**AEROSIL® and SIPERNAT® products help to avoid lumps in sugars and sugar alcohols**

Nearly everyone has discovered the downsides of hygroscopic carbohydrates. Due to the high number of polar hydroxyl-groups in those molecules, they greedily attract moisture from the environment. Agglomerates and hard lumps as a consequence are difficult to process further.

To ensure a convenient as well as efficient handling of sugars without time and resource consuming measures to break agglomerates again, it is important to reduce caking and avoid formation of lumps reliably.

**Some examples of carbohydrates that benefit from the treatment with SIPERNAT® precipitated silica or AEROSIL® fumed silica**

- Fructose
- Confectioner’s sugar
- Glucose
- Brown sugar and caster sugar
- Lactose
- Polysaccharides, such as Maltodextrin, Polydextrose or even starches
- Sucrose
- Sugar substitutes, e.g. Xylitol, Sorbitol and other Polyols

**Reasons for carbohydrate agglomeration and prevention**

As soon as the humidity in the environment reaches a concentration specific to each carbohydrate, they immediately start taking up moisture from the vapor phase. This relative humidity is defined as water activity of the substance.

At which point the carbohydrate commences picking up water depends, amongst others, on the chemical nature of the substance, its purity and particle size. Impurities and smaller particle sizes increase the hygroscopicity, meaning moisture is absorbed already at lower ambient humidity [1].

Figure 1 compares the water content of various sugars and polyols depending on the relative humidity of the atmosphere. In Figure 1 it can be seen, that Fructose tends to take up moisture already at relatively low humidity. It is one of the most hygroscopic carbohydrates, followed by polyols such as Sorbitol and Xylitol.

**Figure 1**

Water uptake of various sugars and polyols depending on relative humidity in the atmosphere. [Based on 1, 2, 3]
The absorbed water builds liquid bridges between the individual particles causing them to agglomerate. At continued exposure to moisture some amount of the carbohydrate becomes dissolved in this water and – when dry again – at lower relative humidity, the carbohydrate will re-crystallize resulting in solid bridges between the particles which leads to the formation of very hard lumps. This principle is demonstrated in Figure 2.

SIPERNAT® precipitated silica and AEROSIL® fumed silica can prevent the formation of these liquid or even solid bridges. The silica absorbs the water film on the surface thereby breaking liquid bridges and avoiding growth of solid bridges.

Additionally, the silica acts as a spacer avoiding direct contact between the individual carbohydrate crystals. Especially very fine powders, such as confectioner’s sugar, tend to cake as the small particles come into close contact to each other. Increasing the distance between the individual particles is important to reducing the attraction forces between those small particles and thus improving the flowability of the fine sugar grades.

Detailed information on the mode of action of silicon dioxide to improve and ensure powder flowability can be found in the Technical Information 1351 "SIPERNAT® and AEROSIL® as Flow Aid and Anticaking Agent" [4].

Example: caking because of moisture uptake

As described in Figure 1, Xylitol is very hygroscopic; it tends to pick up moisture from the environment very fast. Even when stored at room temperature and humidity levels well below the water activity of this polyol, it absorbs water and forms large lumps decreasing the flowability.

Defined glass funnels can be used to determine flow properties of powders. The powder that flows out without interruption through a smaller outlet diameter of the funnel has a better flowability.

Figure 3 (left side) shows the result of keeping Xylitol in a sealed glass bottle at ambient conditions. It is obvious that the material is nearly completely caked and therefore not able to flow through the largest funnel opening. This poor flowability is classified as flow grade 7.

In contrast to this, when the polyol is treated with AEROSIL® fumed silica or SIPERNAT® precipitated silica this is reliably avoided. An example of Xylitol treated with 1 wt% SIPERNAT® 50 S is shown in Figure 3 on the right side. Under the same storage condition the particles do not agglomerate or form lumps. It is still an easy-to-handle, free flowing powder. It reaches the flow grade 3.
Example: poor flowability because of small particle size

Another reason for poor flowability and caking of sugars is the size of the particles. The finer the powder, the worse the flow is.

In the following graphs, two different disaccharides were tested for their caking behavior during storage at elevated temperature and humidity. The first one has a particle size of approximately 130 µm on average, the second one, with a mean-particle size between 50 to 100 µm, being even finer.

With a texture analyzer the hardness of the caked disaccharide was measured and the ability of various anti-caking aids to reduce this hardness was evaluated.

For both carbohydrates a dosage of less than 1 wt% of AEROSIL® or SIPERNAT® products is very efficient in reducing the hardness of the caked material. Figure 4 and 5 summarize the results of those tests.

Besides the obvious reduction in hardness, in Figure 4 the importance of adapting the mixing procedure can be seen. Depending on the material itself, the silica used and the dosage it is necessary to adjust the mixing time in combination with shear-intensity. In many cases, short mixing time at medium shear intensity results in the best anti-caking effect.

The second disaccharide, results are shown in Figure 5, cakes even harder, when untreated. Compared to the example in Figure 4, the hardness of caking here is more than doubled. This is because of the higher humidity during storage but also because of the smaller particles of the confectioner’s sugar. Nevertheless, AEROSIL® and SIPERNAT® grades very efficiently minimize the caking tendency of this sugar, already less than 1 wt% silica addition results in a significant improvement. To reduce the caking of very fine powders, silica with fine particles itself offers the best performance, as for example AEROSIL® 200 F.

As those fine powders are often produced by milling, an efficient way of adding silica is directly during the milling step. After a short period of some seconds (or minutes) the silica is dispersed evenly which improves flowability, reduces caking and avoids clogging the mill. This often improves the throughput of the mill.
**Figure 4**
Reduction in hardness of caked disaccharides depending on type of flow aid added

<table>
<thead>
<tr>
<th>Disaccharide</th>
<th>AEROSIL® 200 F</th>
<th>AEROSIL® 380 F</th>
<th>SIPERNAT® 22 S</th>
<th>SIPERNAT® 25</th>
<th>SIPERNAT® 350</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness of caking [g/mm]</td>
<td>4000</td>
<td>3500</td>
<td>3000</td>
<td>2500</td>
<td>2000</td>
</tr>
</tbody>
</table>

- 0.25 minutes mixing time at 400 rpm
- 0.5 minutes mixing time at 400 rpm

1-α-Glucopyranosyl-1-α-glucopyranosid (mean particle size ~ 130 µm) treated with 1 wt% Silica
Mixing conditions: vertical lab mixer (Somakon) for 0.25 or 0.5 minutes at 400 rpm
Storage at 30 °C, 40% rh, 4 days
Hardness determines the force necessary to scratch off a certain layer of the caked material

**Figure 5**
Reduction in hardness of icing sugar depending on silica dosage

<table>
<thead>
<tr>
<th>Dosage of silica</th>
<th>Untreated icing sugar</th>
<th>0.2 %</th>
<th>0.2 %</th>
<th>0.4 %</th>
<th>0.6 %</th>
<th>0.2 %</th>
<th>0.6 %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness of caking [g/mm]</td>
<td>12000</td>
<td>10000</td>
<td>8000</td>
<td>6000</td>
<td>4000</td>
<td>2000</td>
<td>0</td>
</tr>
</tbody>
</table>

- 100 g of confectioner’s sugar (particle size between 50–100 µm) treated with corresponding silica, dosage given as wt% Silica
- Mixing in lab mixer (Somakon) for 10 sec at 2000 rpm to simulate milling
- Storage at 30 °C, 60% rh for 24 h
- Caking represents the force needed to scratch off a certain layer of the caked icing sugar
Another aspect needs to be taken into consideration: dustiness

By reducing the adhesion forces of fine powders, the individual particles become freely movable and separate from each other. Because of this, it might happen that dustiness of this powder increases. As dust is inconvenient and a possible irritant during manufacturing and packaging processes it needs to be avoided reliably.

Figure 6 compares the influence of various silica grades and dosages on the caking behavior (horizontal axis) versus the effect on dustiness (vertical axis) of powdered sugar. This graph points out the options to reduce the caking of confectioner’s sugar without increasing its dustiness.

It can be seen that especially SIPERNAT® 350 outperforms other flow aids with regard to both attributes. Here a dosage of about 0.4 wt% is already sufficient to reduce the caking significantly but not increase the dustiness. Higher dosages might bring only a limited additional advantage with regard to hardness of lumps, but there is the risk to generate some dust. Consequently, the type of flow aid used, its dosage and the milling conditions need to be optimized carefully.

**Figure 6**
Reduction in caking without dust-generation

<table>
<thead>
<tr>
<th>Caking</th>
<th>Dust</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soft</td>
<td>0.6 % SIPERNAT® 350</td>
</tr>
<tr>
<td>0.4 % SIPERNAT® 350</td>
<td></td>
</tr>
<tr>
<td>0.2 % AEROSIL® 200 F</td>
<td></td>
</tr>
<tr>
<td>0.2 % SIPERNAT® 200 F</td>
<td></td>
</tr>
<tr>
<td>Untreated confectioner’s sugar</td>
<td></td>
</tr>
</tbody>
</table>

Confectioner’s sugar (particle size between 50 – 100 µm) treated with corresponding silica, dosage given as wt%.
Mixing in lab mixer (Somakon) with varying duration at 2000 rpm to simulate milling.
Dust measurement: Optical method, using light scattered by dust, equipment from Fa. Arndt / Germany.
Storage at 30°C, 60% rh for 24 h; caking represents the force needed to scratch off a certain layer of the caked icing sugar.
Summary and recommendation

Depending on the causes of caking and poor flowability of powdered sugars the type of flow aid used and the mode of addition should be chosen accordingly. In Figure 7 the different reasons for caking and the effects of silica are summarized.

<table>
<thead>
<tr>
<th>1) Humidity</th>
<th>Without silica</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture uptake</td>
<td>Liquid bridges</td>
</tr>
<tr>
<td>With AEROSIL® or SIPERNAT®</td>
<td></td>
</tr>
<tr>
<td>Moisture uptake</td>
<td>Silica absorbs liquid</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>2) Particle size</th>
<th>Without silica</th>
</tr>
</thead>
<tbody>
<tr>
<td>Small particles</td>
<td>Close contact</td>
</tr>
<tr>
<td>With AEROSIL® or SIPERNAT®</td>
<td></td>
</tr>
<tr>
<td>Small particles</td>
<td>Silica increases distance</td>
</tr>
</tbody>
</table>

Figure 7
Reasons for caking of carbohydrates and mode of action of silica

In case of hygroscopicity, moisture-uptake is the main challenge, silica with a high absorption capacity, such as SIPERNAT® 50 S, will be beneficial.

When the particle size or fineness of the carbohydrate is the limiting factor for the flowability, flow aids, like AEROSIL® 200 F or AEROSIL® 380 F are a good choice. Because of their particle structure, they very efficiently improve flowability of small-sized powders.

For special applications – where besides flowability dustiness is an important aspect – SIPERNAT® 350 offers a very good performance. Because of its fine particle size, it improves flowability efficiently, but does not increase dust.

For all powders the mode of mixing the flow aid into the material has a significant influence on the effectiveness of the silica.

Adapting the flow aid used, its dosage and the way of processing it depending on the individual condition and requirements, finally leads to a product with optimal powder and handling characteristics.

References

[1] "Zucker und Zuckerwaren", H. Hoffmann, W. Mauch, W. Untze, Behr’s Verlag, p53ff
[4] TI 1351 „SIPERNAT® specialty silica and AEROSIL® fumed silica as flow aid and anticaking agent, Evonik Industries AG

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