resulting torque and normal force signal, various dynamic flow characteristics can be calculated for quality control.(7)

More information can be found in the application report " Basic Flowability Energy Measured by Powder Flow Cell ".

2.10.1 Possible Applications

This measurement can be used as a quick and easy QC tool to quantify external factors affecting the flowability of the powder.

This method can be used with a wide range of powders, e.g. metal powders, food powders, polymer powders, etc. However, due to the limited sensitivity of the measurement method, cohesion strength measurements should be preferred when comparing fluidizable powders. Furthermore, it is recommended to use the powder shear cell for the characterization of very cohesive powders. Shear measurements are absolute measuring methods with additional determination powder parameters such as tensile strength and compressive strength.

2.10.2 Experimental Setup

Necessary equipment:

- Powder Flow Cell
- Helical two-blade stirrer
- Fluidization Set Scientific (optional)

The dynamic flow measurement comprises eleven measuring cycles. Each measuring cycle consists of a sample conditioning and a sample measuring step.

1. Sample conditioning: The powder bed is conditioned before each measuring step. The stirrer moves clockwise up and down the power bed with a constant vertical velocity of -5.3 mm/s and a radial speed of -0.4 s^{-1}.
2. Sample measurement: The powder bed is subjected to shear forces by counter-clockwise rotation of the stirrer.
   Stability Test: The first seven measuring cycles are carried out at constant radial and vertical speed to investigate the flow stability of the powder.
   The vertical velocity and the radial speed are constant at 8.7 mm/s and -0.7 s^{-1}.
   Variable Flow Rate Test: The last four measuring cycles are defined as Variable Flow Rate Test, here the preset parameters are reduced in three steps. The vertical velocity and the radial speed are decreased by 30, 60 and 90 % compared to the values of the stability test.

2.10.3 Analyzing and Understanding the Data

The normal force and torque are recorded while moving the stirrer downwards. The total flowability energy gets calculated over the integral of the normal force plus the torque over the defined measuring position. The total flowability energy is calculated for each of the eleven measuring cycles and leads to the typical energy curve shown in Figure 15.

$$W = \int_0^h \left(\frac{M}{r \cdot \tan \alpha} + F_N \right) dh$$

W: Total flowability energy in mJ
M: Torque in mNm
r: Blade radius of the stirrer in mm
α: Helical angle in °
F_N: Normal force in N
h: Measuring distance (100 mm → 10 mm)

Equation 6: Integral over a defined displacement to calculate the total flowability energy of each measuring cycle.

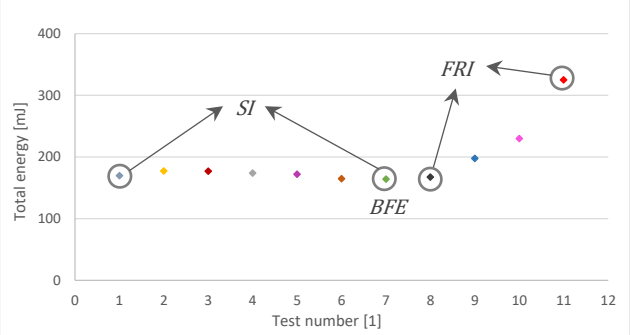

Figure 15: Development of the total flowability energy over the eleven measuring cycles using the example of limestone powder. Display of the total flow energy points used for the calculation of the dynamic flow characteristics.

Basic flowability energy (BFE) describes the flowability of a powder under confined flow and refers to the energy required for downward movement of the blade for measuring cycle seven.

Stability Index (SI) is used to evaluate the stability of the powder under constant measuring conditions. The initial settings of axial and radial movement are repeated seven times and the ratio between the seventh and the first total flowability energy is determined.

Flow Rate Index (FRI) characterizes the sensitivity of the powder to flow rate changes. The FRI is determined during the last four measuring cycles, in which the stirrer's speed gets reduced in three steps. The ratio between the last and the eighth measuring cycle is calculated.

Specific Energy (SE) is defined as the energy which is required during upward movements (not shown in Figure 15). This value can be used as a tool to assess whether the particles tend to interlock with each other.

3 Methods Powder Shear Cell

3.1 Yield Locus Analysis

Yield locus analysis is the most fundamental shear cell method. A yield locus focuses on the border between “solid” behavior and “liquid” behavior. It is based upon the Mohr Coulomb theory which measures the failure plane (similar to a Hookean experiment with solids).

Widely used in equipment design, it gives the user a defined dataset for a load condition under which a powder will flow or not. It can be further combined with both time consolidation measurements as well as environmental control. Time consolidation measurements give insight into the influence of storage time, while controlling the environment opens fully new dimensions of precisely understanding temperature and humidity influence. In essence if a bulk solid is supposed to flow (e.g. in a silo or bin) a shear stress must be applied which is higher than the yield locus function. Conversely when no flow is desired (e.g. an embankment or slope) the maximum stress applied needs to be below the yield locus function.

3.1.1 Possible Applications

The list of possible application is only limited by the knowledge of the process involved. If a precondition can be assigned (load, moisture, temperature, time), then it can be applied to the shear cell and divided into “will flow” and “will not flow”. Prime examples would be silo, hopper or bin design. This of course is not limited to the question what kind of silo must be built for this powder - but naturally also works in the much more common opposite fashion (will this powder get stuck in my existing silo).

Another classic example is the maximum slope angle - the angle of internal friction gained from the method is exactly the angle at which this powder (at the given state of moisture, temperature and pre-compaction) will come to rest, i.e. the steepest angle possible that the bulk solid will assume naturally.

Other examples include quality control, product comparison or even tableting where unconfined yield strength needs to be maximized (in case of non-creeping samples).

3.1.2 Experimental Setup

Necessary equipment:

- Anton Paar powder shear cell
- MCR rheometer
- Optional: CTD for temperature application
- Optional: Moisture Generator for moisture application

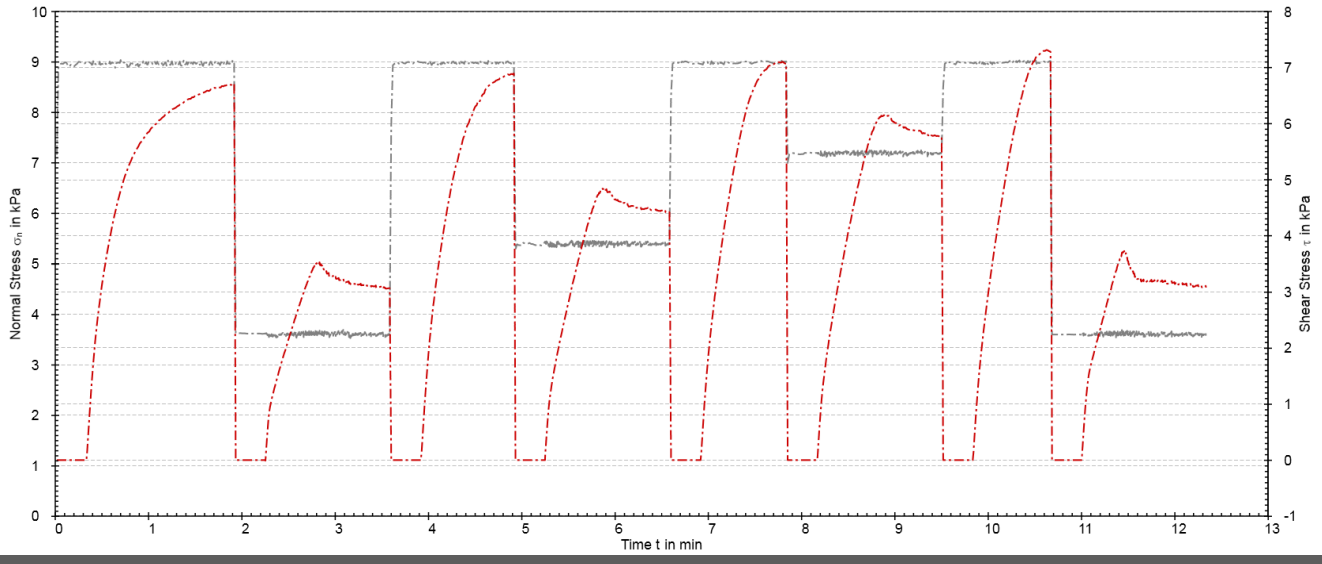

Figure 16: Pre-shear shear sequence of the reference powder BCR 116 (calcium carbonate) at 9 kPa pre-compaction. The red line is the shear stress while the grey line is normal stress.

Before starting the measurements, the sample is filled in to the measurement cell with the sample preparation bench which ensures reduced operator influence. Then, in the first step a defined pre-compaction is applied. This serves to put the sample in a repeatable state. In an opposite fashion to the application of fluidizing air to erase powder memory in the powder flow cell, in the shear cell one uses the pre-compaction to overwrite the residual tensions (or powder memory).

It is advantageous to choose the pre-compaction similar to the pre-compaction loads the sample encounters in “real-life”. This will assure that the gathered data is as close as possible to the real situation. A similar thought is advantageous in regards to moisture and temperature or even time consolidation.

A typical measurement is shown in Figure 16, and consists of repeating preshear and shear steps. The preshear step in general is used to apply a uniform, stress on the sample. Once a steady shear stress (or gap) is reached, the sample is prepared and can be “broken” by the shear phase. This is achieved by lowering the normal stress and thus allowing the powder to “break” (also called “shear-to-failure”). This is where the points for the yield locus are taken.

The points of interest in this measurement are:

- The shear stress maximum during preshear
- The shear stress maximum in each individual shear sequence

These points are then added to a Mohr Coulomb diagram (shear stress τ vs. normal stress σ) which gives the yield locus function (exemplary with 6 kPa preshear normal stress in Figure 17, and Figure 18).

From the yield locus function and the preshear maximum two Mohr circles are drawn which aid in retrieving the wide range of indicators that can be gained by this analysis.

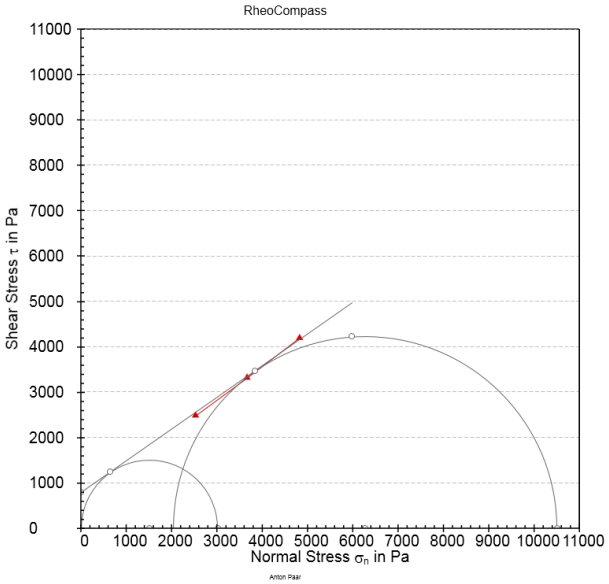

Figure 17: Mohr Coulomb diagram at a normal stress of 6 kPa during preshear.

3.1.3 Analyzing and Understanding the Data

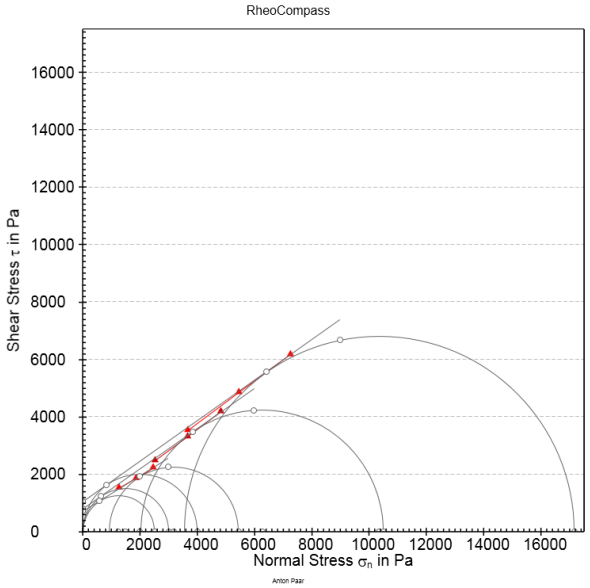

Figure 18: Combined Mohr-Coulomb diagrams at preshear normal stresses of 3 kPa; 6 kPa; and 9 kPa.

The yield loci are further analyzed and yield multiple coefficients of interest:

Coefficient name and symbol	Value
Cohesion τ_c:	1039 Pa
Tensile strength σ_t:	-1473 Pa
Unconfined yield strength σ_c:	4007 Pa
Major principle stress σ_1:	17170 Pa
Coefficient of flowability ff_c:	4.28
Effective angle of internal friction φ_e:	41.17°
Bulk solid density ρ_b:	1.032 g/cm³

Table 3: Major coefficients taken from a Mohr Coulomb diagram of CRM 113 at 9 kPa

The following is a brief description of the coefficients gained:

Cohesion τ_c: The resistance of the powder to flow if no more stress is applied on top. Usually a good indicator of flowability and useful in movement and smoothing processes.

Tensile strength σ_t: The pressure necessary to separate one layer of powder from another. Useful for simulation, quality control and comparative purposes since it is not dependent on the density of the powder.

Unconfined yield strength σ_c: The “strength” of the powder. Useful for tableting and silo design. Used in the following flow factor analysis.

Major principle stress σ_1: The total amount of stress applied to the powder during the test (comprised of both normal and shear stress). Useful to compare the state in the measurement system with uniaxial processes like compression, tableting or silo discharge.

Coefficient of flowability ff_c: quotient of σ_c and σ_1. Very useful flow indicator, discussed in more detail in the next chapter.

Effective angle of internal friction φ_e: Describes the amount of stress deflected from normal to shear direction and vice-versa. In practice, the steepest pile possible with this powder.

Bulk solid density ρ_b: The density of the powder at a given consolidation. Useful for storage, packaging, transport and silo design.

3.2 Flow Function

The flow function and the flow function coefficient (ff_c) were originally conceived by Andrew Jenike in his seminal bulletin 123. It was originally envisioned to aid in hopper bin and silo design and is, in principle the easiest way to judge the flow/non-flow of a silo, hopper or bin. The flow function is the quotient of σ_c and σ_1 and is normally plotted in a diagram of σ_c vs σ_1.

The flow factor diagram is usually partitioned into five areas (Figure 19) from non-flowing to free-flowing.

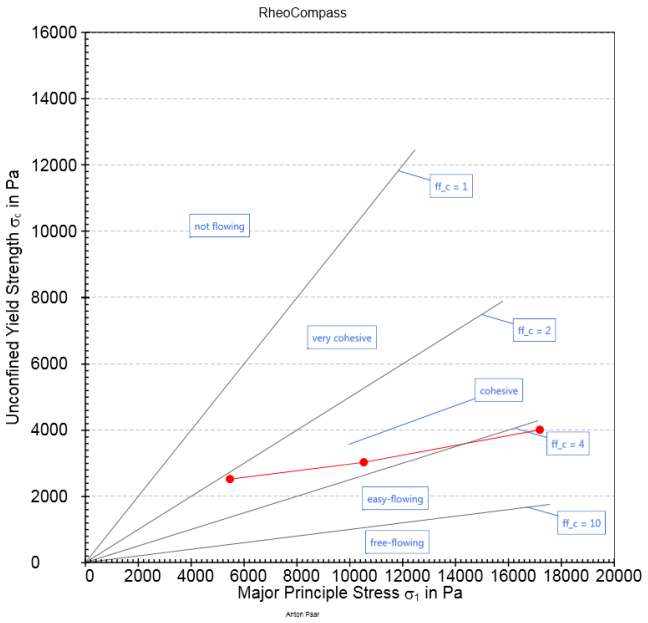

Figure 19: Flow function of calcium carbonate

For all intents and purposes a bulk solid with a flow factor below 1 will not flow.

While the practical mechanics of silo design are established, they would exceed the scope of the current work. However, the ff_c value can be used as a flowability indicator when comparing multiple bulk solids or the same bulk solid in different states (e.g. temperature, moisture or time consolidation).

If used as such, it is necessary to understand that only values gathered at the same pre-compaction can be compared, otherwise the analysis falls prey to the fact that the diagram in itself is designed for the purpose of comparing a material strength (the unconfined yield strength) with an applied force (the major principle stress).

If this precaution is understood, then comparison of flow factors is a useful way to classify and categorize different bulk solid materials and can be used as such.

4 Summary of the Methods

4.1 Powder Flow Cell

Pressure Drop: An air flow is introduced into the powder through a glass frit on the bottom of the powder flow cell. The volumetric air flow is increased until a pressure drop occurs inside the powder flow cell. Method used for determination of incipient and full fluidization for pneumatic transport and filling processes.

Deaeration: Describes behavior of air-holding (retention) capacity. Powders with a low air-holding capacity settle down quickly, while those with a high air retention capacity often show an increase in powder bed height by fluidization. Possible applications are for pneumatic conveying, packing or filling processes.

Cohesion Strength: Gives information about powder flowability (internal resistance). The sample is fully fluidized in order to reset the powder to remove residual tensions and/or agglomerates. After switching off the air flow, torque of a rotational 2-blade stirrer is measured. To compare similar powders, it is necessary to measure at the same gap and use the same amount of sample, preferably by weight. This method is extremely sensitive and repeatable. Cohesion strength can be applied for quality control, flowability measurements.

Warren-Spring Cohesion: Describes measurement of cohesion strength in consolidated state (especially for cohesive powders). This method can be used for: quality control, flowability, and behavior during storage (caking).

Wall Friction: Provides information about friction between the granular media and a solid body. The calculated wall friction angle can be a useful parameter for hopper and flow design along solid walls.

Compressibility: Displays relative volume changes following application or change of pressure. The calculated parameters are the Hausner ratio, Carr index and bulk density. These values provide information about flowability. Combined with the wall friction method, silos can be optimized.

Powder Shear Rate Sweep: It is possible to measure the apparent viscosity by shearing powders. Using the powder flow cell, viscosity can be measured in relation to the powder's state of fluidization. For example, to determine possible difficulties during transport processes: how easily the powder will pass through narrow gaps or kinks, possibility of pneumatic transport following by spraying, as well as the powder's behavior in fluidized bed processes such as reactors or dryers.

Segregation: Describes demixing of solid samples. This depends on their physical properties, such as size, shape, volume, density, etc. This information is important for: bulk amounts and storage, pneumatic transport/conveying, and tableting.

Permeability: During defined compression, air passes through the sample. This describes flow resistance, and is important for: solvation processes, tableting and filling.

Tensile Strength: This method can be applied to powders in un- and consolidated state and is useful for: doctor blading, additive manufacturing, and describing load-independent parameters (i.e. for simulations).

Dynamic Flow: This measurement method provides information on the flowability of the powder under confined (downward) and non-confined (upward) stirring conditions. The samples are conditioned before each measurement cycle to remove local compaction and entrapped air. In comparison to the cohesion strength method, a helical two-blade stirrer is required.

4.2 Powder Shear Cell

Yield Locus analysis: Fundamental shear cell measurement and basis for all further techniques. Yields a wide range of process values based on the differentiation between a solid and non-solid state (non-flow and flow) at different conditions (e.g. load, temperature and moisture). Chief among these process values is the flow function.

Flow Function: An easy index used to compare powders in different states and serve as the essential tool for silo design. Can also be used for flowability analysis and quality control.

Time Consolidation: Gives information on time dependent behavior of powders, like caking. Can be of interest in silo/hopper design, but also in other applications where caked powders hinder further processing.

Compressibility: see powder flow cell.

5 References

1. **Geldart, D.** Types of Gas Fluidization. *Powder Technology.* 1973, Vol. 7, 5, pp. 285-292.

2. **Mohammed, Salam A., et al., et al.** Measuring powder flowability with a modified Warren Spring cohesion tester. *Particuology.* 2011, Vol. 9, 2, pp. 148-154.

3. **Schulze, D.** *Powders and Bulk Solids.* s.l. : Springer, 2008. 978-3-540-73768-1.

4. **Brown, E. and Jaeger, H. M.** Dynamic jamming point for shear thickening suspensions. *Physical review letters.* 103, 2009, 8.

5. **Ehrichs, E. E., et al., et al.** Granular Convection Observed by Magnetic Resonance Imaging. *Science.* 1995, Vol. 267, 5204.

6. **Prescott, James K., Clement, Scott A. and Carson, John W.** *Fluidization segregation tester. 6487921* US, December 3, 2002. Grant.

7. **R. Freeman.** Measuring the flow properties of consolidated, conditioned and aerated powders - A comparative study using a powder rheometer and a rotational shear cell, *Powder Technology*. 2007, Vol. 174, pp. 25-33

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