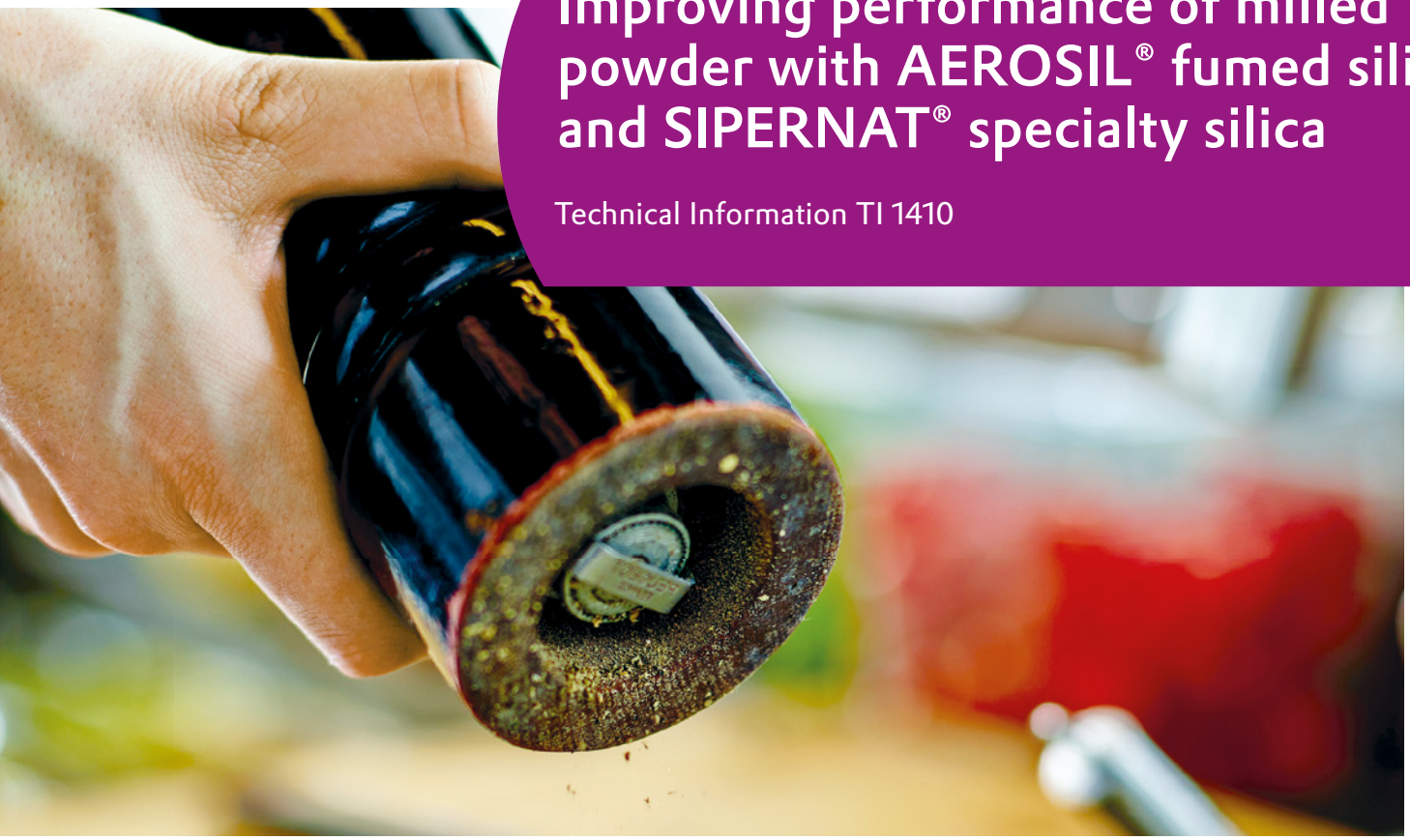
EVONIK INDUSTRIES

Improving performance of milled powder with AEROSIL® fumed silica and SIPERNAT® specialty silica

Technical Information TI 1410



Evonik. Power to create.

Easier milling and improving of free flow / anti-caking properties of milled powders at the same time.

Milling of ingredients is a common process in various industries. A good flowability and an anti-caking property of the milled powder is crucial in many applications for a good handling, reduced shut down periods of the production, suitable transportation ability and an exact dosing manner.

Many ingredients of daily use are being used as milled products like e. g. spice, seasonings, icing (confectioner's) sugar and herbs to facilitate production processes. The handling of several products during the milling process can cause trouble like adhesions at the wall of vessels and mixers, lumping of the product during conveying and others. Reasons for that unfavorable behavior of milled raw materials are e. g. a high content of moisture, oil or fat that can be pressed out and cause stickiness in the mill or of the milled product. Additionally, good flowing powders after milling can be challenging because fine particles are sticking together by the so-called "van-der-Waals-force".

SIPERNAT® and AEROSIL® products as milling aids

How can SIPERNAT® and AEROSIL® products help during the milling step?

- Silica reduces caking of low melting powders during milling process.
- Silica absorbs promptly any released liquid during milling step.
- Silica avoids clogging of the mill and increases throughput.
- Silica improves the free flow and anti-caking properties.
- Silica minimizes the down time of the mill.
- Silica increases the throughput.

When low melting powders or products with a high liquid content are milled, this often leads to caking of material in the mill. As a result, adhesions at the wall of the mill occurs.
To reduce time – and cost-consuming cleaning intervals, SIPERNAT® or AEROSIL® grades can be added to the grinding process or mixed in a step before. The surface of the finely milled powder is coated with the silica immediately. Promptly, any released liquid is absorbed and consequently agglomeration of these particles is inhibited. Besides good powder flow, the mill remains cleaner for longer operation time and the throughput increases. Cleaning intervals of the milling device can be extended and down times minimized.

Figure 1 demonstrates this effect in a hammer mill.

Example: Chili powder in a hammer mill
Without milling aid, the powder sticks to the wall and blocks the mill

With SIPERNAT® product clogging is reduced significantly

The use of Evonik specialty silica and fumed silica is beneficial for hammer mills, pin mills or alike. Applying the silica in this way is especially convenient, as the fine powder is already coated with silica after the milling process, which improves the flow-characteristics of the material for the following processing steps. In table 1 some application examples are listed:

Table 1: Application examples of silica used as milling aid

Application	Examples	Recommended types of milling aid
Food / Feed	Seasonings / Spices Herbs Salt / Sugar Mineral mixes Limestone	SIPERNAT® 50 or 50 S SIPERNAT® 22 or SIPERNAT® 22 S SIPERNAT® 350 AEROSIL® 200 F or AEROSIL® 380 F SIPERNAT® D 17 for feed only
Agrochemicals	Wettable powder	SIPERNAT® 22, SIPERNAT® 320, SIPERNAT® 350, SIPERNAT® 360
Chemical products	Sulphur	SIPERNAT® 320

Test methods:

Procedure of test methods is available on request.

Test equipment:

1. Somakon Lab Mixer (figure 2)
2. Retsch ZM 200, milling device used for tests (figure 3)

Figure 2: Somakon Mixer and used tools

Figure 3: Open milling chamber and collector (by kind permission of Retsch, Haan, Germany)

Lab tests with Retsch milling device

Parameter Retsch milling device:

- Rotor speed: 18,000 rpm
- Rotor circumferential speed: 92.8 m/s
- Ring sieve mesh diameter: 1 mm

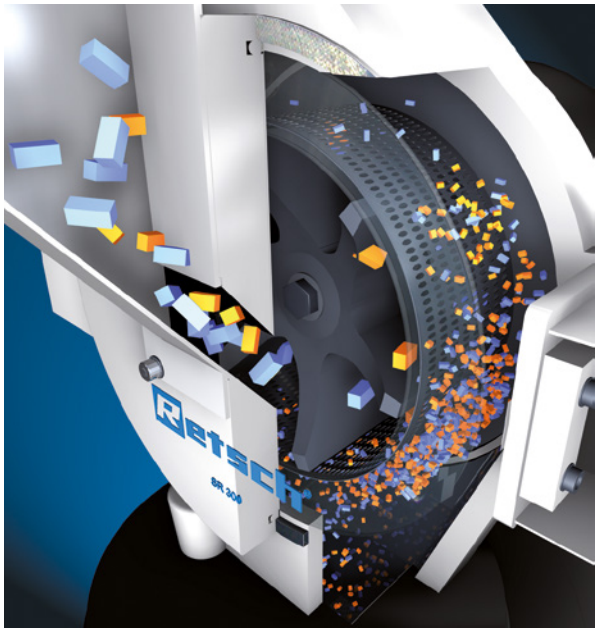


Figure 4: Scheme milling with a hammer mill (by kind permission of Retsch, Haan, Germany)

1. Milling of salt

Crystalline salt has an excellent flowability and no caking tendency as long as it is kept dry. No dust is developed because of the big crystals. When crystalline salt is milled, the salt-powder shows inferior flowability and caking behavior. This is caused by the higher surface area of salt after grinding and the smaller particle size. Besides, the dust content can be increased too. To change those properties of powdered salt SIPERNAT® and AEROSIL® products can help to improve the flowability for a better transport and packaging behavior. SIPERNAT® and AEROSIL® products reduce the caking tendency to keep the mill clean without adhesions at the walls. When salt was ground in our tests without addition of SIPERNAT® and AEROSIL® products the fine salt clumped and stuck at the walls of the mill.

During tests, the following flow aids were used: SIPERNAT® 22 S, 50 S, 22, 50, AEROSIL® 200 F of Evonik and TCP (Tri-calcium-phosphate, grade: Cafos MP of Chemische Fabrik Budenheim).

After the test flow rate (FG in diagram 1, 2, 3), caking behavior and dust value of the mixtures were checked. You can request further information.

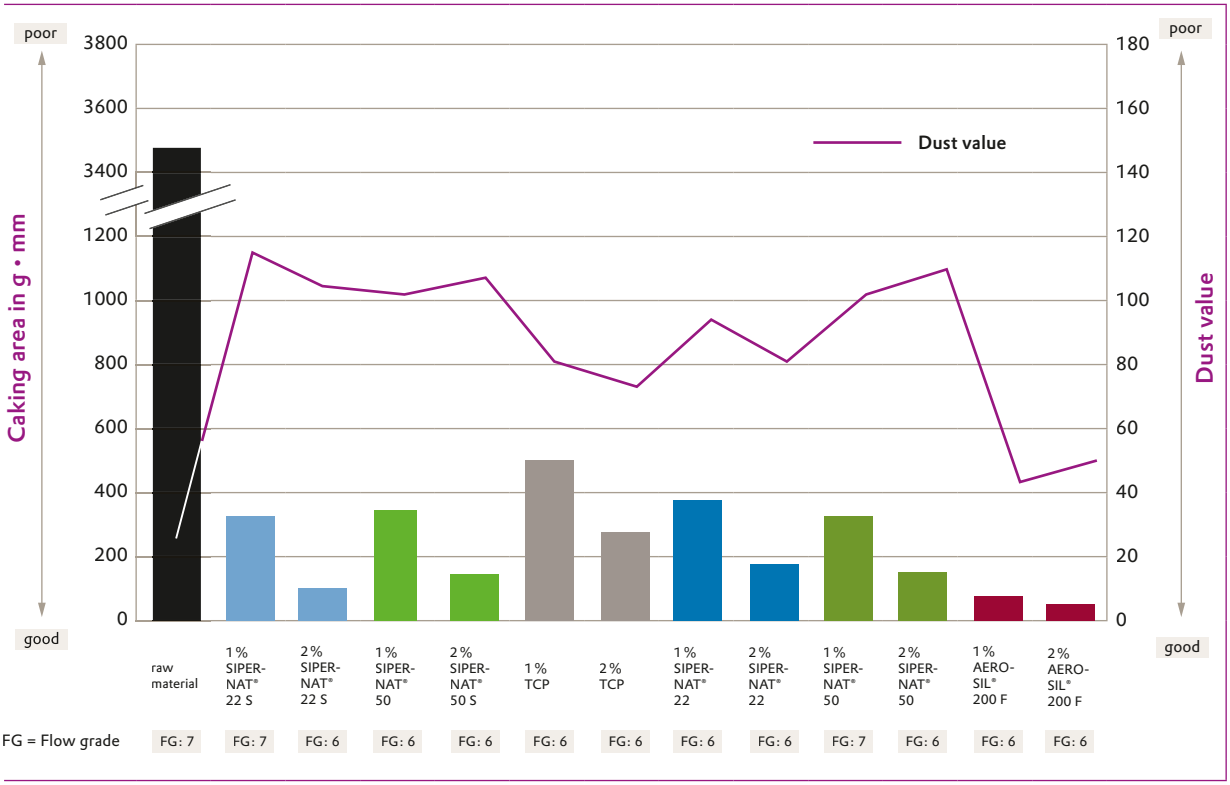
Three different test procedures during the trials were used:

Procedure 1: Direct milling of salt and silica together

Procedure 2: Mixing of salt and silica in a vertical lab mixer followed by the milling process

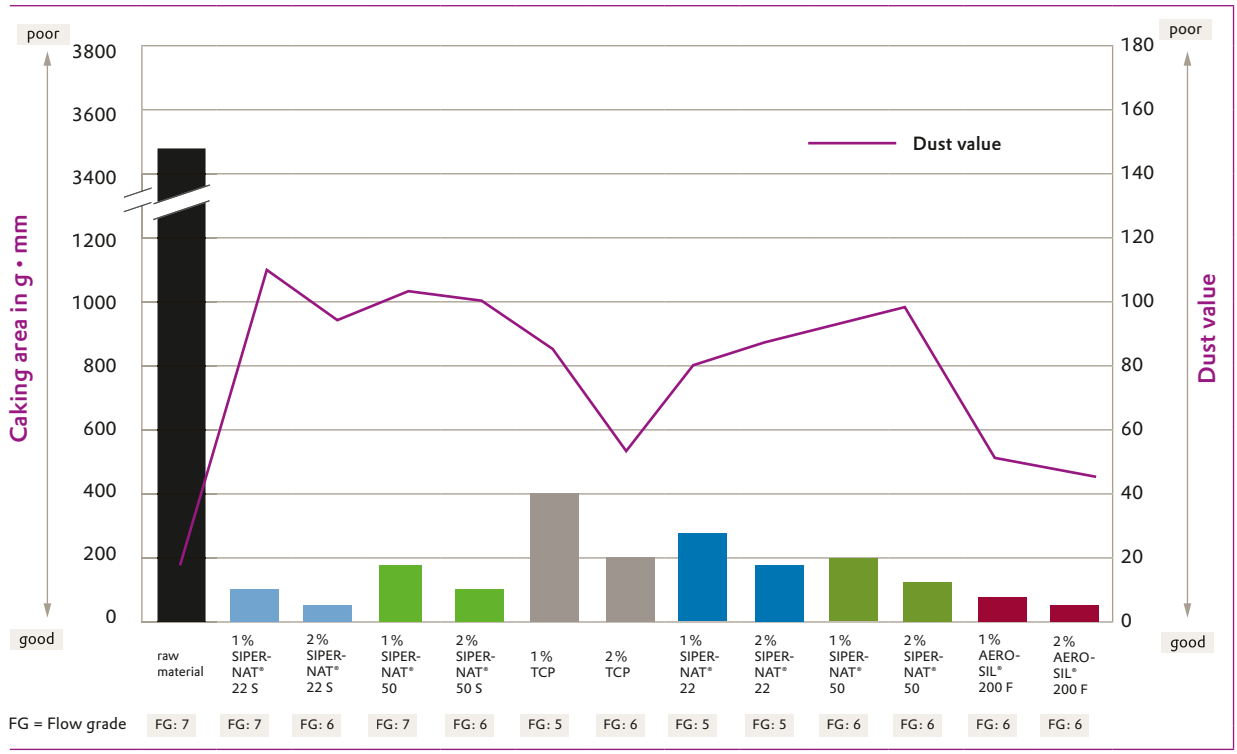
Procedure 3: Milling of salt and post addition of the flow aid in a vertical lab mixer

Diagram 1: Milling of salt and flow aid directly in the mill (procedure 1)



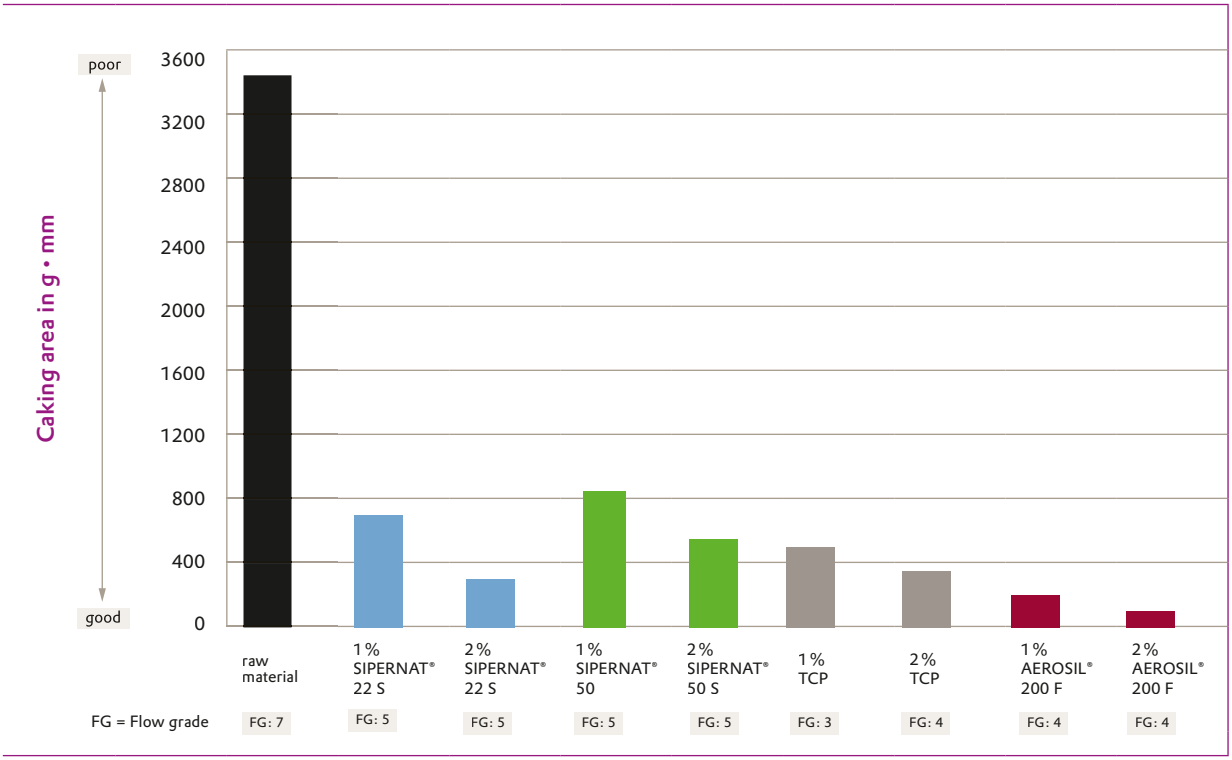
- 2 % SIPERNAT® 22 S, 2 % SIPERNAT® 50 S, 1 – 2 %SIPERNAT® 22, 2 % SIPERNAT® 50, 1 – 2 % AEROSIL® 200 F or 1 – 2 % TCP improved the flow grade (FG).
- All flow aids improved the caking behavior.
- The best anti-caking results and the lowest dust level were achieved with AEROSIL® 200 F.

Diagram 2: Mixing of salt and silica in the Somakon mixer followed by the milling process (procedure 2)



- SIPERNAT® 22 S, SIPERNAT® 50 S, SIPERNAT® 22, SIPERNAT® 50, AEROSIL® 200 F or TCP improved the flowability (FG).
- The lowers dust values were achieved with AEROSIL® 200 F and TCP.
- All flow aids improved the caking behavior. The best anti-caking results were achieved with AEROSIL® 200 F, SIPERNAT® 22 S and SIPERNAT® 50 S.
- The best results and lowest dustiness were achieved with AEROSIL® 200 F.
- SIPERNAT® 22 S, 50 S, 22, 50, AEROSIL® 200 F or TCP improved the flowability.
- The caking behavior was improved by all flow aids.
- SIPERNAT 22® and 50 cannot resp. only minimal reduce the caking behavior of salt when they are not milled with the salt together due to the bigger particle size.

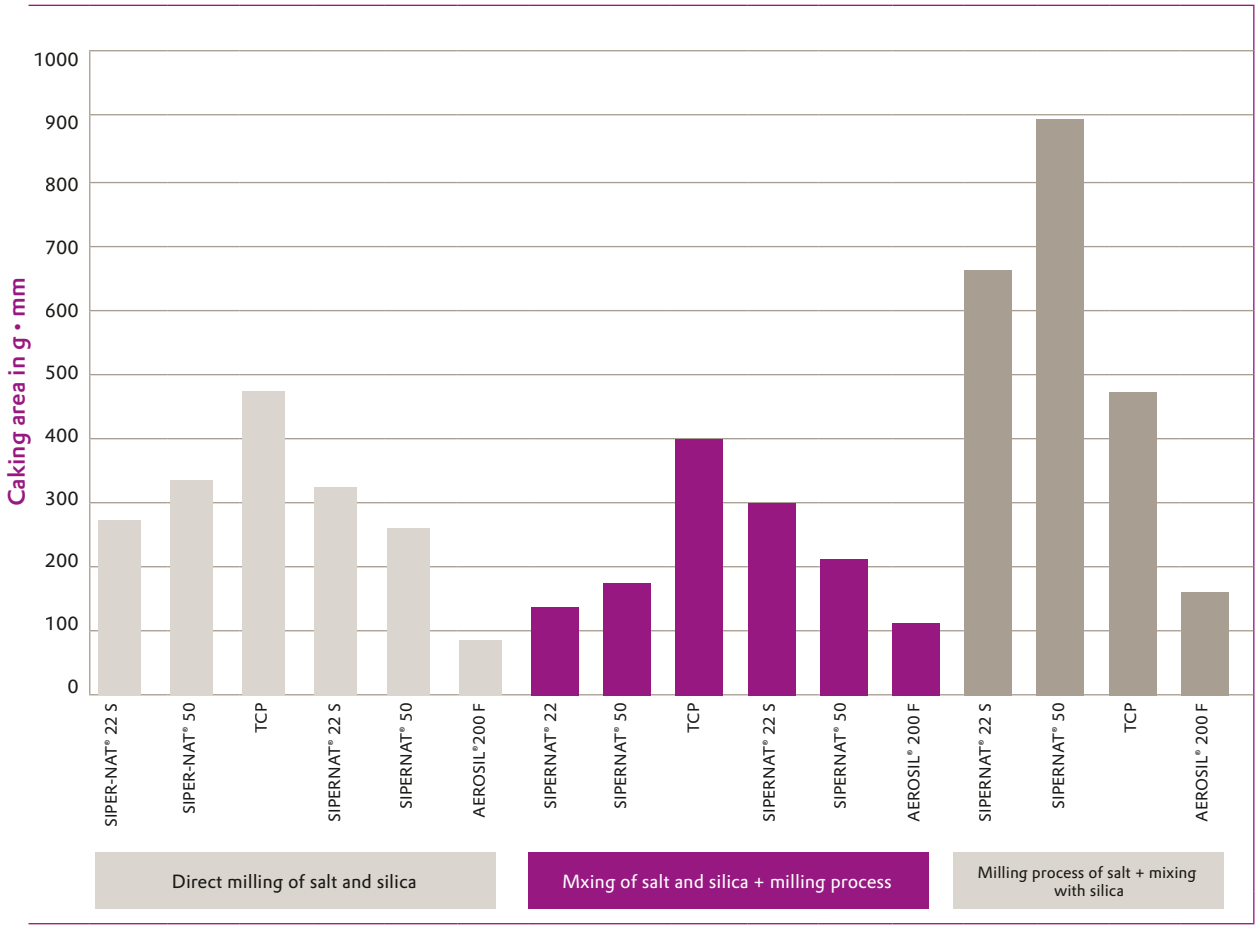
Diagram 3: Milling of salt and post addition of the flow aid in the Somakon Mixer (procedure 3)



Summary Salt (see diagram 4)

- SIPERNAT® and AEROSIL® products show very good anti-caking results in test procedure 1 and 2.
- Mill and the walls were kept particularly clean with SIPERNAT® and AEROSIL® additions reducing time and cost consuming maintenance intervals thereby increasing the throughput by longer operation times.
- In all tests, AEROSIL® 200 F showed the best anti-caking results.
- In procedure 2 SIPERNAT® 22 S and 50 S showed comparable anti-caking results like AEROSIL® 200 F.
- AEROSIL® 200 F achieved the lowest dust levels when milled together with the salt.
- A post addition of the flow aid after the milling step of salt (procedure 3) is not preferable because the mill glued and the milled salt product contains small lumps and agglomerates.

Diagram 4: Anti-caking - overview of mixtures with 1% silica



2. Milling of sugar

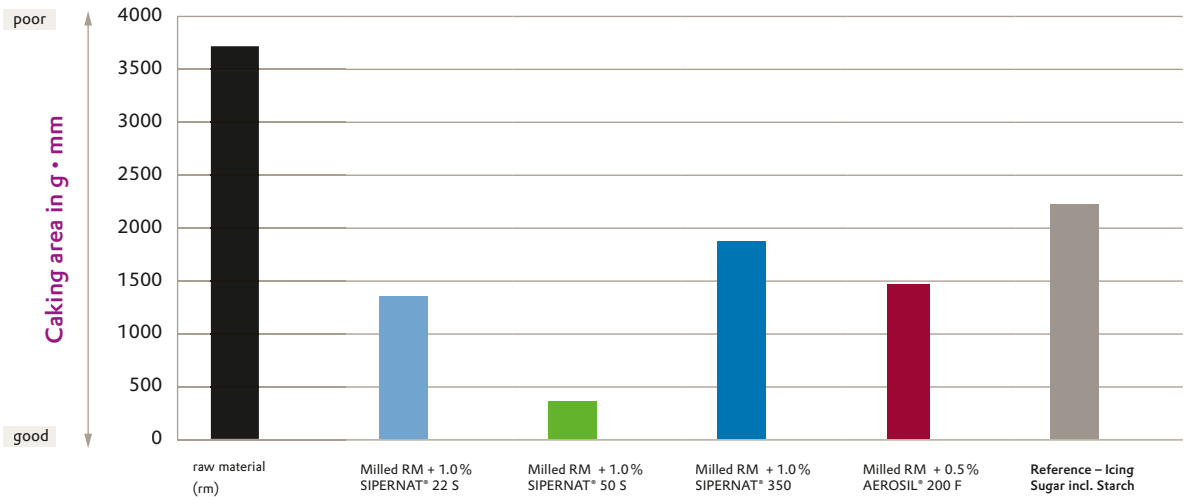
Crystalline sugar has an excellent flowability and no caking tendency arise as long as it is kept dry. No dust is developed because of the big crystals. When crystalline sugar is milled, the icing sugar-powder shows a worse flowability and caking behavior. This is caused by the higher surface area of sugar after grinding and the small particle size. To change those properties of powdered icing sugar silica can help to improve the flowability for a better transport and packaging behavior. Silica reduces the caking tendency to keep the mill clean without adhesions in the mill. Even the dustiness level during transport and packaging can be lowered. When crystalline sugar was ground without addition of silica it clumps together and sticks at the walls of the mill (figure 5). An addition of a flow aid e. g. SIPERNAT® 22 avoids that sugar is clinging at the wall (figure 6).

For caking tests, the following flow aids were used: SIPERNAT® 22 S, 50 S, 350, AEROSIL® 200 F and a reference sample with starch.

Figure 5 Icing sugar without flow aid

Figure 6 Icing sugar milled with 1 % SIPERNAT® 22

Diagram 5: Caking tendency of milled sugar with different flow aids



Summary sugar test

- After milling with SIPERNAT® or AEROSIL® products the remaining adhesions could be removed easier from the collecting pan of the mill.
- After milling the sugar raw material without any flow aid strong adhesions occurred in the collecting pan.
- The sample including 1 % SIPERNAT® 50 S showed a significant reduced caking tendency in comparison to the raw material and the reference sample.

3. Milling of pepper

Peppercorns can be stored for a long time without problems with caking and bad flowability. In comparison, milled pepper has a bad flowability and during the milling step strong adhesions can block the mill. Bad flowability is caused by the fine and hooky particles. No caking tendency occurred in our tests during storage of the milled pepper under pressure, high humidity and temperature in a climate chamber.

Testing:
Peppercorn was milled with SIPERNAT® 22 S, AeroMyl 33 (starch from Südstärke, Germany) and TCP (Cafos MP of Chemische Fabrik Budenheim, Germany). Flowability and dust number were determined.

Results:
- Milling the peppercorn without any addition of silica resulted in strong adhesions on the collecting pan of the mill.
- An addition of 1 % TCP or 1 % AeroMyl 33 (starch) showed no improvement, strong adhesions on the collecting pan were noticed too.
- An addition of 1 % SIPERNAT® 22 S showed less adhesions and an improved the flow grade from 6 to 5.

Diagram 6: Flowability of milled pepper with different flow aids

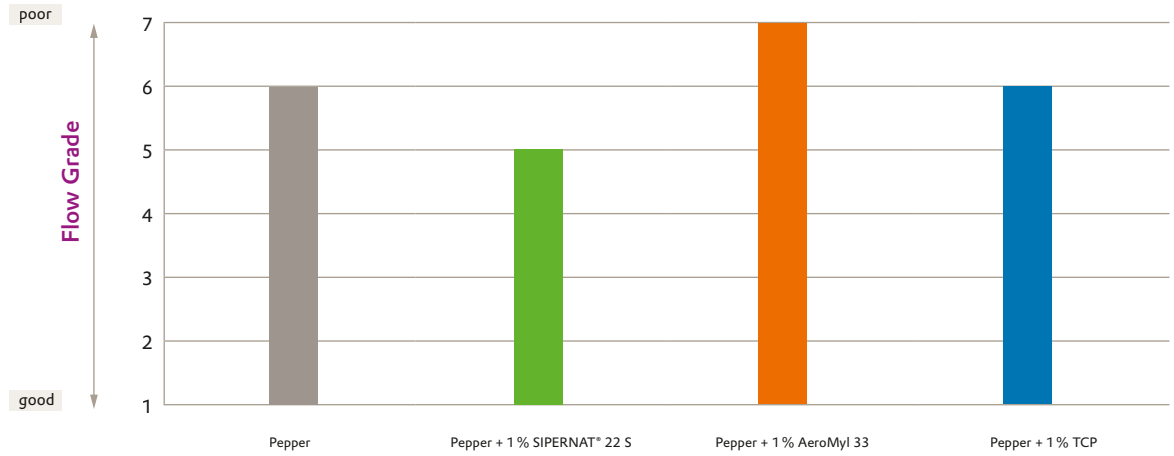
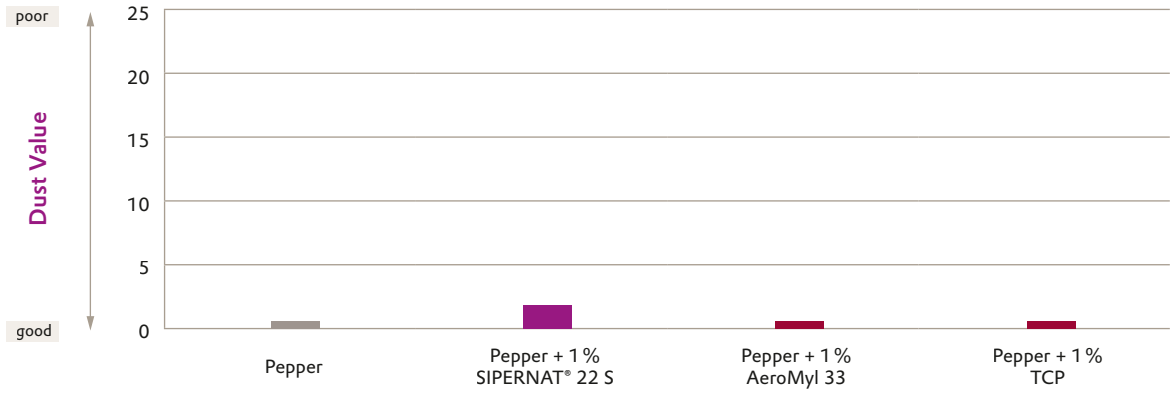


Diagram 7: Dust value of milled pepper with different flow aids



Remark dustiness:

The raw material pepper as well as all samples including SIPERNAT® products or a competitive material had a dust value below 3, in this range no dust problem exists.

5. Conclusion

SIPERNAT® and AEROSIL® products improve the milling step by keeping the mill clean. The flow aids improve the flowability and avoid caking of the milled products enabling convenient handling and dosing with minimized dustiness for you and your customers.

It is important to find out the most suitable silica grade and application conditions such as flow aid concentration etc. by testing.

Our applied technology experts, available in Asia, Europe and the US will be glad to support you.

Examples for AEROSIL® and SIPERNAT® grades

By offering the broadest range of food and feed compliant SIPERNAT® and AEROSIL® products, Evonik is able to address a wide range of properties:

Characteristic physico-chemical data of selected silica grades

Silica	DOA*-absorption in g/100 g Internal method	Particle size in µm Laser diffraction following ISO 13320	Food grade**	Feed grade***
SIPERNAT® 22 S	240	13.5	Yes/E 551	Yes/E 551 a
SIPERNAT® 22	235	120	Yes/E 551	Yes/E 551 a
SIPERNAT® 50 S	280	18	Yes/E 551	Yes/E 551 a
SIPERNAT® 50	295	50	Yes/E 551	Yes/E 551 a
SIPERNAT® 350	170	4.5	Yes/E 551	Yes/E 551 a
SIPERNAT® 320	195	≤ 10 % > 63 µ	No	No
SIPERNAT® 340	235	20	Food grade available in the Americas only	No
SIPERNAT® D 17	Not specified	10	No	Yes/E 551 a
AEROSIL® 200 F	Not applicable	Not applicable	Yes/E 551	Yes/E 551 b
AEROSIL® 380 F	Not applicable	Not applicable	Yes/E 551	Yes/E 551 b

* DOA = Dioctyl adipate, the numbers show the absorption capacity of a product, higher numbers means a larger absorption capacity, measured on original substance
** In Europe HACCP according to food additives EU 1333-2008
*** In Europe according to FAMI-QS
Purple marked product is hydrophobic.
The given data are typical values. Specification on request.

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