

Gloxil WW SL -

Functional matting agent for

water-based clear coats

Authorship: Bodo Essen
 Hubert Oggermüller

Contents

- 1 Introduction
- 2 Experimental
 - 2.1 Base formulation
 - 2.2 Matting agent characteristics
 - 2.3 Formulation variations
 - 2.4 Preparation of batches, application and testing
- 3 Results
 - 3.1 Processing properties and storage stability
 - 3.2 Optical properties
 - 3.2.1 Color
 - 3.2.2 Transparency
 - 3.2.3 Matting
 - 3.2.4 Appearance on wood
 - 3.3 Resistance properties
 - 3.3.1 Water
 - 3.3.2 Alcohol
 - 3.3.3 Ink
 - 3.3.4 Performance comparison
 - 3.4 Performance with simplified preparation
 - 3.4.1 Optimized incorporation and time savings
 - 3.4.2 Foam suppression
 - 3.4.3 Prevention of high shear rates
 - 3.4.4 Low cleaning effort
 - 3.4.5 Maintenance of high technical performance and subsequent addition to finished coating
 - 3.5 Overall performance
- 4 Summary

1 Introduction

Dispersion-based clear coats are becoming increasingly popular as efficient, versatile, and environmentally friendly coating materials with low VOC emissions.

The process of matting these coatings while still maintaining both excellent film appearance and excellent resistance properties is sometimes difficult, especially when using binder emulsions with higher minimum film formation temperature and glass transition temperature. Furthermore, when it comes to dealing with traditional matting agents, there are significant disadvantages presented by their form, which are mostly powder-based.

In a bid to counteract this situation and in response to the high demands of the market, a liquid product in slurry form that offers even better performance has now been developed by Hoffmann Mineral.

This report aims to highlight the performance of the new Gloxil WW SL in comparison to established, powdery silica matting materials using the example of a sensitive, water-borne, acrylate-based standard clear coat for wood. Particular attention is paid to the resulting effects on visual appearance, stain resistance to water, alcohol and ink; and to the improvement of handling and processing properties.

2 Experimental

2.1 Base formulation

Fig. 1 shows the underlying formulation of a universal 1K acrylate dispersion for multi-layer coating of wood surfaces. The formulation is based on a self-crosslinking, relatively hard binder with a minimum film formation temperature of 43°C. The combination of butyl diglycol and butyl glycol is used as a coalescence agent to ensure film formation also at lower temperatures.

In addition to Gloxil WW SL, two commercial silica matting agents are also used and tested for comparison purposes.

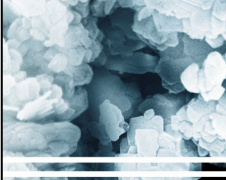

		HOFFMANN MINERAL		
		Base Formulation		
INTRODUCTION <u>EXPERIMENTAL</u> RESULTS SUMMARY 		Derived from guide formulation FP 2514-5 from Alberdingk Boley		
		Description	pbw *	
		Alberdingk AC 2514	Binder, acrylic emulsion	79.5
		Byk 024	Defoamer	0.8
		Butyl diglycol	Cosolvent	6.0
		Butyl glycol	Cosolvent	2.0
		Water demineralized		7.5
		Matting agent	Silica or Gloxil WW SL	varied X
		Aquamat 272	Wax dispersion	3.3
		Byk 346	Wetting agent	0.4
Rheovis PU 1214	Thickener	0.5		
Total		100.0 + X		
* Parts by weight				
VM-0/02.2018				

Fig. 1

2.2 Matting agent characteristics

The competitive silicas used for comparison purposes took two forms – one fumed and one precipitated. As shown in Fig. 2, these are both characterized by high oil absorption, which is based on a very high specific surface area in each case. For the fumed silica, this is based on very fine, fused primary particles with a resulting average agglomerate particle size of about 9 µm. As for the precipitated silica, this comprises significantly larger, segregated primary particles with a high level of porosity that is primarily responsible for its high BET surface area.

Unlike the powdery silicas, Gloxil WW SL features an aqueous dispersion of a comparatively coarse silica with additional additives. The density is noticeably reduced on account of the water content.

		Matting Agents		
		Fumed Silica	Precipitated Silica	Gloxil WW SL
INTRODUCTION		Characteristic		
<u>EXPERIMENTAL</u>				
RESULTS		Appearance		
SUMMARY		Density [g/cm ³]		
		Particle size d ₅₀ [µm]		
		Oil Absorption [g/100g]		
		Specific Surface Area BET [m ² /g]		
		* Agglomerated		
VM-0/02.2018				

Fig. 2

2.3 Formulation variations

The amounts of matting agent added are shown in Fig. 3. The dosage of the comparative products is based on achieving considerable matting level in the range of around 10 to 15 gloss units within the 60° angle. In the formulations with a tiered dosage of Gloxil WW SL, the additive water content of the base formulation was omitted as this was already introduced in the form of the slurry water.

In spite of the higher dosages, the necessary space required for the Gloxil WW SL is comparatively low. It is only at the highest dosage level that it takes in more or less the volume of the precipitated silica, whereas the fumed variant is at a clear disadvantage by being even more voluminous.


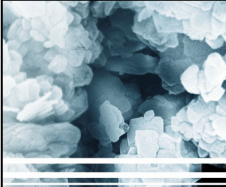
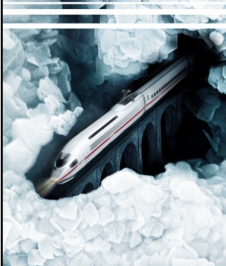
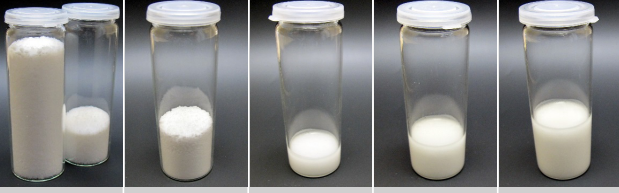
						
 INTRODUCTION <u>EXPERIMENTAL</u> RESULTS SUMMARY 	Formulation Variants					
	pbw					
		Fumed Silica	Prec. Silica	Gloxil WW SL		
	Matting Agent	2.5	2.5	7.7	15.4	23.1
	Bulk / Liquid Volume					
	Water demin.	7.5	7.5	---	---	---
	All other ingredients remain unchanged					
	Total	102.5	102.5	100.2	107.9	115.6
	%					
	Solids Content	38.7	38.7	39.2	38.4	37.7
VM-0/02.2018						

Fig. 3

2.4 Preparation, application and testing

The preparation of the formulations was carried out in a laboratory dissolver equipped with a 4 cm toothed disc. After incorporating the defoamer into the designated binder, the combination of the film forming agents (which have been pre-mixed with water) was slowly added and homogenized. The matting agent was added and the batch was dispersed for 15 minutes at 2500 rpm (5.2 m/s). Including the addition of the other additives, the complete batch typically required a preparation time of approximately 40 minutes.

The following substrates were used for the individual tests:

Contrast cardboard

- Color, CIE L*, a*, b* over white, ISO 7724
- Transparency, CIE L* over black, ISO 7724
- Gloss, DIN EN ISO 1522

Black Leneta film

- Blocking resistance, in accordance with ASTM 4946

Q-Panel Type R 48

- Drying properties, Erichsen Method (no film damage with sliding wire bow)

Wood substrate beech or American walnut

- Transparency, visual assessment
- Water, alcohol and ink resistance, DIN EN 12720, DIN 68861-1

The clear lacquer samples generally were applied in one layer with a 4-sided applicator frame. For tests on wood, three layers were applied at intervals of 3-4 hours for intermediate drying and subsequent intermediate sanding with grain size P220.

If not indicated otherwise, prior to the tests the coating films were dried resp. conditioned for 28 days in an air-conditioned laboratory at 23 °C and 50 % relative humidity.

3 Results

3.1 Processing properties and storage stability

The differences in the physical properties of the matting agents tested in this process have a lasting effect on the incorporation into the liquid formulation batch.

On account of their low bulk density, both powder-based silicas tend to increase the formation of dust, which has a disadvantageous effect on the incorporation times – particularly with the fumed grade. Furthermore, the dispersion batches of the two comparative products result in a greater level of foam formation, whereby a longer deaeration time is required after preparation.

Compared to the powdery silicas, Gloxil WW SL as a liquid slurry avoids dust entirely to ensure reliable, quick, and easy incorporation. Introduction of air during dosage of the matting agent is avoided completely and foam formation in dispersion process is visibly reduced.

After a maturation period of 12 hours, all formulation batches are free from micro-foam and can be processed further.

Overall, the storage stability with Gloxil WW SL can be rated good. As with the silicas in the comparison formulations, the sedimentation tendency is exceptionally low. Only slight settling of the matting agent without formation of hard sediment favors easy re-stir behavior and homogenizability.

The evaluation of the drying time followed a method, where at short time intervals a wire bow is drawn across the drying coating surface with an automated film drawing unit. The time after which no damage of the film surface can any longer be observed, is counted as an index for the length of the drying time.

As shown in Fig. 4, the times for the quick-drying coatings are all extremely similar and are based on the effective total water content in the formulation. The use of Gloxil WW SL results in a shorter drying time in the low dosage, and a drying behavior similar to the competitive level in higher dosages.

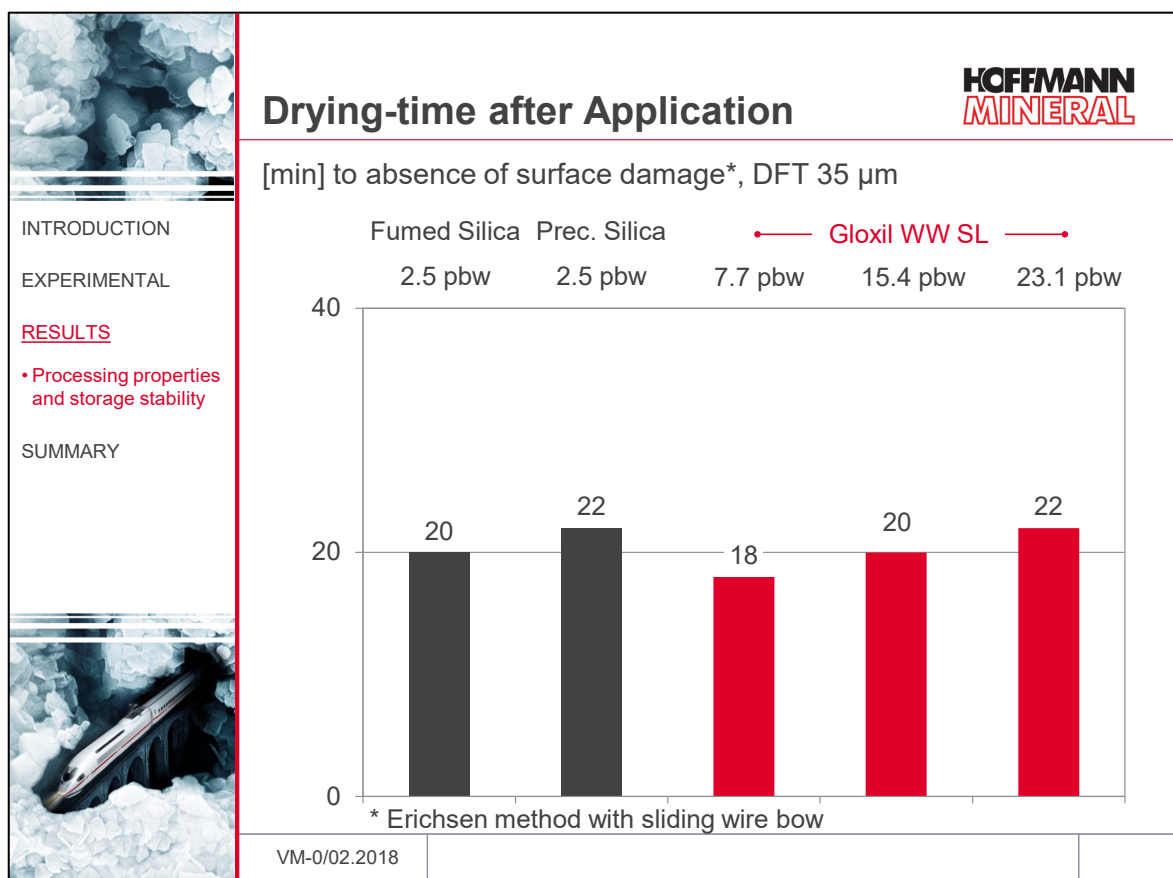


Fig. 4

Fig. 5 highlights the results for determining the early blocking resistance. This property is a criterion for judging the time after which finally coated wood surfaces can be stapled or packed without the risk of sticking together. In the present work, the substrate chosen was Leneta film which after application and 24 hours of drying of the coating was cut into stripes, put together “face to face” and loaded with 100 g/cm² for 24 hours.

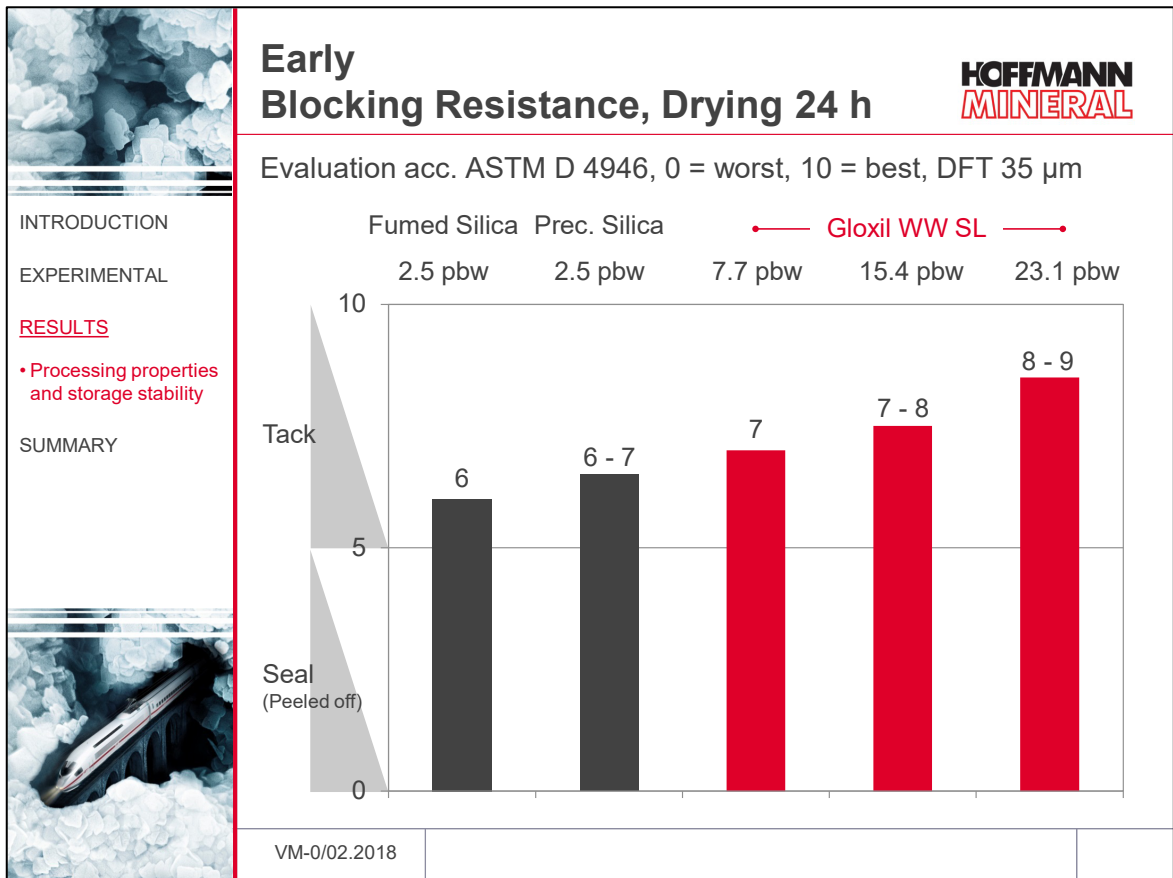


Fig. 5

After 24-hour drying period, all test samples were beyond any signs of peeling off, but tack at a different level was still evident. While the use of the powder-based silicas gives a slightly negative effect, Gloxil WW SL positively reduces the potential for sticking together. It also improves the level of early blocking resistance as the dosage increases.

As a result, the amount of time and storage effort required prior to further processing in the plant is reduced. In manual field of application, the risk of blocking of painted wooden pieces on-site – such as windows/doors in frames – can be reduced.

3.2 Optical properties

3.2.1 Color

Color values were determined over white background on contrast cardboard. As shown in Fig. 6, there are virtually no changes in the brightness L^* or the color values a^* and b^* caused by any of the matting agents. The fairly high b^* value is equal to the result on uncoated substrate and, therefore, is not specific for the tested coating films.

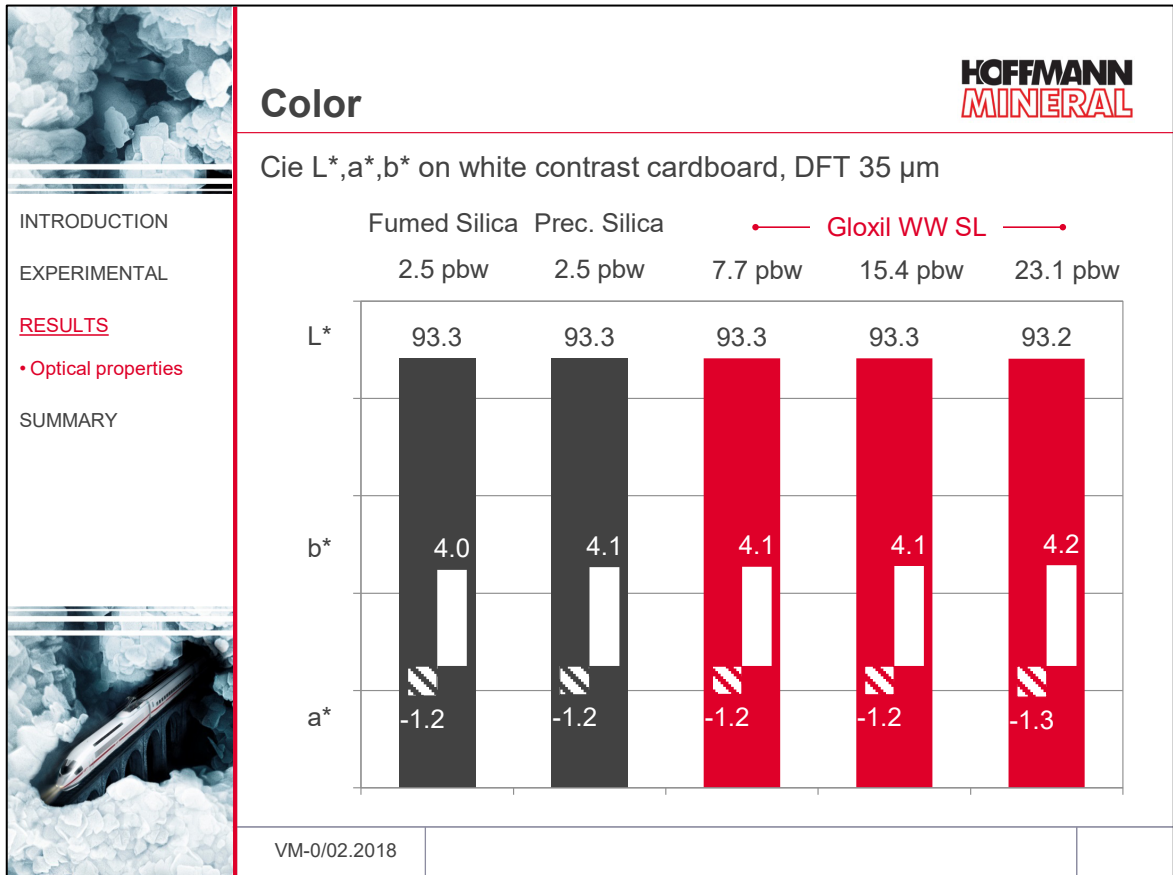


Fig. 6

3.2.2 Transparency

For these tests, initially dried coating films of comparable dry film thickness were measured on contrast cardboard. Clear coats for wood with good transparency over black background basically do not give rise to a change of the brightness index L^* . Poorer transparency is indicated by higher L^* values, as the opacity is increased and the dark background seems to be optically brighter. Inversely, a very low L^* can be interpreted as a quantitative index for good transparency.

The repeated measurements after varying conditioning times show a very stable level in Fig. 7 when using fumed silica.

When matting with precipitated silica, on the other hand, the increasing L^* values demonstrate a clear time dependency of the measurement result along with a noticeable and progressive loss of transparency.

With Gloxil WW SL, the brightness values stabilize in all variants and demonstrate a significantly better transparency behavior – just like the fumed silica. In a direct comparison to this variant, Gloxil WW SL even achieves a slight improvement in transparency in the first two dosage levels of up to 15 parts by weight.

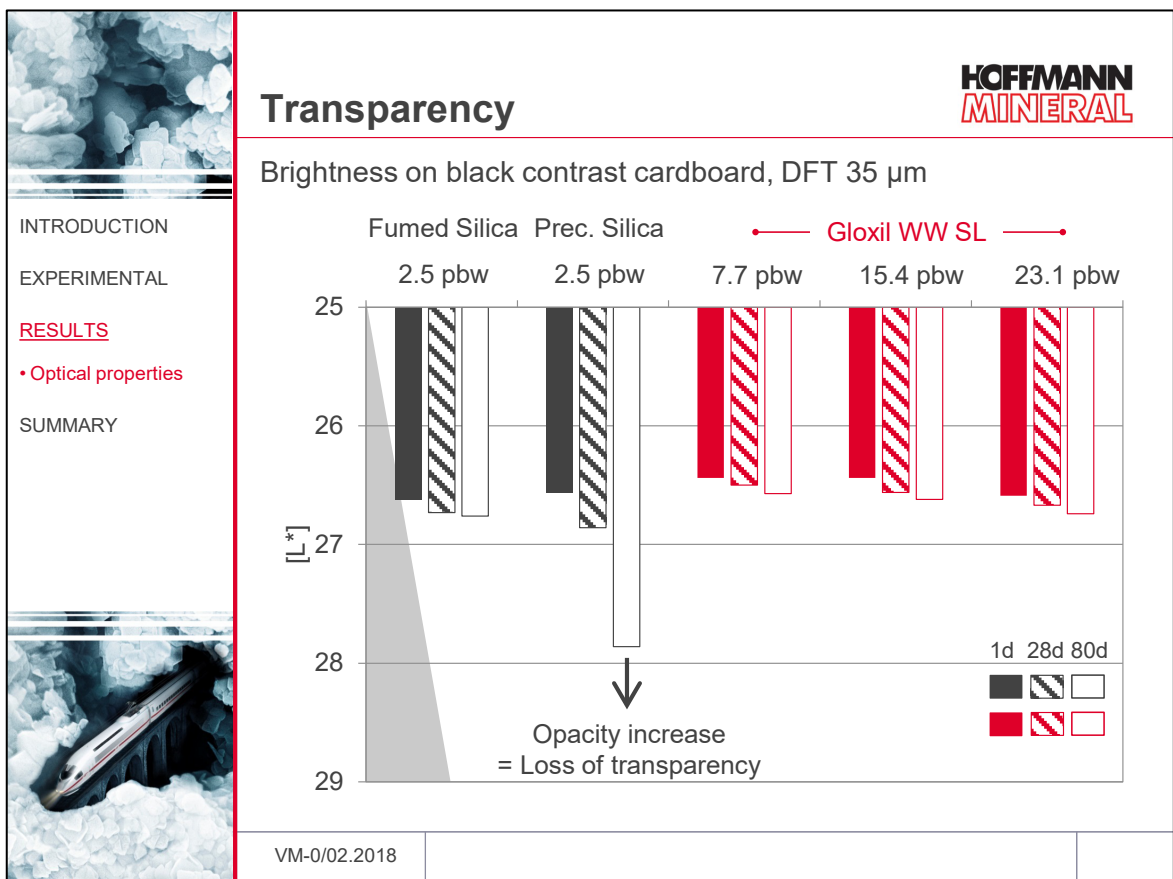


Fig. 7

3.2.3 Matting

In correspondence with the dosing levels of the formulation variants in Chapter 2.2, the matting agents give rise to reduced gloss levels, as shown in *Fig. 8*.

In a direct evaluation of the competitive products, the precipitated silica has proven to be a somewhat more effective matting agent than the fumed grade on account of the lower gloss values.

Gloxil WW SL offers slight matting at a low dosage level; at medium dosage levels, however, there is already an at least comparable matting potential in comparison to the other products. For strong, optimized gloss reduction, especially within 85° angle, the high dosage is recommended.

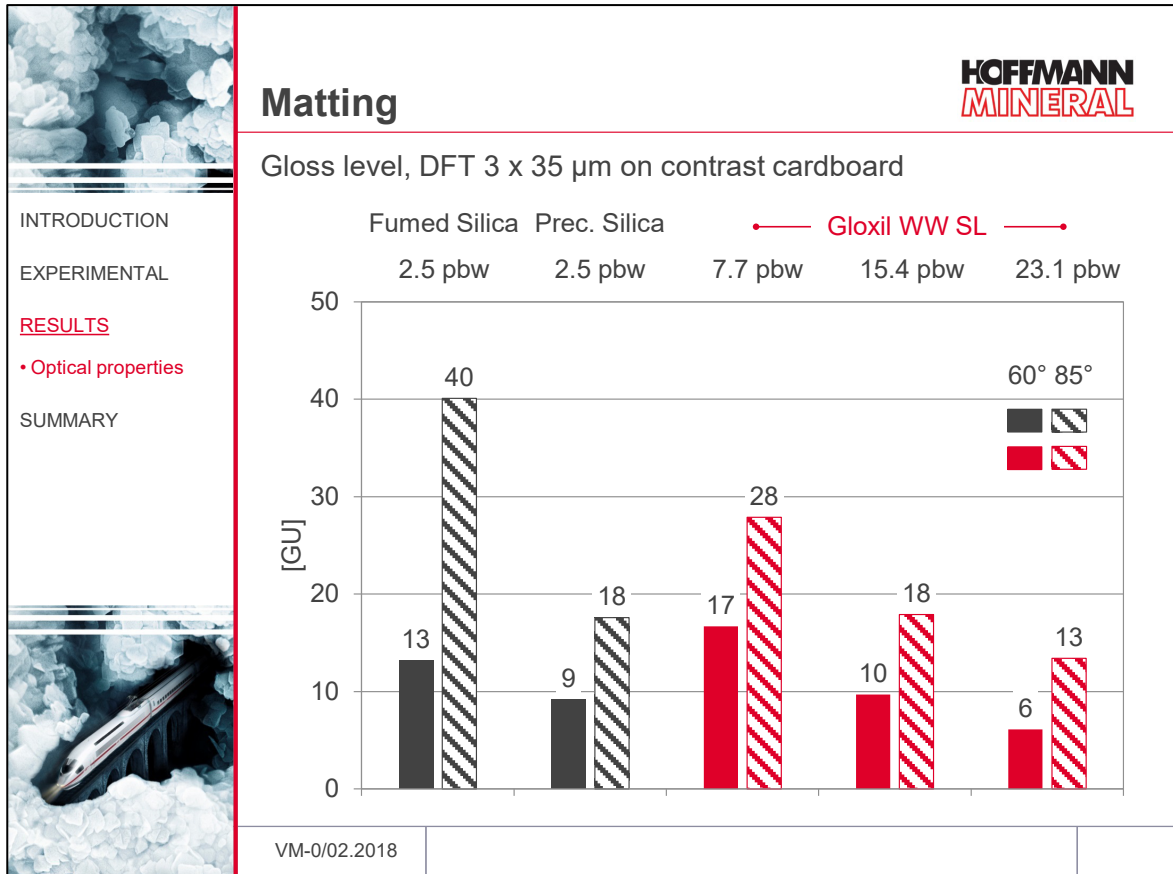


Fig. 8

3.2.4 Appearance on wood

The optical appearance of the formulations applied to wood in three coats using a four-sided applicator frame is extremely different with regard to the competitive silicas.

For the fumed silica, there is an exceptionally high level of transparency on light and dark wood (Fig. 9). In the case of the coating matted with precipitated silica, with increasing time after the application a reduction of the transparency becomes evident, which is in accordance with the brightness tests on black contrast cardboard. The transparency loss increasingly brightens optically up the coating. This effect has a visibly negative impact on the optical appearance of dark woods in particular; the strong contrast of the wood grain is significantly diminished. On beech wood, the dampened, slightly warming reddish color tone comes out reduced.

With Gloxil WW SL, the appearance of the clear coat improves significantly again, slightly exceeding even the wood 'firing' level of the fumed silica. Even in the variant with 23.1 parts by weight of Gloxil WW SL, the level of transparency is excellent. This almost looks unexpected, as with higher matting intensity the diffuse light scattering on the micro-rough surface mostly affects more and more negatively the optical appearance of the coated wood surface. The over the whole only slight decrease of the transparency with increasing addition here points to the high performance of Gloxil WW SL.

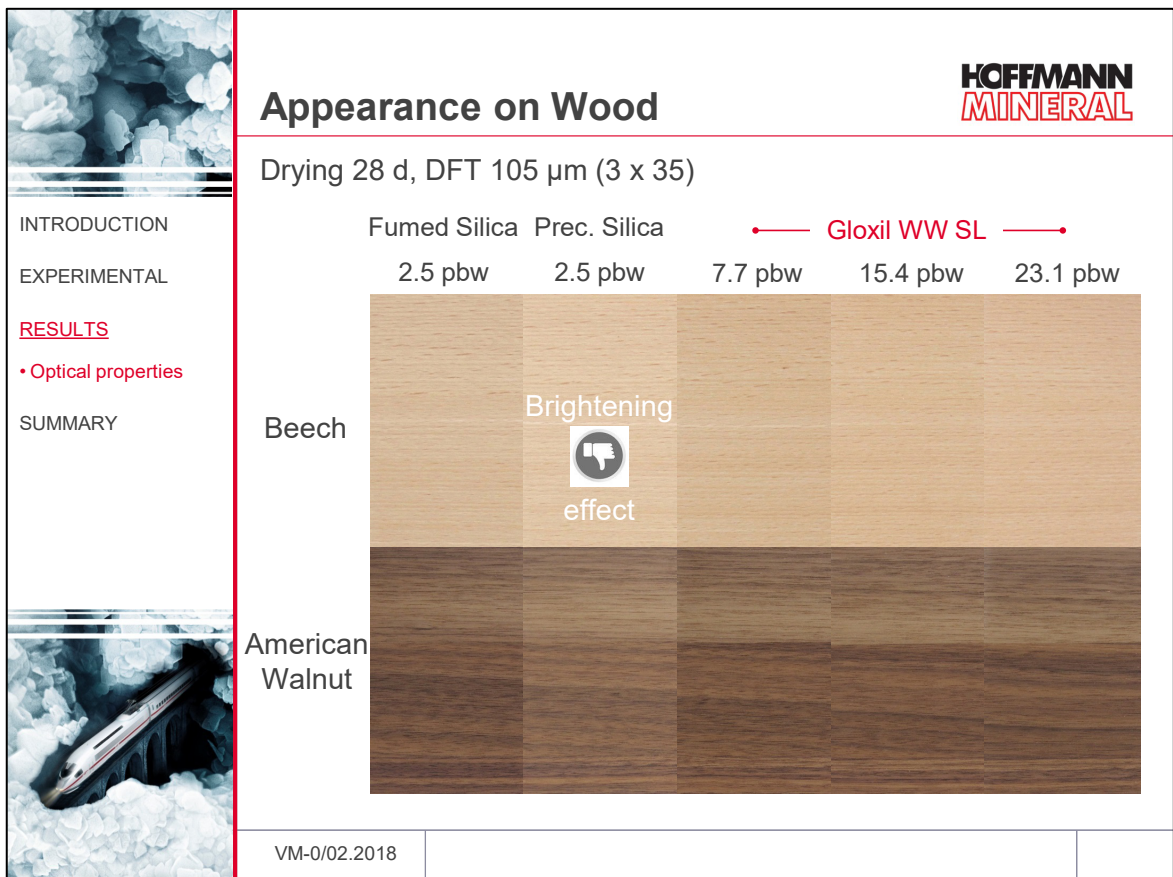


Fig. 9

3.3 Resistance properties

The evaluations of the resistance of the coating surfaces against the chemical attack of liquids were carried out in accordance with the specifications of DIN EN 12720. The assessments were done according to DIN 68861-1, 1A after a drying time of 28 days under 16 hours of exposure, as well as for comparison earlier after only 15 hours of drying. In place of the liquids listed in DIN 68861-1, in particular water, alcohol and blue ink were used, as in far-reaching pre-trials these test liquids had given proof of high sensibility and significance.

3.3.1 Water

The problem of matting while simultaneously maintaining the resistance properties is shown impressively by the highly limited performance when using the powder-based silicas, as shown in Fig. 10. After just a short drying time, there is a significant mark (blushing effect) left behind with both competitive silicas when exposed to water. Prolonging the conditioning time to 28 days prior to the exposure, only the coating with the fumed silica shows visible signs of improvement, whereas the very poor performance with the precipitated silica barely improves.

Only with Gloxil WW SL it appears possible to create an effective matting effect with virtually no loss of water resistance. It is also significant to note the highly positive protective effect of the matting agent even during early stage exposure of the drying coating. This makes Gloxil WW SL particularly suitable for applications involving high requirements for early water resistance.

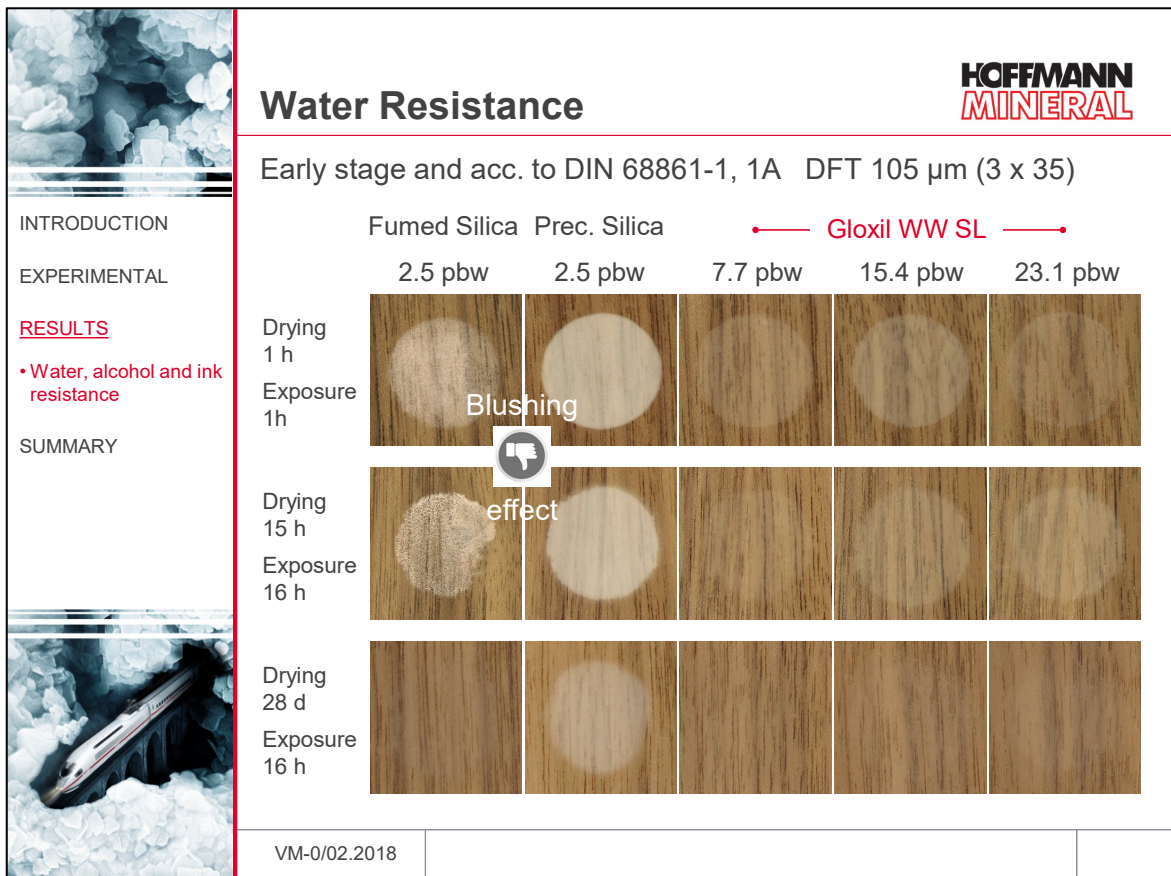


Fig. 10

3.3.2 Alcohol

The evaluation sequence for water resistance is almost comparable to the behavior when exposed to diluted ethanol (48 %). In spite of the aggressive character of this test liquid in relation to water, the exceptional properties for the Gloxil WW SL remain unaffected. While matting with precipitated silica results in insufficient resistance, the Gloxil WW SL offers very high effectiveness at the level of the fumed silica.

Fig. 11 provides a visual representation of the accompanying results using the example of the coating films that were conditioned for 28 days.

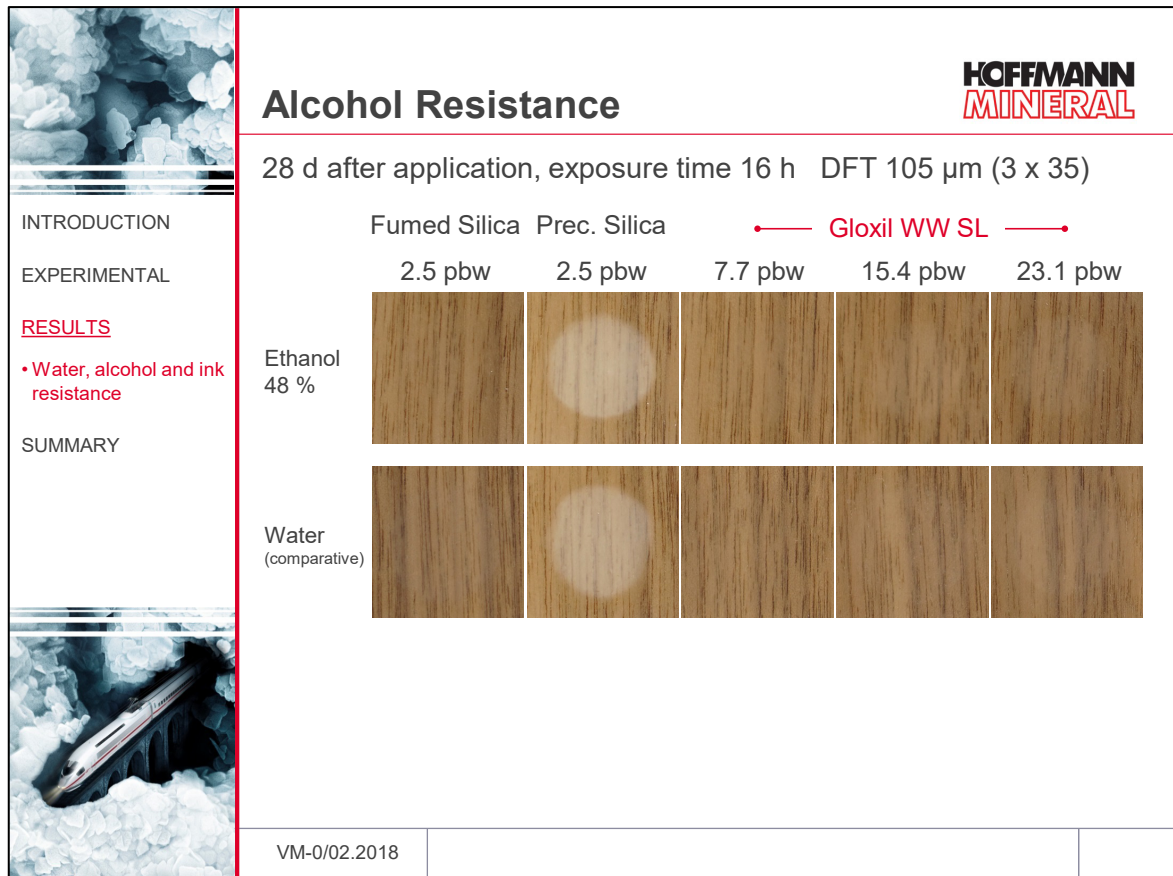


Fig. 11

3.3.3 Ink

Ink in particular has proven a highly sensitive test liquid representing also a number of other substances such as coffee, red wine, ketchup etc., which in households do often cause stains on decorative wood surfaces. In the “spot test”, ink was applied drop-wise to the coatings surfaces as shown in Fig. 12.

The two competitive variants still demonstrate an insufficient level of early resistance on the whole. The use of the powdery silicas lead to noticeable spreading of the applied ink droplet, as a result of which the damaged surface becomes markedly enlarged. As the drying time increases, the spreading effect is reduced. Nevertheless, the coating surfaces are visibly more susceptible to permanent staining. The precipitated silica in particular results in increased staining of the coating film.

Gloxil WW SL already counteracts these effects in the early loading phase, even with a low dosage, and protects the coating layer from even further staining despite the fact that the film formation is still incomplete. Depending on the dosage level, staining can even be avoided entirely. Therefore, Gloxil WW SL is able to ensure excellent spot insensitivity and in appropriately higher concentrations even at prolonged exposure to the liquids maintains almost marking-free coating surfaces.

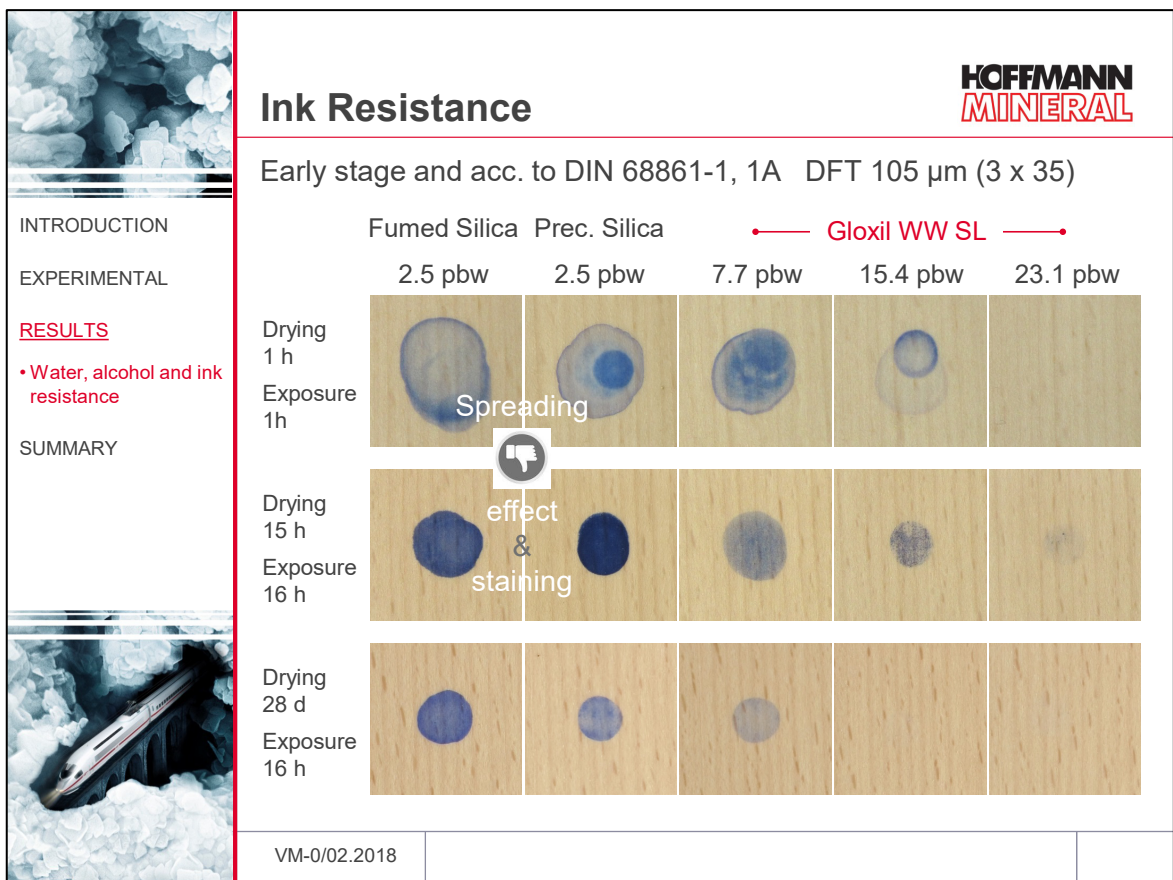


Fig. 12

3.3.4 Performance comparison

In comparison to the powdery matting agents based on silica, *Fig. 13* highlights the special performance of Gloxil WW SL and underlines the following statements, which have been substantiated by a number of further experiments:

- Water and alcohol resistance
 - With Gloxil WW SL, largely independent from loading likewise matting level, whereas pure silicas deteriorate at higher loadings
- Ink resistance
 - Significantly improved with Gloxil WW SL at higher loading and/or matting, whereas silicas remain at a weak performance level

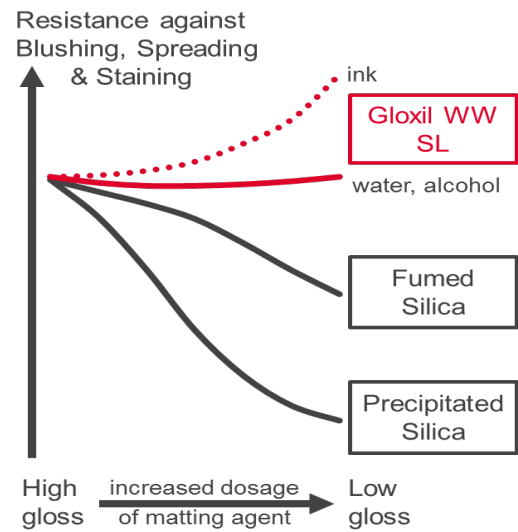


Fig. 13

Independent from the desired degree of matting, Gloxil WW SL offers to the user very good performance which even can be further improved by higher additions. Along with the extraordinary early resistance the result is a high insensitivity towards water or paint splashes directly in the application area or during final use later.

3.4 Performance with simplified preparation

With its excellent processing properties, very good optical properties and high stain resistance, the Gloxil WW SL combines the requirements for a state-of-the-art matting agent into a single product. This results in the following direct benefits for the user:

3.4.1 Optimized incorporation and time savings

Internal laboratory tests confirmed that the usual matting agent incorporation step can still be simplified by some way. Gloxil WW SL is a liquid, pre-dispersed product, which – unlike powdery silicas – does not have to be wetted first but can be easily dosed absolutely dust-free and within shortest time.

In addition, it is possible to dispense with the 10 to 15 minute dispersion process typically associated with classical matting agents. Even using a standard laboratory paddle mixer, Gloxil WW SL clearly saves time by allowing the complete dosing and agglomerate-free incorporation to be achieved within 2 minutes.

3.4.2 Foam suppression

The ability to stir in Gloxil WW SL using a paddle stirrer qualifies the matting agent to be incorporated without creating any foam at all. Fig. 14 shows the conditions in each case two minutes after the end of the mixing process.




		Dissolver		Overhead stirrer
		Fumed Silica	Prec. Silica	Gloxil WW SL
Foaming				
		strong	strong	no ✓
Deaeration		several hours		immediately
VM-0/02.2018				

Fig. 14

For the competitive matting agents, however, the conventional dispersion on a dissolver leads to a very strong, stable buildup of foam. The micro-foam included in the liquid matrix takes up almost the entire formulation volume space here, which is almost twice as large compared to the formulation with Gloxil WW SL. The foam-suppressing effect with Gloxil WW SL has a particularly advantageous effect, since further processing can take place without delay.

3.4.3 Prevention of high shear rates

The incorporation of a matting agent with a simple agitator at a moderate shear rate is a cost-effective alternative to an energy-intensive dissolver. Insofar, the question arises as to whether the simplified manufacturing process also works with powder-based silicas. The laboratory tests also show a reduction in foam formation at this point. Nevertheless, the restrictions clearly described in Fig. 15 exclude the actual application:

The fundamental risk of adhesion to surfaces increases with powder-based silicas further still compared to the usual dispersing process. In addition to the visible sticking/adhesion to the stirrer shaft or container wall, concealed filler nests can form on the propeller at an early stage, which can no longer be mechanically separated further along in the process. Numerous remaining coarser agglomerates in the formula also prove the need to use significantly higher shear forces in order to obtain a satisfactory surface quality of the coating after application.

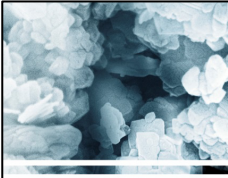






 INTRODUCTION EXPERIMENTAL RESULTS • Combining performance effects SUMMARY 	Customer Benefit				
	Dispersibility Without High Shear Force				
		Overhead stirrer			
	Fumed Silica	Prec. Silica	Gloxil WW SL		
Dispersion problems			no ✓		
Adhesion	lump nests	sticking			
Insufficient particle breakdown	agglomerates		no ✓		
					
VM-0/02.2018					

Fig. 15

With Gloxil WW SL, comparable difficulties do not arise, providing defect-free, high-quality matted surfaces to be achieved.

3.4.4 Low cleaning effort

The incorporation of powder-based matting agent easily leads to higher cleaning effort as the laboratory images in *Fig. 16* already show on account of the dust behavior. Since fumed silica in particular has a strong tendency to create dust, technical measures such as vacuum suction and basic suction devices are therefore already necessary for occupational safety reasons (dust protection) in the environment of the dosing process.

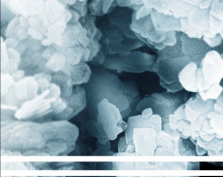


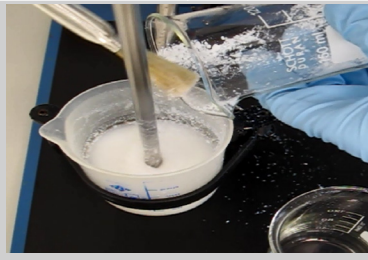





 INTRODUCTION EXPERIMENTAL <u>RESULTS</u> • Combining performance effects SUMMARY 	Customer Benefit				
	Easy Dosing – Easy Cleaning				
		Overhead stirrer			
	Fumed Silica	Prec. Silica	Gloxil WW SL		
Cleaning effort					
During dosing				no ✓	
Subsequently				low ✓	
VM-0/02.2018					

Fig. 16

This additional effort becomes superfluous when using Gloxil WW SL, meaning savings can be made; the remaining effort is limited to cleaning the delivery container and/or metering device if required.

3.4.5 Maintenance of high technical performance and subsequent addition to finished coating

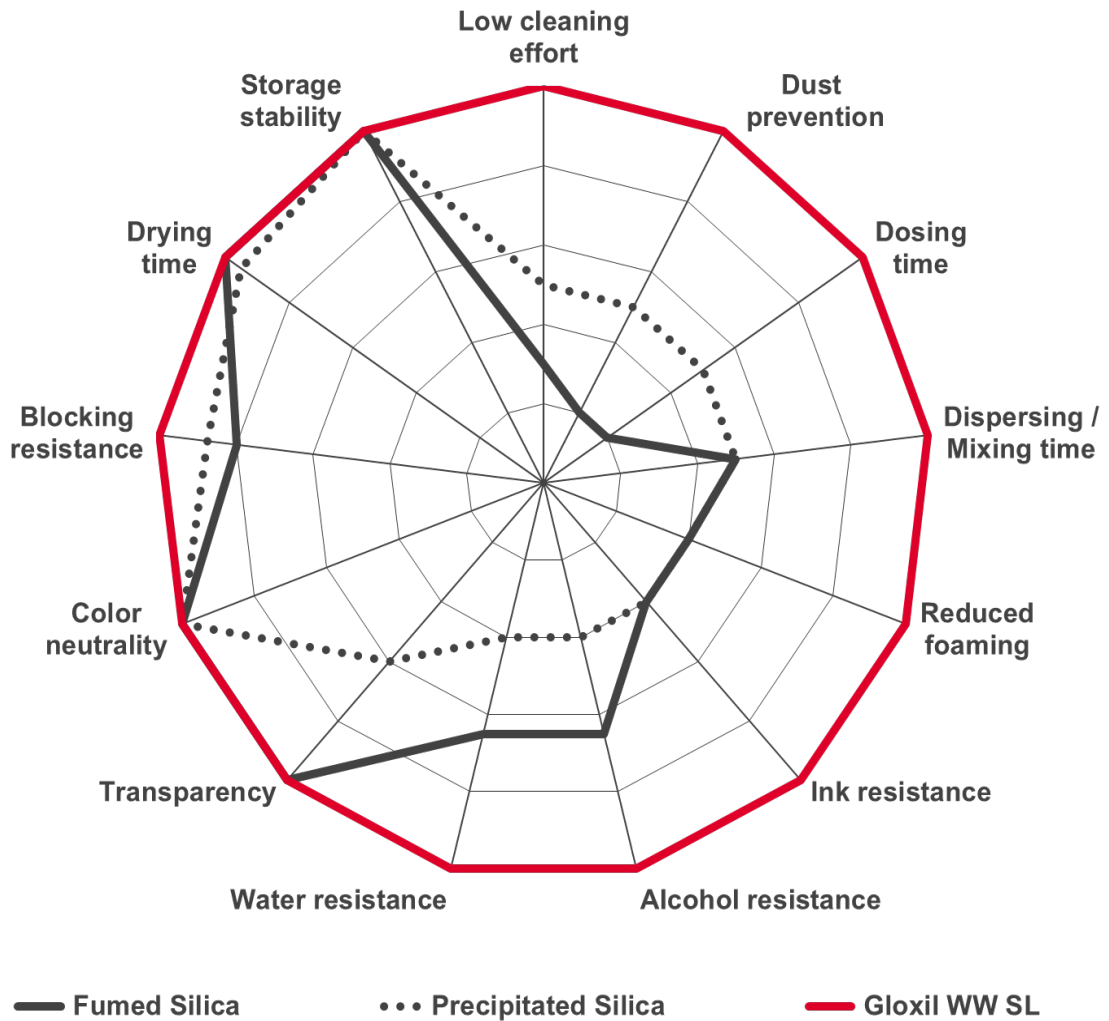
The simplified incorporation compared to a regular dispersing process has no negative effect on the technical performance. The optical property profile and the resistance properties are retained. The option is available to either add the Gloxil WW SL normally as part of the matt coating preparation process or else subsequently to the glossy finished coating. The flexibility gained in this way offers additional customer benefits, as the coating preparation and matting process can be handled separately. Self-tests on the mode of action can be carried out swiftly in advance based on a single formulation batch, which is easily adjustable with Gloxil WW SL to achieve the desired matt finish.

3.5 Overall performance

The relative performance of the matting agents considered in this study is shown graphically in Fig. 17 on the base of a comparable matting level of 10 to 15 gloss units within the 60° angle. For each relevant property, the outer border identifies the best result achieved in the matting agent comparison as the maximum value.

In the rating, the maximum values for all properties ultimately reflect the excellent overall profile of Gloxil WW SL in all areas. With the powder-based silica matting agents, a partly cross-over is found only on the left-hand side of the properties, while on the opposite side it showcases the large losses in performance that have been discussed in detail.

Fig. 17



4 Summary

In comparison to powder-based silica matting agents, Gloxil WW SL offers the following, highly advantageous performance profile for the use in water-borne coatings for surfaces such as wood:

Outstanding handling and processing properties

- Complete dust avoidance without risk of powder adhesion on surfaces
- Easy and fast incorporation without high shear forces
- Foam suppressing effect
- Good storage stability

Support for application-relevant properties

- Rapid drying
- High early blocking resistance

Excellent optical properties

- Strong matting effect
- Very high transparency with good long-term stability
- Good wood grain enhancement, especially on dark wood

Outstanding resistance against exposure to liquids

- Optimized water and alcohol resistance
- Strongly improved resistance against spots from coloring liquids
- Significantly higher early resistance

The results show that Gloxil WW SL is primarily suitable for dispersion-based clear coats, particularly acrylate-based systems, which on matting normally show a high sensitivity towards staining. Here, Gloxil WW SL shows the effects known from classical matting agents, but imparts to the coating surface a markedly higher resistance with simultaneously very good optical properties. As an efficient, highly versatile, liquid matting agent that is both easy and ready to use, Gloxil WW SL provides the option of simplified incorporation and the flexibility of subsequent adjustment of matting level.

The resulting potential for savings in terms of time, energy and work effort significantly contributes to the performance advantage and also makes Gloxil WW SL an ideal and attractive matting agent with improved performance in respect of costs.

Last but not least thanks to the very good handling and processing properties Gloxil WW SL is convincing also in the partial or complete replacement of classical matting agents, and when appropriate can be blended with Neuburg Siliceous Earth.

Additional information about the general use of Neuburg Siliceous Earth grades in formulations for aqueous wood lacquers will be found in the Technical Report "Neuburg Siliceous Earth in Water-based Acrylic Clear Coats for Wood".

Our technical service suggestions and the information contained in this report are based on experience and are made to the best of our knowledge and belief, but must nevertheless be regarded as non-binding advice subject to no guarantee. Working and employment conditions over which we have no control exclude any damage claims arising from the use of our data and recommendations. Furthermore, we cannot assume any responsibility for any patent infringements which might result from the use of our information.