Powder Shear Cell: Temperature Influence on the Flowability of Polyvinylchloride Powder

Relevant for: Polymers, Powder Rheology, (Pneumatic) Transport, Temperature Control, PVC

Polyvinylchloride (PVC) is a commodity plastic used in many applications. During production it is commonly transported in its powdered form through the use of pneumatic conveying. This transport process however reveals surprising complexities that arise from the use of screw extruders and the modification of PVC powder. The powder shear cell proves to be a valuable tool for investigating the impact of these process conditions on the powder behavior of PVC.

1 Abstract

Next to the production itself the transport of a good within or outside of its production site is one of the most important considerations for manufacturing. Multiple steps need to be in sync and improvements to one step do not necessarily transfer to another. Such is the case in this study.

PVC powder is produced in a reactor and then pneumatically transported throughout the plant to the site of screw extruders [1]. Changes in the formulation of the powder, in this case the change of an extruder lubricant can have effects on the whole process chain.

1.1 Introduction

Extruder lubricants are usually paraffins that are added to the powder feedstock to facilitate a clean break from the extruder wall materials at elevated temperatures [2]. It is expected that they have little to no influence on the powder behavior prior to the melting step in the extruder, therefore the effect on the transport process is usually neglected. However, in this case study a substantial increase in pneumatic pipeline blockages was detected after a change of the extruder lubricant.

Prior measurements include Dynamic Scanning Calorimetry (DSC) and Laser Granulometry, both of which yielded no substantial differences in the results.

Soon the pneumatic conveying process itself became a target of concern. When transported through a pneumatic conveying pipe the powder can experience a variety of detrimental effects. The powder itself is still hot when leaving the reactor and the constant deformation of particles colliding in the fluidized stream within a conveying system will heat the powder further. Additionally since PVC is an isolator, tribocharging and frictional heating is another influence [3]. Upon further examination, temperatures exceeding 140 °C were measured at certain bends and kinks in the conveying system. This led to an examination of the temperature dependent flow behavior of the powder and a subsequent resolving of the issue.

2 Experimental

2.1 Samples and measurement setup

Four different samples were investigated as presented in Table 1. They are distinguished by physical characteristics (blocky or round) and the applied extrusion lubricant.

Sample Name	Particle shape	Extrusion lubricant	Color in diagrams
Sample A	Blocky	Formerly used	Red
Sample B	Blocky	New	Black & grey
Sample C	Round	Formerly used	Green
Sample D	Round	New	Blue

Table 1: Investigated PVC samples differing in their particle shape and lubricant

The measurements were performed with an air-bearing-based Modular Compact Rheometer (MCR) of Anton Paar. The MCR was equipped with a convection temperature device (CTD) and a powder shear cell (PSC). The setup is illustrated in Figure 1. The influence of temperature on the powder behavior of the differing PVC powder was characterized.

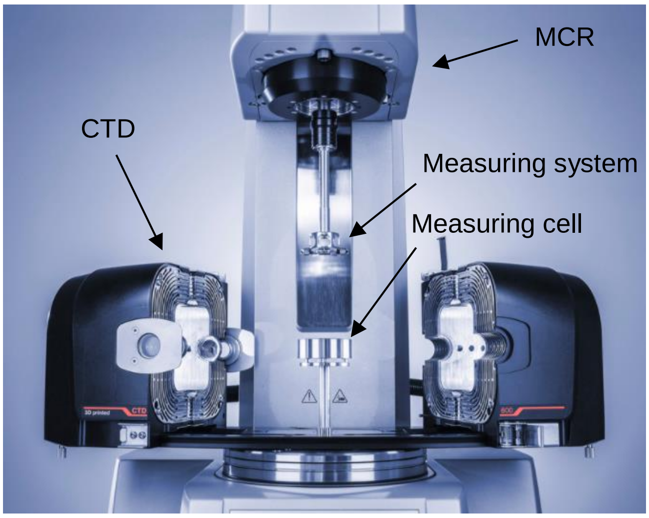

Figure 1: MCR equipped with a powder shear cell and a convection temperature device

In order to combat the excessive free flowing nature of the PVC powders, the conventional preshear-shear sequence was adapted accordingly. Instead of consolidating the powder in the preshear phase until a constant torque is reached, the powder was intentionally overconsolidated. Thus, the preshear phase was stopped only after overconsolidation and when the torque became constant (as shown in Figure 3-5). This typically increases the consolidation of the powder, and consequently leads to a slightly increased major principle stress σ_1 [4]. However, this offset was accepted considering that powders of this nature could not be reliably characterized otherwise by shear measurements and the main purpose of this analysis is comparison.

2.2 Influence of Temperature on Flowability

The PVC powder was filled into the ring shear cell which was placed within the CTD on the MCR (see Figure 2). When measuring at elevated temperatures, the sample was exposed to the set temperature for 20 minutes to equilibrate. The shear measurements were performed at a normal stress σ_{pre} during pre-shear of 1-3-5 kPa at ambient conditions and at 150 °C with a rotational speed of 0.006 rpm. For each pre-shearing phase, 3 shear-to-failure points were performed. Before each shear-to-failure point a pre-shearing was carried out (for further details on shear cell measurements, please refer to the application report “An Introduction to Powder Rheology”).

Figure 2: Powder Shear Cell with measuring system

3 Results

3.1 Influence of Temperature on Flowability

Initially all samples were measured at ambient temperature (25 °C). Neither the differences in particle shape nor in the applied lubricants lead to significantly different results during the shear measurements at ambient temperature. This is exemplarily shown by comparing the shear measurements at 5 kPa precompaction of sample A and B, both consisting of blocky particles but containing the formerly used and the new extrusion lubricant (Figure 3). Thus, samples A through D do not exhibit differences in their flowability at ambient temperature despite of their difference in particle shape and/or the contained extrusion lubricant. The detailed results of the shear measurements can be found in the Appendix section 5.

However, the powder behavior changes substantially when the shear measurements are conducted at 150 °C. Again, the results are exemplarily shown for sample A and B for which different lubricants were applied (Figure 4).

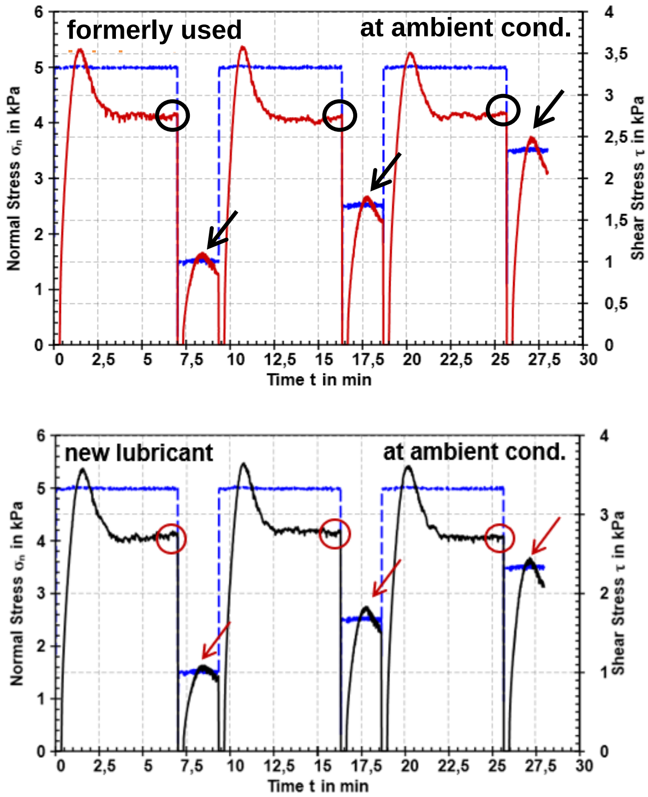

Figure 3: Comparison of preshear-shear curves at 5 kPa precompaction between PVC powder containing the formerly used (sample A; top) and the new extrusion lubricant (sample B bottom). Even though the powders differ in contained lubricant, they show similar behavior during shear measurements at ambient temperature.
Circles: pre-shear points
Arrows: shear-to-failure points

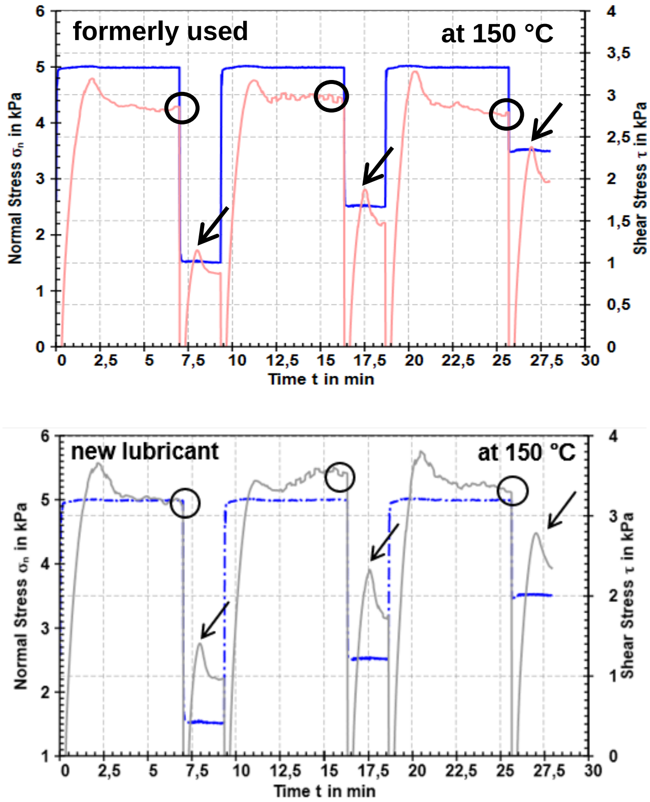

Figure 4: Comparison of preshear-shear curves at 5 kPa precompaction and at 150 °C between the powders containing the formerly used (sample A; top) and the new lubricant (sample B bottom). The powder behavior at 150 °C changes substantially as compared to its behavior at ambient temperature.
Circles: pre-shear points
Arrows: shear-to-failure points

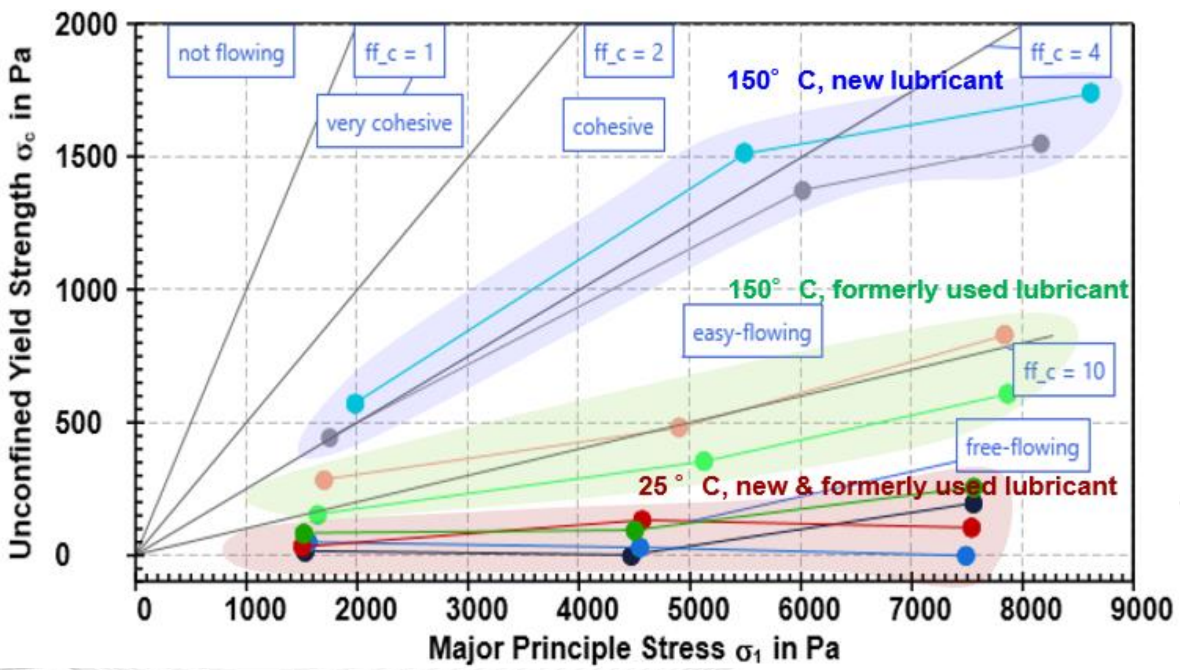

Figure 5: Overview of all measurements showing 3 different resulting clusters for the powders' flowability: one at ambient temperatures for all powders irrespective of lubricant and particle shape (red cloud); two separate clusters at 150 °C for the powders containing the new lubricant (blue cloud) and formerly known lubricant (green cloud) irrespective of the particle shape.

The curves of the shear sections no longer show signs of excessive free flowing especially with the new lubricant. This is demonstrated by the sharp shear to failure points. This is further substantiated in the complete measurement as the cohesive values (tensile strength σ_t, cohesion τ_c and unconfined yield strength σ_c), significantly increased in case of the powders containing the new lubricant (see Appendix section).

Furthermore, the differences resulting from the two lubricants at 150 °C are clearly demonstrated in the ff_c plot of all measured powders and precompactions at the two test temperatures (ambient and 150 °C), see Figure 5. (The ff_c plot helps to classify the powder flowability into categories from free-flowing to not flowing at all. The categorization is based on the individual results from the Mohr cycles given in the Appendix section. More details can be found in the application report "An Introduction to Powder Rheology").

Whereas the flowability presented by the ff_c value did not change significantly at ambient conditions neither in dependence of the particle shape (blocky vs. round) nor of the applied extrusion lubricant (formerly used vs. new), it changed depending on the applied lubricant when determined at 150 °C.

At ambient temperature, all powders were classified in the lower range of the free-flowing area. When increasing the measuring temperature to 150 °C, the flowability of the powders containing the formerly used lubricant decreased and was moved towards the easy-flowing behavior. However, for powders containing the new lubricant, their flowability was categorized mostly cohesive. Thus, their flowability was further decreased. This explained the problems observed when conveying the PVC powder containing the new extrusion lubricant. Most likely this is due to a shorter C-chain paraffin being used in the new extrusion lubricant to increase lubrication in the extrusion process.

4 Conclusions

In conclusion the influence of the type of lubricant for extrusion can be clearly seen to effect values what that are aligned with conveying effects, i.e. inter alia the powder's flowability, whereas the particle shape only showed little impact on the powder's flowability. This shows that despite the results on DSC being similar the powder flow parameters being virtually identical at ambient temperature, once the temperature is raised the differences can be significant.

Using temperature-controlled powder rheological measurements provides a way to identify problems of this type during formulation, and guarantee a smooth operation of the process.

5 Appendix

Powder characteristic	Ambient temperature		150 °C	
Particle Shape	Blocky			
Sample	A	B	A	B
Extrusion lubricant	Formerly used	New	Formerly used	New
Tensile strength σ_t in Pa	39.66	75.89	374.2	582.2
Cohesion τ_c in Pa	27.56	51.95	231.6	405.1
Unconfined yield strength σ_c	105.4	197.1	831.6	1551
Major principle stress σ_1	7542	7560	7841	8168
Angle of internal friction Φ_{sf} in °	28.8	28.82	29.58	32.49
Bulk density in g/cm³	0.781	0.783	0.788	0.666

Table 2: Powder characteristics of PVC powders deduced from shear measurements at 5 kPa precompaction, at ambient temperature and at 150 °C from powder consisting of blocky particles (sample A & B). Differences caused by the lubricant are only detected when measured at 150 °C

Powder characteristic	Ambient temperature		150 °C	
Particle Shape	Round			
Sample	C	D	C	D
Extrusion lubricant	Formerly used	New	Formerly used	New
Tensile strength σ_t in Pa	97.14	0	275.5	714.6
Cohesion τ_c in Pa	67.22	0	170	469.8
Unconfined yield strength σ_c	256.5	0	609.5	1742
Major principle stress σ_1	7563	7491	7869	8618
Angle of internal friction Φ_{st} in °	29.01	29.07	29.33	33.42
Bulk density in g/cm³	0.766	0.748	0.729	0.661

Table 3: Powder characteristics of PVC powders deduced from shear measurements at 5 kPa precompaction, at ambient temperature and at 150 °C from powder consisting of round particles (sample C & D). Differences caused by the lubricant are only detected when measured at 150 °C

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