

# Understanding and solving material caking problems in dry bulk storage vessels

Kerry Johanson    Material Flow Solutions Inc.

**Material caking along your bulk storage vessel's walls can prevent your material from flowing smoothly and discharging completely from the vessel, creating major production headaches. After explaining what's behind caking problems in storage vessels, this solids flow expert gives advice on how you can prevent or mitigate the problems by changing your material or modifying your storage vessel or process.**

Caking is a common problem in bulk solids storage vessels and other equipment. When caking occurs, material can form large lumps, as shown in Figure 1, that can block a vessel outlet and obstruct flow. In a severe caking case, an otherwise free-flowing material can gain enough strength during storage to form an arch wider than 6 feet above the outlet. These caking problems can result in big production losses, including expensive downtime, so you need to avoid them at all costs.

Any of three major mechanisms can cause your material to cake during storage. Some materials cake when crystalline bonds form between adjacent particles. Others cake when particles become cemented together through a chemical reaction. Some materials cake when particle surfaces soften at elevated temperatures — resulting in plastic creep — and then solidify, binding the particles together.

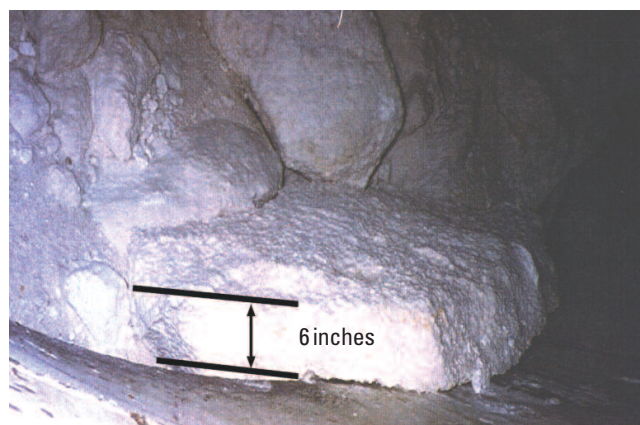
To solve a caking problem, you first need to identify its cause. Then you can modify your material or process to disrupt the cause, make material or process changes to decrease the caking magnitude, or change your vessel or process to induce enough interparticle motion at regular intervals to overcome your material's caking *time constant* (the time required to increase the material's strength).<sup>1</sup>

## Identifying caking mechanisms

The key flow property to measure when identifying a caking problem's cause is your material's *bulk unconfined yield strength* — that is, the major principal stress acting on the bulk material that will cause the unconfined material to yield (or break). The greater your material's caking

Figure 1

Thick layer of caked material in 10-foot-diameter storage vessel



tendency, the larger its unconfined yield strength will be. And, of course, for a caking study, you must measure this strength *after* subjecting your bulk material to a caking stimulus, such as temperature cycling or storage time. [*Editor's note:* You can test your material's bulk unconfined yield strength with a uniaxial compression tester or a shear cell tester; for more information, see the later "For further reading" section or contact the author.]

Let's look at how crystalline bond, cementation, and plastic creep caking mechanisms work and how you can identify them.

**Crystalline bond caking.** Crystalline bonds, as shown in Figure 2a, form between particles when the stored material is somewhat soluble and a water source is present — in the material, in the vapor space between the particles, or in the air (or other gas) surrounding the material. The mechanism starts as moisture condenses or accumulates on the particle surfaces and migrates to the junctions between particles. This liquid dissolves a portion of each particle, creating a saturated solution between particles. A driving force (usually elevated temperature) causes the liquid to evaporate, leaving solid bridges — crystalline bonds — between the particles.

Crystalline bond caking usually results from temperature fluctuations during storage. When the stored material is cool, it induces condensation or water vapor accumulation from the warmer air, which dissolves the material. Then as the material is heated by the air, it drives off the water vapor, leaving solid crystalline bonds between particles. Typical materials that are subject to crystalline bond caking are sodium carbonate, salt, calcium chloride, and urea. Since the caked material's strength generally becomes greater with each temperature cycle, measuring the material's unconfined yield strength during several temperature cycles will help you identify this caking mechanism.

Results of sodium carbonate strength tests showing that the material's strength increased with the number of temperature cycles can be seen in Figure 3a. In the tests, the sodium carbonate was mixed with a *decahydrate* (a hydrate containing 10 water molecules), which served as the water source. Three tests were done with a mixture sample containing 4 percent decahydrate, and three with a sample containing 2 percent decahydrate. A 15-kilopascal consolidation stress was applied to the samples. For both samples, the unconfined yield strength increased with each temperature cycle, although the greater moisture content in the 4 percent decahydrate sample caused its unconfined yield strength to increase faster than that of the 2 percent sample. The crystalline bonds formed during temperature cycling are shown at right in Figure 3a.

**Cementation caking.** Cementation caking, as shown in Figure 2b, occurs when the stored material contains

enough moisture to mobilize ions in the particles. The ions in each particle then seek out and react with other chemicals in that particle or in the material-and-water mixture, forming a gel that builds chemical bonds between particles. In this case, the water in the stored material reacts chemically, changing the bulk material's structure and producing cementation caking. Typical materials subject

Figure 2

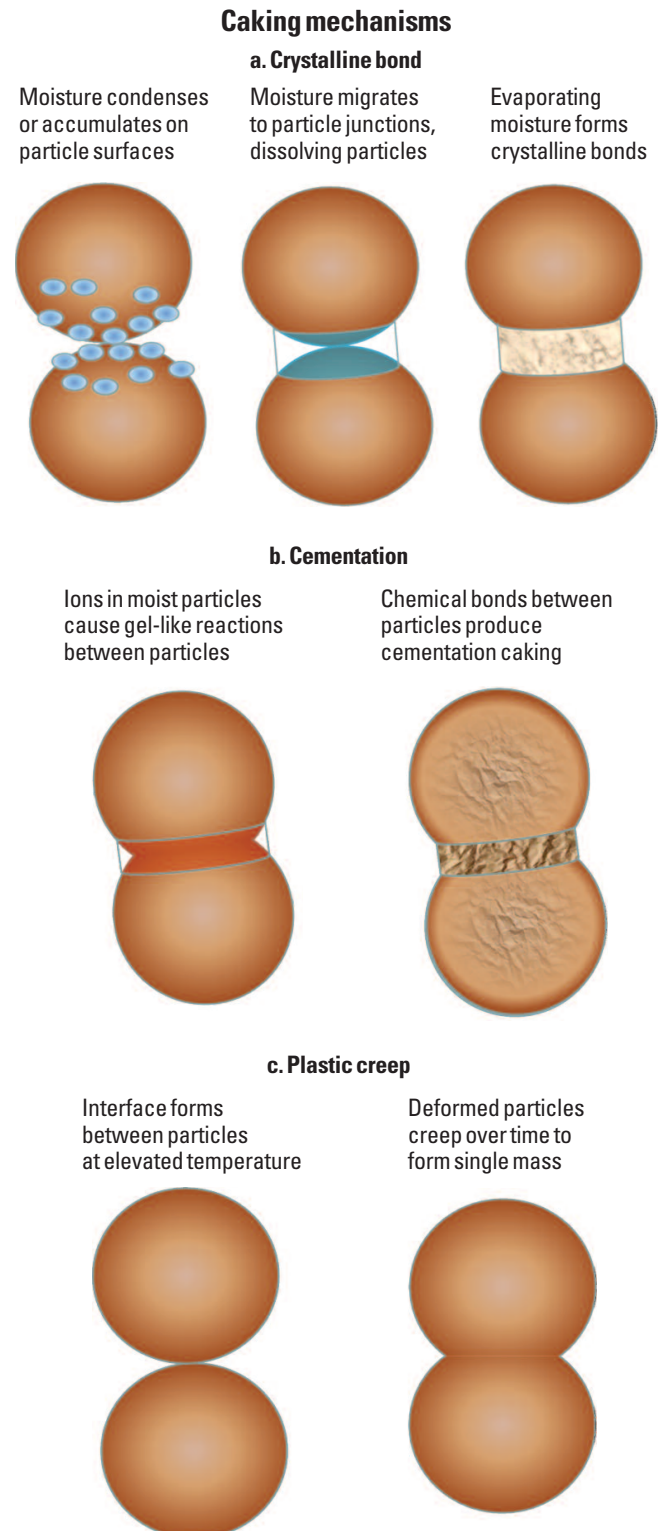
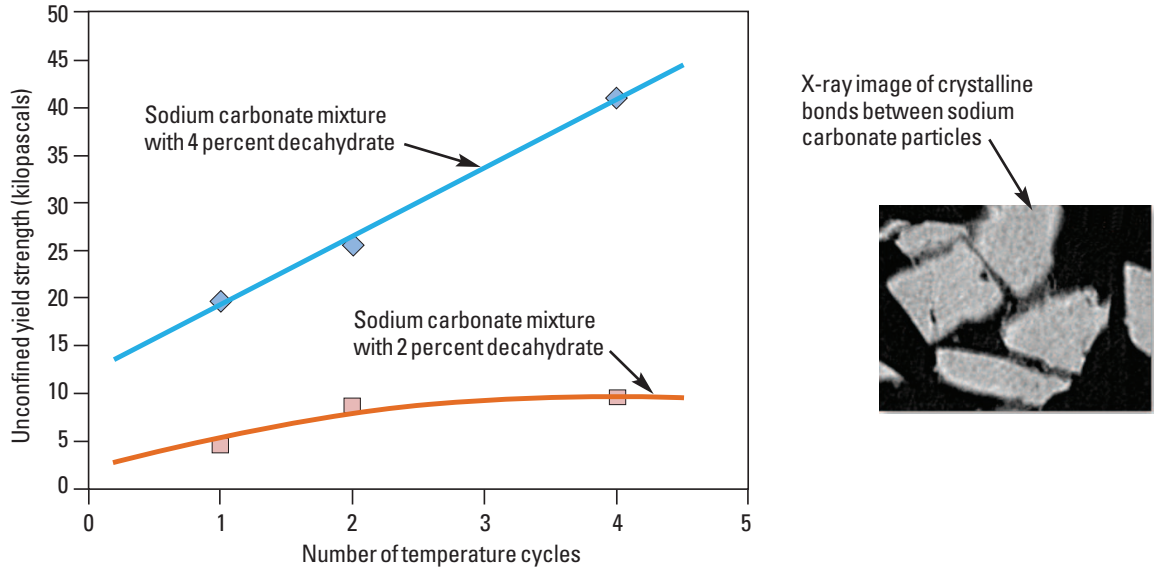


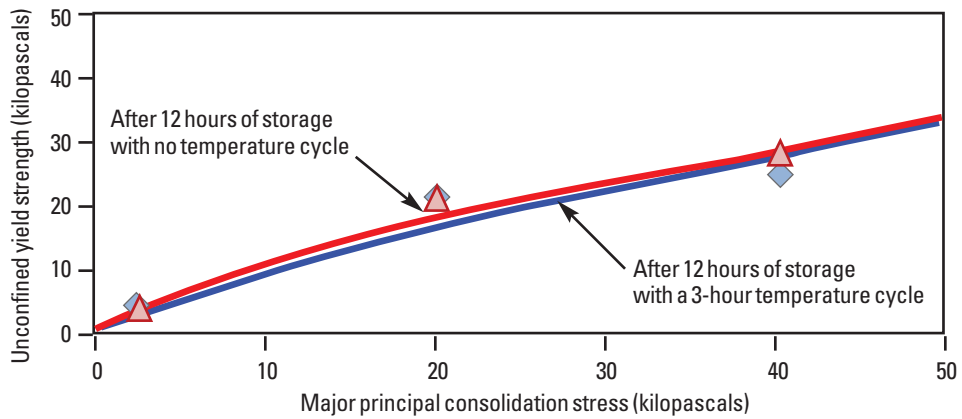
Figure 3

Unconfined yield strength tests identifying caking mechanisms

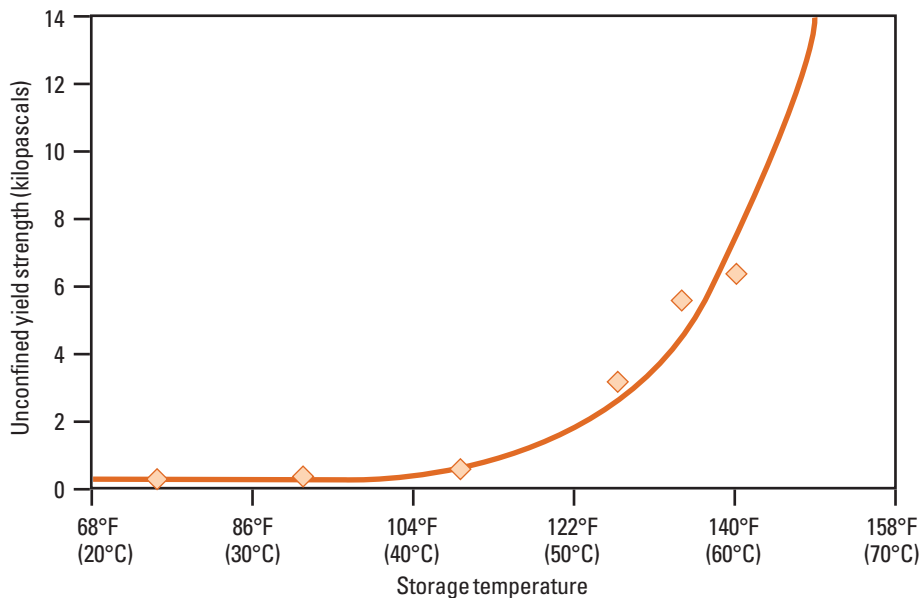
a. Crystalline bond caking of sodium carbonate identified by strength increases with more temperature cycles



b. Cementation caking of fly ash identified by equal strength increases with and without temperature cycling



c. Plastic creep caking of erucamide identified by dramatic strength increase at a critical temperature



to cementation caking include fly ash, Portland cement, gypsum, and some ceramics.

In general, a stored material can experience cementation caking in conditions *without* temperature cycling. Instead, a minimum energy is required to activate the ions. This energy is heat energy generated from an initial exothermic *hydrolysis* reaction (in which water breaks the chemical bond between molecules), and the resulting chemical reaction continues at a rate determined by the local concentration of reacting ions and the local temperature. This means that you can identify the caking mechanism as cementation when the measured material strength is the same both after temperature cycling and after being held at a constant temperature.

You can see this characteristic in the strength test results for fly ash shown in Figure 3b. In the tests, the unconfined yield strength of two fly ash samples was measured under three different consolidation stresses after 12 hours of storage, both with and without a 3-hour temperature cycle. The unconfined yield strength for each sample is the same at each consolidation stress, identifying the caking mechanism as cementation.

**Plastic creep caking.** One form of plastic creep caking, called *thermal creep*, affects materials that can soften at an elevated temperature. Such a material deforms (or creeps) over time when stored at a constant elevated temperature. An interface forms between adjacent particles and, if left long enough, the deformed particles creep and form a single mass, as shown in Figure 2c. Elastomer powder, plastic pellets, soy powder, erucamide powder (a slip agent for plastic films), and lactose powder are common examples of materials subject to thermal creep caking.

Another form of plastic creep occurs when a material sensitive to creep crosses its *glass transition temperature*. In this case, particles stored at an elevated temperature reach an amorphous state that allows the particles to deform, resulting in plastic creep. The glass transition temperature depends on the particle's moisture content, so changes in relative humidity can also initiate glass transition creep.

Neither type of plastic creep caking — thermal or glass transition — requires temperature cycling. However, the increased unconfined yield strength induced by plastic creep caking depends on the temperature difference between the material's softening temperature and the storage (or process) temperature, and the strength generally varies exponentially with this temperature difference. So if you measure your material's strength at several temperatures and the strength appears to increase dramatically at some critical temperature, thermal or glass transition creep caking may be the cause. You can see this characteristic in Figure 3c, where the strength test results for erucamide powder stored for 30 minutes at various temperatures

show a dramatic strength increase beginning at a stored temperature of about 104°F.

### Modifying your material to mitigate caking

You may be able to modify your material to prevent it from caking or reduce the amount of caking. How to change your material will depend on which caking mechanism it's subject to, the material's characteristics, the modification method's cost, and — when the material will be in a product for human consumption — whether the modified material will be toxic or harmful.

**Crystalline bond caking.** You can modify a soluble material subject to crystalline bond caking in several ways. You can coat the material with a moisture-barrier powder that will prevent the material from dissolving and significantly reduce the number of crystalline bonds formed between particles. Or you can add a surfactant to your material, which won't prevent crystallization but will limit the crystal size, thus reducing the strength of the crystalline bonds between particles. Or you can add a moisture scavenger (a drying agent) to your material, which will absorb water vapor and prevent the material from dissolving and forming crystalline bonds.

**Cementation caking.** Modifying a material subject to cementation caking usually requires keeping the material's moisture content *below* the minimum threshold for ion mobilization. In some cases, reducing the temperature at which you store or handle the material, in addition to reducing its moisture content, will slow or eliminate cementation caking. Because cementation reactions are also sensitive to pH levels, adding a powder base, acid, or buffer to the material — depending on your material's chemistry — can prevent extremely basic or acidic pH conditions from causing cementation caking.

---

*Preventing or reducing thermal or glass transition creep caking requires using a powder flow aid to coat your material's particles and keep their softened surfaces apart.*

---

**Plastic creep caking.** Preventing or reducing thermal or glass transition creep caking requires using a powder flow aid to coat your material's particles and keep their softened surfaces apart. The flow aid must have the right particle size to be effective. Flow aids with smaller-diameter particles than your material's particles can become fully embedded in very soft particle surfaces, resulting in little or no separation between the particles, while flow-aid particles with very large diameters may not provide enough surface coverage to prevent the material's soft particles from



touching during shear conditions (when the particles are moving during vessel filling or discharge). In some cases, changing the shape of the large-diameter flow-aid particles can improve coverage of the soft particle surfaces.

### Modifying your storage vessel or process to mitigate caking

You may also be able to modify your storage vessel or process to prevent or reduce caking. Which change will work for your application usually depends on which caking mechanism your material is subject to, but one change — reducing storage time — can mitigate any caking mechanism's effects.

**Reducing storage time.** Caking of any type always yields particles that are bonded or attached to each other by some means *after a certain storage time*. By measuring the increase in your material's unconfined yield strength after various storage times, you can deduce the characteristic bonding time for your material's caking mechanism. You can then design your storage vessel or process to prevent bonding by inducing interparticle motion (moving particles relative to each other) at a *time scale* — that is, storage time — less than that at which your material caked.

Generally, the time required for your material to cake (that is, the time constant) depends on the caking mechanism. Crystalline bond caking requires a temperature cycle, which typically occurs daily, so the time required for this form of caking is usually about 2 to 3 days. The time required for cementation caking can be much shorter — about 3 to 12 hours. Plastic creep (both thermal and glass transition) caking can occur in an even shorter time — as little as 15 minutes — but in some cases may occur only after 4 or more hours. With thermal creep caking, note that two time constants are usually in play: The first is the time it takes to move particles relative to each other during a compression event (such as filling a vessel with material, when force is applied to the material without causing shear), and the second is the time it takes for the particles to deform, which creates greater surface area for adhesion forces between particles. To prevent thermal creep caking, you'll need to measure both of these time constants for your material along with the material's compressibility.

**Designing your storage vessel's convergence geometry to promote interparticle motion.** To prevent areas of stagnant material that will cake, the particles in your vessel must move *relative to each other*. It may seem like using a mass-flow vessel design that produces first-in first-out flow should prevent caking, but this motion can actually promote caking. Material in a mass-flow vessel moves in *plug flow* — all particles move together at a uniform velocity and maintain contact with adjacent particles, with no interparticle motion except at the vessel wall. As a result, if your material shows caking tendencies, you should avoid using any vessel with a tall cylinder section, which

tends to promote perfect plug flow and can cause such a material to cake.

To promote interparticle motion, you don't need to completely empty and refill your vessel. The vessel's cone section must simply have enough convergence or appropriate internal inserts to promote interparticle motion. (This principle applies to both funnel- and mass-flow vessel designs.) The amount of material in motion in your vessel depends on the size of the largest region between converging walls, and the amount of convergence your vessel can have depends on your material's compressibility, which you can measure by measuring the material's bulk density as a function of stress applied to it. [**Editor's note:** For more information on bulk density measurements, see the later "For further reading" section or contact the author.] To promote interparticle motion and prevent caking, the strain induced by passing your material through your cone section's converging geometry must simply be greater than the strain induced by compressing the material.

**Using flow-aid devices.** Using flow-aid devices like air blasters, live-bottom screws, and vibrating bin bottoms to prevent caking works only about half the time. While an air blaster has enough force to break up lightly caked material, the blaster's sphere of influence in the caked material is only from 2.6 to 4.3 feet, depending on the material's characteristics. While the device typically causes significant interparticle motion near the blaster inlet, it causes very little motion farther away.

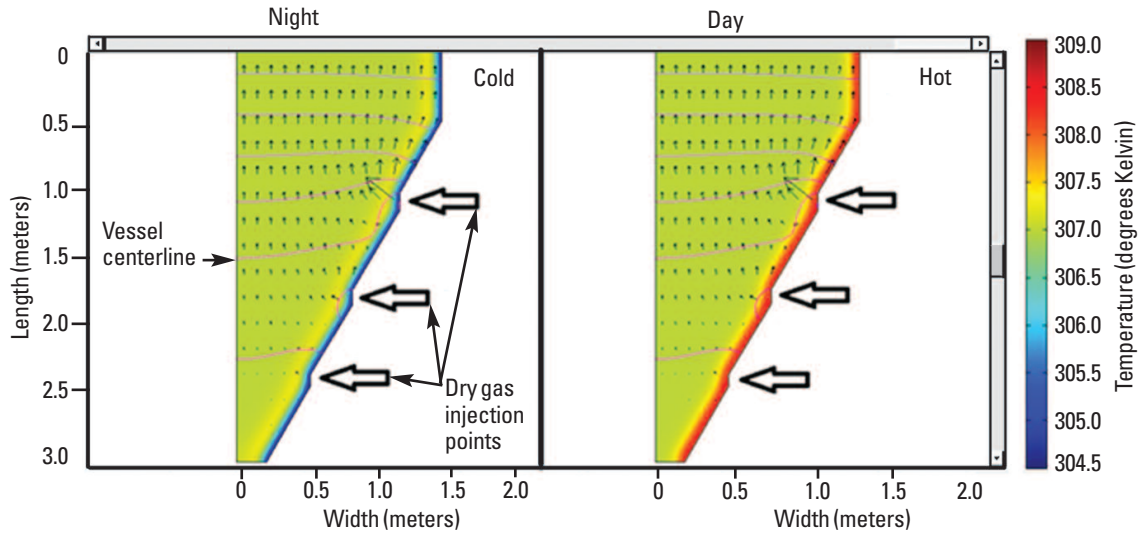
A vessel with live-bottom screws can be effective for mitigating a moderate caking problem. In this case, the material flows uniformly downward in the vessel until it reaches the screws, which induce a large amount of strain on the flow as they carry material out of the vessel. To prevent stagnant regions from forming during discharge, the screws must withdraw material across their entire length.

A vibrating bin bottom doesn't typically induce enough interparticle motion to prevent caking, but when properly designed, it can sometimes induce mass flow and reduce caking. *Note:* Don't use a dished-head vibrating bin bottom if your material tends to cake, because a stagnant region will form in the dished head and make mass flow impossible.

**Using dry gas injection to prevent crystalline bond caking.** If your material is subject to crystalline bond caking, any procedure that disrupts its crystal growth rate will mitigate caking. Crystalline bond caking requires the material's exposure to humid air, which causes moisture to condense on the particle surfaces. Injecting a dry gas into the vessel removes the moisture and prevents condensation, thus reducing caking. Dry gas injection could be the topic of an entire article, but here's a brief look at how you can apply this technique:

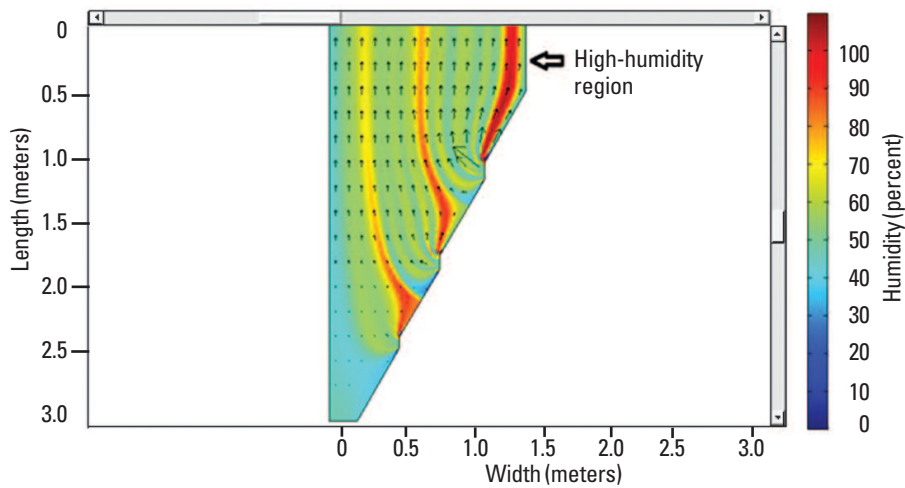
## Figure 4

## FEM analysis for determining placement of dry gas injection points in vessel to prevent caking

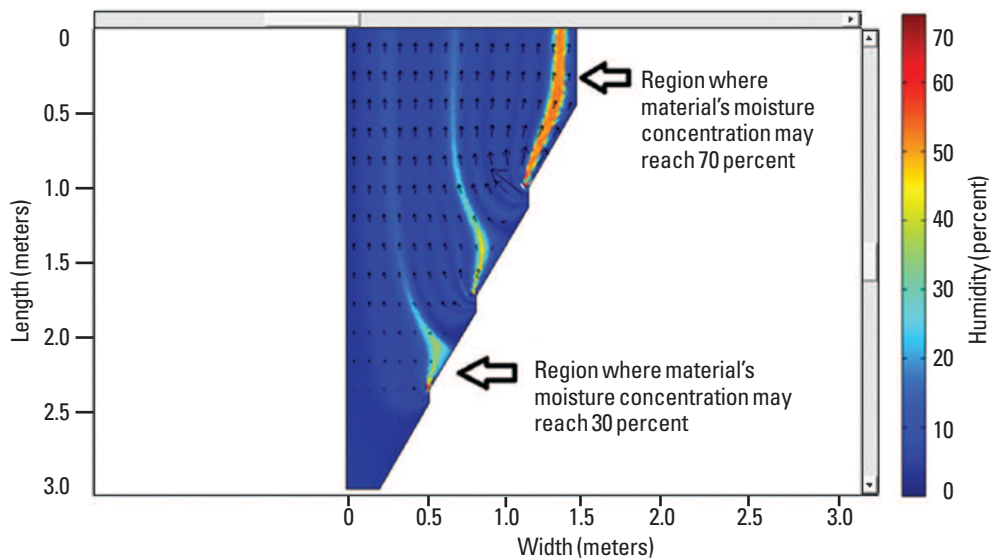
a. Expected temperature profile of material in vessel during night–day temperature cycle<sup>a</sup>

<sup>a</sup>Note: The vertical arrows within the vessel show the gas flow direction; the larger the arrow, the higher the gas velocity.

## b. Expected humidity profile of material



## c. Expected material moisture concentration



First, measure the equilibrium moisture concentration that your material can absorb as a function of the relative humidity it's exposed to. You can do this with a *moisture sorption rate test*, in which the material's rate of weight change (representing moisture absorption) is monitored during exposure to humid air. [*Editor's note:* For more information about this test method, see the later "For further reading" section or contact the author.] The resulting data allows you to compute your material's moisture sorption rate and final equilibrium moisture concentration.

Next, use this data and a process description to generate a finite-element model (FEM) that shows how gas flows through your bulk material.<sup>2</sup> The gas-flow FEM is generated based on data from measuring your material's permeability as a function of stress level. By coupling this gas-flow FEM with a heat-transfer FEM (based on measuring your material's thermal conductivity as a function of stress level) and using the data from your moisture sorption rate test, you can generate a detailed map of moisture flow in your vessel. This map will show which vessel zones have relative humidity values large enough to cause caking. Then, by computing your material's moisture content, you can determine whether it's high enough to cause crystalline bond caking. The results will help you determine where to inject a dry gas into the vessel to mitigate caking.

Consider the FEM analysis shown in Figure 4 for a vessel with a cylinder section, a cone section, and three gas injection points along the cone section's sloping surface. The vessel's outer wall temperature goes through a night-day cycle and is lower at night than during the day, as shown in Figure 4a. Next, the FEM analysis computed the expected humidity profile in the vessel (Figure 4b) based on the injected gas's 50 percent relative humidity, revealing a region with very high humidity above each gas injection point. Each region — especially above the highest injection point — has the potential for crystalline bond caking. The analysis then calculated the material's moisture concentration, which revealed that this concentration is the largest — 70 percent — in the highest region (Figure 4c), indicating that the potential for caking is very high here. Thus, placing another gas injection port at the point where the cylinder meets the cone will reduce the material's moisture concentration enough to prevent crystalline bond caking in this region.

***Maintaining constant temperature to prevent plastic creep caking.*** If your material is caking at some point along the vessel wall as a result of thermal (or glass transition) creep, the temperature at this point becomes high enough during the night-day temperature cycle to cause particles to soften and deform. In this case, you can insulate the vessel or install heat tracing on the vessel to keep the temperature at a relatively constant value below your material's softening point.

**PBE**

## References

1. After one time constant, the material's strength reaches 63 percent of its full value; after two time constants, it reaches 95 percent of the full value.
2. You can contract an independent testing firm to perform this analysis if you don't have access to appropriate test facilities or FEM software.

## For further reading

Find more information on solving material flow problems in articles listed under "Solids flow," "Storage," and "Particle analysis" in *Powder and Bulk Engineering's* article index in the December 2013 issue or the Article Archive on PBE's website, [www.powderbulk.com](http://www.powderbulk.com). (All articles listed in the archive are available for free download to registered users.) You can also find books and webinars on this topic in the website Store.

---

**Kerry Johanson** is chief operations officer at *Material Flow Solutions Inc.* (352-379-8879, [matflowsol@bellsouth.net](mailto:matflowsol@bellsouth.net)). He holds a PhD in chemical engineering from Brigham Young University in Provo, Utah.

**Material Flow Solutions Inc.**

**Gainesville, FL**

**352-379-8879**

**[www.matflowsol.com](http://www.matflowsol.com)**