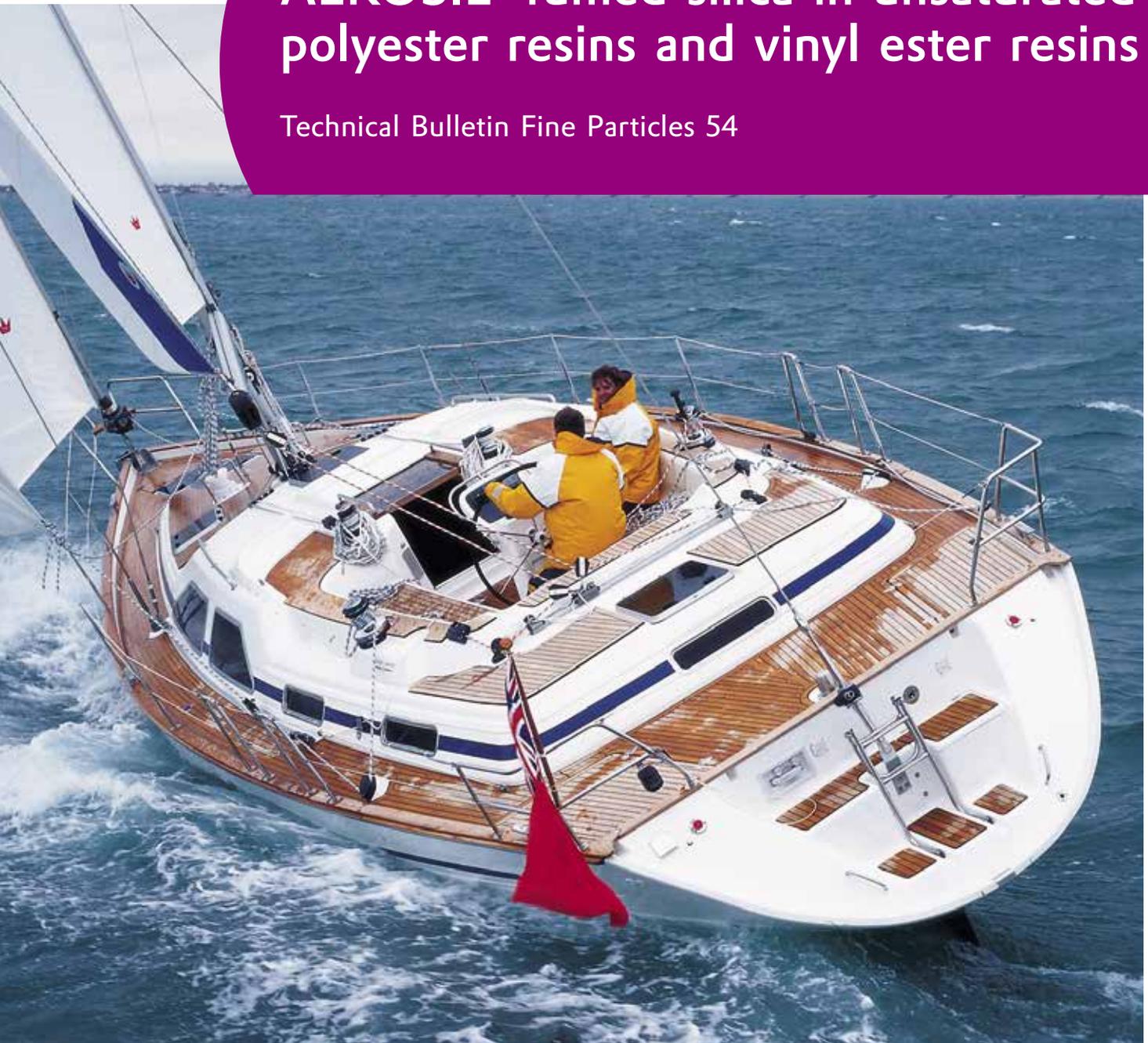


AEROSIL® fumed silica in unsaturated polyester resins and vinyl ester resins

Technical Bulletin Fine Particles 54



The importance of the highly dispersed AEROSIL® fumed silica as a thickening and thixotropic agent for laminating resins and gelcoats based on unsaturated polyester resins and vinyl ester resins has increased with time. Evonik Industries and its affiliate companies manufacture AEROSIL® fumed silica not only in Germany, but also in Belgium, the USA and Japan.

This brochure describes the use of hydrophilic and hydrophobic AEROSIL® types as a thixotrope in various polyester and vinyl ester resins. It specifically deals with the dispersion of AEROSIL® fumed silica using various dispersion units. Relationships between the rheological properties and the dispersibility are addressed. Parameters investigated which influence dispersibility include: direct and master-batch method, mixing time and peripheral velocity of the shearing blade. Topics of practical relevance such as formulation questions are discussed.

Rüdiger Nowak

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Technical Service AEROSIL®

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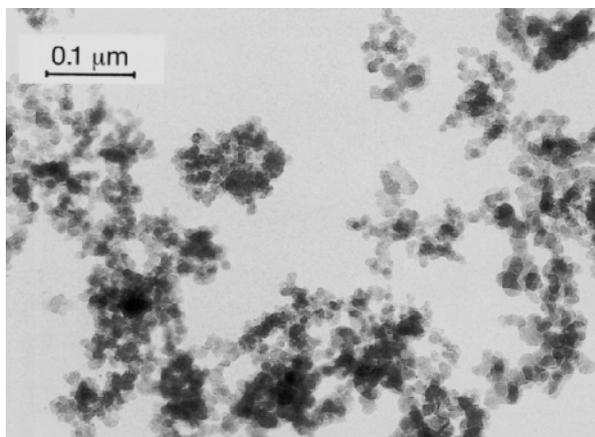
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1 General

1.1 AEROSIL® Fumed Silica

AEROSIL® is a highly dispersed, amorphous, very pure silica which is manufactured by high temperature hydrolysis of silicon tetrachloride in an oxyhydrogen gas flame. AEROSIL® is therefore also referred to as fumed silica.

The primary particles formed in the AEROSIL® process are virtually spherical and are free from pores. In the formation of the primary particles, aggregate bodies are formed which accumulate reversibly into agglomerates. **Figure 1** shows a transmission electron microscope (TEM) photo of AEROSIL® 200, in which the primary particles, aggregates and agglomerates can be seen clearly.

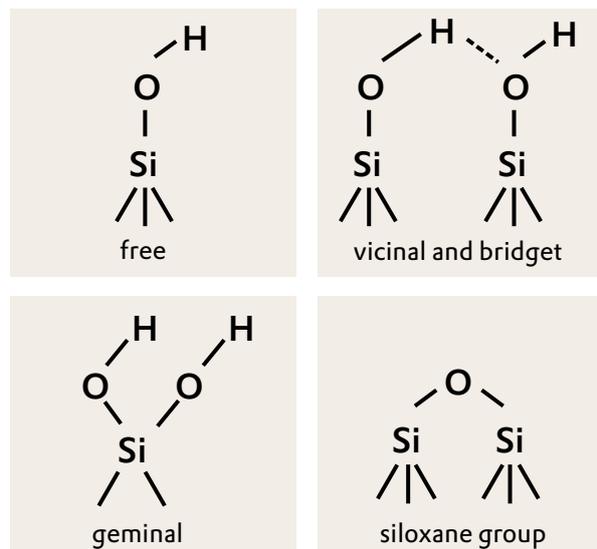


Picture 1: TEM photo of AEROSIL® 200

The average diameters of the primary particles lie between 7 and 40 nm, depending on type. The corresponding specific surface areas are between 50 and 380 m²/g. Siloxane and silanol groups are situated on the surface of AEROSIL® particles. This latter type of functional group in particular is responsible for the hydrophilic behavior of non-aftertreated AEROSIL® types. **Figure 1** shows the surface groups of hydrophilic AEROSIL® fumed silica. For basic details and applications of AEROSIL® fumed silica, see issue number 11 of the pigments publication series [1].

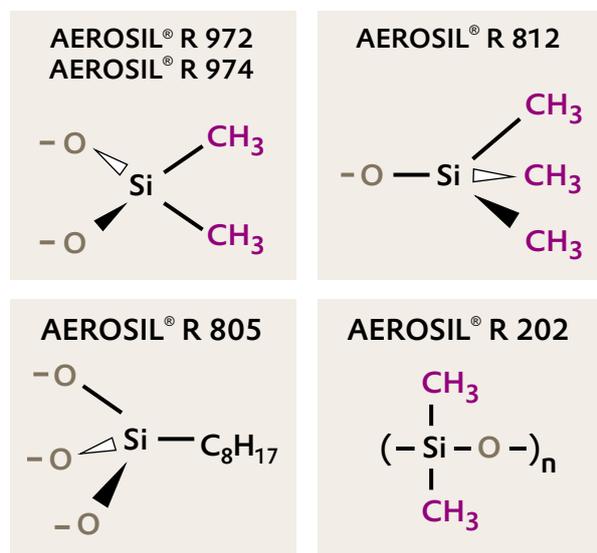
AEROSIL® fumed silica can be surface-modified by reacting the silanol groups with suitable compounds such as silanes. AEROSIL® R 972 is obtained by reaction with dimethyl dichlorosilane, for example; this product exhibits chemically bound dimethyl silyl groups on its surface and can no longer be wetted with water – in other words, it is hydrophobic.

Figure 1 Potential SiO₂ surface groups of hydrophilic silicas



AEROSIL® R 972 became the first hydrophobic silica to be manufactured on an industrial scale in 1962. Further hydrophobic AEROSIL® grades are now available, produced by similar industrial-scale methods using corresponding silanes. **Figure 2** provides a sampling of the many hydrophobic grades available and highlights the difference between them and hydrophilic AEROSIL® grades according to the schematically represented surface groups. All hydrophobic AEROSIL® types carry the suffix „R“ to indicate their water-repellent character.

Figure 2 Hydrophobic AEROSIL® grades and their surface groups



Hydrophilic and hydrophobic AEROSIL® grades have proven effective for numerous areas of application. In view of their use in the plastics industry, the principal properties of fumed silicas are listed below:

- Reinforcing fillers
- Thickening and thixotropic agent for resins
- Anti-sedimentation agent for cast resins and adhesives
- Free flow aid for powder coatings

In contrast to silicas of mineral origin, meaning quartz powder which is used mainly as a „filler“ in the true sense of the word, the desired effects can be obtained by adding only relatively small amounts of synthetic silicas.

A summary of the applications of AEROSIL® fumed silica in general and for plastics in particular is given in [2] and [3]. Since the application-relevant properties of a silica can be optimized for specific applications via the type of surface modification, AEROSIL® R 202, AEROSIL® R 711, and AEROSIL® R 805 were developed.

Theoretical considerations have led to the supposition that longer chain, hydrophobic groups in certain liquid polymer systems—particularly vinyl ester and epoxy resins—ought to make it possible to control the rheological properties more effectively. AEROSIL® R 202, with a surface on which polydimethyl siloxane groups are fixed, and AEROSIL® R 805, which is rendered hydrophobic with octyl groups, fulfill these expectations to a remarkable degree. The hydrophobic character of these two after treated AEROSIL® grades compared with AEROSIL® 300 can be illustrated by means of water vapor adsorption in increasingly humid conditions. **Figure 3** shows the corresponding isotherms.

1.2 Unsaturated Polyester Resins and Vinyl Ester Resins—Significance and Applications

Unsaturated polyester resins (UP resins) are usually produced by the esterification of unsaturated and saturated dicarboxylic acids or their anhydrides with dialcohols at about 200 °C in a molar ratio of 1 : 1 in the melt.

For a standard UP resin (ortho-resin), maleic anhydride, ortho-phthalic anhydride and propylene glycol 1,2 are used, for example. After condensing down to the desired molar mass, which is about 2000 for standard resins, the still warm, highly viscous resins are dissolved in copolymerisable monomers such as 30–40% styrene. In the case of iso-resins, iso-phthalic anhydride is used instead of ortho-phthalic anhydride. The results are better mechanical, chemical and thermal properties. NPG resins contain neopentyl glycol as the alcohol component and likewise exhibit better resistance to chemicals and weathering [4]. Cross-linking of unsaturated polyester resins is achieved by means of graft copolymerisation with styrene and unsaturated dicarboxylic acids. Peroxides and accelerators are used as initiators.

Vinyl ester resins (VE resins) are obtained by the esterification of methacrylic acid with an epoxy resin. In the curing process, the methacrylate groups copolymerise with styrene [16]. Compared with UP resins, vinyl ester resins are more resistant to chemical influences by virtue of the lower number of nonreacted double bonds and ester bonds in the molecular chain [17].

The main area of application for UP and VE resins (approx. 70%) is for fibre-reinforced plastics. The remaining amount is used in non-reinforced applications. The following areas of application are particularly important:

Technical articles, reinforced:

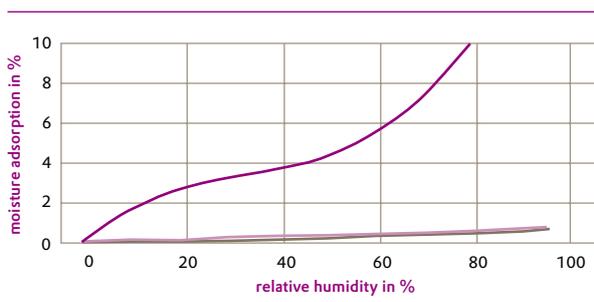
- Construction industry (tanks, containers, pipes)
- Boatbuilding and shipbuilding
- Transport
- Electrical engineering
- Car industry

Technical articles, non-reinforced:

- Moulded parts (buttons, artificial marble)
- Joining technology (adhesives, mortar)
- Surface finishes (putties, cast surfaces and screeds)

Filler compounds and injection fillers

Figure 3 Adsorption isotherm for various AEROSIL® grades at roomtemperature (small samples)

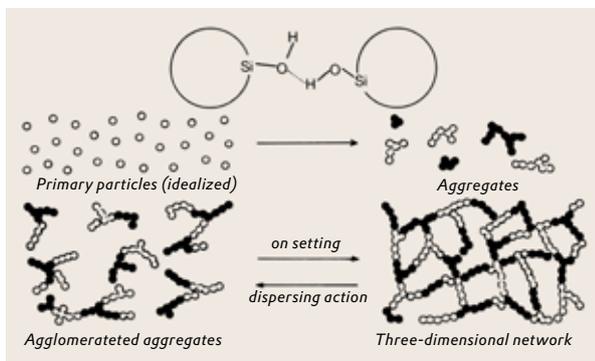


— AEROSIL® 300 — AEROSIL® R 202 — AEROSIL® R 805

2 Mechanism of Thickening and Thixotropy with AEROSIL® Fumed Silica

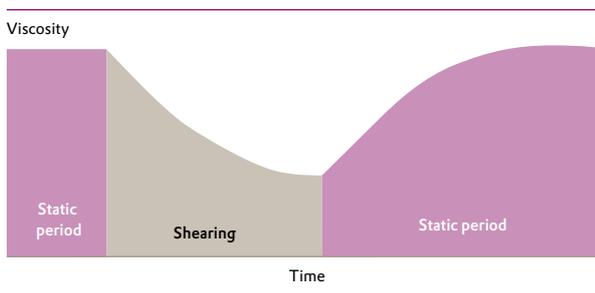
If AEROSIL® fumed silica is dispersed in a liquid, the silanol groups located on the surface interact either directly, or indirectly via the liquid molecules. This affinity is attributable to the hydrogen cross-linking and results in a temporary three-dimensional network structure which becomes „visible“ macroscopically as thickening. If subjected to mechanical loads, whether in the form of intensive stirring or shaking, the network is broken down again and the viscosity of the system is reduced. When static, the AEROSIL® particles interact again and the viscosity rebounds in the original value. This process is referred to as thixotropy and is represented schematically in **Figure 4**.

Figure 4 Schematic graph of the shear thinning effect of AEROSIL® particles in fluids



The concept of the three dimensional network structure can be visualised, as represented schematically in **Figure 5**. In systems thickened with AEROSIL® fumed silica, the original viscosity is almost entirely regained within a matter of seconds after subjecting to a shearing load.

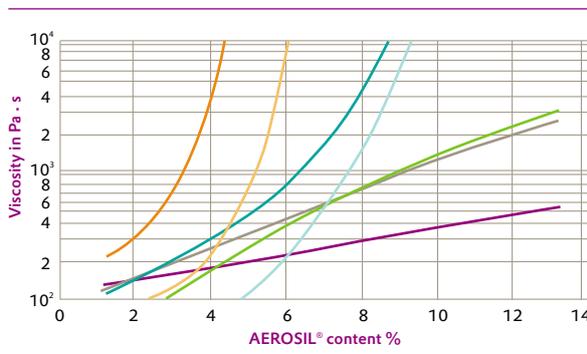
Figure 5 Thixotropy = decrease of the viscosity during the shearing time, and increase again during static period (schematic)



As shown in **Figure 6**, the thickening and thixotropic effect of AEROSIL® fumed silica depends on the system's polarity, with the best results achieved in non-polar systems. In this connection, polarity should be understood to mean the ability of the liquid molecules to

form hydrogen bonding. AEROSIL® COK 84 has proven an effective means of adjusting the rheology of highly polar substances such as water, dimethyl formamide etc. In considering of the rheological effect of hydrophobic AEROSIL® grades, an additional mechanism has to be taken into account because of the specific interaction between silicas and polymers [5,6].

Figure 6 Thickening various liquids with AEROSIL® 200

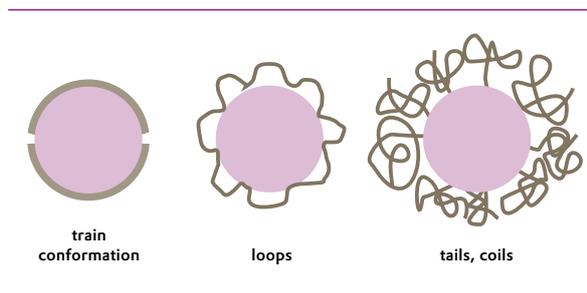


— Carbon tetrachloride — Dibutyl phthalate — Benzene
— Methyl acetate — Acetone — water — Butanol

The polymer molecules in loop and ball conformations are adsorbed at the surface of AEROSIL® fumed silica, with the mobility of the chain segments beyond the polymer/silica boundary layer limited by interlinked and twisted polymer chains.

On AEROSIL® 200, which has a higher density of silanol groups and no functionalisation of the surface, a flat arrangement (train conformation) of polymers is possible (cf. **Figure 7**). In the case of AEROSIL® R 812 S, AEROSIL® R 805 or AEROSIL® R 202, on the other hand, loops or ball shapes (tails, coils) are more probable as a result of surface modification with space filling groups and the lower number of silanol groups (points of attachment).

Figure 7 Potential arrangement for the adsorption of polymers on SiO₂ surfaces



On the basis of this model concept of polymer adsorption on hydrophobic silicas, the following mechanism of rheological effect is proposed:

A thickened system with hydrophobic silica is initially to be considered without shearing. The polymer molecules are firmly adsorbed on the surface and their loop or ball formations protrude far into the solution of binding agent. The adsorbed polymer segments have a cooperative effect, with the mobility of the adjacent polymer segments also restricted. In view of the high molar mass, chains may occur that are twisted and interlinked with the functional groups of silica and reciprocal polymer cross-linking with the adjacent silica particles. A three-dimensional structure is thus created and acts as a flow barrier.

If shearing occurs, this flow barrier is overcome, the chains are disentangled and the static orientation of the polymers is removed. The particles enclosed with polymers and the free polymer molecules move in the shearing field. In the process the polymers assume a thermodynamically unfavourable, extended conformation. After the shearing load, or in other words in the static condition, the polymer segments once again return to their previous static conformation, with intertwining and associations with adjacent silica particles. The system exhibits entropy-elastic properties.

Rheological concepts such as thickening, shear thinning (pseudoplasticity), thixotropy and yield value can be explained clearly with this model concept.

3 Dispersion of AEROSIL® Fumed Silica in Unsaturated Polyester Resins

3.1 Requirements to the Rheological Properties and the Dispersibility

Much has already been written on the fact that by adding approx. 1 to 3% AEROSIL® fumed silica to unsaturated polyester resins, not only is the desired thickening obtained, but the resins are made thixotropic, a vital factor from a processing point of view [7][8].

The thixotropic properties attained by adding AEROSIL® fumed silica mean that the laminating resins, by virtue of their higher viscosity, cannot seep out of the glass fibre material once they have been applied and before they have hardened and that gelcoats applied to sloping or perpendicular surfaces do not run off before they have gelled. The resins moreover exhibit such low viscosity in processing that they are able to soak into and wet the glass fibre materials thoroughly, and can easily be applied by spraying.

A high degree of dispersion of the silicas in the polyester resin is important for the rheological properties and fineness of grind of the silica aggregates in the laminating resins and gelcoats.

The objective of dispersing AEROSIL® fumed silica is to break the silica agglomerates down into aggregates so that the latter are distributed as evenly as possible throughout the resin.

3.1.1 Thickening and Thixotropic Behaviour

It is not difficult to determine the thickening effect by means of viscosity measurement with viscometers; however, reproducible, quantitative measurement of the thixotropic behaviour is only possible to a certain extent. One simple method which makes it possible to reach certain conclusions about the resins' thixotropic behaviour is the "Thixotropic Index". This consists of the quotients from two viscosity values obtained at different speeds. Speeds n_1 and n_2 are usually in an approximate ratio of 1 : 10. In general it can be concluded that a UP resin is sufficiently thixotropic if the Thixotropic Index for laminating resins typically ranges from two to three, and for gelcoats from three to five.

Figure 8 shows the measurement of the viscosity of a UP resin with a rotational viscometer. For the following experiments, the Thixotropic Index was calculated from the viscosities measured at $n_1 = 5$ [rpm] and $n_2 = 50$ [rpm]. Throughout this report, if not explicitly stated, measured viscosity was at 5 rpm.

For gelcoats which are sprayed on airlessly, for example, measurement with a cone-plate rheometer is recommended in addition (see Section 5.3 in this connection).

In this case it is possible to take measurements at the shear rates that are relevant for processing (e.g. 1,000 or 10,000 s^{-1}) and to obtain the desired processing viscosity of e.g. 400 or 500 mPa·s by adding styrene. The cone-plate rheometer moreover makes it possible to record flow curves and to calculate the flow limit and thixotropic area, and to compare them with each other.

3.1.2 Grind Readings

The companies which manufacture laminating resins and gelcoats have adopted the method of measuring the grind readings, a technique that has long been known to and used in the paint industry (DIN 53203 and ASTM No. D 1210-79).

Even if determining the grind reading is somewhat critical for the following limit values, the values which are generally used in practice are given.



Picture 2 Rotational viscosimeter DV-II (Brookfield Engineering Laboratories, Inc., Stoughton, MA, USA, German representative: Brookfield Engineering Lab. GmbH, D-73547 Lorch)

For laminating resins:

Finer than 60 micrometers

For gelcoats:

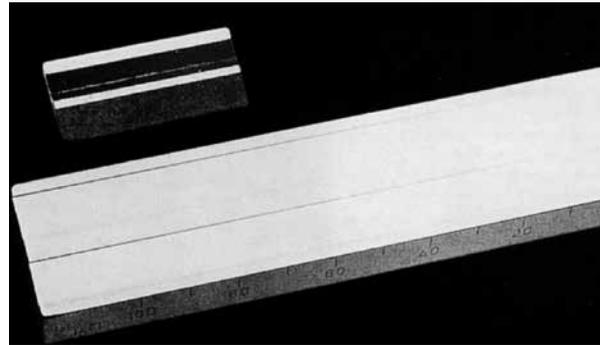
Finer than 40 micrometers

Higher grind readings indicate that the AEROSIL® fumed silica has been insufficiently dispersed, i.e. the agglomerates have not been sufficiently broken down.

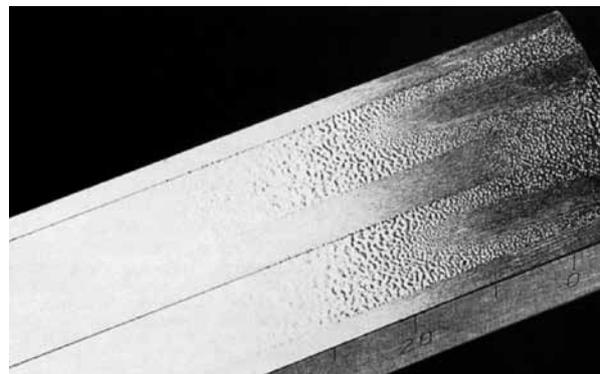
As a consequence of inadequate dispersion, three scenarios may occur in practice:

- 1 The final products exhibit specks and/or other surface flaws.
- 2 The amount of AEROSIL® fumed silica used does not achieve the desired thixotropic effect.
- 3 The thickened UP resins exhibit poor storage stability: sedimentation of AEROSIL® fumed silica may occur in laminating resins, and/or the viscosity originally selected deteriorates the longer the product is stored.

Picture 3 shows the tapered grindometer acc. to DIN 53203 (in increments from 0 to 100 micrometers), and **Picture 4** a UP resin thickened with AEROSIL® fumed silica, with a grind reading of approx. 40 micrometers.



Picture 3 Grind gauge accord. to DIN 53 203 (increments from 0 to 100 µm)

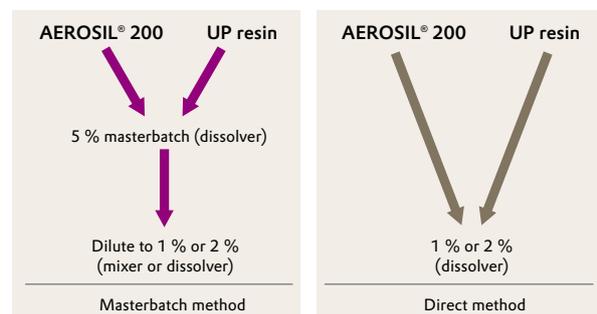


Picture 4 AEROSIL® polyester resin dispersion with a grind reading of approx. 40 µm

3.2 Dispersion of AEROSIL® Fumed Silica with the Dissolver

For dispersing AEROSIL® fumed silica with the dissolver, two different methods can basically be followed, as indicated in **Figure 8**.

Figure 8 Schematic comparison of masterbatch and direct method



In practice, the Cowles dissolver has proven particularly effective. Laboratory and pilotplant dissolvers were therefore used for the experiments described below.

In **Picture 5**, the mixing blade shown is typical of the dissolver (Cowles Division, Inc., Morehouse N. Y., USA).



Picture 5 Lab dissolver blade (Cowles Division, Morehouse, New York, USA)

As different geometrical conditions apply the extent is limited to which the following test results with the laboratory and pilotplant dissolvers can be directly applied on a production scale. The guideline values stated below for the circumferential speed and geometrical dimensions generally apply for production-scale dissolvers [15].

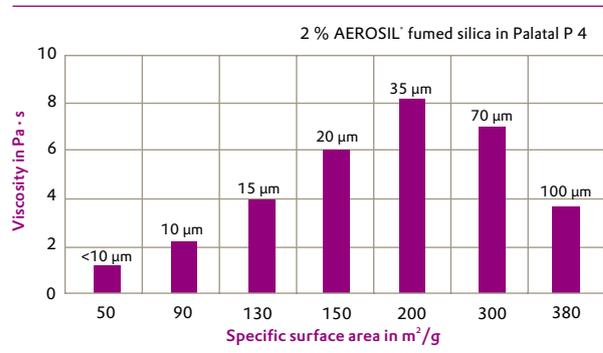
Gelcoats are produced predominantly with the aid of dissolvers. However, other dispersion units such as the Ultra-Turrax can also be used. Laminating resins are produced both with the aid of ultrasonic dispersion equipment and with dissolvers or similar mixers.

- Circumferential speed (m/s):
24 (possible range 22–27 m/s)
Problem: *Critical speed (resonance of shaft) depends on the speed and the diameter of the shaft.*
- Height of mixing container/diameter of mixing container = 1.2/1 (1.0/1.0 for higher-viscosity systems, recommended: Smaller mixer blade diameter)
- Diameter of dissolver plate about 40% of mixer blade diameter (40–60% possible)
- Distance from base approx. 0.5 of the diameter of the dissolver blade

3.2.1 Influence of BET Surface Area of AEROSIL® Fumed Silica

The thickening effect and the dispersibility of various hydrophilic AEROSIL® types with specific surface areas ranging from 50 m²/g to 380 m²/g are compared in **Figure 9**.

Figure 9 Thickening effect and dispersibility of hydrophilic AEROSIL® types in a polyester resin



In each case, 2% AEROSIL® fumed silica was incorporated into and dispersed in Palatal® P4 (Ortho resin, manufacturer: DSM Resins, Zwolle, Netherlands) by the direct method, with the laboratory dissolver. The viscosities of the thickened polyester resins were measured with a Brookfield viscometer at 5 rpm and 25 °C.

AEROSIL® 200 in Palatal P4 exhibits the highest thickening effect. The thickening effect for AEROSIL® 300 is slightly lower. The grind readings increase with the specific surface area, i. e. comparatively little dispersion work is needed for AEROSIL® 200 to achieve a good grind reading compared with AEROSIL® 380.

AEROSIL® 200 is usually used for laminating resins and gelcoats. In special systems such as highly-filled gelcoats, AEROSIL® 300 often exhibits an even better thickening effect than AEROSIL® 200.

The rule that the thickening effect increases from AEROSIL® OX 50 to AEROSIL® 380 only applies for very high-viscosity polymer systems such as PVC plastisols or silicone rubber (cf. also [9] and [10]). In these systems, the shearing forces are so high that an optimum interaction between the aggregates of AEROSIL® 300 resp. AEROSIL® 380 and the polymer can be achieved.

The shearing forces in the relatively low-viscosity laminating resins and gelcoats are lower, with the result that the AEROSIL® 380 can no longer be dispersed to an optimum degree and the increase in viscosity achieved is much lower than for AEROSIL® 200.

3.2.2 Dispersion according to the Masterbatch Method

This method involves the initially making of a concentrate of approx. 5% AEROSIL® 200 or AEROSIL® 300 in a polyester resin (cf. **Figure 8**).

This higher concentration is only used in order to achieve higher shearing forces during the dispersing process than are possible when using the direct method.

Following dispersion with a high AEROSIL® fumed silica concentration, the mixture is diluted to the desired low concentration with more UP resin and styrene. This diluting process involves taking the AEROSIL® concentrate, switching on the mixer at a relatively low speed and adding a pre-weighed amount of polyester resin and styrene. The masterbatch method is used predominantly with solvents and similar dispersing equipment.

3.2.3 Dispersion according to the Direct Method

The direct method involves incorporating the correct AEROSIL® concentration for the final product directly into the UP resin.

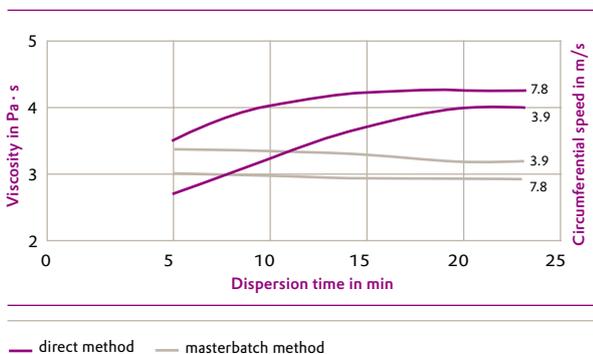
The direct method is used most frequently for producing gelcoats and laminating resins. The reason for this approach is that the diluting process is avoided, consequently rendering the direct method less labour-intensive than the masterbatch method.

Ultrasonic devices and devices which operate according to a continuous circuit have to use the direct method due to their principle.

3.2.4 Influence of Mixing Time

As shown in **Figure 10**, at a laboratory dissolver's given rotating speed in a polyester resin thickened with 2% AEROSIL® 200 by the direct method, the viscosity increases until it reaches a plateau after a mixing time of between 10 and 15 minutes. The mixing container used for these experiments had a diameter of 7.2 cm and the dissolver blade a diameter of 4.8 cm.

Figure 10 Viscosity of a polyester resin (Palatal P 6) with 2% AEROSIL® 200 as a function of the dispersion time.

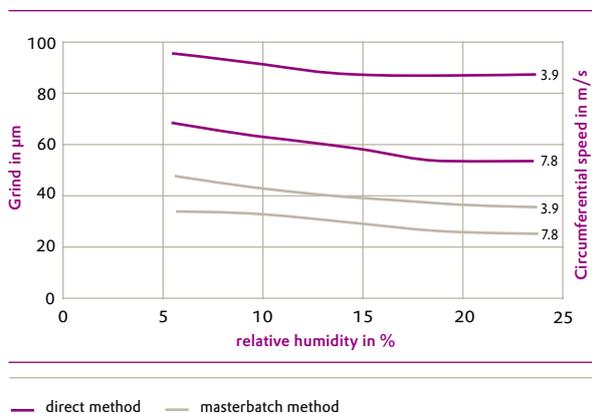


With the same AEROSIL® 200 concentration, lower viscosity values are achieved via the masterbatch method.

Another difference in this two-stage method is that the peak viscosity is measured after a mixing time of 5 minutes. Both statements are clearly substantiated by **Figure 10** and remain valid at a variety of circumferential speeds.

In addition, this illustration shows that lower viscosity values are achieved at higher circumferential speeds with the master-batch method compared with the direct method. **Figure 11** indicates how the grind reading depends on the mixing time at the selected AEROSIL® 200 content of 2%. Here again, the benefits of the masterbatch method compared with the direct method are evident.

Figure 11 Relationship between the grind reading of a polyester resin (Palatal P 6) thickened with 2% AEROSIL® 200 and the dispersion time



3.2.5 Influence of Mixer Blade's Tip Speed

The considerable importance of the mixer-blade's tip-speed (peripheral speed) is evident from **Figure 10** and **11**. As the speed is increased, the more the viscosity values change and the better the grind readings obtained.

Figure 12 Viscosity and grind reading of a polyester resin (Palatal P 6, dispersion time: 5 min) thickened with 2% AEROSIL® 200 as a function of the circumferential speed of the dissolver blade and the method of incorporation

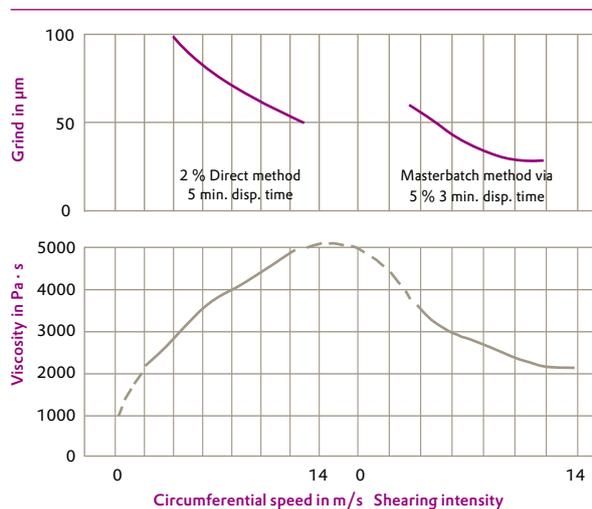


Figure 12 shows the viscosities and the grind readings as a function of the tip speed, with dispersion taking place at a tip speed of up to 13 m/s both for the direct method and the masterbatch method.

If the two viscosity curves are now merged into a single curve, the result is a maximum level beyond which viscosity declines again, for the purposes of obtaining a limit value.

The relatively low viscosity values from the masterbatch method result from the fact that the AEROSIL® aggregates are wetted more effectively by the resin as a result of higher shearing forces. However, these lower viscosity values are balanced by improved storage stability values and better grind readings. If the direct method is used, higher tip speeds between 20 and 25 m/s should be selected. At these high blade tip speeds, even with the direct method the process remains on the right of the viscosity curve, which falls only slightly at this point. This therefore renders it by and large possible to avoid the corrections which are caused by changes in viscosity.

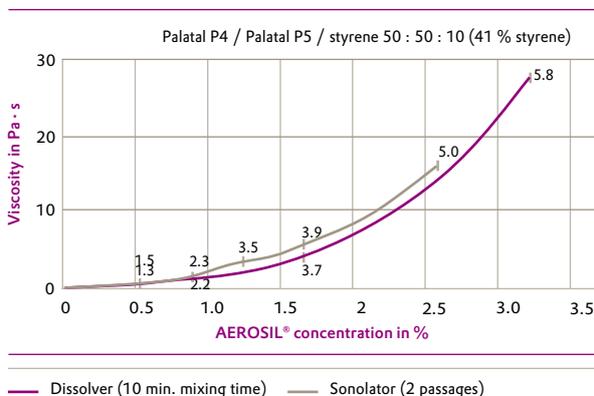
3.3 Comparison of AEROSIL® Dispersion with Sonolator and Dissolver

An ultrasonic dispersion device („ULTRASON®“ homogeniser, manufactured by Branson Ultrasonics Ltd, Hayes, Middlesex, England) was available. A mixture of the Palatal® types P4 and P5 was used for the experiments. The exact conditions of the experiment are described in Section 8.

3.3.1 Influence of AEROSIL® Concentration

Figure 13 shows that the viscosity of UP resins rises exponentially at increasing AEROSIL® concentrations, both with the sonolator and with the dissolver. The Thixotropic Index likewise stated in **Figure 13** as a parameter also increases. Very similar rheological properties are achieved with both dispersing devices.

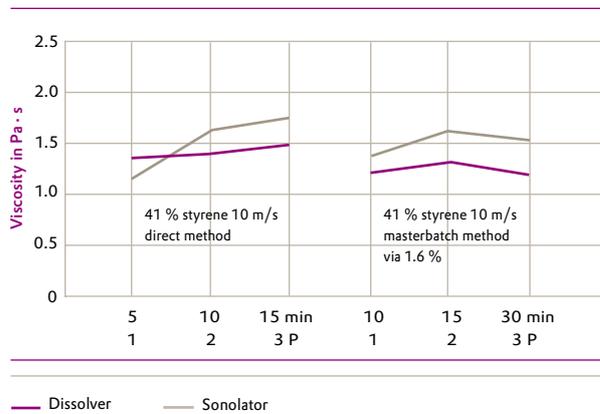
Figure 13 Viscosities of thickened polyester resins in relation to the AEROSIL® 200 content



3.3.2 Influence of Various Dispersion Conditions

Figure 14 shows the viscosity of UP resins with 0.8% AEROSIL® 200 in relation to various dispersion conditions.

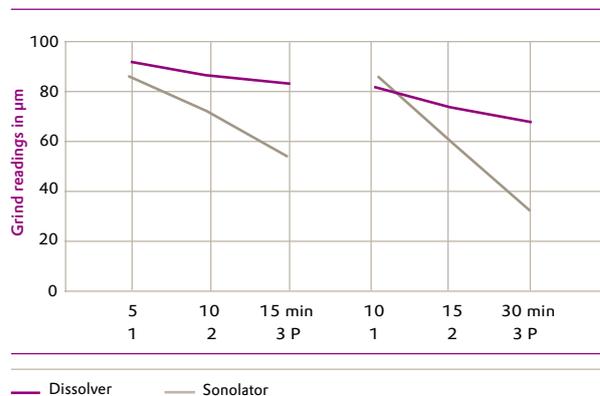
Figure 14 Viscosities of UP resins thickened with 0.8% AEROSIL® 200 in relation to various dispersion conditions



It is notable that for 40% styrene content, 10 m/s circumferential speed and the direct method (left-hand section of diagram), the curves exhibit a rising tendency. In light of the considerations described in Section 3.2.5, this means that the laminating resins have not yet been fully dispersed. The maximum viscosity referred to only appears at the higher concentrations via the masterbatch method (right-hand section of diagram).

Figure 15 confirms the opinion already stated. It is apparent that every additional sonolator passage and any extension to the dispersing duration produces a further reduction in the grind readings as a measure of dispersion quality. The experiments moreover show that maximum fineness is achieved with the sonolator, although a single passage is entirely inadequate to achieve this state.

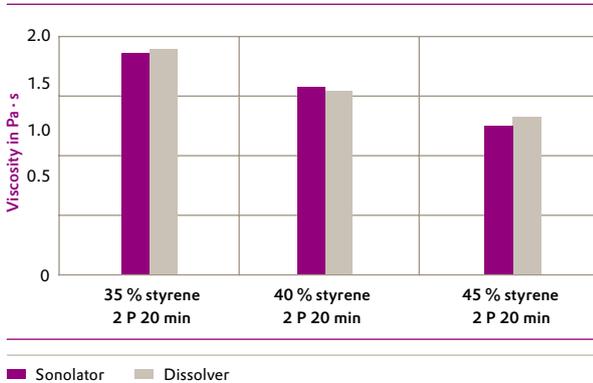
Figure 15 Grind readings of UP resins thickened with 0.8% AEROSIL® 200 in relation to various dispersion conditions as mentioned in Figure 14



3.3.3 Influence of the Styrene Content

Figure 16 shows that the viscosity level achieved with AEROSIL® 200 steadily declines with a rising styrene content. However, the viscosity of the resin that without AEROSIL® 200 also shows a natural decline.

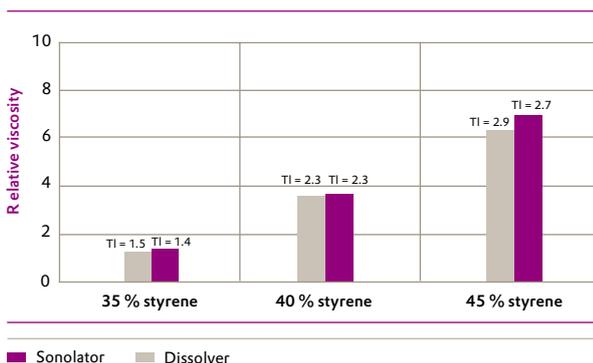
Figure 16 Viscosity of UP resins thickened with 0.8% AEROSIL® 200 in relation to the styrene content



In order to ascertain why the resins with a 0.8% AEROSIL® 200 content exhibit lower viscosity with higher styrene content—i. e. whether this is simply due to dilution with styrene or can be ascribed to a change in the silica’s effectiveness—the term „relative viscosity“ was introduced [11] and defined as the quotient η/η_0 of the viscosity η of the thickened resin and of the viscosity η_0 of the resin without AEROSIL® fumed silica. The „relative viscosity“ therefore serves as a measure of the „thickening capacity“ of AEROSIL® 200.

Figure 17 shows the results of this calculation. It can be seen that the thickening capacity of AEROSIL® 200 increases significantly with higher styrene content. The Thixotropic Index also given in **Figure 17** likewise increases, to the values 1.5, 2.3 and 2.9.

Figure 17 „Relative viscosity“ of UP resins thickened with 0.8% AEROSIL® 200 in relation to the styrene content



The reason for this outcome is that the polarity of the resin/styrene mixture decreases with a rising styrene content, with the result that the silica encounters more favourable conditions for achieving its effect. Since a less polar resin wets the silanol groups on the AEROSIL® surface to a lesser extent, it helps these to form links via hydrogen bonds and to generate a three-dimensional gel structure of AEROSIL® particles.

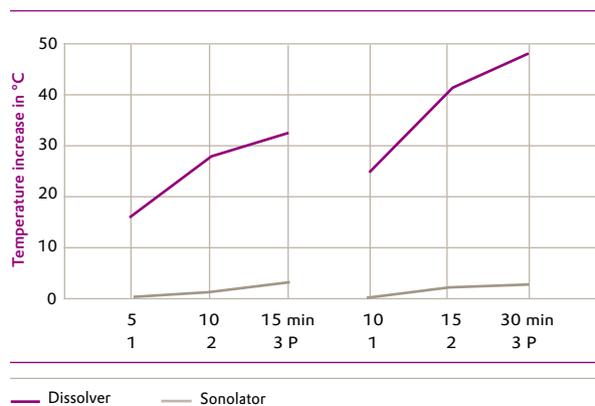
As the styrene content rises, the viscosity of both the resin without AEROSIL® fumed silica and the thickened resin decreases. The shearing forces during dispersion consequently also fall. It was therefore perfectly conceivable that the thickening effect of the silicas could have fallen with a rising styrene content.

However, the fact that the reverse was the case indicates the predominant effect of polarity on the resin’s viscosity following dispersion. These circumstances are also of significance to the rheological properties of gelcoats, cf. also Section 5.3.

3.3.4 Temperature Increase during Dispersion

Figure 18 indicates the measurements for increases in the resin temperature after 1, 2 and 3 passages through the sonolator or 10, 20 and 30 min. dispersion with the dissolver. The experiments carried out show that the temperature increases much less with the sonolator than with the dissolver. Further, it is notable that the increase in temperature after passing through the sonolators was very low even for the 3.2% pre-concentrates, including when higher-viscosity resins (as a result of their lower styrene content) were used. The dissolver, on the other hand, led to a considerable increase in temperature after a relatively short mixing time at the high circumferential speed of 15 m/s, which was selected with a view to achieving good dispersion properties.

Figure 18 Temperature increase during the dispersion process of UP resins thickened with 0.8% AEROSIL® 200 (left) and 3.2% AEROSIL® 200 (right) (35% styrene content)

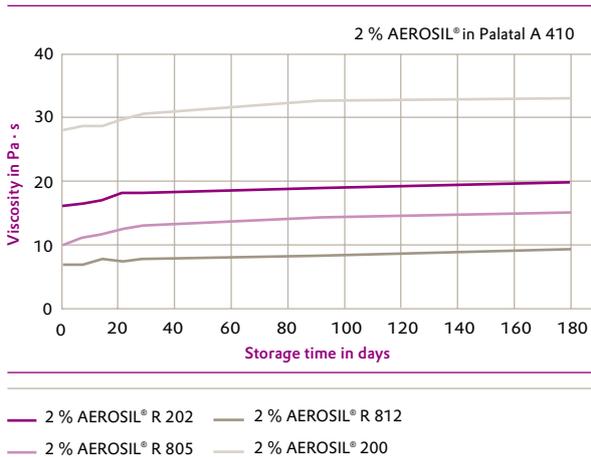


3.4 Storage Stability

UP resins containing AEROSIL® fumed silica exhibit adequate storage stability combined with sufficient dispersion, which goes hand in hand with complete or extensive wetting of the primary particles and aggregates. Because of their relatively high silica concentration of approx. 2 to 2.5% (or higher), storage stability problems are not generally encountered with gelcoats.

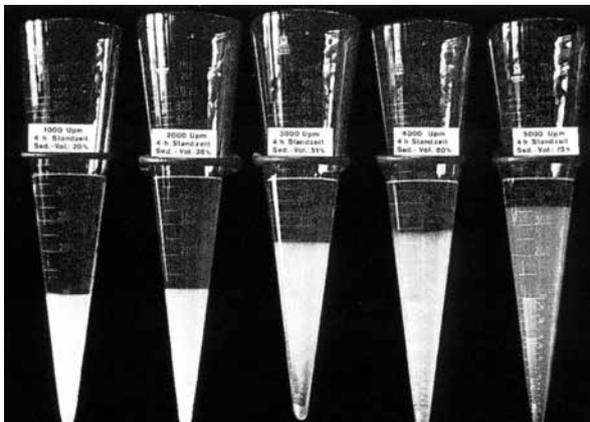
Figure 19 shows the long-term behaviour of Palatal A 410 (45% styrene content, iso resin) thickened with 2% AEROSIL® 200 after a storage time of 6 months.

Figure 19 Viscosity time curves of Palatal A 410, thickened with 2% of various AEROSIL® grades (dissolver dispersion, direct method)



If laminating resins are stored in unfavourable conditions (e. g. at high temperatures), signs of sedimentation of AEROSIL® 200 may occur because of the low silica concentration typically used (approx. 0.7–1.3%). If sedimentation of AEROSIL® fumed silica occurs during storage, this can also possibly be a sign of deficient dispersion. The quality of the dispersion can be measured by the suspension behaviour of a UP dispersion diluted to a low AEROSIL® concentration (about 0.4%).

Picture 6 illustrates the situation after a period of 4 hours; the only change to the dispersion was the mixer blade's altered speed of rotation.

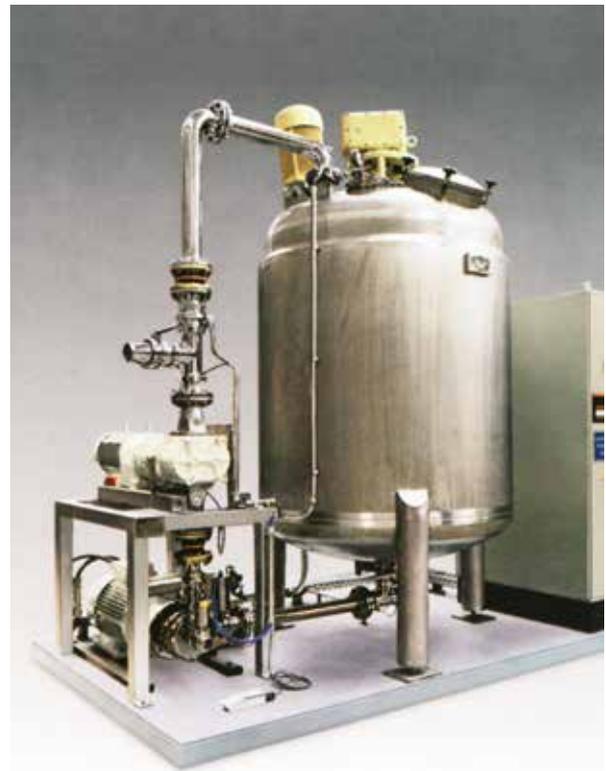


Picture 6 Influence of the circumferential speed of the dissolver (Cowles blade, dia. = 5 cm) on the sedimentation tendency of AEROSIL® 200 in a polyester resin

Further tests have also shown that the temperature of the UP resin during dispersing has a considerable effect on storage stability. The higher the temperature during dispersion, the greater the tendency of the fumed silica to form a sediment in the laminating resin. This effect can be explained by the fact that the viscosities of the UP resins decrease because of the higher temperatures and that the shearing forces during dispersion are therefore also lower.

3.5 Dispersion of AEROSIL® Fumed Silica with the Ystral Conti TDS-4 Mixer

Picture 7 shows the Ystral Conti TDS-4 mixer (manufacturer: Ystral GmbH, 79282 Ballrechten-Dottingen, Germany). This continuous dispersion technology allows silica to be incorporated into the UP resin with virtually no dust formation. The experiments carried out involved incorporating and dispersing AEROSIL® 200 (tapped density 50 g/l) in 200 kg of the styrene-free UP resin Palatal M 671-01 V in various concentrations from 1% to 7% with the standard-shear and high-shear device.



Picture 7 Ystral Conti TDS-4 mixer

The test conditions and results are summarised in **Table 1**.

The test results show that after only 74 seconds dispersing time with the Conti-TDS 4, a grind reading of 43 µm is achieved in the polyester resin thickened with 1% AEROSIL® 200 and a viscosity of 420 mPa·s is reached (sample 3).

Table 1 Dispersion of AEROSIL® 200 (tamped density 50 g/l) in 200kg Palatal M 671-01V

Sample	Head	AEROSIL® conc. in %	Time in sec.	Temp. in °C	Grind in µm	Viscosity in mPa · s
1	Standard	1	14	26.7	59	460
2	Standard	Post-dispersed	30	24.7	48	480
3	High-shear	Post-dispersed	30	26.9	43	420
4	High-shear	2.5	26	28.7	100	740
5	High-shear	Post-dispersed	30	31.9	73	740
6	High-shear	Post-dispersed	30	33.8	59	740
7	High-shear	Post-dispersed	30	36.0	59	740
8	High-shear	4	31	37.0	118	1600
9	High-shear	Post-dispersed	30	39.5	78	1500
10	High-shear	Post-dispersed	90	44.8	65	1400
11	High-shear	Post-dispersed	120	51.3	60	1360
12	High-shear	5.5	33	48.9	-	-
13	High-shear	7.0	93	-	-	-
14	Post-dispersed	Post-dispersed	240	63.9	70	21600
15	-	Palatal M 67 1-0 1 V	-	-	-	360

Comparative tests on the same sample with the laboratory dissolver (5 min. dispersion time, 8 m/s circumferential speed) revealed a somewhat poorer grind reading of approx. 60 µm, and also a slightly higher viscosity of 680 mPa·s on account of the lower shear forces.

Picture 8 shows the mixing container with 7% AEROSIL® 200 in Palatal M 671-01 V. The UP resin, which had already assumed a paste-like consistency because of the high silica concentration, could still be circulated without any problems.



Picture 8 Mixing of 7% AEROSIL® 200 in Palatal M 671-01V (tamped density of 50 g/l)

4 Vinyl Ester Resins

The UP resins Palatal P 4,-P 5,-A 410 and the styrene-free test product Palatal M 671-01 V have been used for tests to date.

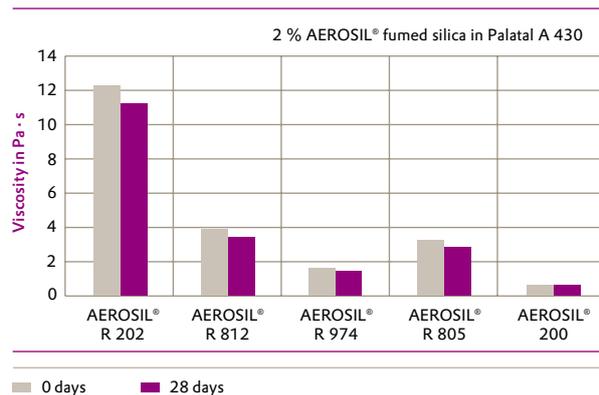
In contrast to conventional UP resins, the thickening of vinyl ester resins with AEROSIL® 200 presents problems, since the hydrophilic AEROSIL® aggregates

are wetted to a greater degree as a result of the larger number of hydroxyl groups in vinyl ester resins, and the thickening and thixotropic effect is therefore too low [12]. The hydrophobic silica AEROSIL® R 202 solves this problem, as illustrated by the two different vinyl ester resins Palatal® A 430 (DSM Resins, Zwolle, Netherlands) and DERAKANE® 411 -45 (Dow Deutschland Inc., 77836 Rheinmunster, Germany) (**Figures 20** and **21**).

Figure 20 Comparison of the thickening effect and storage stability of various AEROSIL® grades in a vinyl ester resin (40% styrene content).



Figure 21 Comparison of the thickening effect and storage stability of various AEROSIL® grades in a vinyl ester resin (45% styrene content).



5 Formula Questions

5.1 Laminating Resins and Gelcoats

For thickening laminating resins, concentrations of between 0.7 and 1.3% AEROSIL® fumed silica are typically used. Gelcoats are normally always thickened and generally contain between 2.0 and 2.5% fumed silica, or in special cases up to 4.0%. The exact percentage of silica depends on the desired rheological properties and the composition of the laminating resins and gelcoats.

Gelcoats can also contain pigments, and possibly fillers such as talc, dispersing agents and deaeration additives, and are diluted to a monomer content of 30–50% with styrene and/or other monomers, depending on rheological and regional regulatory requirements [14].

Simple guideline formulae for laminating resins and gelcoats are given in Section 6.

Before processing, accelerator solution (e.g. approx. 1% cobalt octoate, potassium octoate or dimethyl aniline solution) and peroxide solution (e.g. approx. 2% methyl ethyl ketone peroxide solution) are added to the laminating resin and gelcoat, according to the desired reactivity. Pre-accelerated systems are likewise commercially available.

5.2 Influence of Formula Ingredients

The choice of medium in which to disperse the AEROSIL® 200 is of considerable importance for the rheological properties and storage stability of the thickened UP resins. In order to demonstrate the influence of the dispersing sequence by way of an example, the following variations in the incorporation of AEROSIL® fumed silica were tested:

- 1 UP resin + AEROSIL® 200 (0.5%) dispersed with dissolver; then styrene added.
- 2 UP resin + styrene + AEROSIL® 200 (0.5%) dispersed with dissolver.
- 3 Styrene + AEROSIL® 200 prepared in mixer (5% cone.), stored overnight; then UP resin added with dissolver.
- 4 Styrene + AEROSIL® 200 dispersed for 6 h in a ball mill (5% conc.); then UP resin added with dissolver.

The results are summarised in **Table 2**. The results of the tests indicate that prior wetting with styrene impairs the processing properties of the dispersion. Prior dispersion of AEROSIL® 200 in styrene in the ball mill likewise does not improve this situation.

In the conditions selected here, the best results are obtained if AEROSIL® 200 is stirred directly into the UP resin before it has been diluted with styrene, which results in the silica being dispersed at the highest viscosity, i.e., with the most shear.

Table 2 Relationship between the rheological properties and storage stability of thickened UP resins and the order of addition of formula ingredients.

Incorporated according to method	Viscosity at 22 °C [mPa·s] at rpm		Thixotropic Index	Sedimentation in % of filling level, after number of days			
	20	25		1	7	21	28
1	2080	680	3.1	0	0	0	0
2	420	270	1.6	1	3	6	9
3	250	220	1.2	38	59	65	66
4	240	190	1.2	0	9	19	31

However, the disadvantage of this method is that the high shearing forces which occur during the dispersion process can cause the temperature to rise quite considerably and subsequent addition of styrene necessitates a two-stage process. In practice, fumed silica is therefore often incorporated directly into and dispersed in the pre-formulated UP resin according to method 2, and possibly just a small amount of styrene used to attain the desired rheological properties.

Experiments using pre-accelerated systems have indicated that the accelerators used likewise influence the rheological properties and storage stability of the thickened UP resins.

When an accelerator mixture was used (0.1% dimethyl aniline and 1% cobalt octoate solution) in a laminating resin (45% styrene, 0.5% AEROSIL® 200), the viscosity rose by approx. 10% compared with the non-accelerated laminating resin. In view of this higher viscosity, sedimentation of AEROSIL® 200 compared with the non-accelerated laminating resin was also lower. When only 1% cobalt octoate solution was used as the accelerator, the viscosity compared with the non-accelerated laminating resin fell by approx. 5%, and the sedimentation of AEROSIL® 200 was correspondingly more pronounced.

Further tests with 1% cobalt octoate and potassium octoate solutions in a gelcoat thickened with 2.5% AEROSIL® 200 revealed that the viscosity was approx. 60% higher when the potassium octoate solution was used than with the cobalt octoate solution. Variations in the sequence of addition of the accelerators (addition after dispersion or before the dispersion of AEROSIL® 200) revealed no significant difference in the rheological properties.

5.3 Improving the Spray Properties of Gelcoats

Figure 22 shows the flow curve of a commercially available gelcoat with approximately 4% silica and a specially formulated gelcoat with a simple guideline formula.

It is evident that the shape of the flow curve for the specially formulated gelcoat corresponds exactly to that of the commercially available gelcoat, but that it is located slightly further down due to the lower silica concentration.

The rheological properties of the spraying or spreading compounds, such as yield value and shear thinning flow properties, are important for processing gelcoats effectively (e.g. airless spraying). **Figure 23** shows how these rheological parameters can be specifically obtained by means of fumed silica and styrene.

Gelcoat B contains insufficient or unsuitable thixotropic agents; it exhibits a very low yield value and limited shear thinning flow properties. When the compound is sprayed, fan-like stripes occur and the compound runs down sloping surfaces. The addition of 0.9% AEROSIL® 300 and 10% styrene to gelcoat B ("gelcoat C") and 1.4% AEROSIL® 300 and 20% styrene to gelcoat B ("gelcoat D") raised the yield value and shear thinning (pseudoplasticity) of the gelcoats. Consequently, it can be sprayed more effectively and does not run down sloping or perpendicular surfaces when the coating thickness is within the usual range of approx. 400 to 1000 µm.

These measurements confirm in principle the conclusion reached in Section 3.3.3 that the Thixotropic Index, strictly speaking only a measure of shear thinning, rises when the styrene concentration in the UP resin is increased.

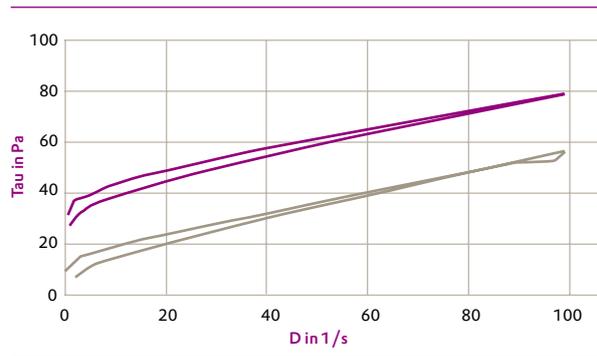
These measurements confirm in principle the conclusion reached in Section 3.3.3 that the Thixotropic Index, strictly speaking only a measure of shear thinning, rises when the styrene concentration in the UP resin is increased.

5.4 Influence of Additives

It has long been known that adding a small amount of a bifunctional liquid additive significantly enhances the thickening behaviour of fumed silicas [12].

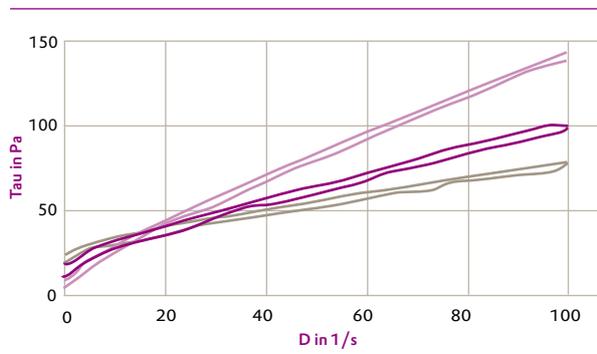
Figure 24 shows that in a UP resin thickened with 1.5% AEROSIL® 200, the viscosity can be significantly boosted by adding only 1% ethylene glycol, polyethylene glycol 200 or polyethylene glycol 600.

Figure 22 Comparison of the rheological properties of a commercially available and a specially formulated gelcoat.



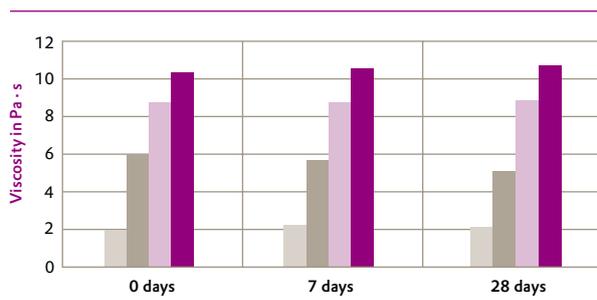
— Gelcoat A — Adjusted formulated Gelcoat with AEROSIL® 200
 79.0 parts Palatal A 410-01
 2.2 parts AEROSIL® 200
 20.0 parts pigment paste 50% (TiO₂,RBK 2 by Kronos in Palatal A 410)
 23.0 parts styrene
 0.6 parts BYK-A 555

Figure 23 Improvement the rheological properties of a gelcoat



— Gelcoat B — Gelcoat C — Gelcoat D

Figure 24 Influence of various additives on the thickening effect of fumed silicas.



■ 1.5 % AEROSIL® 200
 ■ 1.5 % AEROSIL® 200 + 1 % ethylene glycol
 ■ 1.5 % AEROSIL® 200 + 1 % polyethylene glycol 200
 ■ 1.5 % AEROSIL® 200 + 1 % polyethylene glycol 600

6 Guideline Formulas

6.1 Guideline Formula

Gelcoat, Low-viscosity and white in Colour, for spraying.

		Parts	
		by weight	% by weight
Palatal A 41 0-01	(7.1)	79.0	62.0
AEROSIL® 200	(7.2)	2.2	1.7
Pigment paste 50% (TiO ₂ (RBK-2) in Palatal A 41 0-01	(7.3)	20.0	15.6
Styrene		23.0	18.0
Deaeration additive BYK-A 555	(7.4)	0.6	0.5
Methyl ethyl ketone peroxide solution	(7.5)	2.0	1.5
Cobalt octoate solution	(7.6)	1.0	0.8

6.2 Guideline Formula

Laminating Resin

		Parts	
		% by weight	
Palatal P 4	(7.1)	100	
AEROSIL® 200	(7.2)	1	
Dispersing agent Tween 20	(7.7)	0.1	
Methyl ethyl ketone peroxide solution	(7.5)	1	
Cobalt octoate solution	(7.6)	0.5	

6.3 Guideline Formula

Laminating Resin, with Reduced Styrene Emissions

		Parts	
		% by weight	
Palatal P 4	(7.1)	100	
AEROSIL® 200	(7.2)	1	
Dispersion agent Tween 20	(7.7)	0.1	
Paraffin solution (5%) in styrene		5	
Methyl ethyl ketone peroxide solution	(7.5)	1	
Cobalt octoate solution	(7.6)	0.5	

6.4 Guideline Formula

Laminating Resin, on Vinyl Ester Resin Basis

		Parts	
		% by weight	
Derakane 411-45	(7.8)	100	
AEROSIL® R 202	(7.2)	1–1.5	
BYKA515	(7.4)	0.1–0.2	
Methyl ethyl ketone peroxide solution	(7.5)	2	
Cobalt naphthenate solution	(7.6)	0.4	
Dimethyl aniline solution		0.4	
Dimethyl aniline solution		0.4	

7 List of Suppliers

- 7.1 DSM Resins, 8000 AP Zwolle, Netherlands
- 7.2 Evonik Industries AG, 63457 Hanau, Germany
- 7.3 Kronos Titan GmbH, 51373 Leverkusen, Germany
- 7.4 BYK-Chemie GmbH, 46463 Wesel, Germany
- 7.5 Akzo Nobel Chemical, 46446 Emmerich, Germany
- 7.6 Peroxidchemie GmbH, 82049 Pullach, Germany
- 7.7 Deutsche ICI GmbH, 45127 Essen, Germany
- 7.8 Dow Deutschland Inc., 77836 Rheinmünster, Germany

8 Test Procedure for Section 3.3

An ultrasonic dispersion device, type „ULTRASON® HOMOGENISER“, Model B, (manufacturer: Branson Ultrasonics Ltd, Hayes, Middlesex, England) was available. For the setting of up every test, with the gear-type pump running at a constant speed, the „acoustic intensity“ was adjusted to the resonance using the two micrometer screws which are likewise visible on the sonolator.

The orifice, which is very important for the functioning of a sonolator, can be exchanged. The following orifices were used in the test series:

Test series 1: Orifice S2 („standard“)

Test series 2: Orifice IM („undersized“)

Under the pressure of approx. 2.8 to 3.5 N/mm² generated by a gear-type pump, a throughput of about 20 to 30 l/min was achieved depending on the composition of the resin. The resin was not circulated (i. e. processed in a single container), but transferred between two containers to ensure that every volume element of the resin genuinely had to pass through the sonolator. The first container was for incorporating the silica with the aid of a three-vane propeller mixer with conical guide ring and, in the case of several passages, to collect the resin after being pumped through the sonolator to the second container.

Two Cowles dissolvers were available. The following mixing conditions were adopted for the test series:

Test series 1: Mixer plate diameter 7 cm circumferential speed 10 m/s container diameter 16 cm Test amount 2 kg

Test series 2: Mixer plate diameter 10 cm circumferential speed 15 m/s container diameter 24 cm Test amount 8–9.5 kg

A mixture of Palatal®, types P4 and P5 was used for the tests. The tests consisted of two series, with different styrene contents selected in each case:

Test series 1: Styrene content (constant) approx. 41 % by adding 10 parts styrene to 100 parts of the 1 : 1 resin mixture using 35: % styrene.

Test series 2: Styrene content (variable) of 35 %, 40 % and 45 % by adding appropriate amounts of styrene to the 1 : 1 resin mixture using 35 % styrene.

9 Product Safety

All synthetic silicas supplied by Evonik are manufactured either by the precipitation method in an aqueous solution or by flame hydrolysis and are characterised by powder diffractometer pictures. The absence of sharp peaks reveals that the synthetically manufactured silicas have an entirely amorphous form.

When handling amorphous silica, intake by inhalation is of particular significance: if for instance the TLV of 4 mg/m³ is reached when handling amorphous precipitation silicas, no damage to the health has yet been observed. If the TLV is exceeded, it can constitute a mechanical burden to the upper respiratory tract in the same way as other dusts, and protracted exposure can then lead to functional and organic damage to the respiratory passages.

No signs of irritation have yet been observed following brief contact with the skin or mucous membranes. Neither have any sensitisation symptoms been detected in persons who have worked with synthetic silicas over many years. However, the explicit property of adsorbing water and oil can lead to dry, scaly skin after protracted or repeated contact.

Regular medical checks carried out on workers who have been employed for many years at production plants for synthetic amorphous silicas have not detected a single case of silicosis [18-20].

Experiments carried out on animals have largely confirmed the findings for humans on the handling of Evonik silicas. The one-off application of silicas to intact or scarified areas of skin on rats did not produce symptoms of irritation to the skin. As a general rule, no harmful effect was observed. Even the introduction of precipitated silicas into the tear sacs of rabbits did not cause any damage. The acute oral toxicity value, LD₅₀, for synthetic silicas is greater than 10,000 mg/kg for the tests carried out on rats.

The silicas referred to here are supplied in paper sacks. Transport by silo vehicle is also possible. Extensive handling tests have been carried out on synthetic silicas in order to devise practical means of avoiding the formation of dust. The results of these tests are summarised in issue number 28 of the Evonik pigments publication series. These results cover:

- manual or fully-automated debagging,
- internal transport by means of suction or pressure,
- automatic weighing or batching,
- dust-free addition to mixing and dispersion equipment.

According to the chemicals legislation of the European Union, amorphous synthetic silicas are not classified as hazardous substances. They are also not classified as hazardous goods for purposes of transport.

For further information on product safety, see the corresponding safety data sheets and issues 65 and 76 of the pigments publication series.

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11 Physico-Chemical Data of AEROSIL® Fumed Silica

Characteristic-physical and chemical data of AEROSIL® fumed silica

Test methods	AEROSIL®											
	OX 50	90	130	150	200	300	380	R 202	R 805	R 812	R 974	
Behaviour towards water	hydrophilic							hydrophobic				
Appearance	- fluffy white powder -											
BET surface area <i>BET</i> ¹⁾	m ² /g	50±15	90±15	130±25	150±15	200±25	300±30	380±30	100±20	150±25	260±30	170±20
Average primary particle size	nm	40	20	16	14	12	7	7	14	12	7	12
Tapped density ²⁾ <i>approx. value</i>	g/L	130	80	50	50	50	50	50	50	50	50	50
Densified material <i>(suffix „VV“)</i>	g/l	–	120 ¹⁶⁾	120 ¹⁶⁾	120 ¹⁶⁾	120 ¹⁶⁾	120 ¹⁶⁾	120 ¹⁶⁾	–	–	–	90 ¹⁷⁾
Densified material <i>(suffix „VV“)</i>	g/l	–	–	–	50/75	50/75/ 120 ¹⁶⁾	50/75/ 120 ¹⁶⁾	–	60/90 ¹⁷⁾	90 ¹⁷⁾	–	–
Loss on drying ³⁾ <i>(2 h at 105 °C) when leaving the plant</i>	wt. %	≤1.5	≤1.0	≤1.5	≤0.5 ⁹⁾	≤1.5	≤1.5	≤2.0	≤0.5	≤0.5	≤0.5	≤0.5
Loss on ignition ^{4) 7)} <i>(2 h at 1000 °C)</i>	wt. %	≤1.0	≤1.0	≤1.0	≤1.0	≤1.0	≤2.0	≤2.5	4–6 ¹³⁾	5–7 ¹⁴⁾	1.0–2.5 ¹⁵⁾	≤2.0
pH-value ⁵⁾ <i>(4% „aqueous dispersion“)</i>		3.8–4.8	3.7–4.7	3.7–4.7	3.7–4.7	3.7–4.7	3.7–4.7	3.7–4.7	3.5–5.0	4.5–6.5	2.0–3.0	0.7–1.3
C-content	wt. %	–	–	–	–	–	–	–	4–6 ¹⁰⁾	3.5–5.5 ¹⁰⁾	5.5–7.5 ¹⁰⁾	3.7–4.7 ¹⁰⁾
SiO ₂ ⁸⁾	wt. %	≥99.8	≥99.8	≥99.8	≥99.8	≥99.8	≥99.8	≥99.8	≥99.8	≥99.8	≥99.8	≥99.8
Al ₂ O ₃ ⁸⁾	wt. %	≤0.080	≤0.050	≤0.050	≤0.050	≤0.050	≤0.050	≤0.050	≤0.050	≤0.050	≤0.050	≤0.050
Fe ₂ O ₃ ⁸⁾	wt. %	≤0.010	≤0.003	≤0.003	≤0.003	≤0.003	≤0.003	≤0.003	≤0.010	≤0.010	≤0.010	≤0.010
TiO ₂ ⁸⁾	wt. %	≤0.030	≤0.030	≤0.030	≤0.030	≤0.030	≤0.030	≤0.030	≤0.030	≤0.030	≤0.030	≤0.030
HCl ^{8) 11)}	wt. %	≤0.025	≤0.025	≤0.025	≤0.025	≤0.025	≤0.025	≤0.025	≤0.025	≤0.025	≤0.025	≤0.1
Sieve residue ⁶⁾ <i>(by Mocker, 45 µm)</i>	wt. %	≤0.2	≤0.05	≤0.05	≤0.05	≤0.05	≤0.05	≤0.05	–	–	–	–
Unit weight ¹¹⁾ <i>(netto)</i>	kg	10	10	10	10	10	10	10	10			10

The data have no binding force.

¹⁾ acc. to DIN 661 31

²⁾ acc. to DIN EN ISO 787/11, JIS K 5101/18

³⁾ acc. to DIN EN ISO 787/2, ASTM D 280, JIS K 5101/21

⁴⁾ acc. to DIN EN ISO 3262-20, ASTM D 1208, JIS K 5101/23

⁵⁾ acc. to DIN EN ISO 787/9, ASTM D 1208, JIS K 5101/24

⁶⁾ acc. to DIN EN ISO 787/18, JIS K 5101/20

⁷⁾ dried for 2 hours at 105 °C

⁸⁾ ignited for 2 hours at 1000 °C

⁹⁾ special moisture-protective packaging

¹⁰⁾ in water: acetone or methanol = 1:1

¹¹⁾ HCl-content is a part of ignition loss

¹²⁾ contains approx. 0.7–1.3 % chemically bonded carbon

¹³⁾ contains approx. 3.5–5.0 % chemically bonded carbon

¹⁴⁾ contains approx. 4.5–6.5 % chemically bonded carbon

¹⁵⁾ contains approx. 2.0–3.0 % chemically bonded carbon

¹⁶⁾ packaging of densified material: 20 kg

¹⁷⁾ packaging of densified material: 15 kg

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