

What Causes Chemical Salts to Cake or Harden and Free Flowing Solutions

by Mark Mans



One concern in industry is the caking of chemical salts.

Caking, or time-induced consolidation, is the agglomeration of free-flowing particles into a large singular mass. These masses are difficult to discharge from the packaging, often requiring a "brute force" approach from operators, which may result in unproductive manufacturing time and increased risk of injury. Further, caked material may require on-site crushing or milling prior to use, since the particle size of the material may no longer satisfy the customer's specifications or be compatible with their equipment.

Knowledge of the mechanism by which caking occurs is critical to preventing these processing pitfalls. In general, for material to cake, the adhesive forces between particles must increase. For water-soluble materials, such as those produced by Jost Chemical, the formation of material bridges provides an increased adhesion between the particles.

Material bridges are the result of the following process:

- **1** Water molecules are present in the product either as humidity inside the packaging, as residual surface moisture on the product or as waters of hydration.
- 2 These water molecules gather on the surface of a particle, either as a result of a temperature gradient induced moisture migration or simple diffusion of water from the interior of a particle to the exterior of a particle.
- 3 The surface of the particle dissolves in this water, forming a salt solution.
- 4 These salt solutions form liquid bridges between neighboring particles.

- 5 The water in the liquid bridges evaporates resulting in crystallization of the dissolved material.
- 6 The liquid bridge becomes a solid material bridge. What was once multiple discrete neighboring particles has now become a solid network of inter-connected particles.

The formation of material bridges can be influenced by the material's particle size, particle shape, and particle size distribution. Since the potential for caking increases with increased particle-to-particle contact and smaller particles have more particle-to-particle contact, milling an already troublesome product will exacerbate a caking problem.

It is a common misconception that caking is simply the result of an increase in overall moisture content. Although an increase in overall moisture content is one means of forming material bridges, it is most often the insignificant quantity of moisture present in the material which leads to caking. It has been demonstrated that moisture levels as low as 0.05% have been sufficient to induce caking.

The reason an increase in moisture content is not required for caking, is due to the moisture inherently present in the product, which is readily susceptible to temperature gradient induced moisture migration (TGIMM). When a section of a package warms, the local temperature increases as local relative humidity (RH) decreases. The decrease in RH encourages localized desorption of water from the material. The initially static and restricted water is now mobile water vapor, free to move throughout the package. Eventually the The formation of material bridges can be influenced by the material's particle size, particle shape, and particle size distribution.

water vapor diffuses to a section of the package that exhibits a cooler localized temperature. The migration of this extra water vapor causes an increase in the local RH and eventually condensation of the water vapor back to liquid water on the particle surfaces.

TGIMM can result in the movement of significant amounts of water throughout a package. Temperature gradients can exist for a variety of reasons. For example, material packaged while still hot, followed by cooling in a warehouse may result in temperature gradients. Further, packages of product can be routinely subjected to temperature fluctuations during transport. The natural cooling and warming cycles throughout a 24 hour day can establish temperature gradients within the product packaging, since the outside of the container cools or warms before the center of the container. Therefore, free-flowing materials, even when stored in hermetically sealed containers, may still cake if the containers are exposed to temperature gradients large enough to induce moisture migration.

Packaging systems designed to exclude all outside moisture during transport are great at protecting highly hygroscopic materials, but they offer little to no protection from TGIMM inside the package, and thus cannot eliminate caking.

A common industry approach to prevent caking is with the use of anti-caking agents. However the presence of an anti-caking agent is often undesirable to the end user. A key issue is that any material containing anti-caking agents is no longer a single, pure entity. As such it is considered adulterated and therefore cannot be labeled as USP, EP, etc. Jost Chemical has addressed a number of caking issues without incorporating anti-caking agents. Utilizing our expert knowledge of product chemistry and climate controlled packaging suites, Jost Chemical prevents temperature gradients during packaging.

Although the mechanism described above is present in each caking problem, each case is also unique. In some instances, modifying the crystallization conditions can result in a material which remains free-flowing. In other instances, a better approach is to change the physical features of the product. Jost Chemical has a number of particle size modification technologies (i.e. granulation and roller compaction) which keep materials free flowing. Jost Chemical's technology can balance the need for free-flowing material with the need for a specific material performance such as dissolution rate.

Lastly, in particularly difficult instances where material is prone to caking, such as a freeflowing, finely milled product, Jost Chemical can employ special packaging procedures and production schedules so the product is manufactured, shipped, and delivered before excessive caking occurs.

Jost Chemical Co. is a leading manufacturer of high purity chemical salts for use in nutritional formulations, pharmaceutical applications, food, consumer products, and other specialty applications. Many of these products are produced to meet US Pharmacopeia (USP), Food Chemical Codex (FCC), European Pharmacopeia (EP) or American Chemical Society (ACS) compendia monographs. Recognized for its commitment to customers, Jost's team of scientists provide technical assistance with regards to performance, formulation, regulatory and quality of our broad product line.

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