



AEROSIL[®] fumed silica and SIPERNAT[®] specialty silica as flow aid, anticaking agent and carrier

Recommended mixing procedures for powders and granulates
Technical Information TI 1213

1 Introduction

SIPERNAT® specialty silica and AEROSIL® fumed silica are frequently used in numerous applications within many industries. When used as a free flow or anticaking agent, silica acts as an additive which can generate significant improvements even at low addition levels. It can also be used as carrier for liquids, semi-solids or pastes. The effect of SIPERNAT® specialty silica and AEROSIL® fumed silica as a free flow and anticaking agent is dealt with in detail in Evonik's Technical Information TI 1351. Further information on free flow and carrier applications in different industries is given in Evonik's Technical information TI 1360.

As described in the above mentioned literature the use of silica requires, depending on its function, different mixing techniques. In free flow applications fine sized silica grades need to be dispersed in a way that provides a silica layer on the host powder's surface. In contrast carrier silica, which usually consists of free flowing coarse particles, should be handled gently to avoid deagglomeration.

The fundamentals of mixing silica are described in the literature (1, 2). The following pages will provide information on product specific properties and important considerations for the selection of mixing equipment as well as procedures. However, because of the number of parameters influencing a dry mix process this brochure cannot discuss all possible conditions in detail. We look forward to answering your individual questions and providing customized solutions. Contact details can be found at the end of this brochure.

2 Diversity of mixing equipment

The process of mixing solids can be significantly more complex than mixing liquids where a few material properties usually suffice to characterize the ingredients. When it comes to solids, the mixing process is influenced by dispersing properties like particle stability, particle size distribution, specific density, moisture content, and others. The multitude of different mixing systems, combined with the fact that new mixing devices constantly appear on the market, reflect the complexity of the topic this brochure is dealing with. A further challenge is transferring small scale trials to pilot or production scales.

Optimal mixing of silica could be very different depending on the respective application. On the other hand customers are already equipped with mixing devices and the selection of a suitable silica grade needs to be adjusted to both, the existing mixer and the product properties.

The working principle of customary mixing systems varies a lot. Common mechanical mixers, e. g. ploughshare® mixers (**figure 1**), paddle mixers (**figure 2**), cone mixers (**figure 3**) or ribbon blenders, operate by applying a pushing force to powder particles to be mixed; other devices, e. g. tumblers (**figure 4**) or V-Cone blenders mainly use gravitational forces for mixing. An airstream is used to mix/circulate powders in jet mixers, pneumatically mixed silos, or fluid bed mixers. Even devices which are not considered to be a mixer can be used to disperse silica, e. g. dosing silica into mills or spray towers is very common. Furthermore a number of mixing systems are available for batch and continuously operating processes and high and low shear systems are in use.



Figure 1: External view of a LOEDIGE ploughshare® mixer.
 Gebr. Lödige Maschinenbau GmbH
 Elsenerstr. 7–9, D-33102 Paderborn, Germany.

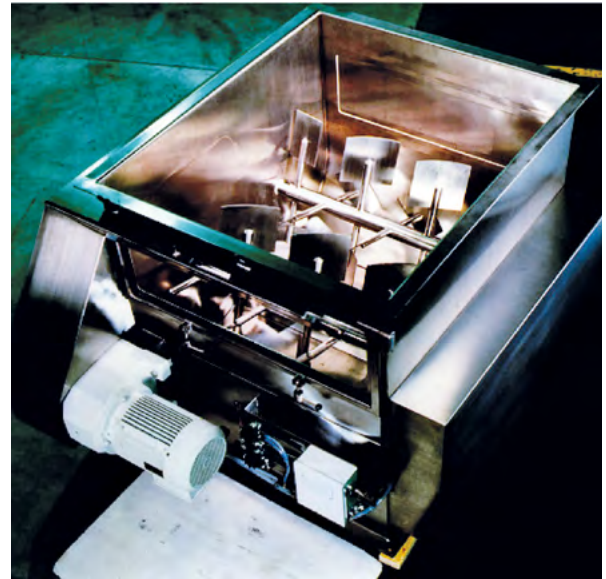


Figure 2: View of a double paddle mixer,
 Halvor Forberg A/S Hegdal, N-3261 Larvik, Norway



Figure 3: Schematic illustration of a Nauta® Mixer
 Hosokawa Micra B.V. Gildenstraat 26,
 NL-7000 AB-Doetichem, The Netherlands

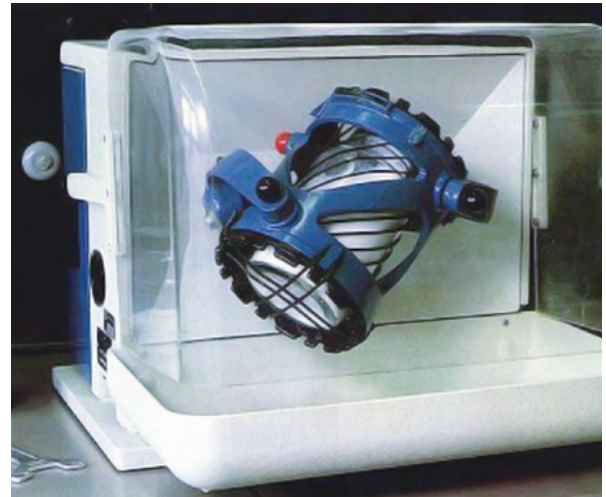


Figure 4: Laboratory example of a tumbler, a so-called
 "TURBULA® Mixer". Willy A. Bachofen AG., Maschinenfabrik.
 Utengasse 15/1, CH-4005 Basle, Switzerland

3 Silica as flow aid or anticaking agent

3.1 Targets of mixing

A lot of powdery substances tend to cake and agglomerate. This makes it difficult to handle these materials especially when conveying, feeding, packaging and storing. Increasing automation requires powdered substances that display good flow behaviour, as well as storage stability. Fluctuating ambient conditions like temperature, humidity or pressure and other factors such as crystallization and sintering processes, moisture uptake, irregular particle shape, etc. can contribute to increased caking of powders.

The addition of silica provides good flowability and reduced caking behaviour. Fine silica particles act as spacer, keeping host powder particles apart from each other and avoid agglomeration. Furthermore they absorb liquids from the surface of host particles and some grades additionally may eliminate electrostatic charging. However, to develop the optimum performance the silica needs to be dispersed in a way that allows covering the host particles.

3.2 Mixing of silica

The degree of dispersion of silica in a host powder is influenced by different parameters. In addition to the silica properties themselves the mixing conditions have a major effect. The following mixing parameters should be noted:

- Mixing time
- Mixing speed (\Rightarrow shear stress)
- Order of addition (e.g. first host powder, then silica; sequential or continuous dosing of silica...)
- Type of mixing system (pneumatically, mechanical, etc.)

In order to give a recommendation on mixing silica the process parameters need to be considered in detail. The following rules of thumb may serve as a first approach.

- Each silica grade has its own optimum mixing intensity, which is needed to reach the best possible performance. Mixing intensity is the shear stress applied for a defined period of time. Too low intensity results in a low coverage of the host powders, too high mixing intensity will cause densification of the silica structure.
- If moisture causes bad flow, gentle mixing is advantageous.
- Dry powders need a more intense mixing to reach the best dispersion of silica.
- The mixing intensity also has to be adjusted to the hardness of the host powder particles as elevated mixing intensity ($>$ speed or time) may force the silica to penetrate into soft host powder particles and reduce their efficiency.
- Hydrophobic silica grades are very efficient and much easier to distribute than hydrophilic grades. They need less mixing intensity and are recommended for moisture sensitive host powders.

Moreover the condition of the host powder itself is sometimes affected by the mixing step. Depending on the kind of movement during mixing agglomeration or deagglomeration can take place. Tumbler mixers (e.g. rotary drum mixers) cause dry but very fine sized powders like titanium dioxide to agglomerate, medium intensive mechanical mixer (e.g. plough share mixer) don't.

Agglomerated host powders or fractal structures may require higher addition levels as silica fills the asperities rather than being available to cover the outer surface of the host powder.

Table 1 shows a selection of products and product groups which have already been successfully treated with Evonik's silica products to improve flow and caking properties.

Table 1

Application areas of SIPERNAT® and AEROSIL® grades for improving flow and caking properties

Product	Addition Level [%]	Evonik Flow Agent
Acrylic polymers	0.1 – 0.2%	SIPERNAT® 22 S, D 10 AEROSIL® R 812
Cellulose ethers	0.5 – 1.0%	SIPERNAT® 22 S, 50 S or 320
Fertilizers	0.1 – 2.0%	SIPERNAT® 22 S
Fire extinguishing powders	0.5 – 1.5%	SIPERNAT® D 17 AEROSIL® R 972
Granulates, powders and dusts in agrochemical applications	0.5 – 3.0%	SIPERNAT® 22 S SIPERNAT® 50 S
Impact modifiers	0.2 – 1.0%	SIPERNAT® D 17, D 10
Inorganic salts	0.5 – 3.0% 0.1 – 0.5%	SIPERNAT® 22 S, 50 S SIPERNAT® D 17
Milk replacer	0.5 – 2.0%	SIPERNAT® 22 S
Mineral- and vitamin premixes	0.2 – 3.0%	SIPERNAT® 22 S, 50 S
Pigments	1.0 – 2.0%	AEROSIL® R 812, R 8200 or R 972
Powder coatings	0.05 – 0.2%	AEROSIL® 200 or SIPERNAT® 22 S, AEROSIL® R 972, AEROXIDE® Alu C
Redispersible powders	0.5 – 3.0% 0.2 – 1.0%	SIPERNAT® 22 S, 50 S SIPERNAT® D 17
Spice mixtures	0.5 – 2.0%	SIPERNAT® 22 S AEROSIL® 200
Thermoplastic elastomers (TPE, TPU)	0.5 – 1.0%	AEROSIL® R 972 or 200 SIPERNAT® 350

4 Silica for spray dried powders

SIPERNAT® and AEROSIL® types can facilitate the spray drying process for oily or sticky materials. They function by preventing these powders from adhering to the side walls of the spray dryer and by absorbing oils or moisture from the surface of the powder particles during the drying process. This can greatly improve the dryer's efficiency and the flow of the final product. The dryer will stay clean longer reducing shut down time. The powder properties are improved as well after drying. Materials are more free flowing and less prone to caking.

Dosing of SIPERNAT® specialty silica or AEROSIL® fumed silica into a spray drying process (figure 7) may be done in several ways and at different dosing points:

1. through the top
 2. through a window, if available
 3. with the hot air stream
 4. by external after-mixing
- Adding the silica through the top (#1) or through a window or inlet opening (#2) will assure the best and most efficient performance. For application a pipe of about 50 mm diameter is led through the corresponding opening ending about 20–50 cm below the wet

zone, depending on the size of the drier. It should be slightly bent in the direction of the air flow. Due to the negative pressure in the drier, the silica is sucked into the tower. At this point the silica can directly coat the freshly dried particles, before they come into close contact. The silica will also most effectively prevent caking of the particles on the side walls or to each other.

- Adding the silica in the hot air supply (#3) is also a good strategy. While not quite as effective as the first two options, it will still yield good results in most cases.
- An alternative to the aforementioned dosing into the spray drier is a treatment directly after drying. Post-mixing is only suggested when the material can be sprayed and dried without difficulty, but flow agent is desired to help convey the material into other processing or into the packaging system. However, this means an additional mixing step.

Overall it can be stated that the earlier a powder is treated with SIPERNAT® specialty silica or AEROSIL® fumed silica the more effective this treatment is.

Figure 5 shows a typical spray drier with the corresponding dosing points.

5 Silica as carrier

While **table 1** provides examples for the improvement of flow properties through addition of AEROSIL® and SIPERNAT® products, **table 2** highlights some applications where liquid, semi-solid or pasty substances are converted into powders through the addition of silica. In these applications, the silica serve as carriers.

Table 2
Application areas of AEROSIL® and SIPERNAT® grades as carrier

Absorbate	Addition Level [%]	Evonik Carrier Silica
Antioxidants	25 – 35	SIPERNAT® 22 or 50
Choline chloride 50 %	33	SIPERNAT® 22
Melamine resins	33 – 35 26 – 29	SIPERNAT® 22 SIPERNAT® 33
Peroxides	33 – 40	SIPERNAT® 22 or 320
Powder defoamers	25 – 35	SIPERNAT® 22, 33, 50 or 2200
Rubber and plastic additives	25 – 35	SIPERNAT® 22 (S), 33 or 50 (S)
Vitamin E-acetate 50 %	47 – 49	SIPERNAT® 2200
Vegetable extracts in pharmaceuticals	33	AEROSIL® 200 Pharma, AEROPERL® 300/30 Pharma
Wettable powders (WP) and water dispersible granulates (WDG) for agro-chemicals	5 – 35	SIPERNAT® 22, 22 S, 50, 50 S

5.1 Mixing techniques for carrier applications

Generally, the carrier silica should be put into the mixer first. The substance to be converted to powder form should then be added in the next step. Liquids should be added continuously and as finely distributed as possible. In some special cases of substances which have been liquefied through increased temperature, improved results can be obtained in case the silica is added to the liquefied substance.

When AEROSIL® and SIPERNAT® products are employed as carriers, gentle mixing is required. Optimum results cannot be obtained with high pressure or high shear forces, because they partly destroy the porous structure, thus pressing out the absorbed liquid.

Figure 6 shows a choline chloride formula with 67% choline chloride solution (75%) absorbed on 33% SIPERNAT® 22 by weight, produced at low shear and thus resulting in good flow behaviour. This material can easily be packed, transported and reprocessed.

Figure 5
Spray drier with possible dosing points

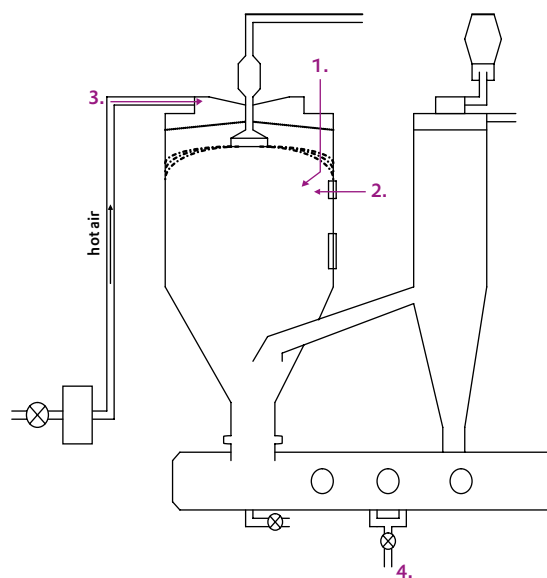


Figure 7 shows a caked choline chloride absorbate with the same composition, but from a mixer with high shear forces.

To optimize flow behaviour and pressure stability of absorbates, the liquid should be introduced as finely divided as possible. The best method is to atomize or spray the liquid. In any case, dripping is still better than pouring. Ploughshare® and paddle mixers as mentioned in section 2 are very well suited to perform these kinds of mixing tasks. They have short mixing times and very low shear forces. In these mixer types, the porous structure of the carrier silica and the high absorptive capacity remain fully intact.

In some modern mixer types, there is the possibility of supplementing the low shear force mixing blades with additional high intensity mixing devices inside the mixing chamber, so-called chopper blades or intensifiers (see **figure 8**). Using these devices leads to a very high energy input into the mixture, which can cause the fluid taken up by the silica to be partially pressed back out of the carrier. With this type of mixing equipment, part of the porous structure of highly absorbent carrier silica can be destroyed irreversibly resulting in a reduced absorption capacity. Microgranular silica structures can even be broken down within a short time with simultaneous formation of fine particles or dust. The use of high intensity mixing tools for a longer mixing time is therefore not recommended.



Figure 6: Freely flowing 50% choline chloride absorbate based on SIPERNAT® 22



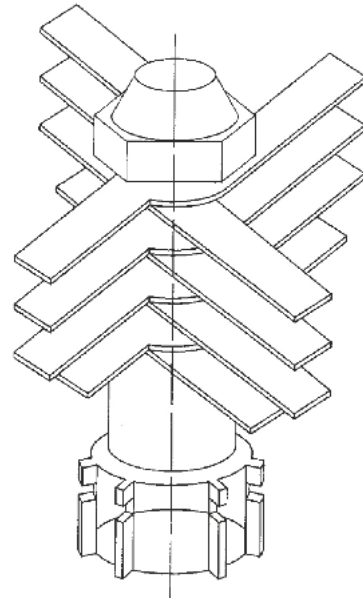
Figure 7: Choline chloride paste created by shearing of the formula shown in figure 8

6 Summary

When using SIPERNAT® and AEROSIL® grades as carrier or as flow agent, various factors have to be taken into consideration with respect to the porous particle structure. The mixing techniques have a significant influence on the quality of the finished product as described in this brochure. Besides this the experts of Evonik's Applied Technology Department are prepared to find the best solution for your challenges. This may include lab testing with different silica grades, levels, and/or mixing tools, as well as testing of storage stability.

Figure 8

Schematic diagram of chopper blades, or intensifier



7 Bibliography

- (1) Vauck, Müller
Grundoperationen chemischer Verfahrenstechnik,
9th Edition, 1972
- (2) Harnby, Edwards, Niemow
Mixing in the Process Industries, Second Edition, 1992
- (3) Prof. Dr.-Ing. Karl Sommer
Hochschulkurs Feststoffmischen, Freising 1998
- (4) Prof. Dr.-Ing. P. Schmidt
Veranstaltungsunterlagen Seminar Mischtechnik, University
GH Essen, 1988
- (5) Technical Information TI 1351
SIPERNAT® and AEROSIL® as Flow Aid and Anticaking Agent,
Evonik Resource Efficiency GmbH
- (6) Technical Information TI 1360
SIPERNAT® and AEROSIL® – an Essential in Industrial Powder
Technology, Evonik Resource Efficiency GmbH

This information and any recommendations, technical or otherwise, are presented in good faith and believed to be correct as of the date prepared. Recipients of this information and recommendations must make their own determination as to its suitability for their purposes. In no event shall Evonik assume liability for damages or losses of any kind or nature that result from the use of or reliance upon this information and recommendations. EVONIK EXPRESSLY DISCLAIMS ANY REPRESENTATIONS AND WARRANTIES OF ANY KIND, WHETHER EXPRESS OR IMPLIED, AS TO THE ACCURACY, COMPLETENESS, NON-INFRINGEMENT, MERCHANTABILITY AND/OR FITNESS FOR A PARTICULAR PURPOSE (EVEN IF EVONIK IS AWARE OF SUCH PURPOSE) WITH RESPECT TO ANY INFORMATION AND RECOMMENDATIONS PROVIDED. Reference to any trade names used by other companies is neither a recommendation nor an endorsement of the corresponding product, and does not imply that similar products could not be used. Evonik reserves the right to make any changes to the information and/or recommendations at any time, without prior or subsequent notice.

AEROSIL® and SIPERNAT® are registered trademarks of Evonik Industries or its subsidiaries.



EVONIK
INDUSTRIES

**Europe/Middle-East/
Africa/Latin America**

Evonik Resource Efficiency GmbH

Business Line Silica
Rodenbacher Chaussee 4
63457 Hanau-Wolfgang
Germany

PHONE +49 6181 59-8118

FAX +49 6181 59-78118

ask-si@evonik.com

www.evonik.com

North America

Evonik Corporation

Business Line Silica
299 Jefferson Road
Parsippany, NJ 07054
USA

PHONE +1 800 233-8052

FAX +1 973 929-8502

ask-si-nafta@evonik.com

Asia / Pacific

Evonik (SEA) Pte. Ltd.

Business Line Silica
3 International Business Park
#07-18 Nordic European Centre
Singapore 609927

PHONE +65 6 809-6877

FAX +65 6 809-6677

ask-si-asia@evonik.com

Evonik. Power to create.