

AEROSIL[®] and SIPERNAT[®] Silica: Versatile Raw Materials for Personal Care Formulations

Technical Information 1251

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Introduction

Evonik Industries is a leading global manufacturer of synthetic amorphous silica (chemically SiO₂) and offers both flame-process (fumed) and wet-process (precipitated) types. Our AEROSIL® fumed silica (available as untreated hydrophilic and surfacetreated hydrophobic "R" types) and SIPERNAT® precipitated silica are well-established problem-solvers in many industries such as food, pharmaceuticals, detergents and coatings. Most personal care manufacturers are familiar with synthetic amorphous silica only as a key raw material for toothpaste. But silica is a very versatile raw material for many other personal care products and we hope this brochure will not only show you the broad application range of our products but perhaps also give you ideas for creating new products or improving existing ones.

I. Silica for Liquid and Semi-solid Systems

Improving Rheology with Silica

AEROSIL® fumed silica can be used as an efficient and reliable rheological aid. In a cosmetic formulation AEROSIL® will create a three dimensional network (Fig. 1) from agglomerated silica aggregates. Since this network can easily be broken down if external shear forces are applied, the resulting mixtures exhibit a thixotropic (non-Newtonian) behavior. The shear thinning effect renders a gel that is easily spreadable during application to the skin without any dripping.

The correct AEROSIL® type to use is dependent on the matrix as well as the viscosity and rheology that needs to be achieved. As a general rule of thumb hydrophilic AEROSIL® grades perform better in non-polar oils (e.g. mineral oil, dimethicone, cyclomethicone). In non-polar oils thickening efficiency is related to the specific surface area (BET) of the silica; high specific surface mostly giving higher viscosity (Fig 2).



Figure 1 Mechanism of thickening of AEROSIL® fumed silica.







For more polar synthetic oils and vegetable oils, hydrophobic AEROSIL® types often are the better choice. AEROSIL® R 974, AEROSIL® R 805 and AEROSIL® R 202 have proven to be especially useful in these systems.

When looking at a cosmetic formulation it is necessary to look at the formulation as a whole since active ingredients, emollients, etc. may completely alter the thickening efficiency behavior of AEROSIL[®]. The optimum product needs to be determined experimentally, taking other parameters like skin feel and gel stability into account.

Table 1 Viscosity achieved with different AEROSIL® products in selected cosmetic oils²

INCI	Product**	Appearance	Viscosity (mPa s)	Concentration (% w/w)
Mineral Oil or Paraffinum Liquidum	AEROSIL [®] 200	clear	Арргох. 30,000	3
Dimethicone	AEROSIL [®] 200	opaque	Арргох. 11,000	2
Caprylic/Capric Triglyceride	AEROSIL® R 202	clear	Арргох. 7,000	6
C12-C15 Alkyl Benzoate	AEROSIL® R 202	opaque	Арргох. 8,500	6
PEG-8	AEROSIL [®] R 805	clear	Арргох. 60,000	5
Helianthus annuus or Helianthus annuus (Sunflower) Seed Oil	AEROSIL® R 202	hazy	Арргох. 20,000	6
Olea Europaea or Olea Europaea (Fruit)	AEROSIL® R 202	opaque	Арргох. 20,000	6
	INCI Mineral Oil or Paraffinum Liquidum Dimethicone Caprylic/Capric Triglyceride C12-C15 Alkyl Benzoate PEG-8 Helianthus annuus or Helianthus an	INCIProduct**Mineral Oil or Paraffinum LiquidumAEROSIL° 200DimethiconeAEROSIL° 200Caprylic/Capric TriglycerideAEROSIL° R 202C12-C15 Alkyl BenzoateAEROSIL° R 202PEG-8AEROSIL° R 805Helianthus annuus or Helianthus annuus (Sunflower) Seed OilAEROSIL° R 202Olea Europaea or Olea Europaea (Fruit)AEROSIL° R 202	INCIProduct**AppearanceMineral Oil or Paraffinum LiquidumAEROSIL° 200clearDimethiconeAEROSIL° 200opaqueCaprylic/Capric TriglycerideAEROSIL° 202clearC12-C15 Alkyl BenzoateAEROSIL° R 202opaquePEG-8AEROSIL° R 805clearHelianthus annuus or Helianthus annuus (Sunflower) Seed OilAEROSIL° R 202opaqueOlea Europaea or Olea Europaea (Fruit)AEROSIL° R 202opaque	INCIProduct**AppearanceViscosity (mPa s)Mineral Oil or Paraffinum LiquidumAEROSIL° 200clearApprox. 30,000DimethiconeAEROSIL° 200opaqueApprox. 11,000Caprylic/Capric TriglycerideAEROSIL° 202clearApprox. 7,000C12-C15 Alkyl BenzoateAEROSIL° R 202opaqueApprox. 8,500PEG-8AEROSIL° R 805clearApprox. 60,000Helianthus annuus or Helianthus annuus (Sunflower) Seed OilAEROSIL° R 202hazyOlea Europaea or Olea Europaea (Fruit)AEROSIL° R 202opaqueApprox. 20,000

* Evonik Industries AG, Business Unit Consumer Specialties

** Evonik Industries AG, Business Unit Inorganic Materials

For highly polar oils and alcohols AEROSIL® R 202 and AEROSIL® R 805 have been proven to be exceptionally useful to control rheology. AEROSIL® fumed silica is not an effective thickener for aqueous solutions.



Figure 3 Flow diagram of polyethylene glycol 400 mixtures with AEROSIL® 200 and AEROSIL® R 805.3

→ 2% w/w AEROSIL® R 805

_____ 3 % w/w AEROSIL® R 805

The refractive index of silica is 1.46 to 1.45 depending on the silica surface chemistry. The closer the silica is to the refractive index of the non-polar base, the greater the clarity can be achieved. If the refractive indices match, the formulation can be completely clear. (see Table 2)

 ² Dispersed 10 minutes using a dissolver with tip speed 15.7 m/s (5 cm blade at 6000 rpm); viscosity measured using Brookfield, 5 rpm.
 ³ Dispersed 10 minutes using a dissolver with tip speed 15.7 m/s (5 cm blade at 6000 rpm);

viscosity measured using a Physica model MCR 300 with cylinder CC27

Table 2

Cosmetic oils that have refractive indices close to the refractive index of silica.

Cosmetic oil INCI	Refractive index
Squalene	1.45 – 1.46
Mineral oil	1.45 – 1.457
Octyldodecanol	1.451 – 1.455
Jojoba oil	1.465
Cottonseed oil	1.45 – 1.46
Phenyl trimethicone	1.45 – 1.46
Cetearyl isononanoate	1.448 - 1.450
Decyl oleate	1.45 – 1.46
Isostearyl isostearate	1.459
	,

The AEROSIL[®] concentration required for thickening is dependent on the desired viscosity as well as on the matrix formulation. Usually concentrations of 0.5 to 6 weight-% are sufficient in the formulation. Higher concentrations of AEROSIL[®] will lead to higher viscosities. In some rare cases a small change of the added AEROSIL[®] quantity may lead to a strong difference in the viscosity of the mixture. In these cases it might be worthwhile to turn to another AEROSIL[®] type with a lower concentration sensitivity regarding the rheological properties.

A prerequisite for taking full advantage of the viscosity increasing effect of AEROSIL[®] is a proper dispersion of the silica in the matrix. As delivered, the products consist of agglomerates that need to be broken up so that the aggregate network can form in the oil. To properly deagglomerate the AEROSIL[®] products high shear mixing equipment like dissolvers or rotor stator mixers with a circumferential speed of at least 15 m/s is recommended. The quality of the dispersion can be judged by a simple grind gauge (Fig. 4).

Various additives can boost the viscosity imparted by silica. Polysorbitol, glycerin and PEGs interact with the silica allowing for even more rheology modification. Each synergist has a different effect on silica rheology.

Polymeric thickeners work through chain entanglement. This type of thickening can lead to specific issues such as a stringy feel in formulations. They are also heat sensitive and can fall out of suspension at elevated temperatures. Repeated shear causes polymers to align and detangle therefore losing their structural integrity. Silica rheological aids do not have these issues.

Details on how to incorporate AEROSIL® into liquid systems can be found in TI 1279 Successful Use of AEROSIL® Fumed Silica in Liquid Systems.





Figure 4 Grind gauge test for dispersion quality

Improving and stabilizing suspensions

Many cosmetic products rely on the homogeneous dispersion of pigments or other insoluble substances in the formulation. Decorative cosmetics products like nail polish and lip gloss are typical examples where pigments need to be stabilized in a primarily organic matrix. But also in AP-deo creams and roll-ons a homogeneous distribution of the active AP salts needs to be maintained throughout the service life of the product.

AEROSIL[®] fumed silica can help to improve and stabilize such suspensions. Primarily hydrophobic AEROSIL[®] grades are used for this application. The AEROSIL[®] network which builds in the formulation will incorporate the particles and stabilize them against settling (Fig. 5)



Figure 5

Schematic representation of pigment particles stabilized in three dimensional network of AEROSIL® fumed silica.

An example of a lip gloss formulation in which effect pigments could be stabilized by the addition of AEROSIL[®] is shown in Fig. 6. Another example for an AP deo formulation is shown later in this brochure.



Figure 6

Lip gloss formulation with effect pigment.⁴ Left: no silica. Right: stabilized with 5 wt.% AEROSIL® 200. On the left the sedimentation of the pigments is clearly visible

AEROSIL[®] grades can provide suspension without a thickening effect in certain formulations. The AEROSIL[®] type and concentration is strongly dependent on the ingredients used. AEROSIL[®] R 202, AEROSIL[®] R 805, or AEROSIL[®] R 974 in a concentration of 3 to 5 % w/w is a good starting point for further formulation development.

Modifying Skin Feel

Silica can modify skin feel in two ways: adsorption of oil and increase of viscosity rebuild time.

Due to their high surface area AEROSIL[®] and SIPERNAT[®] silica can adsorb excess oil. In the case of SIPERNAT[®] products the excess oil can also be effectively bound in the internal pores of the particles. By adding the correct level of silica a formulator can reduce the greasy feel some types of oils can cause. Using 0.5–1.5 % w/w can improve overly oily formulas.

Depending on the surface treatment the degree of silica hydrophobicity can vary greatly. Methanol wettability is one way to classify the degree of hydrophobicity (Fig. 7). The more methanol required to reach 100% sediment volume (or the further to the right on the graph), the more hydrophobic the silica. For non-polar systems the more hydrophobic the silica is, the longer it takes to rebuild viscosity. By increasing the viscosity rebuild time a formulator can decrease the 'draggy' feel that hydrophilic silica imparts.

⁴ Dispersed 10 minutes using a dissolver with tip speed 15.7 m/s (5 cm blade at 6000 rpm); viscosity measured using a Physica model MCR 300 with plate/cone CP 50-1, angle 1°, diameter 50 mm. Figure 7 Relative hydrophobicity of various AEROSIL[®] products as related to their methanol wettability. The higher the methanol content (vol. %, x-axis), the more hydrophobic the product.



Improving water resistance

Hydrophobic AEROSIL[®] types can improve water resistance. We recommend the most hydrophobic AEROSIL[®] products to obtain this effect. By adding 0.5 to 5 w/w % AEROSIL[®] R 812, AEROSIL[®] R 812 S or AEROSIL[®] 202 to a formulation such as a sunscreen or "long-wearing" make-up product the water (sweat) resistance can improve.

Modifying Payoff for Stick Applications

Acting as a rheology aid silica will thicken the oil phase of a wax/oil stick formulation. The increase in viscosity that occurs modifies the stick consistency. The stick consistency can go from soft and malleable to firm depending on the grade and silica amount used. AEROSIL® 200, AEROSIL® R 812 S or AEROSIL® R 202 may be used at 0.5-3.0% by weight to modify payoff.⁵

Payoff is improved in color cosmetic stick formulations due to the silica improving pigment dispersion and preventing pigment re-agglomeration. Silica increases the viscosity of the formulation and higher viscosity results in additional shear which improves pigment dispersion by fully de-agglomerating the pigments. Once the formulation is no longer being sheared the silica forms a three-dimensional network that inhibits pigments from re-agglomerating and settling.

Emulsions

Cold Processing

Hydrophobic AEROSIL[®] types are especially suitable for increasing the viscosity of w/o (water-in-oil) emulsions and producing pseudoplastic flow behavior. AEROSIL[®] fumed silica can also be used for emulsions more readily than other viscosity adjusters. While waxes must be melted, AEROSIL[®] can be used at room temperature. Timeand energy-intensive heating of the phases and cooling of the emulsion are avoided. AEROSIL[®] fumed silica can be dispersed with the usual equipment used in the cosmetic industry. Another advantage of silica-thickened formulations is their low sensitivity to temperature, electrolytes and pH. Emulsion stability can be improved with as little as 0.5-2.0% w/w of AEROSIL[®] R 972, AEROSIL[®] R 974, AEROSIL[®] R 812 or AEROSIL[®] R 812 S.

II. Silica for Powdered Systems

Free Flow

Powder make-up products for the face and eyes often contain irregularly shaped pigments or other ingredients that can cause poor flow. Poor flow can lead to formulations that are not homogenous. When particles are close enough they will be attracted to one another due to van der Waals forces. Silica acts as a particle spacer, preventing van der Waals forces from causing poor flow.

Poor flow may also be caused by irregularly shaped particles. When coated with silica plate-like or irregularlyshaped cosmetic particles are able to slide past each other (Fig. 9). Small amounts (0.5–2.0% w/w) of SIPERNAT* 22 S, AEROSIL* 200 or AEROSIL* R 972 can improve powder flow. The choice of flow aid depends on the particle size of the original powder and the mixing conditions to be used. As a rule of thumb SIPERNAT* grades work better for powders with large particles while AEROSIL* products are to be preferred for smaller particle sizes. The hydrophobic grades are preferentially used in low shear mixers. Details on mixing conditions can be found in our Technical Information 1213 *Silica as Flow Agent and Carrier Substance: Recommended Mixing Procedures for Powders and Granulates*.

Anti-Caking

Hygroscopic materials, such as some of the minerals used in loose powder make up, can attract and bind water causing the powdered cosmetic to cake or become cohesive. Silica can prevent this storage problem by preferentially absorbing water before it can affect the makeup. If the water activity of the silica is higher than the hygroscopic products the silica can still improve the storage stability by spacing the powdered cosmetics far enough apart so that liquid bridging can no longer occur. Products such as SIPERNAT[®] 22 LS, SIPERNAT[®] 50 S or SIPERNAT[®] 500 LS are good choices to produce anti-caking effects. The choice of product depends on the particle size of the original powder and the amount of liquid to be absorbed. The finer the particles of the original powder the more likely it is to successfully use an anti-caking additive of also small particle size.



Figure 8

Scanning electron micrographs of table salt untreated (left) and treated (right) with SIPERNAT[®] precipitated silica showing the reduced number of contact points between the crystals that lead to improved flowability and reduced caking.

Carrying Liquids

Actives that are viscous and difficult to dose can be absorbed, or carried, in the pores of SIPERNAT[®] silica. Carried liquids are absorbed through capillary action and are released through either diffusion or desorption. When the loaded silica contacts water the water is attracted to surface hydroxyl groups which causes the carried liquid to be diffused from the silica.

Due to particle characteristics such as surface area and pore structure each silica grade has a different carrying capacity and absorption/desorption rates (Table 2).

Table 3

Surface area and absorption data of various silica types.

Product	Specific Surface area (BET), m²/g	DOA absorption, g/100g
SIPERNAT [®] 22	190	235
SIPERNAT [®] 22 LS	175	235
SIPERNAT [®] 50	475	300
SIPERNAT [®] 50 S	475	300
SIPERNAT [®] 500 LS	475	285
AEROPERL [®] 300/30	300	235

The absorptive capacity of silica is the basis for the concept of "dry binder", in which binder oils can be turned into a non-cohesive powder for easier incorporation into powder mixtures. Please see the section "Dry Binder" later in this publication for more information.

Mattifying

Adding silica to a formulation can lead to a matting effect by providing an irregular surface on the skin. Unlike smooth surfaces, irregular surfaces refract light away from the source and reduce the gloss (Fig. 9).

Gloss can also be reduced when absorptive silica is used. Due to its carrying capacity silica can bind oils that exude onto the skin which typically cause a glossy appearance.



Figure 9 Schematic diagram of the mattifying effect of silica.

III. Formulation Specific Effects

Improving Hair Care Products with Silica

Bleaching agents⁷

Hair bleaching powders generally contain peroxides such as potassium, sodium or ammonium peroxodisulfate. AEROSIL® fumed silica effectively prevents moistening and caking of peroxides, guaranteeing good free-flow properties and peroxide stability even after extended storage. For special requirements for example, to achieve high flowability of hair bleaching powders hydrophobic AEROSIL® types, such as AEROSIL® R 972 and AEROSIL® R 812 (S), can be used. Another advantage of AEROSIL® products is their high purity. They are almost free of any metals, especially iron, that can catalyze the decomposition of peroxides. In hair bleaching creams, AEROSIL® fumed silica can act as a thickener and stabilizer. AEROSIL® 200 is particularly suitable for hair bleaching agents and is typically used in a concentration between 2.0 and 4.0 % w/w.

Dyes⁷

In hair dyes, AEROSIL[®] fumed silica prevents a premature reaction and caking of powdered colorant components.

AEROSIL[®] 200 is particularly effective as a drying agent in a concentration of up to 4.0% w/w. Especially effective flow regulators are AEROSIL[®] R 972 and AEROSIL[®] R 812 (S). Dispersion dyes can also be stabilized with AEROSIL[®] fumed silica.

Hair rinses and styling products

In hair rinses, hydrophilic AEROSIL* types such as AEROSIL* 200 and AEROSIL* 300, in combination with quaternary ammonium compounds, have a positive effect on the volume of the hair. Typical concentrations used are in the range of 1.0-2.0% w/w, based on the total formulation.

Used in a concentration between 0.5 and 1.0% w/w (based on the finished product), AEROSIL[®] can help increase the volume of the hair. Figure 10 shows scanning electron micrographs of human hair without silica and after treatment with silica. The agglomerates adhere to the rough hair structure, and prevent the hairs from lying flat on one another, thus increasing volume.



Figure 10

Untreated human hair (left), and human hair treated directly with AEROSIL® 200 (right). The agglomerates are clearly visible on the rough hair surface.

⁷ K. Schrader, Hair Bleaching Products, Chapter 5.7 (Volume II) in Cosmetology – Theory and Practice, K. Schrader, A. Domsch, Eds., Verlag für chemische Industrie, Augsburg, 2005

AEROSIL® Fumed Silica for Lipstick

In lipsticks AEROSIL* fumed silica helps to improve the homogeneous distribution of the pigments. Fumed silica can increase the viscosity of a formulation. As the viscosity of a mixture increases more shear has to be applied when mixing. This increase in shear allows pigments to be properly dispersed by breaking down agglomerates that can form. Once allowed to rest, the silica rebuilds a three dimensional network within the oil/wax base of the lipstick preventing the settling or re-agglomeration of pigments.

On the lips, AEROSIL[®] fumed silica can prevent migration ("bleeding", "feathering") of the lipstick into the fine lines of the lips, considerably extending the wear time. This improvement is caused by the silica acting as a structure builder and because of the surface adsorption of oils. By adding rheology and adsorbing excess oils the lipstick can resist migration due to gravity or capillary action.

At higher concentrations, silica can reduce gloss and provide a matting effect for the lipstick.

Depending on the desired product properties, AEROSIL^{\circ} 200, AEROSIL^{\circ} R 972 or AEROSIL^{\circ} R 812 can be used in a concentration between 0.25 and 4.0% w/w. In special formulations, such as primer or lipstick foundations, up to 10% w/w AEROSIL^{\circ} R 972 can be used.

Phase	Ingredient	INCI name	% w/w
1	TEGOSOFT® Liquid*	Cetearyl Ethylhexanoate	9.48
	TEGOSOFT [®] SH*	Stearyl Heptanoate	0.50
	ABIL® Wax 2434*	Stearoxy Dimethicone	0.75
	ABIL® Wax 2440*	Behenoxy Dimethicone	0.75
	REWOPAL® PIB 1000*	Polyisobutene	5.00
	ANTARON V 220	PVP/Eicosene Copolymer	0.25
	Adeps Lanae Lite	Lanolin	31.20
	Crodamol ML	Myristyl Lactate	6.25
	Castor oil	Ricinus Communis	6.70
	Carnauba Wax 2442 L	Cera Carnauba	2.55
	Candelilla Wax 2039L	Candelilla Cera	7.55
	TeCe Ozokerit HH weiss	Ozokerite	3.70
	Paracera W 80	Ceresin	2.50
	A-C Copolymer 400	Ethylene/VA Copolymer	3.00
	Oxynex® K liquid	Ascorbyl Palmitate, Tocopherol, Ascorbic Acid., Tocopherol, PEG-8	0.02
	AEROSIL® R 812**	Silica Silylate	1.00
	COVAPATE BLANC W 9765	CI 77891 & Castor Oil	2.50
	COVAPATE ROSE W 4770	CI 45410 & Castor Oil	9.92
	COVAPATE ROSE W 3773	CI 15850 & Castor Oil	5.05
	COVAPATE BRUN W 8768	CI 77491/77492/77499 & Castor Oil	1.33
	Total		100.00

Guide Formulation: Smooth 'n' Even Lipstick

Heat all ingredients of the wax-oil-phase to approx. 85° C. Mix until homogeneous. Add AEROSIL[®] R 812 while homogenizing. Homogenize for a further five minutes. Add pigment dispersions and stir for at least 30 minutes. Pour into a lipstick mold and place the mold in a freezer at or below 0° C. After 30 minutes remove the mold from the freezer. Immediately remove sticks from the mold and place into lipstick cases.

* Evonik Industries AG, Business Unit Consumer Specialties

** Evonik Industries AG, Business Unit Inorganic Materials

Improving Lipstick Thermal Stability with SIPERNAT[®] Silica

At elevated temperatures wax/oil based products can deform, exude oil ("sweating") or be ruined completely. Addition of SIPERNAT[®] 500 LS or AEROSIL[®] fumed silica may be used to increase the structure of lipstick formulations and lip balm formulations that can limit and prevent both sweating and thermal distortion (Fig. 11).



Figure 11

Lipstick after high temperature storage (4 hours at 65 °C) Left: lipstick containing no silica has deformed due to temperature and has sweated out a large portion of oil. Center and right: lipsticks containing 2% and 4% w/w SIPERNAT* 500 LS (respectively) have retained their shape and oil.

Figure 12 shows how the width of the lipstick changes over time when stored at 60 °C. The original molds were 12 mm in diameter. The closer to 12 mm the stick remains when heated, the less thermal distortion occurred. Results show that once the weight percent of SIPERNAT[®] 500 LS exceeded 2% w/w the silica was able to sufficiently bind excess oil and prevent it from "sweating" out of the lipstick. When the concentration was increased to 3% w/w SIPERNAT[®] 500 LS, little to no thermal distortion was observed.

Figure 12

Relative thermal distortion of lipstick containing various concentrations of SIPERNAT $^{\circ}$ 500 LS (by weight)



In Figure 13 a texture analyzer equipped with a pin probe was used to measure the average hardness of the lipsticks produced with different amounts of SIPERNAT[®] 500 LS. The results (average of twelve replicates) show that at least 5% w/w SIPERNAT[®] 500 LS was required to create a significant change in texture. This allows the formulator to add weight percentages up to 4% without significantly changing the structure of the stick.





Lipstick formulation used for Figures 11 through 13.

Ingredient	INCI name	% w/w
Masterbatch		
TEGOSOFT [®] Liquid*	Cetearyl Ethylhexanoate	11.48
TEGOSOFT [®] SH*	Stearyl Heptanoate	0.50
ABIL [®] Wax 2434*	Stearoxy Dimethicone	0.75
ABIL [®] Wax 2440*	Behenoxy Dimethicone	0.75
REWOPAL® PIB 1000*	Polyisobutene	5.00
Antaron V 220	PVP/Eicosene Copolymer	0.25
TEGO [®] Alkanol 66*	Cetyl Alcohol	32.70
Isododecane	Isododecane	8.25
Castor Oil	Ricinus Communis	6.70
Carnauba Wax	Cera Carnauba	2.55
Candelilla Wax	Cera Candelilla	7.55
Ozokerite Wax White	Ozokerite	3.70
Preservative	Preservative	q. s.
Covapate Blanc W 9765	CI 77891, Castor Oil	2.50
Covapate Rubis W 4765	CI 15850, Castor oil	9.30
Covapate Orange W 2762	CI 15985, Castor oil	7.00
		100.00
Lipstick		
Master Batch		96.00-97.50
SIPERNAT [®] 500 LS**	Hydrated Silica	2.50-4.00

Heat all ingredients of the wax-oil phase to approx. 85 °C, mix until homogeneous. Add pigment dispersions and stir for at least 30 minutes. Refrigerate the mixture. This is the "Master Batch". Reheat the Master Batch to 85 °C and add SIPERNAT® 500 LS. Homogenize for five minutes, and then pour into a mould. Place the mould in a freezer at or below 0 °C. Remove the mould from the freezer after 30 minutes and immediately remove sticks from the mould and place into lipstick cases. * Evonik Industries AG, Business Unit Consumer Specialties

** Evonik Industries AG, Business Unit Inorganic Materials

Silica in Antiperspirants

There are three antiperspirant formulation types which differ regionally in preference: aerosol sprays, roll-ons and sticks. AEROSIL® fumed silica can be used as a suspending and anti-agglomeration agent to improve the performance of solid stick and roll-on formulations.

Antiperspirant sticks require uniform salinity throughout to ensure a quality product. Without the proper structure in the matrix the salt would settle to the top of the mold during cooling and storage resulting in an antiperspirant that works well at first, but declines in effectiveness over time. Adding the right AEROSIL® or SIPERNAT® silica can help improve homogeneous salt distribution by acting as a viscosity builder and suspending agent.

Figure 14 shows the percent difference of salinity between the top and bottom of an antiperspirant stick. Without silica the difference in salinity is 8%. The addition of silica such as SIPERNAT® 22 LS or AEROSIL® 300 gives the stick structure and the salinity difference can be reduced by half or eliminated completely.



Figure 15 AP-deo stick formulated with AEROSIL® R 972 V (left) and hectorite (center and right)8

Figure 14

Difference in salinity between top and bottom of an antiperspirant stick formulated without fumed silica and with 3 % w/w of different silica products.



Other rheology, or structurant aids, such as clays like bentonites and hectorites have inherent disadvantages. Clays can lead to a chalky white appearance which is clearly visible especially on dark clothes. Due to its different particle structure, properly dispersed AEROSIL® fumed silica does not leave the same white deposits behind as shown in Figure 15 where only the residue of the salts are seen.

Figure 16 shows that AEROSIL® fumed silica provides better product appeal in another way. Silica can be used as a titanium dioxide extender due to its lack of own color. Clays, such as the bentonites showed in the pictures, result in a dingy off-white color that would require the addition of titanium dioxide or talc to mask.



(powder)



Hectorite (predispersed gel)



AEROSIL® R 972

(powder)

Hectorite (predispersed gel)

Figure 16

Texture (left) and color (right) differences between antiperspirant sticks formulated with AEROSIL® R 972 V and different hectorite products.8

Guide Formulation: Invisible AP-Deo Stick - Low-whitening formulation with pleasantly dry skin feel

Phase	Ingredients	INCI name	% w/w
1	TEGOSOFT® APM*	PPG-3 Myristyl Ether	5.00
	TEGOSOFT [®] APS*	PPG-11 Stearyl Ether	5.00
	TEGO [®] Alkanol 18	Stearyl Alcohol	16.25
	Cutina HR	Hydrogenated Castor Oil	1.75
2	Cyclomethicone	Cyclopenta (and) Cyclohexasiloxane	47.00
3	Locron P	Aluminum Chlorohydrate	20.00
	AEROSIL® R 972 V**	Silica Dimethyl Silylate	3.00
4	TEGO [®] Deo LYS*	Zinc Ricinoleate & Lysine & Propylene Glycol	2.00
	TOTAL		100.00

Melt Phase 1 at 80-85 °C and stir until a clear phase is obtained. Cool Phase 1 to 75-78 °C. Add Phase 2 while stirring and stir for another 15 minutes. While stirring, add Phase 3 to Phases 1 and 2. Stir until Phase 3 is homogenously dispersed. Add Phase 4 and stir for another 5 minutes. Compensate loss of Cyclopentasiloxane prior to filling (temperature = 68-70 °C).

* Evonik Industries AG, Business Unit Consumer Specialties ** Evonik Industries AG, Business Unit Inorganic Materials

SIPERNAT[®] Hydrated Silica for Face Powders

Many makeup products for the face and eyes, such as facial powder, foundation, blush, and eye shadow, acquire the desired array of features and necessary stability only through the use of AEROSIL® fumed silica. The basis for these features are covered in previous sections and includes concepts such as using AEROSIL® fumed silica acting as an effective anti-caking and free flow agent to provide storage stability and dispersibility of powders. AEROSIL® and AEROXIDE® products such as AEROXIDE® Alu C, can allow for a matting or light diffusion effect, on the skin.

Dry Binder: A New Concept for Pressed Powder

Generally, to manufacture pressed powders, an oil-based binder such as isopropyl isostearate, isopropyl myristate, liquid lanolin, or silicone oil is sprayed onto the pigment containing powder mixture. Homogeneous distribution of the binder oil is difficult to achieve using this method. Without a uniform distribution the pressed cakes could have too much or too little oil. When pressed, the pressed cake can show oil spotting. These issues indicate poor structure for the pressed cake that will lead to chipping when dropped. In addition, oil that is sprayed into a mixing vessel will cake on the walls and lid of the mixer with the formulated pigments and slip aids. This causes a loss in product and the mixing process must be repeatedly interrupted to clean the mixing vessel.

To avoid these problems, the liquid binder oil can first be adsorbed onto SIPERNAT[®] precipitated silica. Because of its high pore volume, SIPERNAT[®] products can absorb up 66 % oil by weight. The resulting "dry", free-flowing binder is then mixed with the other powder components to create a homogeneous mix. When the mixture is pressed, the SIPERNAT[®] silica releases the binder oil causing the formulated powders to form a uniform cake that shows no oil spotting and can have an improved structural integrity than the classic production methods.

Guide Formulation: Eye Shadow Powder with "Dry Binder" – Easy and homogeneous distribution of "dry" binder oil.

Ingredient	INCI name	% w/w	
		green	blue
TALC COVASIL 4.05	Talc & Dimethicone & Trimethylsiloxysilicate	34.00	32.00
Orgasol 2002 N5 HYCOS	Nylon-12 & Hyaluronic Acid	10.00	10.00
SERICA 5 COVASIL 4.05	Mica & Dimethicone & Trimethylsiloxysilicate	10.00	10.00
Amihope LL	Lauroyl Lysine	2.00	2.00
PRESS AID SP	Synthetic Wax	3.00	3.00
ABIL [®] Wax 9814*	Cetyl Dimethicone		2.00
Timiron® Supersheen MP 1001	Mica & Titanium Dioxide	15.00	15.00
Colorona® Majestic Green	Mica & Titanium Dioxide & Chromium Oxide	15.00	
Colorona® Dark Blue	Mica & Titanium Dioxide & Ferric Ferrocyanide		15.00
Preservative	Preservative	q. s.	q. s.
Dry Binder	(see below)	11.00	11.00
Total		100.00	100.00

Combine all powdered ingredients and mix well using a suitable (e.g., a ribbon-type) blender. When uniformly dispersed, micropulverize and return to the blender. Add the Dry Binder Premix and continue to blend until uniformly dispersed. Scrape down powder from sides of the blender whenever necessary. Pulverize through a screen. Press into godets.

Dry Binder Premix

Ingredient	INCI name	% w/w
SIPERNAT [®] 50 S**	Hydrated Silica	30.85
CERAPHYL 847	Octyldodecyl Stearoyl Stearate	69.15
Total		100.00

Weigh SIPERNAT* 50 S into a suitable, clean vessel equipped with, for example, a ribbon-type blender. Mix at low to medium speed and add octyldodecyl stearoyl stearate in 3-4 steps while mixing. Pass through 0.8 mm screen and allow stand overnight.

* Evonik Industries AG, Business Unit Care Specialties
 ** Evonik Industries AG, Business Unit Inorganic Materials

IV. Advanced innovative Formulating

Dry Water Concept

Water or dilute aqueous solutions can be converted to a dry powder with the help of super hydrophobic AEROSIL® R 812, AEROSIL® R 812 S and AEROSIL® R 202. The powdery product can contain up to 95% w/w of an aqueous phase. Under mechanical stress, for example when the product is rubbed onto the skin, the water is released. In this way, solutions containing vitamins, plant extracts and/or other active ingredients can be converted to powdered products.

"Dry Water" is created by producing fine water droplets in the presence of the hydrophobic AEROSIL* during a high shear mixing process. While mixing the fine water droplets are coated by the hydrophobic silica which prevents the water droplets from coalescing. The result is a powdered substance, typically referred to as "Dry Water" (see Fig. 17). The Dry Water concept is based on surface tension. The extremely hydrophobic AEROSIL® particles will not mix with the polar water droplet. This difference in surface tension prevents the droplets from coalescing. However, additives added to the aqueous phase can lower the surface tension allowing droplets to coalesce which can lead to instability. Depending on the effect on the surface tension the basic Dry Water concept allows for the incorporation of up to several percents of additives by weight before the formulation becomes unstable. The more non-polar the additive is, the more the additive will affect the surface tension and the less the additive can be added without the Dry Water becoming unstable. The type of additive can also affect the stability of Dry Water by wetting the surface of the AEROSIL® silica. Certain surfactants and non-polar additives can allow the hydrophobic AEROSIL® types to be miscible with water. Once this happens, the silica moves into the aqueous phase, it can no longer prevent coalescence of the water phase and the formulation will become unstable. Possible applications for Dry Water include bronzing powders, mattifying powders, and hair styling powders.



Figure 17 Schematic diagrams of Dry Water (left) and close-up of the silica layer (right).

Powder-to-Cream Concept

The original Dry Water concept was limited to only a few percent of actives in order to achieve stability. Recently, the concept has been advanced in order to enable more formulation flexibility and much higher active concentrations. By using carrier silica, additives can be incorporated outside of the aqueous environment allowing for many more types of additives to be used successfully and amounts up to approximately 20 % w/w while maintaining a stable powder.

How the concept works

As mentioned earlier, silica works very well as an absorptive carrier that will release the carried additive when in the presence of water. When under mechanical stress, Dry Water turns from a powder into water. This water liberates the additives out of the carrier silica, allowing for formulations that appear as a powder, but apply as a cream (Fig. 18). For more information please see TI 1394 Powder-to-Cream: An innovative concept for cosmetic formulations in powder form.



Figure 18

"Powder-to-Cream". Left: powder containing 66% w/w moisture. Right: The powder turns into a moist cream upon application.

	Effects	Effects							Formulation Concepts		
	Rheology C	Rheology Control Viscosity Increase			Emulsion Stabilization	Suspension Stabilization	Matting Effects	Actives Absorbate	Dry Water	Powder- to-Cream	
	hydrocar- bon / silicone oil	polar liq- uids (e.g. glycerine, PEG)	vegetable oil								
ACEMATT® TS 100*							+				
AEROSIL [®] 200	+			+							
AEROSIL® 300	+										
AEROSIL® R 202		+	+						+	+	
AEROSIL® R 805		+	+		+	+					
AEROSIL® R 812						+			+	+	
AEROSIL® R 812 S									+	+	
AEROSIL® R 972				+	+	+					
AEROSIL® R 974			+		+	+	+				
SIPERNAT [®] 22 LS	+							+		+	
SIPERNAT [®] 22 S				+				+		+	
SIPERNAT [®] 50 S				+				+		+	
SIPERNAT [®] 500 LS	+							+		+	

Product Recommendations

* For nail polish only

	Effects				Formulation Concepts			
	Rheology Control Viscosity Increase	Free Flow/ Anti Caking Agent	Emulsion Stabilization	Suspension Stabilization	Matting Effects	Actives Absorbate	Dry Water	Powder-to- Cream
Antiperspirant stick	+			+				
Antiperspirant roll-on	+			+				
Lipstick	+			+				
Lip Gloss	+			+				
Cream Foundation	+			+				+
Aqua Foundation			+	+				+
Sunscreen			+	+				
Mascara	+		+	+				+
Pressed Powder		+				+		+
Nail polish	+			+	+			
Hair Bleach	+	+						
Hair Dye	+							
Hair Conditioner	+							
Hair Styling							+	+

Products for Toothpaste

	Abrasive	Rheology Control
SIDENT [®] 22 S		+
AEROSIL® 200 F		+
SIDENT [®] 8	+	
SIDENT [®] 9	+	
SIDENT [®] 10	+	

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