Powder Flow Cell: Characterization of Powder Behavior in Manufacturing Processes of Polymers

Relevant for: Polymer Production, Polyacrylamide, Powder Flow, Cohesion, Wall Friction, Powder Rheology

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The manufacturing of powders with consistent behavioral characteristics can be difficult, especially on an industrial scale. Parallel manufacturing lines often generate products with different characteristics despite using the same processing parameters, which can cause problems in the manufacturing process itself and it is also an issue for the final product quality. Therefore fine tuning of processing parameters can help to establish and hold a well-defined output quality. In this report, various characteristics are measured, and their impacts on processes are analyzed. The addressed characteristics are cohesion and abrasion of the tubing, as well as bridging in powder processes.

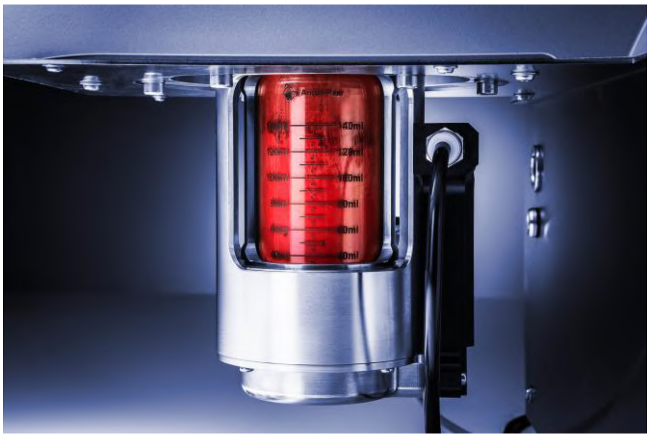

Figure 1: The powder flow cell mounted on a Modular Compact Rheometer (MCR) from Anton Paar.

1 Introduction

Cohesion of a bulk solid is a mechanism leading to an enhanced mechanical resistance to shear of the powder. Various physical characteristics are involved in cohesion, such as particle size distribution, particle shape, particle elasticity, presence of humidity or chemical reactions on the surface.

The cohesion in uncompressed powders can be estimated in the Anton Paar powder flow cell by monitoring the torque needed to rotate a blade inside the bulk at a given speed (see the application report: Introduction to Powder Rheology). However, some applications need to measure the cohesion with a given pre-compaction, which can be achieved with a variation of the Warren Spring Method.

Abrasion between the tubing and the powder particles can be compared quantitatively between samples by using wall friction measurements. The tubing material can be mounted onto a specific measuring tool and will yield the friction coefficient at the applied normal force.

Bridging can occur when both friction inside the bulk (internal cohesion) and the wall friction reach high values. The internal cohesion of the bulk is a powder bulk parameter which can be measured in the powder flow cell with the Warren-Spring geometry. The approach is similar to the method for cohesion measurements; but it takes place on a pre-compressed bulk. The Warren-Spring tool allows one to measure shear strength within the powder itself, thus defining internal cohesion.

This work exemplifies how this set of physical parameters can be used to compare and quantify the behavior of two similar polymer powders.

2 Experimental Setup and Samples

2.1 Samples

The two samples were polyacrylamide (PAM) polymer powders, obtained from an industrial site. Each sample came from a different production line. Both lines were similar in terms of unit operation; but their fine-tuning differed slightly - as is common in industrial processes. Those differences led to production problems within one of the lines, which was particularly sensitive to clogging and flow rate issues.

2.2 Rheometer Set Up

An Anton Paar MCR rheometer equipped with the powder flow cell was used for this study.

The following combined methods were used to fully characterize the samples:

- Compressibility was determined to understand the samples' behavior when they were being consolidated.
- Cohesion strength was measured with a two-blade stirrer after static compaction with the compression tool (equipped with an air-permeable stainless-steel disc). This method was used to address the stress needed to restart flow of powders after they had been compressed to different degrees. This method was in contrast to the usual test for cohesion strength where a powder is fluidized to achieve a repeatable (albeit non-compressed) state. Here however, static compaction was used to simulate powder mass as it is involved in the industrial process.
- Warren-Spring cohesion measurements were carried out with the Warren-Spring geometry to estimate the internal friction of each sample. Here again, the powders were compressed before the measurement.
- Wall friction was measured with the compression tool, which was equipped with a stainless steel disc (similar to industrial tubing).

3 Results and Discussion

3.1 Compressibility

During the tests, the same mass of powder was used in the cell (30 g). The rheometer's normal force detection was used to study the compaction behavior of the powder as the normal stress was increased during the compaction step (Figure 2).

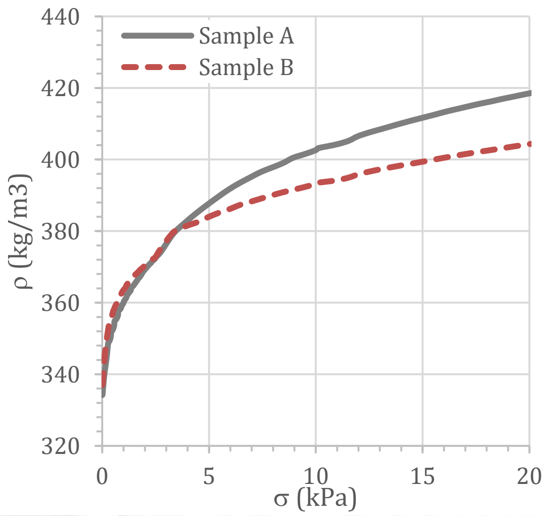

Figure 2: Apparent density of the bulk during the compaction step, depending on the normal stress σ applied.

The two samples behaved similarly up to around 2.5 kPa. At higher normal stresses (between 2.5 and 20 kPa), sample A showed a higher compaction. In this range, the two samples showed distinct compaction behaviors. This method gave more precise and continuous insight into powder behavior than the classical Hausner Ratio or Carr Index.

3.2 Resistance to Flow

The resistance to flow was measured via the torque needed to rotate the two-blade stirrer inside the bulk. To address the specific industrial issue described here, the procedure involved first compressing the powder at various normal stresses and then measuring the cohesion strength for each compaction (Figure 3). The values used for analysis were the initial cohesion strength, as well as the mean value of the last 20 seconds (out of 100 seconds), as displayed in Figure 4.

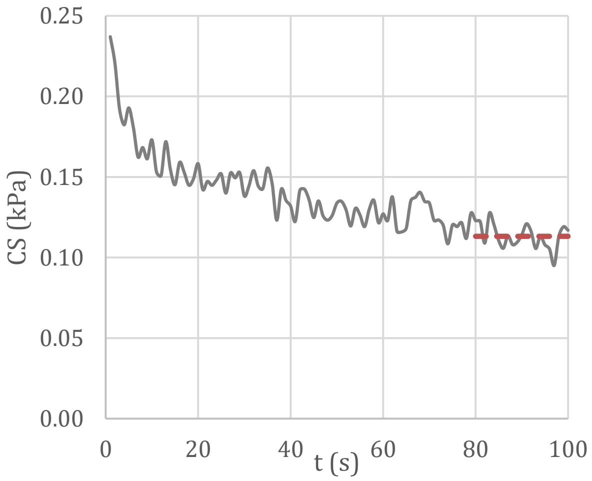

Figure 3: Typical cohesion strength CS (kPa) over time for sample A, pre-compacted at 15 kPa. The initial value was used, as well as the mean value of the last 20 seconds (red dots) for analysis.

The results of the cohesion strength measurement after pre-compaction at 1, 5 and15 kPa are displayed in Figure 4. The gray bars depict the initial value (start of test), while the red bars show the mean value of the last 20 seconds (end of test). Observing the gray bars, it is first noticeable that the torque needed to initiate flow rose as the pre-compaction stress was increased. Both samples required approximately the same stress to initiate flow after compaction (gray bars) with a significantly lower value for sample B at a pre-compaction of 5 kPa, which might suggest an irregularity of the measurement.

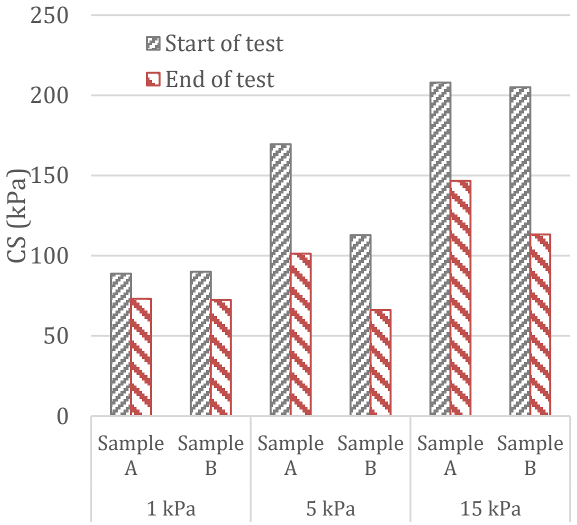

Figure 4: Cohesion strength CS (kPa) measured just after compaction (start of test) and after steady state was achieved (end of test) for the two samples at three compaction normal stresses.

After steady state was achieved (red bars), sample A still required more torque than sample B (mean value over all normal stresses was 30 % higher).

This correlated well with visual observations of both bulk solids, which showed agglomerations which broke up quickly with sample B.

Sample A reached higher compactions (Figure 2) and formed a more cohesive bulk (Figure 4) as soon as it was compressed/compacted.

3.3 Internal Friction

The cohesion strength measured in the previous step was related to free-flowing powder and measured the ability to flow. With the Warren-Spring geometry, the internal friction of the bulk solid was evaluated. The method involved first a compaction of the powder bulk with a piston equipped with an air-permeable disc. The Warren Spring geometry was then inserted into the bulk and measured the internal cohesion by slowly shearing the bulk.

The obtained stress curve presented a peak just before the sample “breaks”, or starts to flow. The maximum value was taken as the Warren Spring Cohesion (C_{WS}) (Figure 5, Table 1).

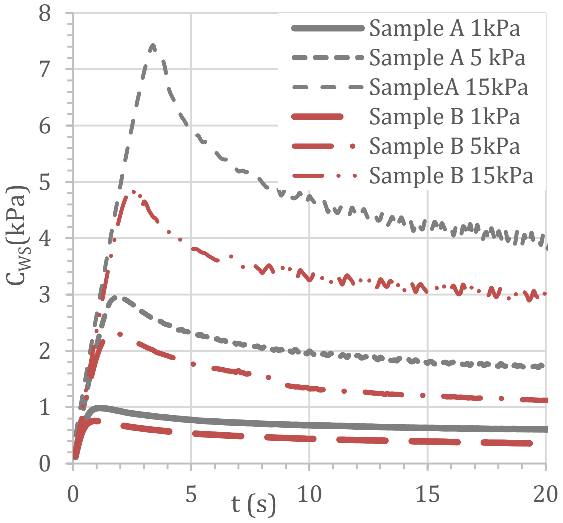

Figure 5: Warren-Spring Cohesion (C_{WS}, kPa) over time (s) for the two samples at 1 kPa, 5 kPa and 15 kPa pre-compaction.

For each pre-compaction, sample A showed a higher cohesion strength than sample B (Table 1 below).

Sample	*A*	*B*	*A*	*B*	*A*	*B*
σ_N *(kPa)*	1		5		15	
C_{WS} *(kPa)*	0.99	0.76	3.0	2.3	7.4	4.9
ΔC_{WS} *(A/B)*	**30%**		**27%**		**52%**	

Table 1: Maximum stress at the break point as a function of pre-compaction stress σ_N for both samples.

This method confirmed that, when compressed, sample A exhibited a higher cohesion among particles for each pre-compaction studied. For moderate pre-compactions (1-5 kPa) the cohesion was about 30 % higher. For more pronounced pre-compactions (15 kPa), the cohesion was 52 % higher for sample A.

3.4 Wall Friction

Bridging can occur in pipes or tanks due to a combination of high powder cohesion and too high wall friction.

This last procedure in this study used a flat stainless-steel disc which rotated on the powder bulk. The normal force was controlled, and the frictional torque was measured. The wall friction angle was then determined for each product.

For these two samples, a linear ramp of normal stress from 0 to 20 kPa was imposed, together with a constant rotational velocity. The results are displayed in Figure 6.

A linear interpolation over the measured points for both products gives the wall friction angle. Sample A exhibited a wall friction angle of 34°, and sample B of 29°.

Once again, sample A exhibited characteristics of a rougher powder than sample B.

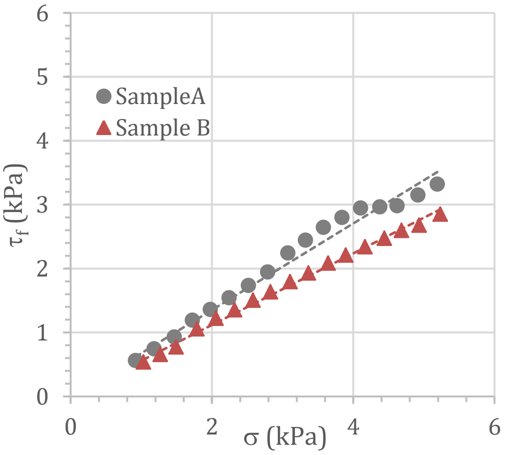

Figure 6: Frictional stress τ_f as a function of the normal stress σ for both samples.

4 Conclusion

The four methods used in this study addressed multiple aspects of powder characteristics using one primary device.

Sample A was found to be more compressible, presented a higher cohesion upon mixing and a higher internal friction. It also led to higher friction forces, despite being the same chemical molecule.

The powder flow cell was proven to be a versatile device capable of addressing industrial issues and giving quantitative values for multiple aspects of powder. The small sample volume and the clean working environment made it easy to qualify the products.

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