Nutraceutical Powders Need Quality Control

Catching Up With The Competition

Nutraceutical powders are like pharmaceutical powders in many aspects. There is an active ingredient (or ingredients) with a host of excipients. This creates a complex mixture of materials which can cause flow problems during a production run. Costly downtime occurs due to flow stoppages and lost time related to the effort involved in identifying the cause and solving the problem.

To ensure production uptime and reduce downtime, a quality control check needs to be made on the material during the production run. This QC test needs to focus on flow behavior of the powder. It also needs to be accurate and timely. The data from the QC check should be compared to flow data from an initial characterization of the material. Pass/Fail criteria are based on how close the flow performance is to the original material. These are the steps that must be taken to keep production up and running.

This article examines the flow behavior of nutraceutical powders, the proper test method for defining their flow parameters, and the implementation of fast and accurate QC checks that ensure a smooth production run.

Nutraceutical Powders

Nutraceutical powders are used to produce the types of consumer products that one would see in a typical health food store: protein powders, shakes, and various types of pressed vitamin tablets. While these products may look simplistic to the end user, they are in fact a complex mixture of active ingredients and excipients. There may be 10, 20 or more different products in a powder blend consisting of the active ingredient, flavorings, glidants and dissolvents. Often the products are milk based. Products like these, which are already cohesive by nature, are susceptible to flow problems due to temperature and humidity changes. Thus, a humid day may significantly affect the flow properties of a powder used to produce a chocolate protein shake. Common flow stoppages for these types of materials occur due to cohesive arching in the discharge hopper and rat-holing in the bin.

In many blends of powders, the bulk densities of the constituent materials may be vastly different. Some powders may be free flowing while others are more cohesive. These variations in bulk densities can also cause flow problems to occur. To test how this mix of bulk densities behaves in the final blend requires not only a density measurement, but also a corresponding flow behavior test.

Conventional methods used to address flow issues include the following: the addition of flow aid, installation of automatic rapping or vibratory systems on plant bins and hoppers, maybe even re-blending of the product for improved flow behavior. But what if these flow issues could be accurately identified beforehand so that they were eliminated before they got to the production floor? Cost for implementing a comprehensive QC test is always a factor. What's needed is a low cost, accurate test method which can characterize these individual component powders initially for flow and density, analyze the blended powder for the same properties, then run subsequent QC checks to ensure that each batch comes close enough in flow properties to the approved formulation.

For testing, analyzing and running QC checks, shear cell technology is the all in one solution.

Figure 1: Shear Cell Instrument: Brookfield Powder Flow Tester

Shear Cell Technology to the Rescue

Shear cell technology is a tried and true scientific method as per ASTM D6128. By shearing a powder at precise consolidation stresses, flow data such as Flow Function, Normalized Flow Function, Arching Dimension, Bulk Density and Rat-Hole Diameter can be gathered and analyzed to determine the flow properties of a material. The Flow Function is the established test in the powder processing industry for characterizing and evaluating bulk solids.

Figure 1 shows an instrument that uses shear cell technology to define a material's flow properties. Once characterized using the Flow Function test, the material can be qualified in the future for acceptance using one or more of the established flow parameters. QC checks on the material can be performed with a quick (10 to 16 minute) test before and during the production cycle. This QC data is compared to the initial characterization test for compliance with set pass/fail criteria. Instrument software accomplishes all of these test requirements, supports quick QA/QC checks and allows for easy analysis and comparison of the flow data.

Flow Function Test and Normalized Flow Function Test

A standard test with a shear cell defines a material's flow properties for common parameters such as Flow Function (a gage of the flowability of the powder), Arching Dimension, Rat-hole Diameter and Bulk Density. One or more of these tests can be used to characterize a product and establish pass/fail criteria.

Consider that the standard flow function represents the strength of the material (its resistance to flow) vs. consolidating stress. The bulk density or self-weight of the material provides the force to overcome the internal strength of the powder to resist flowing. The material is defined into regions of flow from Free Flowing to Non-Flowing as shown in Figure 2.

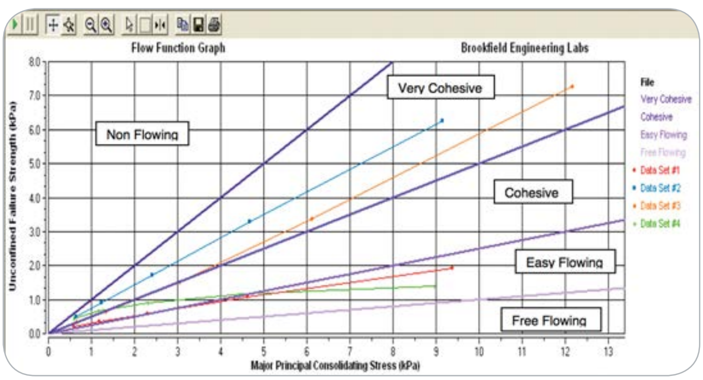

Figure 2: Standard Flow Function with Regions of Flow Behavior

One characteristic of a nutraceutical powder that is frequently overlooked is the differing bulk densities of the powders used to form the blend. Some materials in the blend may be free flowing while others are more cohesive; this may be reflected in their bulk densities. To obtain a valid comparison of a material with these different bulk densities, they need to be normalized to get a better representation of the material.

To accomplish this analysis, the instrument software provides a Normalized Flow Function calculation. This test compares the flowability of materials with differing bulk densities and normalizes them. The normalized Flow Function plot shows Potential Arching Dimension vs. Major Principal Consolidation Stress.

Figure 3 is a Flow Function that has been normalized for bulk density; this gives a valid comparison of different mixtures that may constitute the same end product. The normalized flow function is calculated by dividing the unconfined failure strength by the bulk density and gravity to give units of length (i.e. the arching potential for the powder in the hopper).

In the normalized flow function a free flowing material will have a flow function that is close to horizontal and lies approximately on the consolidation stress axis (i.e. an outlet dimension of 0 at all consolidation stresses). A non-flowing material will have a steeply sloped line. Other blends of materials will fall in-between these two regions based on their cohesiveness. Materials can be rated by the magnitude of the outlet dimension and also by the rate at which the outlet dimension increases with increasing consolidation stress.

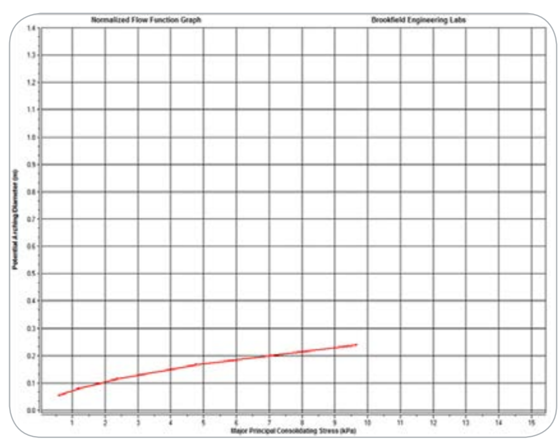

Figure 3: Graph of Normalized Flow Function Shows Arching Dimension vs. Consolidation Stress

QA/QC Data Checks

Once the material is characterized, it is now necessary to implement periodic QC checks on a production batch of material. Once the material is blended, an initial QC check is performed and the production run is begun.

To run QC checks in a timely manner, the instrument software supports quick flow function tests of either 2 points (10 minutes) or 5 points (16 minutes) of data. Thus, QC checks can be run frequently, both before and during the production run, to ensure that the blend hasn't changed. Similarly, the QC check will show whether environmental effects, such as changes in temperature and humidity, are going to cause flow problems.

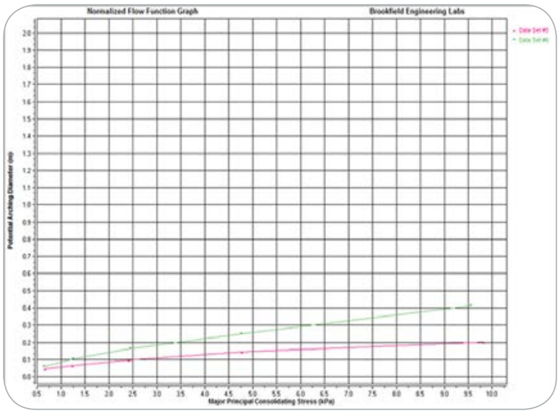

Figure 4: Comparison of Two Normalized Flow Functions

Figure 4 above shows a Normalized Flow Function. Product X Control has been characterized as a material that will flow well at a Major Consolidation Stress of 4.7 kPa with a Potential Arching Dimension of 145 mm. A concurrent QC check on the material as it is being run shows that the Potential Arching Dimension has increased at to 251 mm. This is outside of the acceptable pass/fail criteria, and a flow aid is added. The material is re-blended and then checked again with a subsequent QC test to ensure it is back to specification.

Other flow parameters can also be checked to further qualify the blend, such as rat-hole diameter. This will give additional information on the potential cohesiveness of the production blend.

Conclusion

Shear cell technology is a fast, easy, comprehensive approach for defining materials, analyzing their flow properties and running QC checks to ensure the product meets specifications. Through the use of shear cell technology, a characterization of the material is created for control purposes. By running timely QC checks during production of these nutraceutical materials, potential production showstoppers can be identified, and possibly corrected or avoided altogether. Quality Assurance may chart the test data for each batch and identify the root cause for situations that lead to flow stoppages, such as environmental changes or new suppliers of raw materials. Thus, expensive downtime and lost product can be minimized and production volume maximized.

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