Basics of Powder Caking

Defining caking

Caking occurs when a powder that was uniform and flowable when exiting the dryer, becomes lumpy or even brick-like upon storage (Figure 1).

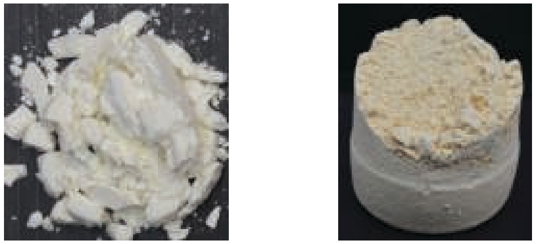

Figure 1. Slightly caked powder and caked powder that has become brick-like.

Caking is not the same as the sticking to surfaces and clumping that occurs during drying of thermoplastic materials (Figure 2) although some dairy powders can have both problems.

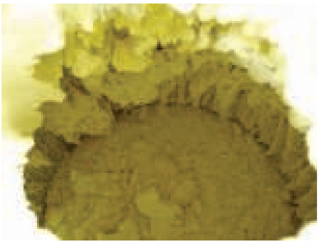

Figure 2. Themoplastic powder adhering to dryer container walls

Cause of caking

Components in the powder that are hygroscopic or water loving cause caking. Hygroscopic components have the ability to attract and absorb water. Proteins, minerals and certain forms of lactose are the hygroscopic components in dairy powders. While proteins are hygroscopic they do not cause caking because they can bind water without becoming sticky. Binding of water by minerals is only an issue at very high moisture contents that are not seen with dairy powders. Some forms of lactose, however, are very hygroscopic leading to issues with caking.

Forms of lactose and affinity for water

Lactose comes in several forms: α-hydrate; β-lactose; α-anhydrous (stable and unstable) and; amorphous. Conditions that allow the formation of different forms of lactose are given in Figure 3. When discussing caking only α-hydrate and amorphous forms of lactose are important.

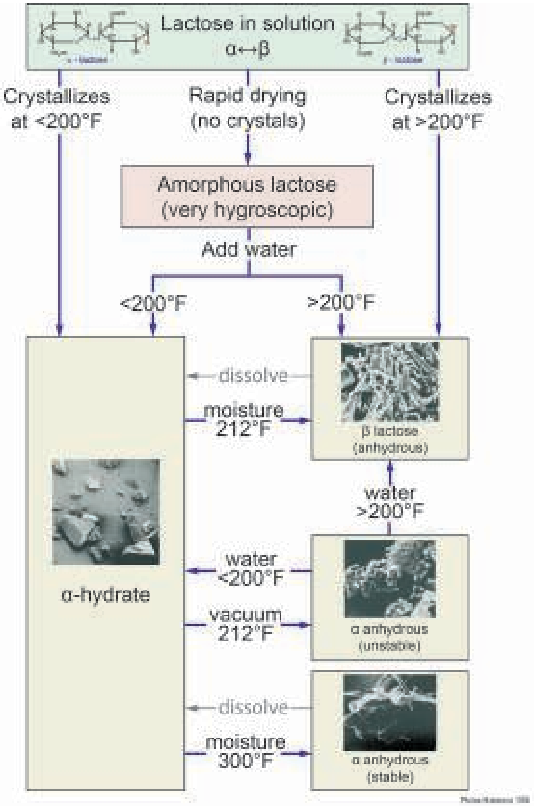

Figure 3. Forms of lactose and conditions of their formation.

Alpha-lactose has one molecule of water built into the crystal and is very stable and not subject to caking. Amorphous lactose, by contrast, does not contain water and is thermodynamically unstable. The terms amorphous and glass are used interchangeably and mean “without structure”. The differences between the structures of α-lactose and amorphous lactose is given in Figure 4. Amorphous lactose appears to be a solid but structurally is a liquid. Water is needed for amorphous lactose to form the stable, non-hygroscopic α-lactose crystal.

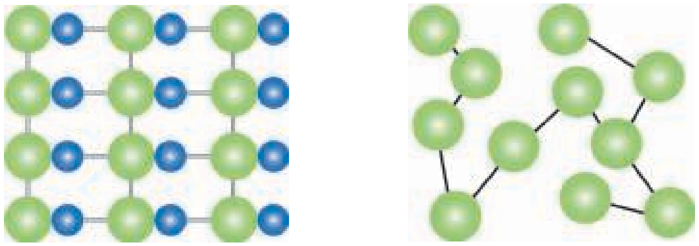

Figure 4. Crystalline α-lactose with associated water molecule and amorphous lactose

Method of caking by amorphous lactose

Amorphous lactose attracts water both from the atmosphere and within the package. Water allows the amorphous lactose to become more mobile such that the molecules can start to align with each other. Some of the amorphous lactose may find enough water that it will crystallize into the α-lactose monohydrate form. Although the conversion of amorphous lactose to α-lactose monohydrate typically is desired, the process can cause problems when it occurs in powders. The remaining amorphous lactose becomes "sticky" because of the presence of water thereby causing the powder to clump together (Figure 5).

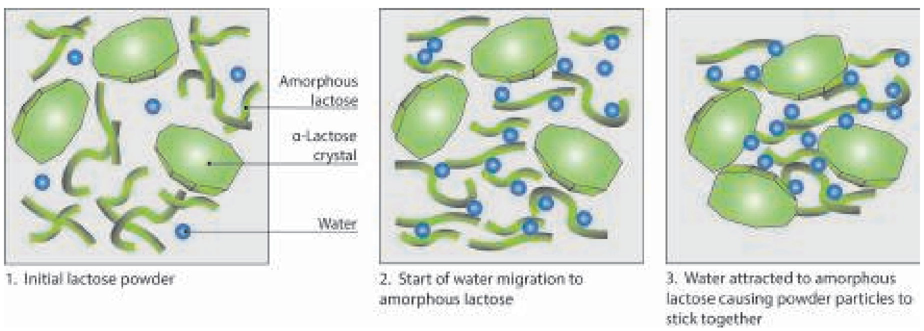

Figure 5. Process of lactose caking

The process changes when protein is present. Protein and amorphous lactose both attract water. Because protein also is attracting water, there is less water available for mobilizing and crystallizing the amorphous lactose. Protein does not become sticky when binding water and since less water is associated with the amorphous lactose, powders containing protein, such as nonfat dry milk (NFDM) and whey protein concentrates (WPC) will exhibit less caking as compared to powders without protein (Figure 6).

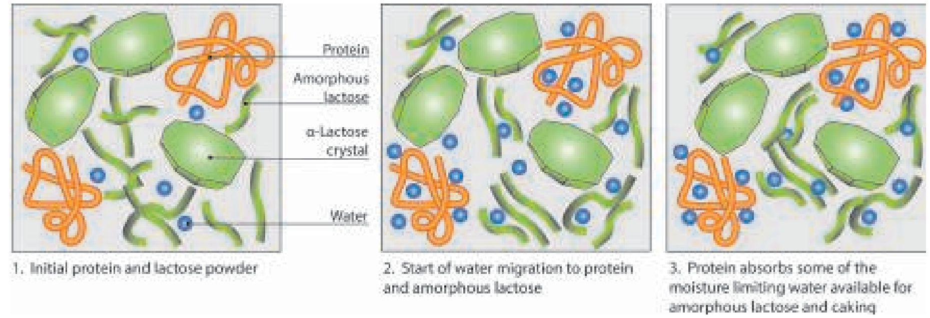

Figure 6. Process of lactose caking in the presence of protein

Determining ability to cake

There are several ways to determine if a powder will cake over time. Some tests are more reliable than others as discussed below.

<u>Moisture</u> - Lactose has both free and bound water. Bound water is part of the α-lactose crystal structure while free water is not associated with the crystal. Together bound and free water make up total moisture or water. Alpha-lactose monohydrate has 5% water built into the crystal based on the molecular weight of lactose and water.

lactose + water = α-lactose monohydrate crystal
342 + 18 = 360 (molecular weights)
95% + 5% = 100%

Depending on the analytical method used, one or both types of water are measured. It is free water that is of concern with caking and product standards.

Loss on drying, typically done with a vacuum oven, measures free water. Standards of identity require free moisture to be less than 1%, however, depending on the type of lactose produced, a loss on drying of less than 0.5% is typical.

The Karl Fischer test measures total moisture. Since bound water is assumed to be 5% of the α-lactose crystal structure, anything above 5% moisture by Karl Fischer would be considered to be free moisture. For example, if Karl Fischer test gives 5.3% moisture for a sample then 5% would be bound water and 0.3% free moisture.

An example of moisture in lactose would be:
Bound + Free = Total moisture
5% + 0.3% = 5.3%

However, not all of the crystallized lactose is in the form of α-lactose monohydrate. Phosphate, which is present in milk, whey, permeate, etc. and often is associated with calcium can combine with lactose to form lactose 1-phosphate. Lactose 1-phosphate does not contain water as part of its structure and can build into the faces of the α-lactose monohydrate crystal. In addition, it may be possible that some β-lactose crystallizes along with the α-lactose monohydrate despite conditions not favoring β-lactose formation. Although crystallized lactose is primarily α-lactose monohydrate some lactose will be in the amorphous form. The exact ratios of α-lactose monohydrate to amorphous lactose depend on the method of production; however, it is possible that 3 to 5% of the lactose is in the amorphous form. Typically more than 90% of the lactose is in the α-monohydrate form. The presence of lactose 1-phosphate, β-lactose, amorphous lactose and minerals, which do not contain water in their structure, has the effect of reducing the expected amount of bound water from 5% to approximately 4.6%.

There are several limitations to the use of moisture for determining susceptibility to caking.

- If free moisture is high then there is a problem, however, if the value is "correct" then there may or may not be a problem with caking.
- There is very little free moisture in lactose therefore small inaccuracies in measurement can cause very different values. Quick moisture methods are not accurate and care is needed with the vacuum method.
- Karl Fischer gives accurate results but they do not always correlate with caking.

<u>Water activity (a_w)</u> - Water activity is a quick and easy test that describes how tightly the water is held by the other components. Water activity values range from 0 to 1 where a water activity of 1 indicates that all of the water is available or free to move about while a 0 means that none of the water is available to react or move.

A plot of product moisture versus water activity gives a curve that indicates product susceptibility to caking, browning, mold growth, etc. The curve for water activity versus moisture content is dependent on the specific product, that is, different products have different water activity versus moisture plots.

The relationship between moisture content, water activity and type of powder is illustrated in Figure 7. In this example, three products with the same moistures would have very different water activities. The hygroscopic powder would have a water activity <0.1, the nonhygroscopic powder 0.3 to 0.4 and the crystalline powder 0.9. The water is tightly held by the hygroscopic powder because of the powders affinity for moisture while the crystalline material is stable as is and therefore does not tightly hold moisture which then is free to move.

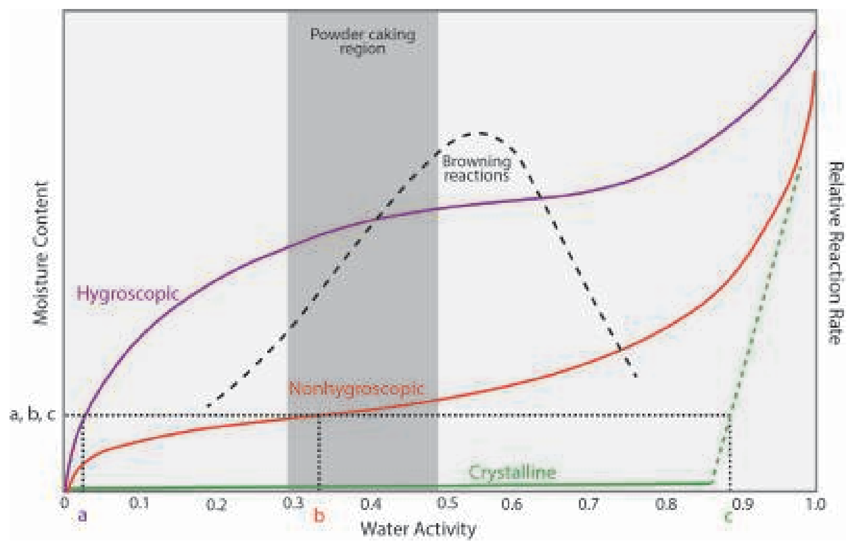

Figure 7. Moisture versus water activity for different types of products

There are several severe limitations to the use of water activity as an indicator of powder caking.

- Water activity of a powder changes quickly with exposure to air so the test must be done immediately after powder is removed from the dryer.
- Different products have different water actvity plots so it is important to know the plot for a given product before drawing conclusions about stability to caking. Water may be free to move, as indicated by a higher water activity, but not be a source of caking because the powder has no affinity for the water. For example, α-lactose monohydrate crystals already have all of the water they need and therefore do not attract additional water that would result in caking.

- A higher than typical water activity may indicate a problem but a “correct” value may or may not mean the powder will cake with storage.
- The water activity test was originally applied to whey powder which has protein. The water activity test is not applicable to crystalline lactose because of the lack of protein and the presence of the α-lactose monohydrate crystal that does not attract water.

Caking test - The caking test is specific for determining if a product will cake upon storage. The test is fast, easy, inexpensive and works for all dairy powders. Details of the test are provided in the Dairy Powder Caking Test handout. The basis of the test is exposing small amounts of powders to very humid conditions and then evaluating their degree of caking. Powders are scored from 1 to 5 with 1 being free flowing and 5 having a crust that does not break up and a gummy texture. Examples of test results are given in Figure 8.

Figure 8. Powders scored 1, 2, 3, 4 and 5 by the dairy powder caking test. The powder scored 4 also shows the results of Maillard browning.

The advantage of the caking test is that it tests specifically for caking. It should be noted that once a powder has caked and the lumps broken apart to make a free flowing powder the powder will not cake again.

Caking Tendency of Powders

Three main factors contribute to the potential for a dairy powder to cake: amount of lactose; amount of protein; and form of lactose present. Products that have large amounts of lactose and little protein along with lactose largely in the amorphous or glass form have a high caking potential. It does not mean that these products will cake but rather these products can cake given the right conditions. Table 1 is a summary of the factors influencing caking and susceptibility to caking for several dairy powders.

Table 1. Summary of factors influencing caking of dairy powders.

Product	Lactose (%)	Protein (%)	Lactose		Caking Potential
			Glass	α-Crystal	
NFDM	53	37	x		Medium
MPC 42	48	43	x		Medium
MPC 70	19	72	x		Low
MPI	<1	92	x		Very low
Whey	77	13	x	x	High
WPC 34	54	34	x		Medium
WPC 80	8	80	x		Low
WPI	2	93	x		Very low
Permeate	90	1	x	x	Very high
Lactose	99.9	0		x	Very low

Possible Strategies to Limit Caking

There are a number of approaches for limiting caking. Some strategies are more practical than others.

Crystallize more lactose

- Run higher total solids from the evaporator. The higher the total solids, the greater the percentage of lactose that can crystallize thereby reducing the overall concentration of amorphous lactose in the final powder.

- Cool to a lower temperature in the crystallizer. The lower temperature allows additional crystallization of lactose.
- Fully crystallize the lactose in the crystallizer. Lactose crystallization is a slow process so it is important to allow sufficient time to complete the process. Lactose crystallizers typically take at least 16 hours to completely crystallize the lactose.
- Use a secondary dryer to convert additional amorphous lactose to α-lactose monohydrate.

Optimize dryer conditions
- Reduce final powder moisture.
- Discharge powder at cool temperatures. Water moves from higher temperatures to lower temperatures. Reducing temperature differences between powder and storage conditions reduces moisture movement and therefore caking opportunities.
- If using a secondary dryer make sure powder enters the secondary dryer with enough moisture that additional amorphous lactose can convert to the α-lactose monohydrate form.

Limit milling of the product
- Milling creates heat and results in production of amorphous lactose on the surface of the α-lactose monohydrate crystal.

Slow absorption of water by the powder
- Use a heavier line in the bags. Heavier liners typically are a better moisture barrier.
- Use a moisture impervious container. Powdered milk originally was packaged in metal cans thereby keeping moisture in the air from reaching the product.
- Add lecithin to coat amorphous lactose. Although people have offered this as a possible solution it is not very practical.

Use smaller packages
- The larger the package, the greater the amount of moisture that can move. Super sacks or totes will have greater amounts of moisture movement as compared to bags.

Use a free flowing agent
- In addition to coating and smoothing rough edges of particles so they flow easily past each other, free flowing agents adsorb excess moisture in the product and atmosphere to help limit caking. Free flowing agents are used in products such as table salt to limit clumping. Examples of free flowing agents include calcium silicate, cornstarch, cellulose powders, sodium aluminosilicate and calcium and magnesium carbonate.
- Some compounds, such as magnesium carbonate, may also be referred to as desiccants because of their ability to absorb water.

Limit temperature and humidity variation in storage
- Warm product in a cool environment encourages moisture to move from the warm center of the package to the cooler outer edges. Caking and mold growth are possible if enough moisture collects in an area of the package. The larger the package, the greater the possible moisture migration.
- Often not practical to control storage conditions.