AEROSIL® – Fumed Silica

Technical Overview

Evonik. Power to create.

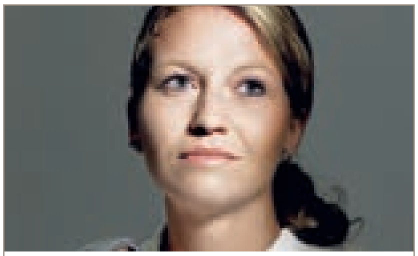
AEROSIL® – Fumed Silica

The series "Basic Characteristics and Applications of AEROSIL®" was first released in 1967. Part 1 of the series, entitled "Basic characteristics of AEROSIL® fumed silica", was later integrated into the Fine Particles series of publications, as text number 11.

The present work, entitled "AEROSIL® – Fumed Silica," is the eighth edition of this standard reference, and has been fully revised. It contains information on the physical and chemical properties of AEROSIL® and its potential applications.

AEROSIL® is a synthetic silica and the production of AEROSIL® began over 70 years ago. The AEROSIL® name is a registered trademark owned by Evonik Industries AG or its subsidiaries with 146 registrations in 99 countries across the globe. Evonik supplies customized fumed silica from various production sites worldwide.

AEROSIL® is the result of many things: past experience and a desire to push research forward, but above all a value-added product philosophy. This philosophy stands for a global production and sales network that provides superb customer proximity (and therefore optimal reliability and certainty), innovative technologies, a wide-ranging product portfolio, great innovative potential, and business relationships that span the long term, not to mention the continuous pursuit of improvement. Our motto: AEROSIL®. Invented to improve.



Inventing.

"As the inventor of fumed silica and as a driver of innovation, we offer the largest range of products. But the search for new and better applications continues. No matter how big the challenges are – we keep opening up new frontiers for our customers."

Improving the good.

"We offer customers more than just perfect surfaces. That's why AEROSIL® is not only a product, but also the fulfillment of many different customer and industry-specific requirements. To live up to this promise, we continuously strive to improve."

Customer proximity.

"We support our customers all over the world. More than 100 sales employees in the regions, global key account teams, local application engineers and industry-specific marketing teams provide direct, personal assistance."

Trust.

"In more than 70 years, we have proved that we are a reliable partner. Long-term business relationships and outstanding quality have made AEROSIL® the leading brand for fumed silica. We return the trust of our customers every day."

Mastering technology.

"Good products at the right time in the right place: With the innovative technologies that our teams in production, supply chain, research and applied technology stand for we offer customers solutions that are fine-tuned to suit their precise needs. And in doing so, we keep setting new benchmarks."

Security.

"Our global production and distribution network offers maximum safety and reliability to our customers. We deliver the required product at short notice, help with changes and are always committed to supporting our customers. Challenge us."

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All fired up about silica...

Introduction

- Natural occurrence
- Synthetic silicas

1. Introduction

1.1 Natural occurrence

The two most abundant elements in the earth's crust are oxygen and silicon (46.6 and 27.7 percent by weight). In nature, silicon is almost always combined with oxygen. Either exclusively (in SiO_2, for example) or in conjunction with additional elements (as is the case for silicates). Examples of silicates include bentonites (such as montmorillonite ($Al_{1.67}Mg_{0.33}$) $[(OH)_2/Si_4O_{10}]$ $Na_{0.33}(H_2O)_4$, talc $Mg_3[Si_4O_{10}(OH)_2]$, and wollastonite $Ca_3[Si_3O_9]$). The natural silicates form the basic raw material for key technical products such as cement, glass, porcelain, and bricks.

Silicon is also present in living things. There are a number of organisms that create structures containing silicon dioxide. The most well-known examples are diatoms (a type of algae), radiolarians (plankton), and hexactinellae (glass sponges).

There are also many plants that contain silicon dioxide in their stems and leaves. Well-known examples include horsetail and bamboo. These plants build a frame from silicon dioxide to achieve greater stability.

Dissolved silica is also present in drinking water and other drinks such as beer (which gets its silicon dioxide from barley). People and animals consume silica as part of their natural diet.

Pure silicon dioxide can occur in amorphous or crystalline form. The main modifications of SiO_2 that occur in nature are listed in table 1.

There are also many other structures in which SiO_2 can occur. For a detailed overview, see the introduction for "silica" in Ullmann's Encyclopedia of Industrial Chemistry [4].

1. Crystalline	
Quartz	Most common modification, pure quartz, quartz sand
Tridymite	Forms at higher temperatures.
Cristobalite	Forms at higher temperatures.
Coesite	High-pressure modification, very rare in nature
Stishovite	High-pressure modification, very rare in nature

2. Amorphous	
Lechatelierite	Natural SiO_2 glass formed by melting as the result of a lightning strike, rare
Opal	Not pure SiO_2, contains water
Kieselguhr	Comes from the SiO_2 in the remains of prehistoric infusoria and diatoms, always impure

Table 1: Modifications of naturally occurring SiO_2

1.2 Synthetic amorphous silicas

1.2.1 Classified by manufacturing process

In the modern world, synthetic silicas are used in many applications, from simple processes and products to highly technical ones.

Different manufacturing processes create SiO_2 products with different properties. The differences in these products are discussed in more detail in section 1.2.3. Figure 1 shows a basic overview of the various types of manufacturing process. SiO_2 products are also divided up between untreated products and those that have undergone chemical/physical treatment.

According to the SRI study "Silicates and Silicas," October 2011, 144,000 metric tons of fumed silica and 1,400,000 metric tons of precipitated silica were produced worldwide in 2010 [5].

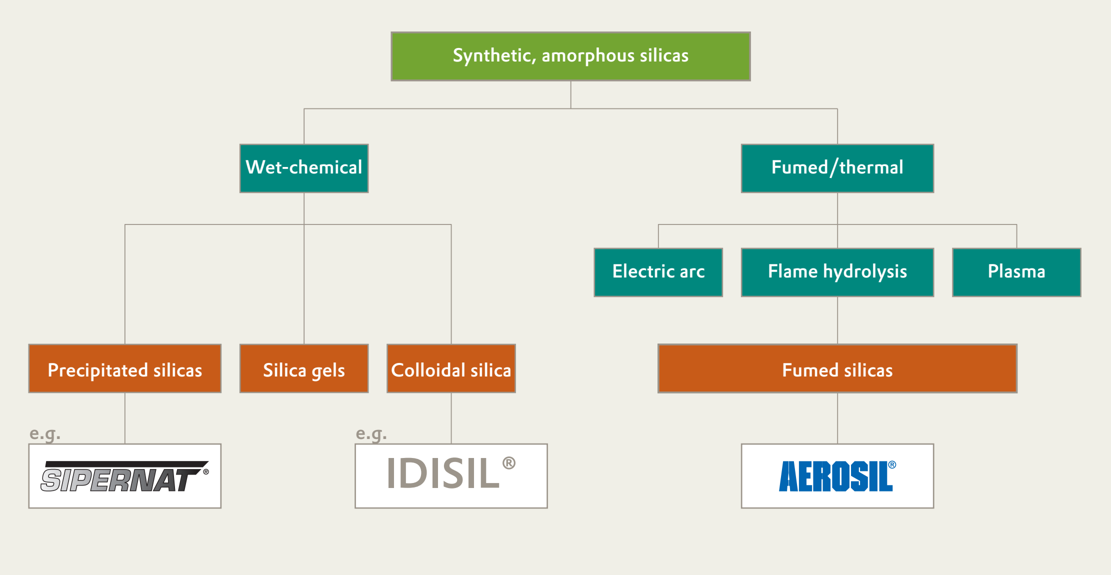


Figure 1: Overview of various manufacturing processes for producing silicas

1.2.2 Silicas produced by Evonik

All SiO_2 products manufactured by Evonik are synthetically produced and X-ray amorphous, as shown in figure 2 with AEROSIL® 200 as an example. The main property of AEROSIL® synthetic amorphous silicas is their lack of a crystalline structure.

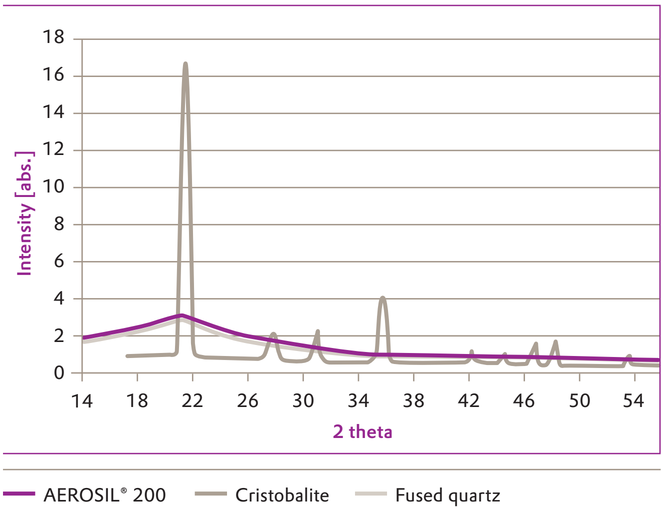
The standard method is X-ray diffraction using an increased percentage of crystalline materials. The detection limit for this method is below 0.05 percent by weight. All silicas produced by Evonik have values below this limit. Accordingly, they are all deemed fully amorphous.

Evonik uses a variety of precipitation methods and flame-based processes to manufacture SiO_2 products.

The oxides that are generated in the flame-based processes are referred to as fumed oxides. These materials include the fumed silicon dioxides that are produced by Evonik and marketed under the trade name AEROSIL®.

The flame hydrolysis method (high-temperature hydrolysis/AEROSIL-process) was conceived and technically developed by Harry Kloepfer, a chemist working for Degussa AG (which went on to become Evonik Industries AG). The aim of his development and research work was to develop a white filler material for the tire industry to replace industrial carbon black. He discovered a way to produce silica with extremely fine particles from $SiCl_4$ via high-temperature flame hydrolysis. This silica was later given the brand name AEROSIL® fumed silica. The first small-scale production took place in 1941. Today, fumed silicas are produced and marketed at various locations worldwide.

Figure 2



X-ray diffraction diagram showing the differences in structure between AEROSIL® and cristobalite. Silica glass has an equally "irregular" structure.

To manufacture AEROSIL® products, Evonik operates production plants in Belgium, Germany, France, Japan, Thailand, and the US.

Evonik, and its predecessor companies, have produced numerous publications that can provide an extensive product overview. Detailed information on products and their applications can be found in the corresponding product information and in the Technical Information texts. These texts and other information can be accessed at www.AEROSIL.com.

Table 2: An overview of the wide range of applications

Application	Effect	Examples/industry	
Raw material/educt	• Purity • Reactivity control • Composition	• Fire protection glass • High-strength concrete • Catalysis	• Optical fibers • Pharmaceutical products
Rheological additives	• Anti-sedimentation • Dispersing agents • Thixotropizing • Thickening	• Agrochemicals • Battery gels • Drilling fluids • Injectable screw anchors • Diamond suspension • Sealants • Paint/coatings	• Adhesives • Cosmetic products • Food • Pharmaceutical products • Rotor blades for wind power plants • Lubricants • Waxes
Filler material	• Antiblocking • Low thermal conductivity • Scratch-resistance • Reinforcement	• Sealants • Films • Golf balls • High-quality technical rubber products • Insulation material • Plastic bags	• Coatings • Shoe soles • Silicone • Thermal insulation • Dental composite fillings • Two-component mortar and concrete
Flow enhancer for powders	• Particle separation	• Agrochemicals • Fire-extinguishing powder • Animal feed • Cosmetics • Plastic granulate • Food	• Grinding additives • Pharmaceutical products • Powder dosage • Powder coating • Toner
Insulation material	• Fineness/pore structure • Low thermal conductivity • Low electrical conductivity	• Cable insulation	• Thermal insulation/vacuum insulation panels
Porous coating	• Pore formation • Pore structure	• Inkjet paper	• Plaster
Abrasives/abrasion enhancers	• Abrasion	• CMP: chemical-mechanical planarization	• Glass polishing • Metal polishing
Controlling triboelectric properties	• Electrostatic charging	• Powder coating	• Toner

1.2.3 Products manufactured in pyrogenic and wet-chemical processes

Some basic physical properties of silicas manufactured via pyrogenic methods and those produced from solutions are compared in table 3:

Table 3: Overview of key properties of synthetic silicas

Property	Unit	Fumed silica	Precipitated silica	Silica gel	Aerogels
Specific surface area (BET) 1)	m^2 / g	50 to 500	30 to 800	250 to 1000	250 to 400
Mean primary particle size	nm	5 to 50	5 to 100	3 to 20	3 to 20
Aggregate or agglomerate size	µm	8)	1 to 40	1 to 20	1 to 15
Tamped density 2)	g / l	50 to 250	50 to 500	500 to 1000	50 to 125
Loss on drying 3)	%	≤ 3	3 to 7	3 to 6	3 to 5
Loss on ignition 4)	%	3,5 to 5,5	3 to 7	3 to 15	3 to 5
pH value 5)		3 to 5	5 to 9	3 to 8	2 to 5
Pore diameter	nm	Non-porous	≥ 30 7)	2 to 20	≥ 25
Pore diameter distribution		8)	Very broad	Narrow	Narrow
Dioctyl adipate absorption 6)	ml / 100 g	200 to 350	175 to 320	100 to 350	200 to 350
Structure of aggregates and agglomerates		Structure predominantly chain-like, branched, or higher	Structure aggregated, low	Extremely high agglomeration, porous particles	Agglomerated particles (distinctly porous)
Thickening effect		Very distinctive	Present	Appears to be present	Present

Typical values for hydrophilic silicas, specifacation on request
1) Based on ISO 9277
2) Based on ISO 787/11
3) Based on ISO 787/2
4) Based on ISO 3262/20
5) Based on ISO 787/9
6) Based on ISO CD 19246
7) Smaller in exceptional cases
8) Not applicable (strongly dependent on dispersion conditions. See section 5.2)

There are some large overlaps in the properties shown, but there are particularly clear differences in relation to the specific surface area (BET), the aggregate and agglomerate size, the tamped density, the loss on drying, and the aggregate structure.

Silicas produced in wet-chemical processes are ground or spray-dried. Precipitated silicas are commonly low-structured aggregated particles. When they are ground or spray-dried, this greatly alters the size and structure of the aggregates/agglomerates.

Silicas produced pyrogenically take the form of chain-like, branched aggregates, resulting in a fluffy powder. They are used without any grinding or spray-drying. Spray-drying, compacting, and chemical processing turn hydrophilic fumed silicon dioxide into technical products of even higher quality (such as hydrophobic products and AEROPERL® grades).

Whether silicon dioxides are produced in wet-chemical processes or in flame hydrolysis, their smallest components (the primary particles) are relatively strongly aggregated and agglomerated. On the basis of [6], the terms primary particle, aggregate, and agglomerate are used in connection with fumed oxides.

It should be noted that the terms "aggregate" and "agglomerate" are sometimes used with the inverse meanings.

Primary particles are the smallest components of a fumed oxide. They do not occur in isolation – they merge to form an aggregate. An aggregate is therefore a group of primary particles that have merged together. If a suitable electron microscope is used, primary particles can be identified in an aggregate and studied. They are identified as "hypothetical particles," under the assumption that they are spherical units that make up the aggregate. To illustrate the definitions, figure 3 shows four primary particles outlined in red. An aggregate is also shown in figure 3, outlined in green.

An agglomerate is made up of several aggregates. Figure 3 shows one agglomerate, made up of four aggregates. The aggregates that make up an agglomerate do not form a unit by merging together. They are held together as the result of weak interactions (such as van der Waals forces or hydrogen bonds).

Figure 3: Diagram illustrating the terms primary particle, aggregate, and agglomerate

Figure 4 a shows some agglomerates of AEROSIL® 200. Dispersion allows the agglomerates to be split up, separating the aggregates. One of such aggregates is shown in figure 4b. The individual primary particles that have merged together and cannot be separated are clearly visible.

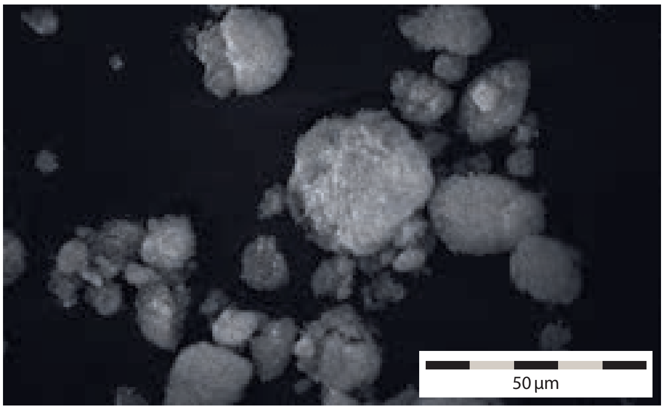


Figure 4 a

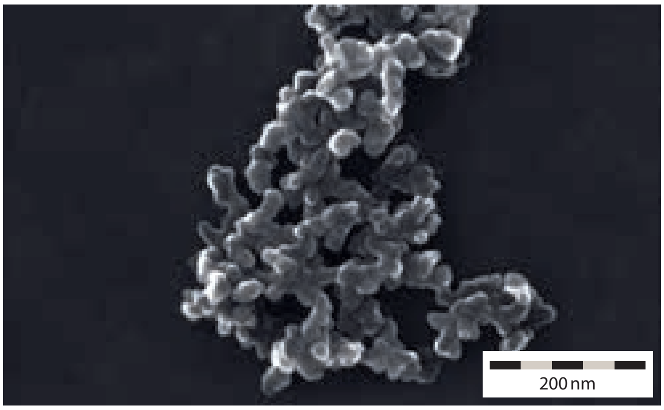


Figure 4 b

The most important material property in the application of synthetically produced silicas is the specific surface area of the materials. Silicas produced via flame hydrolysis have only an external surface. This means that there are generally few pores within the primary particles. Although a certain volume of pores can be detected via physisorption methods (such as Ar physisorption), these pores are located between the primary particles (inter-particle pores), not within the primary particles themselves (intra-particle pores).

Precipitated and fumed silicas display significant differences in their loss on drying, loss on ignition, and purity. Precipitated silicas originate from an aqueous solution and therefore contain a higher content of water (3 % to 7%). They are precipitated from sodium silicate with sulfuric acid, so the finished products contain relatively large quantities of sulfate, alkali, or alkaline earth ions. AEROSIL® contains far fewer impurities. It is made from $SiCl_4$, air, and hydrogen. The synthesis produces hydrochloric acid, which is retained in the final product as HCl, at a rate of <0.025%.

Are you looking for high-quality particles that are produced in a unique quality process?

Manufacture

- Manufacturing hydrophilic AEROSIL®
- Manufacturing other fumed metal oxides
- Mixed oxides and oxide mixtures
- Surface-treated products
- Structure modification
- Granulation
- Dispersion

Introduction

Manufacture

Properties

Products

Application-related effects

Quality and product reliability

Handling and availability

Annex

2. Manufacture

2.1 Manufacturing hydrophilic AEROSIL®

AEROSIL® is manufactured in a continuous flame hydrolysis of a substance such as silicon tetrachloride ($SiCl_4$). In the flame, hydrogen and oxygen (from the air) are reacted in the presence of $SiCl_4$.

The formation of SiO_2 can be described by a combination of an oxyhydrogen reaction, in which water is formed, and a hydrolysis of $SiCl_4$ with this water. The AEROSIL® process can be expressed in the following straightforward equations:

$$2\,H_2 + O_2 \rightarrow 2\,H_2O$$

$$SiCl_4 + 2\,H_2O \rightarrow SiO_2 + 4\,HCl$$

Overall reaction:

$$2\,H_2 + O_2 + SiCl_4 \rightarrow SiO_2 + 4\,HCl$$

One characteristic of the AEROSIL-process is that the educts are fed into the flame in a gaseous state. An initial process step is required to vaporize the $SiCl_4$ before it is mixed with air and hydrogen and fed into the combustion chamber.

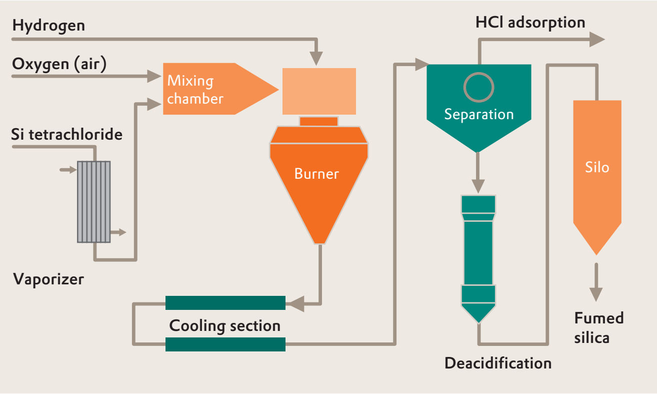


Figure 5: The AEROSIL-process (depicted as a flow chart)

A hydrogen flame that contains $SiCl_4$ burns continuously in the reaction chamber. As water is formed, it undergoes a spontaneous, quantitative reaction with $SiCl_4$ inside the flame, producing hydrochloric acid gas and the desired silicon dioxide.

AEROSIL® is formed in the flame in fractions of a second. A mixture of hot gases and solids (SiO_2 particles) containing hydrochloric acid is produced. This aerosol first needs to be cooled down before being separated into solid and gases in the next process step. The solid still contains relatively large quantities of HCl, adsorbed onto the surface of the particles. A deacidification step is performed to remove HCl, before the hydrophilic product can be packaged. The hydrochloric acid is recycled. A diagram illustrating the process is given in figure 5.

Many important material properties of AEROSIL® are defined in the flame. Varying the concentration of the reactants, the flame temperature, and the gas's dwell time in the combustion chamber enables the particle size, the particle size distribution, the specific surface area, and the surface properties of the silicas to be specified, within broad margins.

Many of the properties of AEROSIL® can be explained in a simplified droplet model.

The model assumes that the reaction gases pass through a region that imposes a sharp increase in temperature followed by a decrease – the flame. At the start of the reaction (at the base of the flame), minute droplets of SiO_2 are formed (nuclides). These nuclides collide with one another stochastically and merge to form bigger and heavier droplets. These bigger droplets can then merge with others and so on – the overall number of droplets keeps decreasing as droplet size increases. This process continues as long as the flame is hot enough to keep the droplets in a liquid state.

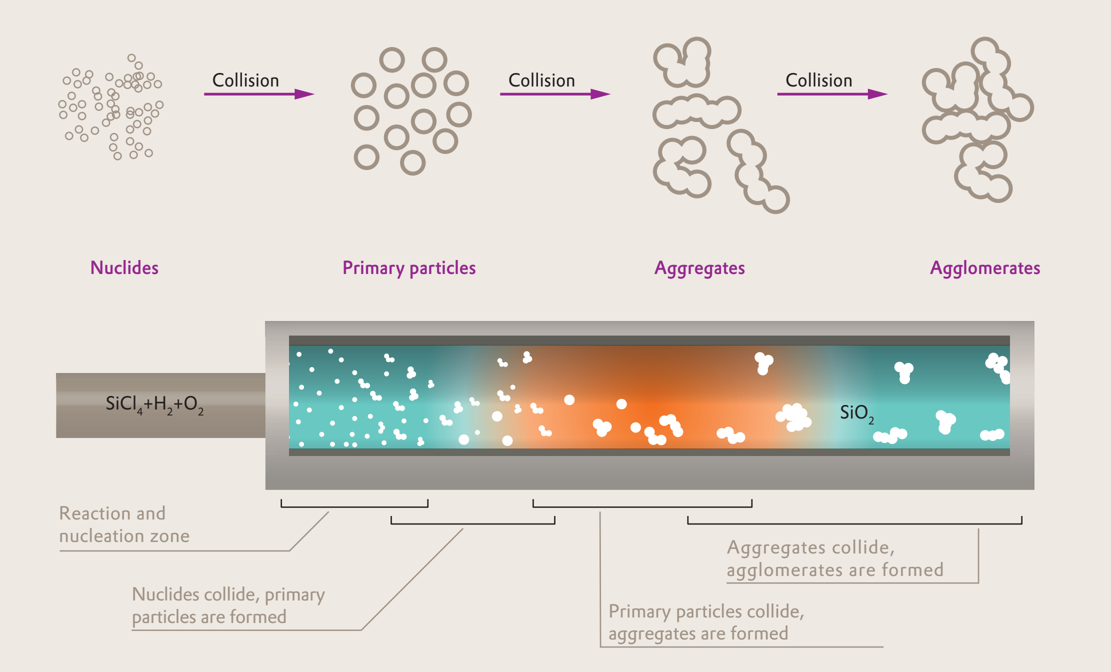


Figure 6: Diagram illustrating a simple model of particle genesis in the flame (cf. figure 41a)

When the droplets enter a colder area of the flame, they solidify (at least partially). When these droplets collide, instead of fully merging and coalescing to form larger spherical droplets, they now only partially merge. This results in aggregates consisting of primary particles (the partially solidified droplets). The aggregates become fully solidified in the colder parts of the flame. When these aggregates collide, they are now unable to merge at all. Instead, the aggregates attach to each other, held together by weak interactions. This results in agglomerates being formed. The droplet model of particle genesis is illustrated in figure 6.

Although it is very simplified, this model of the particle genesis of AEROSIL® is able to explain many of the properties of fumed silicas. For example, the model provides a very effective explanation for why materials with low specific surface areas and large primary particles are formed in hot flames and materials with high specific surface areas and small primary particles are formed in cold flames. The droplets' zone of coalescence is longer in a hot flame than in a cold one, causing larger primary particles to form and creating materials with lower specific surface areas.

The simple droplet model is also able to explain certain aspects of the structure of aggregates and agglomerates. In place of silicon tetrachloride, chlorosilanes such as methyltrichlorosilane and trichlorosilane can be used as raw materials, either alone or in mixtures (including mixtures with $SiCl_4$). If this is done, the flame l and flow conditions need to be varied to create the same product that pure silicon tetrachloride would produce.

Hydrochloric acid is produced with four-molar excess in relation to SiO_2 in the AEROSIL® process. It can be recycled to manufacture $SiCl_4$ or $HSiCl_3$ as shown in the equation below:

$$Si + 4\,HCl \rightarrow SiCl_4 + 2\,H_2$$

The silicon source used in this case is ferro-silicon (FeSi), a product used in the production of steel. The hydrogen is fed back into the AEROSIL® process, creating a large-scale cycle that is environmentally friendly.

Additional sources of silicon for the AEROSIL® process include sidestreams from the manufacture of polycrystalline silicon (PCS) or from Müller-Rochow synthesis.

2.2 Manufacturing other fumed metal oxides (AEROXIDE® products)

For the classic AEROSIL® process, all the educts used need to be transferred into the gas phase. To produce fumed silicon dioxides, silanes such as silicon tetrachloride and methyltrichlorosilane are vaporized and then reacted in the oxyhydrogen flame. Other metal chlorides can also be vaporized and hydrolyzed to form metal oxides in a procedure similar to the AEROSIL-process. This has been performed successfully for many metal and metalloid elements in various research projects. The oxides of aluminium and titanium have been commercialized and are established on the market.

Titanium dioxide, marketed under the product names AEROXIDE® TiO_2 P 25 and AEROXIDE® TiO_2 P 90, is produced from titanium tetrachloride ($TiCl_4$). Titanium tetrachloride, like silicon tetrachloride, is a liquid that is easily vaporized.

The aluminium oxide products AEROXIDE® Alu C, AEROXIDE® Alu 65, and AEROXIDE® Alu 130 are produced from aluminium chloride, which is a solid under normal conditions. A special vaporizer technology is used to transfer the solid into the gas phase and react this gas to form aluminium oxide in a procedure similar to the AEROSIL-process.

Like the production of silicon dioxide, the production of the other oxides can be expressed in simple chemical equations:

$$TiCl_4 + 2\,H_2 + O_2 \rightarrow TiO_2 + 4\,HCl$$

$$2\,AlCl_3 + 3\,H_2 + 1{,}5\,O_2 \rightarrow Al_2O_3 + 6\,HCl$$

The expertise required to produce high-quality metal oxides with precision centers on how the process is carried out. Unlike AEROSIL®, which is amorphous, the oxides of aluminium and titanium are produced in a (partially) crystalline form. These crystal phases can be controlled within wide range via process control. Titanium dioxide occurs in various modifications. AEROXIDE® TiO_2 P 25 and AEROXIDE® TiO_2 P 90 contain the modifications anatase and rutile:

Table 4

	Anatase [wt%]	Rutile [wt%]
AEROXIDE® TiO_2 P 25	Approx. 85	Approximately 15
AEROXIDE® TiO_2 P 90	Approx. 90	Approximately 10

Anatase and Rutile content in AEROXIDE® TiO_2 P 25 and AEROXIDE® TiO_2 P 90

Aluminium oxide can also occur in a various modifications. The short dwell times in the oxyhydrogen flame prevent α-aluminium oxide from being formed. Most of the modifications are metastable forms that would transition to α-aluminium oxide under sufficiently high temperatures. The products AEROXIDE® Alu C, AEROXIDE® Alu 65, and AEROXIDE® Alu 130 are found in the transition phases δ, γ, and θ in varying proportions.

The following table lists further fumed metal oxides that can be produced in the lab or on a technical scale. The oxides in the table can be obtained in pure form or incorporated as mixed oxides in silicon dioxide, titanium dioxide, or aluminium oxide matrices:

Table 5

Process options

- $AlBO_3$
- $AlPO_4$
- BPO_4
- Bi_2O_3
- Cr_2O_3
- ZnO
- Fe_2O_3
- GeO_2
- NiO
- MoO_3
- SnO_2
- ZrO_2
- V_2O_5
- WO_3
- CuO/Cu_2O
- MnO_2/Mn_2O_3
- CeO_2/Ce_2O_3
- Sb_2O_3

A wide range of metal oxides can be produced

As the above demonstrates, the AEROSIL-process is a flexible and versatile procedure that can produce oxides from the corresponding chlorides of many elements. The volatility of the raw material is an important factor in the production of oxides.

2.3 Mixed oxides and oxide mixtures

The flexibility of the AEROSIL-process means that mixed oxides can also be produced. To do this, several educts (such as $SiCl_4$ and $TiCl_4$) are fed into the flame and reacted to form a mixed oxide. Very different products can be produced depending on the educts selected and the flame parameters set.

The composition of the mixed oxides can be configured across wide ranges of the mole fraction. The particle structure is greatly affected by the composition. If a starting material is added in a small quantity in comparison with the second educt, it is mostly homogeneous particles that are produced. In these particles, the metal ion that is present at a lower quantity is homogeneously distributed in the oxide matrix (figure 7, top image).

Mixed oxides are also produced with both components present in more equal relative quantities. In such a case, homogeneous particles (primary particles or aggregates) may be formed (figure 7, top image) or particles that have regions with different stoichiometries. As a result, it is possible to create more specific morphologies in the flame. The most well-known of these is the core-shell structure. Silicon dioxide/titanium dioxide mixed oxides can form core-shell morphologies. This results in a crystalline titanium dioxide core surrounded by an amorphous silicon dioxide shell (figure 7, bottom image).

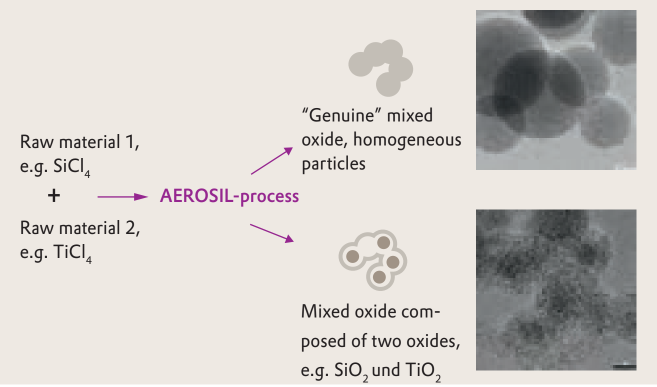


Figure 7: Mixed oxides and core-shell morphology

A distinction must be made between oxide mixtures and mixed oxides that are produced in the flame: Oxide mixtures are oxides that have been mixed together. This means that the oxides are produced in separate processes and then physically mixed in a mixing process. The two starting oxides material do not merge, so the primary particles and aggregates are made up of only one oxide type although the agglomerates may be composed of both types. A diagram of the production of an oxide mixture is shown in figure 8. The important difference between mixed oxides and oxide mixtures is illustrated in figure 9.

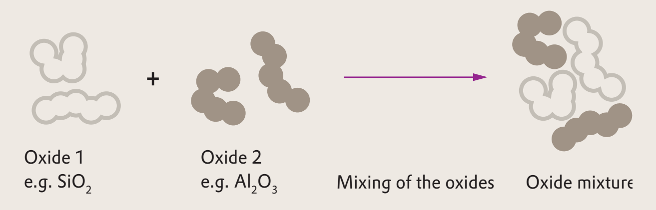


Figure 8: An oxide mixture is formed by mixing two oxide materials

Figure 9: Mixed oxides and oxide mixtures compared

Fumed mixed oxides and fumed oxide mixtures are thus produced in two very different ways. The products created sometimes have opposite application effects, even though they are made up of the same oxides.

Examples of mixed oxides include the SiO_2-Al_2O_3 mixed oxides AEROSIL® MOX 80 and AEROSIL® MOX 170, and the TiO_2-SiO_2 mixed oxides VP TiO_2 1580 S, VP TiO_2 545 S, and VP TiO_2 590 S. In AEROXIDE® TiO_2 PF 2, 1–3 % iron oxide is added to the titanium dioxide.

An example of an oxide mixture is AEROSIL® COK 84, which is produced by mixing 84 % of AEROSIL® 200 with 16% of AEROXIDE® Alu C.

Table 6

Product	SiO_2 [%]	Al_2O_3 [%]	TiO_2 [%]	BET [m²/g]
AEROSIL® COK 84	84	16	–	150–250
AEROSIL® MOX 80	98,3	0,3–1,3	–	60–100
AEROSIL® MOX 170	98,3	0,3–1,3	–	140–200
VP* TiO_2 1580 S	15	–	85	65–95
VP* TiO_2 545 S	5	–	95	35–55
VP* TiO_2 590 S	5	–	95	70–100

Compositions and specific surface areas of selected products

*VP = developmental product. The names of development products start with the letters VP. Commercialization is dependent on the market response.

The values given are non-binding guideline values.

2.4 Surface-treated products

Hydrophobic products

The products described thus far are compounds obtained directly from the AEROSIL-process, that is, via flame hydrolysis. Products created in this way have freely accessible silanol groups (Si-OH) on the particle surface, causing them to be hydrophilic – they can be wetted by water. For all kinds of applications that require special properties, a post-treatment step is required to transform hydrophilic AEROSIL® into its exact opposite: hydrophobic AEROSIL®.

The hydrophobic behavior is brought about by reacting hydrophilic silanol groups with organic groups (see figure 10). After this processing step, these organic groups are anchored so firmly to the surface (via covalent bonds) that they can only be removed by a process such as thermal decomposition, which would also alter the structure of the AEROSIL®.

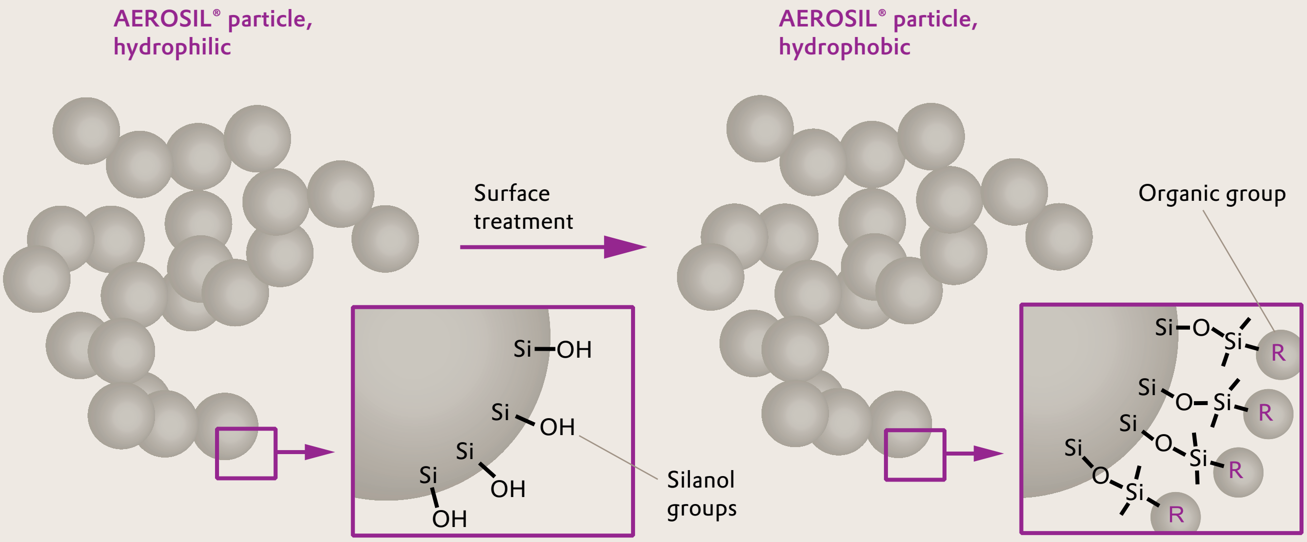


Figure 10: Diagram of a surface treatment transforming hydrophilic AEROSIL® into hydrophobic AEROSIL®

Figure 11: Water with hydrophilic silica (left) and hydrophobic silica (right)

At first glance, hydrophilic and hydrophobic fumed oxides are identical – both are fine white powders. The fundamental difference becomes clear, however, when they are mixed with water: While hydrophilic products can be completely wetted by water, hydrophobic products do not mix with water at all.

This water-repellent behavior is caused by short-chain or long-chain organic groups that are anchored onto the surface of the fumed silica. The following figure displays some examples of hydrophobic AEROSIL® products:

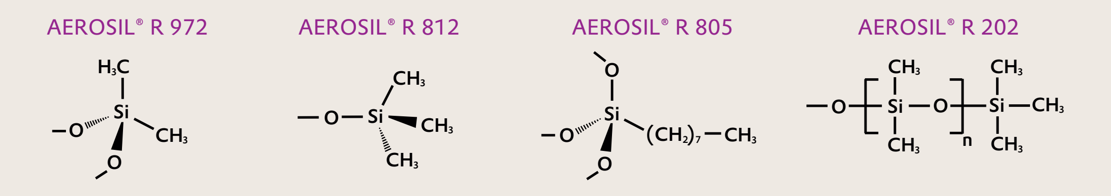


Figure 12: Selection of various hydrophobic AEROSIL® products with their surface groups

There are two basic types of post-treatment: reacting the powder directly after the AEROSIL-process, for example with a halogen silane, and reacting the AEROSIL® surface with silanes, silazanes, siloxanes, etc. Both processes can be carried out continuously and both produce a homogeneous and effective modification of the surface area that can be freely accessed from the outside with organic groups.

In the first method – direct post-treatment – non-compressed AEROSIL® is reacted with a suitable silane. The main factor in a post-treatment reagent's suitability is that it must be sufficiently reactive under the given conditions without being so unstable that the organic components will decompose.

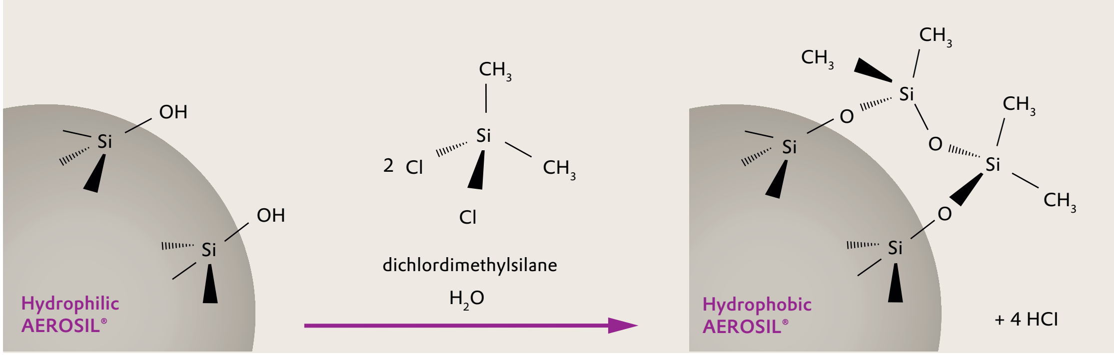
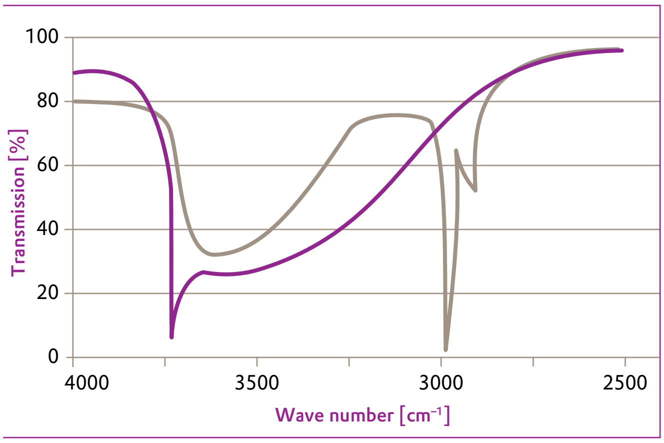


Figure 13: Diagram of dichlordimethylsilane reacting with the silanol groups on the AEROSIL® surface: transition from hydrophilic to hydrophobic

The second method – reacting with silane or siloxane groups other than halogen silanes – is performed after the AEROSIL® production process itself has been fully completed. In this method, more complex organic compounds can be anchored onto the surface, such as those with sensitive organic functionalities.

Both post-treatment methods are controlled to ensure that the capacity of the silica surface is utilized as fully as possible. This utilization is illustrated by comparing the infrared spectra of hydrophilic AEROSIL® and AEROSIL® that has undergone chemical post-treatment. The sharp band in the starting material at approximately 3,750 cm^{-1}, which can be attributed to the free silanol groups on the surface, is removed by the reaction with the modifying reagent, which ultimately produces trimethylsilyl groups. At the same time, the hydrophobic end product displays the formation of a C-H vibration band at <3,000 cm^{-1} (see figure 14).

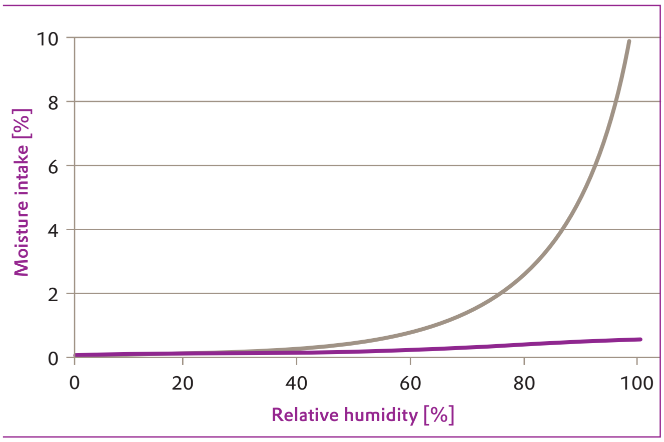
Figure 14



— AEROSIL® 300 — AEROSIL® R 812

Infrared partial spectrum of AEROSIL® 300 before chemical post-treatment and after (AEROSIL® R 812), both pure substance compacted samples, IR device: Perkin Elmer 325

Figure 15



— AEROSIL® 150 — AEROSIL® R 202

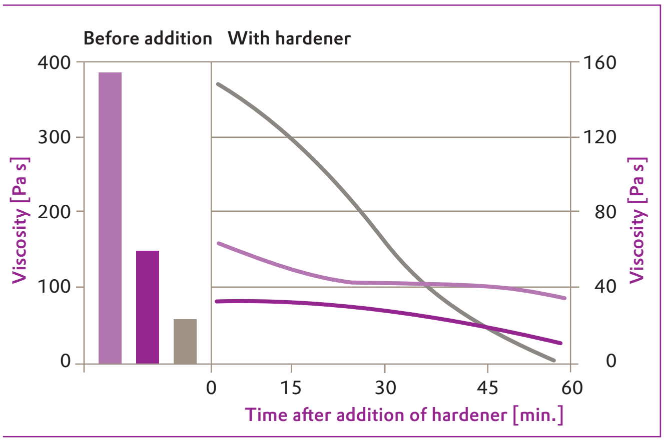
Moisture absorption of AEROSIL® 150 and the hydrophobic AEROSIL® R 202 at room temperature (measured in small samples)

The surface treatment creates totally different physical/chemical properties compared with non-treated AEROSIL® products, which significantly extends the potential applications of fumed oxides. Treated fumed oxides can be used in a wide range of industrial fields such as silicone rubber, paint and coating formulations, toner products, adhesives and sealants, cable compounds, and resin systems.

A very impressive example of the altered surface properties is the absorption of moisture from the air (see figure 15): Thanks to the nonpolar surface of AEROSIL® R 202 compared with AEROSIL® 150 (a product with no surface treatment), interaction with water vapor is extremely reduced, resulting in a much lower capacity to absorb moisture.

Another application engineering example is the thickening effect in a low-viscosity, reactive epoxy resin – a polar medium. The hydrophobic products AEROSIL® R 805 and AEROSIL® R 202 have distinct advantages over the hydrophilic product AEROSIL® 300.

Figure 16



▨ AEROSIL® 300 ▨ AEROSIL® R 202 ▨ AEROSIL® R 805

Viscosity of an epoxy resin with 5.6 % AEROSIL® before the addition of hardener and wetting agent and 3.8 % AEROSIL® after

Products with functionalized surfaces

If a suitable modification reagent is selected, it is possible to customize the chemical functionality on the surface in addition to the polarity. In many applications, making silica hydrophobic is not enough. The suitable chemical functionality needs to be configured to produce the desired effects. To adjust how the AEROSIL® surface reacts with the surrounding substrate with precision, a wide variety of reagents with all manner of functionalities can be used. For example, amines, epoxides, and methacrylates can be anchored to the surface. One example of a functionalized product is AEROSIL® R 711 (figure 17).

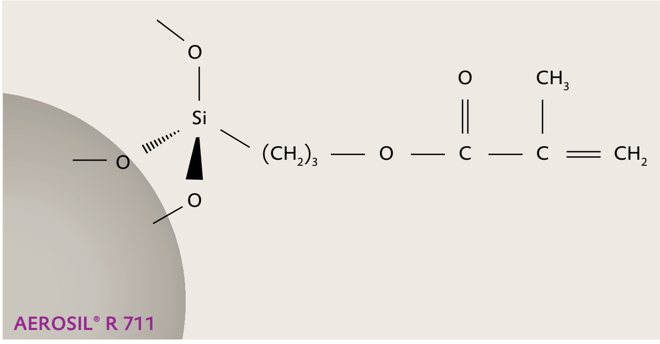


Figure 17:
Diagram illustrating the surface functionality of AEROSIL® R 711

AEROSIL® R 711 is a fumed silica that has been given methacrylate functionalities, producing optimum properties for applications such as special paint and coating systems, high-performance elastomers, and dental composite fillings.

A further field of application for functionalized AEROSIL® products is charge control in toner products. In such applications, it is particularly important for the pigment particles to be given optimum surface charges while preventing the toner particles from clustering together – failing to do so results in suboptimal print quality. Evonik has developed a product for this sensitive field of application, specially configured to fulfill these requirements: AEROSIL® R 504.

This product consists of high-purity AEROSIL® that is made hydrophobic and functionalized with an organic amino group in a special procedure.

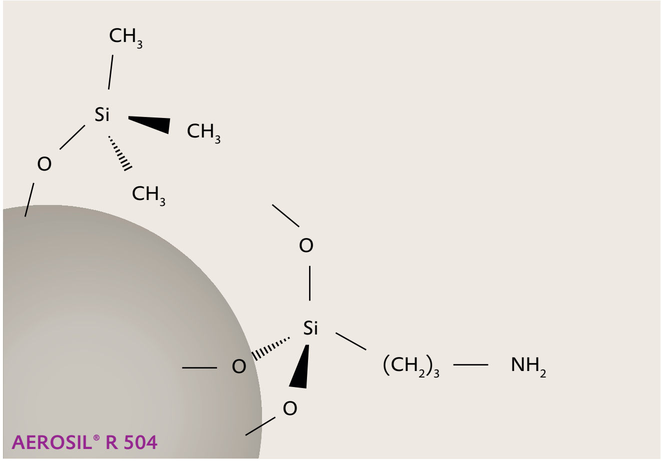


Figure 18:
Diagram illustrating the surface functionality of AEROSIL® R 504

The amino group on the surface provides excellent control of the powder's triboelectric properties, which are essential for demanding toner applications. Positive specific charges on the powder can only be achieved via a functionalizing post-treatment. The unique post-treatment therefore makes AEROSIL® R 504 an optimum toner additive that makes outstanding toner performance possible.

Figure 19

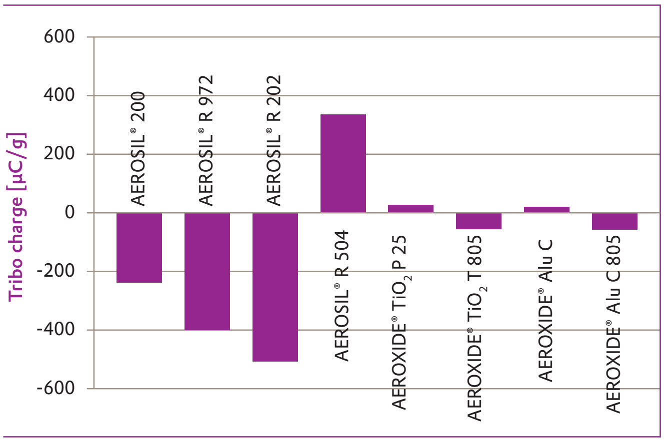


Figure 19 shows the specific charge values (q/m values, charge-mass ratios) for selected products

2.5 Structure modification

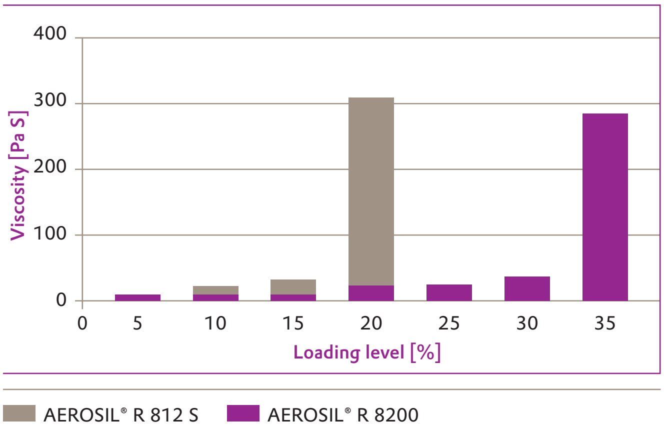
Using the AEROSIL-process to produce fumed silica involves constructing aggregates and agglomerates from primary particles (see section 2.1). The umbrella term structure is used to describe the combination, form, and arrangement of these aggregates and agglomerates. The structure is responsible for many of the characteristic properties of fumed silica. For example, the structure of AEROSIL® is partly responsible for the thickening effect (configuring of the rheological properties) in various media and for the flow properties (thixotropy) of substrates that AEROSIL® has been added to.

A further process step, structure modification, makes it possible to alter the AEROSIL® structure with precision without changing the chemical composition or the surface properties. This allows customized products to be produced with selected properties for special applications.

An example application for an AEROSIL® product with structure modification (the only such products available thus far are AEROSIL® R 7200, AEROSIL® R 8200, and AEROSIL® R 9200) is given below – use as an additive in a silicone polymer. Figure 20 illustrates the increase in viscosity in relation to the proportion of AEROSIL® R 812 S and AEROSIL® R 8200 content, in a two-component RTV (room temperature vulcanizing) silicone. The two materials have been post-treated with the same modification reagent and both have trimethylsilyl groups on their surface. Even at a content percent of 20 %, the viscosity of the polymer mixture with AEROSIL® R 812 S is ten times as high as that of the AEROSIL® R 8200 formulation. AEROSIL® products with no surface modification would display a viscosity of a few thousand pascal seconds under these conditions. Using surface-modified and structure-modified products therefore makes it possible to produce silicone polymers with high content percents, and thus high strengthening properties, combined with low thickening effects.

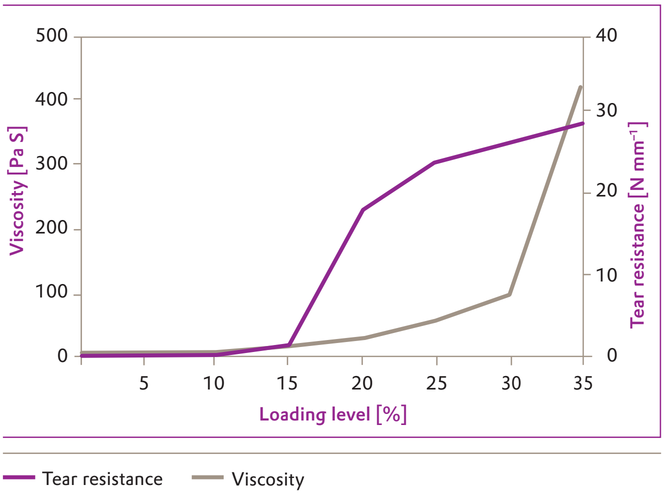
Previously, it was only possible to vary the two main effects in silicone rubber applications – the strengthening properties and the thickening effect of fumed silica – by altering the content percent. The two effects had to be altered in unison. This innovative structure modification, however, allows the two parameters to be controlled independently. Figure 21 below shows that the curves for viscosity and tear resistance (in an RTV-2C silicone rubber) are no longer identical: the two properties have been separated. Using a structure-modified fumed silica such as AEROSIL® R 8200 can therefore provide high mechanical strength combined with low viscosity.

Figure 20



Comparison of the viscosities of AEROSIL® R 812 S and structure-modified AEROSIL® R 8200 in an RTV-2C silicone rubber

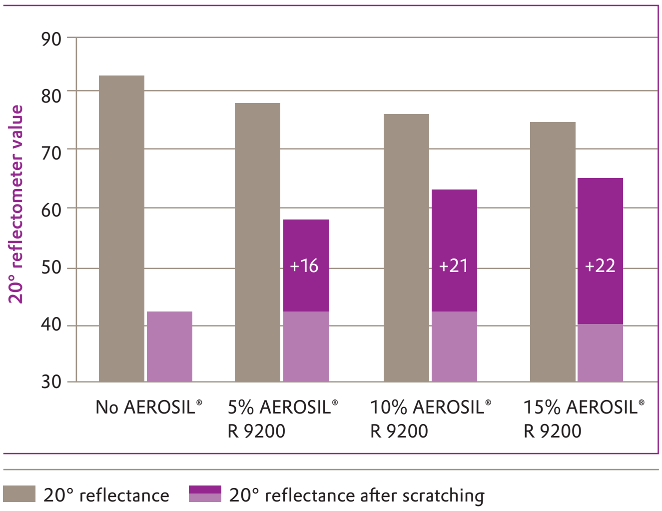
Figure 21



Rheological and mechanical properties of an RTV-2C formulation with AEROSIL® R 8200 in relation to the loading level

Using structure-modified silicas can also significantly improve scratch resistance in coatings. Using AEROSIL® R 9200 in UV-cured clear lacquers can produce a distinct improvement in the luster after scratching (see figure 22). In this UV-cured clear lacquer, increasing the percent of incorporated AEROSIL® R 9200 enabled a further increase in the 20 ° reflectometer value to be achieved with barely any change to the reflectance before scratching.

Figure 22



Using AEROSIL® R 9200 to improve the scratch resistance in a UV-cured clear lacquer based on polyester acrylate. The scratching was applied with an Elcometer 1720 – 40 double strokes with pig bristle brushes, 5 % quartz slurry

2.6 Granulation

One further product group completes Evonik's portfolio of fumed oxides: the AEROPERL® products. These products are granulates. A special granulation process designed for the particular requirements of AEROSIL® enables granulates to be produced with mean particle sizes of approximately 20 to 30 µm. The main features of AEROPERL® are its outstanding flow properties, good incorporation properties, and low-dust handling, which allow the application range of AEROSIL®-based products to be considerably broadened. Figure 23 below illustrates AEROPERL® 300 Pharma – a product specially designed for use in the pharmaceutical industry – as seen under the scanning electron microscope at various levels of magnification.

The process for producing AEROPERL® is free from contamination, so the high purity of the starting material is retained in the compaction. Because of their internal porosity, the granulates are able to absorb large amounts of a wide range of substances and can thus be used as a carrier material. AEROPERL® products display outstanding flow properties even when they are loaded with paste-like substances such as highly viscous oils or formulations with active agents (see figure 24). These properties, combined with the high purity of the granulate, make AEROPERL® products indispensable auxiliary materials, particularly in the cosmetic and pharmaceutical industries.

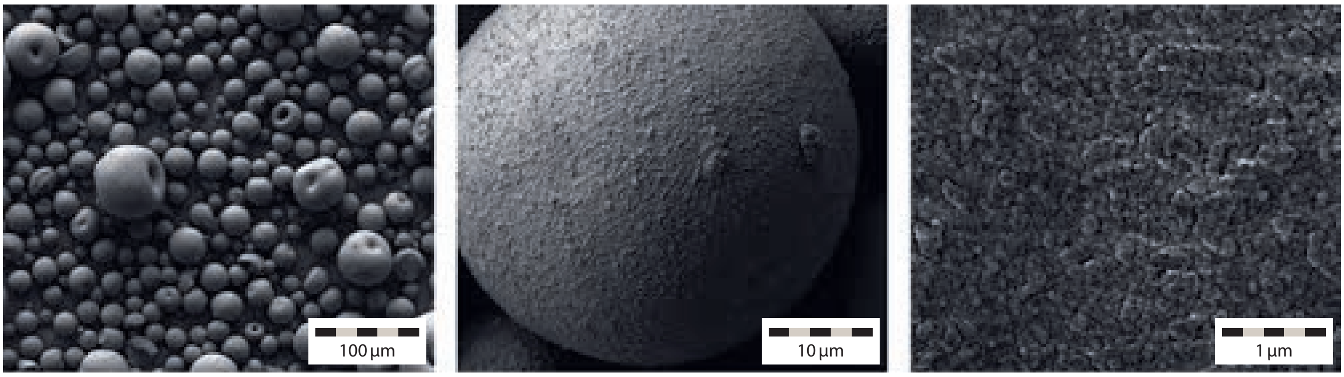
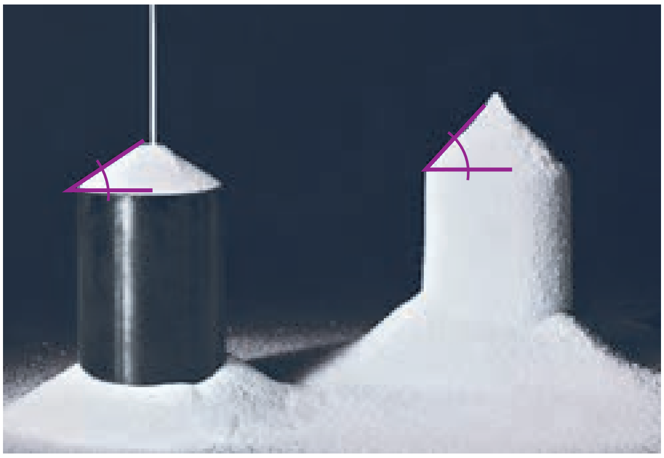


Figure 23: Scanning electron microscope images of AEROPERL® 300 Pharma at various magnification levels



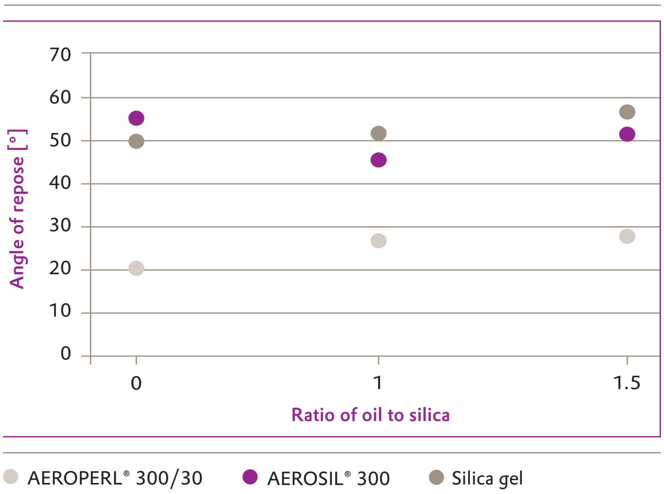


Figure 24

Illustration of how the angle of repose is measured

Flow behavior of silica-based carrier materials loaded with a test oil (MIGLYOL® 840, Sasol Germany GmbH). An angle of repose of <30 ° represents good to very good flow behavior while an angle of repose of >40 ° occurs in powders with mediocre flow

Another outstanding property of AEROPERL® is the high mechanical stability of the granulate, which is particularly important in (heterogeneous) catalysis. AEROPERL®, when used as a stable catalyst carrier, can be retrieved as an intact granulate during the process despite high shear forces.

2.7 Dispersion

Because of the special particle structure/morphology, fumed metal oxides have a relatively low tamped density. The residual moisture is also relatively small (as a result, it has not yet been possible to form all the possible silanol groups for AEROSIL®). This has both advantages and disadvantages. When AEROSIL® is used in gel batteries, it is a great advantage. When AEROSIL® is dispersed in water, however, the amount of wetting is slightly reduced. Special care must be taken when handling this light and airy powder to avoid dust formation.

In principle, powders can be added to the liquid phase from containers such as sacks by hand. If this is done, however, sufficient extraction of the dusts that will arise must be provided. Less dust is produced when a vacuum is used to suck in the product and convey it into suitable preparation containers. Ideally, this mixture of product and air will make close contact with the liquid directly, to enable rapid wetting. Corresponding devices are available. For example, the mixture can be fed directly into the liquid via a submerged tube or the fumed metal oxide can be drawn into the liquid directly via a vacuum dissolver. Devices such as those produced by Ystral (jetstream mixers with vacuum product induction or devices from the Conti-TDS series) can also be used, allowing the material to be fed in directly from the packaging and then dispersed by the same device. For more detailed information, consult our Technical Information [7], "Successful Use of AEROSIL® Fumed Silica in Liquid Systems."

It must be borne in mind, however, that conveying powder in this way can give the powder an electrostatic charge. Relevant information can be found in the brochure "Synthetic Silica and Electrostatic Charges." The conveyor lines should be grounded. If the particles are being conveyed in explosive atmospheres (such as in solvents), it is vital for electrostatic charging of the particles to be prevented or the risk of explosion to be reduced by inerting the container atmosphere. It must, be considered that more air will be dragged in by the product because of its low bulk/tamping weight, and also that the ambient air that is taken in with the vacuum induction can fully undo any inerting of the container atmosphere that may have been present beforehand.

The dispersion process is made up of several substeps. Firstly, the powder must be wetted by the liquid. Once the powder has been wetted, the next step is to expel the air. Energy can then be applied to de-agglomerate the powder.

At this point, it is worth revisiting the differences between primary particles, aggregates, and agglomerates.

The aggregate is made up of sintered primary particles. The sinter necks may be more or less thick and pronounced, depending on the particular product.

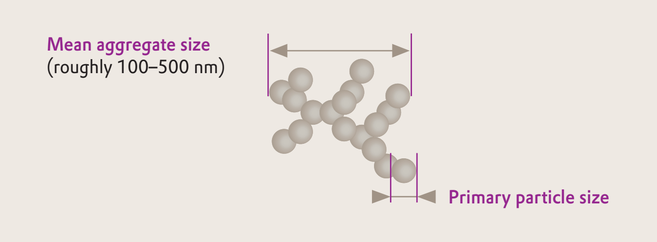


Figure 25: Diagram showing an idealized depiction of primary particle and aggregate size

The agglomerates consist only of aggregates that are held together relatively loosely, by weak forces such as hydrogen bonds, van der Waals forces, and capillary forces. To break up these agglomerates (that is, to de-agglomerate the product), energy is required.

This energy can be applied in the form of shear energy or direct impact. In a mild stirring process, it is normally only possible to apply low amounts of energy. A dissolver can be used for de-agglomeration if used correctly. In this case, the optimum viscosity must be configured. The viscosity of a dispersion is mainly determined by the concentration and fineness of the powder, in addition to the viscosity of the liquid used. The physical/chemical interaction of the particle surfaces is another influencing factor.

Optimum viscosity is achieved in dissolver dispersion when the "donut effect" occurs. If the dissolver is configured correctly (correct container diameter, dissolver disk diameter, and distance between dissolver disk and base), if the speed is sufficient (generally a peripheral speed of 20 to 40 m/s), and if concentration increases, a ring-shaped bulge of product resembling a donut will appear on the surface of the dispersion. Further information can be found in Technical Information TI 1279, "Successful Use of AEROSIL® Fumed Silica in Liquid Systems."

Figure 26: Dispersion with a dissolver

Even when dispersion is performed with other dispersion devices such as rotor/stators, ball mills, homogenizers, or kneading machines, it is always important to observe the optimum viscosity range for the particular procedure and the device used.

Once the energy density has reached sufficient levels, the aggregates can be broken up. Suitable equipment for this includes bead mills with very small grinding media (<0.5 mm) that are able to produce dispersions with a mean particle size of less than 100 nm given a milling duration of adequate length and high milling energy. In many practical examples, aggregates between 100 and 500 nm have been used. It must, however, be noted that the surface area of the grinding beads is not irrelevant and there is a risk of the product being increasingly contaminated with grinding bead material.

In addition to these more mechanical requirements, it is important for additives to be used to ensure that the particles do not lose any fineness in the grinding process. Re-agglomeration must be prevented.

There are four basic principles of stabilization:

1. Electrostatic stabilization

Additives are used to give the particle surface sufficient charge to cause particles with charges of the same polarity to repel each other (a sufficiently high zeta potential). For stabilization purposes, it is irrelevant whether the high surface charges that are brought about are positive or negative. In some cases, this can be achieved merely by configuring a suitable pH value. A suitable pH range can be found by measuring the zeta potential at various pH levels. Unfortunately, this is not always enough. Because of certain chemical reactions that could still take place, the pH range determined to be stable in measurements may differ from the range that has been verified to be stable in practice.

2. Steric stabilization

The effect of surfactants, which generally have molecules with one polar end and one nonpolar end, is well-known. If the polar end, for example, is able to engage in an intensive interaction with the surface, the nonpolar parts of the molecule will face outward. If these parts are sufficiently long, the particles in the liquid can resemble a sea urchin with long spikes. The stabilization effect is not brought about by direct mechanical separators, but by differing local osmotic pressures. If two particles come close enough for their “spikes” to overlap, this creates a higher concentration of “spikes” or molecules in this particular area. The liquid molecules then attempt to dilute this higher concentration by diffusing into this concentration region. This creates a higher pressure (osmotic pressure) in this region. This pressure then causes the particles to separate.

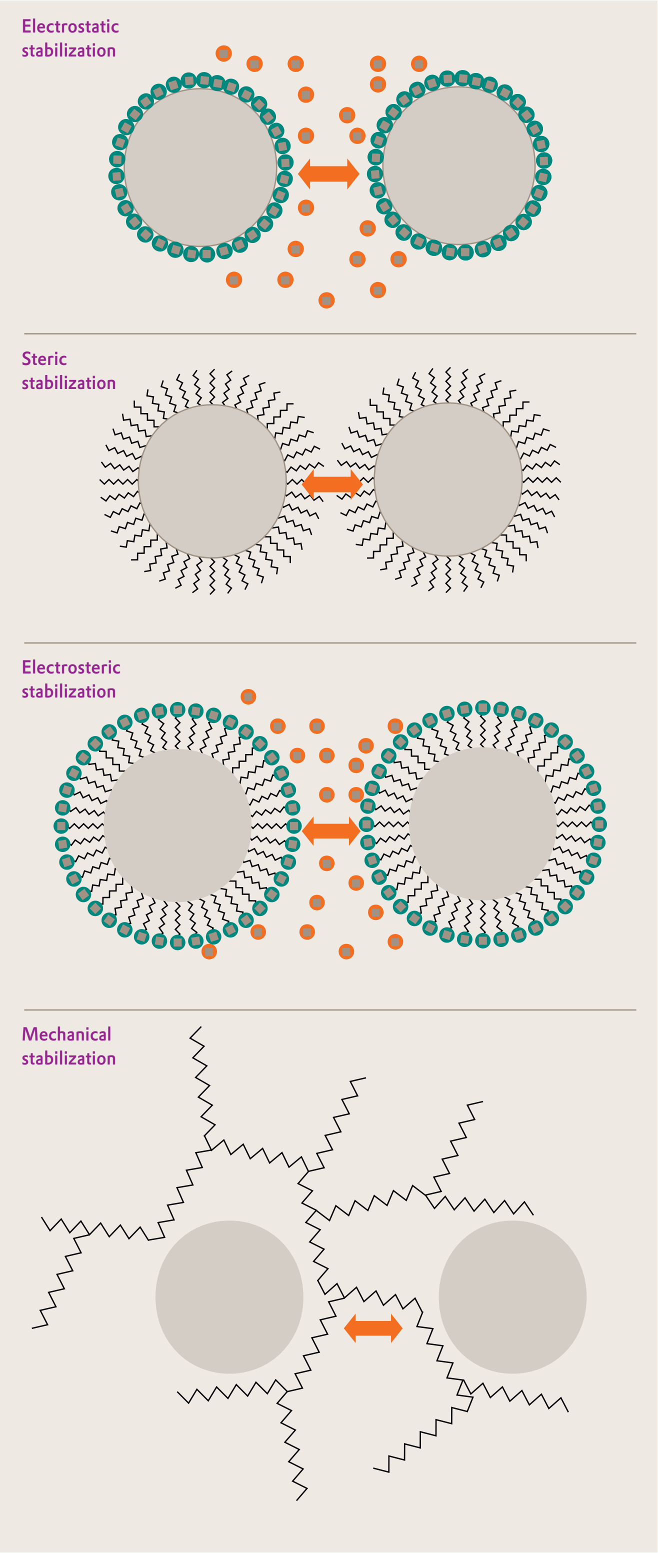


Figure 27: Overview of the four stabilization principles

3. Electrosteric stabilization

The effects described under 1 and 2 can be combined to stabilize the dispersion. The liquid must be sufficiently polar to allow the particle surfaces to become charged.

4. Mechanical stabilization

A fourth principle that can be considered is purely mechanical stabilization. Strictly speaking, this is not a "true" stabilization and it is therefore rarely mentioned in the relevant textbooks. However, practical experience is ultimately what matters. If a material needs to be prevented from re-agglomerating, perhaps because there is no known effective additive or because the effective additive cannot be used, the viscosity of the homogeneous phase can be increased to hinder the movement of the particles in the liquid so much that the material is practically trapped in its highly viscous surroundings.

As mentioned earlier, when sufficiently high energy densities are used with Evonik products, dispersions with particles much smaller than one micrometer can be achieved. This process, also known as the top down process, requires milling devices with adequate strength.

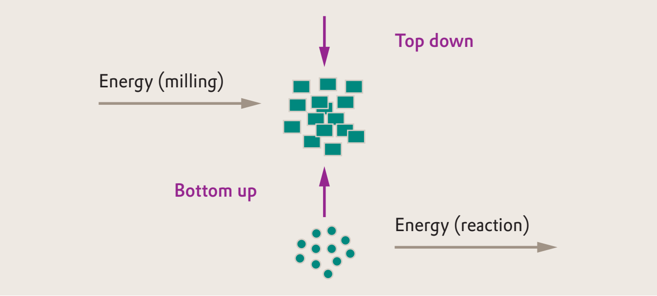


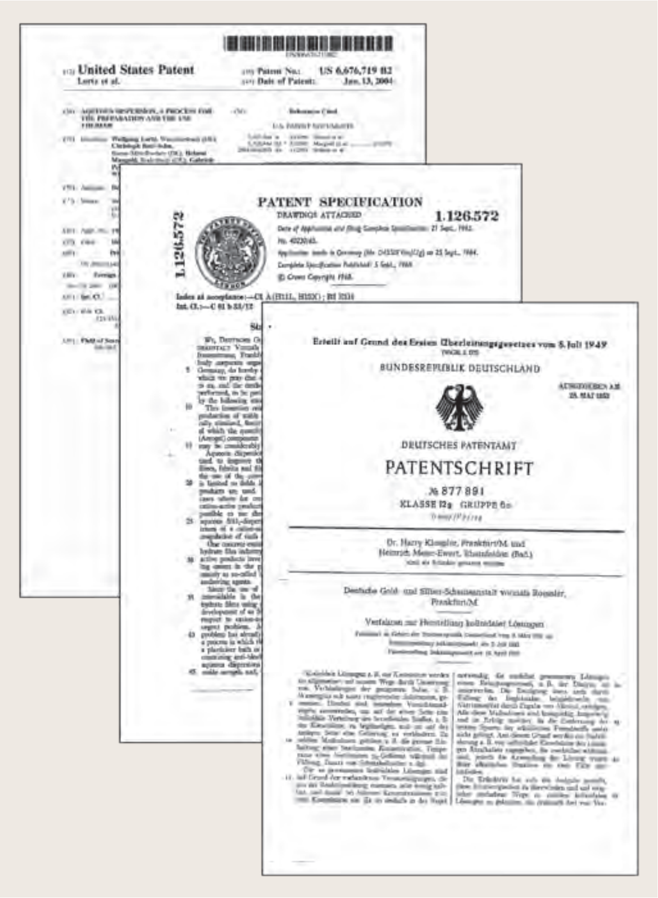
Figure 28: The top down/bottom up process

Such a complex procedure, which requires suitable additives to be found, is not worthwhile for all customers. Evonik has provided several fumed silicas in dispersed form since the 60s.

In the past 10 years, this product range has been expanded. More detailed information on Evonik dispersions can be found in the Industry Brochure "AERODISP® Fumed Silica and Metal Oxide Dispersions." Almost all of Evonik's hydrophilic fumed silicas are now available in dispersed form. Some hydrophobic products are also available in solvents.

These particles can also be provided with surfaces that have no positive or negative charge, thanks to the use of special additives. This surface charge must be taken into account for the particular application and formulation, to avoid incompatibilities. For some applications, however, incompatibility may actually be desired. In high-quality inkjet papers a cationic charge in AEROSIL® particles enables the ink molecules, which normally have a negative charge, to be anchored as near as possible to the paper surface so the water in the ink can be adsorbed by deeper layers. The result is a printed image with intense color and clearly defined edges. More information can be found in the Industry Brochure "AEROSIL® for Photo Inkjet Media."

Thanks to an advanced high-energy milling process, Evonik is able to provide numerous materials as fine dispersions on a non-aqueous basis with high concentrations combined with relatively low viscosities.



United States Patent
Lortz et al.
Patent No.: US 6,676,719 B2
Date of Patent: Jan. 13, 2004

PATENT SPECIFICATION
DRAWINGS ATTACHED
1,126,572

Erteilt auf Grund des Ersten Überleitungsgesetzes vom 8. Juli 1949
BUNDESREPUBLIK DEUTSCHLAND
DEUTSCHES PATENTAMT
PATENTSCHRIFT
№ 877 891
Dr. Harry Kloepfer, Frankfurt/M. und
Heinrich Meier-Ewert, Rheinfelden (Bad.)
Deutsche Gold- und Silber-Scheideanstalt vormals Roessler,
Frankfurt/M.
Verfahren zur Herstellung kolloidaler Lösungen

Figure 29: In 1951 Degussa submitted the first patent for dispersions based on AEROSIL®

Are you looking for a product with excellent basic properties that can be perfectly configured to suit your specific requirements?

Properties

- Amorphous structure, purity
- Particle fineness and surface area
- Special physical/chemical data
- Surface chemistry

Introduction

Manufacture

Properties

Products

Application-related effects

Quality and product reliability

Handling and availability

Annex

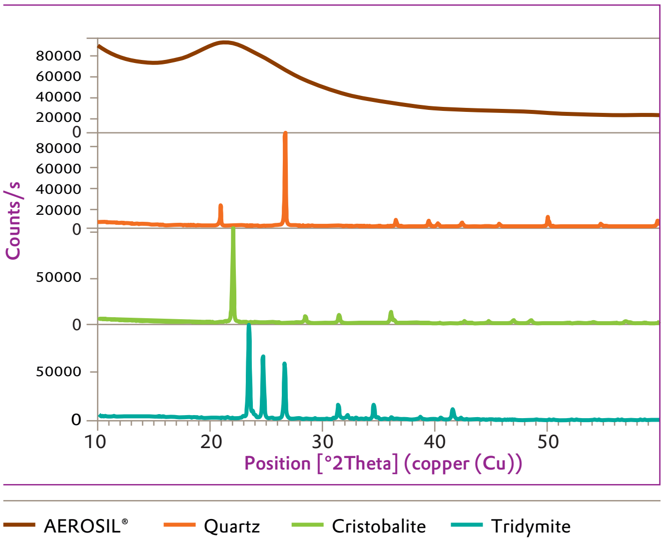
3. Properties

3.1 Amorphous structure, purity

3.1.1 Amorphous structure

The chemical formula of AEROSIL® is SiO_2, as was illustrated earlier. It must be remembered, however, that AEROSIL® is not actually made up of isolated SiO_2 molecules. The silicon atoms form single covalent bonds with the four adjacent oxygen atoms. Each atom satisfies therefore the octet rule. For energy-related reasons, the bonding electron pairs are positioned as far apart from each other as possible, resulting in a tetrahedron arrangement.

Figure 30



X-ray diffraction images of AEROSIL®, quartz, cristobalite, and tridymite

The SiO_4 tetrahedrons form the structural basis of the macromolecular network. In principle, two arrangements are possible: the SiO_4 tetrahedrons can be arranged in an ordered or a completely irregular way. The crystalline silica modifications that occur in nature, such as quartz, cristobalite, or tridymite, are exclusively composed of precisely defined, fully identical structural units, known as unit cells. The regular structure of the crystal lattice causes X-rays to be diffracted at the lattice or network planes with interference effects. All synthetic silicas produced by Evonik show a totally different behavior. The SiO_4 tetrahedrons have an irregular arrangement, as shown in figure 30 by the absence of defined crystalline X-ray diffraction reflections. AEROSIL® is therefore X-ray amorphous. This amorphous nature can also be verified by using a high-resolution transmission electron microscope (see section 3.2.1). Unlike glasses, which form a three-dimensional framework with infinite expansion (measured in atomic dimensions), amorphous AEROSIL® has a particulate structure.

AEROSIL® does not produce clearly defined X-ray reflections. It shows weak, very diffuse amorphous halo. These diffraction phenomena can be interpreted by means of an unorganized network model [8]. They are the result of the short-range order whose scope in non-crystalline substances is always very small in comparison with the particle size of highly dispersed substances (see also page 38).

In fused silica, these states span approximately 1.3 nm, in precipitated silicas approximately 1.2 to 1.0 nm, and in AEROSIL® and electric arc silica approximately 0.9 and 0.8 nm [9]. The transition from an ordered to a disordered state therefore occurs earlier on, after the third tetrahedron coordination sphere. With regard to this tendency toward short-range order, AEROSIL® has the largest structural disorder of all SiO_2 products [9]. It should be stressed that the short-range order regions do not refer to crystallinity.

Figure 31: Diagram of the arrangement of SiO_4 tetrahedrons in AEROSIL®, based on a model by EVANS and KING [10]. The circles represent oxygen atoms. The silicon atoms are located in the centers of the tetrahedrons.

On the basis of the EVANS and KING model, the SiO_2 network can be represented as shown in figure 31. An Si-O bond length of 0.152 nm and an Si-Si bond length of 0.312 nm has been determined on the basis of the calculated radial distribution function. The Si-O-Si bond angle exhibits a considerable fluctuation range of 120 to 180 degrees [11].

3.1.2 Purity

Carbon content

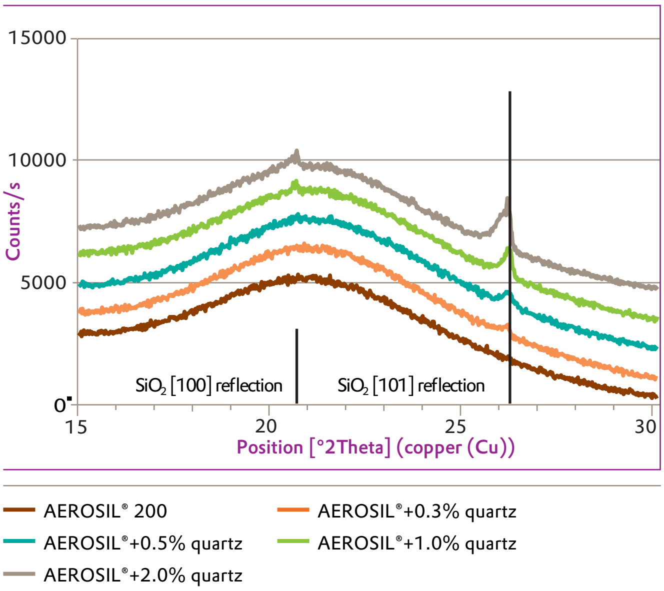
The carbon content of AEROSIL® can be used as a characteristic for the extent of surface treatment. It is determined on the basis of a carrier gas hot extraction analysis (such as can be performed on LECO-analysers, model CS 244 or CS 600). This involves weighing sample material in a ceramic crucible, adding combustion accelerators, and heating it in an induction furnace under an oxygen flow. This procedure leads to an oxidation of the carbon present to form CO_2. The quantity of gas is measured by infrared detectors. Before the measurement is performed, the device is calibrated with a suitable reference material. The carbon detection limit for this analytical method is approx. 300 µg/g.

Amorphous structure of AEROSIL®

As quartz dust and, to a lesser extent, dusts that contain cristobalite, tridymite, and coesite have a silicogenic effect [12, 13], the amorphous structure of AEROSIL® has special significance. IR spectroscopy, differential thermal analysis, and X-ray diffraction have all failed to find any crystalline content in AEROSIL® samples (see section 3.1). Figure 32 shows X-ray diffractograms of AEROSIL® 200 and AEROSIL®-quartz powder mixtures in the angle range of 15 ° to 30 ° (29). It can clearly be seen that pure AEROSIL® 200 does not contain any crystalline reflections. On the other hand, the X-ray deffractogram for the AEROSIL®-quartz powder mixture shows a detectable crystalline reflection at 26.6 ° (2θ) for 0.3 % quartz and higher (figure 32).

The X-ray detection limit for slightly disordered cristobalite in fused quartz is below 0.3 %.

Figure 32



Angle range of [100] and [101] refections of α-quartz, shown on the basis of AEROSIL® 200/α-quartz mixtures. AEROSIL® 200 has no refectionsitself and is therefore X-ray amorphous. Diffractometer X'Pert MPD Pro, CuK α-radiation, 40 kV and 40 mA, measurement time per step80 s

Trace metallic impurities

The educts used for AEROSIL® production are highly volatile silicon compounds that are prepared in a distillation process and are thus highly pure. In flame hydrolysis, gaseous hydrochloric acid is precipitated as the only by-product. It can be almost completely separated from the solid. The result is a product with a high level of purity. The SiO_2 content of AEROSIL® is greater than 99.8 %. AEROSIL® 200, for example, thus meets the requirements of the monographs of numerous pharmacopeias and official specifications (table 6). Evonik provides pharmaceutical products that have been specially developed for the pharmaceutical industry, under the trade names AEROSIL® 200 Pharma, AEROPERL® 300 Pharma, AEROSIL® 300 Pharma, AEROSIL® 200 VV Pharma, and AEROSIL® R 972 Pharma. These products have been tested for the following parameters in accordance with the European, the United States, and the Japanese Pharmacopeias (EP, USP/NF, and JP):

- EP 0434 "Silica, Colloidal Anhydrous": identification, pH, chloride, heavy metals, loss on ignition, assay
- EP 2208 "Silica, Hydrophobic Colloidal": identification, chloride, heavy metals, water-dispersible fraction, loss on ignition, assay
- Determination according to USP/NF "Colloidal Silicon Dioxide": identification, pH, loss on drying, loss on ignition, As, (leachable), assay
- JP XV "Light Anhydrous Silica Acid": identification, chloride, heavy metals, aluminium, iron, calcium, arsenic, loss on drying, loss on ignition, volume test, assay

The purity of AEROSIL® is normally measured on the basis of ICPMS (inductively coupled plasma mass spectrometry). This involves the sample material being dissolved in hydrofluoric acid and the matrix being distilled as H_2SiF_6 at a low temperature. If a complexing agent is added, this process can also be used to determine the boron content. To allow the high-purity qualities to be analyzed effectively, these work steps are performed in a clean room environment.

Table 6

Element	µg/g = ppm	Element	µg/g = ppm
Al	≤ 10	Mg	≤ 0.3
Ca	≤ 0.3	Na	≤ 0.5
Cr	≤ 0.4	Ni	≤ 0.3
Cu	≤ 0.05	P	≤ 0.1
Fe	≤ 3	Ti	≤ 1
K	≤ 0.5	Zr	≤ 0.2
Li	≤ 0.05		

Trace impurities of standard AEROSIL® products (not specifications)

Heavy metals, such as Pb, As, Cd, Hg, Sn, Sb, Bi, and Ti, are all in the range of <0.05 µg/g, as are rare earths.

Higher AEROSIL® purity levels are produced from high-purity precursors. These products only contain metallic trace element impurities in the ppb range (ng/g).

No solvents are used for manufacture or packaging (see section 2.1).

3.2 Particle fineness and surface area

The amorphous structure of AEROSIL® and the irregular arrangement of the SiO_4 tetrahedrons were described in 3.1. This section addresses the macroscopic expansion and form of the particles.

The visual appearance of AEROSIL® is that of a light, white powder. AEROSIL® actually consists of approximately 98 vol% air (density of AEROSIL® 2.2 g/cm³, tamped density of "normal" AEROSIL® product approximately 50 g/l). It can be fluidized with small blasts of compressed air and can therefore be handled in silos without any problems. Figure 33 illustrates this behavior on the basis of a simple lab demonstration.

Figure 33: Simple lab demonstration of the fluidization of AEROSIL®. Compressed air with a pressure of approximately 0.2 bar is applied to the glass frit.

3.2.1 Agglomerate and aggregate size measured in the light scattering procedure

The actual particle size distribution of AEROSIL®, like that of many other powders, is substantially dependent on the particle property being measured (such as the diffraction diameter or solid volume) and the dispersion method (the dispersion medium and dispersion forces). Strictly speaking, the scattering theory only applies for spherical particles. For this reason, a light scattering procedure, such as laser diffraction, can provide the diameter of a sphere with equivalent scattering cross-section.

When AEROSIL® particles are dispersed in a stream of air, the majority of them will be arranged as agglomerates. The laser diffraction method finds distributions that are comparatively similar for the different products (between 4 and 200 µm) with mean values around 40 µm, with no significant portions in the submicron range. Precipitated silicas display slightly broader distributions, in contrast.

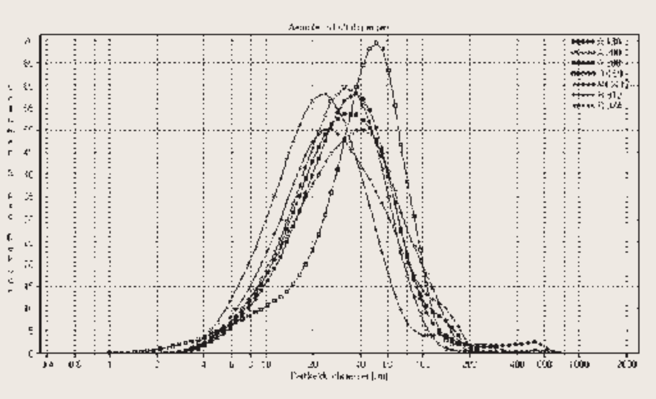


Figure 34: Particle size distribution of various AEROSIL® products dispersed in air, as determined by the laser diffraction method

These particle sizes must not, however, be interpreted as the aerodynamic diameter, which is an important key figure for inhalation behavior. The aerodynamic diameter is a value that is standardized in accordance with the relative density of 1. The particular properties of the silica particles result in them having an effective density (which determines their sinking behavior in still air) that is far lower than their solid density, which results in significantly smaller aerodynamic diameters.

When AEROSIL® is dispersed in a liquid, the particle size distribution varies significantly in relation to the dispersion conditions, for example those arising from ultrasonic treatment.

In aqueous dispersions AEROSIL® particles mainly take the form of coarse agglomerates unless additional dispersion forces are applied (similar to the agglomerates measured in the stream of air). Under a comparatively low ultrasonic influence, they break up into significantly smaller particles (aggregates and more stable, small agglomerates). No primary particles can be detected, however. The hydrophobized AEROSIL® products display slightly higher stability.

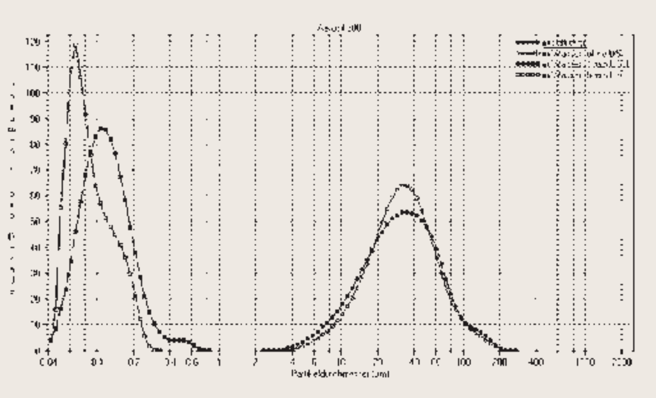


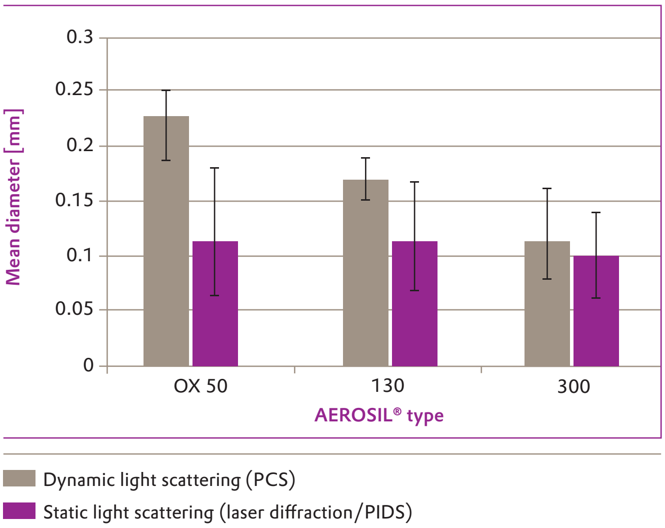
Figure 35: Influence of dispersion energy on the particle spectrum (AEROSIL® 300, laser diffraction method, dispersion in air and water with different ultrasound intensities (0 min, 1 min level 1 and 5 min level 8))

In the case of dispersion with strong shear forces (ultrasound treatment), the AEROSIL® grades mainly take the form of aggregates in the submicron range. Dynamic light scattering, also known as photon correlation spectroscopy (PCS), is particularly suitable for examination in this size range.

Both light scattering methods (dynamic and static) provide comparable results for the various AEROSIL® grades: narrow particle distribution in the submicron range.

As the specific surface area increases, smaller mean particle sizes are detected via PCS and the values approach those obtained via laser diffraction (PIDS). The PCS measurements also show only a monomodal narrow distribution. No particles in the range of primary particles (approximately 10–30 nm, which is within the measuring range for this method) are present in the suspension.

Figure 36



Comparison of the two light scattering methods

3.2.2 Particle size and structure

SEM

Scanning electron microscope (SEM) images display the morphology of AEROSIL® 200 in its delivery state. The material was dusted onto an SEM specimen holder and photographed from above. A field emission scanning electron microscope (FESEM) was used to achieve well-defined images down to the nanometer range.

The SEM overview image in figure 37 a shows spherical structures (agglomerates) in the single-digit to multiple-digit micrometer range. These structures are generated when the sample is taken from the compacted material and applied to the SEM specimen holder as the result of decomposition caused by shear forces (dispersion in the air). Only at higher magnification levels (figures 37b and 37c) does it become clear that this loose group is composed of a strongly branching arrangement of agglomerates that are themselves made up of smaller components (the aggregates). If these agglomerates are dispersed in a liquid medium and this dispersion is then very strongly diluted and then dried on an SEM specimen holder, SEM images such as the one shown in figure 37d are produced at higher magnification levels. The dispersion causes the agglomerates to be separated back into aggregates (figure 37d). It was such aggregates that were compacted to form agglomerates (figures 37a, 37b, 37c) before the product was packaged in transport vessels. Figure 37d clearly shows the structure of a single aggregate: It is made up of primary particles that have merged together and can no longer be separated.

TEM

The scanning electron microscope is not suitable for a precise measurement of the particle size, as a conductive film needs to be applied to the sample. To perform such measurements, transmission electron microscopy (TEM) is required. In the TEM device, the isolated aggregates are radiated with high-ener-

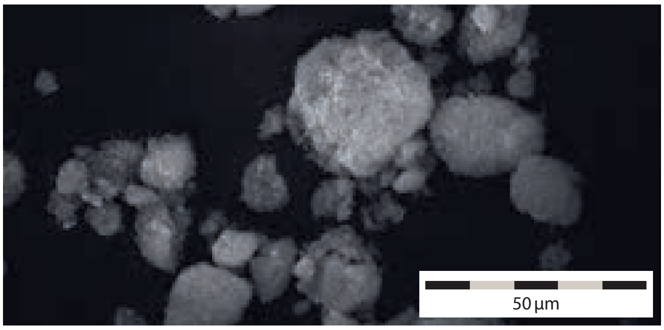


Figure 37 a: Overview image of an agglomerate group

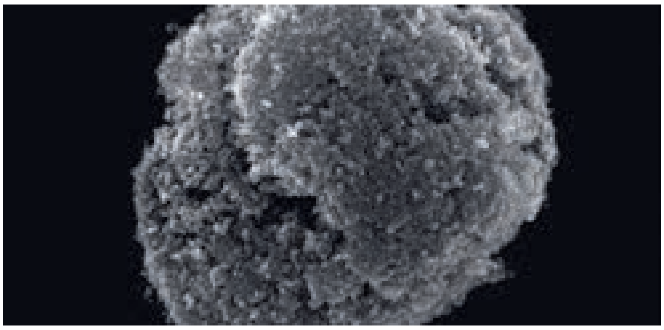


Figure 37b: Overview image of an agglomerate

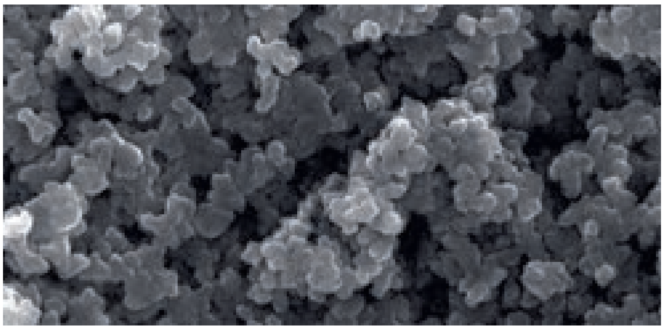


Figure 37c: Fine structure of the agglomerate

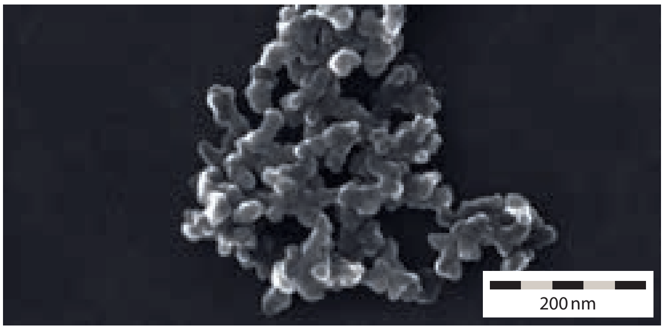


Figure 37d: A single aggregate

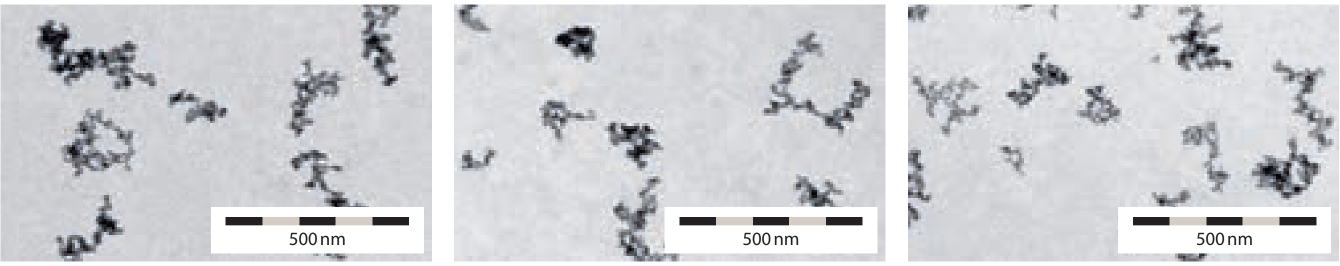


Figure 38: TEM images of AEROSIL® aggregates

AEROSIL® 130 AEROSIL® 200 AEROSIL® 380

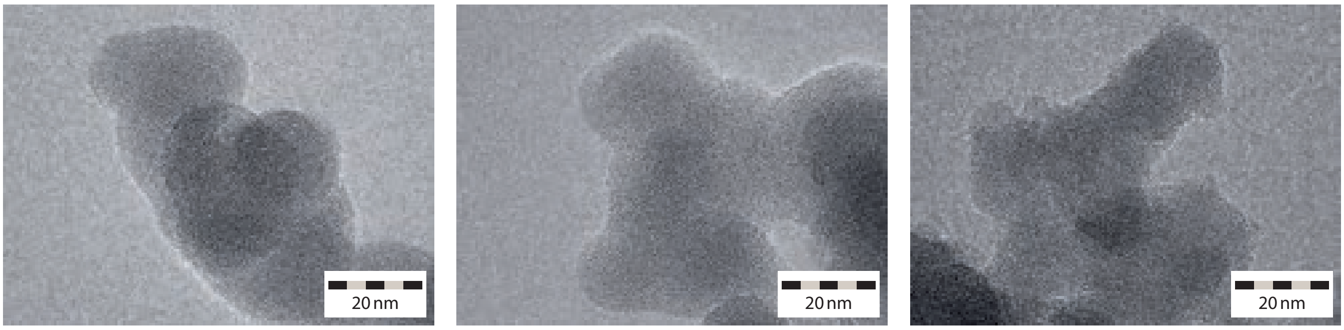


Figure 39: HRTEM images. The merging zones of the primary structures

AEROSIL® 130 AEROSIL® 200 AEROSIL® 380

gy electrons. Charging effects are not relevant in this case and the particles can be photographed in their original state in an undistorted way. TEM is the only direct imaging method that is suitable for identifying and measuring the characteristic aggregate forms and aggregate sizes, which can be customized in the same way as the primary particle sizes in the production process [14].

The TEM images for the product types AEROSIL® 130, AEROSIL® 200, and AEROSIL® 380 illustrate how the particle sizes and particle morphologies vary in flame synthesis. In each case, aggregates are formed from primary particles in the flame process through nucleation, growth, coagulation, and intensive merging.

TEM images with higher magnification (HRTEM) show that the merging zones within these aggregates have intensive overlapping and thus the primary structures have a pronounced degree of penetration – the merging zones are therefore less like a string of pearls with narrow contact points and discrete components, but more like bottlenecks. Separating a primary structure from its aggregate group would therefore involve breaking up numerous O-Si-O bonds and would require very high energy application.

The inside of the aggregates (inside the primary particles and the merging zones) is amorphous (figure 40).

Comparison of HRTEM images

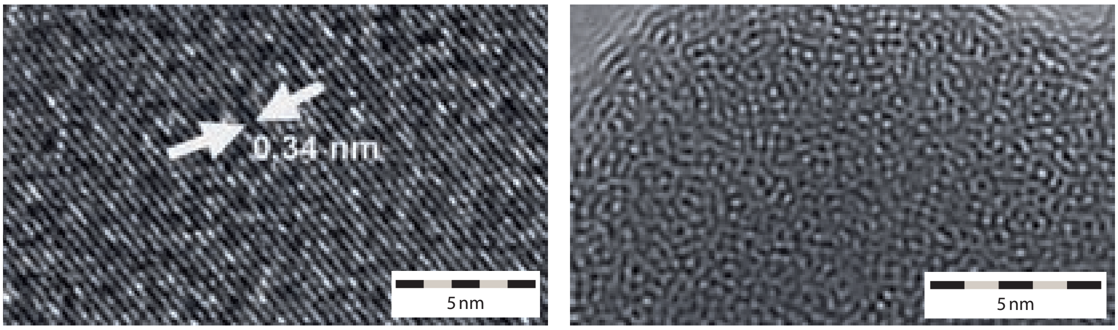


Figure 40: HRTEM images

Crystalline material: quartz, lattice plane image – lattice plane spacing 0.34 nm for hkl 101

Amorphous material: AEROSIL®

These images perfectly correspond to the structure model of amorphous silica represented in figure 31 (section 3.1) and the X-ray findings. AEROSIL® is made up of a three-dimensional network of statistically arranged SiO_4 tetrahedrons with exclusively short-range order. The TEM image shows a two-dimensional projection of the three-dimensional SiO_4 tetrahedron network at the particular focus level of the TEM device.

IINS (inelastic, incoherent neutron scattering)

The findings of the X-ray diffraction and HRTEM investigations of the amorphousness of AEROSIL® are substantiated by a third, independent, method: IINS. The low-frequency vibration state density of AEROSIL® products is significantly different from that of crystalline silica and characteristic of amorphous solids [8, 9, 10, 11, 15].

Infrared spectroscopy (IR, see section 3.4.2.2) provides detailed vibrational spectroscopy information on the O-H stretching vibrations of silica, which occur at low amplitude. At $<1{,}400\,\mathrm{cm}^{-1}$, however, it reaches its limits because of the intensive lattice absorption of the radiation. IINS, as a complement to IR, provides information in this range on low-frequency torsion vibrations and deformation vibrations of silica and demonstrates as an additional, independent method the amorphousness of AEROSIL®.

A numerically exact description of AEROSIL® primary particles and aggregates requires statistical evaluation of TEM images to determine the mean primary particle size and the primary particle size distribution. Depending on the polydispersity of a system and the corresponding precision required for a particle size distribution curve, a particle-size analyzer (TGZ) generally measures approximately 2,000–10,000 particles. The numerical characterization variables given include mean average of the primary particle diameter (DN) and its standard deviation (S), the surface area or the volume for the mean particle diameter (DA, DV), the calculated specific TEM surface area (OEM), and the median values and 90 % margins of the number/weight distributions.

To record and describe the aggregate parameters, the statistical mean values of 1,000–10,000 aggregates were determined (aggregate-size measurement).

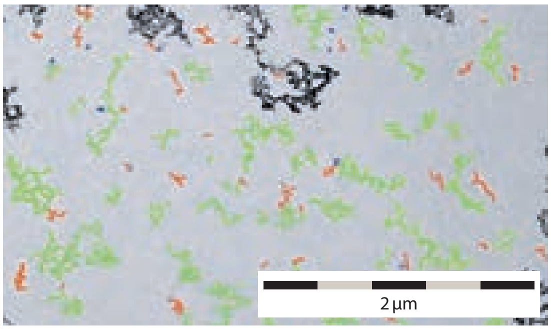


Figure 41: Aggregate-size measurement and digital aggregate classification analysis of AEROSIL® 200 (the samples were dispersed via ultrasound in an isopropanol/water mixture and applied to the TEM grid)

Table 7

Sample		AEROSIL® 200
Particle-size measurement (TGZ)		
Primary particle diameter (DN)*		12 nm
Mean primary particle diameter (DA) derived from specific surface area		14 nm
Calculated specific TEM surface area (OEM)		220 m²/g
Aggregate-size measurement		
Mean projected aggregate surface area		11675 nm²
Circumference		1386 nm
Convex circumference		521 nm
Minimum aggregate diameter		117 nm
Maximum aggregate diameter		198 nm
Void area		28 %
Degree of branching		9.5
Number of primary particles per aggregate		436
Results of the classification analysis		
Percent by class	Spherical	0.88
	Ellipsoid	13.2
	Linear	38.6
	Branched	47.4
Percent by weight	Spherical	0.03
	Ellipsoid	0.72
	Linear	8.6
	Branched	90.7

Selected parameters from the particle-size measurement (TGZ) and aggregate-size measurement. Mean value across 2,000 aggregates. (The samples were dispersed via ultrasound in an isopropanol/water mixture and applied to the TEM grid.) The data are guideline values (not specifications).

*Primary particles do not occur in isolation – they were merged in the production process to form aggregates (figure 6).

This includes parameters such as the mean projected aggregate surface area, the aggregate circumference and the convex circumference, the minimum and maximum aggregate diameter/Feret diameter, the equivalent circular diameter (ECD), the void area, the degree of branching, and the number of primary structures per aggregate.

Aggregates of various sizes can also be divided up into subcategories such as “branched, ellipsoid, linear, and spherical” on the basis of classification analyses and described by means of statistical methods.

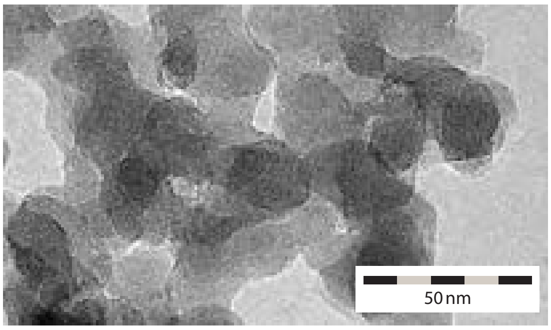


Figure 41 a: Subsection of an aggregate consisting of over 400 merged primary structures

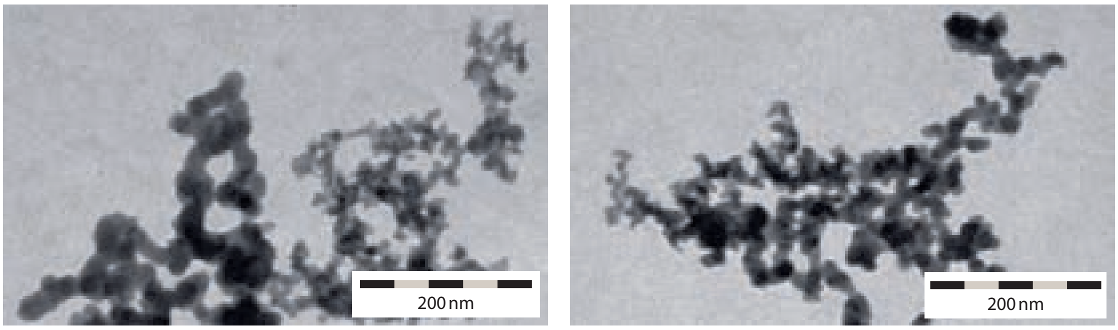


Figure 42: TEM images of AEROSIL® 130 and the hydrophobized product AEROSIL® R 972

AEROSIL® 130

AEROSIL® R 972

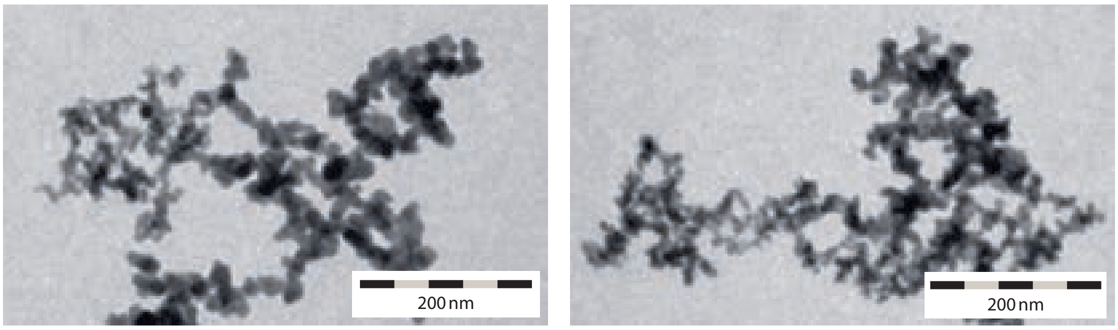


Figure 43: TEM images of AEROSIL® 200 and the hydrophobized product AEROSIL® R 974

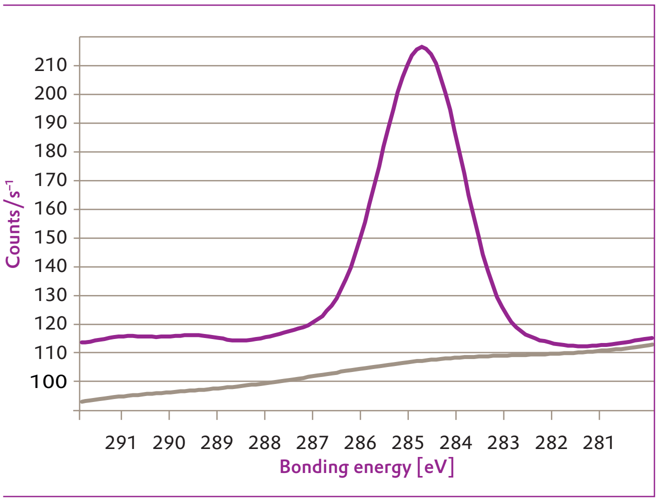
AEROSIL® 200

AEROSIL® R 974

TEM images of hydrophobized AEROSIL® grades such as AEROSIL® R 972 and AEROSIL® R 974 show that giving AEROSIL® surfaces hydrophobic properties significantly improves the separation of the aggregates (it improves the de-agglomeration behavior during the production of suitable TEM preparations to measure the specific aggregate parameters).

Furthermore, statistical evaluations of the mean primary particle size (DN) show that the TEM analyses do not indicate any changes to the primary particle size or the aggregate parameters. When attempts are made to depict a hydrophobization layer that is, as we have seen, molecularly thin, even the resolution capacity of the HRTEM reaches its limits.

Figure 44



XPS C1s-XPS signal of carbon before (gray) and after (purple) reaction with an AEROSIL® surface with an organic hydrophobizing agent

To measure the degree of coverage of AEROSIL® products with organic hydrophobizing agents for the range of the top layer of atoms, it is therefore necessary to use surface-specific methods. For such purposes, X-ray photoelectron spectroscopy (XPS/ESCA) is used (figure 4). This method measures the concentration values for the coverage of Si and O with aliphatic carbon and also reliably detects elements such as nitrogen via spectroscopy.

3.2.3 Specific surface area

It has so far been shown that the primary particle size and the structure of AEROSIL® particles can be derived from electron microscope images. For AEROSIL® products, the relationship between primary particle size and specific surface area can be determined in two completely independent ways, with the two methods providing the same result.

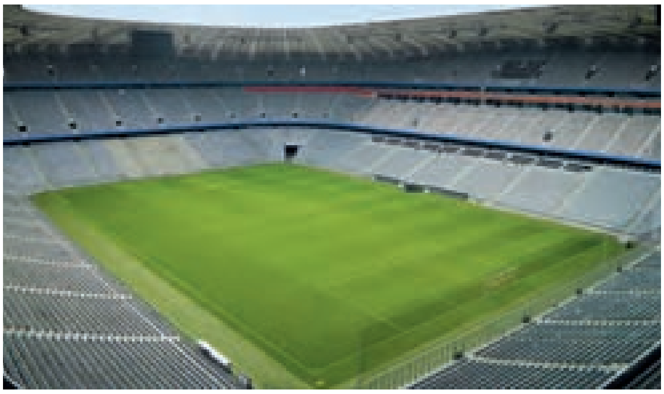


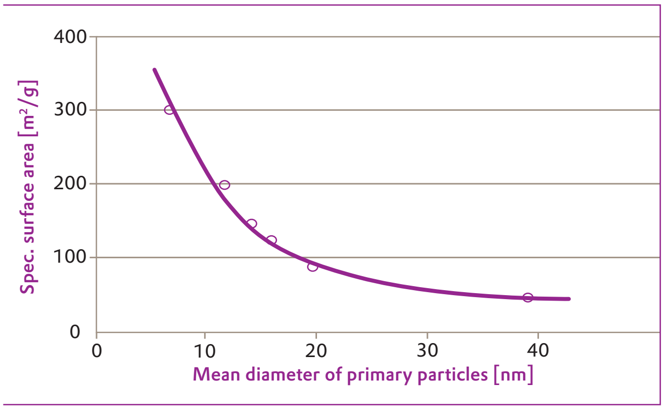
Figure 45:
AEROSIL® 200 (30 g) has the same surface area as a soccer pitch

3.2.3.1 Measuring the specific surface area on the basis of geometry

The fundamental relationship between the primary particle size and the specific surface area can be derived quantitatively from the TEM images (section 3.2.2). In this measurement method, a ZEISS TGZ 3 particle-size analyzer counts several thousand particles using the ENDTER and GEBAUER method and the specific surface area is calculated.

Figure 46 illustrates how the specific surface area increases sharply as particle diameter decreases. 30 g of AEROSIL® 200 has the same surface area as a soccer pitch (figure 45). The following thought experiment should help to illustrate the significance of material fineness:

Figure 46



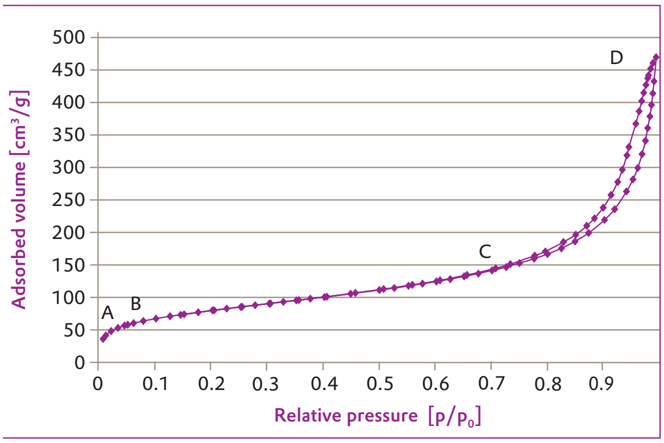
Specific surface area in relation to the mean AEROSIL® primary particle diameter

A cube is cut up to make eight identical smaller cubes, by cutting each edge in half. The total mass of the material naturally remains the same. The total surface area of the eight small cubes, however, is twice as large as the surface area of the original cube, even though the surface area of each individual small cube is smaller. This process can, in theory, be repeated indefinitely. The surface area of each AEROSIL® primary particle is very small. On the other hand, the specific surface area is very high, as the total number of particles is very large. If the primary particles in 1 g AEROSIL® 200 were piled up one on top of the other, the resulting chain would reach to the moon and back 8.5 times!

3.2.3.2 Measuring the specific surface area and pore distribution on the basis of gas adsorption

The most commonly used and most reliable method for measuring the specific surface area and pore distribution of highly dispersed solids is based on gas adsorption as caused by physical interactions.

Figure 47



Sorption isotherms of N_2 on AEROSIL® 300 at 77 K

At low pressures (A) active adsorption sites are occupied initially. These include micropores (pores <2 nm), as these are places where adsorption potentials overlap, causing stronger interaction forces to act on the gas molecules. At (B) there is theoretically a monomolecular adsorbate layer on the surface, which is used to measure the specific surface area. As pressure increases, additional adsorbate layers (B–C) form, which allows the fractal dimension to be measured so the surface roughness can be characterized. As saturation pressure is reached (D), larger pores (mesopores) increasingly fill up with condensate as the result of capillary forces. The desorption curve then exhibits hysteresis (D–C).

The outer surface and the inner surface, which is accessible in the case of porous substances, are determined on the basis of the BET method (Brunauer, Emmett, and Teller). The process is described in reference 16. For AEROSIL®, N_2 is generally used as the measuring gas and adsorbed at 77 K. A preferable alternative is the adsorption of Ar at 87 K, as N_2 molecules engage in specific interaction with the silanol groups on the SiO_2 surface and position themselves vertically to the surface, which causes the specific area requirement of the molecule to be lower than the value recommended for calculating the surface area (0.162 nm²).

The BET surface areas and the derived TEM surface areas generally show a good degree of agreement. AEROSIL® 380 is an exception to this, however. In comparison with AEROSIL® 300, the particles are not finer. Rather, they show a certain degree of surface roughness.

Micropore volume (pores <2 nm) and geometrical surface area (outer surface area outside the micropores) are derived from the adsorption data on the basis of the isotherm comparison method (t-plot method as described in [17]: comparison of the measured adsorption isotherms with a reference isotherm of a nonporous material).

Most AEROSIL® products, with the exception of AEROSIL® TT 600, and precipitated silicas have no significant microporosity, unlike silica gels, for example.

An additional way to characterize the surface structure on the basis of isotherm data is to use the distribution of mesopores in the 2–100 nm range in accordance with the Barrett-Joyner-Halenda (BJH) method as described in [18].

Table 8

AEROSIL®	Spec. surf. area [m²/g]	Pore vol. cm³/g (2–100 nm)
AEROSIL® OX 50	50	0.06
AEROSIL® 130	130	0.26
AEROSIL® 300	295	0.56

Correlation between measured pore volume of fumed silicas and spec. surface area

3.3 Special physical/chemical data

The following variables are often relevant for technical matters:

- Specific surface area based on [16]
- Mean size of primary particles
- Density after tamping based on [19]
- Loss on drying based on [20]
- Loss on ignition based on [21]
- pH value [22]
- Foreign oxide content
- Chloride content
- Residue on sieve based on [23]

Descriptions are given for the analytical testing methods, while the corresponding physical/chemical data are listed in the annex of this publication.

The high temperature resistance of hydrophilic AEROSIL® (up to 850 °C for prolonged exposure) is important when AEROSIL® is used for thermal insulation, for example.

The hydrophobic AEROSIL® products have lower temperature stability than hydrophilic AEROSIL® because of their carbon content (see table 9). However, a headspace analysis of AEROSIL® R 972, for example, performed with gas chromatography/mass spectrometry at 100 °C found no organic compound content above the legal limits after a period of two hours.

Table 9

Refractive index	1.46
Solubility in water (pH 7.25 °C)	150 mg/l
Specific weight 1)	2.2 g/cm^3
Heat capacity c_p of AEROSIL® 200	**10 °C:** 0,79 J/g K **50 °C:** 0.85 J/g K
Heat of wetting of water for AEROSIL® 200	$-150 \cdot 10^{-7}$ J/m^2
Molar adsorption for free silanol groups (3,750 cm^{-1})	(4.4 ± 0.4) • 105 cm^2/mol
Temperature resistance of hydrophilic AEROSIL®	850 °C
Ignition temperature of AEROSIL® to DIN 51794 2)	**AEROSIL® R 972:** 550 °C **AEROSIL® R 974:** 530 °C **AEROSIL® R 805:** 480 °C **AEROSIL® R 812:** 460 °C **AEROSIL® R 202:** 440 °C
Resistance to acids to 5 % ammonia to 5 % sodium hydroxide solution to oxidizing agent to reducing agent	Excellent 3) Poor Very poor Excellent Excellent

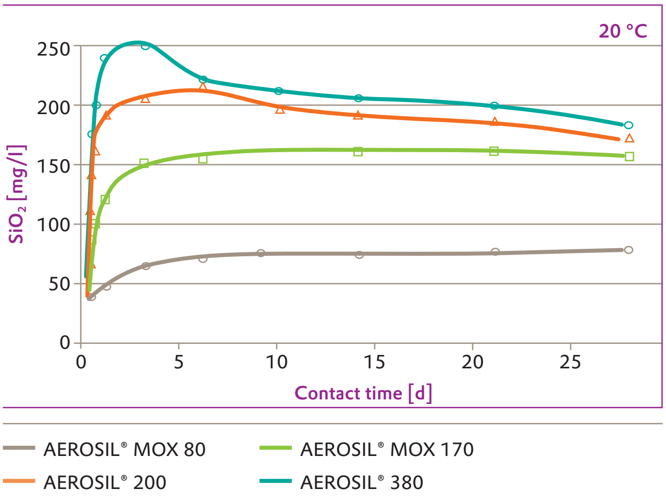
Specific physical/chemical data of AEROSIL®, not part of the specification

1) Density of a compacted sample, air comparison pycnometer, helium
2) It is obviously not possible to ignite hydrophilic AEROSIL®
3) With the exception of hydrofluoric acid

3.3.1 Water solubility

Although quartz is considered practically insoluble in water at room temperature, it actually dissolves at a rate of 0.01 % at room temperature and a pH of 7. This also applies for all hydrophilic AEROSIL® products in a state of equilibrium. The dynamics of the dissolving process are, however, very different: Quartz only reaches the equilibrium value after long contact periods, while AEROSIL® products form supersaturated solutions rapidly because of their fineness and their amorphous nature.

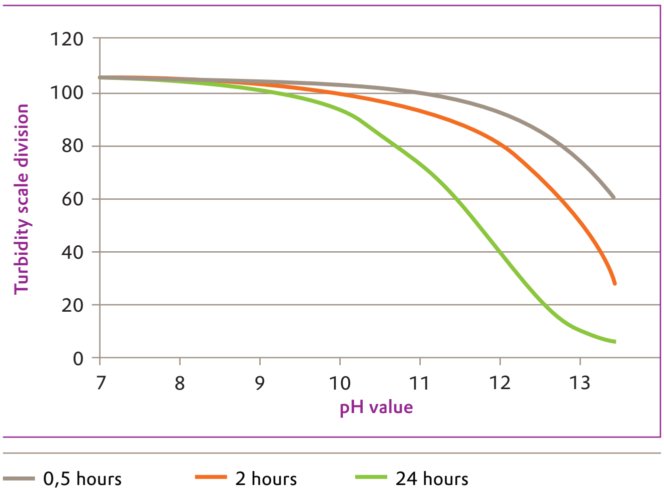
Figure 48



Solubility of various AEROSIL® products in water at 20 °C > in relation to the contact time

Figure 48 shows the solubility of various AEROSIL® products in water at 20 °C. As alkalinity rises, silica formation increases rapidly in AEROSIL® products. As figure 49 demonstrates, this process already becomes clearly noticeable around a pH level of 10.

Figure 49



Solubility of AEROSIL® 200 in sodium hydroxide solution, turbidity after various exposure times, 1 % aqueous dispersion

The solubility is measured by means of an adjusted version of OECD test 105 with the piston method at 37 °C, followed by measurement of the Si content in solution by means of ICP-OES (inductively coupled plasma optical emission spectrometry) at room temperature. Samples are taken after 72, 96, and 120 hours.

The following water solubility values were measured in accordance with OECD test 105:

Hydrophobic AEROSIL®, e.g. AEROSIL® R 972, AEROSIL® R 974, AEROSIL® 812 S is water soluble in the range of 0,05 – 0,1g/l, whilst hydrophobic grades the solubility increases in the range of 0,05 – 0,016 g/l, in accordance with OECD test 105. ≤0.05 – 0.1 g

3.3.2 Thermal conductivity

AEROSIL® products are ideal for insulation applications, thanks to their pore structure and low density. The solid thermal conductivity is suppressed by extending the pathways and by the interfaces of the framework-forming aggregates and agglomerates, while the pore structure has a positive influence on the gas-conducting portion of the thermal transport. A diagram illustrating this is given in figure 50.

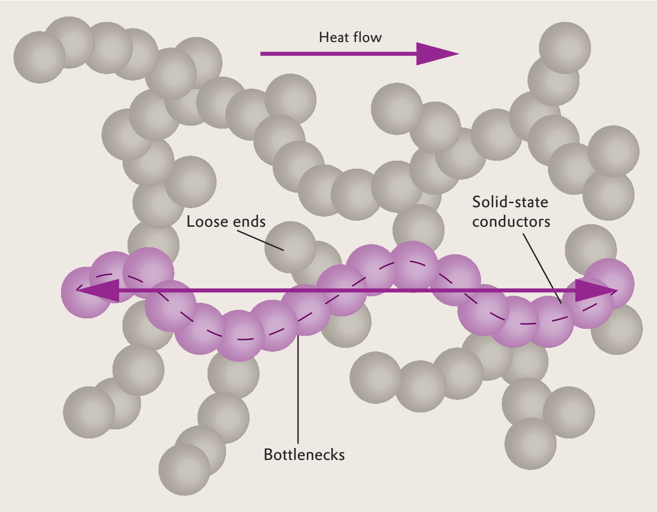
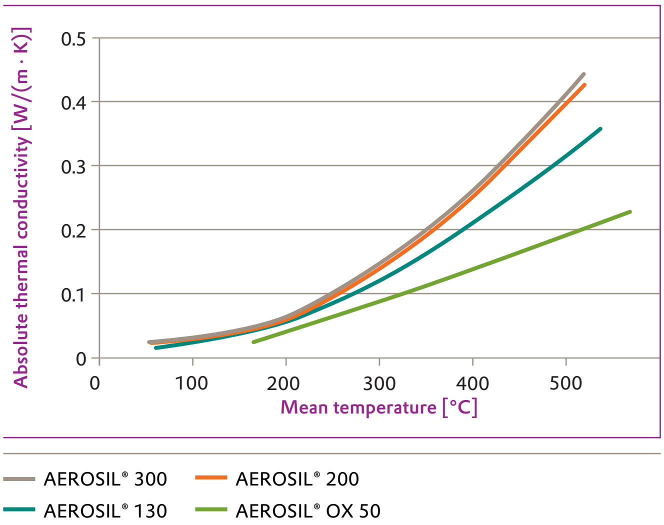


Figure 50: Diagram showing heat conduction through a porous molded body consisting of AEROSIL®, cf. figure 41a [source: ZAE Bayern – Bavarian Center for Applied Energy Research]

The absolute thermal conductivity of certain AEROSIL® products in relation to the mean temperature of the heat transfer is shown in figure 51.

Figure 51

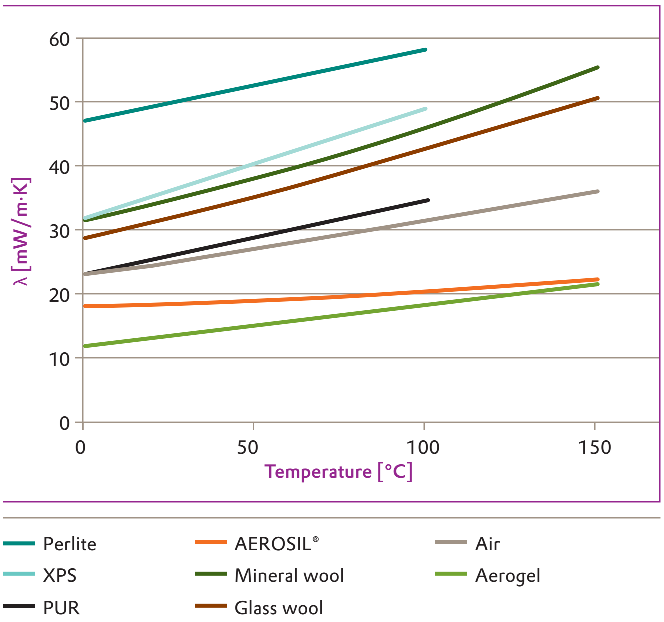


Absolute thermal conductivity of certain AEROSIL® products, compressed density 200 g/l

Adding infrared opacifiers such as suitable carbon black, silicon carbide, rutile, or magnetite powders to the silica allows thermal conduction to be lowered even more by further suppressing the radiation portion of the conduction.

Typical thermal conductivity levels for standard high-performance insulation materials are shown in figure 52. Although the thermal conductivity of many insulation materials significantly increases as temperature rises, the increase in thermal conductivity is moderate for a thermal insulation mixture containing AEROSIL®.

Figure 52



Temperate-dependence of thermal conduction for selected insulation substances

The excellent insulation properties and the high temperature resistance of silica-based insulation materials make them especially suitable for applications in higher temperature ranges.

3.3.3 NMR spectroscopy

Silicon contains the NMR-active isotope ^{29}Si at a level of 4.9 %. ^{29}Si NMR spectroscopy (NMR = Nuclear Magnetic Resonance) can be used to examine and characterize post-treated hydrophobic AEROSIL® and, to a lesser extent, hydrophilic AEROSIL®. As AEROSIL® cannot be dissolved in a solvent, special solid-state NMR techniques (MAS = magic angle spinning, CPMAS = cross polarization magic angle spinning) are used for spectrum scanning [25]. With the characteristic positions of the signals in the spectrum used as a reference, it is possible to distinguish between similar groups such as methyl-, dimethyl-, and trimethylsilyl groups. M, D, T, and Q groups are described in the nomenclature (figure 53). The typical signal positions (chemical shifts) of these groups are summarized in table 10. Figures 54–56 show ^{29}Si NMR spectra for different types of AEROSIL®.

NMR spectroscopy can reveal whether a reagent, such as a hydrophobizing agent, will bond with the AEROSIL® surface. If so, the signal intensity of the hydrophobizing agent will increase and that of the free silanol groups (Q2 and Q3 groups) will decrease.

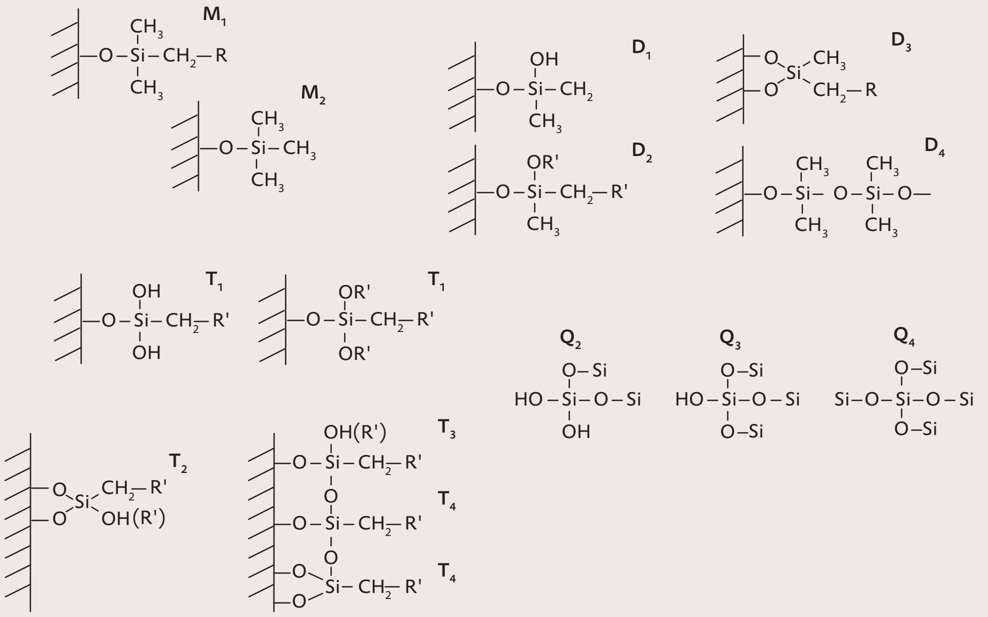


Figure 53: Diagram of the various molecular structural elements that can be distinguished between with the help of ^{29}Si NMR spectroscopy (R, R′ = alkyl or functional residue) [24]

Table 10

Structural element	δ ^{29}Si [ppm]
M_2	8
D_1	−4
D_2	−7
D_3	−10
D_4-D_4'	−14 to −21
T_1	−46
T_1'	−50
T_2	−56
T_3+T_3'	−59
T_4+T_4'	−64 to −70
Q_2	−91
Q_3	−101
Q_4	−110

List of the silane signals (chemical shifts) of ^{29}Si nuclei for various structural elements (for their arrangements see figure 53) relative to OMCT (octamethylcyclotetrasiloxane = −20 ppm) [25–28]

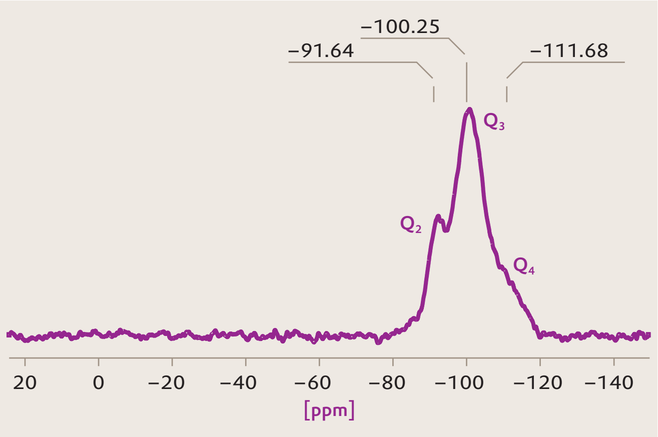


Figure 54: ^{29}Si solid-state NMR spectrum (CPMAS) of AEROSIL® 200. The various Q groups with various silanol groups are visible in the spectrum (see section 3.3.3)

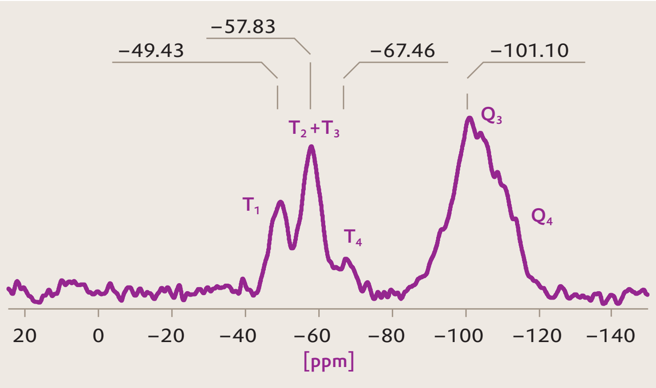


Figure 55: ^{29}Si solid-state NMR spectrum (CPMAS) of AEROSIL® R 805. Various T groups of the octyltrialkoxy residue are present in addition to the Q group

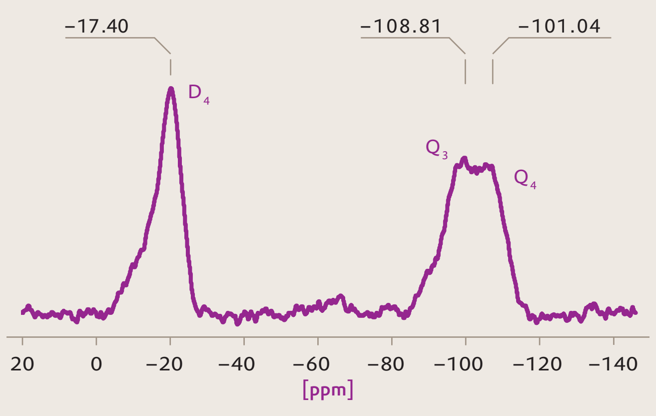


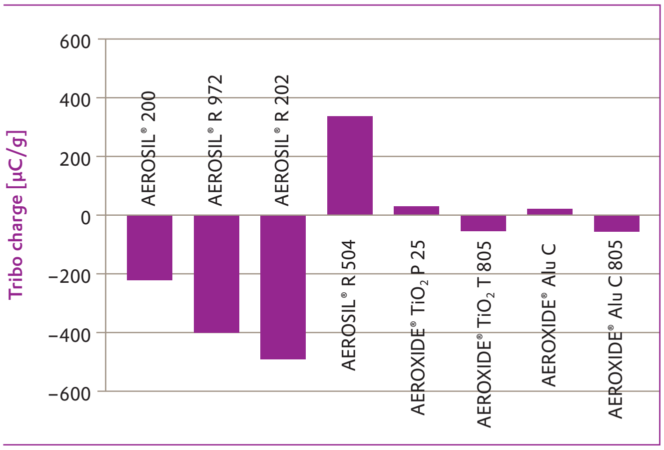
Figure 56: ^{29}Si solid-state NMR spectrum (CPMAS) of AEROSIL® R 972, an AEROSIL® that is reacted with DDS (dimethyldichlorosilane). In addition to the Q group, D4 groups preferentially occur for $(CH_3)_2SiO_2$ fragments, indicating that the DDS has reacted on the surface to a large extent

3.3.4 Triboelectricity

Triboelectric properties are important in certain applications such as toners. Figure 57 contrasts the specific charge values (q/m values, charge-mass ratios) for certain selected products.

As illustrated in the diagram, hydrophobizing can shift the triboelectricity of the particles to negative values and strengthen it. Corresponding chemical post-treatment also enables powder to be produced that can be positively charged.

Figure 57



Specific measurement of the tribo charge [µC/g] of fumed oxides, measured with an uncoated ferrite carrier by means of in-house Evonik methods

3.3.5 Refractive index

AEROSIL® products all have a refractive index of approximately 1.46, with only marginal differences between varieties. The indices are measured using the immersion method. This involves suspending the AEROSIL® sample in a suitable liquid (such as a mixture of limonene and ethanol). A turbidity-temperature curve is produced. The refractive index of the AEROSIL® variety is equivalent to the refractive index of the liquid at the turbidity minimum. As the refractive index of the liquid at the temperature of the turbidity minimum is known, the refractive index for the particular AEROSIL® variety can also be deduced.

3.3.6 Density

Density is mass divided by volume. In the case of a porous substance or bulk particle material, the volume is made up of the solid volume and the pore volume. The definitions of density vary depending on which volume percents are considered and which pores are included in the solid volume. This assortment of definitions gives rise to a diverse range of related terms and analytical methods, including bulk density, tamped density, apparent density, gross density, absolute density, solid density, true density, and helium density, to name but a few.

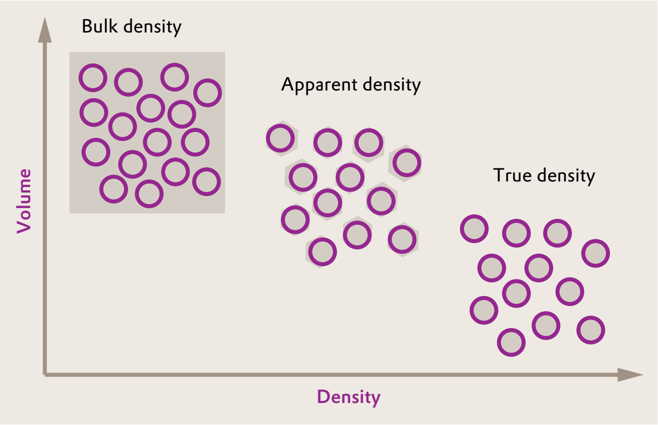


Figure 58: Volumes and densities of solids

Solid density

To determine the density of a solid of any particular form with any open pores excluded from the volume (that is, the true density), the main method used is pycnometry. In pycnometric methods, the pore volume and the inter-particle space are measured with the help of a displacement liquid or a gas.

Pressurized mercury, water, or high-boiling organic liquids can be used as displacement liquids. To enable the pores to be filled, the samples are evacuated in advance (the air is removed by centrifuging). Depending on the wetting properties and the molecule size of the liquid, the liquid may fail to fill the smallest pores, however.

To ensure that all the accessible pores are filled, gases can be used (the most common being helium). Certain pores being closed or the measuring gas being adsorbed or absorbed by the solid may distort the measurement. Gas pycnometry measurements are easier to carry out than pycnometric measurements in liquid.

In the gas pycnometer (figure 59) the volume of the test substance is measured on the basis of the pressure change of an inert gas (helium) in a cell with changeable volume. The volume can be changed by opening the valve to a second chamber. All the cell volumes are measured by means of calibration with a calibration block. The test substance is dried before the measurement and repeatedly purged with the measuring gas in the measuring chamber to release remaining impurities (such as trapped air). To calculate the density, weighing is performed before or after the volume measurement. The process is described in DIN 66137-2/EP 2.9.23 [29].

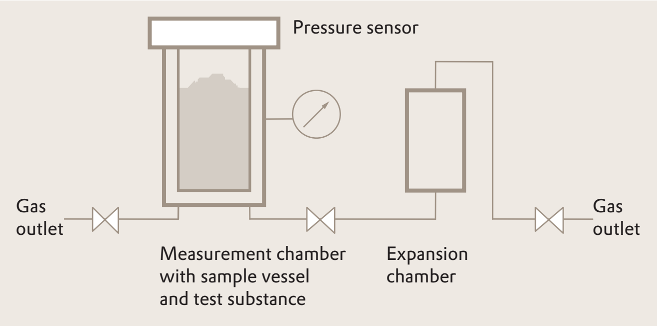


Figure 59: Helium pycnometer

The typical value for solid state densities of AEROSIL®, as measured by helium pycnometer, is 2.2 g/cm³

Bulk density

Bulk density is a key parameter in silica applications. The sample is loosely filled into a 250 ml measuring cylinder up to the top marking, allowing the sample time to settle. The sample is then weighed. The sample should not be tapped to make it more compact.

Tamped density and tamped volume

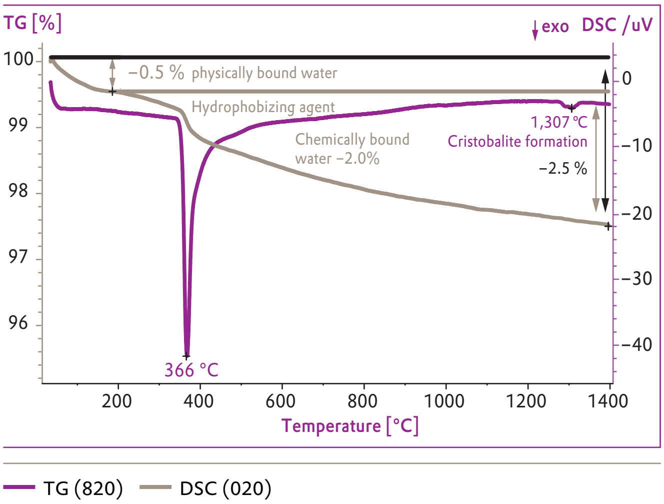
Tamped density or tamped volume is an important specification parameter for silicas. It provides more reproducible values than bulk density and is measured in the tamped volumeter. Approximately 200 ml of sample is filled into a measuring cylinder. A camshaft then lifts the measuring cylinder by 3 mm and drops it 1,250 times in a period of 5 minutes.

Hydrophilic and hydrophobic AEROSIL® have tamped densities of approximately 50–60 g/l. AEROSIL® OX 50 has a value of approximately 130 g/l, because of its limited structure. Compacting can increase the tamped density of hydrophilic AEROSIL® to approximately 120 g/l and that of hydrophobic AEROSIL® to 90 g/l.

3.3.7 Thermal stability

Simultaneous thermal analysis (STA = thermogravimetry TG with simultaneous differential scanning calorimetry DSC) can provide an overview of the processes taking place while the substance is heated.

Figure 60



STA diagram of a hydrophobic AEROSIL®, measured in the presence of air

The TG curve initially displays the release of physically bound water (moisture). Depending on the AEROSIL® variety, the additives (such as hydrophobizing agents) begin to burn away at around 300 °C, which is accompanied by an intense exothermic reaction.

The emission of the chemically bound water (silanol group condensation) takes place at higher temperatures and spans a wide area.

Finally, at around 1,300 °C, the DSC curve displays the exothermic cristobalite formation.

During the cooling process, this cubic high-temperature form of cristobalite transitions into the tetragonal low-temperature form at approximately 250 °C in an exothermic, displacive transformation (involving shifting of the atom layers and change in the Si-O-Si bond angle). This reversible phase transformation appears as an endothermic peak in a similar temperature range in a second heating.

When AEROSIL® and precipitated silicas are compared, they display noticeably different crystallization temperature, heat of crystallization, and crystallization rate. Precipitated silicas crystallize within a narrower temperature range (with a sharper exothermic DSC peak), starting as low as 1,000 °C.

TG/MS/FTIR evolved gas analysis (thermogravimetry combined with mass spectrometry and infrared spectroscopy) provides additional information on the composition of the gases released during the heating process and what temperatures they occur at (their emission curves).

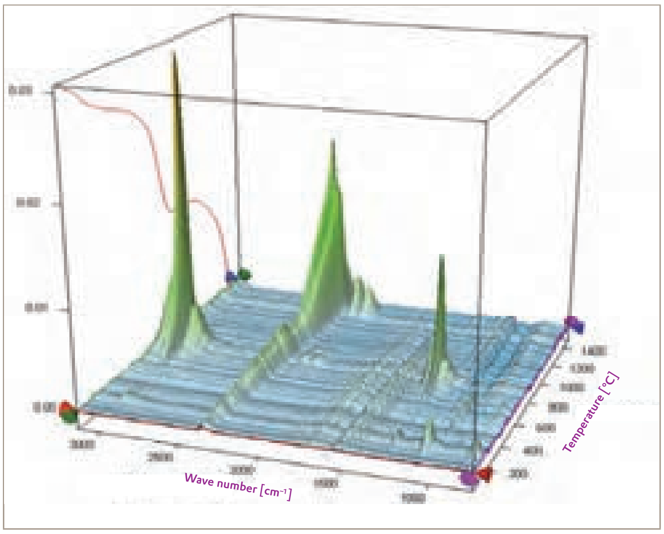


Figure 61: 3D depiction of the IR spectra for AEROSIL® R 104 with TG curve added, measured in the presence of helium

Figure 61 illustrates this with a surface-modified AEROSIL® used as an example. Water begins to be released when heating is started, and this continues with varying intensities until approximately 1,300 °C. From approximately 100 °C onward, the emission of various organic substances is registered, such as octamethylcyclotetrasiloxane with an emission peak at 190 °C and hexamethylcyclotrisiloxane at approximately 570 °C. The intensive methane emission takes place during a second weight loss stage, peaking at 750 °C.

Carbon dioxide and carbon monoxide mainly occur during a third decomposition stage at approximately 1,360 °C, and then again with less intensity at 760 °C.

3.4 Surface chemistry

The most important property of AEROSIL® fumed silica is the high specific surface area, in addition to its particle fineness. As mentioned above, the specific surface area is determined by the mean size of the primary particles. As AEROSIL® products have a large surface area relative to their mass, surface chemistry is an important factor that determines many application-related properties.

3.4.1 Two functional groups determine the chemistry

There are two main functional groups that are relevant for AEROSIL®: the silanol groups and the siloxane groups, as shown in figure 62.

The silanol groups have a hydrophilic nature, which means that these groups readily mix with water. Furthermore, hydrophobic varieties of AEROSIL® can be produced thanks to the chemical reactivity of these silanol groups.

The siloxane groups, on the other hand, are largely chemically inert (that is, chemically inactive). They also display hydrophobic (water-repelling) behavior. In AEROSIL® varieties that do not undergo post-treatment, however, the hydrophilic nature of the silanol groups is predominant. These two functional groups give rise to a highly complex reaction chemistry. One of the reasons for this is that distinctions must be made between the following groups:

- Isolated silanol groups
- Bridged silanol groups
- Geminal silanol groups
- Vicinal silanol groups
- (Strained) siloxane groups

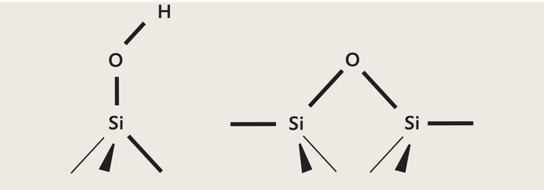


Figure 62: Silanol groups (left) and siloxane groups (right)

The individual groups shown in figure 63 will be discussed in further detail below.

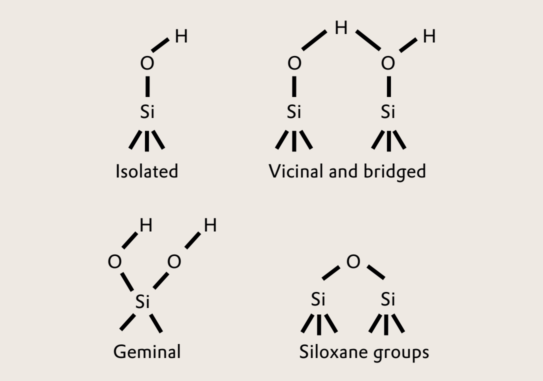


Figure 63: SiO_2 surface groups

We will start by discussing the quantification of silanol groups, however, as this group has special significance as mentioned earlier.

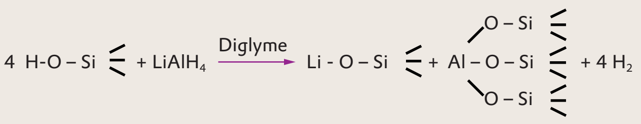
3.4.2 Determining the concentration of silanol groups

Because of their reactivity, the silanol groups can be quantified by various methods. In the literature, the following methods for determining the SiOH concentration are described:

- Igniting dried AEROSIL®
- Chlorinating ≡ SiOH
- Reacting ≡ SiOH with phenyllithium
- Diazomethane and alkylmagnesium halides
- Reacting ≡ SiOH with B_2H_6
- Reacting ≡ SiOH with $LiAlH_4$
- Infrared spectroscopy

3.4.2.1 The lithium alanate method

One of the most precise and straightforward methods of quantifying the SiOH concentration on the AEROSIL® surface is reacting dried AEROSIL® with $LiAlH_4$ in accordance with the following equation:



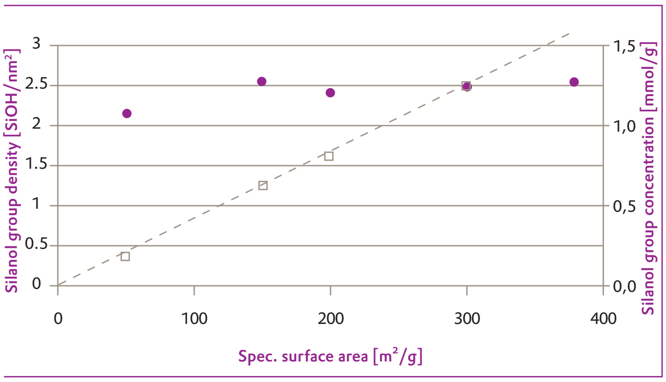
$$4\ \mathrm{H{-}O{-}Si}\equiv\ +\ \mathrm{LiAlH_4}\ \xrightarrow{\text{Diglyme}}\ \mathrm{Li{-}O{-}Si}\equiv\ +\ \underset{\diagdown \mathrm{O-Si}\equiv}{\overset{\diagup \mathrm{O-Si}\equiv}{\mathrm{Al-O-Si}\equiv}}\ +\ 4\ \mathrm{H_2}$$

This method involves measuring pressure to determine the amount of hydrogen formed and thus the silanol group density. As the hydride ion, functioning as an aggressive agent, is very small and highly reactive, all the silanol groups on the surface are detected, including the bridged ones. This is in agreement with the determination of the residual silanol group density of hydrophobic AEROSIL®, which contains practically no free silanol groups according to the IR spectroscopy findings.

As figure 64 illustrates, an initial approximation reveals no relationship between silanol group density and specific surface area. In aged material (stored longer than one week after production, that is, normal product), approximately 2.5 SiOH/nm² is measured – only AEROSIL® OX 50 displays slightly lower SiOH densities (approximately 2.2 SiOH/nm²), as the result of the production process.

The absolute concentration of the silanol groups increases linearly as the specific surface area increases. This explains the high surface areas of AEROSIL® products (assuming good dispersibility).

Figure 64



Total silanol group concentration of hydrophilic AEROSIL® as determined by the $LiAlH_4$ method

3.4.2.2 Infrared spectroscopy

In addition to the $LiAlH_4$ method, infrared (IR) spectroscopy is an important tool for the qualitative and the quantitative detection of silanol groups in the lab [30, 31, 32].

The polar SiOH group displays intensive bands in the IR spectrum. The band position and the band form are dependent on whether the silanol group is isolated or hydrogen bonds have been formed with other OH groups of adjacent SiOH groups. Characteristic IR absorption bands for AEROSIL® and modified AEROSIL® are summarized in table 11.

Table 11

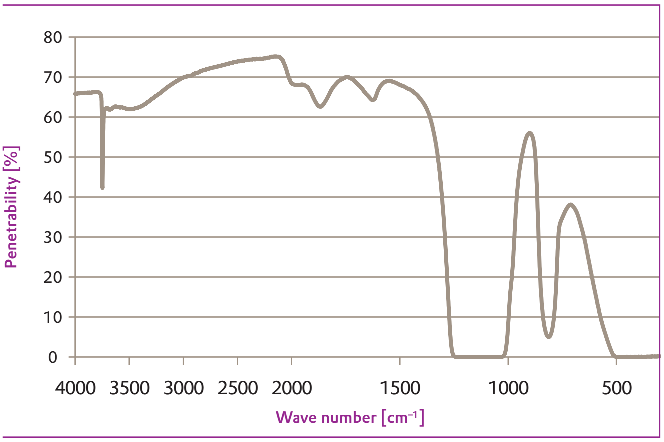
Structural element	cm^{-1}
H_2O	5,200
SiOH (combination vibration)	4,550
SiOH (isolated)	3,750
SiOH (bridged) proton acceptor	3,715
SiOH proton donator	3,510
SiOH (bridged)	3,000 – 3,500
-CH	2,900 – 3,000
-OD	2,760

List of characteristic IR absorption bands for AEROSIL® and modified AEROSIL®

For the scanning of the spectra, the sample material is prepared in a dispersing agent in the form of fine layers of powder, a compacted sample, or a powder bed. Quantitative results can then be obtained either relatively (in comparison to a reference band) or absolutely (on the basis of the Lambert-Beer law). This requires the values for the layer thickness and the molar extinction coefficient of the silanol groups. For isolated silanol groups at 3,750 cm^{-1}, a value of 4.4 · 105 cm^2/mol was determined by Mathias and Wannemacher, for example [33].

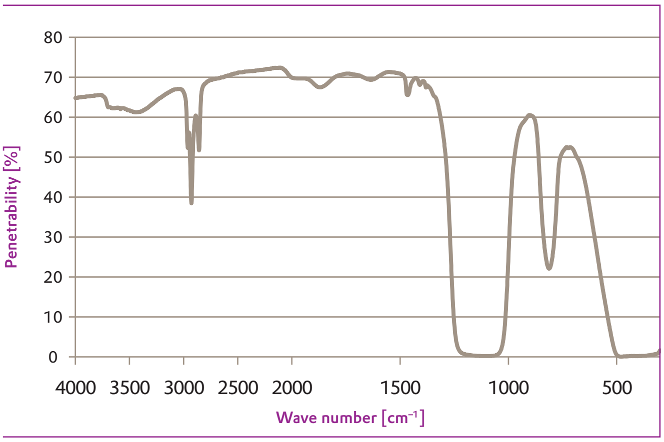
The exchangeability and accessibility of silanol groups in silica and AEROSIL® can be verified in deuteration experiments. This involves treating the sample with D_2O and replacing the protons of the silanol group with deuterium (H-D exchange). This makes it possible to determine the proportion of inaccessible silanol groups, for example [34].

Figure 65



IR spectrum of AEROSIL® 150. At 3,750 cm^{-1} the sharp SiOH band of isolated silanol groups is clearly visible

Figure 66



IR spectrum of AEROSIL® R 805: Isolated silanol groups are no longer visible. In their place, the alkyl bands of the hydrophobizing agent can be seen

3.4.3 Interparticle interactions

To describe the rheological properties of dispersions that contain AEROSIL® and the structure of powdered AEROSIL® it is essential to consider the ways in which the SiO_2 particles interact with one another and with the dispersion phase. These interactions could include the following:

- VAN DER WAALS attraction forces
- Electrostatic interactions (COULOMB interactions)
- Acid/base interactions
- Orbital interactions

3.4.3.1 Hydrogen bonds

Hydrogen bond interactions are considered a subgroup of acid/base interactions. In a model developed by E. R. Lippincott and R. Schroeder, hydrogen bonds are depicted as superposed protomeric contributing structures. In figure 68 the proton has two stable layers within the hydrogen bond. The proton delocalization across the region of the two potential wells is performed by means of a tunnel effect with high frequency (comparable with ammonia inversion vibration).

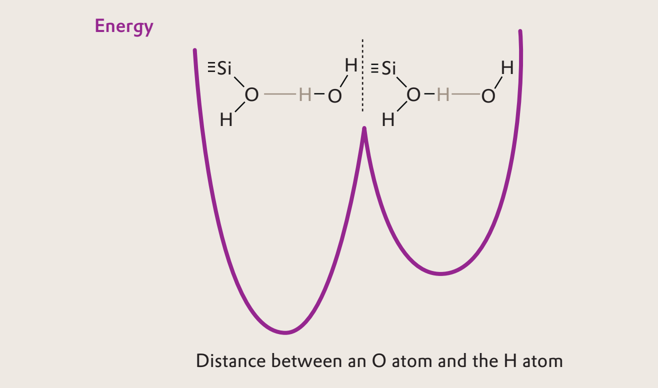


Figure 67: Double potential trough of the hydrogen bond between the O atom of a silanol group and the H atom of a water molecule (diagrammatic, the O-O distance remains constant)

The energy of the hydrogen bond (4–40 kJ/mol) is determined by the OHO angle. It has a minimum level when the three atoms are arranged in a linear formation.

Compared with a covalent C-H bond (approximately 360 kJ/mol), the hydrogen bond is a moderately weak interaction.

It is, however, stronger than the VAN DER WAALS forces. Hydrogen bonds play a very important role in nature. The mean kinetic energy of translation is approximately 4 kJ/mol at human body temperature. Hydrogen bonds splitting and new ones being formed are therefore elementary metabolic processes. Complex molecular structures can only be maintained if the nature of the H bonds is configured correctly. At the same time, H bonds allow structural changes to be performed rapidly.

Similar “reactions” take place constantly on the AEROSIL® surface. The loose structure of the AEROSIL® agglomerates can be explained by the simple forming and splitting of hydrogen bonds.

Because of the low silanol group density of approximately $2.5/nm^2$ there is no way for intraparticle bonds to be formed (in contrast to precipitated silicas). The presence of isolated silanol groups has also been substantiated in IR spectroscopy tests, so only interparticle hydrogen bonds need to be considered.

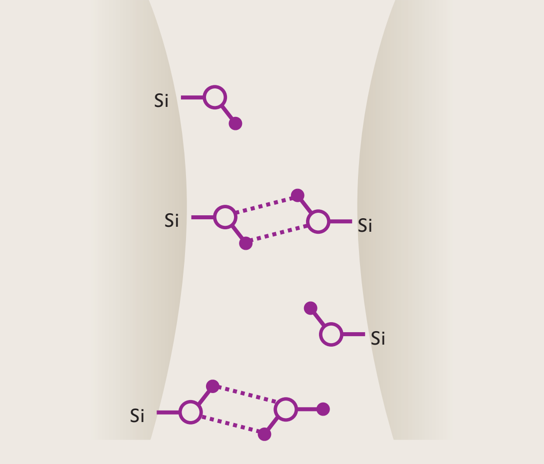


Figure 68: A hydrogen bond interaction between two AEROSIL® primary particles

In the infrared system, the frequency of vibrations between the oxygen atom and the hydrogen atom in the OH bond decreases in the case of bridged systems, which is equivalent to a loosening of the OH bond. A silanol group’s probability of finding a suitable neighbor to form an H bond with rises as particle fineness increases. Accordingly, the density of the free silanol groups ($SiOH/nm^2$) decreases as specific surface area increases (figure 69). This is also reflected in the dispersibility of AEROSIL®, which is reduced as the specific surface area increases.

3.4.3.2 Moisture balance

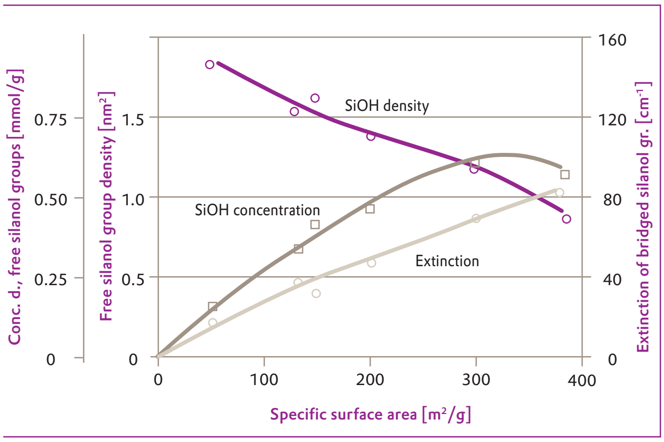
3.4.3.2.1 Moisture balance at room temperature

Water molecules can be anchored to silanol groups and thus to the AEROSIL® surface via hydrogen bonds, as discussed in the previous section. Water vapor therefore has a relatively high affinity for hydrophilic AEROSIL® and is adsorbed well (wetting heat of water with AEROSIL® 200: −150 x 10 − 7 J/ 2, compared with quartz, which has a higher affinity for liquid water: −610 x 10 − 7 J/m^2).

The intake of water from the atmosphere is dependent on the humidity and is reversible (figure 70).

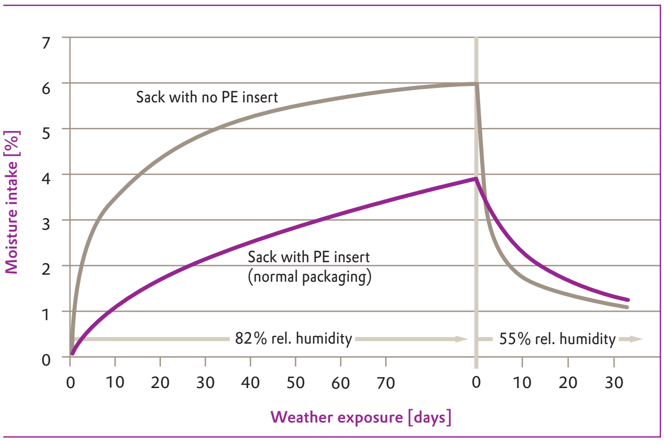
Depending on the storage conditions, the moisture in the air can be adsorbed with significant speed and released just as quickly. In practice, an equilibrium is reached slowly, as the outer layer in a sack of AEROSIL® shields the portion that is deeper inside. The moisture balance is then mainly determined by diffusion processes.

Figure 69



Silanol group density as a function of the specific surface area of AEROSIL® (IR, samples 1 year old)

Figure 70

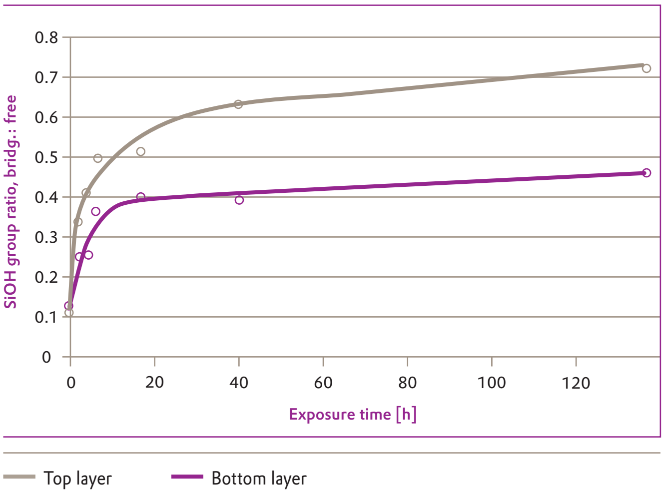


Moisture intake (storage at 82 % rel. humidity) and release (storage at 55% rel. humidity) of AEROSIL® 200 (10 kg sack, closed, red line: PE coating = normal packaging, gray line: sack with no PE layer). Additional notes on storing AEROSIL® can be found in section 7.4, Storage durability of AEROSIL®

This behavior can be demonstrated in a simple weather exposure test. Test tubes filled with AEROSIL® 150 are stored at a relative humidity of 90 %.

The water adsorption changes the absolute amounts for the extinction values relating to the free and the bridged silanol groups. The change in the quotient of bridged and free extinction depicts the ongoing intake of water. Figure 71 shows that the top layer of AEROSIL® differs considerably from the bottom layer. The results can be explained by a slow diffusion of moisture toward the inside of the AEROSIL® bulk material.

Figure 71



Comparison of the silanol group ratios in the bottom and top layer of AEROSIL® 150 in a test tube in relation to the duration of moisture exposure (90 % relative humidity)

The maximum water intake of hydrophilic AEROSIL® (a thin layer) rises as specific surface area increases and thus correlates to the total number of silanol groups. With the largest BET surface area, AEROSIL® 380 naturally takes on the most water.

For the hydrophobic AEROSIL® varieties, the total number of silanol groups and thus the moisture intake is significantly reduced.

AEROSIL® is not hygroscopic. While it does take moisture from the air, it readily releases this moisture again under slightly different “normal” conditions. In each case, equilibrium states are reached, given sufficient storage time.

At least 95 % of the water adsorbed after genesis is removed simply by applying a vacuum (4–10 mbar), even at room temperature. This can be easily verified on the basis of the IR spectra.

It is well known that solid hygroscopic substances tend to melt or clump, but AEROSIL® remains unchanged under the conditions mentioned.

3.4.3.2.2 Aging

The Si-O-Si bond angle in a siloxane bond can vary between 120 ° and 180 °. Strained siloxane bonds display greater reactivity than water.

As shown in table 12, this hydrolysis causes the silanol group concentration to increase across the storage period. The amount of water required for this is very low (approximately 0.25 %). Directly after flame hydrolysis, AEROSIL® receives this quantity of moisture in the form of physically bound water.

Table 12

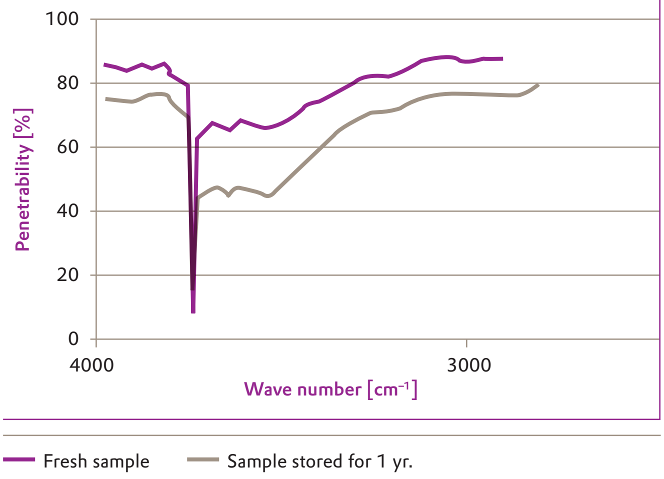
	3 days old	Stored for 1 yr.	Determination method
Isolate SiOH/nm^2	1.5	1.15	IR
Total SiOH/nm^2	1.8	2.65	$LiAlH_4$

Silanol group density of AEROSIL® 200 stored for one year directly after production (original sack)

As the IR spectra in figure 72 illustrate, the physically bound water also accumulates on the free silanol groups over time, while they transition into bridged silanol groups.

Figure 72

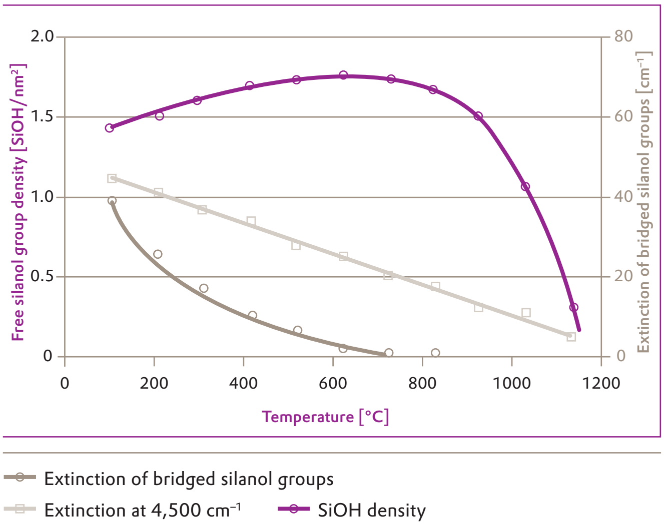
IR spectra of AEROSIL® 200



3.4.3.2.3 Moisture balance at elevated temperatures

If AEROSIL® 200 is heated, the concentration of free silanol groups increases up to 1.8 SiOH/nm^2 (at approximately 600 °C). At the same time, the intensity of the bridged silanol groups drops to zero (at approximately 700 °C) – see figure 73. Minor changes in the band form at 3,750 cm^{-1} during heating between 450 °C and 1,100 °C are, according to E. Knözinger, P. Hoffmann, and R. Echterhoff, the result of a change in concentration in the geminal silanol groups (= $Si(OH)_2$).

Figure 73



Separation of silanol groups from AEROSIL® as caused by thermal treatment (IR inspection, 1 h tempering time)

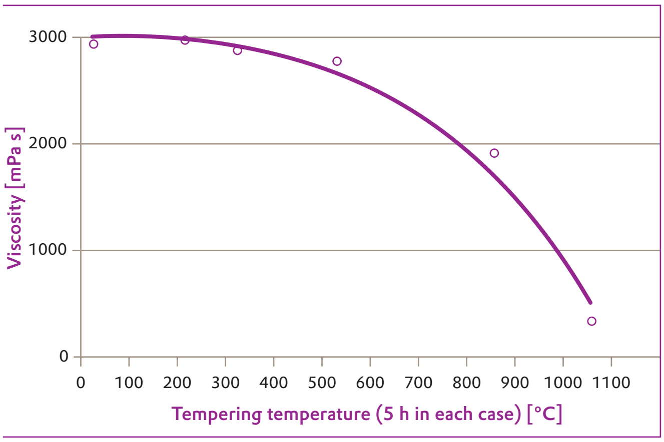
IR spectroscopy was used to track the affinity of tempered AEROSIL® for water: The samples treated at 500 to 900 °C take on water more readily than AEROSIL® tempered at 1,100 °C. It is evident that strained siloxane bonds, at high temperatures, can transition as the result of small framework rearrangements into fewer strained systems, which are later not accessible to a subsequent hydrolysis.

The AEROSIL® sample tempered at 1,100 °C displays simultaneous growth of two IR bands at 3,715 cm^{-1} (proton acceptor) and 3,510 cm^{-1} (proton donator). The intensities are the result of a chemisorption of water and can be attributed to H-bridged vicinal OH groups. Bridging chains of greater length, $(OH)_3$, $(OH)_4$, etc., were not observed.

At lower tempering temperatures (approximately 900 °C) the pure adsorption of water molecules by present silanol groups can also be observed. AEROSIL® that was only treated at 450 °C (or even lower temperatures) shows an additional band at 3,675 cm^{-1} in its reaction with water. Tempered samples do not exhibit these vibrations, indicating irreversible processes at high temperatures.

The reduced thickening effect of AEROSIL® treated at high temperatures can also be attributed to an irreversible loss of silanol groups – see figure 74.

Figure 74



Thickening effect of tempered AEROSIL® 200 (unsaturated polyester resin, tempering time: 5 h in each case)

You want to choose from a vast assortment of products for a diverse range of applications?

Products

- Hydrophilic products
- Post-treated products
- For the food industry
- For the pharmaceutical industry

Introduction

Manufacture

Properties

Products

Application-related effects

Quality and product reliability

Handling and availability

Annex

4. Products

In 1941, Degussa, as the company was known at that time, began to develop a white filler material for the tire industry to replace industrial carbon black. All the filler materials that were in use could only be produced from oil.

One year later, small-scale production began for an extremely fine silica in a high-temperature flame synthesis process. This silica now forms the foundation of many technical processes and products. The idea of using fumed silicas, as they are called today, in tires did not come to fruition in the first few years of development.

The silicas produced in the process developed by Harry Kloepfer have been marketed under the brand name AEROSIL® since 1943. In 1944 production began in Rheinfelden and in 1952 large-scale production started, based on a newly developed flame procedure.

With its experience, development, and research, Evonik was able to further expand its position as the leading manufacturer of fumed silica.

There are all kinds of things that make up part of everyday life that owe their existence to fumed silicas.

AEROSIL® is used, for example, in earthquake-proof foundations for buildings, in silicone sealants for bathtubs, in insulation material, in the food industry, in the pharmaceutical industry, as a separator for ink particles in toners and in paints and coatings that are much easier to apply thanks to AEROSIL® as an additive (see table 2, page 10).

Figure 75:

Dr. Harry Kloepfer, chemist at Degussa (now Evonik) and inventor of AEROSIL®, the silica produced in a pyrogenic process

4.1 Hydrophilic grades

The different varieties of AEROSIL® are divided up into two groups: untreated (hydrophilic) grades and grades that have undergone chemical post-treatment (hydrophobic).

The hydrophilic grades can be further divided into pure silicon dioxide and mixed oxides. The names of the products with no additives – the standard AEROSIL® grades – are made up of the brand name AEROSIL® and a particular number. The number represents the product's specific surface area (BET), which is the most important property when it comes to distinguishing between the different fumed silica types.

These AEROSIL® products form the basis of additional variants, such as the pharmaceutical and food products that are produced and analyzed to IPEC-GMP* and HACCP** standards.

Evonik uses various compacting techniques to produce compacted variants of certain AEROSIL® grades, which are essential for various industrial applications. These variants have a higher tamped density and bulk density than their non-compacted counterparts and are available for almost all grades of AEROSIL®.

An overview of the basic physical/chemical key figures for individual hydrophilic AEROSIL® grades is given in table 13.

Table 13: Hydrophilic fumed silicas

AEROSIL® grades	BET surface area [m²/g]	Loss on drying [%]	pH
AEROSIL® 90	75–105	≤ 1.0	3.7 – 4.7
AEROSIL® 130	105–155	≤ 1.5	3.7 – 4.5
AEROSIL® 150	135–165	≤ 1.5	3.7 – 4.5
AEROSIL® 200	175–225	≤ 1.5	3.7 – 4.5
AEROSIL® 255	230–280	≤ 1.5	3.7 – 4.5
AEROSIL® 300	270–330	≤ 1.5	3.7 – 4.5
AEROSIL® 380	350–410	≤ 2.0	3.7 – 4.5
AEROSIL® OX 50	35 – 65	≤ 1.5	3.8 – 4.8

The values given are non-binding guideline values.

* **IPEC-GMP** = International Pharmaceutical Excipients Council – Good Manufacturing Practices – quality management for pharmaceutical excipient products

** **HACCP** = Hazard Analysis and Critical Control Points – quality management for the food and animal feed industries

4.2 Surface-treated grades

4.2.1 Hydrophobic AEROSIL®

A large number of hydrophobic AEROSIL® grades have been developed to solve particular technical problems. They are created by subjecting hydrophilic grades to chemical post-treatment with alkoxysilanes, silazanes, or siloxanes. In the end product, parts of the post-treatment agent have formed a firm chemical bond with the previously hydrophilic oxide. The products, which are now hydrophobic, display low moisture uptake, particularly good dispersion properties, and an ability to configure rheological behavior, even in polar systems.

The free-flowing characteristics of powders can be increased by adding hydrophobic AEROSIL®. Furthermore, these highly dispersed silicas can improve the water resistance of certain systems. Thanks to the low moisture uptake of hydrophobic AEROSIL®, it can make textile coating water-repellent when used as an additive. A further positive effect is that adding hydrophobic silica to moisture-sensitive pigments, superabsorbers, and fire-extinguishing powders can significantly improve their storage stability. Pigments containing hydrophobic AEROSIL® disperse more easily and have greater color intensity.

Positive effects of hydrophobic AEROSIL®

- Optimum rheology during use
- Thickening of polar liquids such as epoxy resins
- Reinforcing silicone elastomers
- High content levels, for impression materials, etc.
- Improving water-repellent properties, for rust protection, etc.
- Improving dielectric properties, for cable compounds, etc.
- Flow enhancer for powders, in fire extinguishers, etc.
- Increasing scratch resistance, in coatings and plastics, etc.

An overview of the physical/chemical key figures for selected hydrophobic AEROSIL® grades is given in table 14.

Table 14: Hydrophobic fumed silicas

AEROSIL® grades	BET surface area [m^2/g]	Loss on drying [%]	pH	Carbon content [%]
AEROSIL® R 972	90–130	≤ 0.5	3.6 – 5.5*	0.6 – 1.2*
AEROSIL® R 974	150–190	≤ 0.5	3.7 – 4.7	0.9 – 1.5*
AEROSIL® R 104	125–175	≤ 1.5	≥ 4.0	1.0 – 2.0
AEROSIL® R 106	220–280	≤ 0.5	≥ 3.7	1.5 – 3.0
AEROSIL® R 202	80–120	≤ 0.5	4.0 – 6.0	3.5 – 5.0
AEROSIL® R 208	80 – 140	≤ 0.5	4.5 – 6.5	4.5 – 6.5
AEROSIL® R 805	125 – 175	≤ 0.5	3.5 – 5.5	4.5 – 6.5
AEROSIL® R 812	230 – 290	≤ 0.5	5.5 – 8.0	2.0 – 3.0
AEROSIL® R 812 S	195 – 245	≤ 0.5	5.5 – 9.0	3.0 – 4.0
AEROSIL® R 816	170 – 210	≤ 1.0	4.0 – 5.5	0.9 – 1.8
AEROSIL® NAX 50	30–50	≤ 0.5	5.5 – 7.5	0.45–0.85
AEROSIL® NY 50	20–40	≤ 0.5	5.0–6.0	2.0–3.8
AEROSIL® RX 200	115–165	≤ 0.5	5.5–8.5	1.5–3.5
AEROSIL® RX 300	180–220	≤ 0.5	6.0–8.0	3.0–4.0
AEROSIL® RX 50	25–45	≤ 0.5	6.0–8.0	0.5–1.0
AEROSIL® RY 200	80–120	≤ 0.5	4.0–7.0	3.0–5.5
AEROSIL® RY 200 L	80–120	≤ 0.5	4.0–7.0	4.4–6.6
AEROSIL® RY 200 S	65–95	≤ 0.5	4.5–6.5	3.0–5.0
AEROSIL® RY 300	110–140	≤ 0.5	4.5–5.5	6.0–8.0
AEROSIL® RY 50	15–45	≤ 0.5	4.5–7.5	3.0–4.0
AEROSIL® NX 90 G	50–80	≤ 0.5	5.0–7.5	0.7–1.5
AEROSIL® NX 90 S	50–70	≤ 0.5	5.0–7.0	0.8–1.2
AEROSIL® NX 130	85–115	≤ 0.5	5.0–7.5	1.0–3.0

*A narrower range may apply in certain regions.
The values given are non-binding guideline values.

4.2.2 Functionalized silica

In addition to subjecting AEROSIL® to surface treatment to create hydrophobic grades, there is another way of functionalizing the surface of the particles. This involves post-treatment steps, similar to the hydrophobizing process. In this case, however, the silica is given specific chemical functionalities rather than just an altered polarity.

In special applications, a suitable functional group is required to achieve the desired interaction between the matrix and the incorporated silica. For example, AEROSIL® products with methacrylate functionalities can be used in special coatings, resins, or plastic compounds with the particles functioning as an integral part of the polymer. This is done by linking the reactive groups on the particle surface to the surrounding components. Composite systems of this type boast outstanding functionalities such as scratch resistance, or specific mechanical properties.

An overview of the physical/chemical key figures for functionalized AEROSIL® grades is given in table 15.

Table 15: Functionalized silicas

AEROSIL® grades	BET surface area [m^2/g]	Loss on drying [%]	pH	Carbon content [%]
AEROSIL® R 711	125–175	≤ 1.5	4.0 – 6.0	4.5 – 6.5
AEROSIL® R 504	125–175	≤ 1.5	8.5 – 10.5	2.0 – 4.5
AEROSIL® NA 200 Y	100–150	≤ 0.8	6.5–9.5	3.0–6.5
AEROSIL® NA 50 H	30–50	≤ 0.8	8.0–10.0	≤ 2.0
AEROSIL® NA 50 Y	25–45	≤ 0.8	6.5–9.0	2.0–4.0
AEROSIL® RA 200 HS	120–160	≤ 0.8	8.0–10.0	≤ 3.5
AEROSIL® REA 90	40–70	≤ 1.0	7.5–10.0	3.0–6.0
AEROSIL® REA 200	110–150	≤ 1.0	8.0–10.0	5.0–7.5

The values given are non-binding guideline values.

4.2.3 Structured grades

Structure-modified AEROSIL® grades open up even more possibilities for supporting customers in developing new products and enhancing existing ones.

An example of this is the high content levels combined with low viscosity increases in liquid systems.

An overview of the physical/chemical key figures for individual structured AEROSIL® grades is given in table 16.

Table 16: Structured silicas

AEROSIL® grades	BET surface area [m^2/g]	Loss on drying [%]	pH	Carbon content [%]
AEROSIL® R 7200	125–175	≤ 1.5	4.0 – 6.0	4.5 – 6.5
AEROSIL® R 8200	135–185	≤ 0.5	≥ 5.0	2.0 – 4.0
AEROSIL® R 9200	150–190	≤ 1.5	3.0 – 5.0	0.7 – 1.3

The values given are non-binding guideline values.

Further information on structured grades can be found in our Technical Information 1209 "AEROSIL® R 8200 for Silicone Rubber – Fumed silica with high reinforcing properties and a particularly low thickening effect" and Technical Information 1284 "AEROSIL® R 9200 to Improve the Scratch Resistance of Paint and Coating Systems."

4.2.4 Granulated fumed silica

Unlike AEROSIL®, Evonik's core product with fine particles, AEROPERL® (AEROPERL® 300/30 and AEROPERL® 300 Pharma) is a spherical, mechanically stable granulate. It has a mean particle size of approximately 30 micrometers (this is equivalent to 30 millionths of a meter). This highly porous granulate made from pure silicon dioxide is perfect for use as a carrier for liquid or paste-like active agents, scents, flavorings, and other fluids. The granulate nature of AEROPERL® makes it particularly easy to handle, simplifying modern manufacturing processes.

Positive effects

- Low-dust, very easy to handle
- High purity
- Very good flow performance, in loaded or unloaded state
- High adsorption and soaking capacity
- Easily separated from liquids and gases

More detailed information on granulated grades of AEROSIL® can be found in our Technical Information 1341, "AEROPERL® Granulated fumed oxides."

4.2.5 AERODISP® – liquid AEROSIL® grades

AERODISP® dispersions are the perfect solution when it comes to the dust-free handling of powdered AEROSIL® in liquid media. These fumed oxides available in pre-dispersed form, with outstanding dispersion characteristics. AERODISP® dispersions are ready to use and easy to handle. In addition to the product range shown here, Evonik offers a large number of customized dispersions based on various fumed silicas and metal oxides. Either water or ethylene glycol is used as a dispersion medium. Dispersions in non-aqueous organic solvents are available on request.

Positive effects

- High ink adsorption in photo inkjet papers
- Improves surface properties by cleaning, polishing, and coating metals, papers, textiles, etc.
- Optimizes the rheological properties of paints, coatings, and battery gels
- Improves visual properties in wood stains
- Optimizes adhesive performance in self-adhesive stickers, etc.
- Improves anti-blocking properties in PET films

An overview of the physical/chemical key figures for individual AERODISP® grades is given in table 17.

Table 17 AERODISP® dispersions

AERODISP® grades	Solid content1) [wt%]	pH2)	Viscosity3) [mPas]	Density [g/cm^3]	Stabilizing agent/comments
SiO_2 dispersions, alkaline					
AERODISP® W 7520	20	9.5–10.5	≤ 100	1.12	Ammonia
AERODISP® W 7520 N	20	9.5–10.5	≤ 100	1.12	Sodium hydroxide solution
AERODISP® W 7520 P	20	9.0–10.0	≤ 300	1.12	Potassium hydroxide solution
AERODISP® W 7622	22	9.5–10.5	≤ 1,000	1.13	Ammonia
AERODISP® W 1226	26	9.0–10.0	≤ 100	1.16	
VP Disp. W 1250	50	9.5–10.5	≤ 2,000	1.38	Ammonia
AERODISP® W 1244	48	10.6–11.2	≤ 200	1.44	Potassium hydroxide solution
AERODISP® W 7330 N	30	9.5–10.5	≤ 1,000	1.20	Sodium hydroxide solution
AERODISP® WR 8520	20	10.0–11.0	≤ 100	1.13	2-Dimethylaminoethanol (DMEA)
AERODISP® W 7225 P	25	9.8–10.8	≤ 300	1.16	Potassium hydroxide solution
SiO_2 dispersions, acidic					
AERODISP® W 1714	14	5.0–6.0	≤ 100	1.08	Phosphate / 4)
AERODISP® W 1824	24	5.0–6.0	≤ 150	1.15	Phosphate / 4)
AERODISP® W 1836	34	4.0–6.0	≤ 200	1.23	Phosphate / 4)
AERODISP® W 7215 S	15	5.0–6.0	≤ 100	1.09	Ammonia
AERODISP® W 7512 S	12	5.0–6.0	≤ 100	1.07	Ammonia
SiO_2 dispersions, cationic					
AERODISP® WK 341	41	2.5–4.0	≤ 1,000	1.28	Cationic polymer / 4)
AERODISP® WK 7330	30	2.5–4.0	≤ 1,000	1.20	Cationic polymer / 4)
Al_2O_3 dispersions					
AERODISP® W 630	30	3.0–5.0	≤ 2,000	1.26	
AERODISP® W 440	40	3.0–5.0	≤ 1,000	1.38	
AERODISP® W 925	25	3.0–5.0	≤ 1,000	1.20	
TiO_2 dispersions					
AERODISP® W 740 X	40	5.0–7.0	≤ 1,000	1.41	
VP Disp. W 2730 X	30	6.0–8.0	≤ 5,000	1.28	
Solvent-based dispersions					
AERODISP® G 1220	20	–	≤ 300	1.23	Ethylene glycol
VP Disp. G 6020 X	20	–	≤ 200	1.24	Ethylene glycol
AERODISP® 1030	30	–	≤ 10 [Pa s] 3)	1.16	1-Methoxy-2-propyl acetate (MPA)

Additional dispersions of hydrophilic and hydrophobic silicas and metal oxides in organic solvents are available on request. The values quoted are non-binding guideline values.

Development products include "VP Disp." in their name. Their commercialization is dependent on their performance on the market. As a result, future availability cannot be guaranteed, although the product is produced on a commercial scale currently. In some cases, VP Disp. products may not have undergone all tests.

More detailed information on AERODISP® grades can be found in our Industry Brochure "AERODISP® Fumed Silica and Metal Oxide Dispersions."

1) Solid content may vary by ±1 percent point for technical reasons

2) Based on ISO 787-9

3) Measured in accordance with ISO 3219 at a shear rate of 100 s^{-1}

4) Contains stabilizers

4.3 For the food industry

Demand is increasing for food products that are easy to work with and of high quality. Such products are often made from powdered ingredients such as salt, sugar, spices, milk powder, and powdered eggs. Because of their fineness and/or their hygroscopic nature, these food ingredients tend to have poor flow characteristics, which makes them difficult to work with and hard to measure out precisely. Adding AEROSIL® 200 F or AEROSIL® 380 F represents an inexpensive way to make these products easy to handle and to ensure consistent product quality; when food manufacturing processes run smoothly, output increases.

AEROSIL® 200 F and AEROSIL® 380 F significantly improve the free-flowing characteristics of powdered products, enhancing flow by encapsulating the powder particles of the product and keeping them separate. Another benefit of using AEROSIL® products is their good absorption capacity. This enables them to absorb traces of water or oil, reducing the powder's tendency to become caked. Using AEROSIL® 200 F and AEROSIL® 380 F allows the product to retain its color intensity.

In fluid foodstuffs such as marinades made with oil, AEROSIL® 200 F and AEROSIL® 380 F regulate viscosity and thus prevent herbs and spices from settling. AEROSIL® 200 F and AEROSIL® 380 F are hydrophilic fumed silicas with a respective average specific surface areas of 200 and 380 m^2/g. The special grades are produced in compliance with the requirements of the HACCP* guidelines. The production and packaging process has been audited – it meets the European specifications for food and animal feed additives and has FAMI-QS certification.

Positive effects

- Configuration of the rheology of liquids
- Use as anti-settling agent, thickening agent
- Improves the free-flow properties of powders
- Absorption agent to stop powders from soaking up moisture and caking
- High chemical purity, low residual moisture
- Neutral taste
- No alteration of the natural coloring of powder formulations

* HACCP = Hazard Analysis and Critical Control Points – quality management for the food and animal feed industries

4.4 For the pharmaceutical industry

Reliable and cost-optimized production processes are essential for the pharmaceutical industry. As an effective glidant, AEROSIL® has become one of the most important and most frequently used pharmaceutical excipients. Producing tablets and capsules with high-speed machinery calls for optimum flow properties of the powdered starting materials. When it is used as a powder flow additive, AEROSIL® helps to ensure that equal amounts of active pharmaceutical ingredients (API) are present in each tablet or capsule, enabling high throughput in production. AEROSIL® can also optimize the production and the properties of numerous other dosage forms, such as ointments, suppositories, and transdermal therapeutic systems.

The highly regulated pharmaceutical industry has very high expectations when it comes to the purity and reliability of the raw materials used. AEROSIL® is a shining example of a product that can meet such demands, thanks to its high purity and extremely low heavy-metal content. There are other requirements that apply for pharmaceutical products, however. For example, they need to be analyzed on the basis of methods set down in the monographs of pharmacopeia and they must be produced in accordance with validated processes. In order to meet these requirements, Evonik provides special products for the pharmaceutical industry, all with names ending with the word "Pharma." All these products are produced in compliance with the Good Manufacturing Practice (GMP) standards issued by the International Pharmaceutical Excipients Council (IPEC). For more information, consult our Industry Brochure "AEROSIL® Colloidal Silicon Dioxide for Pharmaceuticals."

You need a highly versatile product, customized to suit your specific application?

Application-related effects

- Reinforcement and scratch resistance
- AEROSIL® – a simple thickening model
- Free flow and anticaking
- Transparency
- Heat stabilization

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5. Application-related effects

In addition to producing AEROSIL®, our teams of specialists in research and development ensure that our products are continuously improved. They work in close contact with the application engineering teams, to enable solutions to be found for specific customer needs. Numerous special applications are listed in table 2 on page 10.

5.1 Reinforcement and scratch resistance

When AEROSIL® is used as a reinforcement filler material in elastomers, it can improve the mechanical properties of the silicone rubbers with long-lasting effect, such as their tensile strength, elongation, and tear resistance.

Silicone rubbers with AEROSIL® as an active component play an important role in earthquake-proof foundations. The technology, developed in Japan, that enables silicone rubber to be used as a main component in foundations gives buildings in high-risk regions additional protection from earthquake damage, even when they are exposed to intense mechanical stress. In a special technique, layers of silicone rubber that have been configured precisely are combined with conventional building materials.

In this application, RTV-2C (two-component) silicone rubbers are used not only as sealants but also to make the foundation particularly elastic even at low or high temperatures.

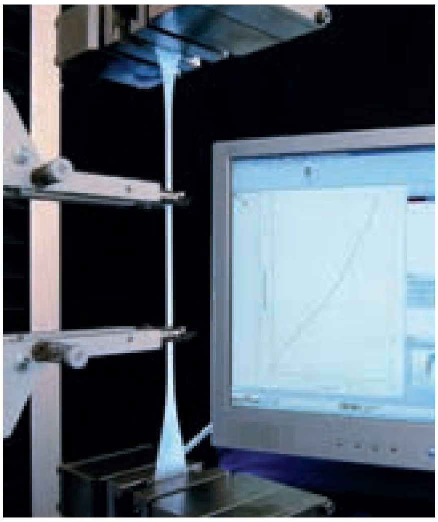


Figure 76

AEROSIL® is used as a strengthening component in silicone rubber products for earthquake-proof foundations

Tensile testing machine for measuring mechanical properties of silicone rubbers, such as tensile strength, elongation, and tear resistance

Additional typical application fields

- Liquid silicone rubber (LSR)
- High temperature vulcanizing rubber (HTV)
- Room temperature vulcanizing silicone rubber (RTV)
- Fluoroelastomers
- Natural rubber (NR)
- Styrene-butadiene rubber (SBR)

In this Fine Particles series, text no. 63, "AEROSIL® Fumed Silica and SIPERNAT® in sealants," describes these applications in detail.

The strengthening is fundamentally brought about by silanol groups being interacted with or Si-methyl bonds being radically cleaved and the fragments being incorporated into the polymer. In coating systems, AEROSIL® can significantly strengthen the coating films even at a low concentration, which is sufficient for rheology control.

AEROSIL® increases the glass transition temperature of the coating layers. This strengthening effect is also reflected in an increase of the shear modulus and the modulus of elasticity. This effect is assumed to be caused by the binding agent molecules being held in a strong adsorptive bond with the AEROSIL® surface, which then significantly restricts the mobility of the polymer chain segments. For modern coating systems, the scratch resistance often plays a central role, in addition to the usual mechanical properties.

To address this need, a new generation of fumed silicas have been developed that enable the scratch resistance of UV-cured, high-solid, and conventional solvent-based 2C systems to be considerably improved. Thanks to a combination of chemical and mechanical post-treatment, these structure-modified AEROSIL® products have only a minimal effect on the rheology of the coating systems. Quantities as high as 10 wt% in relation to the solids in the system can be incorporated without any significant increase in viscosity. This elevated content percent of AEROSIL® R 9200 causes a distinct increase in the mechanical stability with regard to scratches.

Alongside the well-known products AEROSIL® R 7200 and AEROSIL® R 8200, AEROSIL® R 9200 for UV-cured, high-solid, and conventional solvent-based 2C systems has also been launched on the market successfully.

AERODISP® 1030 is a 30 % dispersion of AEROSIL® R 9200 in 1-methoxy-2-propyl acetate (MPA). This user-friendly dispersion offers users a range of benefits. In addition to considerably improving the scratch resistance of UV-cured, high-solid, and conventional solvent-based 2C systems, it provides optimum visual properties for the coatings.

5.2 AEROSIL® – a simple thickening model

For the majority of solvents or dispersing agents, the molecules require relatively little force to glide past each other while flowing, as they are relatively small. Accordingly, their internal friction is low – they have a similar consistency to water. These systems therefore tend to have low viscosity.

In dispersions containing AEROSIL® that have open fractal aggregate structures with high relative density, associates and network structures are created in accordance with the surface chemistry. The flow curves of such systems are highly complex. As a result of the network formation, components such as heavy pigments can be held in suspension, and a "fluid" dispersion layer may be prevented from running when applied to vertical surfaces. Effects such as these cannot normally be configured effectively on the basis of thickeners alone.

For a simple thickening model of AEROSIL®, it is necessary to consider two, and only two, properties (and these particular properties need to be compatible).

The first property is a preferential aggregate size of approximately one hundred to several hundred nanometers. The second is a surface chemistry that allows the aggregates to build network structures via hydrogen bonds or van der Waals forces – either with one another or with other filler materials such as pigments, additives, resins, solvents, etc. (see figure 77).

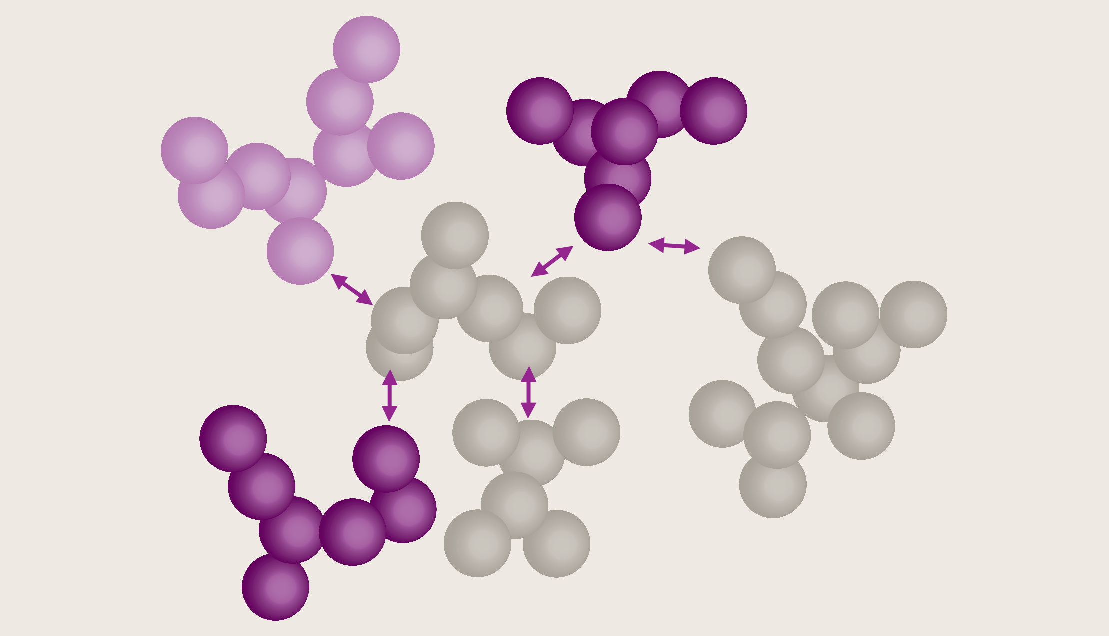


Figure 77: Customized AEROSIL® aggregate structures and "matching" surface chemistry (symbolized by the arrows) are important criteria for rheology control with AEROSIL®.

Ideally, the steric and electronic conditions of the AEROSIL® products will be suited to the particular customer formulation, in accordance with the lock and key model. For example, AEROSIL® R 805 can often be used to provide effective rheological control of polar coating systems that have polar and nonpolar regions, as this functionalized silica also has polar and nonpolar regions with a suitable structure. Polar interactions can then be set up effectively via hydrogen bonds with the free silanol groups, and nonpolar interactions via van der Waals interactions, perhaps with the involvement of the methylene groups from the silane residues (in hydrophobic AEROSIL® grades).

The importance of the network structure, and thus the size of the AEROSIL® aggregates, can be explained quite simply: When colloidal silicas have particles only a few nanometers in size, barely any thickening effect is achieved even when they are added at a rate of 50 wt%. AEROSIL®, on the other hand, can have an aggregate size of >100 nm and still create highly transparent systems. This is because the aggregates are not complete SiO_2 spheres. They actually mainly consist of the dispersion agent, embedded in the fractal AEROSIL® framework. Consequently, the aggregates scatter light with a refractive index that has been correspondingly averaged out. This refractive index will come very close to the index of the pure dispersion agent, resulting in a very low scattering effect for AEROSIL® dispersions.

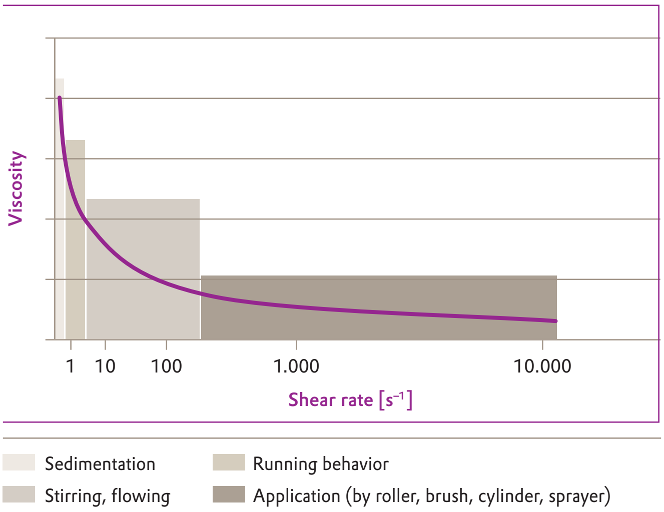
5.2.1 Rheology control

When it comes to rheology control, AEROSIL® is a versatile problem-solver in a large number of applications. Examples of this broad range of application fields include paints and coatings, unsaturated polyester and epoxy resins, pastes, resists, blood fractionation, no-maintenance batteries, drilling fluids, diamond suspensions, and oil and water emulsions.

The rheology of coatings is discussed below, as rheology plays a crucial role in a coating's effectiveness.

A coating should have high viscosity when it is under low mechanical stress, for example during storage or transport, to prevent the pigments or filler materials from settling. When it is under high mechanical stress, such as when it is being sprayed, the viscosity should be low, however, to allow the coating to be applied smoothly. As soon as the coating adheres to the substrate, it should return to its original viscosity, to prevent running (see figure 78).

Figure 78



Ideal viscosity profile of a coating with AEROSIL®

5.2.2 Thixotropy

In general, AEROSIL® generates a shear thinning that is connected with relatively pronounced thixotropy. Shear thinning means that the viscosity of a liquid changes in relation to the shear rate or shear stress – it is dependent on the force acting on the coating film parallel to the substrate. At higher levels of shear stress, lower viscosity is observed. This viscosity increases when the shear stress is reduced. The effect of a liquid increasing in viscosity as the duration of shear exposure increases, while the shear intensity remains constant, is referred to as thixotropy [35, 36, 37]. Both shear thinning and thixotropy influence the application qualities of a coating.

A low viscosity at high shear stress levels ensures good application, particularly in the case of spray application. The high viscosity of the coating at low shear stress levels or in a rest state ensures that the paint does not run on vertical surfaces and allows the maximum possible wet film thickness to be significantly increased by using AEROSIL®. Nevertheless, sometimes a compromise must be reached between the running behavior of the coating and the self-leveling of the coating surface, although self-leveling is mainly dependent on the surface tension, which is generally influenced little by AEROSIL®.

If AEROSIL® is used, intensive dispersion is generally required. It is ordinarily advisable to make a parent paste or a grinding mixture from some of the binding agent, the solvent, and (where possible) the pigments. A good dispersion can be ensured by using a dissolver, a rotor-stator device such as an Ultra Turrax® or a Kady mill, a Skandex mixer, a bead mill, or a ball mill, as appropriate for the starting viscosity. In most cases, jet dispersants have also provided excellent results.

5.2.3 AEROSIL® as an antisettling agent

Hydrophilic and hydrophobic AEROSIL® are used both as a dispersion aid and to stabilize pigments and filler materials during the production and storage of dispersions and preparations such coatings and paints. There are clearly two effects at work here. The AEROSIL® particles form a shell of sorts around the pigment particles, preventing re-agglomeration. At the same time, the settling of the pigments and filler materials is prevented or reduced by the greater viscosity created by AEROSIL® and the consequent flow limit of the coating.

Figure 79

Increasing the antirunning properties in spray applications (55 µm dry film thickness)

No AEROSIL®

0.3 % AEROSIL® R 812 S

White 2C PUR enamel based on oil-free saturated polyester/isocyanate (high solids)

AEROSIL® R 972 holds siliceous filler materials and matting agents made from silicon dioxide in suspension very effectively. In this application, additive quantities of 0.3 % – 0.5% in relation to the overall formulation are generally used. Specific heavy pigments can also be held in suspension. An example of this is the use of AEROSIL® R 972 or AEROSIL® R 805 in corrosion-protection paints such as zinc dust paints or micaceous iron ore paints. Hydrophobic AEROSIL® is also used in electrically conductive coatings that contain metal powder or metal flakes. When the conductive components are metal flakes, AEROSIL® R 805 is the better option, as it has no negative influence on the lamellar alignment despite providing a pronounced suspension effect. Even when AEROSIL® is unable to prevent settling entirely, the sediment that forms is not solid and can be stirred up easily.

Figure 80: Separation after eight weeks' storage at room temperature
Left: No AEROSIL®, right: 0.8 % AEROSIL® R 972 Water-based filler based on PUR dispersion / melamine resin

5.3 Free flow and anticaking

The process steps of dosage, drying, conveying, filling, and other powder-handling procedure are part of day-to-day operations in the chemical, pharmaceutical, animal feed, and food industries.

Problems caused by caking or bad flow properties in powders, such as blocked filters, nozzles, or mills and imprecise dosage, complicate these processes and can result in unwelcome downtime.

This leads to increased costs resulting from production failure and fluctuations in product quality, which are noticed by customers directly and can cause increased complaints or even customer loss.

Both precipitated silicas (SIPERNAT®) and fumed silicas such as AEROSIL® products allow the flowing properties of powders to be configured perfectly, ensuring an effective industrial processing of powdered products. At the same time, improved anticaking properties have a positive effect on the storage and transport of the end products, even in varying climatic conditions.

Various factors can cause powders to flow poorly or cake, including the following:

- The van der Waals forces between the particles (see figure 81)
- Liquid bridges between the particles
- Plasticity of the particles
- Particle shape
- Particle size
- Electric charges

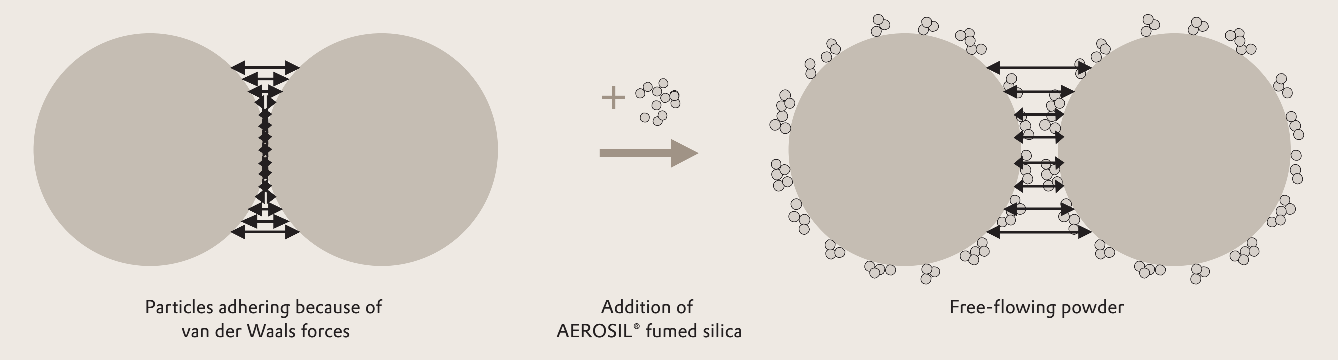


Figure 81: AEROSIL® covers the surfaces of the powder and functions as a separator

For products such as spices, properties like good free-flow behavior with no large clumps forming and preservation of color intensity are essential for customers and consumers. To create high-quality powders that have these properties, AEROSIL® products can be mixed into the spice powders during the milling stage.

Further information on the effects of silicas on the above-mentioned factors can be found in our Technical Information [38] titled “SIPERNAT® specialty silica and AEROSIL® fumed silica as flow aid and anticaking agent” and in the additional brochures for special applications such as spices and seasonings [39], sugars and sugar alcohols [40], instant beverages [41], and other relevant industrial applications.

Figure 82: Free-flow properties of chili powder Left with no flow enhancer, right with 2 % AEROSIL® 200 F

5.4 Transparency

Thanks to the use of AEROSIL®, silicone rubber is colorless and highly transparent. These properties have made the material popular in a diverse range of applications, thanks to its visual appeal. Silicone rubber can be produced in practically any color, but highly transparent formulations are also sold. They are preferred in medicinal applications, such as tubes and catheters, and also in everyday products such as pacifiers and diving goggles.

The transparency is influenced by two main factors: the particle size of the silica and the refractive index of the polymer and the filler material.

For highly transparent silicone systems, AEROSIL® grades with relatively high surface areas are particularly suitable. However, modifying the silicas with silanes or silicone oil can also give the silicas a refractive index similar to that of the silicone rubber and provide a lasting improvement in the transparency of the silicone system.

Figure 83: High-transparency diving goggles and snorkel mouthpieces made from HTV silicone rubber

5.5 Heat stabilization

Silicone rubbers are elastomers, with physical and mechanical properties that are largely unaffected by temperature. The reason for this high resilience to temperature is the stable O-Si-O framework. The polymers do not undergo thermal decomposition until temperatures exceed 200 °C. The thermal degradation of silicone involves a loss in weight. The methyl groups are cleaved away via oxidation, until only SiO_2 remains. For improving the thermal stability of silicone rubber, the use of 0.5 % – 5% AEROXIDE® TiO_2 P 25 or 0.5% – 3% AEROXIDE® TiO_2 PF 2 has proved to be effective. Both products are manufactured by flame hydrolysis of titanium tetrachloride. The addition of FeCl gives the more effective product, AEROXIDE® TiO_2 PF 2, mixed with 2% Fe_2O_3. The assumed mechanism of action is that these metal oxides promote the recombination of formed radicals.

You demand quality, and not just on paper?

Quality and product reliability

- Quality control
- Environmental conduct
- Toxicology and hygiene at work
- Disposal
- Legal considerations in Germany
- Registration status of AEROSIL® products

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6. Quality and product reliability

6.1 Quality control

All AEROSIL® products worldwide are produced in compliance with strict quality standards, and Evonik goes to great lengths to ensure that this compliance is maintained. For example, all production sites are certified to the ISO 9001 quality management system, as are all research and application sites. Regular external and internal audits and constant systematic improvement of the quality of our products, services, and processes supports adherence to the high quality standards.

All invoice recipients are given, a quality document for the related product and a copy of the agreed product specifications. Before products are released for dispatch to the customers, tests are performed in the quality control lab of the production plant to ensure that the products comply with specification limits.

All analysis data are saved in a laboratory information management system (LIMS).

The data are also evaluated regularly as part of statistical quality control (SQC). This involves determining mean average μ and standard deviation, in addition to the values for C_p and C_pK. The process capability indices C_p und C_pK are key figures that are used to statistically evaluate a process in production technology. They indicate the process's ability to achieve the specified targets.

While the C_p value only indicates the relationship between the specified tolerance and the process distribution, the C_pK value also incorporates the positioning of the mean value in relation to the specified tolerance midpoint. The C_pK value is thus always smaller than or equal to the C_p value [42].

Figure 84 shows four distributions of measured values relative to the specification limits. The related C_p and C_pK values are also indicated.

C_p and C_pK values of at least 1 are generally aimed for all products. This means that at least 99.73 % of the produced quantity meets specifications in terms of the quality features under consideration.

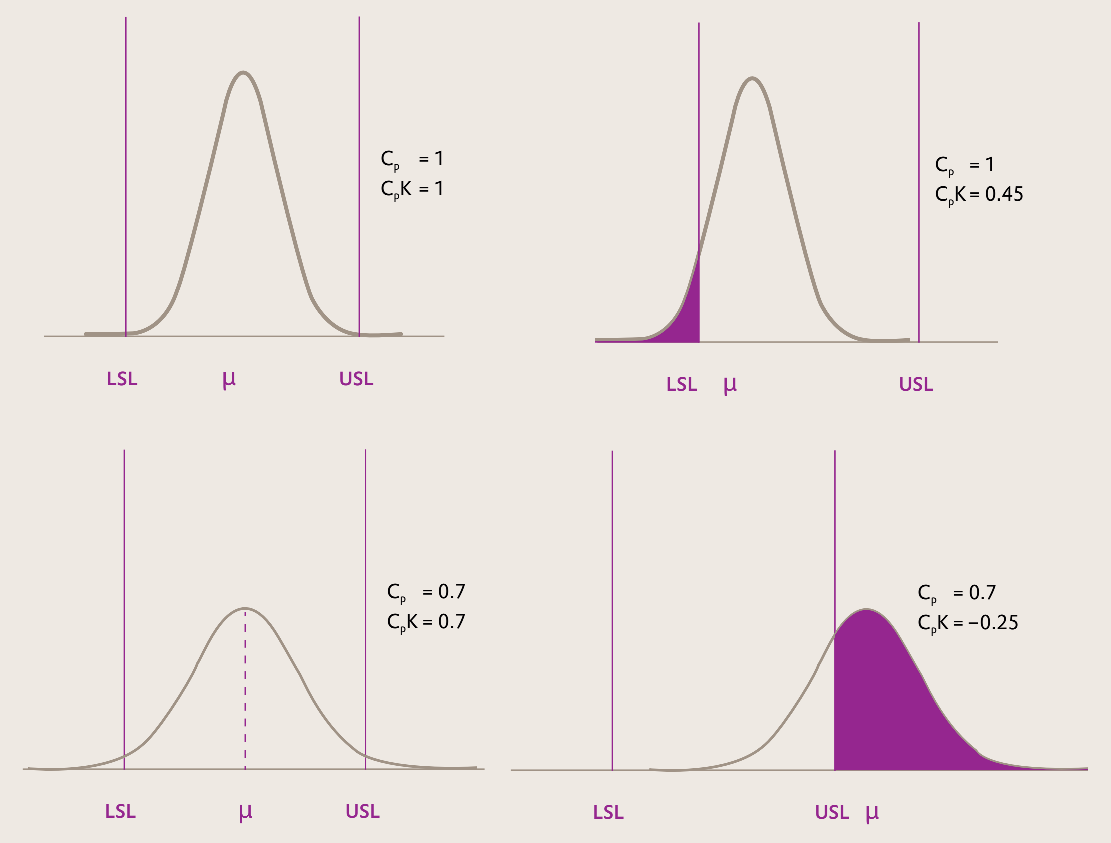

Figure 84: Examples of process capability indices
LSL = lower specification limit
USL = upper specification limit

6.2 Environmental conduct

Silicon dioxide is one of the most abundant substances in nature. Thanks to its hardness, its chemical stability, and its UV resistance, it is particularly resistant to weathering. As part of silicates or clay minerals, it forms the main component in the earth's crust, making it the most common silicon compound. Noncrystalline amorphous silicon dioxide is a major component in the skeletons of radiolarians, diatoms, and sponges. Synthetic amorphous silica is an inert, solid inorganic substance. In the environment, it accumulates in sediments or soils and can enter the natural silicon dioxide cycle as silicic acid as a result of weathering.

Silicon dioxide dissolves in water slowly and poorly. Its bioavailability for aquatic organisms is therefore low. Amorphous silicon dioxide showed no harmful effect in acute fish and daphnia tests performed to the OECD standard, even at very high concentrations (1,000 or 10,000 mg/l). Because of silicon dioxide's physical/chemical properties and the low acute ecotoxicity, chronic effects and accumulation in organisms are not to be expected. Silicon dioxide is an inorganic substance that is not broken down by microorganisms. The international standardized regulations for determining biodegradability (such as OECD and EEC guidelines) can only be applied in relation to organic substances.

The German Commission for the Evaluation of Substances Hazardous to Waters (KBwS) has classified synthetic amorphous silica as not hazardous to waters (KBwS no. 849). Silicon dioxide is included in the Pose Little or No Risk to the Environment List (PLONOR). The substances listed are recommended for use in offshore waters by the OSPAR commission.

6.3 Toxicology and hygiene at work

The main feature of AEROSIL® synthetic amorphous silicas, which are produced via flame hydrolysis, is their lack of crystalline components. The method used to detect crystalline structures is electron diffraction combined with transmission electron microscopy and X-ray diffraction. The detection limit for this method is less than 0.05 weight percent. The synthetic amorphous silicas produced by Evonik have been found to be fully amorphous.

Synthetic amorphous silicas are used in products and processes. They bring about no acute toxic symptoms, whether administrated orally or via the skin. Studies evaluating the toxicity of skin or eye contact have shown that synthetic amorphous silicas do not irritate skin or eyes. Chronic skin contact is known to cause dry skin or generative eczema, however. These reactions can be avoided by intensive skin protection/care. The data collected from occupational health examinations throughout the decades of production and use have revealed no indications of sensitization potential. No cases of contact allergy have been reported. Synthetic amorphous silicas have no mutagenic effect. No teratogenic effects have been observed. After repeated oral ingestion, no treatment-related effects could be detected.

In inhalation studies, none of the synthetic amorphous silicas resulting in permanent damage to lungs or progressive harm comparable to silicosis. Multiple epidemiological studies of workers with chronic exposure found no indications of a silicosis. The available data provide no indication of lung cancer or other sustained respiratory diseases. On the basis of all the available data, synthetic amorphous silicas are clearly not harmful to health within the applicable limits. Many countries have workplace exposure limits for synthetic amorphous silica. The precise values can be found in the latest safety data sheets. It may be necessary for suitable dust extraction to be performed or for dust masks to be worn.

6.4 Disposal

All AEROSIL® grades are largely ecologically harmless. They have no effect on the groundwater – in fact, dissolved silica is often present in drinking water. When AEROSIL® or waste containing AEROSIL® is being disposed of, the local waste disposal laws and regulations must be observed. During disposal dust generation should be avoided.

6.5 Legal considerations

AEROSIL® products can be selected as additives in food and animal feed, food contact materials, toys, cosmetics, and pharmaceuticals.

6.6 Registration status of AEROSIL® products

Detailed information on the registration status of AEROSIL® products is available on request (please email us at sds-hu@evonik.com). It can also be found in the product information (www.AEROSIL.com).

You are looking for international, reliable customer service that exceeds expectations?

Handling and availability

- Handling AEROSIL®
- Packaging for AEROSIL®
- Storage durability of AEROSIL®
- Safety technology
- Reliable supply

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7. Handing and availability

7.1 Handling AEROSIL®

In a time in which there is increasing emphasis on automation, rationalization, and hygiene at work, the handling of AEROSIL® is of vital importance. Evonik has continuously expanded its operations relating to handling over the past decades to protect its workers and to fulfill environmental standards.

Handling refers to the techniques required to transfer the products into the customer's work operations after delivery. The following topics can be considered focal points:

- Emptying packaging
- Siloing
- Internal transport
- Dosage
- Placing the material in the customer's processing machines

Handling also covers the measures required to perform the above steps:

- Filter technology
- Fill level measurement technology
- Weighing technology
- Discharge technology

Evonik endeavors to offer customers prompt assistance whenever they experience problems with handling or are replanning their operations. The customer service features described here are provided free of charge by Evonik to existing customers worldwide. The type and the scope of consultation depends on the customer's particular issue.

To identify particular issues affecting the handling of AEROSIL® and to optimize them, Evonik has set up a technical center for handling in the Wolfgang industrial estate in Hanau (see figure 85). This technical center can be used to demonstration purposes for customers. The majority of the handling steps can be showcased here. The handling department may also assist with trials at the customer's own premises in special cases.

Planning dust-free and automated systems requires knowledge of our products and experience in process engineering. As this combination of skills cannot always be provided by the vendors delivering the machinery and equipment, consultation and assistance can be provided to our customers personally by handling technicians and engineers – either at the Evonik site in Hanau or at the customer's premises. Where necessary, trials (such as trials for pneumatic dense phase conveying) may also be arranged at external technical centers (on the equipment manufacturer's premises, for example).

In addition, basic information on handling the products has been published, for example in the brochure "Handling of synthetic silicas and silicates" a range of Technical Information texts to address specific topic areas.

Figure 85: The Silica Business Line's technical center for handling in Hanau, AEROSIL® dispatched in sacks, in FIBCs, and as a bulk good.

7.2 Packaging for AEROSIL®

Standard packaging

Standard AEROSIL® packaging is a multi-layer paper sack, in some cases fitted with a PE-coated layer. The filling weights of the sacks, which vary for the different types of AEROSIL® and the corresponding tamped density, range from 10 to 20 kg (for NAFTA also 10 lbs).

Flexible intermediate bulk container (FIBC)

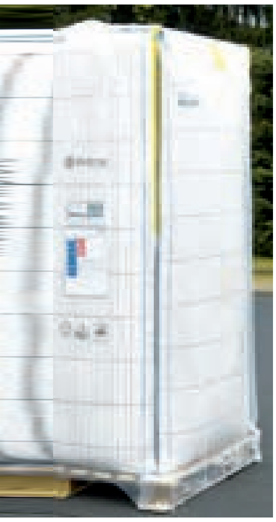
As a semi-bulk packaging, Evonik provides AEROSIL® in FIBCs (flexible intermediate bulk containers). FIBCs are flexible containers made from - woven polypropylene with lifting loops attached at the top corners. The discharge spout, which has been customized to suit the Powder Emptying System (PESy) developed and patented by Evonik, is located at the center of the base. This emptying device is provided for our customers free of charge to allow the FIBC to be emptied easily with no dust being formed. The fill quantities vary depending on the product and compaction type. More information can be found in Technical Information 1219 [43], "Semi-bulk packaging for AEROSIL®."

Silo transport

For large consumption quantities, AEROSIL® can be dispatched in silo vehicles. In such a case, the customer must have a storage silo of sufficient size. The supply quantities for silo dispatch depend on the degree of compaction and the vehicle volume.

Packaging for AERODISP® dispersions

The AERODISP® dispersions are delivered in 60 kg canisters, 220 kg barrels, and in 1,000 kg intermediate bulk containers (IBCs). In the colder months, the containers are fully covered with a metalized bubble wrap to protect them from low temperatures during transport. The insulation packaging can vary depending on the container and transport type.



7.3 Storage durability of AEROSIL®

AEROSIL® is largely chemically inactive and under suitable storage conditions it will not change chemically even after decades. It must be ensured that AEROSIL® does not come into contact with hydrofluoric acid or strong alkalis, as these substances in principle will react with silicon dioxide.

Because of the highly dispersed nature of AEROSIL®, there is a risk of it adsorbing vapors and gases. Interactions with the silanol groups can occur, particularly in compounds with amine or hydroxyl groups. In the case of water, adsorption is reversible. Accordingly, the loss on drying for hydrophilic AEROSIL® increases as humidity rises and decreases again as humidity drops (see also section 3.4.3.2.1, Moisture balance at room temperature). In hydrophobic AEROSIL®, the adsorption capacity is significantly reduced and the uptake of moisture is much lower. These adsorption processes can influence the application engineering properties of the product. AEROSIL® should therefore always be kept away from potential sources of contamination.

The storage temperature should not exceed 50 °C (or 35 °C, in the case of AEROSIL® R 7200, AEROSIL® R 805, AEROSIL® R 711).

AEROSIL® has a relative density of only 3 % with a tamped density of 50 g/l. There is some risk that AEROSIL® may become compacted in the case of prolonged storage. This can result in an increase in the tamped density and a change in the structure.

Although AEROSIL® can in principle be considered suitable for long-term storage given suitable conditions (there is no risk of aging or decomposition), we recommend that the above-mentioned products be used within two years of the production date. The production date is given in the reference number that is printed on each sack.

More detailed information on storing AEROSIL® can be found in Technical Information 1373 [44], “How to store AEROSIL® fumed silica.”

AERODISP® dispersions should be stored with protection from heat and frost and used within 6 to 12 months after the production date. The production date is given in the reference number that is printed on each package.

7.4 Safety technology

AEROSIL® has the form of a powder. If dust formation cannot be fully prevented during handling, dust extraction is recommended.

If high concentrations of dust are present, a dust mask with particle filter should be worn (see safety data sheet).

When silica is being handled, electrostatic charging can occasionally occur, for example when the silica sack is being emptied. To avert any risk during handling, measures should therefore be taken to prevent electrostatic charging, such as grounding and possibly inerting of the supply tank. More information can be found in the brochure “Synthetic Silica and Electrostatic Charges.”

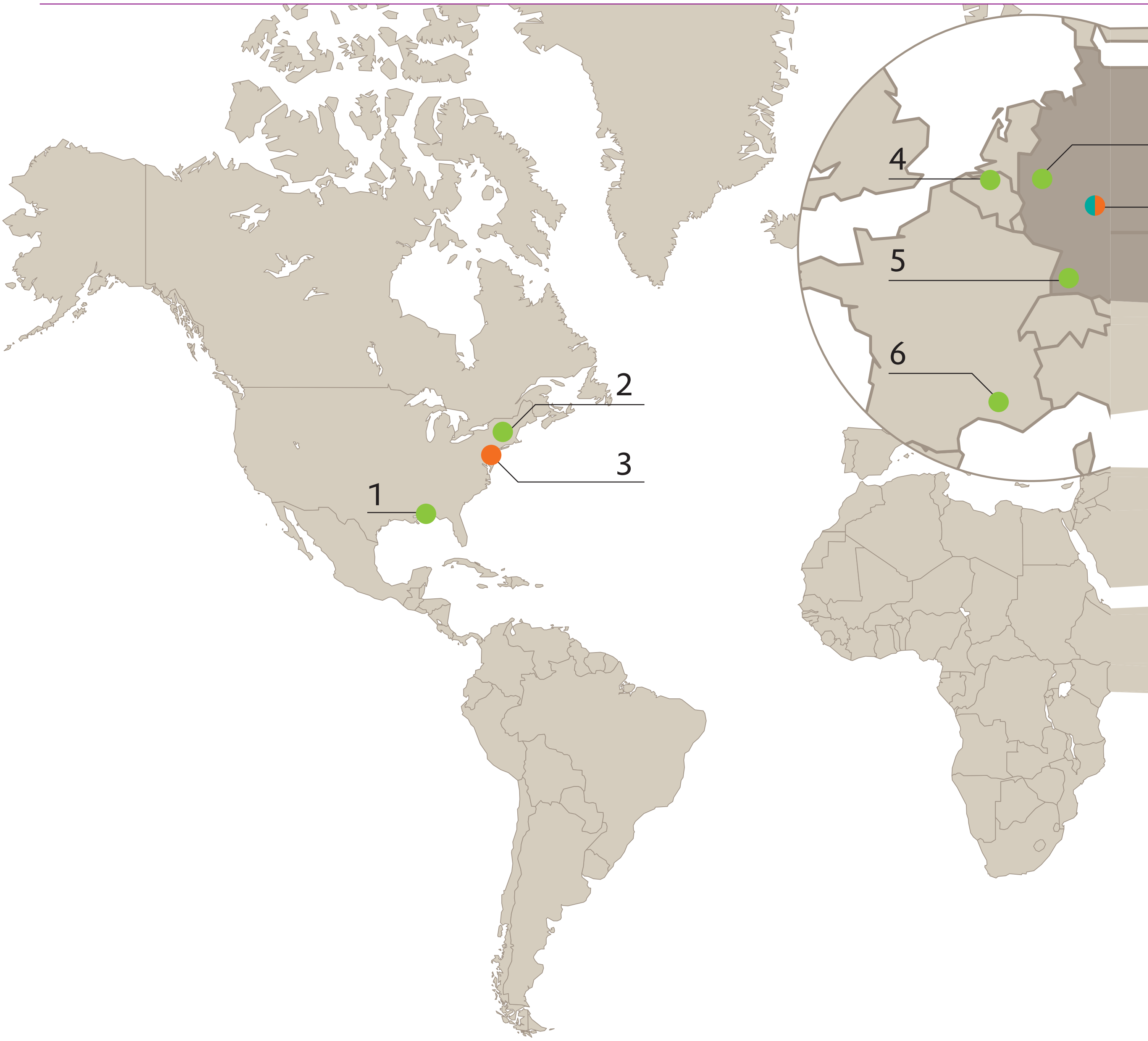
Hydrophilic, fumed synthetic amorphous silicas (SiO_2) are not flammable. A dust explosion of pure SiO_2 is therefore not to be expected.

Hydrophobic synthetic amorphous silicas have been surface-treated and are highly reactive because of the quantity of organic substances on their surface. As a result, some AEROSIL® grades may exhibit self-heating behavior. This volume-dependent reaction should be noted in the case of AEROSIL® R 7200 and AEROSIL® R 805, for example. It should be borne in mind when production plants are being set up and when storage facilities are being designed. More detailed information can be found in the safety data sheet and Technical Information TI 1373, “How to store AEROSIL® fumed silica.” Additionally, for some surface-treated AEROSIL® products, such as AEROSIL® R 805, there is a risk of dust explosion. Appropriate protective measures must be taken when these products are used. For more information, refer to the safety data sheet and Technical Information TI 1363, “Dust-Explosion Protection in Handling Systems for Surface-Modified AEROSIL®.” These resources also provide information on the protective measures derived from the key safety data.

If synthetic, amorphous fumed silica makes contact with the skin and leaves a dry sensation, the silica should be washed off with water and skin cream should be applied.

Spilled material should be collected up without any dust being formed and placed in a suitable container.

7.5 Reliable supply



● Production

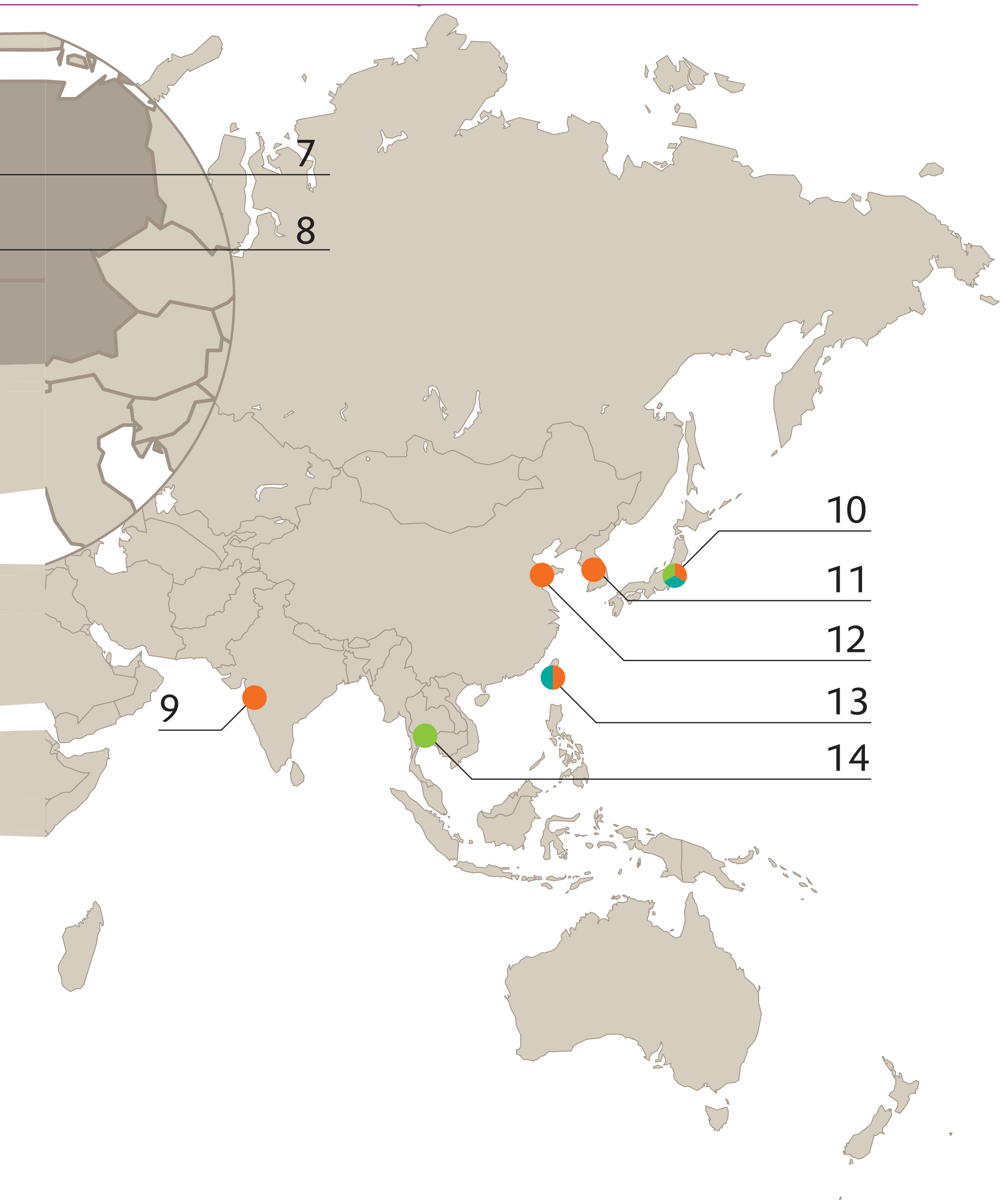
1	Mobile, USA
2	Waterford, USA
4	Antwerp, Belgium
5	Rheinfelden, Germany
6	Roussillon, France
7	Leverkusen, Germany
10	Yokkaichi, Japan
14	Maptaput, Thailand

● Research & Development

8	Hanau, Germany
10	Yokkaichi, Japan
13	Ta Yuan, Taiwan

● Applied Technology

3	Piscataway, USA
8	Hanau, Germany
9	Mumbai, India
10	Yokkaichi, Japan
11	Seoul, Korea
12	Shanghai, China
13	Ta Yuan, Taiwan



With numerous production sites for AEROSIL® and AERODISP® in Europe, the US, and Asia and over 100 sales offices worldwide, Evonik has a unique service and production network. This guarantees highly reliable supply and direct transport routes, ensuring that the right product will arrive no matter where in the world it is sent to – rapidly, in outstanding quality.

You would like more details on properties, references, and technical terms?

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- Physical/chemical data of AEROSIL®
- Bibliography
- Glossary

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8. Annex

8.1 Physical/chemical key figures for AEROSIL®

Hydrophilic AEROSIL®

Test methods	Unit	AEROSIL® 90	AEROSIL® 130	AEROSIL® 150	AEROSIL® 200	AEROSIL® 255	AEROSIL® 300	AEROSIL® 380	AEROSIL® OX 50
Behavior in relation to water	Hydrophilic								
BET surface area [1]	m^2/g	75–105	105–155	135–165	175–225	230–280	270–330	350–410	35–65
Tamped density [2] Approximate value Standard product	g/l	80	50	50	50	50	50	50	130
Loss on drying [3] (2 hours at 105 °C) on leaving the supplier factory	%	< 1.0	< 1.5	< 1.5	< 1.5	< 1.5	< 1.5	< 2.0	< 1.5
pH value [4]		3.7–4.7	3.7–4.5	3.7–4.5	3.7–4.5	3.7–4.5	3.7–4.5	3.7–4.5	3.8 – 4.8
Al_2O_3	%	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.08
Fe_2O_3	%	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.01
TiO_2	%	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
HCl	%	< 0.020	< 0.020	< 0.020	< 0.020	< 0.020	< 0.020	< 0.020	< 0.025
Sieve residue [5] (Mocker method, 45 µm)	%	< 0.05	< 0.025	< 0.025	< 0.025	< 0.025	< 0.025	< 0.025	< 0.200
SiO_2 content	%	> 99.8	> 99.8	> 99.8	> 99.8	> 99.8	> 99.8	> 99.8	> 99.8

1) Based on ISO 9277

2) Based on ISO 787/11

3) Based on ISO 787/2

4) Based on ISO 787/9

5) Based on ISO 787/18

6) Based on the requirements of the HACCP guidelines

7) Tested to Ph. Eur. and USP/NF monographs

The SiO_2 content of AEROSIL® is >99.8 % in relation to the tempered substance

The given data are typical values, specification on request.

	AEROSIL® TT 600	AEROSIL® MOX 80	AEROSIL® MOX 170	AEROSIL® COK 84	AEROSIL® 200 F6)	AEROSIL® 380 F6)	AEROPERL® 300/30
	150–250	60–100	140–200	155–215	175 – 225	350 – 410	270–330
	60	60	50	50	50	50	280
	< 2.5	< 1.5	< 1.5	< 1.5	< 1.5	< 2.0	< 3.5
	3.6 – 4.5	3.6 – 4.5	3.6 – 4.5	3.6 – 4.3	3.7 – 4.5	3.7 – 4.5	4.0–6.0
	< 0.05	0.3 – 1.3	0.3 – 1.3	14–18	< 0.03	< 0.03	/
	< 0.003	< 0.01	< 0.01	< 0.1	< 0.003	< 0.003	/
	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	/
	< 0.025	< 0.025	< 0.025	< 0.1	< 0.020	< 0.020	/
	< 0.05	< 0.1	< 0.1	< 0.1	< 0.025	< 0.025	/
	> 99.8	> 99.8	/	/	/	/	99.0 - 100.5

More detailed information on the container sizes of AEROSIL® can be found in Technical Information TI 1231, “Types of Packaging for AEROSIL®.”

Hydrophobic or surface-treated AEROSIL®

Test methods	Unit	AEROSIL® R 972	AEROSIL® R 974	AEROSIL® R 202	AEROSIL® R 208	AEROSIL® R 805	AEROSIL® R 812	AEROSIL® R 812 S	AEROSIL® R 104
Behavior in relation to water	Hydrophobic								
BET surface area 1)	m^2/g	90–130	150–190	80–120	80–140	125–175	230–290	195–245	125–175
Tamped density 2) Approximate value Standard product	g/l	50	50	60	60	60	60	60	50
Loss on drying 3) (2 hours at 105 °C) on leaving the supplier factory	%	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Carbon content 4)	%	0.7–1.0*	0.8–1.4	3.5–5.0	4.5 – 6.5	4.5–6.5	2.0–3.0	3.0–4.0	1.0–2.0
pH value 5)		4.0 – 5.5*	3.8 – 5.0	4.0–6.0	4.5 – 6.5	3.5–5.5	5.5–8.0	5.5–9.0	> 4.0
Al_2O_3	%	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Fe_2O_3	%	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
TiO_2	%	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
HCl	%	< 0.05	< 0.05	< 0.025	< 0.025	< 0.025	< 0.025	< 0.025	< 0.02

Test methods	Unit	AEROSIL® RY 50	AEROSIL® NY 50	AEROSIL® RY 200	AEROSIL® RY 200 S	AEROSIL® RY 200 L	AEROSIL® RY 300	AEROSIL® RX 50	AEROSIL® NAX 50
Behavior in relation to water	Hydrophobic								
BET surface area 1)	m^2/g	15 – 45	20 – 40	80 – 120	65 – 95	80 – 120	110 – 140	25 – 45	30 – 50
Tamped density 2) Approximate value Standard product	g/l	130	60	50	50	30	50	170	60
Loss on drying 3) (2 hours at 105 °C) on leaving the supplier factory	%	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Carbon content 4)	%	3.0 – 4.5	2.5 – 4.0	4.0 – 6.5	3.0 – 5.0	4.0 – 6.0	6.0 – 8.5	0.5 – 1.0	0.5 – 1.0
pH value 5)		4.5 – 7.5	5.0 – 6.0	4.0 – 7.0	4.5 – 6.5	4.0 – 7.0	4.5 – 5.5	6.0 – 8.0	5.5 – 7.5

* A narrower range may apply in certain regions.

1) Based on ISO 9277

2) Based on ISO 787/11

3) Based on ISO 787/2

4) C content based on ISO-3262-20

5) Based on ISO 787/9

7) Tested to Ph. Eur. and USP/NF monographs

The SiO_2 content of AEROSIL® is >99.8 % in relation to the tempered substance

The given data are typical values, specification on request.

AEROSIL® R 106	AEROSIL® R 816	AEROSIL® R 711	AEROSIL® R 7200	AEROSIL® R 8200	AEROSIL® R 9200	AEROSIL® R 504	AEROSIL® R 972 Pharma[6]	AEROSIL® R 976 S
220–280	170–210	125–175	125–175	135–185	150–190	125–175	90–130	215 – 265
50	60	60	230	140	200	50	50	50
< 0.5	< 1.0	< 1.5	< 1.5	< 0.5	< 1.5	< 1.5	/	< 0.5
1.5–3.0	0.9–1.8	4.5–6.5	4.5–6.5	2.0–4.0	0.7–1.3	2.0 – 4.5	/	1.8 – 2.1
> 3.7	4.0–5.5	4.0–6.0	4.0–6.0	> 5.0	3.0–5.0	8.5 – 11.0	/	4.0 – 5.5
< 0.05	< 0.05	< 0.05	< 0.05	/	/	< 0.05	/	/
< 0.01	< 0.01	< 0.003	< 0.003	/	/	< 0.01	/	/
< 0.03	< 0.03	< 0.03	< 0.03	/	/	< 0.03	/	/
< 0.025	< 0.025	< 0.01	< 0.01	/	/	< 0.025	/	< 0.020

AEROSIL® RX 200	AEROSIL® RX 300	AEROSIL® NX 90 G	AEROSIL® REA 200	AEROSIL® REA 90	AEROSIL® NA 50 Y	AEROSIL® NA 200 Y	AEROSIL® NA 50 H	AEROSIL® RA 200 HS
115 – 165	180 – 220	50 – 80	110 – 150	40–70	25 – 45	100 – 150	30 – 50	120 – 160
50	50	40	50	50	40	50	50	40
< 0.5	< 0.5	< 0.5	< 1.0	< 1.0	< 0.8	< 0.8	< 0.8	< 0.8
1.5 – 3.5	2.5 – 5.0	0.7 – 1.5	5.0–7.5	3.0 – 6.0	2.0 – 4.0	3.0 – 6.5	< 2.0	< 3.5
5.5 – 8.5	6.0 – 8.0	5.0 – 7.5	8.0 – 10.0	7.5 – 10.0	6.5 – 9.0	6.5 – 9.5	8.0 – 10.0	8.0 – 10.0

More detailed information on the container sizes of AEROSIL® can be found in Technical Information TI 1231, "Types of Packaging for AEROSIL®."

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8.3 Glossary

A – C

Adsorption
A substance adhering to the inner and/or outer surface of a solid (as defined in DIN 28400).

Adsorption isotherms
Adsorption isotherms show the relation between the amount of substance adsorbed on the adsorbent and the concentration or partial pressure of the substance/gas being adsorbed in a state of equilibrium at constant temperature.

AERODISP®
Registered trademark of Evonik Industries AG or its subsidiaries for dispersions of fumed silica and fumed metal oxides (trade name AEROSIL® in aqueous or organic media, such as ethylene glycol).

AEROPERL®
Registered trademark of Evonik Industries AG or its subsidiaries for a spherical granulate made from AEROSIL® or metal oxides with high tamped density and consistently large surface area. The microgranulate displays good flow properties and low-dust handling.

AEROSIL®
Registered trademark owned by Evonik Industries AG or its subsidiaries for synthetic amorphous silicas produced using the high-temperature hydrolysis method.

AEROXIDE®
Registered trademark owned by Evonik Industries AG or its subsidiaries for fumed aluminum and titanium oxides produced using the high temperature hydrolysis method.

Affinity
When two elements or compounds join, the more heat that is released, the stronger their affinity (THOMSEN-BERTHELOT principle).

Agglomerates
Loose collections of → *primary particles* and/or aggregates that can be split up during dispersion (as defined in DIN 53 206).

Aggregates
As defined in DIN 53206, a merged group of particles positioned next to one another in one plane, with a surface smaller than the total surfaces of the → *primary particles*.

Aliphatic
Aliphatic hydrocarbons (such as alkanes) are non-aromatic organic compounds that consist of carbon and hydrogen. Unsaturated hydrocarbons (compounds that contain double or triple carbon bonds – alkenes or alkynes) are also aliphatic compounds.

Amorphous
shapeless, structureless, as opposed to → *crystalline*.

Anatase
Anatase is one of the three crystal modifications in which titanium dioxide occurs, the other two being → *rutile* and → *brookite*. It is a relatively commonly occurring mineral that crystallizes in the tetragonal crystal system.

BET
→ *Brunauer-Emmett-Teller* specific surface area, in Journal of the American Chemical Society 60 (1938), p. 309

BET method
Measuring the specific surface area on the basis of gas adsorption

Brookite
Brookite is one of the three crystal modifications in which titanium dioxide occurs, the other two being → *anatase* and → *rutile*. It is named for the British mineralogist H. J. Brooke (1771–1857).

Catalysis, heterogeneous
In heterogeneous catalysis, the catalyst and educts/products are in different phases (for example a solid catalyst with reacting substances in the gas phase).

Chemisorption
A gas, liquid, or dissolved solid attaching/adhering to the surface of a solid or a liquid with a chemical bond being formed.

Chromatography
A physical separation technique involving material being distributed between a stationary and a mobile phase.

Coagulation
From the Latin coagulatio (clot). Small dispersed particles combining under the influence of forces, such as those occurring in Brownian motion or electrostatic attraction.

C – H

Coalescence
Separate colloidal particles merging (in the present context, it refers to droplets merging in AEROSIL® particle genesis).

Coesite
A high-pressure modification of SiO_2. Detected as a naturally occurring mineral in the sandstone in meteor craters.

Coherent
In physics, two waves are coherent if they have a constant phase relationship.

Compactate
Result of compaction, also referred to as granulate. Solid particles that have been compressed to form larger (ideally more compact) balls, grains, slugs, etc.

Covalence, covalent bond
A covalent bond is a type of chemical bond responsible for holding atoms firmly together in molecular compounds.

Cristobalite
A stable modification of silicon dioxide that is stable between 1,470 °C and the melting point of quartz (1,710 °C). Forms small, cloudy, milk-white crystals in the cavities of volcanic rocks.

Crystalline
A substance is crystalline if its components are arranged in regular crystal lattices. The opposite of → *amorphous*.

Deformation vibration
Vibrations of bound atoms in a molecule involving a change to the bond angle. Characteristic deformation vibrations are used to determine the structure of molecules in IR spectroscopy.

Desorption
A substance being separated or released from the inner and/or outer surface of a solid. The opposite of → *adsorption*.

Desorption curve
The desorption curve depicts the separation process of → *desorption*. For example, it may depict N_2 physisorption in relation to relative pressure.

Differential thermal analysis
A thermometric analysis method. Allows phase transformations to be observed on the basis of their heat tones and is particularly suitable for studying structural changes in solids at higher temperatures.

Diffraction rings
→ *X-ray diffraction*.

Diffusion
Different gases, liquids, or solids that have been brought into contact gradually mixing together without external stimulus.

Diglyme
An abbreviation for diethylene glycol dimethyl ether.

Dioctyl adipate
Abbreviated as DOA as per DIN EN ISO 1043-3:2000-01, refers to dioctyl adipate/bis(2-ethylhexyl)-adipate, a plasticizer. A colorless liquid with barely any odor that belongs to the group of dicarboxylic acid esters (adipates). Highly soluble in acetone, diethyl ester, and ethanol.

Dipole
A molecule with asymmetrically distributed charge.

Dipole-dipole interaction
Forces that act as the result of → *dipoles* being present.

Disagglomeration/de-agglomeration
A generally mechanical process involving aggregate groups with relatively loose attachments being broken up (for example by dispersion).

Dispersing
Using dispersion machines to evenly distribute powdered substances in liquids.

Dispersion
From the Latin dispersio (scatter). As defined in DIN EN ISO 862:1995-10, a system (a disperse system) consisting of multiple phases, with one continuous phase (the dispersing agent or dispersant) and at least one finely distributed phase (the dispersed phase). Examples of dispersions: emulsions (immiscible liquid phases), aerosols, suspensions. Forms of energy that can be used to form a dispersion include chemical, electrochemical, electrical, and mechanical energy.

Dissolvers
Dissolvers are used to disperse substances, most commonly in liquids. The dissolver provides the energy required for → *disagglomeration*.

Doping
In semiconductor production, doping is the standard term for intentionally reducing the purity of extremely pure germanium and silicon crystals by adding minute amounts of foreign material.

Educt
The starting material in a chemical reaction.

Elastic modulus
The elastic modulus (Young's modulus) is a material constant used in materials science. It depicts the relationship between tension and expansion in the deformation of a solid body in the case of a linear elastic response.

Electric arc silica
Fumed silica produced by reducing quartz sand with coke in an electric arc furnace and then oxidizing the SiO that is formed.

Emulsion
Systems with relatively low thermodynamic stability, consisting of a fine distribution of two liquids with low miscibility.

Extinction
From the Latin extinctio (quench). The logarithm of the quotient of the intensity of the primary beam to the intensity of the sample beam.

Flame hydrolysis
A substance such as silicon tetrachloride reacting with the water that forms in the oxyhydrogen flame.

Fumed
Produced in a flame.

Fused quartz
Quartz glass, glass made from pure silicon dioxide, free from impurities

Fused quartz
Fully transparent clear glass with a high melting temperature (1,720 °C) and high chemical resistance.

Fused silica
Cloudy, non-transparent silica glass that is unsuitable for optical purposes.

geminal
relating to two substituents attached to the same atom, → *vicinal*.

Handling
Measures and practices implemented to produce specific operating procedures, particularly those involving powdered raw materials, in compliance with regulatory guideline values and limits.

Headspace analysis
A type of analysis of the air space (headspace) above a solid or a liquid, mainly involving gas chromatography (→ *chromatography*).

High temperature form
→ *Modification* that is formed preferentially at higher temperatures.

Highly dispersed
Finely distributed.

Homogeneous
Of the same kind, consisting of the same substance.

Hydrogen bond
A bond that forms between a hydrogen atom covalently bonded to an electronegative element (→ *proton donator*) and the lone electron pair of another electronegative atom (→ *proton acceptor*).

Hydrolysis
Cleaving a chemical compound by reacting it with water.

Hydrophilic
Attracted to or able to be wetted by water. The opposite of → *hydrophobic*.

Hydrophobic
Water-repellent. The opposite of → *hydrophilic*.

Hysteresis
From Greek hystéresis (lag behind). A physical variable causing an effect with some delay, combined with a residual effect that continues after the cause has ceased.

I – R

IDISIL®

Registered trademark of Evonik Industries AG or its subsidiaries for highest quality colloidal silica products. IDISIL® materials cover particle sizes from 5 to 150 nm, with a range of stabilizing ions, pH and particle size distribution at various concentrations.

Inert

Inactive, not chemically reactive.

Inerting

Turning substances into chemically inactive (inert) compounds.

Infrared (IR) spectroscopy

An optical procedure that uses the absorption spectra of solid, liquid, or gaseous compounds, mainly organic ones, in the infrared range to perform qualitative or quantitative analysis, determine structure, and the like.

Interference

Phenomenon that occurs when waves of the same wavelength superpose.

Irreversible

Of a chemical reaction, not able to be reversed, → *reversible*.

Kieselguhr

Synonym for infusorial earth, diatomaceous earth, diatomite. Very fine-grained, lightweight, normally light gray powder consisting of 70–90 % → *amorphous* silica, a few percent of → *crystalline* silica, 3–12 % water, and small amounts or organic impurities.

Macropores

IUPAC defines macropores as pores in a solid with diameters >50 nm. → *micropores* <2 nm and → *mesopores* between 2 and 50 nm.

Mesopores

IUPAC defines mesopores as pores in a solid with diameters between 2 and 50 nm. → *micropores* <2 nm and → *macropores* >50 nm.

Metastable

A state between stable and unstable. A metastable state is stable with regard to minor changes but unstable with regard to larger charges.

Micropores

As defined by IUPAC, micropores are pores in a solid with diameters <2 nm. → *mesopores* 2–50 nm and → *macropores* >50 nm.

Modification

Various states of chemical elements or compounds, displaying different physical properties despite having the same composition.

Monomodal

If a particle size distribution curve has only one maximum, the distribution is referred to as monomodal.

Morphology

Study of the origin and development of shapes and forms.

Nanometer

One millionth of a millimeter = 1 nm.

NMR

Nuclear magnetic resonance. A spectroscopic technique for determining structure.

Octet rule

The tendency of atoms to engage in chemical bonds to achieve an outer electron shell with eight electrons (the noble gas configuration).

Orbital interactions

The electrons of atoms or molecules have particular zones that they occupy. These occupation zones overlap or almost touch, with energy possibly being released (producing a stable state).

Osmotic pressure

Pressure generated by the tendency of a concentrated solution to dilute itself with the pure solvent when the two are separated by a semipermeable membrane.

Oxyhydrogen reaction
Gaseous hydrogen (H_2) reacting with oxygen (O_2), with water being produced as a reaction product. The conversion (detonation) normally produces a loud bang.

Pascal second
Unit of dynamic viscosity [Pa*s]

Paste-like
Of a liquid, (highly) viscous.

Phases
Here: the various crystal forms in which a pure chemical substance can occur.

PIDS
Polarization intensity differential scattering. This technique works with polarized light, which leads to a better resolution. As a result, smaller particles can be measured than is the case for the conventional laser diffraction method. This technique was developed by the company Beckman Coulter.

Precipitated silicas
Synthetic → *silicas* created by reacting sodium silicate with sulfuric acid.

Precursor
A general term for a starting compound that is typically involved in one or more reactions until the target molecule is produced.

Primary particle
As defined in DIN 53206, the smallest particle (individual particle) that a powdered solid can consist of. Such a particle can be recognized as an individual unit under an electron microscope.

Proton acceptor
A compound that can accept protons (H^+ ions) (a BRØNSTED base). The opposite of a → *proton donator*.

Proton donator
A compound that can donate protons (H^+ ions) (a BRØNSTED acid). The opposite of a → *proton acceptor*.

PU
Abbreviation for polyurethane.

Pure quartz
A particularly pure, transparent form of quartz, resembling glass. It often forms trapezohedral-hemihedral individual crystals.

Quartz
The main → *crystalline* → *modification* of SiO_2. The most common mineral in the earth's crust. Generally found in crystalline druses that are colorless, white, with vitreous or greasy luster, opaque, milky, or transparent.

Reflectometer
A device used to measure the reflectance of a substance/surface. Used to determine the luster of coatings on substrates.

Reversible
Of a chemical reaction, able to be reversed. The opposite of → *irreversible*.

Rheology
The study of flow. A branch of physics concerned with describing, explaining, and measuring the phenomena that occur as bodies deform in flow.

Rotor-stator device
A type of dispersion machine used to mix a pigment or a filler material. Consists of an outer stationary ring and an inner concentric rotating ring. Both rings have openings (holes or slits) that the liquid has to pass through under a high shear load.

RTV
Room temperature vulcanizing

Rutile
Rutile is one of the three crystal modifications in which titanium dioxide occurs, the other two being → *anatase* and → *brookite*. It is a relatively commonly occurring mineral that crystallizes in the tetragonal crystal system.

S – Z

Sedimentation
Settling behavior.

SEM image
A scanning electron microscope image.

Short-range order
Approximately ordered regions of an otherwise disordered substance.

Silanes
In the stricter sense, silanes are binary compounds of silicon with hydrogen, with the general formula Si_nH_{2n} + 2. They are thus the Si equivalents of alkanes. The term silane is also used more broadly, to refer to derivatives in which some or all of the hydrogen atoms are replaced by other groups.

Silanol groups
Surface-resident groups on the silica surface with the formula Si-OH.

Silica
Collective term for compounds that share the chemical formula SiO_2. Silica can be either → *fumed silica*, such as → *AEROSIL®*, or → *precipitated silica*. The different forms of silica differ in their physical/chemical properties, such as the size of the → *specific surface area*, the size of the particles, the loss on drying, or the loss on ignition. → *silica gel*, → *electric arc silica*.

Silica gel
Porous → *silica*, manufactured from acid and sodium silicate in the wet production method. The counterpart of → *precipitated silica*.

Silicone rubber
Highly viscous silicone oils that can be cured with peroxides or by other principles to form elastomers. The basic polymer for silicone rubber is dimethylpolysiloxane.

Silicosis
Occupational illness caused by inhaling dust with quartz content.

Siloxane groups
Si-O-Si units produced by the condensation of → *silanol groups*.

Sinter neck
The connection point between two or more particles, created during the sintering process.

Sintering
Sintering involves compacting individual solid particles (such as primary particles, aggregates, etc.) at high temperatures. The sintering process is performed below the melting temperature, so no actual melting takes place. The solid particles gain a certain degree of plasticity that allows them to be firmly joined after they have been pushed together without merging completely (as melted droplets would).

SIPERNAT®
Registered trademark owned by Evonik Industries AG or its subsidiaries for precipitated silica.

Specific surface area
As defined in DIN 66131, the surface area of a solid in relation to its mass, measured in m^2/g. It is generally measured on the basis of the → BET method (Brunauer, Emmett, Teller, in Journal of the American Chemical Society 60 (1938), p. 309).

Stishovite
A very hard, impact-resistant SiO_2 mineral. It is created when relatively large meteorites fall to the earth's surface and the intense blast wave alters the quartz granules embedded in the earth. The silicon atom is surrounded by six equidistant O atoms, rather than four, as is the case for standard SiO_2. Stishovite can also be manufactured synthetically under extremely high pressures.

Stretching vibration
A special form of vibration that molecules perform after they have absorbed electromagnetic radiation. Characteristic IR vibrations (vibration bands) allow the structure of molecules to be determined. In a stretching vibration, the bond lengths of the affected atoms change within a molecule.

Structure modification
A technique for altering the structure (generally breaking down the structure) of fumed metal oxides. The microscopic 3D network of aggregates and agglomerates is changed to create products with new properties. The main characteristics of these products are reduced thickening effect and greater tamped density.

Surface functionality
Functional (organic) group that can be accessed from the outside. Found on the surface of particles.

Tamped density

ISO 787-11 defines tamped density as a measured variable that describes the amount of volume lost by a powdered solid when it is shaken or packed down firmly.

TEM image

A transmission electron microscope image.

Tetragonal

In a tetragonal system, the four bonding electron pairs point to the corners of a → *tetrahedron* and are thus separated by the greatest possible distance (with an angle of approximately 109 °).

Tetrahedron

A solid with four faces, all of which are equilateral triangles.

Thixotropy

The extent to which a liquid's → *viscosity* decreases in accordance with the shear intensity and shear duration and returns to its original state when the shear stress has been removed.

Torsional vibration

A special form of vibration that molecules perform after they have absorbed electromagnetic radiation. Characteristic IR vibrations (vibration bands) allow the structure of molecules to be determined. A torsional vibrations is a deformation vibration in which the bond angle changes while the bond gaps remain approximately constant. The vibration occurs from the molecular level.

Toxicology

The study of substances that are harmful, and in some cases fatal, in excess doses.

Triboelectricity

Static electricity. The generation of voltage when two different isolators become electrically charged with opposite polarities when they are rubbed together. Often characterized as a quotient of charge per mass.

Tridymite

→ *High temperature form* of SiO_2. Commonly hexagonal tabular crystals, 1–4 mm in size, colorless, with a vitreous luster or milky, transparent to translucent. → *Modification* of silica.

Tunnel effect

When atomic particles are faced with a potential hill that has potential energy higher than their kinetic energy, they can overcome it, with a certain probability, by "tunneling" through the hill.

UP

Abbreviation for unsaturated polyester resin.

Van der Waals forces

Named for Johannes Diderik van der Waals. Intermolecular forces that occur as weak bonding forces between inert atoms and saturated molecules, mainly in real gases but also in liquids and in solids.

Vicinal

Relating to substituents attached to adjacent atoms, → *geminal*.

Viscosity

DIN 13342 defines viscosity as substance's ability to absorb a shear stress that is dependent on the shear rate through shear deformation.

X-ray analysis

Physical technique for chemical analysis, with the sample exposed to → *X-rays*.

X-ray diffraction

Technique for analyzing crystal structure, involving → *X-rays* being diffracted by the electrons of lattice atoms. Superposing the diffraction waves produces regular → *diffraction rings*.

X-rays

Short-wave electromagnetic radiation, with a wavelength of approximately 10^{-9} to 10^{-12} m.

Zeta potential

Even in a suspension, solid particles (such as AEROSIL®) carry charges on their surfaces that are compensated/screened by solvent molecules on the outside. This minimizes the potential. When the particles move (as the result of Brownian motion, stirring, etc.), friction is caused, which allows this layer of solvent molecules to be sheared off from the surface of the particle. The surface charge of the particles is now no longer neutral on the outside, creating a potential: the zeta potential.

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