

TECHNICAL INFORMATION 1417

# AEROSIL® and SIPERNAT® Products in Powder Detergents



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# 1. Introduction: Adapting to Shifting Trends in Powder Detergents

Globally, powder detergents remain the most popular product form for cleaning garments. Yet, even powders must evolve to accommodate stringent demands of the modern consumer. The formulation of products that deliver on performance and also support initiatives to reduce environmental impact are critical. For both large and small powder detergent manufacturers, challenges include ease-of-use, compaction, concentration and cleaning performance.

Driven by consumer perception and retailer push, package reduction has become one of the most critical areas of research so as to comply with the “green” movement, while empowering the consumer to work with a more concentrated formulation. The rise of single-dose forms that aim to deliver concentrated, easy-to-use products lead to less time spent on laundry, while maintaining quality of the wash.

To address these emerging trends, the next generation of raw materials must not only allow for higher concentrations, but also enable a higher level of compatibility between formulation ingredients.

Evonik has worked to actively pursue developments to alleviate these formulation hurdles. Our specialty silica line offers a wide range of products to provide various benefits, from higher surfactant loading in powders to improved shelf life.

Silica products manufactured under the SIPERNAT® and AEROSIL® brands can play an important role in the modern detergent formula. Focusing specifically on detergent powders, silica can greatly increase overall surfactant content while reducing packaging size. In addition, SIPERNAT® and AEROSIL® grades can improve the flowability of powder detergents and prevent them from caking or forming lumps during storage.

By capitalizing on the extensive absorption capacity of SIPERNAT® specialty silica, the processing, form and appearance of a finished product can be improved. Table 1 associates applications of Evonik silica particles to powder detergent formulation needs.

Table 1

Formulation Needs	Silica Application
Concentrated detergents	Carrier
Improved process efficiency	Free-flow
Robust shelf-life independent of region	Anti-caking

## 1.1 Benefits at a Glance



Free-Flow



Carrier



Anti-Caking



Spray Drying – © GEA Niro GmbH

## 2. SIPERNAT® Products as Carrier for Surfactants, Fragrances and Additives

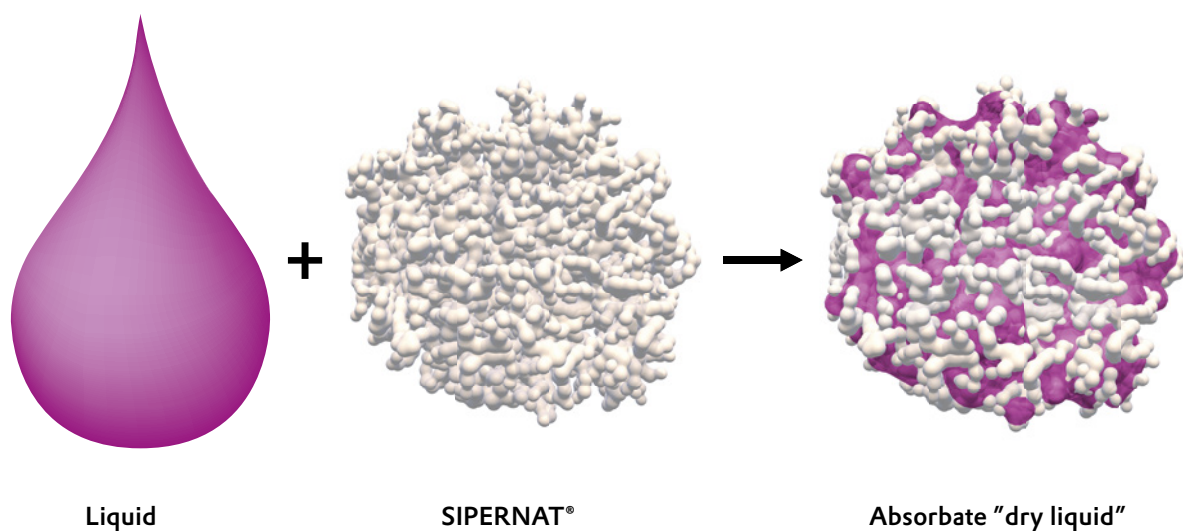
Formulation space in concentrated detergents is limited. The use of highly absorptive substrates like SIPERNAT® specialty silica facilitates the loading of liquid ingredients, while maintaining the appearance of the finished product.

The following depiction shows schematically the absorption of a liquid into the porous silica structure. The liquid is drawn into the pores by capillary forces.

From a formulation perspective, this can be accomplished through two methods:

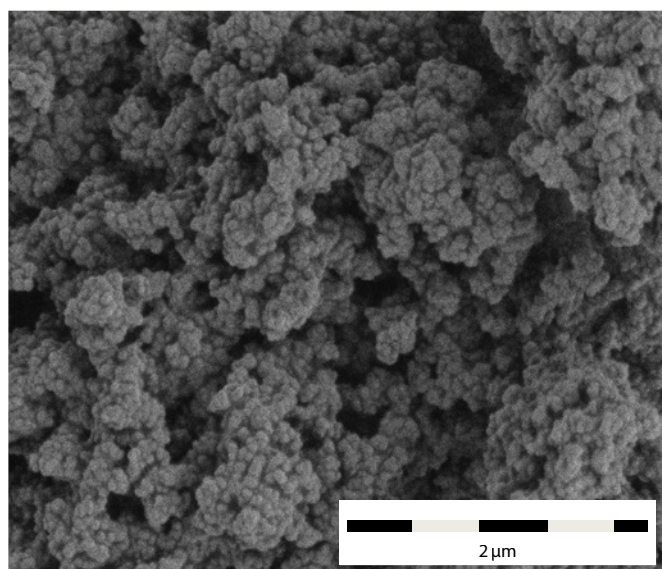
- Creating a surfactant absorbate using silica and dry-blending this with the rest of the detergent formulation
- Direct addition of silica along with the rest of the detergent components

**Figure 1**



Schematic depiction of the absorption process

**Figure 2**



SEM picture of the porous SIPERNAT® structure

Fragrances, enzymes and anti-redeposition polymers can also be loaded onto silica substrates in a similar way. This could provide increased detergent efficacy, while maintaining

compatibility between the ingredients of the formulation. Table 2 compares various types of SIPERNAT® silica with the properties most relevant to their performance as carriers.

**Table 2**

Analytical data of selected SIPERNAT® grades.

Product name	DOA Absorption in ml/100g following ISO 19246	Particle size (d50) in μm following ISO 13320-1	BET Surface Area in m <sup>2</sup> /g following ISO 9277	Tamped density in g/l following ISO 787-11
<b>SIPERNAT® 50</b>	290	40	500	175
<b>SIPERNAT® 50 S</b>	280	18	500	105
<b>SIPERNAT® 500 LS</b>	270	10.5	500	100
<b>SIPERNAT® 22</b>	240	120	190	260
<b>SIPERNAT® 22 S</b>	235	12.0	180	100
<b>SIPERNAT® 2200</b>	225	320	190	250
<b>SIPERNAT® 340</b>	235	20	175	160

The numbers are typical data; specifications on request.

The amount of liquid that can be absorbed varies between silica grades. The DOA (Dioctyl Adipate) absorption value allows users to compare absorption capacities of different SIPERNAT® grades. Furthermore, the physical properties of liquid components should be considered when optimizing a formulation containing silica.

While the DOA value gives insight into the relative loading capacity of a silica, surface area must also be considered, as it has a direct impact on its rate of absorption. Thus, optimization relative to the liquid component involving both silica surface area and DOA is necessary.

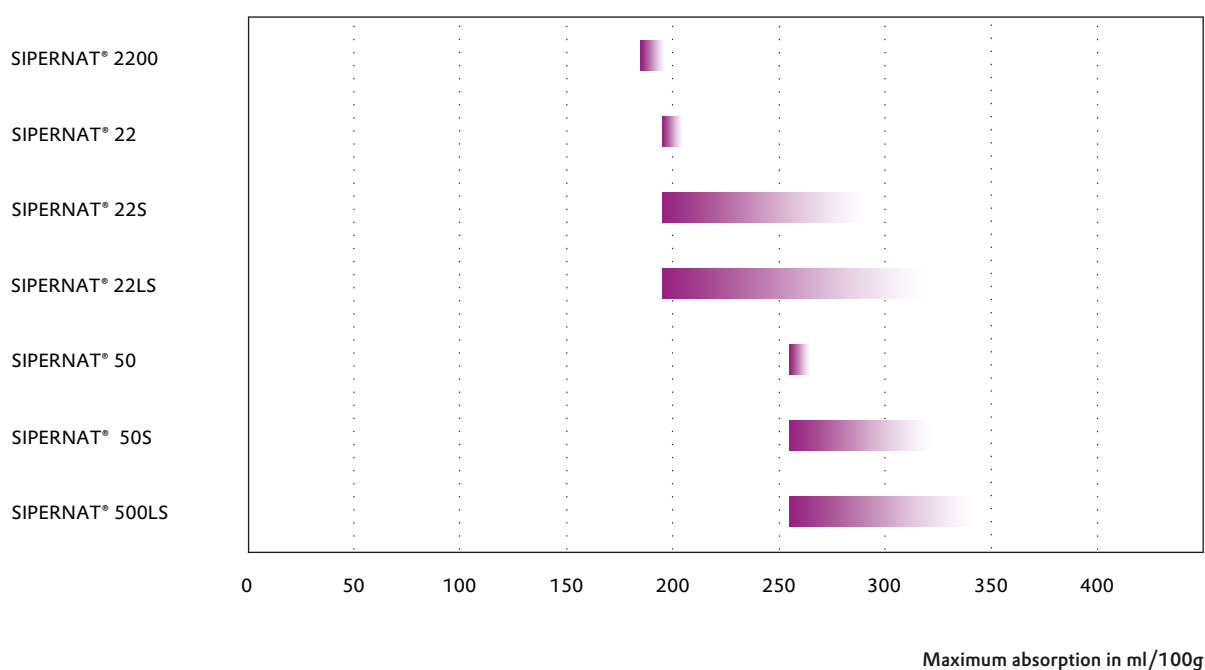
## 2.1 Carrier Processing

The DOA number gives a relative comparison of the absorption capacity of a silica only. The loading level that can be achieved in practice is also influenced by the mixing process. The following graph (Figure 3) shows the range of loading levels that can be achieved depending on the mixing conditions. A model liquid was mixed with the silica with different shear intensities. The maximum absorption given on the x-axis is the volume of liquid that can be absorbed before the liquid leaks out and the absorbate becomes wet. With appropriate mixing technology, it is possible to agglomerate the fine particle silica during the absorption process in a controlled way, resulting in higher absorption capacities. The left end of

the bars in the diagram symbolizes the capacity that can be achieved with regular mixing, the fading right end symbolizes the higher capacities that can be achieved with a very gentle and more sophisticated mixing. This higher absorption capacity might be lost when high shear forces are applied, depending on the SIPERNAT® grade which is used.

Furthermore, the liquid should be dosed uniformly during production. Other parameters such as silica to surfactant ratio, liquid temperature (since this affects the viscosity), mixing duration and speed should all be optimized for each individual liquid.

**Figure 3**



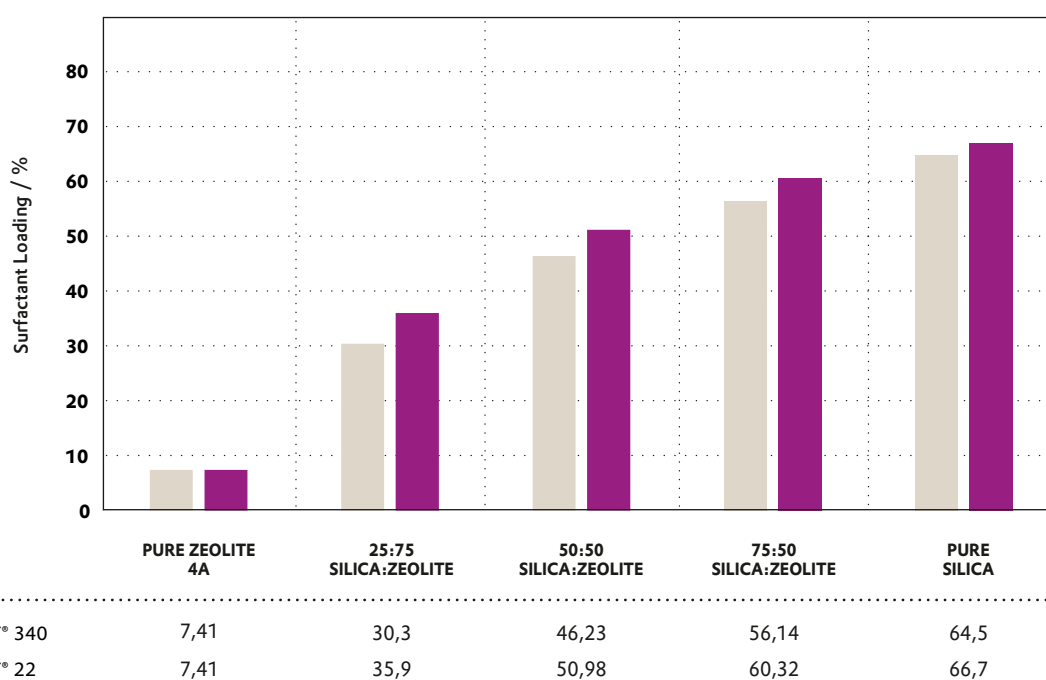
Absorption capacity of different silica; range depending on mixing conditions

## 2.2 Synergies of SIPERNAT® Silica and Builders

Builders such as zeolites are commonly used in detergents to reduce the hardness of the wash water, which improves the detergency of surfactants. These aluminosilicates have long been the standard formulation option for conventional detergents. However, their small size and low absorption makes

them poor carriers and restricts the quantity of liquid surfactant that can be added. By substituting zeolites with different precipitated silica grades, a dramatic increase in surfactant loading can be achieved, along with a significant improvement of powder characteristics.

**Figure 4**

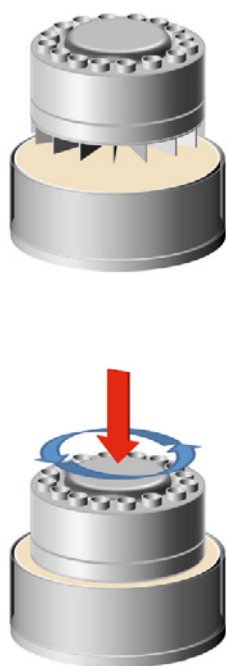


Example surfactant absorption data for blends of different silica with zeolite 4A (Valfor® 100 from PQ corporation)

Figure 4 shows the increase in loading of a non-ionic surfactant (an alcohol alkoxylate CAS# 68154-97-2) when two different grades, SIPERNAT® 340 and SIPERNAT® 22, are blended with a zeolite at different weight ratios. Dry powder blends of the SIPERNAT® grades and zeolite were created at different wt. ratios by mixing on a Turbula™ mixer for 15 minutes. The non-ionic surfactant was added to

these blends intermittently at a rate of 1 g/min at a low mixing speed of ~100 rpm on a Somakon™ mixer. The maximum loading capacity was evaluated based on the flowability of the carrier mixture. This was evaluated by a combination of visual detection of the saturation point and confirmed using powder rheology testing.

**Figure 5**

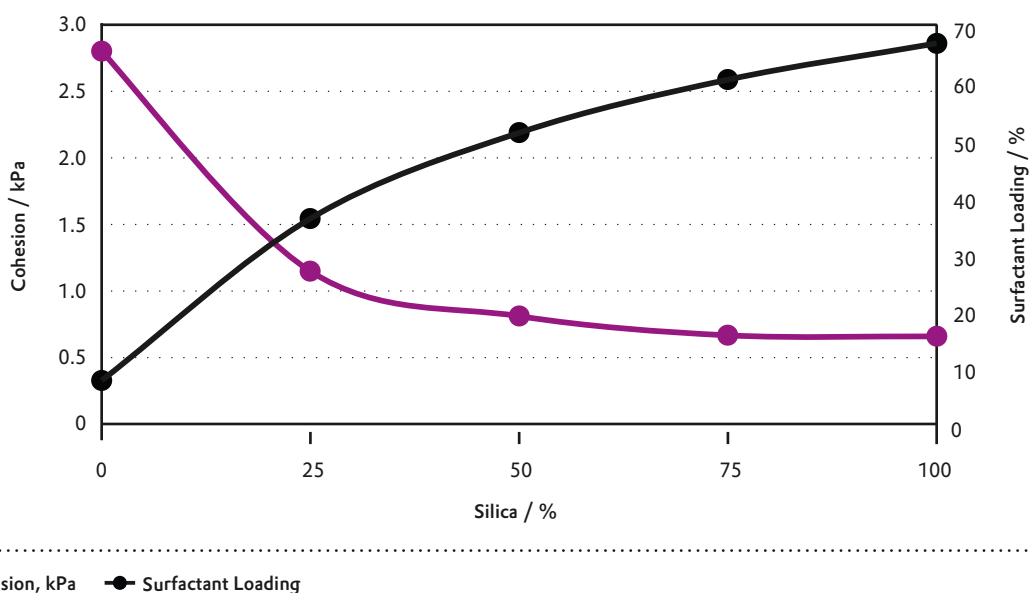


The benefits of adding silica to the carrier blends are characterized using powder rheology testing. Specifically, Shear Cell analysis is used to measure the cohesiveness, or “stickiness”, of the powder. This is done by applying a low shear force to the top layer of the powder bed, while keeping the bottom layer stationary. The shear force is increased until the powder bed yields and the upper layer slips against the bottom layer. Higher forces indicate a more cohesive powder.

Figure 6 below shows Shear Cell test data on various Zeolite 4A/SIPERNAT® 340 carrier blends. Prior to shearing the powder, a pre-defined compression force is applied to each sample. This allows for the determination of the relative cohesiveness of the various blends. As the silica concentration goes up, we see a noticeable increase in the amount of surfactant that can be loaded onto the blend. Furthermore, the graph shows how the powder cohesiveness, or stickiness, drops with increasing silica content, while achieving a much higher surfactant loading. This would equate to a more concentrated finished product, while maintaining a robust free-flowing powder.

Schematic of Shear Cell analysis – Images reproduced with the permission of Freeman Technology Ltd

**Figure 6**



Cohesion and surfactant loading as a function of silica wt. %

### 2.3 Dispersibility and Release

Dispersibility is an important consideration in concentrated detergents due to higher surfactant loading. The detergent must disperse readily and release the surfactants.

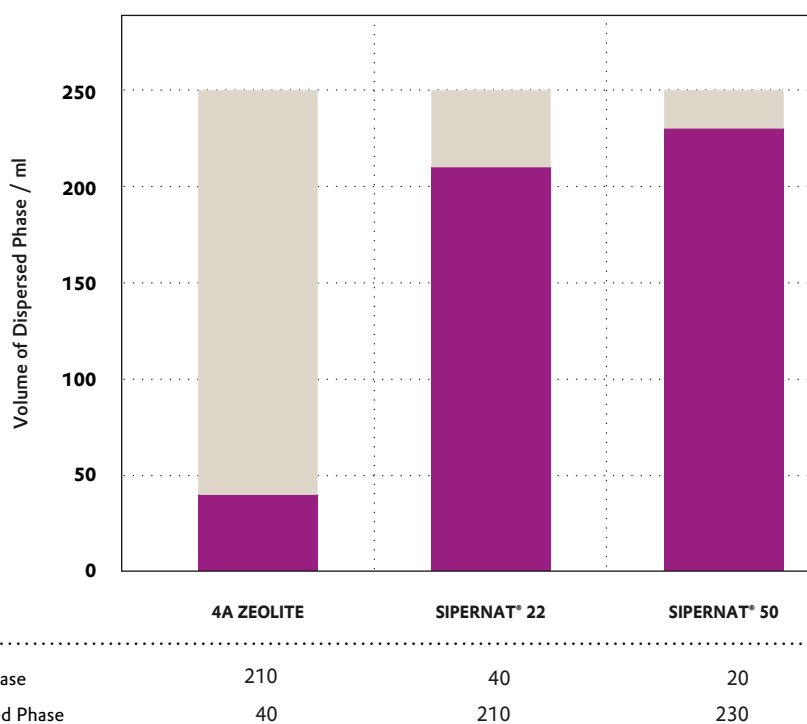
Figure 7 (below) shows results from a dispersibility test.

The carriers in these studies were made by mixing the powder blends at 500 rpm on the Somakon™ mixer and the surfactant (an alcohol ethoxylate) was added over 2 minutes.

The surfactants were heated to 60 °C for optimal processing. The SIPERNAT® 22 carrier was made at 64.2 % and the SIPERNAT® 50 carrier was made at 68.7 %.

Dispersibility was determined by taking 5 g of the silica carriers and mixing it with 250 ml of water at 4200 rpm for one minute. The settling behavior of the carriers were visually observed in graduated cylinders.

**Figure 7**



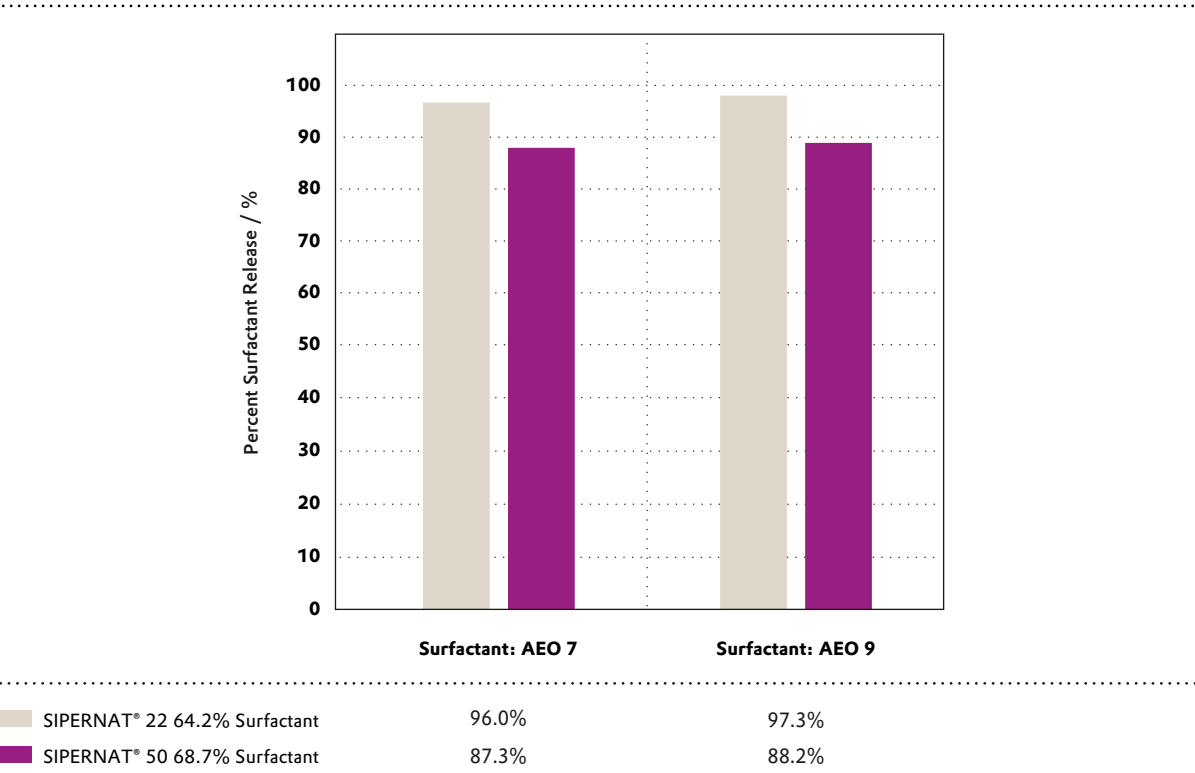
Dispersibility of surfactant carriers in water

Once liquid is incorporated into carriers, it can be released by displacement, wherein a liquid with a higher affinity for the silica displaces the liquid being carried in detergent systems. In a detergent carrier, the wash water permeates the hydrophilic surface of the SIPERNAT® silica and saturates the porous network, triggering the release of the contained active. A critical parameter in this context is the ability of the silica to release liquids efficiently with respect to both rate and completeness. This will have a direct impact on detergent performance, since incomplete release would result in less surfactant available in the wash.

Figure 8 below highlights the effective release of surfactants from various SIPERNAT® 22 and SIPERNAT® 50 precipitated silica grades. It is critical that absorptive substrates show complete release under wash conditions.

The release of AEO 7 & 9-loaded silica carriers was determined by dissolving 2 g of the carriers in 1 liter of deionized water at 60 °C and stirring at 350 rpm for 3 minutes. The silica was filtered and dried at 105 °C for 2 hours. The surfactant release was determined gravimetrically.

Figure 8



Release efficiency of surfactant carriers

### 3. SIPERNAT® Products to Ensure Flowability and Prevent Caking of Detergents

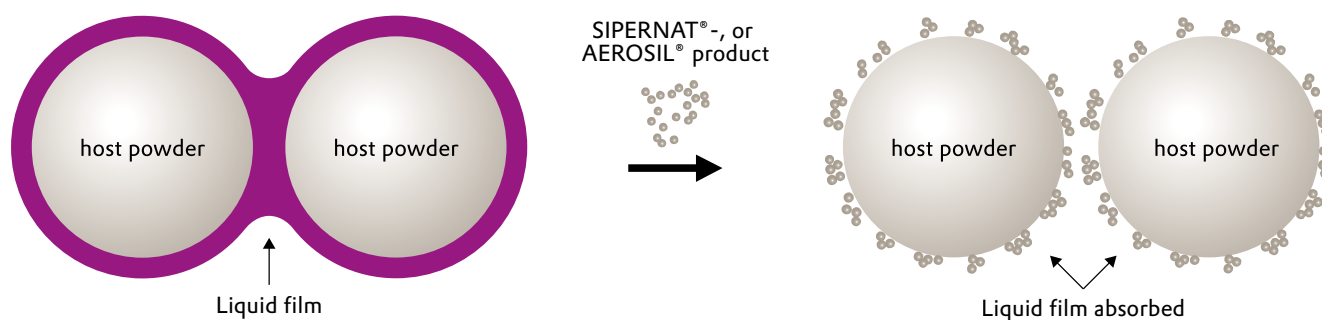
In detergent formulations, SIPERNAT® and AEROSIL® grades inhibit the formation of hard agglomerates or clumps. This ensures good flowability that subsequently leads to improved handling and processing of the powder. The shelf life of the finished product is also improved through the elimination of issues such as hardness and sensitivity to moisture in storage.

Flow aids like SIPERNAT® precipitated silica and AEROSIL® fumed silica are fine powders that can coat the surfaces of a host powder to create surface roughness, which reduces

inter-particle attractive forces. They also reduce tackiness of the finished product by functioning as spacers, keeping sticky host powder particles isolated from each other.

Residual liquids in a formulation can also cause processing and flow issues. The high absorptivity of SIPERNAT® allows for the disruption of liquid bridging between host particles, preventing clumps from forming in the detergent powder. As a result, particle size distribution of the final formulation can be tightly controlled during manufacturing and in storage.

Figure 9



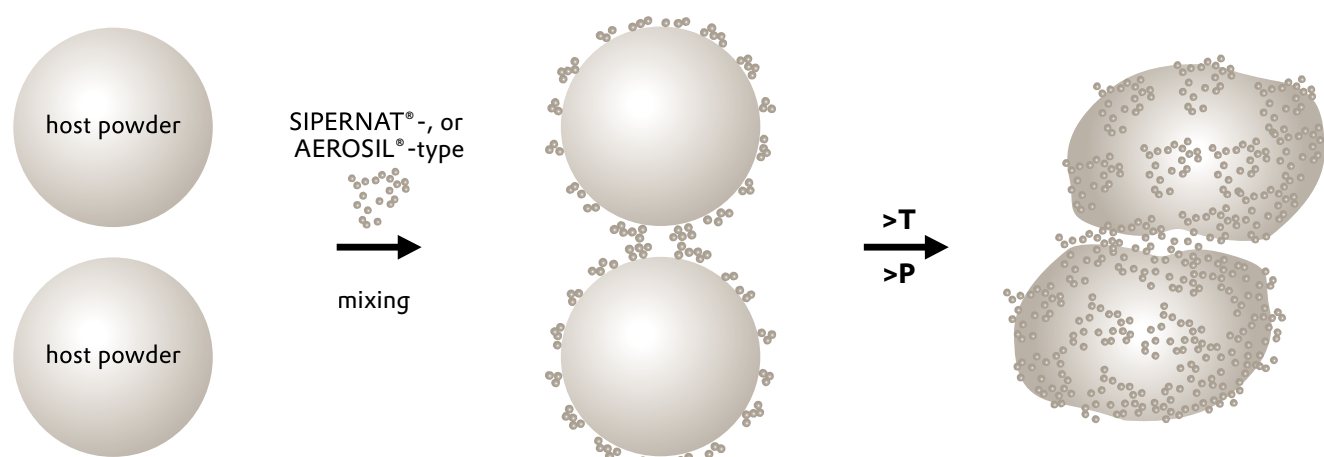
Silica absorbing liquid from the surface of a host powder

Other factors which affect flowability of detergents include:

- **Particle size:** Fine powders tend to cake more than coarse materials
- **Particle shape:** Irregular shape can create particle interlocking
- **Plasticity:** Malleable, soft powders stick at high temperature and pressure conditions
- **Electrostatic charging:** Charged particles stick to the inner walls of containers and hoppers

The following figure shows how silica can reduce the caking tendency of a malleable powder by covering the surface of the powder particles.

**Figure 10**



Schematic depiction of anticaking effect

### Detergent test formulation

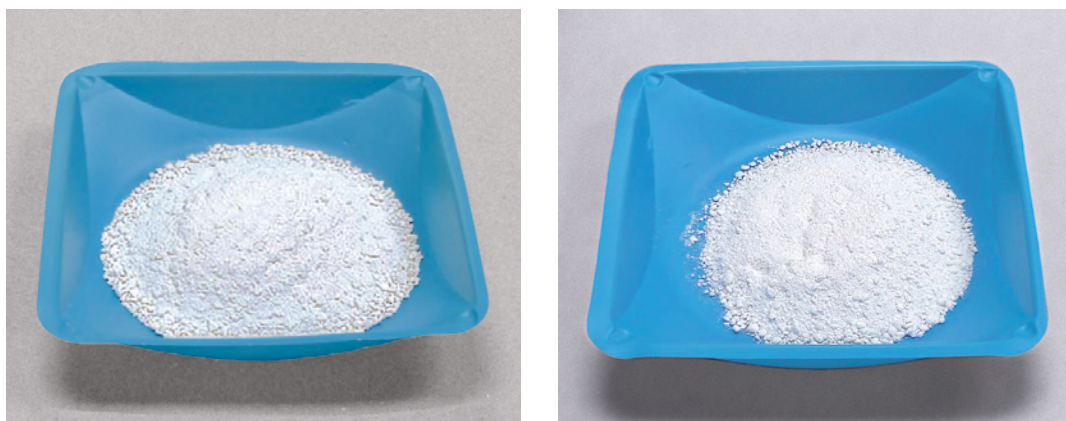
To show the benefit of a carrier silica, a formula (shown in Table 3 below), containing a mixture of surfactants along with other powder ingredients such as zeolites, silicates and calcium sulfate, was evaluated.

**Table 3**

Ingredients	Standard Detergent (wt. %)	Concentrated Detergent (wt. %)
Zeolite 4A (Valfor <sup>®</sup> 100)	30.0	30.0
Hydrous Polysilicate	7.0	7.0
Sodium Carbonate	15.0	15.0
C12-C15 linear alcohol, 7 moles EO	4.0	10.0
Linear alkylbenzene sulfonic acid	10.0	10.0
Carboxymethylcellulose	1.0	1.0
Sodium Sulfate	33.0	25.0
SIPERNAT <sup>®</sup> 22	-	2.0

Example formulations for detergent testing

**Figure 11**



Left – Standard detergent with ~14 % surfactant and no silica, Right – Concentrated detergent with 20 % surfactant and 2% SIPERNAT® 22.  
The samples look similar even though there is a ~42 % increase in surfactant loading

Figure 11 above shows the two model formulations. The image on the left shows the standard detergent formulation containing a total of 14 % by wt. of surfactant in the formulation without any silica. The image on the right shows the

concentrated formulation containing 20 % by wt. of surfactant. This represents ~42 % increase in surfactant concentration while maintaining flowability.

#### **4. SIPERNAT® and AEROSIL® Products as Spray Drying Aids**

Spray drying, as implemented in the manufacturing of detergent powders, is typically achieved by first combining the surfactants, sodium silicate, additives and fillers with water. This slurry is then spray-dried under high temperatures, yielding the base powder.

During spray drying processes, various problems including wall deposition of powder, low product yields, low product quality, high maintenance efforts and frequent shut down for cleaning can occur.

At Evonik, extensive time and effort has been invested into researching the effects of silica materials in spray drying processes. Internal studies have focused on determining the effects of silica type and its point of addition on the finished spray-dried powders. The results of these studies indicate that optimum performance can be achieved by addition of the silica at the top of the spray tower, close to the atomizer, but separate from the slurry.

Additional details can be found in our Technical Information 1365.

## 5. Methodology to Determine Flow and Carrier Characteristics

When processing powders, it is important to understand the factors that affect flowability and caking. Flowability measurements determine whether a powder can be conveyed easily and dosed accurately. Caking test describes whether the powder remains free flowing during storage and transport. The tendency of a powder to form dust is another important aspect that is especially relevant when packing or emptying bags. The following section highlights equipment that are used routinely for powder analysis.

### 5.1 Powder Rheology Using the FT4

Powders are complex materials that contain multiple, distinct phases of solid particles, liquid additives, air entrapped between particles, and, in most cases, moisture from the atmosphere. As a result, they exhibit complex behaviors that require specialized testing procedures for complete characterization. Powder rheology techniques allow us to address these complexities and quantify the effect of silica in detergent powders.

The FT4 Powder Rheometer by Freeman Technology (UK) has emerged as a versatile tool in powder testing. A number of tests such as Stability and Variable Flow Rate, Shear Cell, Permeability, Aeration, Compressibility etc. enable a thorough understanding of a test powder and the effect of different additives, mixing and processing variables.

### 5.2 Flow Funnels

Alternatively, a quick and simple method to measure the flow grade can be achieved through the use of flow funnels. For this method, a defined set of flow funnels with different outlet diameters is used. The performance of how a powder flows through the funnel -whether it completely goes through or a blockage is formed- allows for its relative characterization.

Figure 12



FT4 Powder Rheometer - Image reproduced with the permission of Freeman Technology Ltd

### 5.3 Dust

One key objective when developing a powder formulation is to reduce or avoid dustiness of the material, especially when the dust generated could be toxic or flammable. Therefore, measuring the dust generated by the powder is important. This can be done using optical methods to determine the attenuation of light in a dust-laden atmosphere. Furthermore, the dust settling behavior over time can be determined.

Figure 13



Set of flow funnels with different outlet diameters

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