Temperature-Controlled Powder Rheology Measurements in a Fluidized and Consolidated State

Relevant for: Temperature Impact on Powder Behavior, Powder Rheology, Powder Flow Cell, Powder Shear Cell, Powder Characterization, Flowability, Cohesion Strength, Deaeration, Air-retention

Temperature can have a strong impact on the behavior of powders. At elevated temperatures the powders can become sticky or exhibit partial melting resulting in a decreased or increased flowability. Additionally, at sub-zero temperatures freezing can occur which also will impede flowability. These changes will affect the behavior in a consolidated state, and influence for example the flowability, cohesion or angle of internal friction. However, it also influences fluidized powders and can change their behavior e.g. regarding their air-retention.

1 Abstract

Knowing the properties and behavior of powders such as cohesion or flowability is essential as they have a strong impact on production processes. Temperature can strongly affect the behavior of powders and therefore it is of importance to be able to control it to predict these behavioral changes. Anton Paar's powder flow cell is used to analyze powder behavior while fluidized or under low loads, while the shear cell is used for consolidated powders. In this report, in order to simulate the environmental conditions as close to reality as possible the *temperature-controlled* powder flow cell and powder shear cell were used. The influence of temperature on these properties is demonstrated using a mix of precipitated silica and monolaurin.

2 Introduction

Material properties such as density and viscosity of liquids are known to be sensitive to changes in temperature. Additionally, a fluctuation in temperature also results in change in the ambient humidity, which in turn can cause further changes in behavior.

With powders and granular media, changes in temperature (and humidity) can also have a significant impact on properties such as flowability. This in turn can lead to complications during silo discharging and also in filling and dosing processes. The behavior of powder in a consolidated (i.e. as in a silo) is easily characterized with a powder shear cell. Anton Paar rheometers can be equipped with shear cells and combined with convection devices, which enable control of temperature and humidity during the measurement.

However, not only the properties in a consolidated state change with temperature. Temperature will also affect how the powder behaves when fluidized. Consequently, the need arises to also control the temperature when characterizing fluidized powders. The powder flow cell is an accessory for Anton Paar rheometers and makes it possible to measure a wide range of different properties of fluidized and aerated powders. The cell used for measurements in this report was modified by Anton Paar to also allow temperature control of the sample and fluidizing gas in the range -5 to 80 °C.

In this report the influence of temperature on precipitated silica mixed with monolaurin (also known as glycerol monolaurate) will be studied. Monolaurin is produced from lauric acid, which is found in coconut milk and human breast milk. This substance was chosen for demonstration purposes because of its wide range of applications such as

- Pharmaceuticals
- Food supplements
- Food additives
- Surfactant in cosmetics (deodorants)
- Emulsifier

3 Experimental Setup and Methods

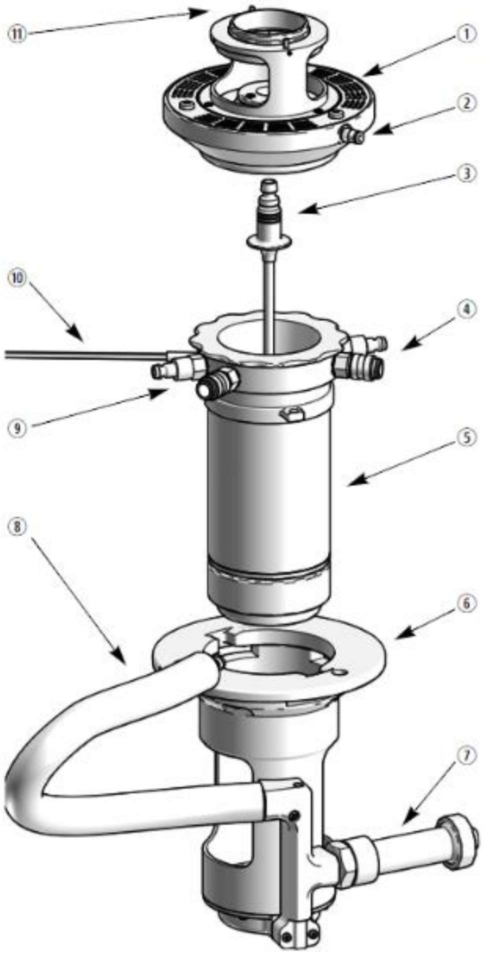
3.1 Sample

The analyzed powder sample is, as described above, precipitated silica with monolaurin. It was reported, that this sample tended to show flooding during silo discharge in summer, while in winter it would discharge without any issues. Therefore, a temperature range from 20 to 60 °C was selected for the measurements in this report. Monolaurin has its melting point at 63 °C, therefore a behavior change of the sample can be expected as that temperature is approached.

3.2 Setup

3.2.1 Powder Flow Cell for Fluidized Measurements

The measurements were performed with a Modular Compact Rheometer (MCR) equipped with the temperature-controlled powder flow cell (PFC-LTD80). This cell enables temperature dependent measurements in the range -5 to 80 °C. A schematic configuration of the PFC-LTD80 is shown in Figure 1. The configuration for the PFC-LTD80 includes a dust protection hood (1) and an air connection for sealing air to prevent loss of fine particles. Located at the upper end of the measuring cell (5) are water inlet/outlet connections (4) for the fluid circulator. The flow of the fluid through the circulatory system inside the cell cools/heats it. A secondary circulatory system in the cell is used in turn to cool/heat an air flow. The cooled/heated air passes by a pressure manifold with a pressure sensor (7) and is inserted into the powder bed from the bottom by a porous glass frit and thereby can be used to fluidize the powder. The volumetric air flow rate is controlled by a mass flow controller (MFC), in these experiments in the range 0.05 to 5 L/min. the measuring tube has a diameter of 50 mm and is made of stainless steel. For the cohesion strength measurements a two-blade stirrer (measuring system) was used.



1 Dust protection hood with sealing air connection
2 Air connection for sealing air
3 Measuring system
4 Water inlet/outlet from fluid circulator
5 Measuring cell
6 Cell holder
7 Pressure sensor
8 Air connection for fluidization
9 Air inlet/outlet
10 Temperature sensor
11 Index pins

Figure 1: Overview of the PFC-LTD80 setup used for the fluidization measurements.

3.2.2 Powder Shear Cell for Consolidated Measurements

Further rheological measurements with the MCR were carried out with the powder shear cell (PSC). The shear cell geometry PSC43 with temperature option (convection temperature device – CTD) was used for the shear measurements. An image of the setup is shown in Figure 2. Shear cell measurements are commonly used for silo design. Time consolidation (caking), wall friction angle and compressibility are further important methods that can be performed with the shear cell, providing the user with additional valuable information. [2]

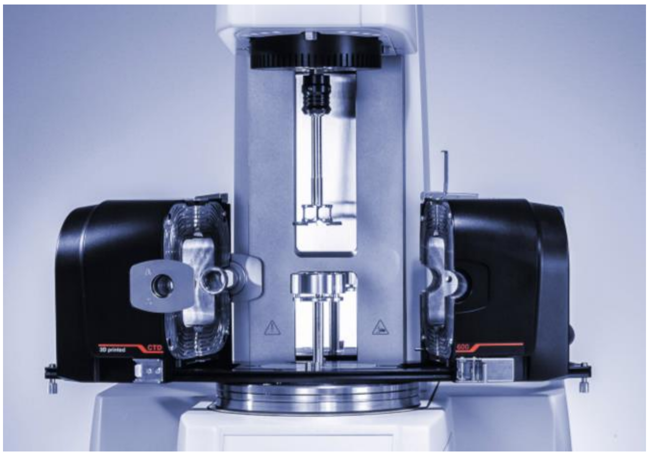


Figure 2: Shear cell mounted in an MCR rheometer, equipped with a CTD temperature device.

3.3 Measurement Methods

3.3.1 Temperature-Controlled Cohesion Strength Measurements

The cohesion strength provides relative information about the flowability of an aerated powder, or in other words, about its internal flow resistance. This method stands out due to its high repeatability, reproducibility and sensitivity. Short mounting and measuring times and easy handling make this method an ideal solution for quality control since even small changes in the sample can be detected. The measurement consists of two steps – preparation and measurement. For the sample preparation step, stirring was carried out at 8 rpm with an air flow of 2.3 L/min for 60 seconds to fully fluidize the powder in order to remove residual tensions within the bulk. The measurement is then carried out by stirring a 2-blade stirrer at 8 rpm (without any air flow) while recording the torque. The measurements were performed at temperatures of 20 and 60 °C.

Additionally, a measurement with a stepwise temperature increase from 20 to 60 °C in 10 °C steps was performed.

The cohesion strength is a function of measured torque and of a constant geometry factor, the latter being gathered by calibration using CRM-116 limestone powder. The calculation of the cohesion strength is carried out when steady-state flow is achieved. The final 20 measurement points are averaged and used to compare the different samples.

Note: The cohesion strength, measured in the flow cell, cannot be directly compared with the cohesion measured in a shear cell. The cohesion strength in the flow cell is a relative measurement in a deaerated state, whereas the cohesion from the shear cell is a property measured in a consolidated state.

3.3.2 Temperature-Controlled Deaeration Measurements

The deaeration time or air holding capacity provides information on the time necessary for particles to settle down after fluidization. It mainly depends on the particle size, density and morphology. Determining the air holding capacity is for example important for pneumatic transport or for filling and dosing processes.

Deaeration experiments were carried out at temperatures of 20 and 60 °C. For this test, first the powder is fully fluidized and then the air flow is stopped. The time necessary for the powder to settle is measured by analyzing the pressure signal under the powder bed. The experiments were carried out with a fluidization air flow of 2.8 L/min for 10 minutes and a rotational speed of 8 rpm.

3.3.3 Temperature-Controlled Shear Cell Measurements

Information and the theoretical background on powder shear cell measurements is well covered in literature, e.g. in "Powders and Bulk Solids” from D. Schulze.[1]

The Anton Paar MCR is equipped with a convection temperature device (CTD) and the powder shear cell (PSC) was used to run the measurements. The shear measurements were carried out at temperatures of 20, 50 and 60 °C. The pre-shear measurements were performed at a normal stress of 6 kPa and the shear-to-failure points were measured at normal stresses of 2.4/3.6/4.8 kPa while using a rotational speed of 0.003 rpm.

4 Results

4.1.1 Temperature-Controlled Cohesion Strength Measurements

Figure 3 shows cohesion strength measurement curves of the sample at 20 and 60 °C. At the beginning of the measurement (interval time 0 – 10 s) a strong increase of the signal can be observed for both temperatures, followed by a constant signal. The increasing signal represents the settling powder bed after fluidization, while the constant value signifies a steady-state flow which is used as a result to compare measurements. Comparing the measurements at 20 and 60 °C, the cohesion strength is significantly lower at higher temperature, indicating that the flow behavior of the powder changes as well. At 20 °C a cohesion strength of CS = 36.2 Pa, and at 60 °C CS=33.6 Pa was measured. The sample shows an improved flowability at higher temperatures. At 60 °C the sample is close to the melting point of monolaurin

(63 °C), therefore the monolaurin seems to be acting as a lubricant at elevated temperatures.

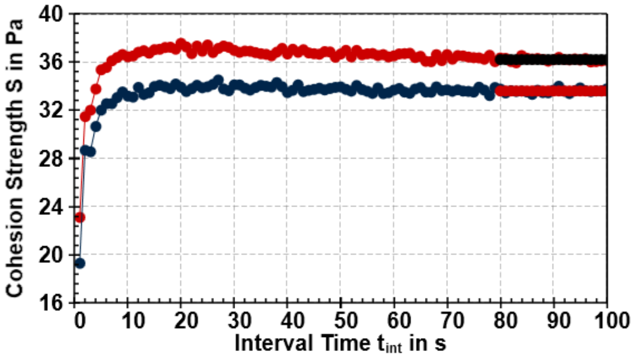


Figure 3: Cohesion strength measurements at 20 °C (red) and 60 °C (dark blue).

Figure 4 shows the change in cohesion strength as a function of temperature. A stepwise increase of temperature was performed from 20 to 60 °C with 10 °C steps. During the temperature increase (at 0.5 °C/min), fluidization was carried out at 1.2 L/min, so well below the flow rate for fluidization (2.3 L/min as described in 3.3.1). This was done to avoid loss of fine particles by the fluidization, while still ensuring a homogeneous and fast temperature distribution within the bulk by the heated air.

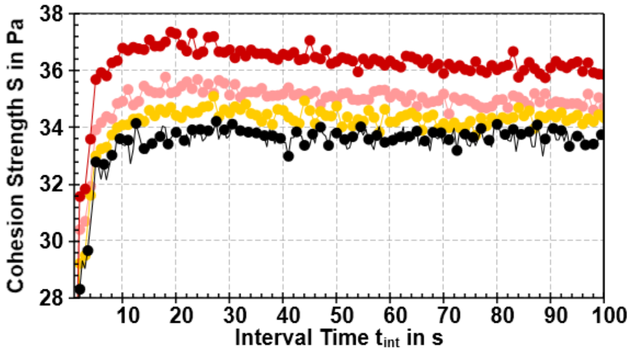


Figure 4: Cohesion strength measurements at 20 °C (red), 30 °C (pink), 40 °C (yellow), and 60 °C (black).

The results of the measurements in Figure 4 are displayed in Table 1. The largest change in cohesion strength can be observed in the first step from 20 to 30 °C (ΔCS = 1.3 Pa). The change gradually decreases for increasing temperature down to a ΔCS = 0.3 Pa from 50 to 60 °C), indicating that the temperature increase has the highest impact in the 20-30 °C range.

Temperature [°C]	Cohesion Strength [Pa]
20	36.2
30	34.9
40	34.4
50	33.9
60	33.6

Table 1: Results of the cohesion strength measurements at different temperatures.

4.1.2 Temperature-Controlled Deaeration Measurements

The measurement curve of the deaeration time is displayed in Figure 5. The pressure curve shows three distinct phases during the deaeration. The first phase (up to approx. 0.8 s) is characterized by a rapid decrease of the pressure signal. A fast release of the surplus air takes place. In the second phase a slower pressure decrease can be observed. The third phase shows a constant pressure signal, which indicates that the samples are fully settled.

For the measurement at 20 °C (red curve) the first and second phase finish after 1.25 s. Consequently, this leads to the conclusion that at 20 °C the sample is fully defluidized after 1.25 s.

The deaeration at 60 °C (black curve) shows a significantly different behavior. The first and second phase are longer than for the cold sample. The deaeration time is therefore significantly extended with 2.9 s. This parameter provides crucial information for discharge, filling and dosing processes, for pneumatic transport (e.g. dilution vs. dense flow), but also for the behavior in a fluidized bed reactor. As can be seen the ambient and process temperature must also be considered in order to be able to set the optimal process parameters. If for example the residence time inside a silo or feeder is under the deaeration time then this can result in uncontrollable outflow (flooding) during discharge.

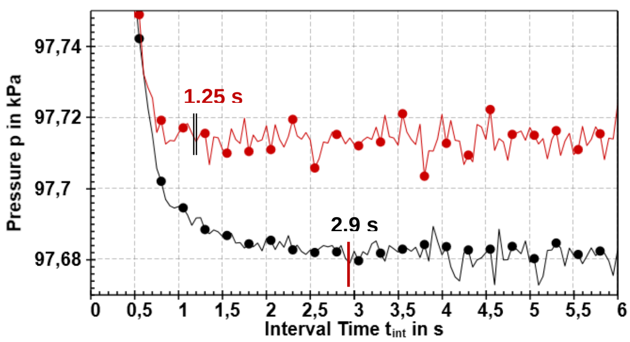


Figure 5: Deaeration time at 20 °C (red) and 60 °C (black).

4.1.3 Temperature-Controlled Shear Measurements

Figure 6 shows a typical shear measurement curve. The shear cell measurement starts by pre-shearing the samples. In this case the measurements were performed at a normal stress (precompaction σ_n) of 6 kPa during pre-shear. The end of the pre-shear step is reached when the shear stress reaches steady-state flow indicated by a constant value and highlighted with black circles in the diagram. After each pre-shear, a shear to failure point is measured at lower normal stresses (here at 2.4/3.6/4.8 kPa). These are indicated by the black arrows in Figure 6.

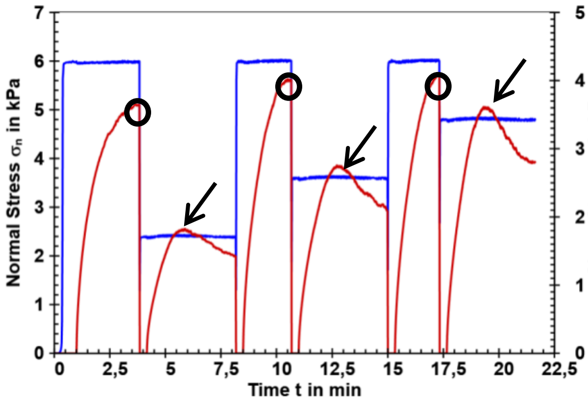


Figure 6: Shear measurement with a precompaction of 6 kPa at 20 °C. Red: shear stress; blue: normal stress; circle: pre-shear; arrow: shear to failure.

Mohr-Coulomb diagrams are used to analyze the raw data to understand the stress conditions within the powder and yield multiple powder parameters, such as σ_c (unconfined yield strength), σ_1 (major principle stress), σ_t (tensile strength), τ_c (cohesion), Φ_{sf} (angle of internal friction) or ρ_b (bulk density). Their definitions are listed in Table 2. Information on how the raw data is analyzed to create a Mohr-Coulomb diagram can be found in the application report “Introduction to powder rheology”.[2]

Parameter	Description
σ_t [Pa]	**Tensile strength** – value of the yield locus for zero shear stress (where it intersects the horizontal axis τ=0). Force necessary to separate a layer of powder from itself.
τ_c [Pa]	**Cohesion** - value of the yield locus for zero normal stress (where it intersects the vertical axis). Resistance to flow when no stress is applied.
σ_c [Pa]	**Unconfined yield strength** – “strength” of the powder” threshold where it begins to flow.”
σ_1 [Pa]	**Major principle stress** – total amount of stress acting inside the bulk (normal and shear stress).
Φ_{sf} [°]	**Angle of internal friction** – during stationary flow, in essence the angle of repose.
ρ_b [g/cm³]	**Bulk solid density** - density of the powder including entrapped air/gas.

Table 2: Parameters resulting from the Mohr-Coulomb diagram.

The Mohr-Coulomb diagram of the shear measurements at 20 and 50 °C are displayed in Figure 7. The results of the measurement at 60 °C are not displayed as the powder was so strongly impacted by the temperature, that the Mohr-Coulomb theory does not apply anymore. The Mohr-Coulomb diagram yields multiple parameters, a brief explanation of them is given in Table 2. The resulting values of the measurements at 20 and 50 °C are compared in Table 3. The sample shows a drastic change in the behavior with increasing temperature. At 50 °C the tensile strength and cohesion drop to very low values, the same is observed for the unconfined yield strength. The major principle stress does not show such an extreme change, indicating that the force transmission between particles remains similar.

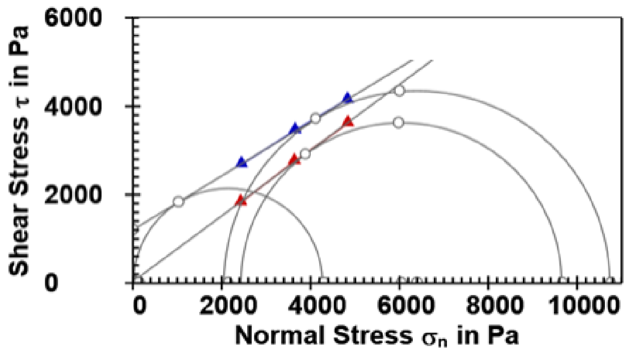


Figure 7: Mohr-Coulomb diagram at a precompaction of 6 kPa, at 20 °C (blue) and at 50 °C (red).

	@ 20 °C	@ 50 °C
σ_t [Pa]	1976	49
τ_c [Pa]	1206	36
σ_c [Pa]	4296	143
σ_1 [Pa]	10750	9677
Φ_{sf} [°]	35.9	31.2
ρ_b [g/cm³]	0.383	0.523

Table 3: Results of the shear measurements at a temperature of 20°C and 50 °C.

The flowability sets into relation the major principle stress and the unconfined yield strength ($ff_c = \frac{\sigma_1}{\sigma_C}$), and helps to numerically characterize it. The graphical representation of ff_c is displayed in Figure 8. At 20 °C the sample shows a cohesive behavior (ff_c = 2.5). When the temperature is increased to 50 °C it shows a drastic change to extremely free-flowing behavior (ff_c = 67.5).

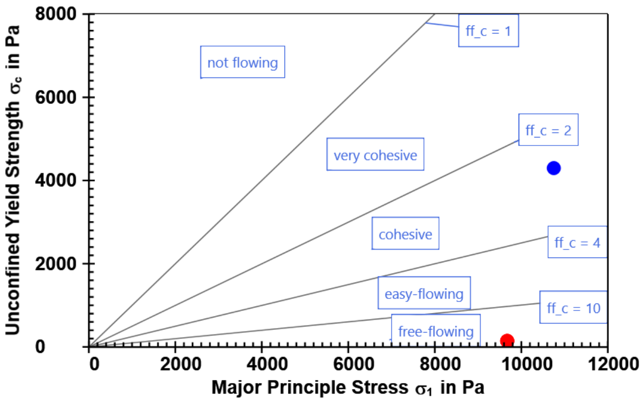


Figure 8: Flowability at 20 °C (blue) and 50 °C (red).

5 Conclusion

The application of Anton Paar's temperature-controlled powder rheology cells were demonstrated in this report. The change in behavior of the powder at different temperatures was characterized both on the powder flow cell and on the powder shear cell. It was shown that a changing temperature has a significant impact on the behavior in a fluidized state, an aerated state, as well as in a consolidated state. Furthermore, the results from both cells correlate well with each other and reveal an increase of flowability with increasing temperature.
Moreover, even small changes can be detected by means of cohesion strength measurements. This method is particularly suitable for incoming and outgoing quality control. In addition, there are often temperature fluctuations during transport, storage, manufacturing processes, etc. which cause a change in the powder properties. This makes temperature-controlled measurements useful for predictions on the behavior during processing as well as storage.

6 References

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

[2] “Introduction to Powder Rheology”, Application Report available on anton-paar.com

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