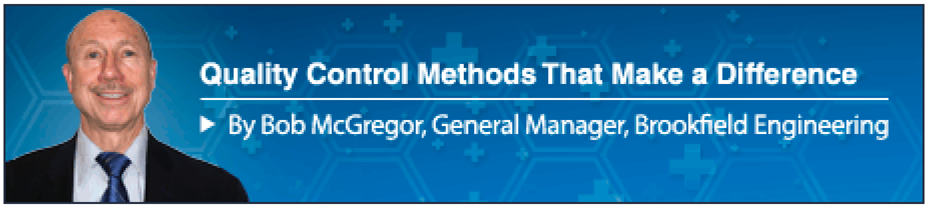


# QC METHODS THAT MAKE A DIFFERENCE:
## The Challenge Of Measuring Powder Flowability

*By Bob McGregor, Brookfield Engineering*

What does it take to put effective qualification tests into QC for powder processors? Pharmaceutical products that satisfy not only manufacturability criteria, but also provide a high level of consumer satisfaction, undergo a battery of physical property tests before being introduced into production. Using affordable instrumentation to perform these QC tests is an essential part of today's successful business.

Corporations willingly invest in devices used to test physical properties which measure flowability of powders. Proper flow behavior is essential to keep the production line running smoothly without interruption. Powders comprised of multiple components require a throughput process which preserves this "mixed" condition. The possibility of desegregation of component particles is a potential pitfall that accompanies "core flow" behavior, a "first in – last out" flow pattern that is more the norm than the exception. (See figure 1)

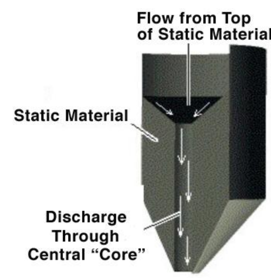


Figure 1

When unsure about effective QC test methodology for evaluating powder flow behavior, manufacturers contact potential instrument suppliers, review product literature, and request demonstrations. Can businesses, both large and small, command similar attention from vendors? And how expensive is the outlay to bring these test methods in house? In most companies, R&D has the responsibility for defining the QC test method. The bottom line is to know how different methods work, why they add value, and to find the best purchase price once the decision is made to go forward. Equally important is to make sure that QC has a clear understanding of how the method works, the relevance of the data, and when to question the results.

Pharma industry formulators come up with new powder mixes on a weekly basis for a variety of reasons. Some relate to adding to or modifying the mix of active ingredients, some pertain to new excipients that will enhance processability, and others arise because new suppliers are providing equivalent raw materials, perhaps at a reduced cost. The challenge in each case is to make sure that the new formulation flows acceptably when scaled up.

The Challenge Of Measuring Powder Flowability

Flowability in gravity discharge through a hopper is one area that requires attention, because the popular test methods, while simple to do, are unfortunately inaccurate. Tests using the Flodex Cup or Angle of Repose measurement do not focus on the pertinent parameter that affects flow behavior of powders. (See figure 2) Consolidation of powder in the bin due to its own self-weight is the key variable of interest when evaluating whether the powder will flow. Shear-cell methodology is the proper technique for this challenge because it truly simulates the physical conditions in the bin as powder particles move relative to each other. The components of the typical shear cell appear in figure 3.

Figure 2

Figure 3

The Flodex Cup is the prevalent tool in the QC Lab for both historical reasons and low cost to purchase. Discs with holes of varying diameter are individually placed in the bottom of the cup and the powder sample is poured in. If the powder flows out of the cup, the technician will report the hole size that allowed the powder to discharge. Based on earlier trial and error, there will most likely be a correlation with the dimensions for the opening in a hopper that equates with the size of the hole in the disc. The missing detail in this test procedure is that the powder in the cup was in a loosely consolidated condition, which is not the same as the powder sitting in a bin awaiting discharge.

Shear-cell methodology applies a compressive load to the powder sample which simulates the effect of the powder weight on itself in the bin. (See figure 4) Increasing the compressive load on the powder sample simulates the increasing height of the powder fill level in the bin. At each compressive load, the shear cell causes the powder particles to slide against each other by rotating the powder in the trough relative to powder in the lid. This technique measures the sliding friction between the particles, which in fact, is the best indication of "flowability".

Figure 4

The established technique for conducting a shear- cell test produces a data set called the "flow function". (See figure 5) The x-axis shows the amount of consolidating stress or pressure applied to the sample by the instrument. The effect of the pressure, which simulates the self-weight of the powder contained in a bin, is to squeeze the air out of the sample, thereby moving the powder particles closer to each other.
The y-axis shows the amount of strength in the powder, which basically indicates its resistance to moving under the influence of gravity.

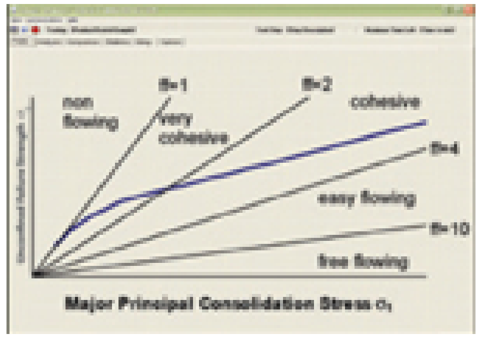


Figure 5

Industry has agreed on different regimes for powder flow behavior, ranging from "free flowing" to "nonflowing". Review the flow function curve shown in figure 5 and note that the flow behavior changes as the consolidating stress experienced by the powder increases. At low consolidation stress, the powder is "highly cohesive", whereas at higher consolidation stress, it becomes "cohesive" which means that there is less resistance to flow. Remember that the consolidating stress correlates with the fill level of the powder in the bin.

The "Flow Index" of the powder is the slope of the line from the origin to the data point associated with the highest consolidating stress. This numerical value is an approximate measure for flowability.

One useful calculation, which derives from the Flow Function, is the "arching dimension". This numerical value is the capacity of the powder to build a stable structure or arch in the bottom of the bin, thereby creating an obstruction which prevents the powder from discharging. Arching pertains specifically to powders that discharge in "mass flow" behavior, which is a "first in – first out" flow pattern. (See figure 6) Although most powder processing operations will never achieve mass flow conditions, this number for arching dimension is still relevant in terms of giving a second value which characterizes the powder.

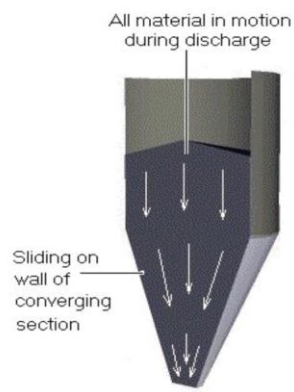


Figure 6

Another useful calculation is the "rathole diameter". This value characterizes the annulus of powder that can remain in the bin after "core-flow" discharge. The strength of the consolidated powder contained in this annular ring is greater than the discharging force due to gravity, which means that the powder will not move. The powder that had been in the core discharges while the powder outside the "rathole diameter" stays. Powder processors are all too familiar with the hopper bashing that takes place in an attempt to dislodge the powder that gets hung up in ratholes.

Science supports the shear cell method which has been practiced for almost 50 years. ASTM D6128 characterizes the original test procedure which derived from analytical work in the minerals industry during the 1960s. Advances in equipment design and data processing have progressed to the point where shear cells are now much more affordable, easy to operate, and automatically generate the information described above. The remaining challenge is to spend sufficient time characterizing individual powders and interpreting the data. The results will lead to decisions on optimal use of flow additives and potential equipment design changes.

Perhaps it's time to investigate the shear cell and find out how the test data can be used in your process.

**About The Author**

Robert G. McGregor is the General Manager and Global Marketing for Brookfield's High-End Lab Instrument Sales. He graduated from MIT in Cambridge, Massachusetts and holds M.S. and B.S. degrees in mechanical engineering.