Powder Pointers

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Brought to you by: **Material Flow Solutions, Inc.**

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In This Issue

Particle Breakage: A Study in Prediction

Creating the particle in Just the Right Size – an Introduction. The breakage of particles in handling systems is critical in the chemical, energy, and pharmaceutical industries. The desire is to create particles with a prescribed behavior: for example, a catalyst with a significant surface area and prescribed particle porosity, yet sufficiently robust to maintain integrity in unit operations such as fluid bed reactors. Creating such a catalyst is often a compromise between the porosity and the particle size degradation. There is a movement in industry to use nano-particles to create certain chemical behaviors that add value to everyday products. However, the addition of these small particles into the matrix of other materials can lead to particle design issues where the optimal particle is very porous and, therefore, not sufficiently robust to be handled by consumers. There is also a movement to create low weight, high strength materials through novel particle production processes involving sintering product mixtures. In this case, the green materials are easily broken and must be treated carefully prior to the sintering process to avoid breakage. The final product is robust, but the intermediate process steps require handling products that are friable. In all cases, particle size degradation is a key flow property that controls the product or process design.

To design the optimal particle or process, we must first determine the expected breakage behavior in a given process or unit operation. It is critical to understand what causes particle breakage.

- A particle may be sensitive to breakage by fracture (i.e. a tendency to break in half during an impact or stress-strain event).
- A particle may be sensitive to breakage by abrasion (i.e. impact or shear events tend to break off corners).
- Some particles are sensitive to breakage through fatigue (i.e. repeated stress-strain or impact events eventually cause the particle to fracture).
- Some particles are only sensitive to cutting or tearing in order to create finer particles. Pinch points and close mechanical tolerance points can result in particle size degradation of these materials.

In addition, the structure of the particle may determine how the particle will break. Often a particle is comprised of smaller particles combined to form an agglomerate or extruded structure. In these cases the breakage behavior of both the primary particles, as well as the agglomerated or extruded structure, becomes crucial. It is important to match the breakage characteristics to the type of action in unit operations behavior. For

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Recapture Lost Revenue: Solve Segregation Issues

A single instrument measures:

- *Segregation by Particle Size*
- *Segregation by Sifting*
- *Segregation by Fluidization*
- *Segregation by Angle of Repose*
- *Segregation by Air Entrainment*
- *Segregation by Chemical Composition*

The SPECTester

- FAST – 10 to 30 minutes to run an analysis.
- Measures a mixture of up to 6 unique components
- Identifies primary segregation mechanism out of 4 specific mechanisms
- Identifies segregation by particle size, sifting, fluidization, angle of repose, chemical component and air entrainment
- Provides data about component concentration, particle size differences, product uniformity
- Identifies process design parameters and quality control issues
- Results scalable to process conditions – mimics actual process conditions
- 50 segregation points measured within a sample
- Fully automated, reports how much as well as why the material mixture is segregating
- Touch-screen/pad control
- Provides uniformity index for sample, and segregation variance data
- Data can be exported in Excel format
- Certified CE Compliant

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example, a pneumatic conveying system is predominantly driven by impact events. Thus, particles sensitive to breakage during impact cause significant fines generations in pneumatic systems. A rotary valve maintains close tolerance between the rotors and the valve housing during operation. Particle breakage in this unit operation will predominantly depend on stress-strain events. This suggests that measuring particle breakage due to impacts, stress-strain, and cutting events will provide data to help design processes that resist particle breakage. In this article we describe some of the method used to determine these key breakage behaviors. They provide an attrition fingerprint of a particular material and aid engineers to design proper systems to mitigate the breakage.

Impact Degradation. Typically a range of velocities exist in typical operations. Hence, when testing for impact degradation, it is important to measure breakage at various impact velocities. To measure for impact degradation, material is placed in a cylindrical container and oscillated at high speed as shown in figure 1. The cylinder moves relative to the contained material, eventually causing the material to impacts on the opposite end of the container. Each cycle of oscillation creates two impacts as material is thrown against each end of the cylinder. During these impacts the particles interact with the cylinder wall and other particles within the cylinder to break due to repeated impacts. The frequency of the oscillation governs the impact velocity and the length of time dictates the number of repeated impacts. This mode of degradation has direct application to unit operations where impacts are prevalent. For example, in a pneumatic conveying system the particle bounces off pipe walls as air currents carry the particles. In a straight section of pipe these impacts are generally glancing blows and the actual change in impact velocity is small. However, as particles approach an elbow, the trajectory causes more than just a glancing blow and the change in impact velocity is much greater. If we can estimate the number of impacts in a typical conveying system as well as the velocity of these impacts, we can determine the particle breakage due to transport through the system. This system is a series of repeated impacts much like the degradation test. Thus, data from this type of test can be used to estimate breakage in pneumatic feed systems, and free fall into process equipment and high velocity blenders where impact is a large fraction of the motion. However, it would be useless to use this data to describe the breakage that may be present in flowing equipment subject to high stress levels or excessive strains. Another type of degradation test is required.

Impact Box for Measuring Degradation Potential

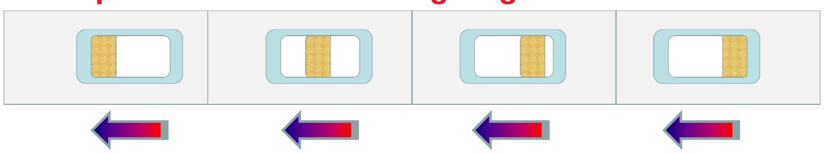

Figure 1. Schematic of impact degradation box showing ½of impact cycle. As the box move left, the material moves right – relative to the box – and eventually impacts on the opposite side of the box.

Stress-Strain Degradation. In this case, the predominant breaking action is due to fracture or fatigue caused by large stress or strain conditions. Therefore, we measure the degradation after exposing the material to pure strain or shear at a known stress condition. The material is placed in a rectangular test cell and allowed to deform as shown in Figure 2.

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Powder Pointers Preview

Coming Next Quarter – Segregation management

Segregation is a primary cause of poor quality product in the chemical and pharmaceutical industries. Powders and granulates segregate for a variety of reasons. Understanding segregation of bimodal mixtures is relatively easy. However, real materials are usually a mixture of more than just two components. Interaction of all components in a mixture leads to complex segregation behavior that is not intuitively obvious, nor explained by popular academic bimodal segregation theories. Knowing why and how much segregation is occurring in the system allows formulators and engineers to design robust products and processes that increase product quality, reduce product loss, and increase customer acceptance. Our next Newsletter will address how to characterize and mitigate multi-component and multi-mechanism segregation issues.

Future Topics

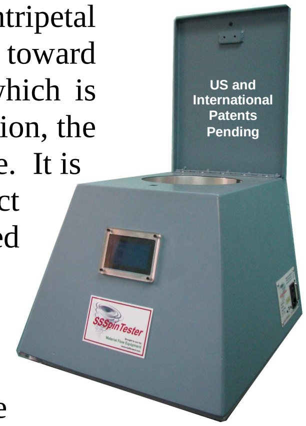
To put you at the cutting-edge

- Eliminating material hang-up
- Blending of powders
- Robust product design
- Making the process work for you

We encourage and welcome your suggestions and special requests for powder flow topics which you would like to see included in future editions of *Powder Pointers.*

Contact: Susan at 352-379-8879

Novel Powder Strength Tester – Release in 2012



The Science of Centrifugal Force. Reactive centrifugal force is the reaction force to centripetal force. A mass undergoing curved motion, such as a circular motion, constantly accelerates toward the axis of rotation. This centripetal acceleration is provided by a centripetal force, which is exerted on the mass by some other object. In accordance with Newton's Third Law of Motion, the mass exerts and equal and opposite force on the object. This is the reactive centrifugal force. It is directed away from the center of rotation, and is exerted *by* the rotating mass *on* the object that originates the centripetal acceleration. The concept of reactive centrifugal force, as used in mechanics and engineering, is referred to as just *centrifugal force*. (Wikipedia)

The *SSSpinTester* Revolutionizes the Chemical and Pharma Industries

In Pharmaceuticals. All drugs must be "packaged" somehow with excipients in order to be marketable. Material bulk properties MUST be measured at some point in the development process to quantify drug formulations for use in tablet press, tablet fill, and segregation modeling. The *SSSpinTester* can be used to measure bulk properties of pharmaceutical powders at formulation time. In the formulation step of drug development, only a few grams of material are created due to very high cost of production. The *SSSpinTester* is the only instrument that can measure material bulk properties (strength) during the formulation step in drug development—the amount of sample required by other testers prohibits measuring these properties until much later in the process. Having this data sooner rather than later narrows the R&D path, speeding time to market for new products by at least as six to eight months. Optimization and characterization of products can now be done at the formulation stage. Low pressure measurements simulate filling of dies—characterization and quality control can now be achieved on a capsule-to-capsule, tablet-to-tablet basis.

How it works. The *SSSpinTester* uses the science of centrifugal force to measure the unconfined yield strength of fine powders by first consolidating material using centrifugal force and then causing the compacted material to yield using centrifugal force. Using state-of-the-art technology, it allows measurement at forces as small as 0.2 KPa and as large as 6000 KPa (ASTM standard base limestone). We no longer must rely on inherently inaccurate extrapolation for answers. Current methods of measuring strength of a powdered material require at least 50 gram of sample—and some as much as 300 gram—usually hard to come by in the pharmaceutical and chemical industries. If you can generate sufficient sample to run a particle size analysis, you've got a sample of sufficient quantity to measure strength with the *SSSpinTester*.

In Chemicals: Measured values of material strength under stress are critical to proper design and utilization of both process system equipment and product characterization. Material properties of fine powders often change with exposure to fluctuating environmental conditions. This is particularly true in the chemical industry. The single sample *SSSpinTester* analysis can be conducted in under ten minutes, making it an essential tool from the formulation process, all the way through end-product quality assurance.

Using the Tester. Material sample size is based on the parameters of the testing cell: a conical frustum with a top aperture 0.25 inches and 0.20 inches deep. Depending on sample density, this translates into a bulk weight between 0.01 and 1.00 grams material to conduct the single test necessary to determine the unconfined yield strength. Materials tested to date include ASTM standard limestone reference powder, herbiceuticals, household cleanser, time-release allergy compound, sodium sulfate, lactose monohydrate, multiple spices and mixtures, powdered drink mix, light-weight polyethylene powders, pigment powder, tungsten/lead mixture, calcium carbonate, titanium dioxide, infant formula, sodium citrate dehydrate, fabric care compound, gelatin, cosmetics, vitamins, talc, sleep aid compound, and more.

Specific Machine Features:

- *Quantifies the strength of fine powders in as little as 15 minutes.*
- *16x18 inch footprint makes the tester easy to accommodate in any testing lab.*
- *Requires ONLY ~0.05 grams of material to run a full strength analysis.*
- *Testing range from 0.2 to 6000 KPa.*
- *Ships with pre-programmed computer and test cells*

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This deformation induces nearly perfect shear and causes the particle to experience strain at a controlled stress condition. As the test cell osculates between a square and a rhombus shape, we can measure breakage at a prescribed stress level for several total strains. It is obvious that this degradation date would be useless when applied to transfer in pneumatic transfer systems. However, it exactly describes the breakage that may occur in silos, screw feeders, rotary valves, low shear mixers and other process equipment that moves material slowly.

Shear Box for Measuring Degradation Potential

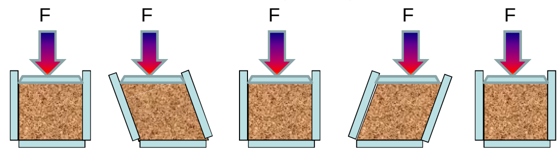


Figure 2. Schematic of stress strain degradation test

Relating Test Data to the Process. Consider bran cereal that has been subjected to stress-strain events. We use the difference in cumulative particle size between the unstrained and strained materials as a measure of particle breakage (Figure 3). This is expressed as a difference in cumulative particle size as indicated in Figure 4. Note that the peak in Figure 4 corresponds to the greatest difference between the two cumulative curves before and after subjecting the cereal to stress-strain events. After computing similar degradation profiles for other stress and strain conditions, this information can be used to determine the degradation of a material in a particular process geometry. For example, consider the bin below that is 20 foot in diameter with a 40 foot tall cylindrical section and necks down to a 2-foot diameter outlet (Figure 5). We have shown the typical stress and strain profile that would exist in this storage bin. A series of stress-strain degradation tests could be conducted to cover the range of stress level as well as strain levels in this bin, and that data could be used to compute the expected particle breakage in this bin.

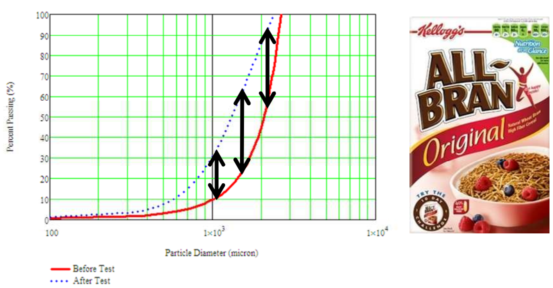


Figure 3. Typical degradation due to stress-strain events at a prescribed stress and total strain: stress of 414 psf and a strain of 52 in/in

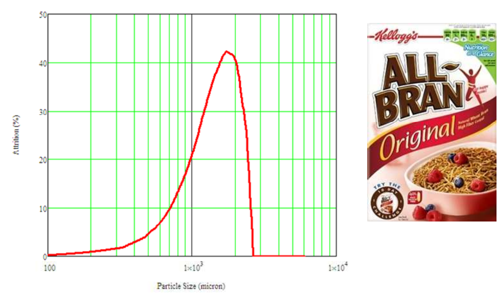


Figure 4. Cumulative degradation at a stress of 414 psf and a strain of 52 in/in

Figure 6 shows the expected particle size degradation for the bin configuration shown in Figure 5. In the zero strain zone, crushing occurs. In the hopper section, the high stress and low stain region shows a rapid increase in particle breakage followed by a slow increase in particle breakage in the low stress and high strain section. This behavior suggests that taking steps to retrofit this bin and minimize stress levels will mitigate particle breakage.

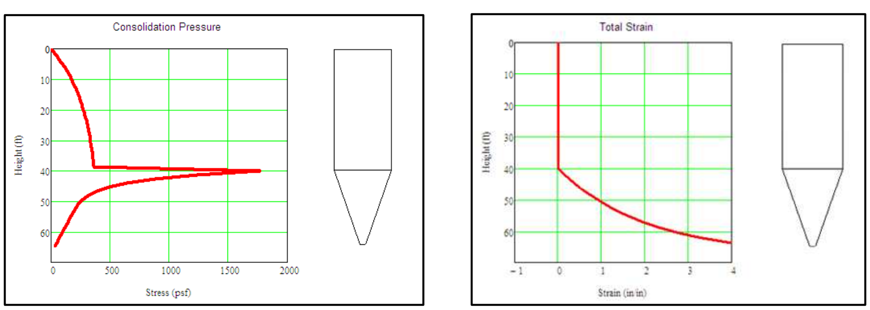


Figure 5. Typical stress and strain profile in bin

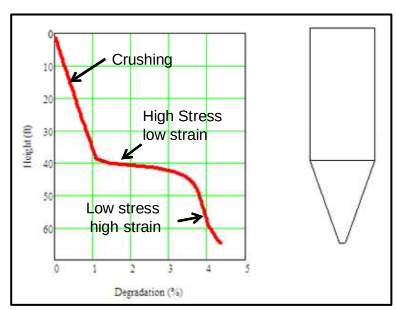
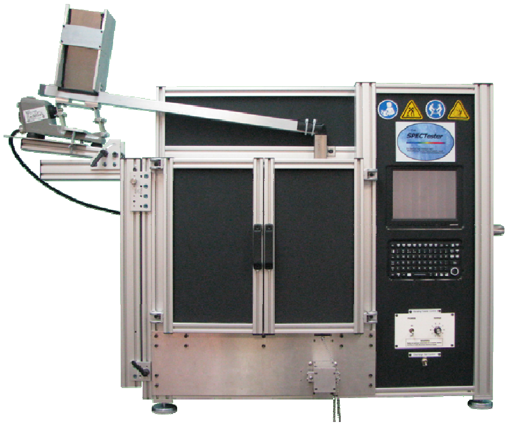


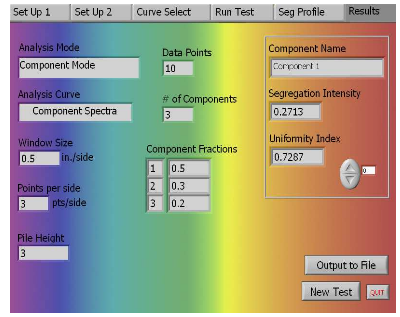
Figure 6. Typical degradation profile in bin (400 mm particles)

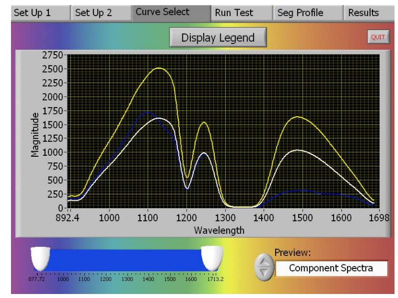
Examining a degradation problem from this mechanistic viewpoint allows engineers to design a solution to address troublesome particle breakage issues. This approach saves time and money and allows minimal down time to make process changes. The key pieces of information needed are a correct description of the degradation behavior of your material and knowledge of stress-strain profiles in the unit operation. Similar analyses can be carried out for screw feeders, belt feeders, rotary valves, blenders, transition points, pneumatic conveying systems, blow pots, and stock piles. Measuring the degradation due to impact and stress-strain events is a valuable characterization tool.

Mitigate Segregation with the SPECTester *(Continued from page 1)*



Data is presented numerically and graphically

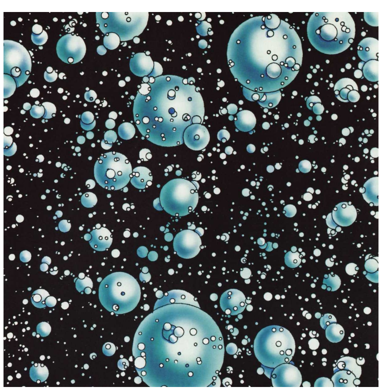




Segregation, also called separation, of granular and powder materials is one of the three main causes of process failure with systems that handle powder materials. It is a global problem, affecting all industries: conservative estimates suggest that 30% of all unscheduled process downtimes are due to segregation and quality issues. The SPECTester's spectrophotometer acquires data to analyze the segregation potential of a material mixture by scanning spectral reflectance of the top layer (edge) of the material pile in the testing hopper. It compares that gathered data to the previously acquired spectras of the individual ingredients in the mixture by de-convoluting the data to identify the presence and concentration of the various components and their locations in the hopper. The SPECTester will measure up to 50 squares along the pile edge, with a matrix of up to 49 points within each square. Concentration data identifies the magnitude of the segregation. Using state-of-the-art spectroscopic technology, the innovative SPECTester measures samples containing up to six unique components and reports how much, as well as why, your material is segregating. Fully automated, the SPECTester identifies: component concentrations, particle size differences, differences in chemical composition, product uniformity, and up to four specific segregation mechanisms.

Learning the Trade – Particle Size Distribution

Knowing and understanding key material properties is power to characterize bulk material flow behavior. We will empower you quarterly as we discuss one of these fundamental flow properties and its industrial application.



Particle-Size Distribution (PSD). The particle-size distribution of a powder or granular material defines the relative amounts of particles in a sample mixture, sorted according to size.

Particle size is an important parameter that can be used for product and process design scale up. There are many methods available for measuring particle size. Some of the most widely used include, but are not limited to: optical techniques, laser diffraction, sieving, settlement, and electro-sensing. Often particle-size distribution is the primary factor in understanding how a bulk material will segregate, blend, dissolve, and fluidize. It is one of the process variables that engineers have moderately good control over. It is related to bulk unconfined yield strength and can be used to predict cohesive flow properties if the structure and surface characteristics of a particle assembly are known. Thus, we can predict cohesive flow problems with knowledge of particle size distribution, moisture content, particle surface energies, and shape changes.

At Material Flow Solutions, our particle analysis method also allows us to characterize particle shape and bulk granularity. These techniques can be extended to look at the structure of individual particles or agglomerates and determine if the agglomerate structure is robust. Particle size is the key parameter used to determine the attrition in a process or characteristics of a particular material. We measure material particle size with both standard and proprietary optical methods in order to recommend optimal process parameters so your product will be what you want it to be – the first time.

All Bran® pictures courtesy of: ***http://www.mrbreakfast.com/cereal_detail.asp?id=6***