

## Influence of Moisture Content on Powder Flowability During Extruder Feeding

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#### Introduction

When running an extrusion process, many parameters like the thermal-mechanical energy input induced by the extruder barrel, the screw configuration used, or the applied screw speed play an important role for optimal processing. However, the continuous flow of raw material into the extruder is of utmost importance. To ensure a constant feed, raw materials are fed into the extruder in granular form via single or twin screw feeder before entering the extruder barrel and passing into several conveying and kneading zones during the extrusion process itself.

Flowability is a key factor when it comes to establishing an effective process to ensure that the desired amount of material is fed into the extruder. The flowability of granular materials highly depends on particle shape and size as well as particle surface properties, among other things. Additionally, the moisture content of the materials must be considered; as moisture content increases, liquid bridges tend to form between particles. This can lead to agglomeration, hence increasing cohesiveness and lowering flowability. Modern rheometers can be used to investigate the flowability of various samples. This application note intends to showcase the usage of powder rheological techniques to characterize the flowability of potato starch powder with increased moisture content with respect to its feedability for food extrusion processes.

### Materials and methods

For this study two potato starches with respective moisture contents of 17.4 % and 19.7 % were used.

To characterize the flowability of the powders, a Thermo Scientific<sup>™</sup> HAAKE<sup>™</sup> MARS<sup>™</sup> iQ Rheometer equipped with the powder rheology measuring geometry was used. Figure 1 shows the utilized measuring geometries.



Figure 1. Overview of powder rheology measuring geometries powder vane rotor (left), porous piston (middle), and powder shear rotor (right), needed to perform powder shear tests.

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The powder flowability was characterized by a powder shear test following ASTM D 7891.

Figure 2 shows the measurement procedure in the Thermo Scientific™ HAAKE<sup>™</sup> RheoWin<sup>™</sup> Software.



Figure 2. Measurement routine for powder shear test with a pre-shear stress of 3 kPa in the HAAKE RheoWin Software.

First, the sample is loaded into the funnel and an initial conditioning cycle with a tip speed of 40 mm/s is performed using the powder vane rotor to remove all loading related stresses and to ensure that the sample is homogenously filling the cup. After this, the rotor is changed to a porous piston to pre-consolidate the powder with a constant normal stress of 3 kPa for 1 min. The porous body of the piston allows air to pass through the rotor during this phase.

After that, the sample split is performed by sliding the funnel sideways onto the reservoir to remove excess and to obtain a defined sample volume of 13.3 ml in the powder shear cup. By removing the cup from the triangular adapter plate and placing it on a scale, the powder mass can be obtained and entered in the HAAKE RheoWin Software to calculate the conditioned bulk density (CBD). Afterwards, the porous piston rotor is exchanged for a powder shear rotor with profiled surface and lowered onto the powder bed. After a normal stress  $\sigma_{ps}$  of 3 kPa is reached, the test starts.

The powder shear test consists of several shear cycles in which the powder is first subjected to multiple pre-shear steps. This is followed by a single shear step during which the failure of the sample is observed. At this point, the internal structure of the powder can no longer withstand the applied stress, and the sample starts to flow.

During the pre-shear phase as well as the shear step, the rotor rotates with a fixed rotational speed of 0.05 rpm.

During pre-shear, the sample is subjected to a pre-shear normal stress  $\sigma_{\rm ps}$  to consolidate the sample and the resulting shear stress  $\tau_{\rm ps}$  is recorded. The aim of this phase is to allow the sample to reach a steady state flow condition and to attain a well-defined and reproducible state of consolidation. This is also referred to as critically consolidated state.<sup>1</sup>

The pre-shear phase is successfully completed when the recorded shear stress of two consecutive steady state pre-shear phases is within a tolerance  $\Delta$  of 2 %. In case no steady state is reached or if the tolerance is not met, another pre-shear step is conducted.

During the following shear step, the powder is subjected to a lower normal stress and the shear stress is recorded up to the point in which the powder bed fails and the powder starts to flow. The resulting maximum in the recorded shear stress curve as a function of the applied normal stress represents to a single yield point value of the powder sample.

The principle of applying a pre-shear normal stress  $\sigma_{ps}$  before obtaining any yield point at a normal stress  $\sigma_i$  lower than  $\sigma_{ps}$  is illustrated in Figure 1.



Figure 3. Example shear cycle based on pre-shear phase and shear step.

To obtain a set of powder shear measuring data, a whole series of yield points at different normal stress levels is recorded, each preceded by a new pre-shear phase. Table 1 shows the used normal stresses for each pre-shear and the respective yield points.

	Normal stress $\sigma$
Pre-shear	3 kPa
1. Yield point	2.1 kPa
Pre-shear	3 kPa
2. Yield point	1.75 kPa
Pre-shear	3 kPa
3. Yield point	1.5 kPa
Pre-shear	3 kPa
4. Yield point	1.25 kPa
Pre-shear	3 kPa
5. Yield point	1 kPa

Table 1. Normal stresses for pre-shear and yield points used for this application report.

Out of the series of yield points one can generate the yield limit of a consolidated bulk solid in a ( $\sigma$ ,  $\tau$ ) diagram, the so-called yield locus. This yield locus is only valid for one defined consolidation state which has been achieved during pre-shear. Hence, different pre-shear stresses will lead to different yield loci.<sup>1</sup>

With respect to the yield locus and the pre-shear point, different evaluation parameters can be obtained. By applying a linear curve fit through the yield locus, the τ-axis interception corresponds to the cohesion C of the sample. However, samples that are not free-flowing, usually exhibit a slightly curved yield locus.<sup>2</sup> In such cases, the cohesion parameter obtained by the T-axis intercept of the idealized linear curve fit may not correspond to the actual sample behavior. Besides this, two Mohr stress circles are constructed based on the vield locus and the pre-shear point, where a small circle is drawn tangential to the yield locus passing through the origin. The greatest value at which the small circle intercepts the x-axis is called the unconfined yield strength (UYS or  $\sigma_{\rm c}$ ). The second large circle is also drawn tangential to the yield locus but passing through the pre-shear point ( $\sigma_{_{\rm ps}},\,\tau_{_{\rm ps}}$ ). The greatest value at which the large circle intercepts the x-axis is called the major principal stress (MPS or  $\sigma_{i}$ ). The center of both circles is located per definition on the  $\sigma$ -axis. Based on the ratio of MPS to UYS, the flow function (FF) can be calculated. According to Jenike,<sup>3</sup> the flow function can be used to group flow of granular materials into different categories (Table 2).

Expected powder flow	Flow function (FF)	
Not flowing	FF > 1	
Very cohesive	1 < FF < 2	
Cohesive	2 < FF < 4	
Easy flowing	4 < FF < 10	
Free flowing	FF > 10	

Table 2. Classification of powder flowability according to Jenike<sup>3</sup>.

Lastly, the effective angle of internal friction (eAIF) can be determined by a line that starts from the origin of the diagram and touches the larger Mohr circle.

Figure 4 shows the evaluation of the different parameters based on an example set of shear test data.



Figure 4. Example shear test data with respective evaluation based on two Mohr stress circles.

To understand the feeding performance of the two potato starch samples in the actual process, calibration curves were recorded to understand the achievable throughput as a function of screw speed using a gravimetrical Kubota Brabender MiniTwin MT feeder.

#### **Results and discussion**

A stable extrusion process relies on a constant and well-defined supply of raw material. Hence, the throughput of the feeder needs to be calibrated, so that the amount of granular material which is fed into the extruder can be precisely controlled. Figure 5 shows such a calibration curve for the two potato starch samples investigated in this study.



Figure 5. Feeder calibration curve for potato starch with 17.4 % and 19.7 % moisture content.

At screw speeds below 50 % of the maximum scale, there is almost no difference between 17.4 % and 19.7 % moisture content. However, above 50 %, the throughput of the potato starch sample with less moisture increases less compared to the other sample. This indicates, the difference of 1.7 % moisture is enough to change the feeding behavior resulting in less powder fed into the process at high screw speeds. Therefore, the extruder cannot process extrudates with the same quality and texture with higher throughput, as less potato starch is processed.

To understand this change in powder flowability, powder shear tests can be conducted as described above. Figure 6 shows the powder shear test results of both samples with a pre-shear normal stress of 3 kPa.



Figure 6. Results powder shear tests on potato starch with 17.4% and 19.7% moisture content.

The evaluation results utilizing the two Mohr stress circles are summarized in Table 3.

The results show only marginal differences in cohesion as well as unconfined yield stress. This is in accordance with the work of David J. Sun et al.<sup>4</sup> who found out that there is a proportionality between powder cohesion determined from the  $\tau$ -axis intercept and UYS derived from shear cell testing. However, this also implies that the cohesiveness of the sample does not change significantly with only 1.7 % difference in water content.

Besides this, both the major principal stress and the effective angle of internal friction show a stronger dependency on the moisture content.

The influence of moisture content on the effective angle of internal friction is in accordance with Stoklosa et al.<sup>5</sup> who found that the effect of moisture sorption of powders from surrounding air is reflected by a change in the resulting effective angle of internal friction. The increased eAIF with increased moisture content indicates higher friction between different planes inside the powder sample, corresponding to less flowability. Hence, higher levels of moisture can lead to reduced powder flowability due to changes on the particle's surface resulting from moisture absorption. This leads to the formation of liquid bridges between particles.

	Cohesion (C) in kPa	Unconfined yield stress (UYS) in kPa	Major principal stress (MPS) in kPa	Effective angle of internal friction (eAIF) in °	Flow Function (FF)
Moisture content 17.4 %	0.44	1.52	4.04	40.5	2.64
Moisture content 19.7 %	0.40	1.53	4.41	46.4	2.88

Table 3. Evaluation powder shear test results of potato starch with a pre-shear normal stress of 3 kPa.

Despite only little difference in UYS, the flow function of 2.88 shows a slightly more cohesive flow behavior of the sample with 19.7% compared to 2.64 value determined for 17.4 % moisture content. However, this is only valid for the stress state chosen for this study. When performing powder shear tests with a different pre-shear consolidation stress, the flow behavior might be different. Depending on the application at hand, it can therefore be beneficial to further investigate the shear behavior at different stress states by choosing one of the other pre-defined stress tables for 6, 9 or even 15 kPa pre-shear stress available in HAAKE RheoWin Software.

#### Conclusion

In this application note, the influence of moisture content on the flowability of potato starch was investigated and linked to the throughput of a gravimetrical feeder for food extrusion applications. An increase in moisture content of potato starch had a negative effect on the throughput, hence posing a potential issue for establishing an efficient extrusion process. Powder shear testing proved to be a valuable tool for differentiating the different moisture contents based on their effective angle of internal friction as well as their flow function. Based on such findings, powder performance for extrusion processes can be evaluated, controlled and optimized to allow for efficient production of extrudates with the desired textures.

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