Powder Pointers

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

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Robust Products Through Proper Design Principals

Background. When engineers think of product design they typically think about satisfying the end use that the product was designed for. Typically the product must have the right chemistry to accomplish the task at hand. For example, a pharmaceutical powder to cure the common cold should have a chemical makeup to actually be able to cure the common cold. In some cases the product must have the right physical characteristics required by the user. For example, the catalysis particle must have the right pore structure and available active sites to convert one molecule to another. These product design descriptions are primary product design constraints. Typically much of the research done on a product falls into making sure these primary product design parameters are satisfied. These types of design requirement are very specific to the end use of the product, and optimization of these requirements is intimately connected to use. While these design constraints are paramount, we will not consider these types of product design requirements in this newsletter.

There are a set of product design requirements that are secondary requirements which play a major role in maintaining quality and/or ease of use of the product. Once the chemistry is set, these are the product properties that will make or break a production facility. These product requirements must be optimized to maintain end user acceptance. For example, you may have a pharmaceutical formulation that can cure the common cold. Yet, if it cannot be made with consistent quality or in units that have a controlled quantity in the package, then the product can not maintain market place acceptance and will fail. Likewise, a food product that segregates excessively will not remain in the market place unless steps are taken to mitigate this issue.

Finally, there are some product requirements that are simply nice to have but will not significantly impact the quality or salability of the product. For example, it is always nice to have a product that limits the dust formation during handling and processing. But this characteristic of the product will not make or break a process unless the process design is driven by EPA standards.

The Questions. How does one characterize the right aspects of the material so as to have the information to rank a product as good or bad and how can one change these key parameters to design the product with the right attributes? Books could be written on this process, but we will merely introduce the concept in this Newsletter. You may contact us for more details as they apply to your products. *(Continued on page 3)*

In This Issue

Announcements:

THE UNIVERSITY OF MISSISSIPPI
School of Pharmacy

Hands-On Course in Tablet Technology

Dr. Kerry Johanson will present a keynote lecture on:

"Mixing and Blending"

Dates:

- March 2-7, 2014
- June 1-6, 2014
- September 21-26, 2014

Measuring the interaction of a two-component mixture is simple. As one component concentrates in a particular region, the other must be sparse in that same region. However, very few real mixtures are limited to just two components. When three or more components are in the bulk mixture, interaction of the different particles becomes more complex and not easily predicted by simple models or rules.

To address this very issue, Material Flow Solutions has developed a novel segregation tester to be utilized to solver real segregation problems in the real world. We call it the SPECTester (US patent: 8,467,066). The SPECTester measures individual components of a combined sample and provides data that can be used to determine the magnitude, type and reason(s) for this mixture separation. It accomplishes this by measuring the segregation (separation) potential of a bulk composite material of up to six (6) components. The SPECTester's ability to analyze mixture samples of multiple ingredients is significant because it can be used not only during the formulation process, but actually on the production line as a quality control measure. The SPECTester is the only testing machine currently available that can do this.

A comparison of on-the-market segregation testers is most easily referenced with a table:

Testing Capabilities	*SPECTester*	Jenike Sifting Tester™	Jenike Fluidization Tester®
Identifies primary segregation mechanism	**YES**	no	no
Identifies process design parameters	**YES**	no	no
Identifies process quality control issues	**YES**	no	no
Results scaleable to process conditions	**YES**	no	no
Provides uniformity index for sample	**YES**	no	no
Provides segregation variance data	**YES**	no	no
Segregation by particle size	**YES**	YES	YES
Segregation by sifting	**YES**	YES	no
Segregation by fluidization	**YES**	no	YES
Segregation by angle of repose	**YES**	no	no
Segregation by chemical component	**YES**	no	no
Segregation by air entrainment	**YES**	no	no
Measures segregation upon hopper discharge	**YES**	YES	no
Measures segregation within hopper	**YES**	no	YES
Number of segregation points measured within sample	**up to 50**	-	3
Number of components in sample	**up to 6**	2	2
USB output to thumb-drive (included) Excel-ready data	**YES**	no	no
Automatic analysis	**YES**	no	YES
Additional equipment required for data acquisition	**NONE**	required	none

The *SPECTester* outshines all competition

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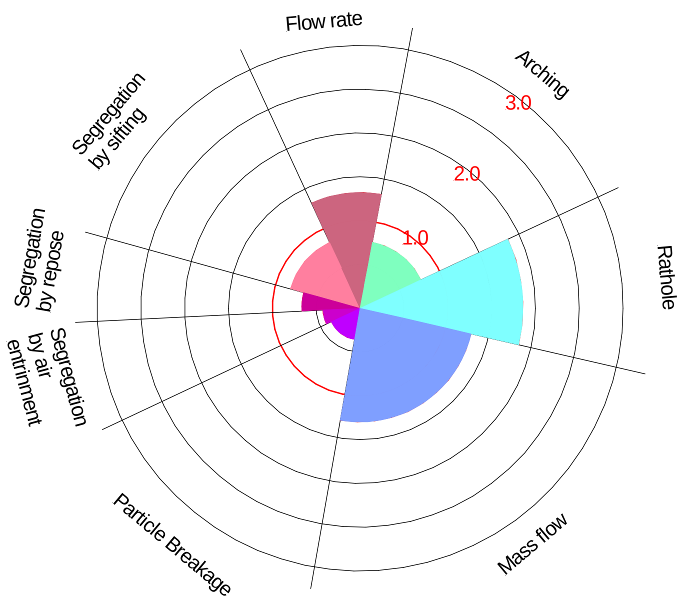
The Answers. First, it is important to point out that there is likely not one key parameter that makes a product good or bad. The concept of a good or bad product requires combination of several key parameters. A bulk powder material may cause arches in feed systems while, at the same time, be subject to limiting flow problems or segregation issues. Although these effects often depend on each other, they can easily be mutually exclusive. Second, it is important to note that the definition of good and bad product depends on the handling system or issues around the consumer's point of use. What is good for one process may be defined "bad" for another process. Therefore, whatever system we use as a ranking system must incorporate both of these concepts.



The concept that good or bad is a multi-parameter entity can be described by the use of a spider plot. A spider plot relates several attributes as distance from a common apex. However, the issue with this type of plot is that each leg or ray identifying the attribute must be scaled in some sensible way that has meaning with respect to either the process or the constraints on the consumer point of use – and that scaling on each leg does not generate any bias for that attribute. It is also important to group similar attributes together and recognize that a spider plot is actually a representation of a bar chart since there is no guarantee that neighboring attributes have any correlation to each other. There is also a need to provide a means in this description to allow user bias to rank the importance of the attribute without generating any bias in the scaling relative to the process. Thus, we will use the following system as a means of comparison of materials. The spider plot will be divided into pie shaped sections where the angular section size is based on the user bias. The greater the importance based on the user weighing, the wider the piece of the pie. The distance from the center represents the behavior relative to the process and this ranking will be described below. A number of 1.0 will represent the critical point for the process and a number exceeding 1.0 indicates a potential issue with the material selected and the process. Thus, a good material would have all pie slices within the 1.0 circle. We have grouped the cohesive properties together and the segregation and blending attributes together to give the engineer a general feeling of how the flow properties may govern the process behavior. Now let's discuss how to generate the scaling relative to the process. We will assume that the smallest outlet in the process (D_{out}) is 8-inches equivalent conical diameter. This suggests that a material with a critical conical arching dimension (AI) of 8-inch would cause problems in the process. Thus, the obvious choice for ranking for the arching attribute (R_{arch}) is the following:

$$R_{arch} = \frac{AI}{D_{out}}$$

Equation 1

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Powder Pointers Preview

Coming Next Quarter – Make the process work for you: optimize your design

Plants that handle powders or bulk material are currently running at about 60% efficiency, often due to unscheduled downtimes. Many downtimes occur as a result of poor material flow through systems. Therefore, design of robust systems is of paramount importance. Our next Powder Pointers Newsletter addresses powder process design to avoid hang-ups, prevent flow stoppages, control process flow rates, and prevent mixture segregation based on measurement of key flow properties at process conditions and using sound design principals based on these flow properties. The relationship between flow in process vessels and the material properties that control process behavior can be used to determine which solutions will work for your particular process and which should be avoided.

Future Topics

To put you at the cutting-edge

- Managing agglomeration
- Controlling particle breakage
- Preventing caking
- Designing non-segregating materials

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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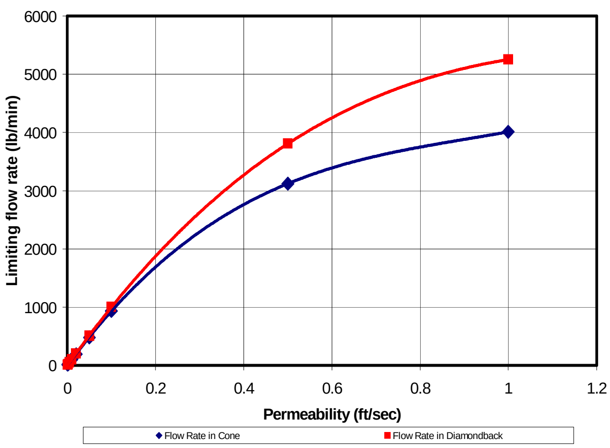
Likewise, a similar technique can be used to determine if the material in the process will require mass flow bins or if partial mass flow bin might be used to overcome cohesive effects. In this case, the guiding attribute is the critical rathole diameter (*RI*). If the critical rathole diameter is greater than size of the bins (D_{bin}) or containers in the process, then the entire container or bin must be designed to induce flow along the walls for reliable process operation. Thus, the scale ranking for the rathole attribute ($R_{rathole}$) is:

$$R_{rathole} = \frac{RI}{D_{bin}} \qquad \textbf{Equation 2}$$

Generally, the process will have a hopper angle or flow channel angle design parameter that sets the shape of the active flow channel. Associated with this design flow channel is a design friction angle. Thus, the process design is governed by a characteristic mass flow angle. A bad material would be one where the design mass flow angle is (θc_{design}) larger than recommended mass flow angle (θc_{rec}) of the material in question requiring a hopper steeper than the process was designed for. Thus, the scale ranking for mass flow ($R_{massflow}$) is.

$$R_{massflow} = \frac{\tan(\theta c_{design})}{\tan(\theta c_{rec})} \qquad \textbf{Equation 3}$$

Most often, the process will be associated with a design process flow rate through a characteristic outlet area (A_{out}). However, the actual flow rate can be less than the required depending on the bulk density (γ) and permeability (K) and the flow channel angle (θ). Thus, the ranking for flow rate limitations is defined as the ratio of the process flow rate to the computed limiting flow rate:



Computed limiting flow rates

$$Qs_{\min} = \frac{\gamma(\sigma_{out}) \cdot A_{out} \cdot K(\sigma_{out})}{1 - \dfrac{\gamma(\sigma_{out})}{\gamma(\sigma_{bin})}} \qquad \textbf{Equation 4}$$

$$Qs_{\max} = \gamma(\sigma_{out}) \cdot A_{out} \cdot \sqrt{\frac{D_{out} \cdot g}{2 \cdot a \cdot \tan(\theta)}} \qquad \textbf{Equation 4}$$

$$Qs_{\lim it} = Qs_{\max} \cdot \left[-\frac{1}{2} \cdot \frac{Qs_{\max}}{Qs_{\min}} + \frac{1}{2} \cdot \sqrt{\left(\frac{Qs_{\max}}{Qs_{\min}}\right)^2 + 4} \right] \qquad \textbf{Equation 4}$$

$$R_{flowrate} = \frac{Qs_{process}}{Qs_{\lim it}} \qquad \textbf{Equation 4}$$

Computing a ranking factor for the particle breakage and segregation attributes is a little more difficult. Due to brevity, we will simply outline the calculation method. In the case of the particle breakage attribute, the process presumable was designed with a minimum allowable particle breakage for the product ($Break_{process}$). Tests can be done on the material to measure particle breakage due to stress/strain events and impact events. These test data can be used along with an understanding of the process to compute the stress/strain magnitude in the process as well as the expected impact velocities in the process. This data can then be used to predict the breakage in the process due to both effects ($Break_{computed}$). For more information on how to accomplish this, please contact us. Ranking for the particle breakage is then defined as:

$$R_{flowrate} = \frac{Break_{computed}}{Break_{process}} \qquad \textbf{Equation 8}$$

The segregation ranking (R_{seg}) factor is done in a similar manner. The simplest method is to generate an overall segregation intensity variance and compare that to the allowable variance in the product based on consumer acceptance ($SegVar_{reg}$). The segregation intensity (SI_i) is defined as the standard deviation measured relative to the mean concentration (c_i) and can be computed using obtained from segregation testers such as the SPECTester.

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Ranking factor values greater than values greater than 1.0 indicate that segregation is great enough to cause some packages to have concentrations outside user acceptability.

$$R_{seg} = \frac{\sqrt{\sum_i (c_i \cdot SI_i)^2}}{SegVar_{reg}}$$

Equation 9

Sometimes we are more interested in the causes of segregation. In this case, a ranking factor can also be computed. The measured segregation intensity values (SI_i) are still required, as well data for particle size, angle of repose and particle density. This data is used to determine a quantitative amount of segregation expected from each mechanism. Segregation due to sifting requires inter-particle motion. Thus, the process can be analyzed to determine the fraction of the process that may be subject to inter-particle motion. Likewise, angle of repose segregation requires pile formation and the process can be analyzed to determine the percent of the process that forms piles. The same can be done for air entrainment segregation by determining the fraction of the process that is subject to air currents. This method of ranking will not be part of this Newsletter, but please contact us if you wish to rank your systems based on these segregation mechanisms.

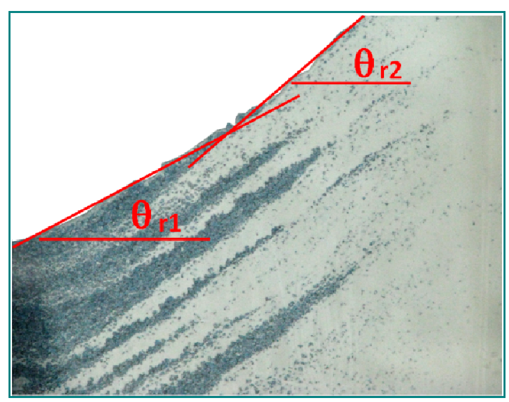
Summing Up. A spider diagram, when used as described above, can provide a snapshot of the system that allows the user to base product acceptance on scientific analysis of the bulk material. It also allows the user to impose their own weighting factor basis without changing the overall scientific basis.

Learning the Trade – Mechanisms of Segregation

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.

Segregation occurs through several mechanisms. Identification of the segregation cause and pattern produced through handling is critical to prevent de-mixing during handling and packaging. Any property difference between materials can cause separation of critical material components, although there are five common causes of segregation problems in typical handling systems. In this Newsletter, we will discuss angle of repose segregation, as well as best practices to eliminate or mitigate the condition. Subsequent issues will discuss other mechanisms and their prevention.

Angle of repose. 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. Materials may have frictional behavior between particles when differences in inter-particle friction cause different materials to flow at different velocities down a pile. Significant segregation results because these surface friction differences are highly dependent on particle shape, size, and surface roughness. Repose angle differences of just two (2) degrees can result in significant segregation. Sometimes adhesion forces between dissimilar particles mitigate the separation effect. Generally, repose angle segregation produces a radial pattern as material forms a pile in process equipment.



Two ingredients with distinct angle of repose values – note the segregation pattern

Identifying the cause of segregation allows the formulator or engineer to avoid processing that will induce the problem. Knowing the pattern of segregation helps in determining a means of re-mixing material, if required. Understanding the segregation mechanism also helps in the determination of what must be done to the material to create a product that is less likely to segregate. Combining segregation potential tests with particle size analysis and repose angle measurements, formulators obtain a detailed quantitative picture of segregation for any powder mixture. Mitigating angle of repose segregation can often be accomplished by changing the particle size and/or shape of one or more of the components in a mixture. However, rather than utilize a time consuming trial-and-error method to determine which ingredient(s) much be changed to which particle size(s)/shape(s), these analyses become a powerful tool that brings sound science to the mitigation of complex segregation problems which have been vexing the industry for years – taking the guess-work out of the process.