Hot Gas Filtration: A Simple Screening Method for Optimized Processing Conditions with a Powder Shear Cell

Relevant for: Powder Stickiness, Powder Fusibility, Hot Gas Filtration, Sulfuric Acid Regeneration, Powder Rheology

Hot gas filtration is used to remove particulate matter in process gas flows at high temperatures up to 1000 °C. This filtration method is used for sulfuric acid regeneration, the incineration of hazardous materials, but also in petrochemical processing and many other industrial processes. At critical temperatures the particulate matter can show a tendency to fuse or become sticky, resulting in the substantial issue of clogged filter candles. This application report presents a simple powder rheology screening method to find the critical temperature of fusibility/stickiness of filter dust, ensuring optimized processing conditions for maximum efficiency.

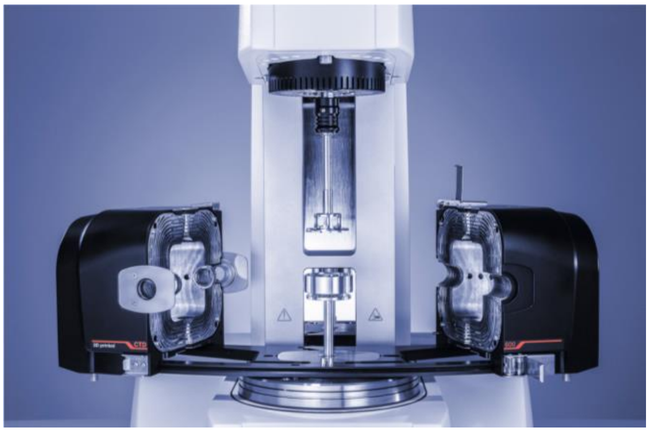

Figure 1: MCR rheometer equipped with a powder shear cell and a CTD convection oven.

1 Abstract

Removing particulate matter with hot gas filtration is a powerful tool to clean process gas flows. This filtration method can clean considerable amounts of matter load up to 1000 °C. A major problem for this filtration method is the clogging of the filter candles at critical temperatures caused by the fusibility/stickiness of the particulate matter, resulting in the need of cleaning and in the worst case demanding a shut-down of the plant. This application report discusses a novel screening and analysis method using a powder shear cell to find the critical temperature where the filter dust starts to be sticky and clog the filters. The powder rheological screening method is verified by wall friction measurements. With this method it becomes possible to easily know until which maximum temperature the filtration system can be run without clogging.

2 Introduction

Since the 1970s hot gas filtration with woven ceramic filter candles is employed to clean hot flue gases from various industrial incineration processes. It is advantageous as it helps to conserve the environment and also protects downstream equipment like catalyst beds, heat exchangers, scrubbers or turbines from erosion, fouling and clogging. The high temperature of the gas stream ensures the maximum yield of thermal efficiency of the overall process, requiring filter elements (e.g. ceramic candle filters) with a high mechanical stability. [1]

The filter systems are run at high temperatures, usually in the range 150 °C to 600 °C, but can even go up to extreme temperatures of 1000 °C. At these high temperatures, particles can stick together and thereby clog the filters. In the worst case this results in a non-operability of the plant making a cleaning of the filters necessary. Modern hot gas filters are cleaned by high pressure pulsed air jets, but if they were clogged by sticky particulates then laborious manual cleaning would often be the only way to get the system ready for operation again. Several elements like alkali metals and alkaline earth metals can lead to a lower melting point and increased stickiness, resulting in clogged filters which cannot be cleaned with compressed air jets.

Analyzing particulate matter of a process gas stream is crucial to predict the performance of a hot gas filter. At the moment the particle assessment is done only by the application of SEM-EDX, STA, PSD-measurements and other classical methods which are usually carried out at room temperature.

In order to ensure maximum utilization and efficiency of the plant, it is necessary to know at which temperatures the powders start to stick together in order to avoid this operating regime. Since this critical temperature is strongly dependent on the composition of the powder, it must be analyzed separately for each system. This “stickiness” is currently being analyzed on the basis of DIN 51730 (Testing of solid fuels - Determination of fusibility of fuel ash), which however, leaves much to be desired in terms of informative value.

Until now no reliable possibility to screen the behavior in dependence of temperature has been reported. Therefore, the goal of this application report is to report a novel qualitative screening method. The screening method is derived from common wall friction tests [2] which are carried out in an annular ring shear cell and allow a comparatively quick analysis at which temperatures the filter dust starts to change its behavior. Common wall friction tests in shear cells can be carried out by rotating a flat disc on the powder bed. The wall material is usually chosen to reflect the powder´s environment (i.e. a stainless-steel disk would be used to analyze the wall friction of a powder in a stainless-steel silo). The data of the screening method is compared with standard wall friction measurements at different temperatures, in order to relate it with a known measurement method.

With the powder rheology screening method explained in this report it becomes possible to measure at what temperatures hot gas filtration powders become sticky/fusible. This knowledge can be used for the design of filtration applications as well as for filter candle manufacturers.

3 Samples and Experimental

3.1 Samples

Three industrial samples were provided from a plant manufacturer. The samples are from plants in which sulfuric acid is regenerated, and consist of varying components.

Sample 1 and 3 showed considerable alkali metal concentrations in a SEM-EDX analysis. In the filtration process these samples led to severe problems in the cleaning step, which hampered the lifetime of the applied filter candles. Sample 2 showed low alkali metal concentrations, which correlated with a good cleaning performance via pulse cleaning with back-flushed compressed air.

3.2 Experimental

An Anton Paar Modular Compact Rheometer (MCR) equipped with a convection temperature device (CTD) and a powder shear cell was used to run the measurements. The CTD enables to control the sample temperature up to 600 °C and can therefore be used to analyze the change in behavior in dependence of temperature.

3.2.1 Screening Method

The novel screening method is carried out with a wall friction geometry (flat disk) of stainless-steel. The sample is loaded into the ring shear cell and placed within the CTD on the MCR. The wall friction geometry then consolidates the powder bed with a defined normal stress and shears at a constant rotational speed of 0.05 rpm, while a temperature ramp from room temperature to 600 °C is carried out. The torque is recorded and used for further analysis, where the noise (i.e. slip-stick) is plotted over temperature.

3.2.2 Wall Friction Measurements

The results of the screening measurements are used to run wall friction measurements at specific constant temperatures.

The powder is consolidated with a flat disc at a defined normal stress and sheared with 0.05 rpm, while the torque is recorded. This measuring step is performed at different normal stresses (usually at 9, 6 and 3 kPa). The torque is converted into shear stress and the resulting value pairs (shear stress / normal stress) are plotted in a graph to analyze the adhesion and wall friction angle.

4 Results and Discussion

This chapter first focuses on presenting the screening method and its validity with one sample, before other samples are analyzed.

4.1 Screening Measurement and Validation of Sample 1

4.1.1 Screening Measurement on Sample 1

The result of the screening (rotation of a disk at a defined normal stress at a constant rotational speed while a temperature ramp is carried out) of sample 1 is displayed in Figure 2. From room temperature to 270 °C the torque signal remains relatively stable. Between 270 °C and 420 °C the torque signal drops slightly, but shows the same general behavior as for lower temperatures. In the range 420 °C to 450 °C the powder starts to show what appears to be an increasing amount of noise coming from slip-stick in the powder. From 450 °C to 530 °C this noise gradually increases with the rising temperature, showing even a strong outbreak (see maximum constant stress 500 °C). The behavior seems to stabilize and reach its maximum effect once 550 °C is reached and remains relatively constant until 600 °C.

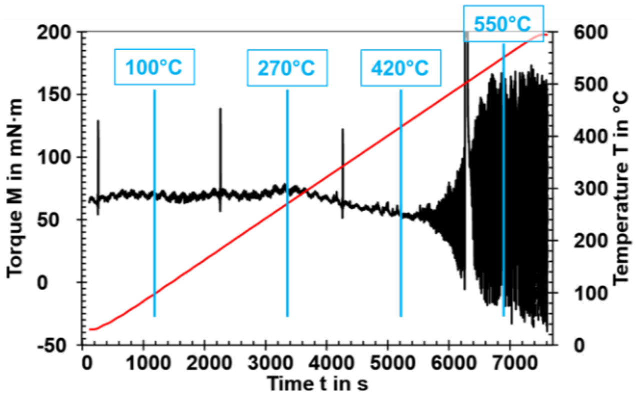

Figure 2: Sample 1 screening (temperature ramp). Black: torque. Red: temperature. Blue lines indicate at which temperatures wall friction measurements were carried out (see 4.1.3).

4.1.2 Analysis of Screening on Sample 1

The torque signal is analyzed in such a manner, that finally a relative shear stress signal can be plotted over the temperature, as shown in Figure 3. The relative signal change is quite low until 450 °C is reached (blue arrow), where the signal starts to rapidly increase. From this critical temperature on a change in behavior is expected – this will be verified with wall friction measurements in the next subchapter.

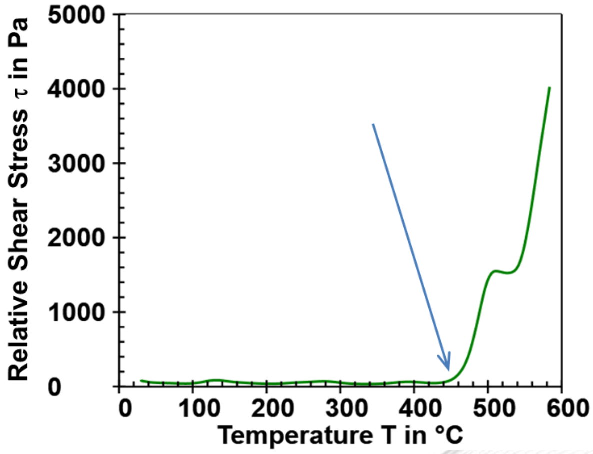

Figure 3: Sample 1 analyzed data of screening - relative shear stress over temperature. Arrow at 550 °C indicates where the sample starts to change its behavior.

4.1.3 Validation with Wall Friction Measurements on Sample 1

Slip-stick and stickiness do not necessarily directly correlate but are often related, depending on the sample. In order to ensure this relation is valid for these samples, wall friction measurements (see Figure 4) are carried out at defined temperatures as indicated by the blue vertical bars in Figure 2. The wall yield locus (gray line, fitted for the three measurement points at 9, 6 and 3 kPa normal stress) remains relatively stable for low temperatures (100 °C, 270 °C, 420 °C), indicating little change in the sample for this range. At 550 °C the wall yield locus shows a very different behavior, as well as a much higher adhesion. The adhesion is defined by the intersection of the wall yield locus with the vertical axis and indicates the tendency to stick towards the wall material. The adhesion is very low for temperatures were no slip-stick was observed (below 420 °C), while for 550 °C a high adhesion can be observed. This indicates a strong increase in stickiness of the particulates in this temperature regime.

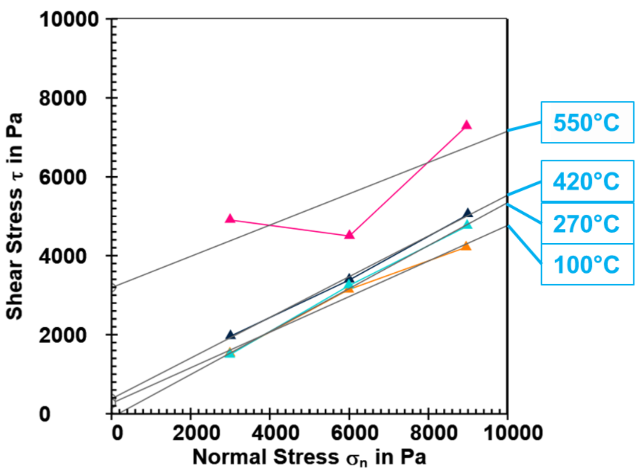

Figure 4: Sample 1 wall friction measurements at 100 °C (orange), 270 °C (turquoise), 420 °C (black) and 550 °C (pink).

4.2 Sample 2

4.2.1 Screening Measurement on Sample 2

The result of the screening of sample 2 is displayed in Figure 5. Here the signal shows some variation in intensity, but does not show any change in behavior.

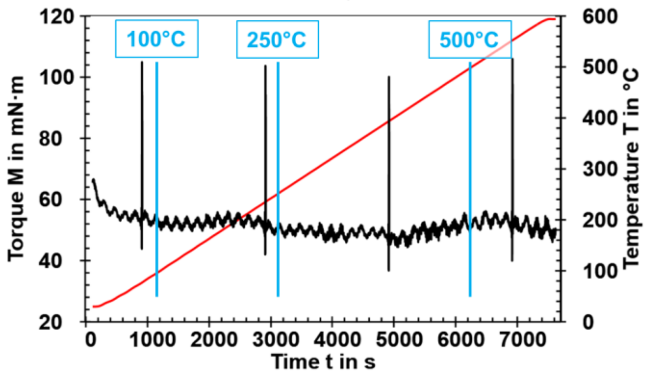

Figure 5: Sample 2 screening (temperature ramp). Black: torque. Red: temperature. Blue lines indicate at which temperatures wall friction measurements were carried out.

4.2.2 Analysis of Screening on Sample 2

The analysis of the torque (or shear stress) signal of the screening of sample 2 (from Figure 5) is displayed in Figure 6 (with the same scale as sample 1 in Figure 3). As already observed above, the sample does not show any significant change in the range from room temperature to 600 °C.

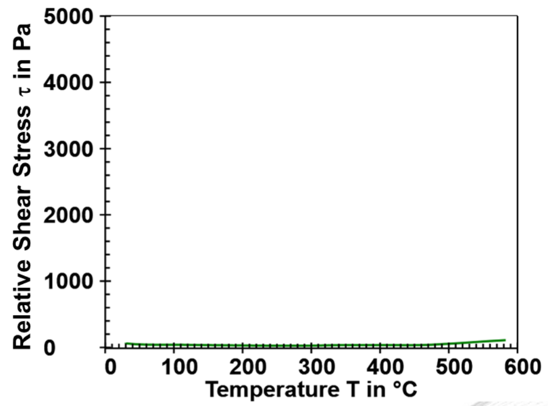

Figure 6: Sample 2 analyzed data of screening - relative shear stress over temperature.

4.2.3 Validation with Wall Friction Measurements on Sample 2

The wall friction measurements at the selected temperatures (100/250/500 °C) are shown in Figure 7. Unlike sample 1, for this sample the wall friction angle, as well as the adhesion do not show any significant change with temperature, confirming the validity of the screening method.

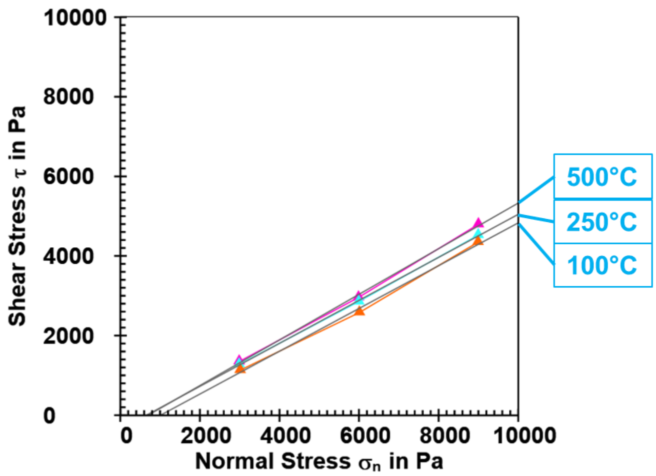

Figure 7: Sample 2 wall friction measurements at 100 °C (orange), 250 °C (turquoise) and 500°C (pink).

4.3 Sample 3

4.3.1 Screening Measurement on Sample 3

The screening measurement of sample 3 is displayed in Figure 8. This sample shows remarkable behavior with some peaks and increasing/decreasing wall friction (e.g. at 120 °C, 380 °C, 450 °C). The change in slip-stick of this sample seems to take place for temperatures higher than approximately 520 °C.

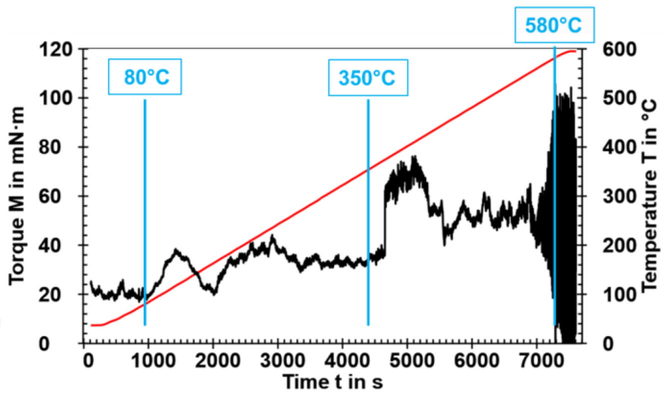

Figure 8: Sample 3 screening (temperature ramp). Black: torque. Red: temperature. Blue lines indicate at which select temperatures wall friction measurements were carried out.

4.3.2 Analysis of Screening on Sample 3

The analysis of the screening data (Figure 9) shows a less regular behavior, already at lower temperatures. But the significant change in the behavior seems to start to take place for temperatures higher than the critical temperature of 520 °C.

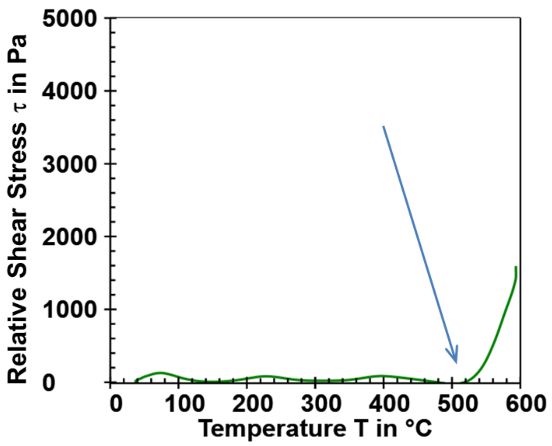

Figure 9: Sample 3 analyzed data of screening - relative shear stress over temperature. Arrow at 520 °C indicates where the sample starts to change its behavior.

4.3.3 Validation with Wall Friction Measurements on Sample 3

Wall friction measurements were carried out at 80 °C, 350 °C and 580 °C (Figure 10). As expected from the screening and its analysis, at low temperatures (80 °C and 350 °C) no significant difference can be observed, above the critical temperature (580 °C) a strong increase in adhesion as well as wall friction angle becomes visible, confirming the screening results.

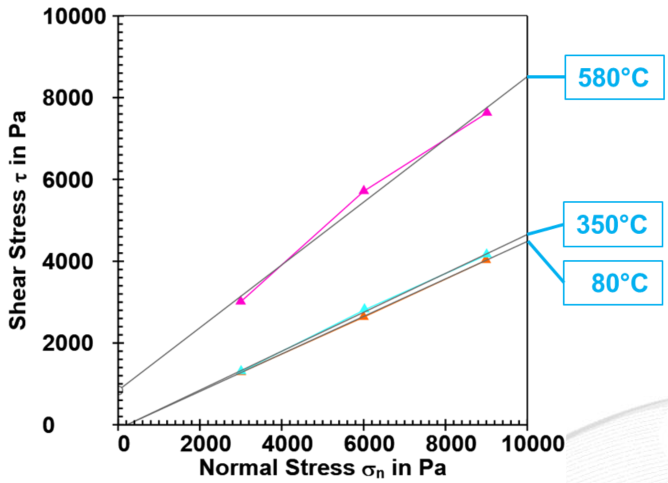

Figure 10: Sample 3 wall friction measurements at 80 °C (orange), 350 °C (turquoise) and 580 °C (pink).

5 Conclusion

A screening and analysis method for the determination of dust filter stickiness in dependence of temperature was demonstrated and its validity was proven with wall friction measurements at selected temperatures.

The three compared samples showed strongly diverging behavior. While one sample showed no change in its powder behavior from room temperature to 600 °C, the other two samples exhibited a strongly diverging behavior. With the screening method and its analysis, it was possible to determine that the two other samples exhibit a stickiness for temperatures above 520 °C and 550 °C. These results were also validated with wall friction measurements.

This enables to reliably predict the critical temperature at which the filter dust becomes sticky and would start to clog the filter candles. As a consequence, it is now possible to know at which maximum temperature the plant can be run for a maximized efficiency without clogging of the filter candles.

The convection oven which was chosen for these measurements can be used to cover the temperature range starting from -160 °C up to 600 °C. In order to analyze filter dust behavior at even higher temperatures another convection oven could be used with a maximum temperature of 1000 °C.

6 References

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2. **Shamlou, P.A.**, Handling of Bulk Solids, 1990, 0-407-01180-3

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