



Powder Pointers



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

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Segregation Management

Background. In the pharmaceutical industry, preventing the segregation of the active drug in the mixture is critical to assure a potent tablet or capsule. However, in some cases preventing segregation of the lubricant component is equally important, to assure reasonable dissolution and non-sticking behavior in tablet presses. Thus, we are always concerned with understanding multi-component segregation. In the chemical industry, each additive to the mixture performs a unique function in the product.

Overabundance or lack of a particular component in a mixture changes the behavior that the formulator desired to impart to the mixture, creating poor product quality and, often, User non-acceptance. Thus, preventing segregation goes directly to acceptance of a given product by the end User. In the food industry, segregation of key components causes material to have different taste or nutritional characteristics than the product was designed for. The result of this segregation is often a rejection of the product by Users of discriminating taste. It is critical when creating a new product that the quality of the product be good from the start. The old adage “you only have one chance to make a good first impression” applies to consumer market places. Consumers are fickle. If you supply them with a quality product from the start, they remember, and you have often earned a customer for life. However, one poor quality product can sour the consumer on all future products you may produce. This implies that you should strive to understand and prevent segregation that may be occurring in your process to “get it right” the first time. So, let’s discuss segregation.

Mechanisms of segregation. Segregation is a mechanistic driven process that results in the separation of key components when the product undergoes a stimulus during handling and packaging that affects the key components in different manners. It is critical to understand the reason behind the separation of these key components, the magnitude of segregation, and the pattern of segregation that may be occurring in the mixture. Understanding the cause of segregation leads us to modify the process and remove the stimulus that causes separation. Knowing the magnitude of segregation of each key component in the mix helps designers focus on designing the process to limit segregation of the most important key components. Understanding the segregation pattern allows the process design engineer to modify the process in such a way as to induce velocity profiles which remix material during handling or maintain uniform velocity profiles to prevent segregation in the first place.

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ANNOUNCEMENT: SSSpinTester Granted Patent

January 15, 2013:

Material Flow Solutions is pleased to announce that our innovative powder strength, *SSSpinTester*, has been granted a full US patent.

US 8,355,129

SSSpinTester uses the science of centrifugal force to measure unconfined yield strength



of fine powders using a sample of just 0.06 gram. Current methods of measuring the bulk strength of a powdered material require at least one liter of sample – usually hard to come by in the pharmaceutical and chemical industries. Cutting-edge technology extends the testing range to 0.2 KPA with accuracy, which allows direct measurement for arching. We no longer must rely on inherently inaccurate extrapolation for answers. If you can generate sufficient sample to run a particle size analysis, you've got a sample of sufficient quantity to measure strength with *SSSpinTester*.

GET YOURS TODAY

We will discuss a few of the causes of segregation that are common in industry. However, note that any particle scale property that is different between the components can be used to induce segregation in the bulk if the material is subjected to a stimulus during handling to which that property is sensitive.

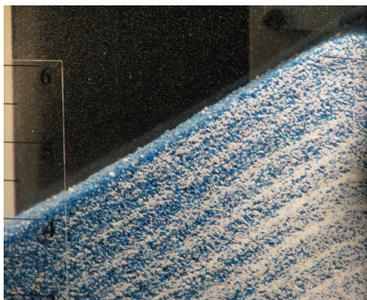


Figure 1. Sifting segregation

Sifting: Fines may sift through a matrix of coarse particles during handling when the void space between adjacent particles is large enough to permit passage of fine particles. Generally, this requires a particle size difference of about 3:1. Inter-particle motion provides the means of exposing empty void spaces to fine particles. The fines must be sufficiently free flowing to prevent arching or clinging between adjacent particles, and void spaces must be empty enough to accept finer particles. Usually, sifting segregation produces a radial pattern as material forms a pile in process equipment. Fines accumulate near the pile charge point, and decrease in concentration toward the pile edge.

Angle of repose differences: Two materials may have different angles of repose, resulting in overlapping piles where the material with the steeper repose angle accumulates near the top of the pile and the material with the flatter repose angle accumulates near the pile edge. Generally, there is a distribution of these two materials along the pile's surface. Repose angle differences of about 2 degrees can result in significant segregation. Material of different particle sizes can possess sufficient difference in repose angles to cause this type of segregation. Sometimes

adhesion forces between dissimilar particles mitigate the separation effect by changing the repose angle to make the repose angle of dissimilar products the same. Generally, angle of repose segregation produces a radial pattern as the material forms a pile in process equipment.

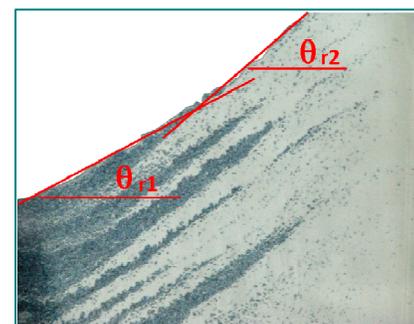


Figure 2. Angle of repose segregation



Figure 3. Air entrainment segregation

Air entrainment: The mixture may contain fines small enough to be carried by air currents in the handling system. Fines drop out of the air stream when gas velocities decrease below the entrainment velocity causing separation of fine and coarse in handling systems. Fines generally deposit near the container walls. Air entrainment segregation requires a source of air currents in process equipment. This segregation typically causes a radial pattern during pile formation. However, fines are at the bottom of the pile, and not the top.

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Powder Pointers Preview

Coming Next Quarter – Eliminating Material Hang-up

Two conditions must be satisfied for trouble free process operations using powder materials: the outlet must be large enough to overcome cohesive arching of bulk materials, and the active flow channel must be larger than the critical rathole dimension. But, how large is “large enough,” and what is the “critical” dimension which will mitigate rathole formation? All powders have flow properties that can be measured as a function of time and exposure. The properties include, unconfined yield strength, density, permeability, and wall friction angle. Both arching and rathole conditions must be overcome to assure reliable flow. In the next issue of Powder Pointers, we will focus on the use of measured material flow properties that affect material hang-up in bins, hoppers and other pieces of process equipment – and how to use this data to eliminate material hang-up.

Future Topics

To put you at the cutting-edge

- Blending of powders
- Robust product design
- Making the process work for you – optimize your design
- Managing agglomeration

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

Managing segregation. We have recently developed a segregation tester that can measure the potential of a multi-component mixture to segregate. We call it the SPECTester. The SPECTester forms a pile of



Figure 4: The SPECTester

material and then scans the pile using a spectral reflectance probe to measure the spectra of the mixture along the pile length. The spectra of the pure components are also measured, and this information is used to compute the concentration profile of all key components along the pile. These concentration profiles can be used to rank the segregation potential of key components, provide important information concerning the segregation pattern, and help identify the

cause of segregation when used in conjunction with a few easily measured particle scale properties. This testing is a critical step in mitigating segregation in a particular process. The question, then, is: How do we reduce the segregation of key components that exit the handling process? In general there are two ways to attack segregation issues in process design.

First. First, knowing the segregation mechanism, we can look at the process and determine if the process will induce separation of product via the mechanism of interest. For example, if

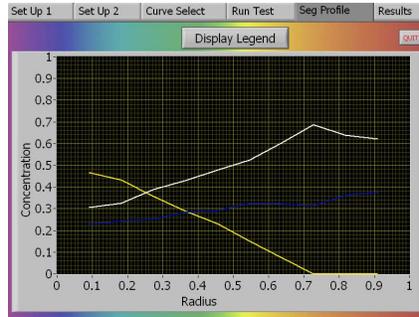


Figure 5. Typical segregation profile of a three-component mixture

segregation potential tests indicate that repose angle is a key segregation mechanism, then minimizing pile formation will help mitigate segregation in the process. However, in some cases the natural velocity profile in the process equipment causes the formation of piles during discharge. Thus, one could optimize segregation prevention by the use of distributing equipment when filling key process vessels and controlling the velocity profile during discharge to avoid pile formation (i.e. form many small piles during process fill and design the vessels to induce uniform flow). It is important to note that simple mass flow (flow along the walls) is often not enough to prevent segregation during discharge. We need a velocity profile that induces a more uniform flow across the container to limit the inverted pile formation during discharge. The use of inserts such as the cone-in-cone can help control the velocity profile during discharge. However, these systems must be properly designed.

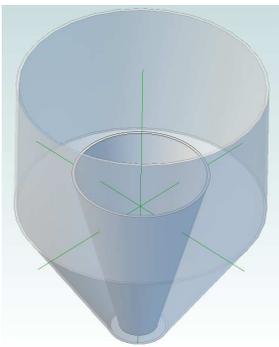


Figure 6. Cone-in-cone insert

Second. Second, we can simply look at the pattern of segregation and design a process that induces the right velocity profiles to re-mix the product together during discharge. For example, suppose that the material segregates to form a radial pattern with fines at the bottom of the pile and, further suppose, that the discharge is funnel-flow which creates a large spike of fine material exiting the hopper during discharge. In this case, one could insert an in-line blender just before the packing unit operation to smooth the spikes caused by discharge in poorly designed process equipment. Generally, this in-line blender

Upcoming Conferences

Dr. Kerry Johanson will be presenting at several upcoming conferences around the world:

Please Join Us



April 23, 2013

Nuremberg, Germany

Poster Presentation #12

“Solving Segregation by Using Cohesive Flow Properties”



Powder & Bulk Engineering

Midwest Conference

& Powder Show

May 23, 2013

Columbus, Ohio

Thursday morning, 8:00 – 11:00

“Optimizing Product Quality through Particle Size Reduction”

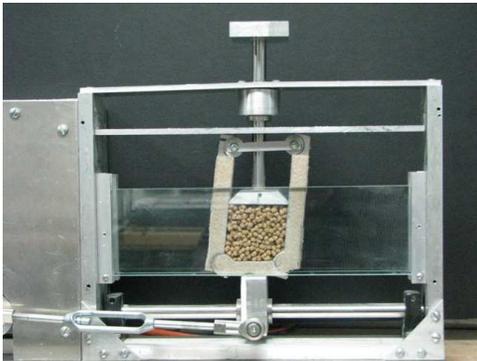
works on a continuous flow stream and has a wide residence time distribution that mixes time sequence material flow to make them more uniform. This can be done without significant headroom loss. In this case, we allow the segregation to occur in the system and then use the segregation pattern and design a prescribed velocity profile in downstream unit operations to provide a means of re-mixing segregated material.

In many cases the segregated material leaving a piece of process equipment has a prescribed segregation profile where material segregates at the beginning and end of the discharge cycle. In some cases, it is feasible to use the flow from two identical pieces of equipment to control segregation by mixing the high concentration profile from one bin with the low concentration leaving the other bin. This can be designed into the discharge system by measuring the segregation potential of the material and then utilizing mathematical models to predict the outcome from the two flow streams. These streams are then combined in such a way as to mitigate the overall segregation profile.

In all cases, it is critical to measure the material properties that govern flow behavior in the process equipment as well as the segregation potential of the mixture. With this information we can compute and design an optimal system to mitigate segregation behavior. Alternatively, we use the same information to create a new system to prevent segregation.

Learning the Trade – Attrition / Degradation

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.



Device measures stress/strain degradation

Attrition or Degradation. Attrition, also called degradation, describes particle breakage during processing. In general, particles within a piece of process equipment contact each other during shearing and impact events. These shear or impact events cause particles to break through one of three mechanisms: Fracture, Abrasion, or Fatigue.

Fracture results when stress acting on particles during shear reaches the plastic limit, causing particle failure. Fracture is also the result of impact with brittle material. **Abrasion** occurs as normal and shear stresses between particles cause subsurface cracks, which result in small flakes of material peeling off the surface of the particle. Impact may also cause abrasion as a glancing blow knocks off corners of rough particles. **Fatigue** is time-sensi

tive and requires a series of impacts or contacts that generate a minimum repeated stress on the particles. Cracks propagate at a given rate prescribed by the stress applied during a shear or impact event. The local micro-structure of the particles greatly influences fatigue behavior. Often the rate of particle size degradation changes during processing due to fatigue events. We measure degradation of material using both stress/strain and impact testing methods. We can quantify the amount and type of particle breakage in your system. Practical reasons for measuring **attrition or degradation** include, but are not limited to:

- Characterizing particle robustness
- Predicting breakage in pneumatic systems
- Predicting breakage in bins and feeders
- Predicting milling operations
- Designing robust particles
- Population balance model studies