Taste the Difference–A New Method of Measuring Segregation: Steak Seasoning, a Case Study

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Abstract

Bulk solid processing generally fails due to one of three problems. The material may hang-up in the system, the material flow rate cannot be controlled by volumetric feeders, or the material segregates during handling and processing creating quality problems. The focus of this paper will be diagnosing and solving segregation problems. There is a significant lack in the ability to measure segregation especially when dealing with more than two components. Almost all bulk solid mixtures are a combination of at least three or more materials; yet our ability to measure and mitigate segregation tendencies in multi-component mixtures is significantly below our ability to create these mixtures. There is a critical need for segregation testers that can estimate the segregation profiles of mixtures of multiple components. This paper presents a method to measure the segregation potential of a mixture comprised of multiple components using spectral methods. Steak seasoning with five components was chosen as a case study mixture to show the effects of segregation in multi-component systems. Changing the physical characteristics of just one component in the system can often affect segregation of several components in the system. This study shows how changing just the size of the salt component affects at least two other components.

Key Words: Segregation, Sifting, Angle
of Repose, Reflectance Spectra

Introduction

There are two uses for segregation data. Engineers may wish to use the segregation data to optimize product design creating a product with minimal segregation. Engineers may also wish to modify the processing to 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 recommendations.

Bulk segregation can occur due to a variety of mechanisms [1,2]. Finer particles sift down through a matrix of coarse particles [3], separating as they slide down a pile. Air currents generated by free fall carry fines to regions in the bin where gas velocities decrease enough to precipitate particles [4]. Differences in repose angles can cause materials to slide down the pile at different velocities, creating a separation of materials based on particle friction characteristics [5,6,7]. In order to mitigate the segregation issues existing with bulk materials, it is necessary to understand the mechanisms causing segregation as well as the magnitude of the segregation. It is also valuable to know the pattern of segregation in process vessels. Attempting to cure a segregation problem caused by air entrainment of fines requires modifying the amount of air entrained in the free falling solid. However, modifying the free fall height will not affect segregation caused by sifting.

One problem with current segregation testers is that they do not relate well to the process. Any two or three materials can segregate if there is a difference in properties and the mixture is subject to enough external stimuli. Adding enough fluidization gas will separate materials of different densities, even though these extreme conditions may not occur in the actual process. The real question to be answered 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, the feed should be controlled so as to allow the measured segregation to be scalable to process conditions. Second, the pattern of the segregation should be included as part of the mechanism in order to predict the expected concentration leaving process equipment. Finally, the magnitude of the segregation should be quantified to provide guidance in determining if the mechanism is actually a potential problem in the process.

Segregation Measurement Device and Procedure

The ideal segregation test would be to, first, feed material into a small container in a manner similar to the actual process conditions. Then, section the pile at many (15 to 50) distinct points and, finally, measure concentration of key components in a small amount of material collected at these points. This procedure would yield enough information about the segregation magnitude, mechanism, and pattern of multi-component materials to be useful in process and product design.

In the past researchers have fed bulk materials onto a pile, sectioned a conical pile into annular sections, and performed chemical or size analysis on the material collected in each annular section [8, 9]. This is a long and tedious method which results in only a very few measurements collected along the pile. Plant personnel often use thieves to sample piles in process equipment. This method also yields only a few measurements along the pile and disturbs the surface, thereby changing the segregation pattern. Others measure segregation pattern by placing material in a funnel flow hopper and then discharging the hopper, collecting the output stream and measuring the quality of the mixture by chemical composition or particle size [10, 11]. This convolutes the segregation with the flow pattern from a particular hopper or bin. Unless the bin design in the tester is precisely the same as the bin design in the process, there is no guarantee that the composition during discharge from the tester will be similar to that found in the actual process. Additionally, the cohesive properties of the bulk material can impede flow from the segregation tester hopper, even though hang-ups may not occur in the process with the same material.

It is the author's contention that it is better to measure the segregation pattern from the pile formation segregation tester then compute the velocity pattern from flow properties. Once the segregation pattern and velocity profile are known, the composition of material leaving the hopper can be estimated. This method possesses the added benefit that, once the segregation pattern is measured, the concentration output from any series of general bins, hoppers, and other process equipment can be computed to yield the segregation effect on the entire process. Thus, this methodology provides a powerful tool to predict and mitigate segregation problems in feed systems and not just within a single bin or hopper.

The majority of real process mixtures contain more than two components. However, quite often segregation studies are done only for two component combinations [12-19]. This is done because analysis for multiple materials is difficult and time consuming. The interaction of two materials is usually simple and follows predictable rules. One example of a simple rule is that the fines fill the voids. In a two component system this causes finer particles to concentrate at the top of the pile. Simple rules applied to two component system generally result in predictable behavior.

The real challenge is to predict what happens in systems with multiple components subject to one or more segregation mechanism. Some researchers have formulated models that incorporate multi-particle effects [20 – 22]. In multi-particle flows, simple rules may not apply. The segregation pattern of a mixture containing more than two components is significantly more complex due to interaction of multiple sets of particles. Sometimes this can lead to predictable patterns. For example, materials with differing repose angle will generally concentrate in order of their repose angle along a pile surface with the flattest repose angle at the bottom of the pile and the steepest at the top. However, suppose that a material is subject to both angle of repose segregation and sifting segregation. Increasing the local concentration of fines increases the local repose angle on the pile, causing a material that was not previously segregating to separate from the bulk because of local repose angle differences. Likewise, partially filling the voids between particles with the right size particle can create a smoother surface that can facilitate segregation of other materials in the mixture as they slide down a pile. Changing one component often affects the segregation profile of all the materials in the mixture. In any event, the ability to measure the segregation pattern of a mixture with multiple components is critical to understanding the segregation behavior of that mixture.

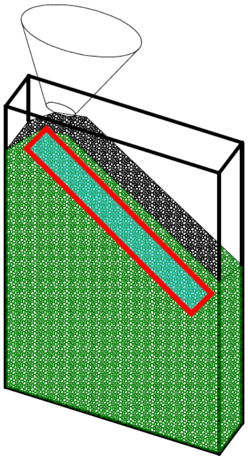


Figure 1. Schematic of segregation tester

Dump material into box and observe the change in color intensity along the pile as measured just below the top surface of the pile (rectangle section).

These changes in color intensity are an indication of differences in either chemical composition or in particle size and can be used to estimate the segregation of key components in the system.

One way to gain access to the pile cross-section is to fill a slice model with material and observe the segregation pattern through the side of the slice model using optical techniques (Figure 1).

Observation of segregation through the sides of a slice model will be biased to the optical pattern that exists at the wall of the segregation tester. Therefore, care must be taken when loading the tester to assure a representative sample is visible through the side of the tester. Filling the tester across the width of the slice model will distribute the material to the tester wall, creating a uniform material across the tester. Limiting the thickness of the slice model will also help with distribution of representative material to the tester wall. Extremely thin slice models are subject to banding due to wall effects that would not be present in wider slice models. For typical materials, this limits the slice model to a minimum of approximately 25 mm thick.

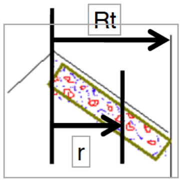


Figure 2. Measurement zone along pile top surface

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.

Reflectance spectroscopic methods can be employed to measure subtle differences in color. Since we are dealing with a discrete particle system, multiple measurements are required to determine the average concentration within a given view-port. Segregation measurement is also a scale issue. The size of the chosen view-port should be large enough to contain a representative number of particles, yet small enough that differences in local compositions are not lost in the averaging scheme. The ideal segregation tester will allow the user to change the size of the view-port, as well as the number of measurements within the view-port, to accomplish segregation measurement.

Based on the principles addressed above, a novel segregation tester (Figure 3) was created to measure the segregation pattern of various materials. The tester consists of a vibratory feed system to control the rate and fall height of material fed into the tester slice model. The back of the slice model is made of glass so the reflectance probe can view the side of the pile. This probe is connected to an x-z stage that moves to observe any portion of the slice model hopper.

Figure 3. Segregation tester 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

The light from this probe is fed to a spectrophotometer, which generates spectra of the material in the view-port area. There are also up to six component trays that contain the pure components materials and be viewed with the spectrophotometer. The front of the test unit is opened during filling and closed during spectral measurement. The black glass doors are designed to keep ambient light from influencing the spectral imaging results.

For the purposes of demonstrating the use of the tester as a means of providing product design recommendations, the material selected was steak seasoning which consists of five components of different size, shape, and color. Let's suppose that there is freedom to select one of three grades of salt for use as one of the components in the seasoning mixture. The question to be answered is: what grade of salt gives the most uniform mixture and is least sensitive to segregation issues? Steak seasoning consisting of 47% salt, 28% minced garlic, 19% black pepper, 2% red pepper, and 4% dill seed was used in this experiment. The following procedure was used in the generation of steak seasoning segregation data.

1. Create a mixture of steak seasoning using one of three possible grades of salt.
2. Feed this mixture into the segregation tester slice model at a rate of 1.5 lit/min through a drop height of 152 mm inches.
3. Place salt, garlic, black pepper, red pepper, and dill seed in the individual component measurement trays and

measure the reflective spectral response of each material for 1825 wavelengths between 420 nm to 850 nm.

4. Select a measurement zone that is parallel to the pile surface and positioned 20 mm below the top surface of the pile.

5. Select the view-port to be a square 15 mm by 15 mm and measure the spectra of 36 (a 6x6 matrix) distinct points within this view-port. Average these 36 values to create an average spectral reading of the material within the selected view-port.

6. Select 20 points along the pile to measure the average view-port spectra (total 720 complete spectral measurements per pile).

7. Using the spectra of the pure components and the average mixture spectra along the pile, compute the concentration of the pure components from numerical spectral de-mixing techniques. This will give the concentration of the different materials along the pile as a function of radial distance to the pile's edge.

8. Repeat steps 1 through 7 for each salt grade used.

Figure 4 shows the averaged spectra of the five components used in the mixture. Notice the different peaks at various wavelengths. The garlic and salt have similar spectra. The dill seed and black pepper are similar, and the red pepper has a unique spectra. These average spectra were created by filling the component holders with pure materials and averaging 10 individual spectral images of each pure component. These pure component spectra include the effect of surface texture, color and particle size. The 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 Fmixj(λ) would be a simple linear combination of the spectral intensity of pure components (Fi(λ)) based on the local fraction (xi,j) of each component (equation 1).

1) $$ix_j(\lambda) = \sum_i x_{i,j} \cdot F_i(\lambda)$$

However, in this case the smaller particles fill the voids between coarse particles, creating a shadow effect for the coarse particles. These fine particles within the voids occupy a proportionally greater percent of area than the volume fraction would suggest. This indicates that a linear combination of pure spectra will not account for the mixture spectra. The mixture spectra will be more biased towards the fine materials. This effect can be modeled by adding a weighting factor (Wi) to the linear combination of pure spectra (equation 2).

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

Figure 5 contains a list of the computed optical weighting factors for the steak seasoning. The tester compares the computed intensity curve with the actual measured curve and adjusts the weighting factors and local fractions to minimize the error between the two curves. The data in Figure 5 shows the actual measured intensity at one point in the tester and the computed weighted spectral combination for steak seasoning using the overall spectral weighting factors. There is good agreement between the measured curve and the computed weighted linear combination.

The error between the computed spectra using weighted spectral averaging and the actual measured spectra

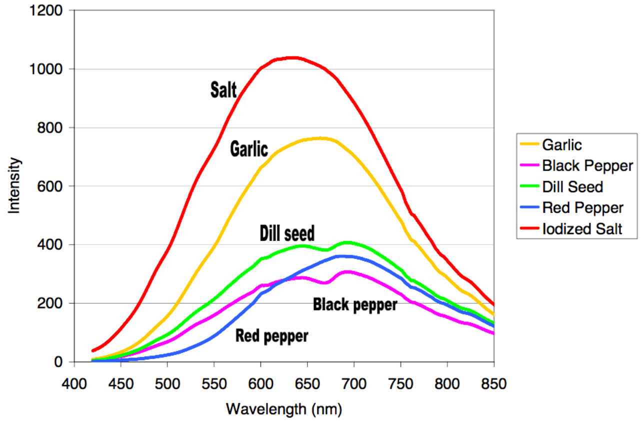
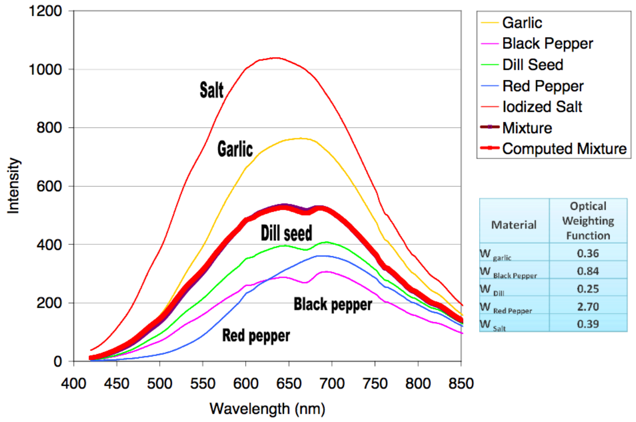


Figure 4. Spectra of steak seasoning components



Material	Optical Weighting Function
W_{garlic}	0.36
$W_{Black\ Pepper}$	0.84
W_{Dill}	0.25
$W_{Red\ Pepper}$	2.70
W_{Salt}	0.39

Figure 5. Sample mixture spectra resulting in a composition of 27.1% Garlic, 19.7% Black pepper, 4.0% Dill seed, 2.0% Red pepper, and 47.2% iodized Morton® salt

from the reflectance probe was minimized to obtain the local concentrations of spices in the steak seasoning mixture. This gave concentration profile data for each of salt grades as shown in Figures 6 through 8.

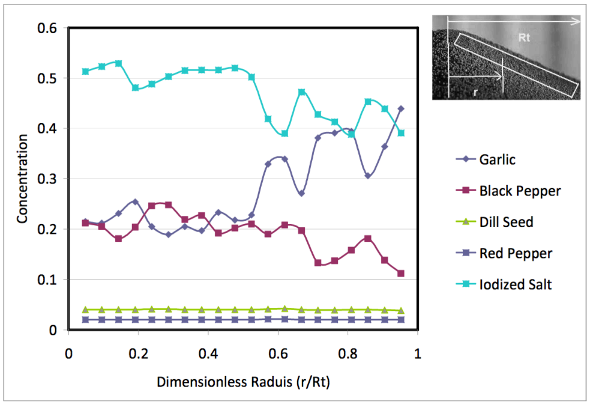


Figure 6. Segregation Test results for steak seasoning with iodized salt

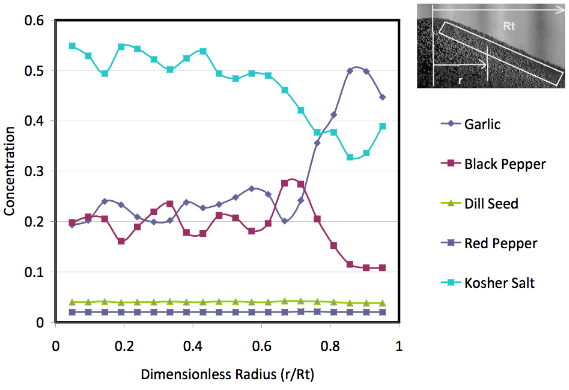


Figure 7. Segregation Test results for steak seasoning with kosher salt

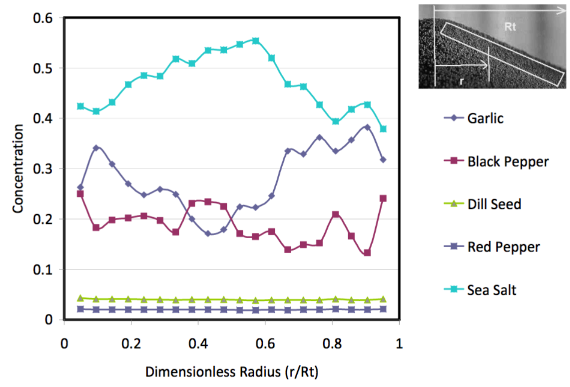


Figure 8. Segregation Test results for steak seasoning with sea salt

The dill seed and red pepper show no signs of segregation. However the garlic, salt and black pepper exhibit some interesting interaction. The iodized salt has the finest particle size of all the salts used and is subject to sifting segregation with both garlic and pepper. However, there is also some angle of repose segregation between black pepper and garlic. The finer salt accumulates near the top of the pile while the garlic accumulates at the bottom of the pile. However, making the salt particle size larger (kosher salt) actually increases the segregation magnitude. Apparently the angle of repose segregation of the coarser particles creates more segregation than does sifting segregation.

There is another way of representing the segregation occurring in a bulk material that helps characterize the pattern of segregation (Figure 9 through 11). One can compute the cumulative concentration of various compounds in the mixture and normalize them by dividing by the average concentration of each component. This cumulative data can be plotted against the radial dimension. All cumulative concentration data would vary from 0 to 1 over the radial distance down the pile. A uniform material then yields a straight line between 0 and 1. Any deviation off of this line would indicate segregation. A positive deviation off of this line indicates accumulation near the top of the pile. A negative deviation off of this line indicates accumulation toward the bottom of the pile. The overall deviation from this line is a quantitative measure of segregation. Figures 9 through 11 suggest that the worst segregation occurs with the kosher salt. The least segregating salt is the sea salt. This reduces the overall segregation of all components.

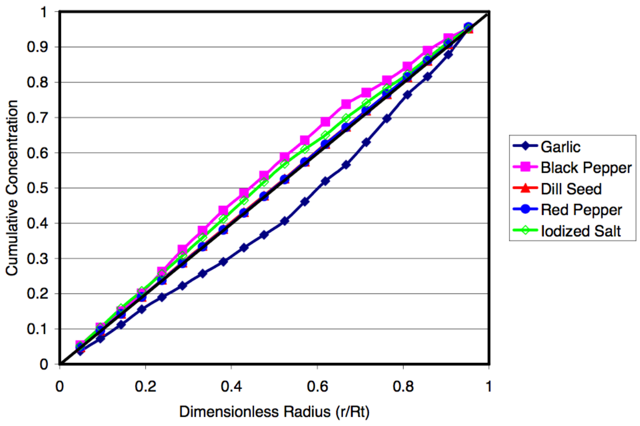


Figure 9. Cumulative steak seasoning distribution as a function of radial dimension for iodized salt

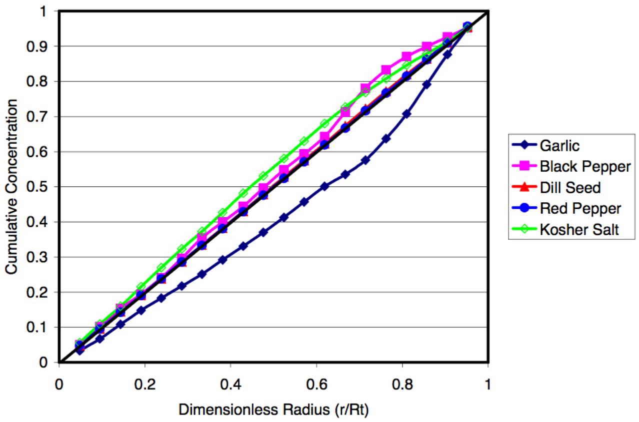


Figure 10. Cumulative steak seasoning distribution as a function of radial dimension for kosher salt

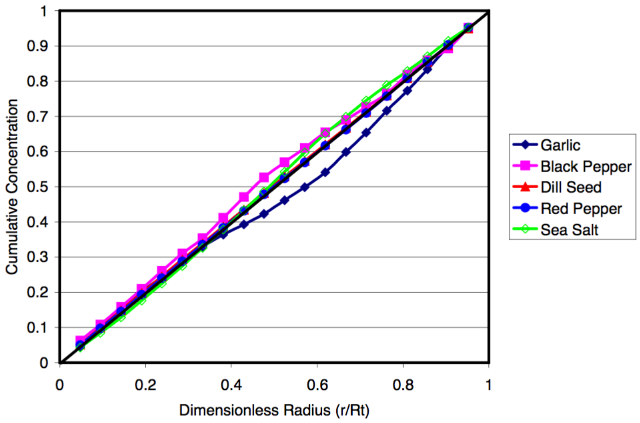


Figure 11. Cumulative steak seasoning distribution as a function of radial dimension for sea salt

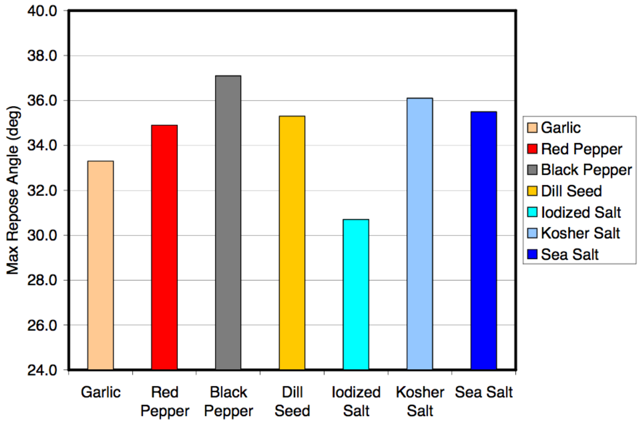


Figure 12. Angle of repose for steak seasoning components.

We also measured 6 repose angles for each of these materials and selected the maximum repose angle to be representative of the bulk material (see Figure 12). If repose angle were the only mechanism of segregation with this material then the materials should segregate in order of repose angle down the pile. The top of the pile should be rich in black pepper, followed by dill seed, then red pepper, garlic and finally iodized salt. However the reality is that the red pepper and dill seed do not segregate. This is likely due to the low percentage of these materials in the mix. There is not enough particulate material to effectively form a unique repose angle and the repose angle of these two ingredients are close to the median response angle. Thus, there is little driving force to cause these two materials to segregate. The black pepper has the largest repose angle and it always show a positive deviation from the cumulative concentration plots indicating that black pepper accumulates near the top of the pile. This is validated by the repose angle measurements. However, the repose angle data would suggest that the salt should accumulate at the bottom of the pile. In reality the segregation tests indicate that iodized salt accumulates at the top of the pile. This is due to the fact that the iodized salt has a finer particle size and will also sift down through the void of coarse particles depositing near the pile top. Thus, the angle of repose driving force and the sifting segregation driving force oppose each other decreasing the overall segregation of salt. However, Kosher salt has some fine particles that can promote sifting and the angle of repose of Kosher is close to that of black pepper. Thus both the angle of repose driving force and the sifting driving force promote salt accumulation at the top of the pile. This is validated by the results of the segregation tester which shows Kosher salt as the worst material to use for steak seasoning mixture. The sea salt repose angle is similar to the dill and red pepper angle and is a little smaller than the kosher salt repose angle. The particle size of the sea salt is larger than the kosher salt so both the angle of repose segregation driving force and the sifting segregation driving decrease making the sea salt the optimal salt to use for steak seasoning.

It is obvious from the analysis above that this novel segregation tester plays an important role in product design. It can provide guidance in product selection when multiple segregation mechanisms are significant. It is important to point out that the optimal mixture still exhibits some segregation, but it is the best that can be done subject to the constraint of only changing one material. Further work could be done to optimize the segregation profile by changing other components. Each segregation test requires about 10 minutes to perform. Thus, in one to two hours an engineer can determine the optimal steak seasoning mixture, whereas previous methods have taken days or weeks to gather the necessary data. This segregation tester can reduce the time required to generate optimal product recommendations using lab scale batches. It is a powerful tool to understanding segregation of bulk materials.

Nomenclature

$Fmix_j(\lambda)$	reflectance spectral intensity of the mixture in the tester as a function of wavelength
$F_i(\lambda)$	reflective spectral intensity of the pure component as a function of wavelength
r	radial position from the top of the pile to the point of interest
Rt	radial dimension of the bottom of the pile
W_i	optical weighting factors for each particle based on all data measure on the pile
$x_{i,j}$	fraction of the ith pure component at the jth position on the pile
λ	wavelength of light

Conclusions

The new spectral method of measuring segregation presented above is a valuable tool for enhancing product design. This test method provides an easy way to measure the potential segregation behavior of prospective or current mixtures. The measured segregation tendencies can then be used to optimize the mixture by selecting the right components for combination. This should facilitate the production of new materials using lab scale quantities.

An important side note is that segregation of multi-component mixtures is often due to multiple segregation mechanisms. The tester can identify up to four prominent segregation mechanisms in addition to particle size and chemical composition. It also shows the influence of the segregation of one component on other components in the system. The segregation potentials of all components are inter-related, and changing one component often changes the segregation potential of all components in the system – usually in a complex way. Observing these details is possible since the tester measures many concentration points, providing valuable information and allowing the user to obtain more segregation features than traditional segregation testing will allow.

It is clear that this tester is valuable for successful product design. However, the fact that segregation magnitude is dependent on changes in one product, and that these differences can be measured, means that the tester can also be used to provide quality control information in the plant. A sample of material can be obtained from the plant and placed in the tester. This tester then measures the segregation of material and compares this with the segregation potential of an ideal mixture. Small changes in either the size or shape of single components will cause subtle differences in the measured segregation potential and segregation pattern. One of the keys to successful process control is the ability to directly measure the influence of key properties on critical bulk behavior. Wherever possible, direct measurements of critical process behavior should be used as the control variable. In other words, if product segregation is a key process behavior to control, then it is much more advantageous to measure segregation directly than measure wall friction angle and cohesion and infer segregation behavior. This tester allows direct measurement of product segregation.

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