Powder Pointers

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

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In This Issue

How to Prevent Caking

Background. One property of material that can cause severe flow problems is a phenomenon called caking. Caking is a term that describes a dramatic increase in the bulk unconfined yield strength of a granular or powder material during storage. During a caking event a free flowing powder can gain sufficient strength to cause arches over very large outlets and form stable ratholes in funnel flow bins. Sometimes caking material can form large, hard lumps in equipment that later attempt to flow through the outlet and block the flow of material (Figure 1).

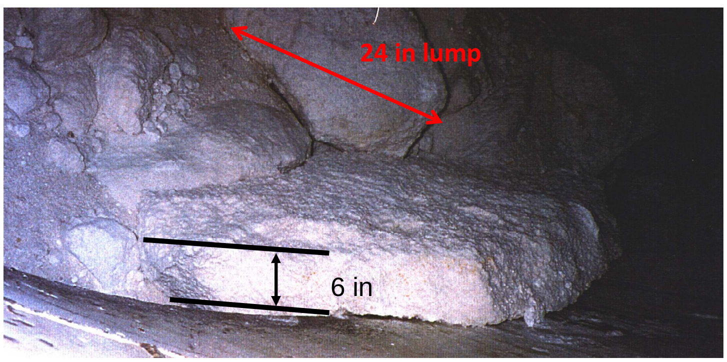

Figure 1. Large caked mass in 10 foot diameter bin with 10 inch outlet

Caking events cause significant loss of production, including expensive downtimes, and should be avoided at all costs. So how do we characterize caking and how do we design processes that handle caking materials?

Caking is a mechanistic event. In some cases caking arises from recrystallization occurring between particles. Moisture within the material pools at the particle-particle junctions and dissolves some material, creating a saturate liquid solution. Then a change in temperature or relative humidity causes the dissolved solids in solution to precipitate out, forming solid bonds between adjacent particles and resulting in caking. In other cases, caking occurs because surfaces become soft and plastic creep, followed by solidification, bind particles together. Finally, in some cases the local moisture content of the material initiates a gel reaction that triggers the transition of the material to a cementitious material. The solution for caking problems depends largely on the cause of the caking. Thus, to characterize this phenomenon, we must identify the cause or mechanism, determine the magnitude, and measure the time constant for the caking events. Once we have this key information, we can either modify the material or the process to disrupt the cause, induce process or product changes to decrease the magnitude of the event, or generate sufficient inter-particle motion at regular intervals to overcome the caking time constants.

Examples presented in this article deal with fine powders often found in the chemical industry. The same principles apply in pharmaceuticals and any industry handling fine powders. *(Continued on page 2)*

Upcoming Events:

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"

March 13-19, 2016
May 22-27, 2016
September 11-16, 2016

PARTEC 2016 International Congress on Particle Technology

Nürnberg, Germany

April 19-21, 2016

Dr. Kerry Johanson will present a lecture entitled:

"General Framework to Predict Segregation Behavior in Multi-Component and Multi-Mechanism Materials"

And a poster entitled:

"Using a Measure of Bulk Strength and a Limiting State Model to Predict Channeling Behavior in Fluidized Beds"

Tuesday, April 19, 2016

How to Prevent Caking

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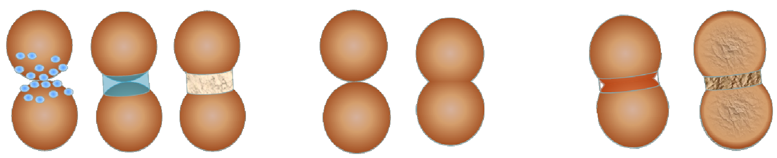
Clearly, the key flow property to measure is the unconfined yield strength of the material. Unconfined yield strength is defined as the major principle stress acting on a bulk material that will cause that unconfined material to yield or break. The greater the caking tendency, the larger the strength will be. However, unconfined yield strength must be measured after subjecting the bulk material to a stimulus that will induce a caking event.



Schematic of crystallization caking *Schematic of creep caking* *Schematic of cementation caking*

Figure 2. Different caking mechanisms

Consider the case of a material sensitive to creep caking events. Plastic pellets, low melt-ing organic powders, and many food particles subject to a glass transition temperature may experience caking due to plastic creep. One such material is erucamide prills. It is a low melting material often used as a lubricant plastics production facilities. As erucamide approaches a critical temperature, the surface of the pellets becomes soft. The soft pellets press against each other during storage and cause local creep at the solid surfaces. The rate of occurrence for this phenomenon is temperature dependent. The closer the temperature is to the melting point of the material, the greater the tendency to creep. The bulk strength increase is also temperature dependent. Strength grows exponentially as the storage temperature approaches the melting, softening, or glass transition point (Figure 3). Thus, we know that the caking trigger with this type of material is a critical storage temperature. With erucamide prills, we must keep the temperature below 45 deg C to assure free flowing material. 45 deg C is a relatively low temperature and many processes may be operating near this temperature on a hot summer day. The question that really needs to be answered is "if I am forced to operate at temperatures above some critical value, how long do I have before significant caking will occur?" We can answer this question by measuring the bulk unconfined yield strength at some critical consolidation pressure and storage temperature for several storage times.

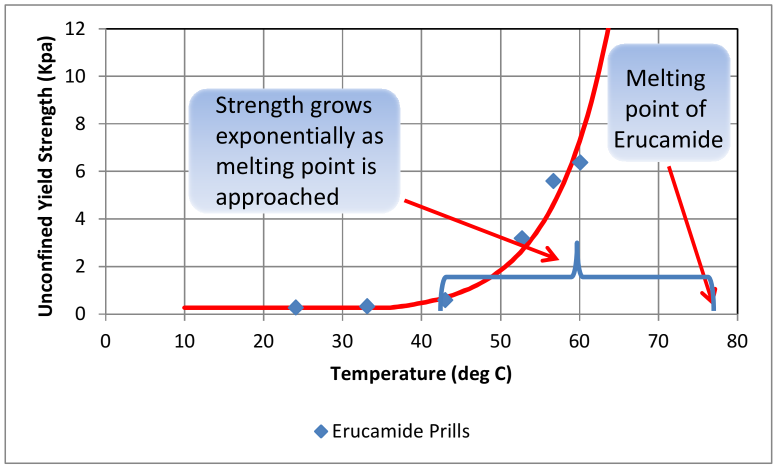


Figure 3. Effect of creep caking events on bulk strength

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Powder Pointers Preview

Coming Next Quarter – Product Design to Avoid Segregation

Segregation is a critical issue in the pharmaceutical, food, cosmetic, chemical, powder metals, and ceramic industries. Segregation is a mechanistic driven process that has many causes. One intriguing question is: "can we perform a simple test on a mixture and determine the root causes of the segregation?" The second question is: "if we can fully quantify the segregation of a particular material, can we then modify the material in some systematic way to prevent segregation of the mixture due to any process induced stimulus?" In other words, can we create a material that does not segregate, no matter how we handle it? Our next Newsletter will present a systematic method of identifying the root cause of segregation, fully quantify it, and then use that information to make decisions on how to modify the material to minimize segregation.

Future Topics

To put you at the cutting-edge

- Fine powder flow problems
- Controlling particle breakage
- Moisture migration issues

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.*

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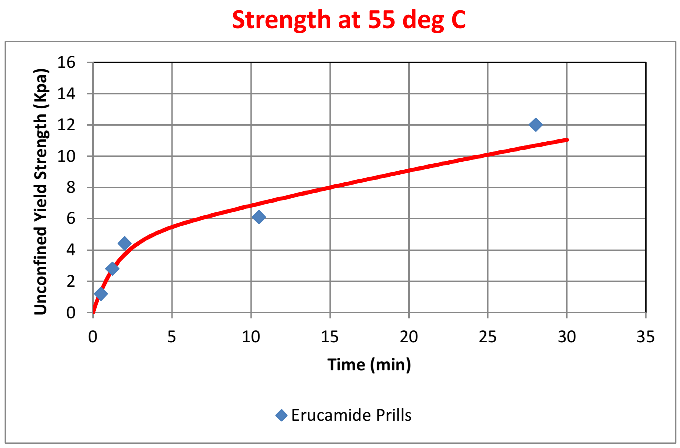
By plotting the yield strength as a function of storage time we can deduce how fast the caking events are.

It turns out that a combination of two first order rate events describe the caking of this particular material (Figure 4). Thus, there are two causes of bulk unconfined yield strength increase; one with a time constant (τ_1) of 1.5 min and one with a time constant (τ_2) of 80 min. One time constant is the amount of time required to reach 63% of the maximum strength (fc_1, fc_2) associated with the caking event. It is likely that on event is due to the viscous arrangement of particles and the other event is due to plastic creep between particles. The total maximum strength is a sum of The maximum strengths of the two events. About 16% of the strength happens with the short time constant and the remainder of the strength will be subject to the longer time constant. Next we look at the process and ask the question: “what is the critical strength value that would cause problems in this process due to either arching or rathole formation?”

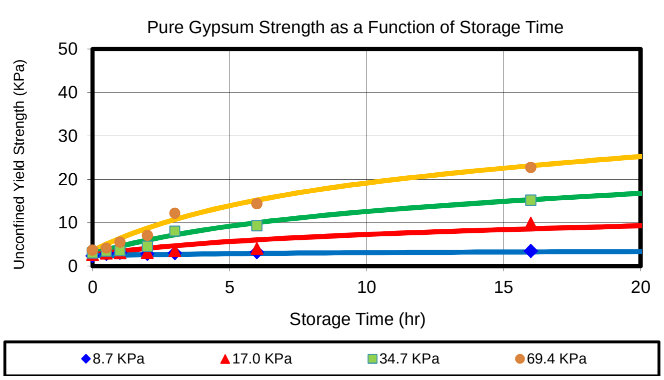


σ1 (KPa)	21.7
fc0 (KPa)	0.00
fc1 (KPa)	4.31
τ1 (min)	1.5
fc2 (KPa)	21.56
τ2 (min)	80

$$fc = fc_0 + fc_1 \cdot \left(1 - \exp\left(\frac{t}{\tau_1}\right)\right) + fc_2 \cdot \left(1 - \exp\left(\frac{t}{\tau_2}\right)\right)$$

Figure 4. Time constants for creep caking events of erucamide

There will be some critical condition that describes bad flow behavior in the process. Suppose, for the sake of example, that critical strength value was 6 KPa. The curve in Figure 4 suggests that the critical storage time is only 7 minutes. Thus, we would need to induce inter-particle motion every 7 minutes to prevent critical hang-ups in the process. This same analysis can be applied to different caking mechanisms. For example, consider the case of a typical cementation reaction (gypsum). In this case, if the material is exposed to some critical moisture content the cementation reactions initiate. Caking is a strong function of the consolidation pressure acting on the material. The greater the consolidation pressure, the greater the bulk unconfined yield strength. Consider the case of gypsum powder at 10% moisture content. We can measure the bulk unconfined yield strength as a function of storage time at various consolidation pressures (Figure 5). This data also indicate the presence of two caking events. One caking event has a time constant of ~4.4 hours while the other event has a time constant of ~55 hours. In this case, about 7% of the strength is present before any caking event occurs. 20% is due to the caking reaction that has a time constant of 4.4 hours. The remaining strength increase is subject to caking with a time constant of 55 hours. If the maximum stress in the process is 35 KPa and the critical strength to prevent hang-ups is ~10 KPa, then you would need to induce inter-particle motion in the bulk material within ~6 hours to pre-vent hang-ups. Obviously, under-standing the relationship between bulk unconfined yield strength



σ1 (KPa)	69.42	34.71	17.36	8.68
fc0 (KPa)	3.60	2.90	2.60	2.40
fc1 (KPa)	10.50	6.00	3.00	0.50
τ1 (hr)	4.40	4.50	4.60	4.60
fc2 (KPa)	38.00	26.00	12.00	1.50
τ2 (hr)	57.00	55.00	54.00	53.50

$$fc = fc_0 + fc_1 \cdot \left(1 - \exp\left(\frac{t}{\tau_1}\right)\right) + fc_2 \cdot \left(1 - \exp\left(\frac{t}{\tau_2}\right)\right)$$

Figure 5. Time constants for cementation caking events of moist gypsum

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and the mechanisms that induce caking is critical to recognize how to handle a caking material. But this is not all that is required. Motion of the material is critical to the solution of a caking problem.

There are many process designs that can induce mass flow and motion of a material during discharge (Figure 6). These range from simple conical hoppers to arch-breaking Diamondback® hoppers or chisel-shaped hoppers. Both the container storing the material and the feed system must be reviewed to assure the principles of mass flow are followed so as to avoid stagnant zones during operation. However, it is important to note that simple mass flow is not always the solution to a caking problem with a given material. It is true that the formation of stagnant or dead zones is mitigated by the use of mass flow designs. Certainly the formation of stable ratholes is prevented in a mass flow design. However, the solution to a caking problem requires inter-particle motion, not just bulk motion. In some cases a mass flow solution will result in a situation where all of the material is in motion, but not subject to inter-particle motion. Traditional mass flow designs may induce zones of nearly perfect plug flow. The solution to a caking problem is to induce inter-particle motion at some critical time based on the flow properties. Thus, to solve caking problems, we often need a mass flow design with sufficient velocity profile to induce inter-particle motion and prevent the cementation or caking of material. Caking prevention is all about velocity control and not just mass flow.

Relative headroom of several mass flow hopper designs

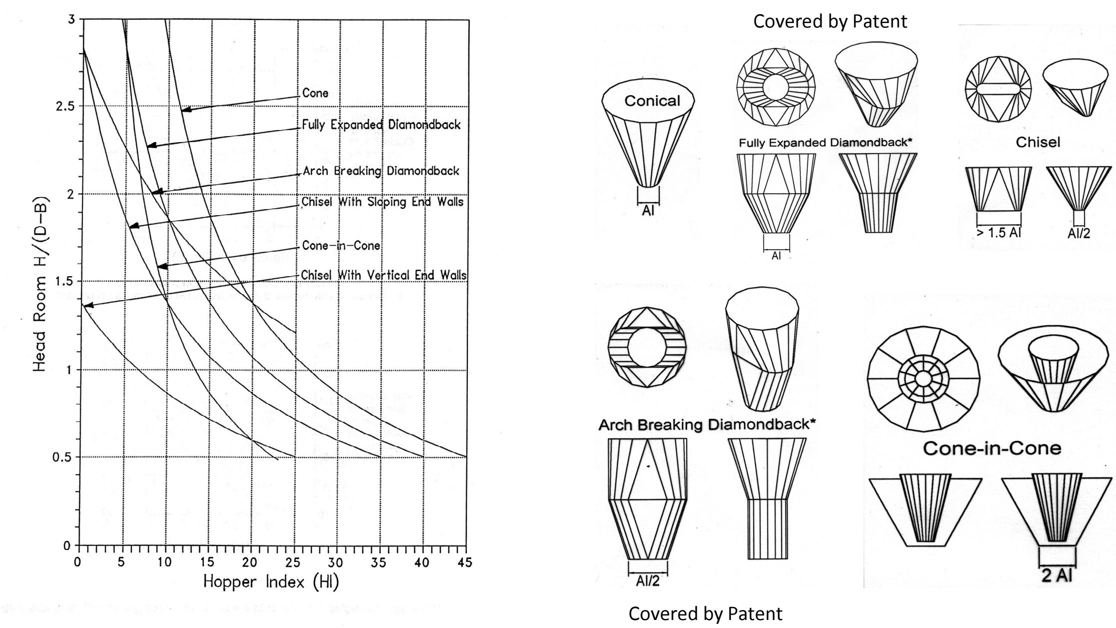

Figure 6. Typical mass flow hoppers

In summary. To solve caking problems we need to characterize the unconfined strength properties of your bulk material subject to the situation that might cause caking or the building of solid bonds between particles. In some cases this will be a temperature induced event. In some cases this will be induced by a temperature cycle, in some cases caking will be induced by a critical moisture content. It is important to understand what initiates a caking event. In general, we need to measure the bulk strength at various storage times to determine the expected caking rate behavior. Next we need to determine the maximum stress level acting in the process equipment and the maximum strength value that would cause a hang-up condition in that particular equipment. Finally, we need to determine the time constant of the caking events and predict the time required to induce inter-particle motion of material in the process based on how fast the strength reaches the critical value for the given process. This systematic approach to caking will help prevent serious caking problems in process equipment.

For more info or to discuss your application contact: Kerry @ 352-303-9123 or kjohanson@matflowsol.com

Customized Seminars:
Your Process – Your Personnel – Your Place

Material Flow Solutions, Inc. offers a set of seminar topics specifically for your process and product design engineers to help them design material handling systems, design better products, and successfully select unit operations that are compatible with critical material properties. This proven approach allows your engineers to optimize plant performance and increase your plant and operation productivity. Our seminars are available in 1-day, 2-day or 3-day venues. Customize your seminar by choosing from a wide range of available topics that best meet your company's needs. Further optimize your seminar by adding a half- or full-day plant visit that will include an on-site review of your current process.

Mix and Match. Our engineers will assist you in designing a seminar program to optimize your time and personnel investment, and assist you in increasing your company bottom line. Simply choose from our shopping list of topics and you are on your way to enhanced company profit and productivity. We will travel to your facility with a customized presentation that exactly meets your needs and parameters. Our goal is to help you "get it right the first time" through education.

Some Available Seminar Topics

- Successful food plant design
- Successful food product design
- Segregation prevention
- Bin and hopper design
- Feeder design
- Optimal blender selection
- Minimizing attrition
- Agglomeration unit operations
- Blender operation
- Mill operations

Avoid Costly Plant Downtime

Arching

Ratholes

Segregation

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 air entrainment segregation.

Air entrainment segregation. A powder blend may contain fines that are small enough to be carried by air currents in the handling system. These fines drop out of the air stream when gas velocities decrease below the entrainment velocity. This causes separation of fine and coarse particles in handling systems. The fines generally deposit near the container walls. This type of segregation requires a source of air currents in process equipment. This source of air can come from free fall of a compressible material or from air injection ports deliberately placed in the system. When the falling stream impacts the material level, the entrained air is pushed out of the interstitial pores and carries the fine particles in the resulting dust cloud – resulting in segregation. This segregation typically causes a radial pattern during pile formation, but the fines are at the bottom of the pile and not the top. They are, therefore, the first to leave the system. The result is an end product that is heavy in the fine particle ingredient at the beginning of the batch and light on that ingredient at the end of the batch.

3-component blend (dry milk, sugar, cocoa powder) showing air entrainment segregation issues

When material segregation is the result of air entrainment, the first step toward mitigation is to reduce – to the extent possible – the air source. If the air presence is the result of high drop height, retrofit your process to reduce drop height. Conversely, adjusting the particle size of one or more ingredient in the blend can minimize air entrainment segregation. Quantifying this critical segregation mechanism, then, becomes a significant device in the design engineer or formulator toolbox.