Powder Shear Cell: How to Characterize the Response of a Polymer Powder (PEG) to Thermal and Hygrometric Conditions

Relevant for: Polymer Production, PEG, Powder Storage, Cohesion, Angle of Internal Friction, Flowability, Powder Flow, Powder Rheology

The processing, handling and storage of powders on an industrial scale are subject to various problematic effects. Changes in relative humidity and temperature can have a strong impact on the powder behavior. Food product, chemicals or drug products are especially concerned by these issues, with the possibility of a major impact on processability and quality. In this report the sensitivity of a PEG polymer towards thermal or hygrometric changes is characterized. The mechanical parameters extracted from the shear cell – cohesion, flow function, angle of internal friction– are shown to describe well the changes in powder behavior.

Figure 1: The powder shear cells which can be equipped in a Modular Compact Rheometer (MCR) from Anton Paar.

1 Introduction

The annular ring shear cell is a scientific tool which provides precise characteristics of a powder. By contrast to "normative" usual tests (such as tapped density, angle of repose), the shear cell allows the user to parametrize the protocol and adapt it to the issue to be simulated. In detail, the shear cell allows a dynamic mode of testing, combining compaction levels, rotation speed, time delays and in the case described here even environmental control. [1]

Many powders show a severe behavioral dependence of the environmental conditions they are subjected to (e.g. temperature and humidity). These behavioral changes express themselves as changes in properties such as flowability, cohesion, angle of internal friction – and many more.

This report focuses on the effects of different temperature and relative humidity levels on the mechanical powder behavior. The objective is to demonstrate how the shear cell can be used to anticipate the changes that the powder properties undergo during transport, storage or variations in the environmental conditions of the day to day production.

This report will not delve into the theoretical depths of shear cell measurements, the interested reader can find detailed explanations on how these measurements are carried out and analyzed in the application report "Introduction to Powder Rheology".

2 Experimental Setup and Samples

2.1 Samples

The studied sample is a polyethylene-glycol (PEG) powder, obtained from an industrial site. PEG is used in many different medical and industrial applications, for example as excipient or as anti-fouling layer. [2] The melting point of PEG strongly depends on the molecular weight, but as expected with a polymer material starts to soften when approaching the melting temperature, resulting in a change of flowability, cohesion etc.

2.2 Rheometer Set Up

A Modular Compact Rheometer from Anton Paar (MCR) equipped with a powder shear cell, a convection temperature device (CTD) and a humidity generator was used for this study.

The following methods were used to characterize the samples:

Shear cell protocol 3-6-9 kPa

Environmental conditions:

- 25°C, dry air
- 25°C, high humidity air (70 ± 2 %rH)
- 40°C, dry air

The measurement protocol involves three preshearing steps while consolidating, each followed by a failure test (shear-to-failure) performed at a lower compaction. This allows to test the powder bed resistance at three intermediate compression stresses below the initial compaction stress. The result of the measurement is expressed in terms of yield locus, cohesion and angle of internal friction (refer to the “Introduction to powder rheology” application note for further details on the shear cell measurement and the associated Mohr theory).

In this work, the powder sample was measured at 3, 6 and 9 kPa of initial compression to simulate low to high powder loading.

3 Results and Discussion

3.1 Raw Data

As described above, for each initial compaction, the measurement routine involves a total of 6 steps allowing successive preshear and shear-to-failure tests. Normal stresses and shear stresses are recorded during the steps, an example is displayed in Figure 2. Their evolution is useful to check the correct steady state during the compressions and shows abrupt spikes for each failure step.

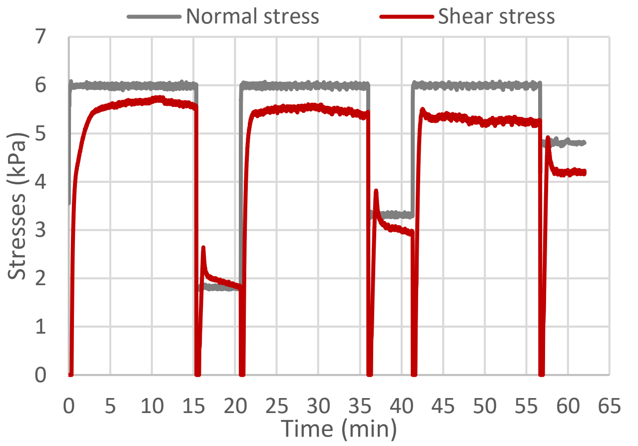

Figure 2: A typical shear cell measurement: 3 preshear steps, each followed by a shear-to-failure test.

The obtained raw data is then processed automatically by the RheoCompass software with respect to the Mohr theory.

3.2 Mohr Circles and their Interpretation

In the Mohr plane, shear stresses are plotted against normal stresses (Figure 3). Specific points appear on the graph, which are the mean initial compaction taken on the 3 preshear steps (circle at normal stress 9 kPa and shear stress 7.1 kPa) and the three shear-to-failure points (black triangles). A linear model is fitted on these 3 shear-to-failure points, which is the yield locus of the powder. Two Mohr-circles are then drawn tangent to the linear fit, one passing by the origin, and the second by the preshear point.

The two maximum normal stresses of each semi-circles are particular values known as the unconfined yield strength (σ_c) and the major principal stress (σ_1). The ratio of these two stresses represent a flowability parameter known as the Flow Function ffc of the powder. The Cohesion is another parameter derived from this figure and is taken as the intercept of the yield locus with the vertical axis. The angle of internal friction (not displayed in this figure) is the angle of the preshear point in regard to the origin.

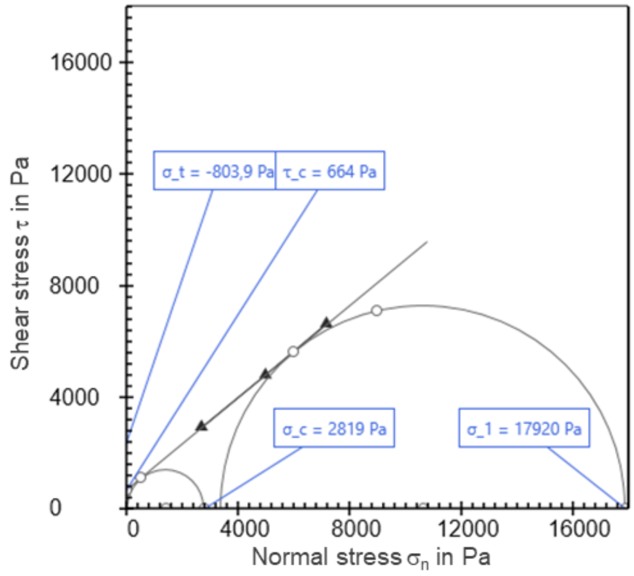

Figure 3: Mohr circles of a typical result, with resulting parameters: σ_t tensile strength, τ_c cohesion, σ_c critical or unconfined yield strength, σ_1 major principal stress.

The next section presents the evolution of these parameters with the different compaction levels and the environmental conditions.

3.3 Results

The cohesion of the PEG powder is displayed in Figure 4 at three precompaction values and with three different environmental conditions.

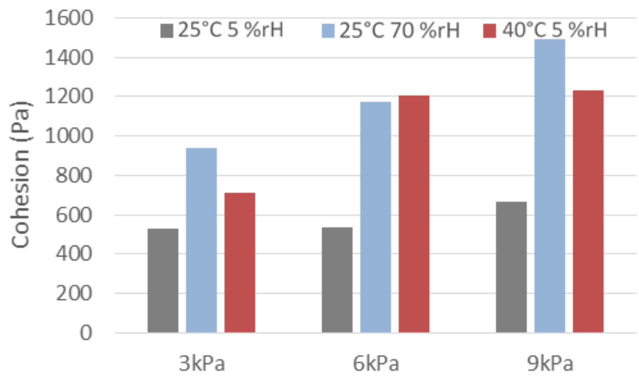

Figure 4: Cohesion in dependence of compaction and environmental conditions. Gray: 25°C and 5 %rH. Blue: 25°C and 70 %rH. Red: 40°C and 5 %rH.

Compared to the reference condition (25°C dry air), the PEG powder exhibits approximately a twice as high cohesion in both 25°C humid condition and 40°C dry condition. What is particularly interesting is the similarity of the cohesion between these two conditions. Humid air at room temperature impacts the powder behavior in a similar way as higher temperatures without humidity. The PEG powder will then tend to form aggregates in these conditions.

The angle of internal friction in dependence of the environmental conditions and the compaction is displayed in Figure 5. For all conditions, a decreasing angle can be observed with increasing compaction. The reference condition (25°C dry air) shows the lowest angle, while the two other conditions are higher and are like the cohesion relatively similar.

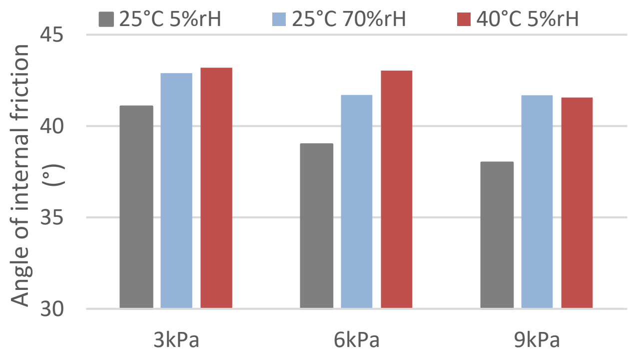

Figure 5: Angle of internal friction in dependence of compaction and environmental conditions. Gray: 25°C and 5 %rH. Blue: 25°C and 70 %rH. Red: 40°C and 5 %rH.

The flowability (ffc) for the three conditions is shown in Figure 6. In a first observation of the ffc diagram, the general behavior (so for all ambient conditions) shows an improving flowability as the stress conditions increase (i.e. going from left to right). This is simply due to a load effect, which allows the powder to flow more easily as there is more mass above itself.

Taking also into account the ambient conditions, the same general behavior can be observed in the flowability as was for the cohesion and angle of internal friction – that 25°C in dry environment has the best flowability, while 25°C humid condition and 40°C dry condition flow less well and are in very close proximity to one another.

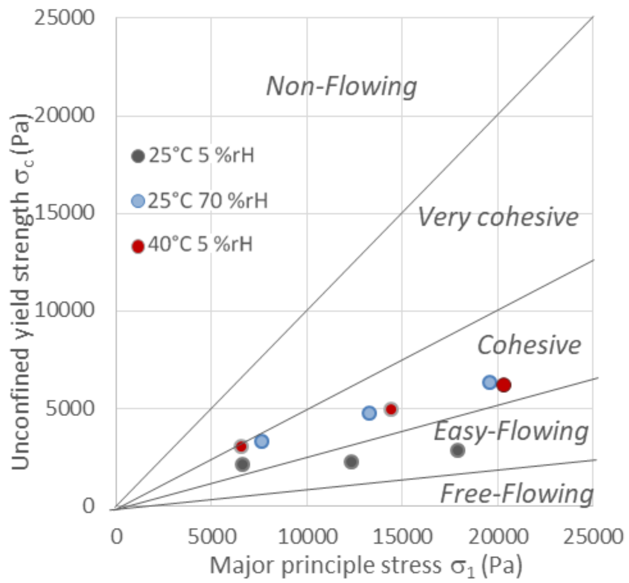

Figure 6: PEG powder flowability ffc classification. Gray: 25°C and 5 %rH. Blue: 25°C and 70 %rH. Red: 40°C and 5 %rH.

Note: Measurements were also carried out at 50°C. At this temperature, the powder behaves in such a manner that we suspected some melting to occur. As the theoretical background of the measurement relies on bulk solid/powder behavior, the resulting points are not shown because the product is no longer purely a powder and therefore not a useful comparison.

4 Conclusion

The shear cell can be used as a tool to obtain focused insights on powder behavior. This application note shows how easy it is to quantify the impact environmental conditions have on the behavior of a polymer powder.

Compared to room temperature and dry conditions, this particular powder (polyethylene glycol, or PEG) showed a clearly changing behavior for humid conditions at room temperature, as well as for dry conditions at elevated temperatures. In both cases an increase in angle of internal friction, as well as a strong increase in cohesion was observed. For the sample measured, a high relative humidity at ambient temperature acted on the powder in a very similar way as the elevated temperature.

The data analysis also shows the powders tendency to form aggregates and quantifies how the powder flowability deteriorates with humidity level or temperature rise.

5 References

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

2. **Aschl, T.** *Biochips based on silicon for detecting the interaction between aptamers and pathogens*, 2016.

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