Powder Pointers

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

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Preventing Segregation by Proper Product Design

The Segregation Problem. Segregation is responsible for a significant amount of lost product due to poor quality. Predicting and controlling segregation is critical to optimize product design or mitigate quality issues with bulk powders and granules. Segregation can be caused by several effects. Fine particles sift down through a matrix of coarse particles. Differences in repose angle can cause separation during pile formation. Air currents may carry fine particles, depositing them preferentially in different parts of the bin. In general, segregation is caused by a difference in a particle scale property. Thus, the segregation driving force is some function of these key flow properties. Cohesion can often be used to mitigate segregation. However, the effect of cohesion is different depending on the segregation mechanism.

The Key. To solve a segregation problem we need a reliable means of measuring segregation tendencies in bulk materials. Engineers use segregation data to optimize product design, creating a product with minimal segregation, or to modify the process and minimize the effect of segregation in their plant packaging process or handling facility. In either case, the segregation pattern, segregation mechanism, and magnitude of segregation are key parameters in process or product design.

Any measurement of segregation must relate to the process. Segregation occurs due to differences in key particle scale properties and almost all materials will segregate if exposed to an external stimulus that induces different behaviors based on a key property. For example, fines can be carried by air currents. However, if the process has minimal air current events, then segregation will not be prominent. The real question is: will the mixture segregate when exposed to a feed behavior similar to that present in the process? Therefore, any measurement of segregation tendency should have three key elements. First, feed must be controlled to allow the measured segregation to be scalable to process conditions. Second, the segregation pattern must be included as part of the measurement to predict the expected concentration leaving process equipment. Finally, the magnitude of segregation must be quantified. We can form a pile at conditions similar to what might exist in the process and then measure the concentration of key components down the pile using a slice box similar to that shown in Figure1. The back of this slice model is made of glass, allowing access to a view of the segregation pattern in the bulk material. Observation view ports are chosen and reflectance spectroscopic methods employed to measure subtle

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In This Issue

Dr. Kerry Johanson Will Teach at Upcoming Events

April 26-29, 2010
World Congress on Powder Technology – WCPT6
Nuremberg, Germany
Cutting-Edge Lecture / Discussion
"Using Segregation Test Data to Predict Quality of Mixtures in Handling Systems"

May 4-6, 2010
Process Technology for Industry: International Powder & Bulk Solids Conference – PTXi
Chicago, Illinois
Thursday, May 6:
Full Day Workshop on Segregation and Blending
(9:00 am-12:00 pm and 2:00 pm-4:00 pm)

June 7-8, 2010
ASME Continuing Education
Houston, Texas

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October 14-15, 2010
ASME Continuing Education
Chicago, Illinois
2-Day Course entitled "Producing Quality Powder Products"

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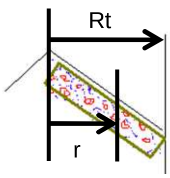
color differences in a user-defined view port. Since segregation measurement is a scale issue, the view port size must be large enough to contain a representative number of particles, yet small enough that local composition differences are not lost in the averaging scheme.

Figure 1. ***SPECTester used in experiment showing the variable speed vibratory feed system and the pile formed in the tester for analysis (note right door is open to expose the segregation hopper)***



Define the size of the view port and measure the spectra along the top of the pile. Adjacent viewports can overlap and tester can measure concentration at up to 50 locations along pile.

Figure 2. ***Measurement zone along pile top surface***

The SPECTester Solution. If the spectra of the pure components are known, and the spectra of the mixture in different view port boxes along the pile are known, then the pure component spectra can be used to compute the concentration of components along the length of the pile. A new segregation testing device called ***SPECTester*** measures segregation potential of a bulk material using both visible and NIR reflectance spectroscopic techniques. Up to six components can be measured. Experiments indicate that this spectroscopic measurement method corresponds very well to actual concentration segregation profiles measured on piles. This new tool allows the user to evaluate the effect of changing particle scale properties on segregation behavior, making it the ideal product design tool. We have applied this technique to hundreds of materials, including pharmaceuticals, foods, chemical mixtures, and even the same material at different particle sizes.

The experiment. Consider the effect of cohesion caused by liquid bridge between particles on the segregation of bulk materials. Three differently colored sands – blue 70%, yellow 15%, and white 15% were – were used to generate segregation data. Figure 3 shows the averaged spectra of the three sand components used in the mixture. Note the different peaks at various wavelengths.

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Powder Pointers Preview

Coming Next Quarter – PAT Implementation

The pharmaceutical industry is requiring that critical product properties be monitored and key process variables changed in an attempt to create quality product all the time. Other industries are implementing this type of control with looser constraints. The bulk of PAT work has focused on inline monitoring methods. However, a portion of PAT methodology relates key process and product variables to process behavior. Robust processes must be developed with enough variables to adjust process behavior and relate this to sound models that predict process behavior from knowledge of key material flow properties. Our next Newsletter will examine how to do this for typical handling systems and solids processing unit operations.

Future Topics

To put you at the cutting-edge

- Successful agglomeration
- Process simulation and predicting behavior
- Milling – new techniques

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

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How it works. These pure component spectra include the effect of surface texture, color and particle size. The mixture spectra also are affected by particle orientation. If the particles were spherical and all the same size, then there would be no optical difference between the particles in the mixture and pure particles placed in the component trays. In this case, the mixture spectral intensity $Fmix_j(\lambda)$ would be a simple linear combination of the spectral intensity of pure components ($F_i(\lambda)$) based on the local fraction ($x_{i,j}$) of each component. However, smaller particles fill the voids between coarse particles, creating a shadow effect for the coarse particles. The fine particles within the voids occupy a proportionally greater percent of area than the volume fraction would suggest. This effect can be modeled by adding a weighting factor (W_i) to the linear combination of pure spectra (equation 1). The tester compares the computed intensity curve $Fmix_j(\lambda)$ with the actual measured mixture curve $Fact_j(\lambda)$ and adjusts the weighting factors W_i and local component fractions down the pile $x_{i,j}$ to minimize the error between the two curves using a least squares approach. The result is a radial concentration profile. Now consider the effect of adding cohesion to the bulk material by comparing the segregation profiles given in Figure 4. A dimensionless radius value of 0.0 corresponds to the top of the pile; a value of 1.0 corresponds to the pile bottom. Consider the concentration profiles for the sand mixture without oil. The blue sand accumulates at the bottom of the pile while the yellow and white sands accumulate at the top of the pile. When oil is added to the mixture then the cohesive forces between the particles prevent segregation resulting in a nearly uniform concentration as a function of radial distance. The standard deviation relative to the mean of each component concentration, or segregation intensity (SI_i), is used to quantify the overall segregation potential of the mixtures (equation 2).

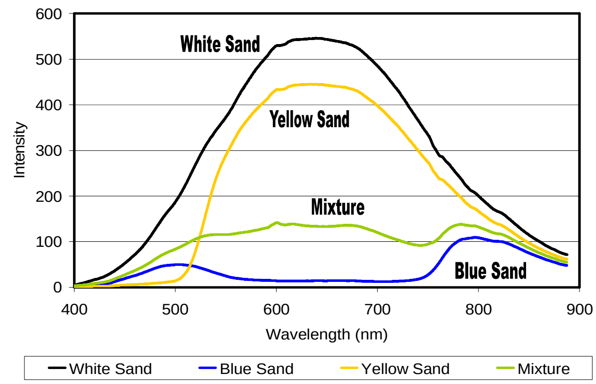

Figure 3. ***Spectra of three colored sand components and sample mixture spectra resulting in a composition of 12.5% white, 79.3% blue, and 8.2% yellow sand***

$$Fmix_j(\lambda) = \sum_i W_i \cdot x_{i,j} \cdot F_i(\lambda)$$

Equation 1

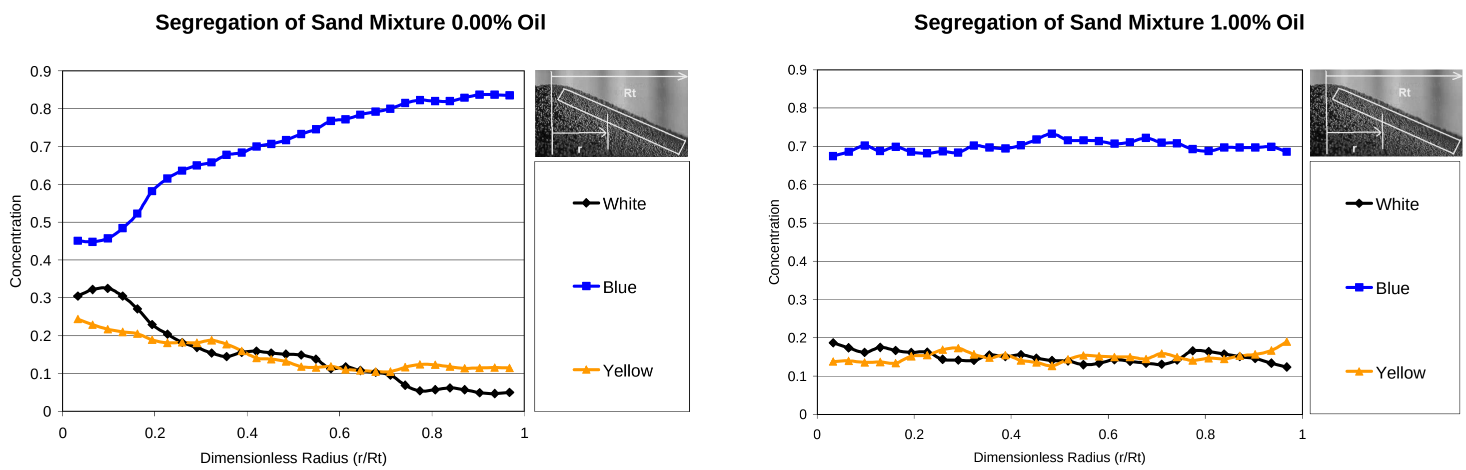

Figure 4. ***Segregation Test results for sand mixture 0% and 1.00 % oil***

Mitigating segregation through product design. Segregation intensity varies between 0 for uniform mixtures to 1.0 for completely segregated mixtures. Thus, a simple change in product properties can prevent segregation from occurring. Likewise, one could have changed the particle size or shape of key components and mitigated segregation. The trick, however, is having the ability to easily measure segregation potential quickly and accurately. The new ***SPECTester*** allows both accuracy and speed, making product design a very simple task. Since the tester requires only 10 to 15 minutes to run, many potential mixtures could be evaluated

$$SI_i = \sqrt{\frac{\sum_j \left(1 - \frac{x_{i,j}}{xavg_i}\right)^2}{n-1}}$$

Equation 2

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in just one day's time, providing the necessary data to truly optimize product design. Figure 5 shows the reduction in segregation intensity as the oil content is increased, demonstrating how a small change in formulation can mitigate segregation.

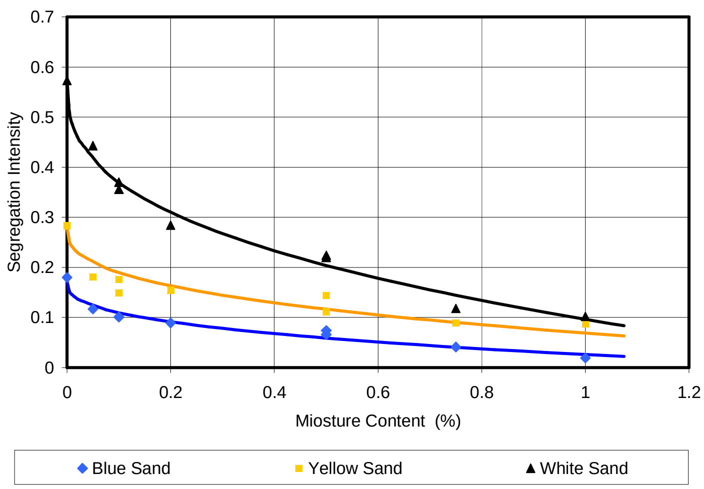

Figure 5. Segregation Intensity Index as a function of oil content for a 3-color sand mixture

Consider using this technique to help solve your next segregation problem, or to characterize your current materials.

The *SSSpinTester* (patent pending) Will Revolutionize the Pharmaceutical and Chemical Industries

Spotlighted in the March issue of ***Tablets & Capsules*** magazine. Planned release for this revolutionary tester is Spring of 2010.

In its final stages of development, the ***SSSpinTester*** measures the strength of fine powders using a sample as small as 0.05 gram. Current methods of measuring the unconfined yield strength of a powdered material require at least one liter of sample – usually hard to come by in the pharmaceutical and chemical industries.

Able to quantify the strength of fine powders in as little as 15 minutes, this novel tester takes its user to the cutting-edge of productivity. Its 14x16 inch footprint makes it easy to accommodate in any testing laboratory.

The ***SSSpinTester*** will arrive at your facility, complete with bonus testing cells, a preprogrammed laptop, and instruction manual with demonstration video on CD.

For more information: ***Contact Kerry at 352-303-9123***

Important Announcement: Growth Results in a Move

Material Flow Solutions is pleased to announce that we have outgrown our previous facility and have moved across town to a new location. Our new contact information is:

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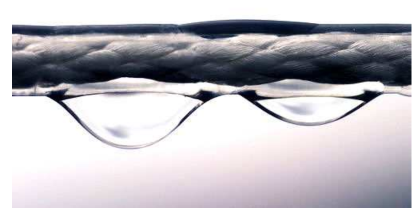
Learning the Trade

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.



Surface Tension. Surface characteristics of particles dictate the magnitude of inter-particle forces between adjacent particles. These adhesive bonds between particles result in bulk cohesion. Liquid bonds between particles can cause significant forces to hold particles together, resulting in a material that will arch over outlets and form stable ratholes in process equipment. We have developed models relating inter-particle forces to cohesive behavior. These can be used to estimate the cohesion in a process during the product design phase of the project. Thus, the design engineer provides a shorter path to market as well as a more robust process design with limited bulk property information. Practical application of ***surface tension*** data include, but are not limited to:

- Segregation prevention
- Hang-up prevention
- Risk analysis
- Blending analysis
- Process design during formulation development
- Process control
- Flow rate prediction
- Drying efficiencies