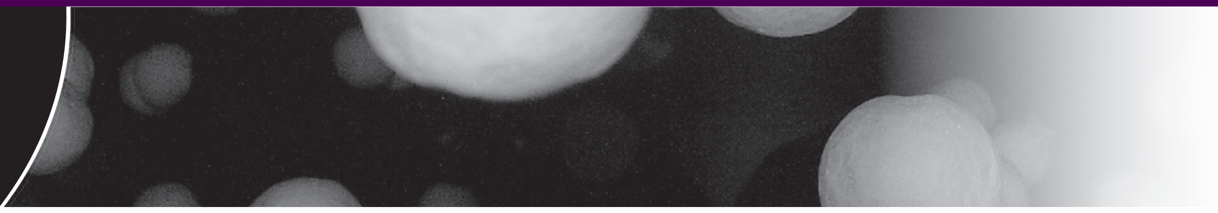

You Know the Particle Size…But Will the Powder Flow?

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Particle Size Analysers are well established instruments used without question in R&D laboratories to evaluate new formulations. They measure the particle size distribution for a powder sample that gives important information about the nature of the mixture. Experienced plant managers may be able to infer whether there will be potential flow problems, based on particle size information alone, but the truth is that this data does not really address flowability. Why? It is simply because particle size does not assess the frictional forces between particles that are fundamental to predicting flow behaviour in gravity discharge from a bin or flow down a chute.

Figure 1. Example of Flow Cup Used to Predict Powder Flowability

Instruments often found in test labs along with Particle Size Analysers include Flow Cups, Angle of Repose Testers and Tap Testers. This combination of instruments may be used to make predictions about flowability of the powder, both for new formulations in R&D and for batch qualification by QC. The first device would seemingly be most relevant based on name alone. *Figure 1* shows a popular example for this type of test too. Flow Cups come with discs that have holes of differing diameter in the centre. After a specific disc is placed in the bottom of the cup, then filled with powder, the test provides a simple go/no-go indication for whether the powder might flow through an opening of that size. The test is performed on a small volume of powder, usually less than 500cc, and takes no more than a minute or two. While useful for free flowing materials, like salt and granulated sugar, cohesive powders do not perform well in this type of test and leave the processor uncertain as to how problematical the flow behaviour will be.

Angle of repose measurement is another standard test performed on powders along with particle size analysis. As the name implies, it measures the angle relative to horizontal for a small pile of powder. Although this data has little to do with flow behaviour, manufacturers will attempt to correlate the results with predicting processibility in their plant, oftentimes with wrong predictions. The one useful application for angle of repose data is the floor space requirement for stockpiled powder in an open area. Concrete manufacturers, for example, may use this type of measurement for their sand and gravel materials.

Tap Tests are popular with the pharmaceutical industry. A full column of powder with defined volume is tapped 100 or more times to determine compressibility. The reduced volume in the column is measured and two common calculations are made to determine the Carr Index and Hausner Ratio. *Figure 2* shows the equations. These values are then correlated empirically with potential flow behaviour. The more compressible the powder, the more likely it will exhibit flow problems during processing.

- Hausner Ratio

$$HR = \left[\frac{\rho_{Poured}}{\rho_{Tapped}}\right]$$

- Carr Index

$$Carr\% = \left[\frac{\rho_{Tapped} - \rho_{Poured}}{\rho_{Tapped}}\right] \cdot 100$$

Figure 2. Equations for Carr Index and Hausner Ratio

Annular shear cells have become the scientific instrument of choice for measuring flowability. A defined sample volume of powder is placed in a trough *(see Figure 3)*, weighed, and placed on the instrument turntable. The powder is compressed to a predefined consolidating stress by the vaned lid *(see Figure 3)* that descends on the sample. Powder particles at the intersection between lid and trough are sheared against one another when the trough rotates through a small angle. The lid is connected to a torsion spring that measures the amount of torque that must be overcome before the lid stops moving along with the trough. Powder failure strength is equated with this measured torque value.

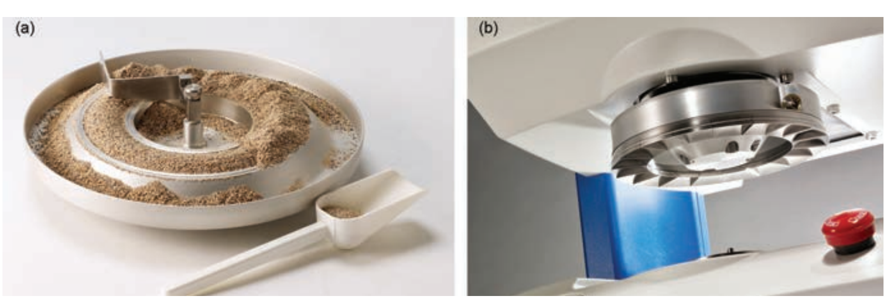

Figure 3a. Annular Trough Containing Powder Sample 3b: Vaned Lid

This measurement procedure is repeated at increasing levels of consolidating stress on the powder sample. Increasing height of powder contained in a bin correlates with these higher levels of consolidating stress. Data from the test is plotted on a graph *(see Figure 5)* that has the consolidating stress on the x-axis and the powder failure strength on the y-axis. 'Flow Function' is the name of the curve that describes the flow behaviour of the powder.

Figure 4. Brookfield Powder Flow Tester – Example of Annular Shear Cell Instrument

Industry has agreed to classify powders into different regions of 'flowability'. *Figure 5* shows that they range from 'free-flowing' to 'non-flowing'. Those that cause the most problems with flow behaviour tend to be 'cohesive' and 'very cohesive'. Note that a powder can be 'cohesive' or even 'easy flowing' at high consolidating stress, but transition to "very cohesive" at low consolidating stress. This suggests that powders may change in flow behaviour during processing depending on the fill level of the powder in a feed bin.

Another important flow characteristic that the shear cell can measure is the phenomenon known as 'time consolidation'. When a powder is left in a bin for any period of time, settling will gradually take place due to gravity. As the air trapped between powder particles is squeezed out, particles move closer together and the fill level in the bin will drop. Similar to the process of making a snowball, the powder may consolidate and pack together tightly, preventing further flow out the bottom of the container. When the shear cell applies a consolidating stress, the failure strength can be measured right away and then measured again at a later time interval to determine whether the failure strength has increased. R&D may specify different time intervals to mimic specific situations that occur on the plant floor, such as temporary stoppage for a few minutes or an equipment breakdown that lasts several hours.

Although shear cells have been available for decades, they have become highly popular in the last few years due to several recent improvements. Included are lower cost to purchase, ease and speed of test set up, automatic execution of test without operator involvement, and straightforward interpretation of data. There is little doubt that shear cells provide useful test information that makes a plant manager's life less problematic when dealing with flow issues. Recent reductions in test cycle time that allow a Flow Function to run within 15 minutes give strong argument to consider use of shear cells in QC as well as R&D. The investment is likely to pay off within the first few months if it can eliminate costly stoppages in processing related to powder jams.

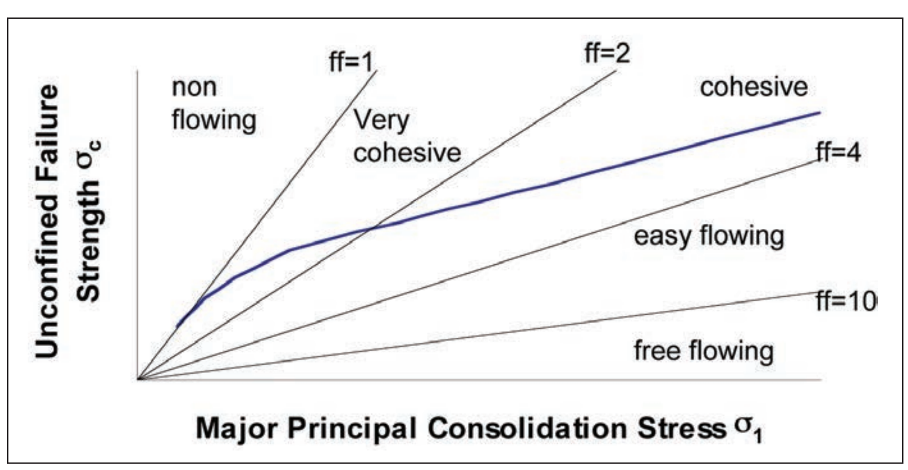

Figure 5. Flow Function Graph for a Powder Sample