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Powder Rheology Using A Novel Friction Tool Measuring System

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Rheology is defined as the science of flow and deformation of matter. Rheological instrumentation and measurements have become essential tools in the analytical laboratories of companies for characterizing ingredients and final products, as well as for predicting product performance and consumer acceptance. Rheological measurements can assist scientists in both formulating the optimal products and improving processing efficiency.

Yield stress is defined as the minimum shear stress required to initiate flow. Yield stress can also be defined as the stress below which the material will not exhibit fluid-like behavior. This means that subjecting a material to stress less than the yield stress will lead to a nonpermanent deformation or a slow creeping motion over the time scale of the experiment. The yield stress value depends on the time scale of the experiment, and almost all substances will eventually flow if the time scale for imposing a shear stress is long enough. However, in general, the higher the static yield value, the more readily a material will maintain its particles intact. Thus, the magnitude of the static yield value can be used as one of the criteria for studying the interfacial wall friction within the particles in a powder material. This interparticulate friction defines the flow properties of powders. Studying the flow behavior of powders is critical for smooth processing, handling, and transportation. Powder rheology finds varying applications, especially in the pharmaceutical and food industries; thus it is essential to understand and characterize powders. In this article, powders are characterized by studying the flow properties using yield stress measurements.

The authors utilized the newly designed Friction Tool measuring system (**ATS RheoSystems/REOLOGICA Instruments**, Bordentown, NJ) (*Figure 1a* and *b*) to study the behavior of four disparate calcium carbonate powder samples. The powder samples were categorized as A—untreated and B—untreated, and A—treated and B—treated. Samples categorized as "treated" were coated with a modifier. The A samples had a lower average particle size compared to the B samples. The experiments were conducted on the fully automated NOVA Rheometer (**ATS RheoSystems/REOLOGICA Instruments**) (*Figure 2*) with a 25-mm Friction Tool and cup geometry. The NOVA Rheometer is equipped with the patented Differential Pressure Normal Force (DPNF) sensor providing high normal force accuracy and sensitivity. Measurements were done to study the flowability and frictional properties of the powder samples. A Friction Tool test method was developed, which characterizes the powder by utilizing a yield stress ramp measurement. This method is used for measuring the interfacial wall friction during powder flow. Therefore, a typical method of characterizing the powder flow is obtained in shear rate condition evidenced by a yield like flow behavior.

Loading the samples with the same exact loading history is very important in order to obtain reproducible results. The packing state of the powder has a profound influence on its flow behavior. Here the samples were tested in a relatively consolidated state. A known volume of approximately 11.5 mL was used for all samples due to different sample density or packing values. This volume was used to get a height of approximately 2 cm in the cup. Zeroing gap was done 1.5 cm above the bottom of the cup. After filling the cup with the sample, the cup was tapped 20 times for each sample before starting the test. A 5.0 N loading force was used for all samples to maintain the same loading history. An equilibrium time of 300 sec allowed the normal force to equilibrate. A stress ramp increased logarithmically from 500

a b

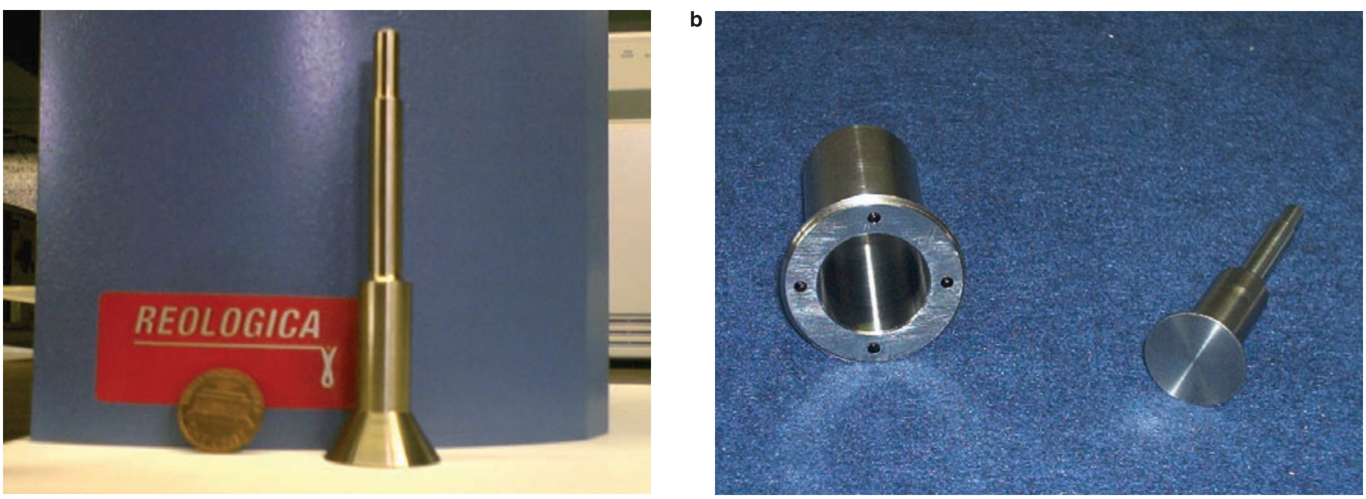

Figure 1 *Friction Tool (25-mm) (a) and corresponding lower cup (b).*

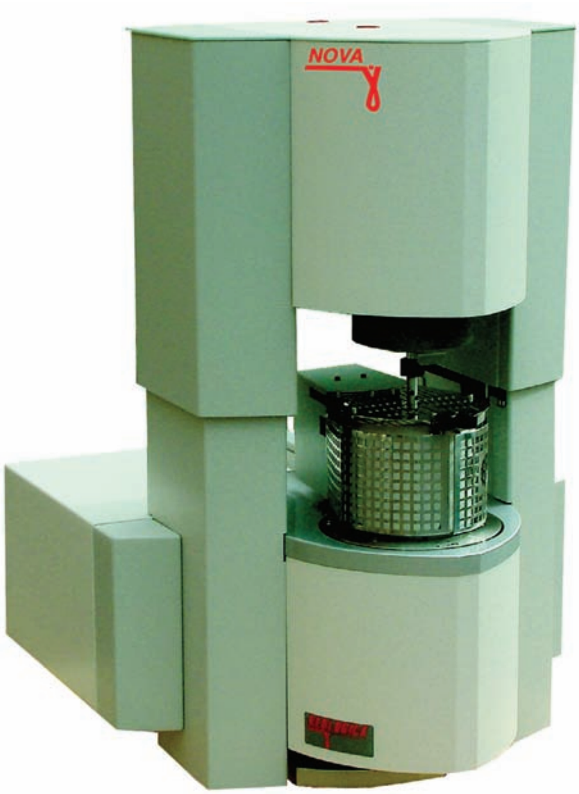

Figure 2 *NOVA Rheometer.*

to 10,000 Pa in 500 sec was used for all measurements. This test essentially subjects the sample to a series of shearing stresses and monitors the deformation. The data are displayed as viscosity versus shear stress. The viscosity value starts out very high, since the powder behaves like a solid. As deformation commences, the value drops at an increasing rate. The yield stress is determined as the stress at which the highest rate of change occurs.

Figure 3 shows viscosity (Pa s) vs stress (Pa) for sample A from the yield stress experiment. Also shown in Figure 3 is the normal force (N), which was maintained constant throughout the test indicating constant compaction. The data are shown in duplicate, which demonstrates good reproducibility. Using the built-in software tools, the yield stress was obtained for all the experiments and has been tabulated in *Table 1*. The table shows the average yield stress values for various samples from duplicate tests.

Untreated samples (A and B) had lower yield stress values compared to the corresponding treated samples, indicating better flowability. Samples A (treated and untreated) had lower yield stress values than the corresponding B samples, indicating better flowability. These trends are consistent with the known particle size differences and observed powder flowability in the actual

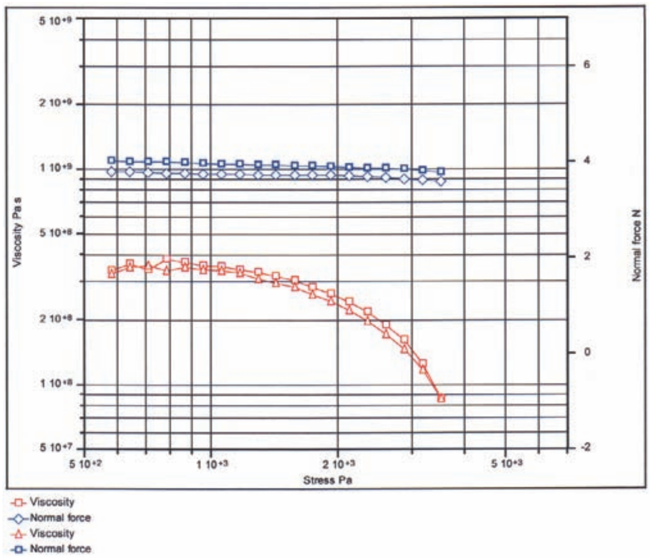

Figure 3 *Viscosity (Pa s) and normal force (N) of sample A showing reproducibility.*

process for samples A and B. Thus, simple rheological yield stress measurements can be used to predict powder flowability, and also provide an indication of powder's friction coefficient.

The above measurements were done where stress was ramped logarithmically. It is also possible to perform measurements where the stress is ramped linearly, which is a common practice. Table 1 indicates that when ramped linearly, the yield stress for A—treated sample is about 48% higher than the A—untreated sample, whereas for the logarithmic stress ramp, the yield value for A—treated is 23% higher than the A—untreated sample. In addition, the yield stress obtained via the linear ramp is significantly higher than that obtained via the logarithmic ramp based on the incremental steps being more uniform. As a result, the differences between the samples are magnified in the linear stress ramp test. For powder samples, it appears that the yield stress test under linear stress ramp differentiates between the sample better than the logarithmic stress ramp.

This article presents yield stress measurements on powders using the NOVA Rheometer. The rheological characterization of powders provides important information for engineers and scientists to improve and optimize their products and manufacturing processes. The Friction Tool successfully characterizes powder samples based on the difference in particle size and treatment. Significant differences were observed within the powders in terms of flowability. Powder with lower particle size had lower yield stress and hence better flowability, whereas treated powders had higher yield stress than the untreated powders.

Table 1 Yield stress values for treated and untreated samples

Sample	Average yield stress log ramp rate (Pa)	Average yield stress linear ramp rate (Pa)
A—untreated	3050	3800
B—untreated	3095	—
A—treated	3740	5640
B—treated	4599	—

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