

# Preventing moisture caking, the unwanted agglomeration

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**Moisture caking occurs in many materials, particularly when being stored or transported. This article will describe characterization tests that can determine whether a material will cake due to moisture, and will discuss possible post-process solutions for preventing moisture caking during storage and transportation.**

Agglomeration is a natural phenomenon. It happens under certain conditions whether you want it to or not. Unwanted agglomeration, typically called *caking*, refers to the state of a bulk solid material that has lumped or agglomerated because strong bonds have formed between the material's particles. Often caking occurs to an extent that it causes serious operating difficulties and customer complaints.<sup>1</sup>

For instance, do any of these complaints sound familiar? "Sometimes the product flows well and at other times it doesn't. Your competitor's product *always* flows well." "Some bulk bags are caked so hard that we can't unload the material from the bags. We have to break it up by ramming the bags against a wall." "Some bulk containers are taking excessive time to unload. This is costing us too much money." "Some packages that leave the plant are full of lumps. They need to be reground at the customer's site, and we have to pay for this." "Some batches are full of agglomerates that don't dissolve."

Bear in mind that your customer will see your caked material as a poor-quality product even if you're using the most advanced technology to produce it. If your customers can

find a supplier that can provide the same material in a more desirable form, they'll turn to that supplier.

## Caking mechanisms

To solve a caking problem, you first have to understand caking mechanisms. Then you can modify your process, storage, or transportation practices to prevent or remedy the undesired agglomeration.

The main mechanisms that can cause particles in a material to bond, resulting in caking, are mechanical interlocking, plastic-flow bonding, electrostatic charging, physio-chemical bonding (which includes moisture caking), and a combination of these.<sup>2</sup>

Your material's particle size, particle size distribution, and particle shape play important roles with any of these mechanisms. The potential for caking increases with increased particle-to-particle contact. Therefore, in general, the smaller the particle size and the less uniform the particle shape, the more likely the material will cake.

## Moisture caking in the bulk solids industries

Moisture caking, which is one form of physio-chemical bonding, is the most common caking mechanism in the bulk solids industries. For this reason, this article will concentrate only on moisture caking. Moisture caking particularly affects foodstuffs and crystalline powder systems, such as salts, and involves dissolution and recrystallization, solid-solid phase transitions, and sublimation. [*Editor's note:* Find more information about these in reference 2.]

**How it happens.** In moisture caking, liquid interparticle bridges (with some moisture-related particle surface dis-

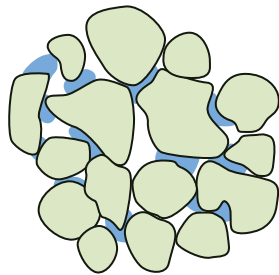
solution) form and subsequently solidify under dry conditions. Storage conditions, including humidity, temperature, time, and consolidation pressures, also influence moisture caking. Moisture or water can also act as a plasticizer, softening the particles and allowing them to deform and come into closer proximity, which increases their bonding strength.

The moisture that causes caking comes from various sources, including direct entry from the upstream process, the material's inherent moisture, and ambient humidity.<sup>3</sup> At high *water activity* levels (normally, when the relative humidity is above 60 percent), liquid bridges begin to form between the particles due to *capillary condensation*, as shown in Figure 1a. As more moisture becomes available, liquid bridges form at all contact points between the particles, causing agglomeration (Figure 1b). When the material becomes fully saturated, all voids or porous spaces between the particles are filled with water, and lumps form

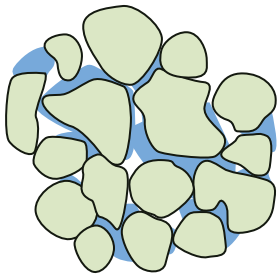
## Figure 1

### Moisture caking stages

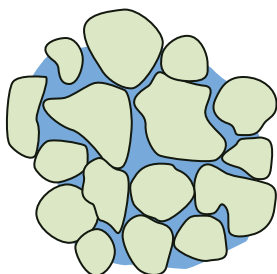
#### a. Liquid bridges beginning to form



#### b. Liquid bridges forming at all particle contact points, causing agglomeration



#### c. Liquid filling all voids or porous spaces between particles, creating lumps



(Figure 1c). If the liquid bridges contain soluble matter and if the liquid is subsequently removed by drying or *moisture migration* (discussed in the next subsection), then strong solid bridges form, causing severe moisture caking and lumps, as shown in Figure 2.

**The problem of moisture migration.** Moisture migration caused by uneven water concentration, called a *water concentration gradient*, can produce localized areas of high water activity in a packed material bed, causing caking.<sup>4</sup> The gradient may occur between the material stored in the container and the atmospheric air in contact with the container's surface (for example, when a moisture-permeable bag is used) and between air in the container's head space and the stored material.

Unfortunately, having a perfectly moisture-impermeable container isn't a guarantee that relatively high water concentrations won't occur inside the packed bed. Temperature gradients during packaging can also induce moisture migration and caking.<sup>5</sup> This is better explained with an example: Consider a crystalline powder or salt that leaves a dryer at about 140°F (60°C) and is placed in a bulk bag with surfaces that are exposed to a cooler temperature, such as the warehouse's ambient temperature. Initially, the air surrounding the particles in the container will be near the air's equilibrium relative humidity. While the surrounding air's absolute humidity is initially constant throughout, its relative humidity near the cooler bag walls will become higher as the air temperature drops. The air's moisture will condense and adsorb onto the particles nearest the wall, causing liquid bridging. The absolute humidity near the wall will then be reduced, and the moisture will migrate from the bulk bag's or container's center to the wall.

At the same time, the transport of moisture away from the material bed's hotter regions will cause moisture desorp-

## Figure 2

### Lumps caused by severe moisture caking, collected from a bulk bag after shipping



tion from the material, further facilitating moisture transport. This process will continue until the temperature and moisture reach equilibrium. If this process is subsequently reversed (that is, the ambient temperature becomes hotter than the packed material), solid bridges can form near the bag surface, causing caking.

When a material is packed and transported, it's often subject to daily and nightly temperature swings. Even when stored in moisture-impermeable containers, material can still cake as moisture is drawn to the surface and evaporates when the material is warm and then condenses when the material is cooled.

### Caking characterization techniques and measurement

You can determine how likely your material is to cake by performing bench-scale tests on representative samples of the material under expected processing, storage, and transport conditions. You can conduct *sorption tests* to quantify the material's interaction with ambient humidity and *shear*

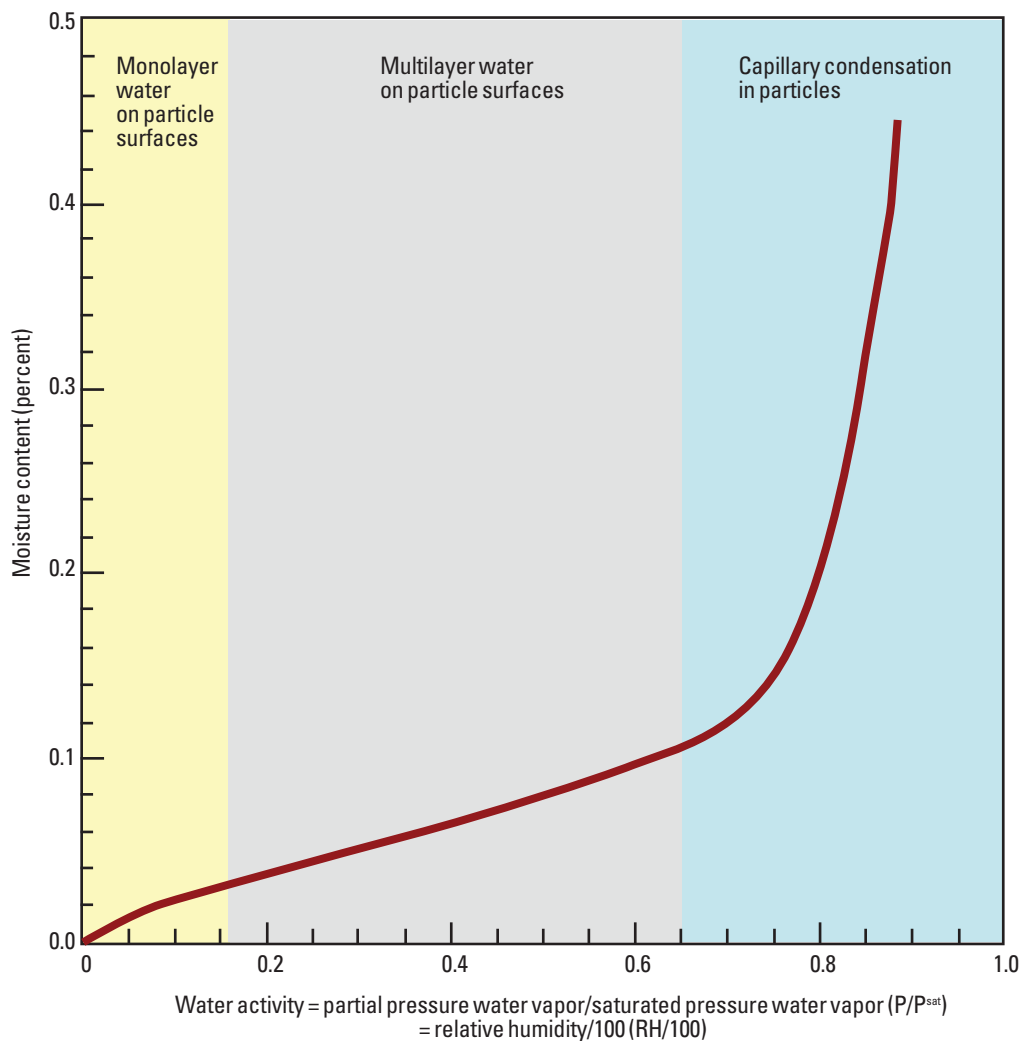
*tests* to determine how likely the material is to cake under controlled temperature, humidity, and consolidation levels<sup>6</sup>.

**Sorption tests.** You can quantify ambient humidity's effect on your material's moisture uptake by conducting a series of sorption tests using a microbalance inside a chamber at controlled temperature and humidity conditions and recording the results on a curve called a *sorption isotherm*. In the tests, small samples of the material are placed on the microbalance and exposed to a range of relative humidities at a constant temperature, starting with near 0 percent relative humidity. Sufficient time is allowed to attain equilibrium between the air and the particles. The change in the material's weight at each step is recorded, representing the equilibrium moisture content as a function of the relative humidity in equilibrium with the material.

The shape of the sorption isotherm contains useful information on the type of interaction that occurs between the material and ambient humidity. The isotherm in Figure 3 shows the typical behavior of a foodstuff or crystalline ma-

Figure 3

### Sorption isotherm



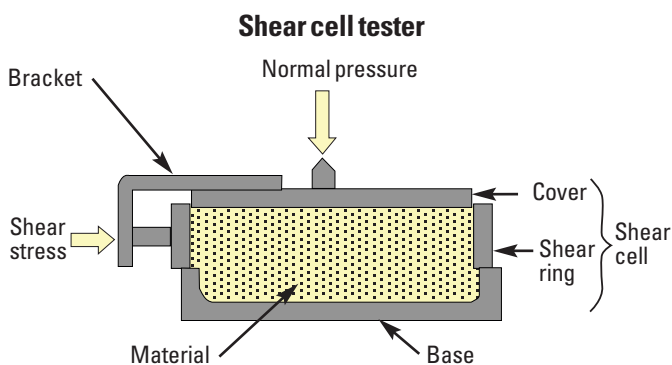
material that's prone to caking due to moisture migration. This isotherm shape indicates that only small amounts of moisture need to be redistributed in the packed material to yield localized regions of high water activity, where liquid bridges begin to form.

**Shear tests.** In addition to humidity, moisture caking is influenced by consolidation pressures during storage, along with the ambient temperature, relative humidity, and time. When testing your material, your test method must provide good control of these critical variables and must be quantitative. Because caking can be defined by the gain in a material's cohesive strength when held at consolidating pressures for a prescribed period of time, a shear test is ideal for testing what consolidation pressures cause your material to cake under certain ambient conditions: The test applies consolidation pressures that simulate those you expect when your material is stored. The test apparatus, called a *shear cell tester*, is shown in Figure 4. The tester measures your material's cohesive strength as consolidation pressure is applied at controlled levels to the material.

You can apply the shear test results with confidence, since tests conducted with small samples of your material will replicate conditions expected for large scales.

As an example, to determine if your material will cake in a bag after it's been stored for a week, you can measure the cohesive strength at consolidating pressures that correspond to those in the bag at continuous flow conditions and after a week of time at rest.<sup>6</sup> You can also control the sample's temperature and the ambient relative humidity. To simulate the consolidation pressures during storage, you place samples of your material in cells and load each into a consolidation bench, as shown in Figure 5, under a static load for the prescribed period of time and ambient conditions. After the time expires, you place the cells in the tester, which shears them to determine the material's gain in cohesive strength compared to its strength under continuous flow conditions.

**Figure 4**



## Preventing moisture caking during storage and transportation

Sorption and shear tests can give you a good understanding of your material's caking behavior under storage conditions so you can develop strategies to prevent moisture caking. For example, the sorption test results provide insights into the material's moisture uptake as a function of humidity and temperature, so you can use this information to specify ambient conditions in your warehouse, containers, or process. The shear test results can help you identify your material's caking sensitivity to storage conditions, so you can minimize caking by controlling variables such as material moisture content and ambient humidity and temperature, time, and consolidation pressures during storage.

Unfortunately, to prevent or remedy moisture caking problems, running these tests alone may not be sufficient. This is especially true when moisture migration resulting from temperature gradients during storage in your warehouse or during transportation causes the moisture caking. What's more, the simple solution of implementing air control systems in the warehouse or using air-conditioned containers sometimes isn't economically feasible.

In these cases, you can use published mathematical models<sup>4,7,8</sup> to simulate moisture diffusion inside packed material beds. These models can help you predict the temperature gradient needed to exceed a specified water activity (readily identified during bench-scale testing) at a cold surface for a given initial water activity and tempera-

**Figure 5**

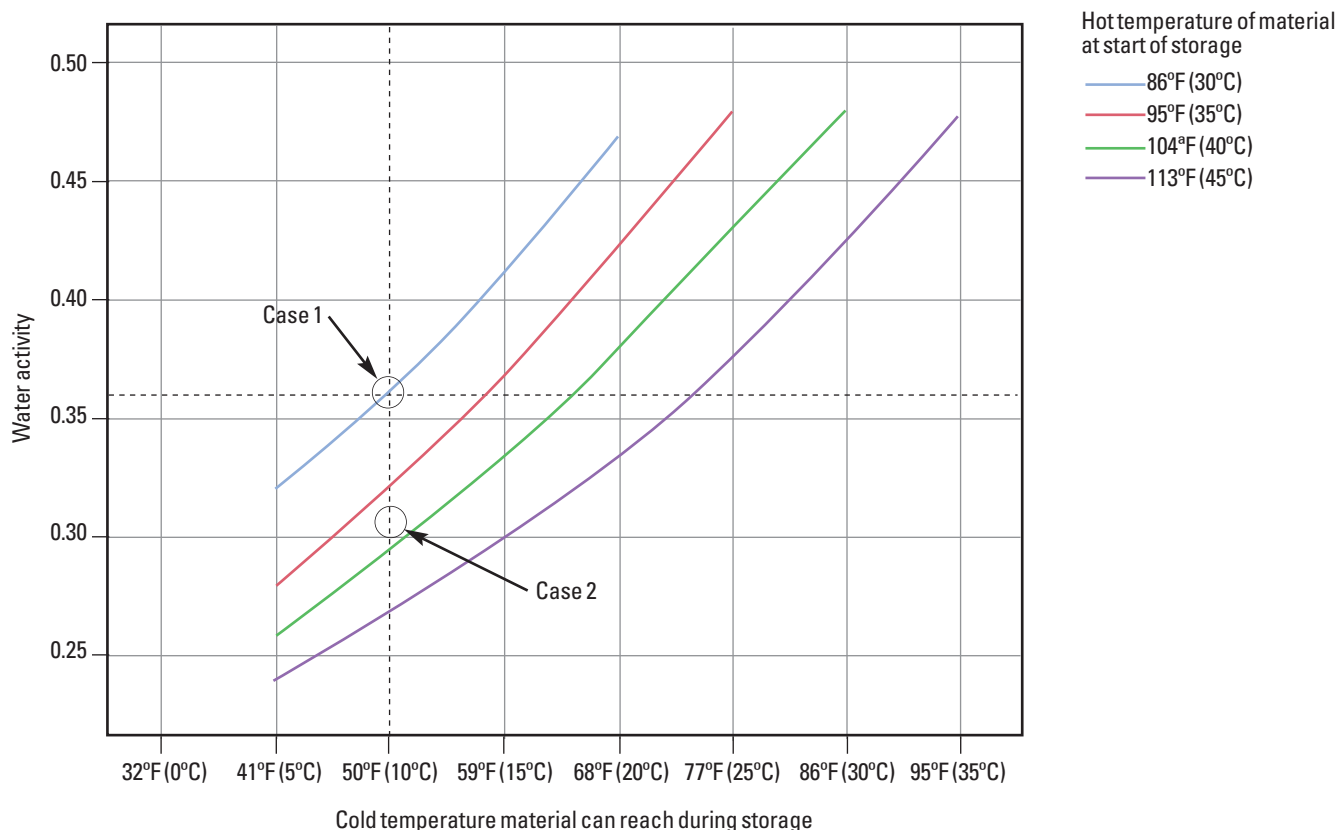
### Consolidation bench





Figure 6

**Moisture migration model for a crystalline powder showing where it reaches 60 percent relative humidity after various process stages**



ture of the packed material. (Be aware that you'll need to validate these models with your material.)

Figure 6 shows the results of running a moisture migration model for a crystalline powder that caked. Each curve represents the material's temperature gradient (from hot to cold) during storage after exiting a different process stage. To use the graph, you note the water activity at the point where each curve intersects the cold temperature, then apply the water activity measurement in the sorption isotherm to find the corresponding material moisture content. This tells you the maximum moisture the material should contain when stored at the noted cold temperature to avoid moisture caking. You can use results like these either for determining whether your material has been dried enough to prevent caking during your process's cooling stage or inside your silos prior to packing, or to prevent caking inside the bags during transportation.

Here are two examples of how the graph in Figure 6 might be used:

**Case 1:** If the material's temperature at the packaging stage is 86°F (30°C) and the temperature of the warehouse (or the

shipping containers where the bags are stored) is 50°F (10°C), then the material must be dried to a maximum water activity of 0.36 (corresponding to a specific moisture content taken from the sorption isotherm) before it goes into the bag to prevent caking during the cooling cycle.

**Case 2:** If the water activity is 0.3 and the minimum temperature reached during shipping is 50°F (10°C), then, to avoid caking, the material must be cooled to 100°F (38°C) before it's bagged.

### A final word

Moisture caking is a complicated phenomenon. Using bench-scale testing, along with validated moisture-migration models, can help you prevent caking by identifying acceptable ranges for critical process variables. **PBE**

### References

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### For further reading

Find more information on this topic in articles listed under "Storage" and "Solids flow" in *Powder and Bulk Engineering's* comprehensive article index (in the December 2013 issue and at *PBE's* website, [www.powderbulk.com](http://www.powderbulk.com)) and in books available on the website at the *PBE* Bookstore. You can also purchase copies of past *PBE* articles at [www.powderbulk.com](http://www.powderbulk.com).

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