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Lactose caking: influence of the particle size distribution and the water content

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Abstract

Industrially, after the crystallisation of lactose from mother liquor, the crystals are separated by centrifugation and then dried in a flash drier followed by a fluid bed drier. It is known that if the moisture content and/or particle size is not correct then lumping and caking of the lactose can occur in the flash drier causing operational problems.The aim of this study was to characterize the influence of water content and particle size distribution on the caking of lactose powders.

Powders with different d_{50}'s and different water contents had their caking/clumping characteristics analyzed with a texture analyzer, a sticky plate and a blow tester to determine their cohesion. A theoretical approach looking at the capillary interactions of the liquid bridges between lactose crystals was taken from the literature to explain the results obtained.

The results showed that lactose powder with a low d_{50} can cake easily as soon as the water content is higher than 3%. This cohesion between lactose crystals is due to liquid bridges linking them together. The number of bridges formed affects the level of cohesion: the more numerous they are, the more cohesive the lactose crystals are. Therefore, small particles cake more easily because the number of bridges in a given volume is higher than for bigger particles.

It was predicted that the capillary force cannot hold together particles with d_{50} bigger than 400µm and that was confirmed by experiment.

Finally, in order to prevent the caking of lactose powders obtained by processes such as a decanter centrifuge, the lactose crystallization step should be controlled to obtain large crystal (with d_{50}>300µm) and/or the final water content should not be higher than 3%.

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Key Words;Lactose; Caking; Capillary force; Liquid bridge; Particle size distribution

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Nomenclature

D1	Diameter of first particle [m]
D2	Diameter of second particle [m]
F_C	capillary force between two spheres [N]
Fc_{tot}	total capillary force [N]
F_g	force on liquid bridge due to gravity on particle 2 [N]
F_T	surface tension force [N]
g	acceleration due to gravity [$m.s^{-2}$]
h1	distance of liquid bridge on particle 1 [m]
h2	distance of liquid bridge on particle 2 [m]
h_n	height h in the particle n [m]
N_b	number of bridges between one particle and the particles around it.[-]
R1	radius of liquid bridge [m]
R2	radius of curvature of liquid bridge [m]
v	volume of the powder [m^3]
$v_{paticule}$	volume of one particle [m^3]
Δp	pressure difference [Pa]
$\rho_{lactose}$	density of lactose crystal [$kg.m^{-3}$]
γ	surface tension [$N.m^{-1}$]
Φ_M	maximum packing parameter of the powder

1. Introduction

After lactose has been crystallised from whey it is separated from the mother liquor by centrifugation. After washing the last step before drying is again a centrifugation step which determines the moisture content of the crystal cake that is fed to the dryer (1, 2). The water content of the crystal cake is a function of the scroll speed in the centrifuge and water retaining properties of the cake. This water content affects the behavior of the final product by inducing the formation of solid lumps or the caking of the lactose crystals. Industrial experience has shown that it is critical to get this moisture content low enough so that clumps do not form in the drier. It is also known that bad batches containing lots of small crystals make it harder to achieve suitable moisture contents. There is no literature quantifying these effects for lactose so the aim of this study was to characterize the influence of water content and particle size distribution on the caking of lactose powders.

1.1. Theory

Water can exist in at least three states between the crystals as a function of the amount of water in the lactose powder: a hydroscopic state in which water forms thin adsorption layers on the crystal surfaces (3), a pendular state in which liquid bridges occur between individual particles (4), and a saturation state in which all the pores of the bed are filled with liquid (5). In the pendular state, the water distribution creates attractive forces between the particles due to the surface tension and the pressure difference between the air pressure and the liquid pressure across the liquid bridge interface. This difference is called capillary pressure (3). This adhesion between the lactose crystals determines the flow and the structure of the lactose powder. Moreover, this cohesion between the particles depends of the number of liquid bridges (3) while the volume does not have a strong effect on the total cohesion. It was shown that the presence of small particles in soils influence the water retention and the cohesion of this granular media (6). Therefore, the induction of capillary force into lactose powder depends of the amount of water in the powder and the crystals size.

The presence of a liquid bridge between two particles (Fig.1) leads to their interaction through a capillary force, Fc [N], owing to the pressure difference (Δp [Pa]) or Laplace pressure across the curved interface and the action of the surface tension force (F_T [N]) exerted around the annulus of the meniscus (7).

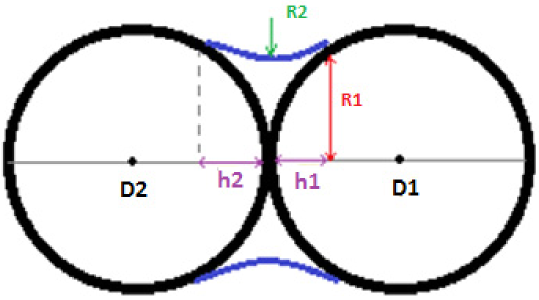

Figure 1: Geometrical representations of the liquid bridge between two uneven-sized particles.

The Laplace pressure across the curved interface can be calculated directly knowing the radii R1 and R2 by the equation (1)(8):

$$\Delta p = \gamma\left(R1 + R2\right) \tag{1}$$

The surface tension force is given by the equation (8):

$$F_T = 0.4 \times \gamma \times 2 \times \pi \times R1 \tag{2}$$

R_2 is a function of the diameter of the two particles:

$$R2 = \frac{h1 + h2}{2} \tag{3}$$

And

$$h_n = \frac{Dn}{2} - \sqrt{\left(\frac{Dn}{2}\right)^2 - R1^2} \tag{4}$$

Thus the capillary force is function of the diameter of the particles as shown in Fig. 2. If the particles are arranged vertically with the number two particle being below, then the gravity force applying to particle 2 can be calculated from equation (5):

$$F_g = \frac{4}{3}\pi\rho_{lactose}\, g\left(\frac{D2}{2}\right)^3 \tag{5}$$

When Fg>Fc, the two particles can no longer be held together by the liquid bridge. This is shown in the Figure 2 where the capillary force between one large particle has been plotted as a function of the second particle size. The figure assumes R1 (the radius of the liquid bridge) is 1µm in radius as it was found that the capillary force does not change much as R1 gets bigger.

In a compact stacking the number of bridges between spheres can be very high. Indeed, one sphere can be linked by 6 to 12 bridges to the other spheres around it, depending of their disposition (9). However, in a polydisperse lactose powder, crystals are not spherical and their sizes are not homogeneous, so the number of bridges can be higher. Thus the total capillary force induced by all the liquid bridges between spherical particles in a given volume of powder can be calculated by equation (6):

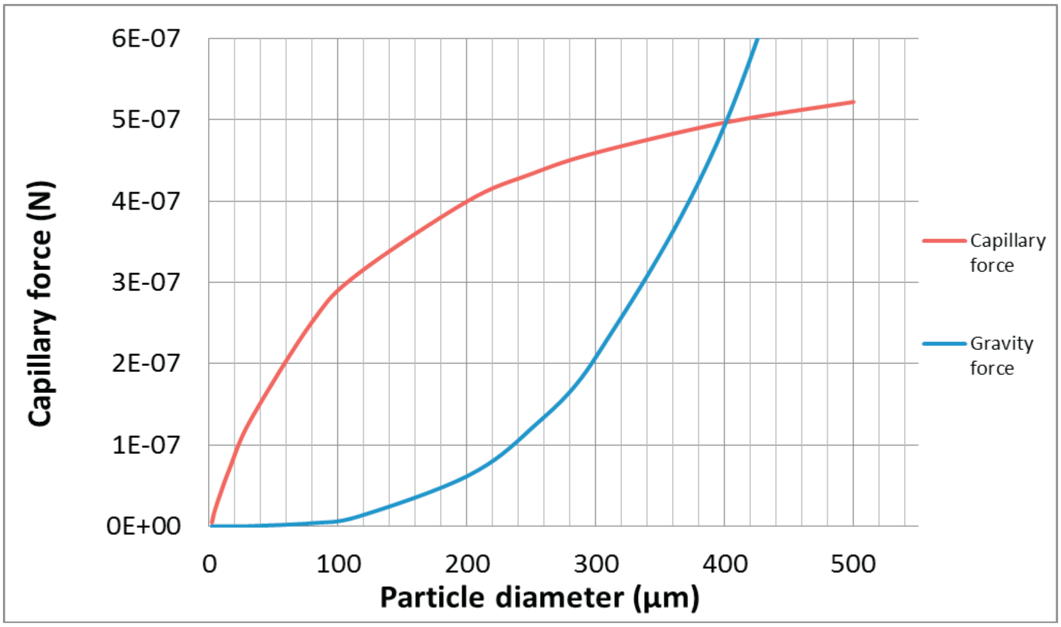

Figure 2: Capillary force for 1µm radius capillary bridge and gravity force applied on two lactose spheres in function on their diameter.

$$F_{C_{Total}} = F_C \times N_b \times \frac{v}{v_{particle}} \times \Phi_M \qquad (6)$$

Φ_M, the maximum packing parameter of the powder was taken as an average value of 0.619 for spheres of equal size (10).

2. Materials and methods

Alpha lactose monohydrate crystals from Fonterra, New Zealand were used in the study. Different particles size distributions were obtained by sieving the lactose powder with sieves of 300µm, 250µm, 212µm, 150µm, 106µm, 75µm and 45µm. The particle distribution of the sieved powders was analyzed with the Malvern Particle Sizer S. Five powders with d_{50} of 504,26µm, 301,69µm, 211,56µm, 135,74µm and 84,21µm were kept for the study.

Figure 3 : The blow tester

Each powder was mixed with different amounts of water to obtain free moisture contents of 5%; 4%; 3%; 2%; 1% (w/w). The powders were mixed with distilled water in a kitchen mixer, at speed 3 (100 rotation/minute) for 3 minutes and stored 2 hours in plastic bags to let them equilibrate. After the homogenization time, 6g of each powder was put into small weighing cups (3.2x3.2x0.8cm). Petri dishes were filled up with powder and the surface smoothed by scrapping a straight edge across the top for the blow tester tests. Five replicates were made for each powder and each test.

The blow test method is described by Paterson et al. (2001)(11) and the blow tester is shown in Fig. 3. The measured variable was the air flow value (corrected for pressure effects) ($L.min^{-1}$) for which a channel is carved into the lactose powder. Once a channel in the selected segment was observed, the air flow was stopped and the value of the air flow was read simultaneously on the rotameter of the blow tester.

A texture analyzer (TA-XT2, Stable Micro Systems, England) fitted with a cylindrical probe round end (8mm diameter) was used. The samples in the weighing cups were turned upside down on a Petri dish lid to form lactose cakes (dimensions: 3.1x3.1x0.8cm) (Fig.4). These cakes were analyzed by a compression method at 1.0 $mm.s^{-1}$ and were compressed until 90% of their height with a 5 kg load cell. The area under the compression curve (work) and the maximum compression force was recorded to estimate the cohesion of the samples.

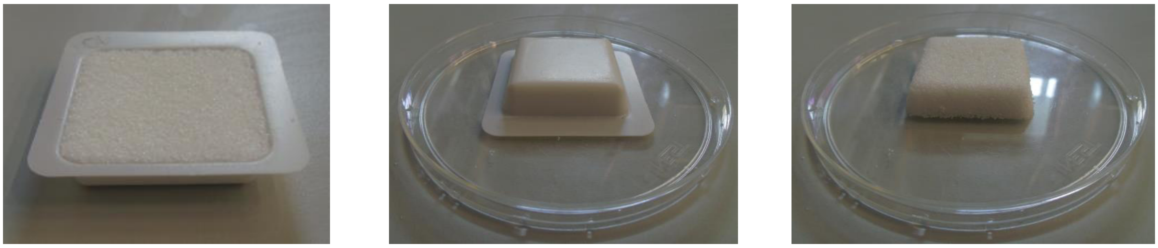

Figure 4: Lactose powder molding into weighing cup for the experiments.

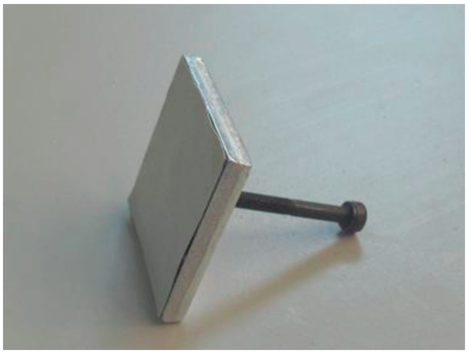

Figure 5: The sticky plate showing the double sided tape and handle

The sticky plate consists of a 30 mm x 30 mm steel plate (3.5mm of thickness) with a 30 mm screw inserted as a handle. The surface was coated with double sided tape (Fig. 5).This sticky plate was put on scales and tared. Then the samples in weighing cups were turned upside down on a Petri dish lid. The sticky plate was put on it, taken off immediately and weighted one more time. The weight of crystals stuck on it was recorded. The tape was renewed for each sample.

3. Results and discussion

Fig. 6 shows that the weight of powder that adhered was very low for water contents below 2%. At these levels the amount of water in the powder was not enough to create a pendular domain so the number of liquid bridges between crystals was not enough to hold them together. The graph also shows that the weight of crystals that stuck to the plate decreased for larger particle sizes. Indeed, as predicted in Figure 2, the capillary forces cannot hold particles together when the diameter is above 400µm because for particles larger than this the gravity force becomes larger than the capillary force.

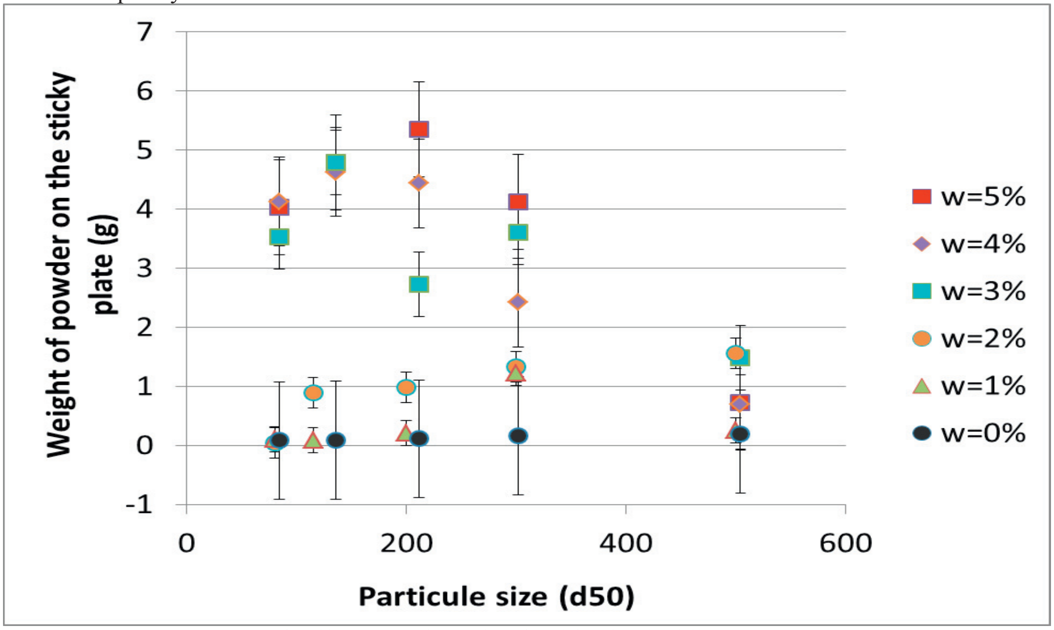

Figure 6: Weight of lactose powder stuck to the sticky plate for different water contents (w %).

Figures 7 and 8 show that the force required to break the capillary forces between the particles increase with the amount of water. Furthermore, for water contents higher than 3%, the cohesion between crystals becomes constant. Richeufeu et al. (2006)(12) showed that the failure threshold of capillary force is nearly independent of the volume of the liquid bridges. Thereby, for water content below 3%, the number of bridges between crystals may increase to arrive at the maximum number. After that, the volume of the bridges is increasing without having a strong effect on the capillary force between particles. As the liquid content is increased, the liquid bonds may coalesce, at least locally, affecting the general cohesion of the particles by reducing the number of bridges.

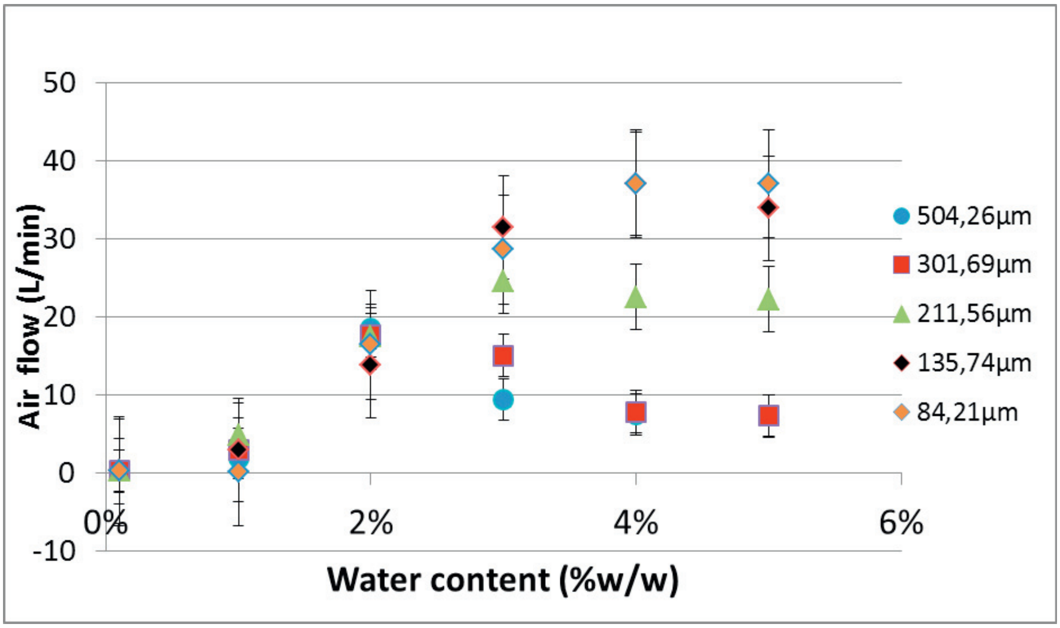

Figure 7: Air flow applied on the lactose powder samples in function of water content and for powders with different d_{50}.

The cohesion of powders as measured by the blow tester (Figure 7) and the work needed to compress the samples (Figure 8) with d_{50}'s of 504 µm and 302 µm have low values while the smaller particles are strongly cohesive. Indeed, even if the capillary force per bridge increases with the diameter of the particle (Figure 2), the number of small particles in a given volume is higher than for big particles so the force needed to break all these bridges will be higher.

These results are confirmed in Figure 9 where the maximum force needed to penetrate the lactose powder increased for smaller particles. For particles larger than 300µm, the force needed to break the capillary forces in the volume of the texture analyzer probe was much higher than predicted by theory, even for small amounts of water. Possible reasons to explain these results are: the theoretical calculations were made for monodisperse powder made of spherical particles while lactose powders are polydisperse systems of non-spherical (tomahawk) crystals. Additionally the surface of the crystal is not flat and the roughness of the crystals can interfere with the capillary forces (13). Indeed, more capillary domains may be observed as a function of the water content (14):

- for very small amounts of water in the powder, an 'asperity domain' is created and small liquid bridges are formed between asperities of crystals.
- when more water is added, a 'ruggedness domain' is observed and the volume of the liquid bridges is increased to fill the asperities of the particles.
- for sufficient amounts of water, a 'spherical domain' is observed and the ruggedness of the particle does not influence the capillary forces between crystals.

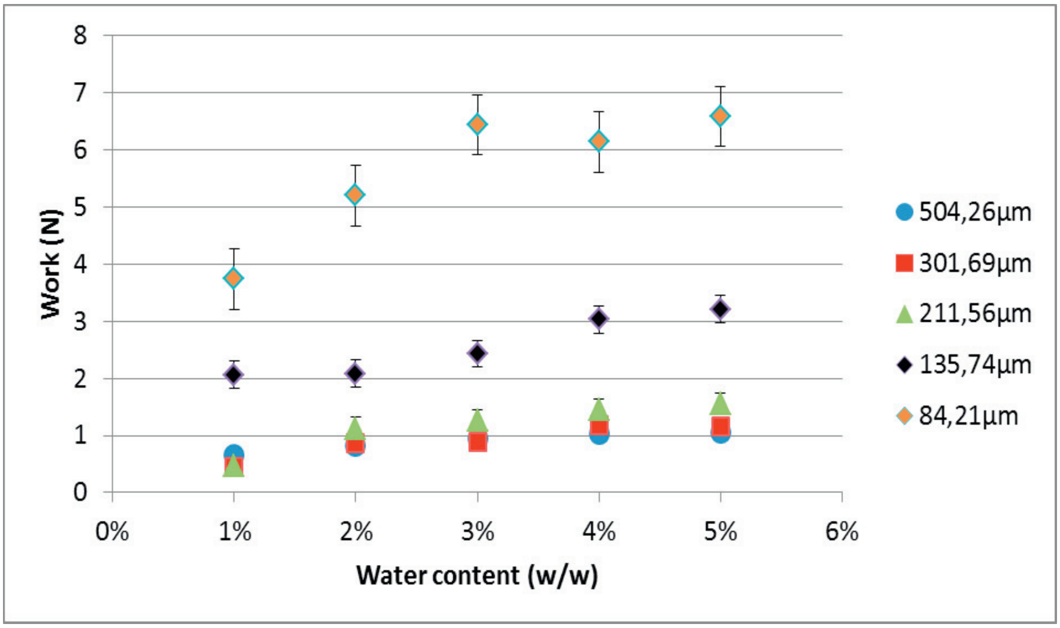

Figure 8: Work of probe of the texture analyzer in function of water content and for powders with different d_{50}.

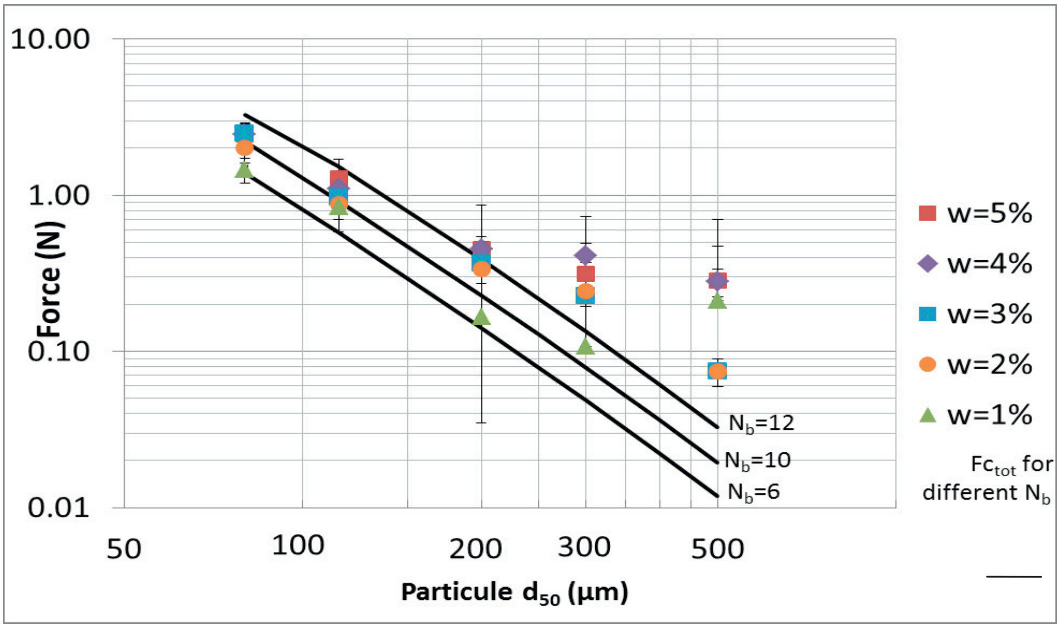

Figure 9: Maximum penetration force measured by the texture analyzer in function of particle size for different water content, compared to the theoretical total capillary force Fc_{tot} for different number of bridges (N_b)

In the spherical domain, the capillary force between crystals should be close to the theoretical predictions. For lactose crystals with d_{50}'s greater than 300 µm, it is possible that the 'spherical domain' needs more water to be established because their asperities are bigger than with small crystals.

For crystals with d_{50} below 115µm, Figure 9 shows that with the number of bridges between 6 and 12 per crystal for water contents between 4% and 5% the theory predicts the experimental results.

4. Conclusions

This work results showed that small lactose crystals with a d_{50} less than 300 µm can cake easily as soon as the water content is higher than 3%. This cohesion between lactose crystals is due to liquid bridges linking them together. The number of bridges formed affects the level of cohesion: the more numerous they are, the more cohesive the lactose crystals are. Therefore, small particles powder cake more easily because of the number of bridges in a given volume is higher than for larger particles. It was predicted that the capillary force cannot hold together particles with a d_{50} larger than 400µm and this was confirmed by experiment.

Finally, in order to prevent the caking of lactose powders in the driers, the lactose crystallization step should be controlled to obtain large crystals (with d_{50}>300µm) and/or the final water content should not be higher than 3%.

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