Neuburg Siliceous Earth

in water-based

acrylic clear coats for wood

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Contents

1 Introduction

Clear aqueous lacquers based on acrylate dispersions are primarily used for wood and furniture coatings. Contrary to the frequently applied classical solvent-based cellulose nitrate lacquers, the water-based systems represent environmentally highly friendly and physically drying systems which are more and more used as primers, topcoats and also as multi-layer lacquers in industrial as well as craftsmen's coatings.

By applying so-called „self-crosslinking" arcylate dispersions, the protective efficiency of the coatings on heavily used wood surfaces can further be improved.

According to the mode of preparation and application resp. the performance requirements and the wood substrate, the following properties (selectively) can be of importance:

- ✓ Drying behavior
- ✓ Sandability
- ✓ Blocking resistance
- ✓ Transparency - wood warmth („Anfeuerung")
- ✓ Gloss / Matting
- ✓ Abrasion and scratch resistance
- ✓ Water and chemical resistance
- ✓ Stain resistance

The present report will show inhowfar the property profile of aqueous wood lacquers can be optimized by the use of functional fillers based on Neuburg Siliceous Earth. For this, typical filler effects on processing, optical, mechanical and other relevant properties will be shown on the example of three different base formulations.

2 Experimental

2.1 Base formulations

The starting point of the trials was a wood filler and two universally used multi-layer lacquers based on binders and guide recipes from Alberdingk Boley.

The wood filler serves to fill pores and other irregularities on the wood surface. Furthermore, it can be used as a primer and base for the subsequent application of a coating system. The major requirements here refer to high filling power and rapid good sandability.

In multi-layer lacquers, generally the primer coating and the following topcoat - sometimes with an intermediate layer - will be based on the same formulation. For better adhesion between the layers and film leveling after drying, the surface will be subject to a fine grinding step. Compared with the filler alone, in these systems, apart from the optical properties, above all mechanical and chemical resistance is of importance.

In all coatings, the addition of film forming additives - organic cosolvents in low concentrations - improves the coalescence of the binder polymer particles which improves the film formation also at lower temperatures.

A detailed summary of the ingredients of the studied formulations will be found in the „Results“ section.

2.2 Neuburg Siliceous Earth

Neuburg Siliceous Earth, as exploited near Neuburg on the Danube, represents a naturally formed mixture of corpuscular Neuburg silica with lamellar kaolinite: a loose conglomerate that cannot be separated by physical methods. The silica portion, as a result of its natural formation, has a rounded grain shape, and consists of aggregated primary particles about 200 nm diameter.

Fig. 1 shows the most important characteristics of the siliceous earth grades studied. The two non-treated product variants differ above all in their fineness, which is reflected for Sillitin Z 89 in slightly higher figures for the oil number and the specific surface area.

For surface-treated filler, an Aktisil grade based on Sillitin V 88 was tested. Aktisil MAM has a surface modified with a methacryl functional group.

Filler Characteristics

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		Neuburg Siliceous Earth		
		Non surface treated		Surface treated
		Sillitin Z 89	Sillitin V 88	Aktisil MAM
Density	[g/cm³]	2.6	2.6	2.6
Particle size d_{50}	[µm]	1.8	4	4
Particle size d_{97}	[µm]	8	18	18
Oil absorption	[g/100g]	55	45	45
Spezific surface area BET	[m²/g]	11	8	7

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Fig. 1

Preliminary trials have confirmed that Neuburg Siliceous Earth is able to fulfill the major optical requirements of incorporation in a clear lacquer. The markedly higher transparency of the coatings as compared with classical competitive fillers shows off even at high loadings (*Fig. 2*, right) and a PVC of 25 %, which corresponds to a weight portion of 20 % calculated on the total formulation (wet).

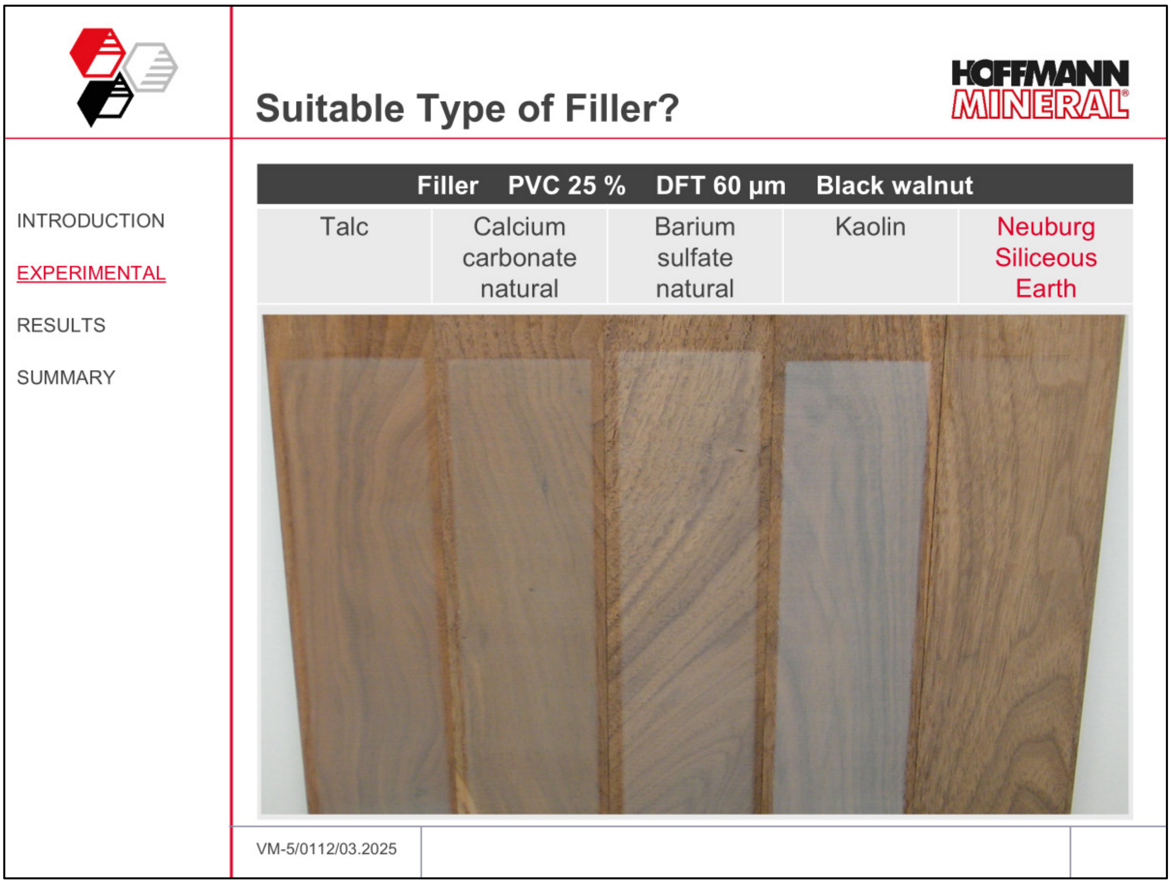

Fig. 2

2.3 Preparation of batches, application and tests

Mixing of the raw materials was carried out in a dissolver equipped with a toothed disc. The film formation additives and additional water were premixed separately and were added slowly. The Neuburg Siliceous Earth, after adding, was incorporated by dispersing for 15 min at a peripheral speed of 5.2 m/s.

After completing the formulations and maturing over night, a part of the wet paint was separated for assessing the storage stability. For testing the dry films, one layer was applied with the aid of a doctor blade predominantly at a gap clearance of 150 µm corresponding to 30-35 µm dry film thickness. The substrates were selected according to the following tests:

Contrast cardboard

- Gloss, DIN EN ISO 1522
- Abrasion resistance, Taber CS17, according to ASTM 4060

Black Leneta film

- Sandability, manual test with abrasive paper fixed to hammer head
- Blocking resistance; according to ASTM 4946

Q Panel type R 48

- Drying behavior, Erichsen method (no film damage with sliding wire bow)
- Hardness, Pendulum damping test according to Koenig, DIN EN ISO 1522

Wood substrate beech resp. American walnut

- Transparency, visual assessment
- Water, chemical and stain resistance, DIN EN 12720, DIN 68861-1

For the tests on wood, the multi-layer lacquers were applied in three layers with intermediate drying periods of 3-4 hours and subsequent P220 sanding.

If not indicated otherwise, the drying and conditioning of the coating films as well as the tests took place in an air-conditioned laboratory at 23 °C and 50 % relative humidity.

3 Results

Note: The tests reported in the experimental section represent a selection of the studies carried out within the framework of this project. Results which are particularly relevant for the application will be discussed in detail and this above all for high filler loadings. Further properties have been considered in assessing the overall performance of the systems.

3.1 Filler

Fig. 3 summarizes the details of the starting-point wood filler formulation. The binder is a non-self-crosslinking acrylate dispersion with a minimum film formation temperature (MFFT) of 40 °C. By adding butyl glycol the MFFT can be lowered and a sufficient film formation realized already at room temperature.

As representatives of Neuburg Siliceous Earth, Sillitin Z 89 and Sillitin V 88 were incorporated into the control formulation at the high loading of 20 parts by weight. The addition of a dispersing / wetting agent in these variants favors a good incorporation of the filler into the mixture of water and cosolvent.

The water content of the filled formulations furthermore was increased in a way to obtain a comparable dry film thickness at the application of identical lacquer volumes. Under these conditions (comparable solids content volume) during the drying process the evaporating water portion will become identical and allow an objective assessment of drying times as well of the sandability after defined periods of time.

Filler Formulations

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	Control		Same solid volume	Neuburg Siliceous Earth
	%	pbw		pbw
Butyl glycol	7.7	5.0		5.0
Water demineralized	13.3	8.6	3. increased	
Tego Dispers 750 W	----	----	2. added	
Filler	----	----	1. added	20.0
Alberdingk AC 31	77.3	50.0		50.0
Byk 024	0.8	0.5		0.5
Rheovis PU 1214	0.9	0.6		0.6
Total	**100.0**	**64.7**		**100.0**
Solid content w/w	[%]	39.8		46.9
Solid content v/v	[%]	**37.2**		**37.2**
PVC	[%]	0		25.3

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Fig. 3

3.1.1 Drying and Sanding

Similar to the control batch, with Sillitin Z 89 or Sillitin V 88 results a comparable period of time, i.e. about 20-25 minutes, after which the film (dry thickness 30 µm) comes out no longer damaged by an applied and pulled-through sliding wire bow. Analogous tests with higher film thickness gave equivalent results with Neuburg Siliceous Earth. Neuburg Siliceous Earth, therefore, does not significantly affect the drying rate, which seems to depend in the first place on the amount of water in the drying film.

As the wet lacquers compounded with Neuburg Siliceous Earth (in particular with Sillitin V 88) show a lower viscosity than the control batch, there exist chances for optimizing via a reduction of the water portion. This way, an appropriate adjustment towards the rheological profile of the filler-free formulation can be realized, along at the same time with more rapid drying (cf. also the chapters Multi Layer 1 and Multi Layer 2). The higher solids content volume additionally has a positive effect on the filling behavior and layer buildup of the coating.

For the assessment of sandability, the blow side of a hammer was covered with abrasive paper of grain size 220, which then served for a sequence of 50 double rubs under a load of 125 g/cm^2 on the tested coating surface.

In manual tests, Sillitin Z 89 and Sillitin V 88 impress with significantly earlier sandability and markedly finer sanding dust (*Fig. 4*). Already after a drying time of 40 minutes an excellent result is obtained with grinding through of the coating, whereas the control without filler even after a threefold longer time shows unsatisfactory sandability via formation of little rolls.

The favorable properties of Neuburg Siliceous Earth can equally be verified at higher dry film thicknesses of 60 µm.

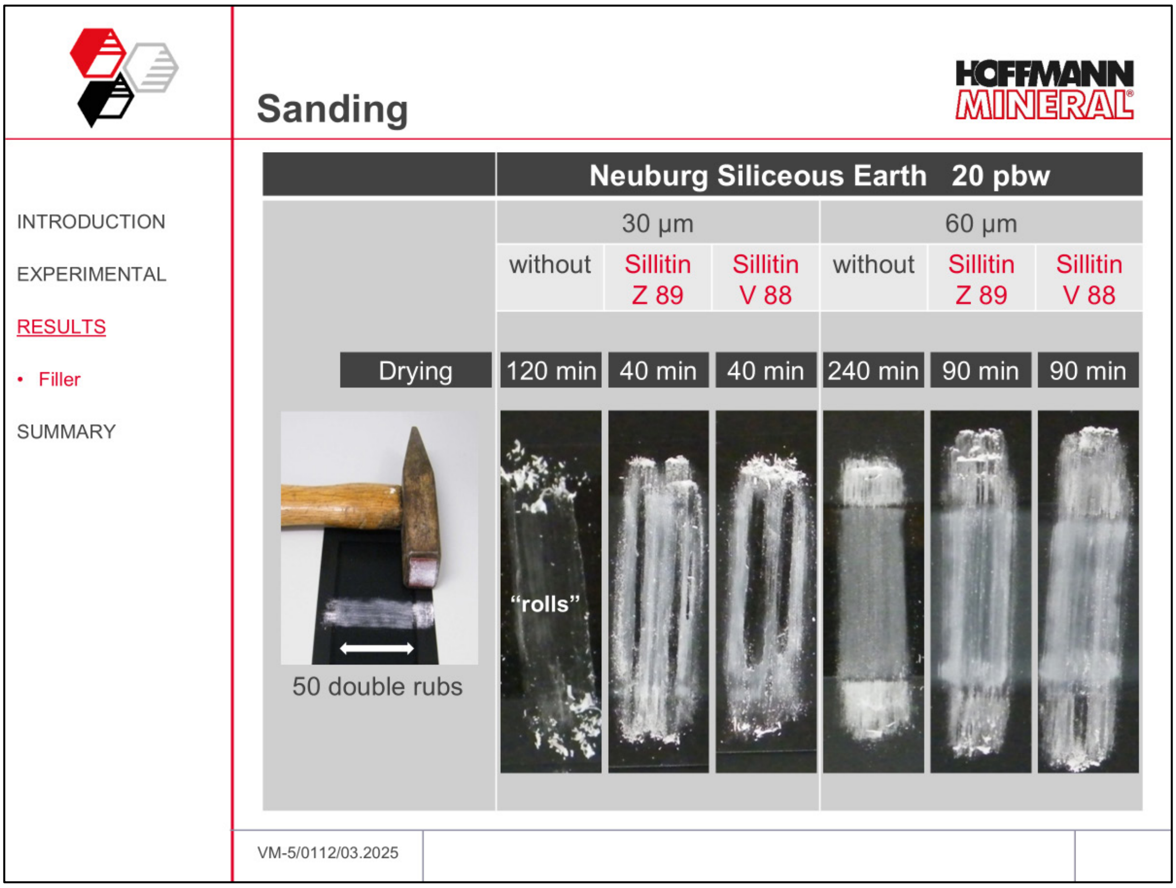

Fig. 4

3.1.2 Blocking resistance

A high resistance against blocking is an important property for avoiding damages or tackiness of the applied coating under pressure or when stacking at the end of the coating process.
In order to test this property, lacquer films were applied to Leneta foil, after 60 minutes drying time cut in strips, fixed with the coated surfaces against each other and pressed for 1 hour with a load of 100 g/cm³. Specific indications relative to variations in the conditioning and loading conditions can be seen in *Fig. 5.*

Neuburg Siliceous Earth compared with the filler-free compound at an early time «takes out» the tack und thus avoids large-scale tearing phenomena as they can be seen with the control batch. By partially forced drying in an circulating-air oven at elevated temperature the coatings overall become more highly resistant against blocking, and with Sillitin V 88 even reach the state of complete freedom from blocking.

An increase of the temperature to 40 °C during the loading cycle of the control compound gives rise to a marked negative effect of partial tear-off. By contrast, the Sillitin grades Z 89 and even more so V 88 give evidence of a much lower sensibility and markedly better blocking resistance.

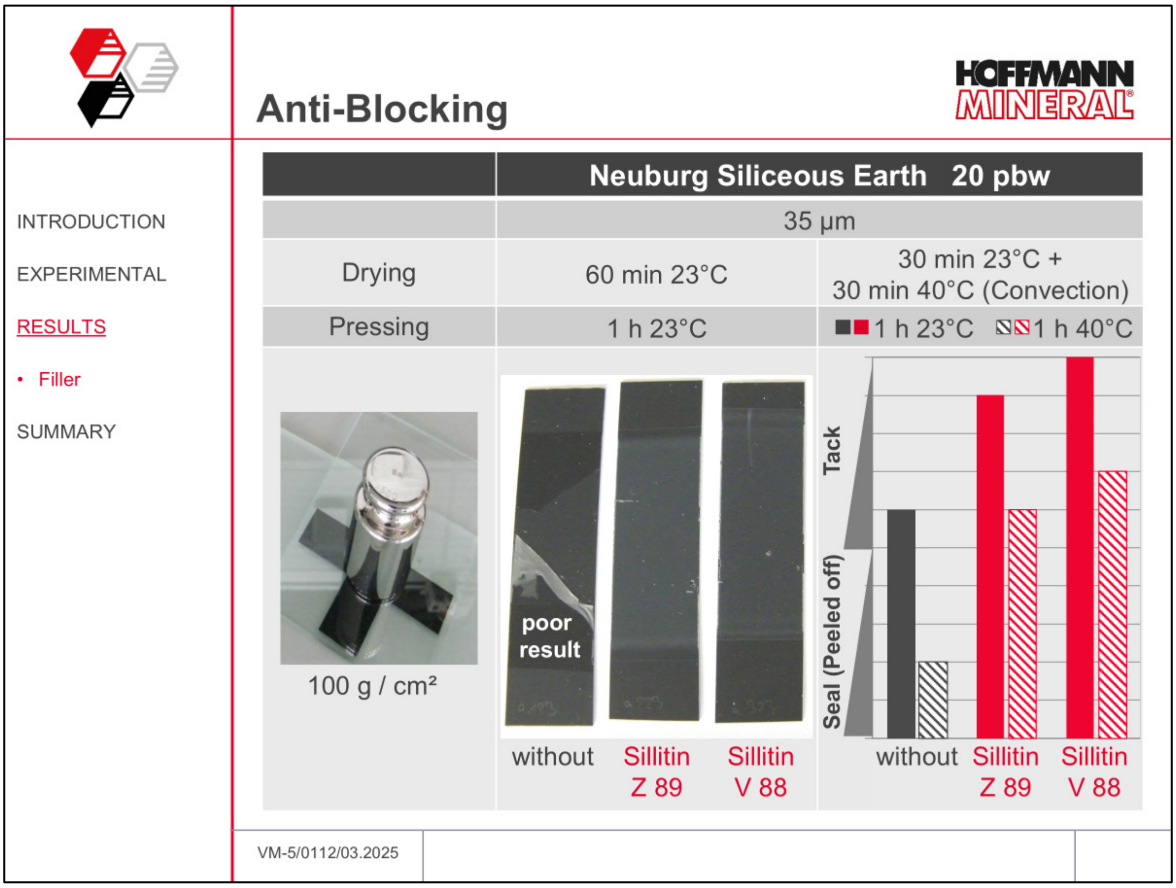

Fig. 5

3.1.3 Overall performance

Fig. 6 compares the results of the study in the wood filler formulation.

The batches with Neuburg Siliceous Earth throughout offer good storage stability. While Sillitin Z 89 gives rise to an excellent result without any sediment, the slight sedimentation tendency with Sillitin V 88 can, if necessary, be compensated by some addition of a layered silicate thickener (e.g., Laponite RD) or of fumed silica.

Comparable with the drying properties, the hardness level of the coatings is maintained when working with Neuburg Siliceous Earth.

The Neuburg Siliceous Earth grades through their considerably better sandability and an early high blocking resistance are able to impart marked benefits already during the application process. Further tests have shown that a comparable property profile cannot be obtained by just using conventional grinding aids such as zinc stearate, but there is room for optimizing by using them as additional additives (1-2 pbw).

The moderate reductions in transparency primarily are due to the strong matting effect of the filler and the resulting stronger diffuse light scattering on the micro-rough coating surface. Continued removal of the filler by sanding and overcoating with a filler-free clear lacquer as topcoat will eliminate this effect and lead to the optically favorable appearance of the unfilled system.

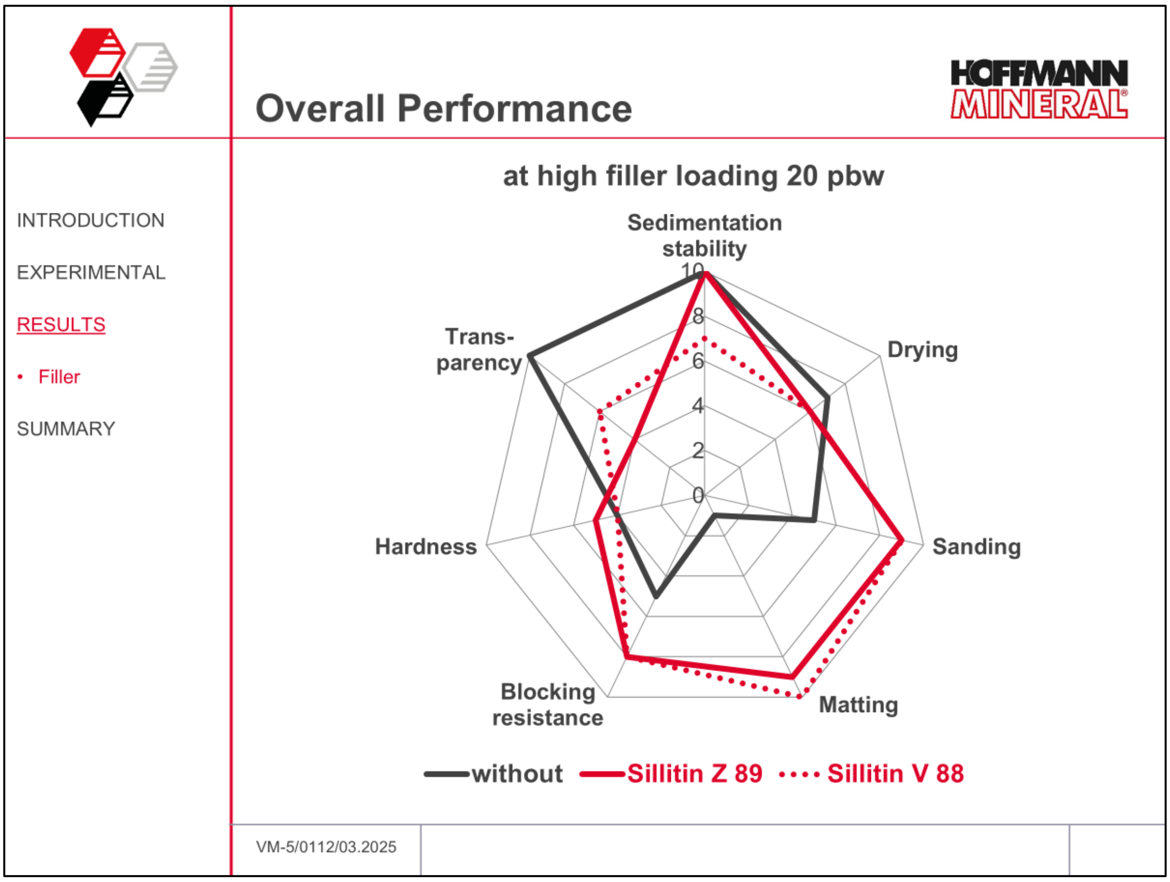

Fig. 6

3.2 Multi Layer 1

In practice, the viscosity for the application of a multi-layer lacquer is frequently specified as the flow time out of a defined cup. When coating furniture, or equally at the general coating of wood substrates for interior application, the viscosity will be adjusted to a corresponding adequate level according to the coating process (spreading, rolling, spraying, flow-coating, dipping) and to the shape of the object (rather flat: table plates, furniture frontsides, or three-dimensional with vertical areas, e.g. chairs).

The flow time of 75 seconds in *Fig. 7* already is situated in a somewhat higher range, and allows a certain sag safety when coating vertically positioned wood surfaces.

Alberdingk AC 25381 is a binder based on polymer particles with a hard core and a surrounding softer phase (core-shell technology). The core assures early mechanical stability and good blocking resistance. The soft phase favors the flow of the polymer particles and the interdiffusion of the polymer chains during the drying step and thus facilitates the film formation.

Fig. 7 summarizes the formulation variants based on the control recipe.

The matting effect of Neuburg Siliceous Earth in the filled batches allows to eliminate the matting agent, here Acematt TS 100. The increased viscosity with Neuburg Siliceous Earth under low shear conditions makes a cost-favorable reduction of the used rheological additive possible. At the same time, in order to adjust a comparable efflux time and thus viscosity increase at medium share, a lower amount of water can be added, which also positively helps towards a higher solids content.

Multi Layer 1
Formulations

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	Control	Efflux time 75 s DIN-4 Cup	Neuburg Siliceous Earth	
	pbw		pbw	
Alberdingk AC 25381	74.5		74.5	74.5
Tego Foamex 822	0.6		0.6	0.6
DPM	5.0		5.0	5.0
DPnB	2.0		2.0	2.0
Water demineralized	13.8	4. reduced	6.0	7.5 – 8.0
Acematt TS 100	0.5	2. replaced	----	----
Filler	----	1. added	10.0	20.0
Aquacer 539	3.0		3.0	3.0
Byk 346	0.3		0.3	0.3
Rheovis PU 1214	0.3	3. reduced	0.15	0.15
Total	**100.0**		**101.55**	**113.05 - 113.55**
Solid content w/w [%]	37.7		46.4	50.3 – 50.6
PVC [%]	0.7		10.6	19.1

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Fig. 7

3.2.1 Drying and Sanding

As a result of the higher solids and lower water content, the necessary drying times in comparison with the filler-free control formulation can be decreased. In particular with high filler loadings drying times will be shorter by 30 to 40 % vs. the control, which means the drying step will take up only 17 to 20 vs. 29 minutes.

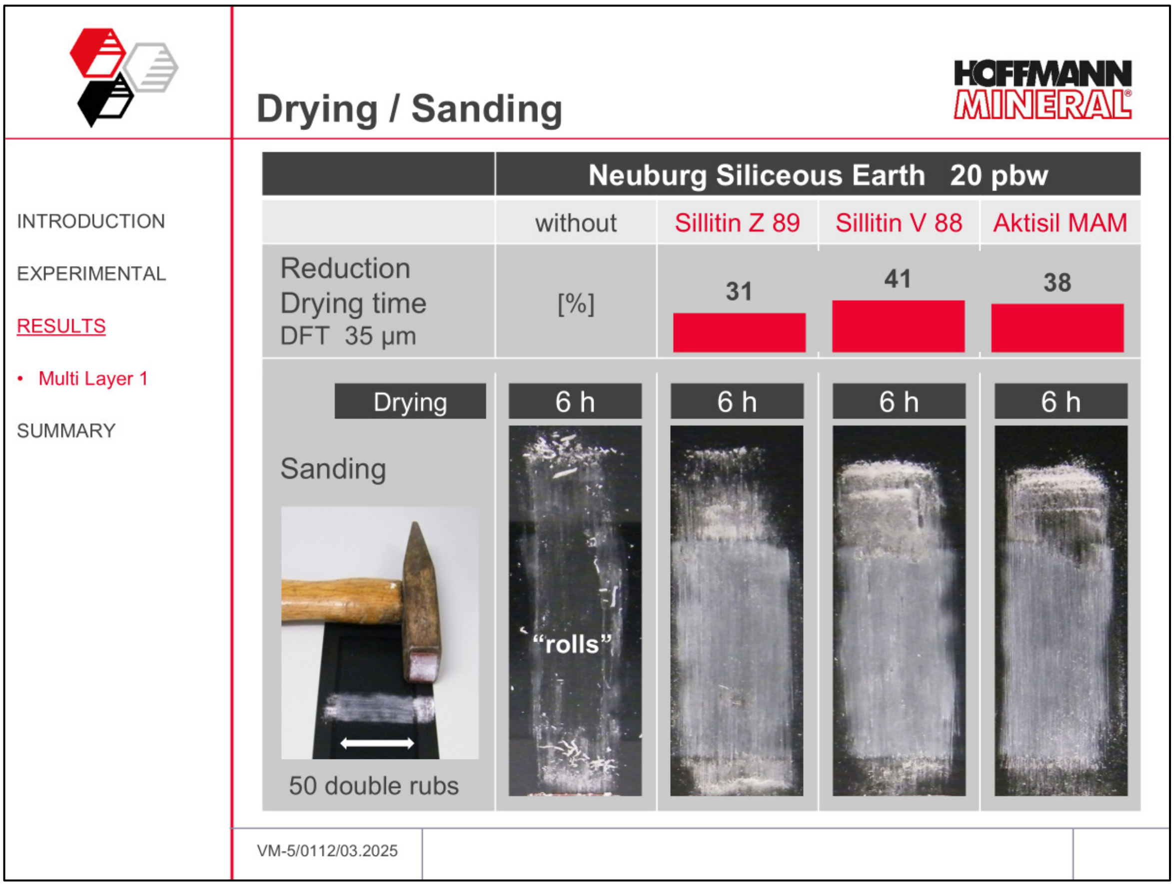

Fig. 8

Freshly applied films with Neuburg Siliceous Earth after already 6 hours offer an excellent sandability as evident from very fine grinding dust. The unfilled control batch in this respect shows a more elastic behavior. As a result, the coating during the sanding step becomes increasingly hotter and gives rise, as seen in *Fig. 8* even after drying for 24 hours, to a sticky, roll-like material removal.

3.2.2 Optical Appearance

Fig. 9 in the upper part demonstrates graphically the strong matting effect of Neuburg Siliceous Earth. The effect with Sillitin Z 89, because of its higher grain fineness and the resulting lower roughness of the film surface (as confirmed under the microscope), comes out somewhat less pronounced than with the two other siliceous earth grades. The matting effect combined with very high film transparency imparts a decently natural appearance to the surface, close to allegedly uncoated wood. On dark wood substrates, however, a certain brightening effect as a result of the strong light scattering at the coatings surface must be taken into account.

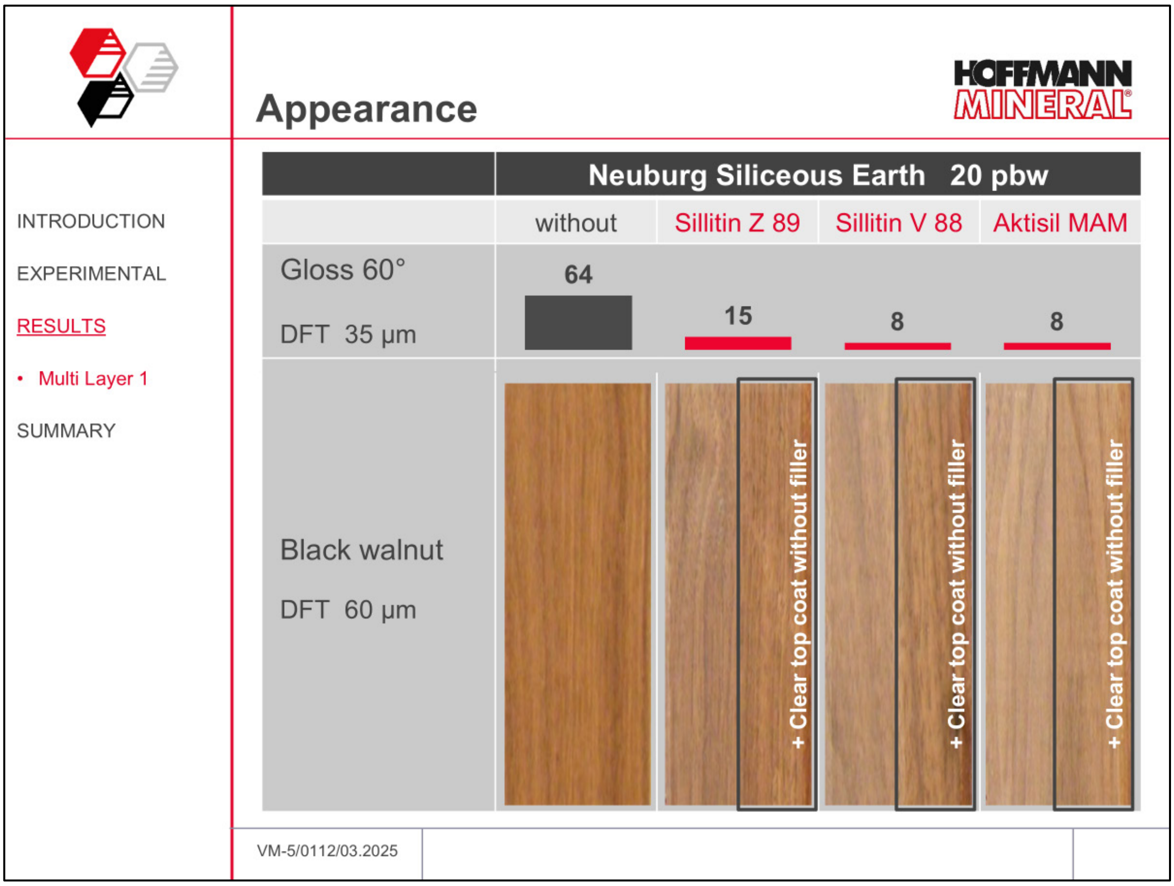

Fig. 9

If for optical reasons a higher gloss is desired, according to *Fig. 10* a reduced filler loading is suggested of preferably Sillitin Z 89, or also an overcoat with the filler-free formulation. At the same time, the increasing wood warmth will bring out the color and grain of the wood, and this gives a particularly positive effect on dark woods.

In comparison, Sillitin V 88 and also Aktisil MAM ensure the optimum compromise between appearance and transparency at a specified low gloss level.

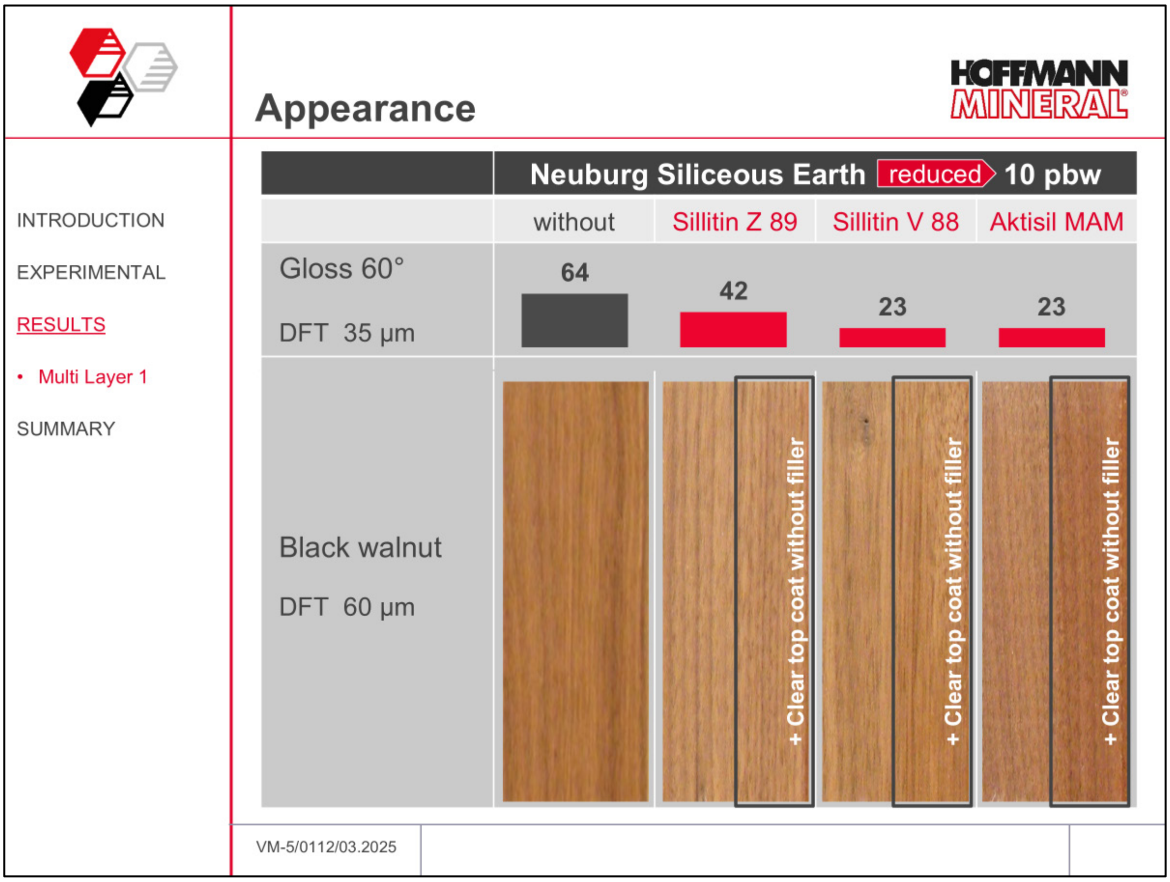

Fig. 10

3.2.3 Water, Chemical and Stain resistance

The resistance towards the exposure to cold liquids or pasteous substances overall has to be judged very good, and largely fulfills the high requirements of DIN 68861-1 Class 1B.

Fig. 11 illustrates the results at different filler loadings as a function of the tested material and loading time on beech wood.

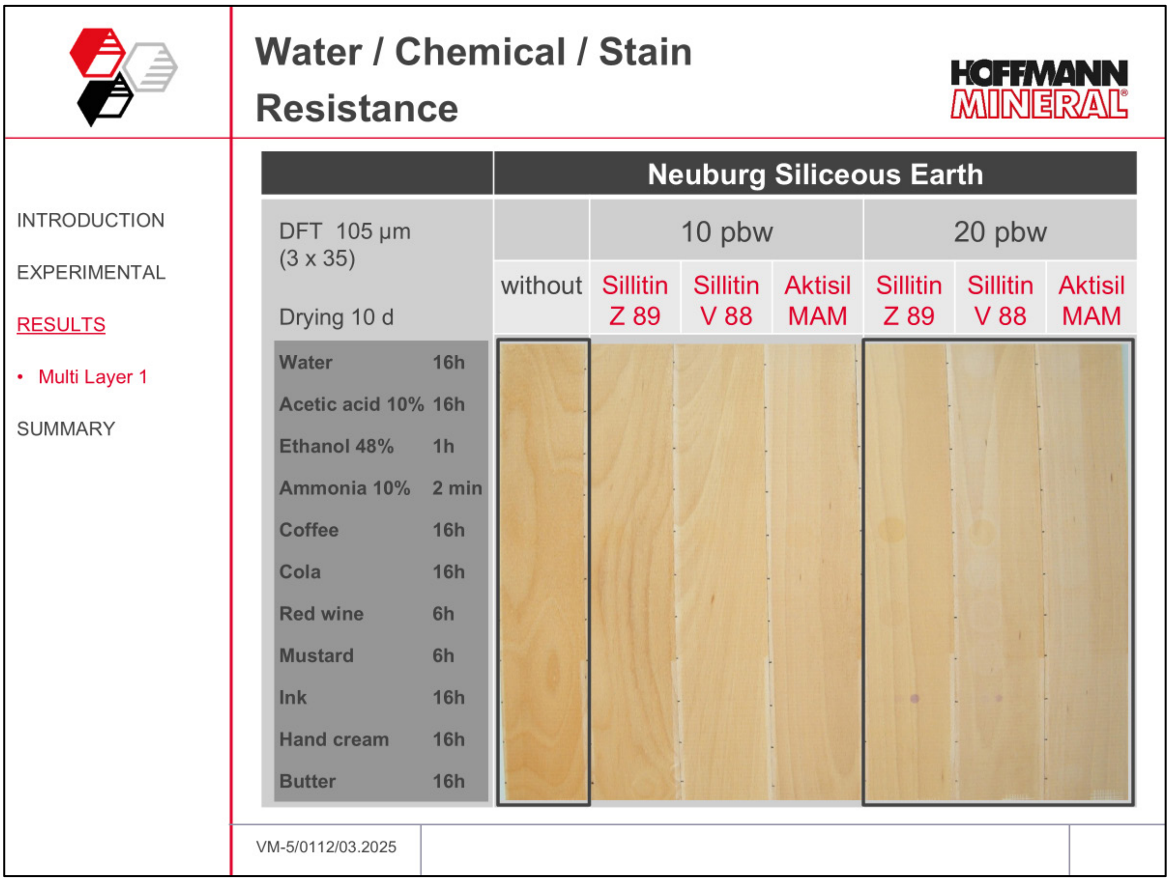

Fig. 11

Even at high filler loadings an excellent resistance against water and cleaning chemicals is obtained as well as against fatty substances (hand cream, butter). Only with high filler additions of 20 pbw and with strongly coloring substances such as coffee and ink, small visible traces are left behind. The use of the methacryl functionalized treated siliceous earth Aktisil MAM, however, prevents even at loadings of 20 pbw any coffee or ink stains.

3.2.4 Overall performance

The base formulation can be improved through the use of Neuburg Siliceous Earth with respect to important processing properties, and can be adjusted via the filler loading relating to optical requirements such as gloss, transparency and appearance. Filler specific differences are shown in *Figs. 12/13*: Compared with Sillitin Z 89, the coarser Sillitin V 88 or Aktisil MAM has a slightly positive effect on transparency and matting, along with a slightly negative effect on water and chemical resistance. Abrasion resistance and stain resistance against coloring materials, however, are improved with the studied coarser Neuburg Siliceous Earth grades, and further optimized through the surface modification of Aktisil MAM.

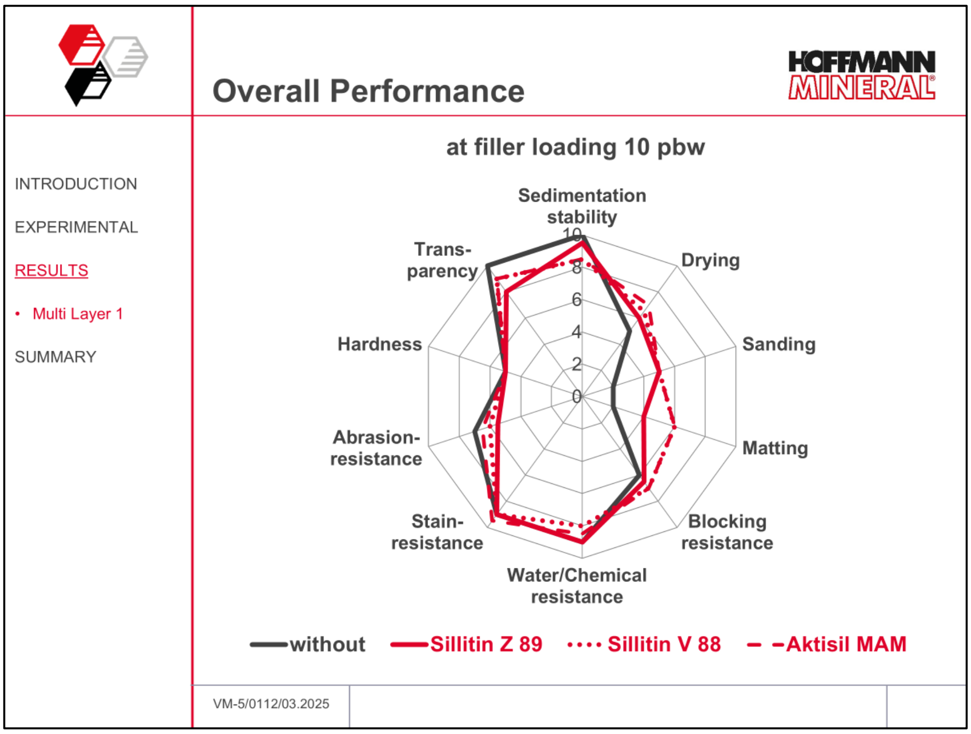

Fig. 12

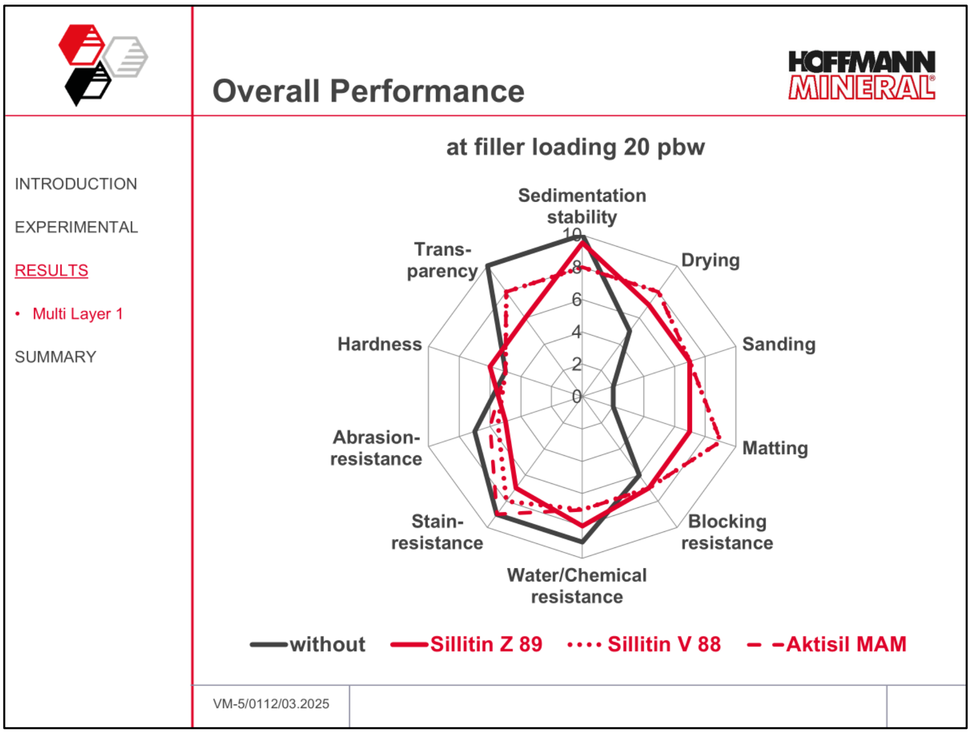

Fig. 13

3.3 Multi Layer 2

The second formulation included in the study of filler effects in multi-layer lacquers again is based on a self-crosslinking binder, but this time without core-shell structure. *Fig. 14* gives the details of the starting point recipe which was used as a control reference for the variants with Neuburg Siliceous Earth.

Compared with Alberdingk AC 25381, the grade AC 2514 is characterized by a higher minimum film formation temperature and hardness along with markedly lower viscosity. The cup flow time, therefore, comes out overall at rather low levels. In the correspondingly adjusted formulations with Neuburg Siliceous Earth (*Fig. 16*), a comparable level can be reached by completely eliminating the rheological additive and reducing the added amount of water.

Multi Layer 2
Formulations

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	Control	Efflux time 20 s DIN-4 Cup	Neuburg Siliceous Earth	
	pbw		pbw	
Alberdingk AC 2514	79.4		79.4	79.4
Byk 024	0.8		0.8	0.8
Butyl diglycol	6.0		6.0	6.0
Butyl glycol	2.0		2.0	2.0
Water demineralized	7.5	4. replaced / reduced	----	4.0
Acematt TS 100	0.5	2. replaced	----	----
Filler	----	1. added	10.0	20.0
Ultralube D 816	3.0		3.0	3.0
Byk 346	0.4		0.4	0.4
Rheovis PU 1214	0.4	3. replaced	----	----
Total	**100.0**		**101.6**	**115.6**
Solid content w/w [%]	37.7		46.3	49.1
PVC [%]	0.7		10.6	19.4

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Fig. 14

3.3.1 Drying and Sanding

In analogy to the earlier results, the reduction of the water portion in the formulations with Neuburg Siliceous Earth leads to a drying time shortened by 30 to 35 % (*Fig. 15*, upper part: control batch 29 min, siliceous earth variants 19-20 min).

The higher hardness of the binder already in the unfilled batches allows an early sandability with, however, unsatisfactory material removal. The latter is markedly improved when working with Neuburg Siliceous Earth, which combines early and good sandability.

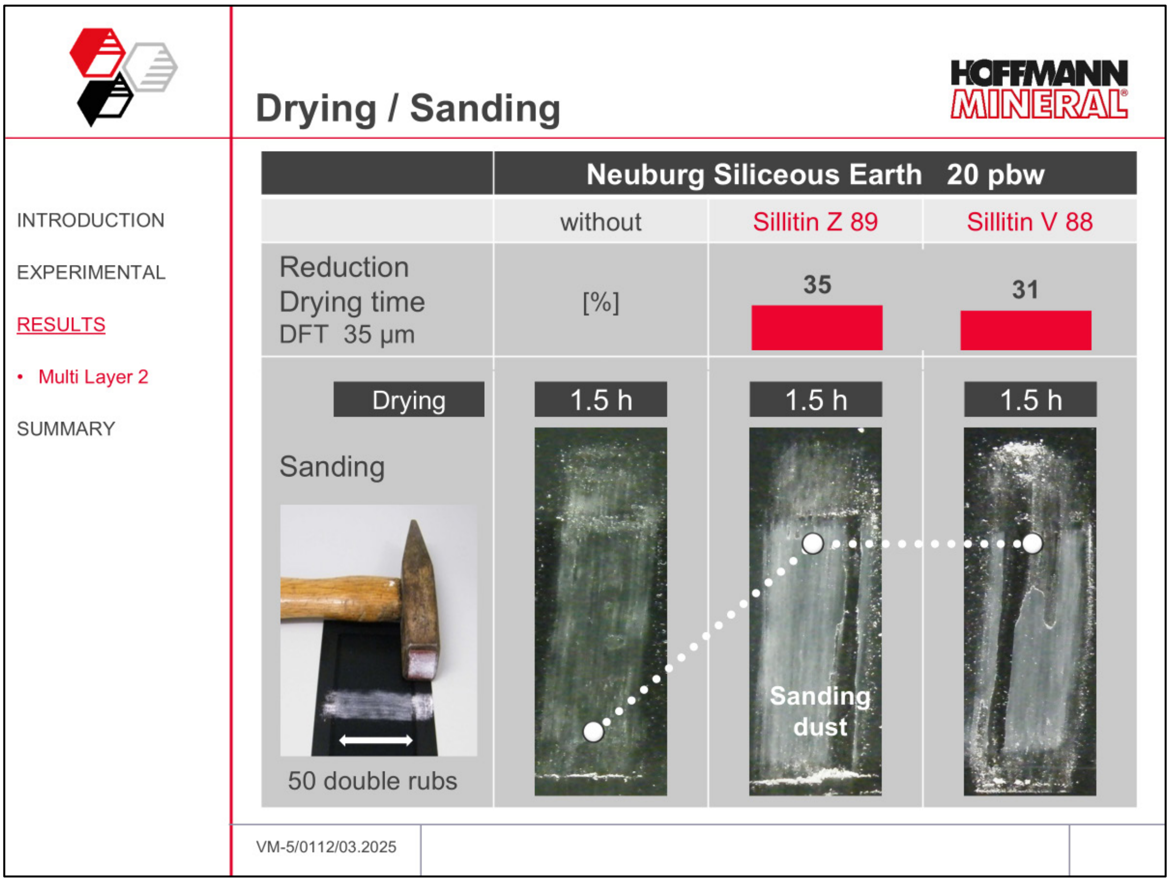

Fig. 15

3.3.2 Optical Appearance

In the Multi-layer Lacquer 2 the filler exerts a particularly marked effect on the application properties. The comparably high loading of 20 pbw reduces the transparency (*Fig. 16*) with the result of a slight glazing effect on a definitely matted surface. Overcoating with an unfilled clear lacquer results in a glossy surface with an optically comparable appearance.

The reason for the reduced transparency is probably due to a more difficult film formation, which primarily goes back to the different polymer characteristics of the binder (no core-shell morphology, higher MFFT and hardness). Under these aspects the selected filler loading already appears so high that it goes beyond a critical limit with respect to ideal coalescence and polymer entangling at the drying of the film.

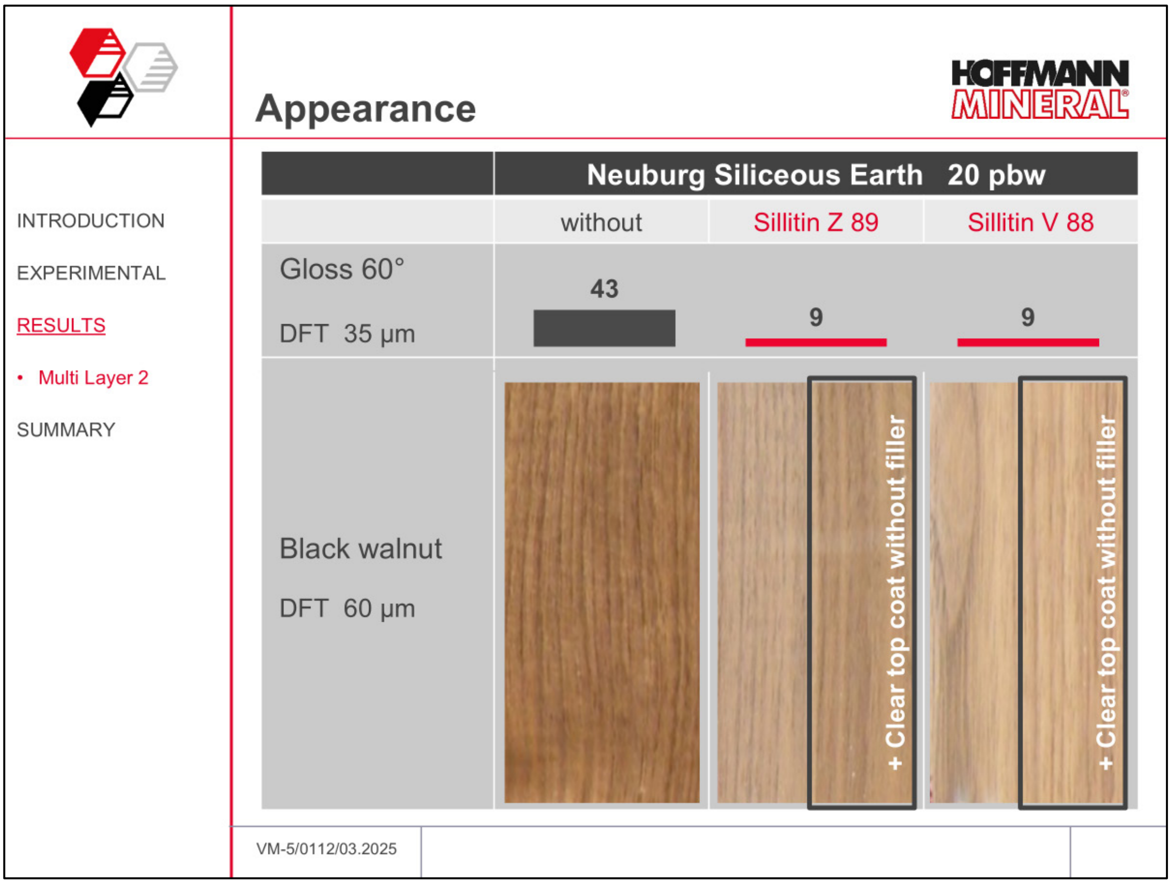

Fig. 16

In order to obtain good transparency also on dark wood substrates, according to the requirements it looks recommendable to reduce the filler loading in this coating system to 10 pbw maximum, as illustrated in *Fig. 17*. At the resulting moderate matting effect, the optical appearance and the wood warmth and grain accentuation come out much better, which means close to the control formulation.

As an additional possibility to improve the transparency, the film formation additives can be varied by replacing butyl glycol with dipropylene glycol monomethyl ether (DPM).

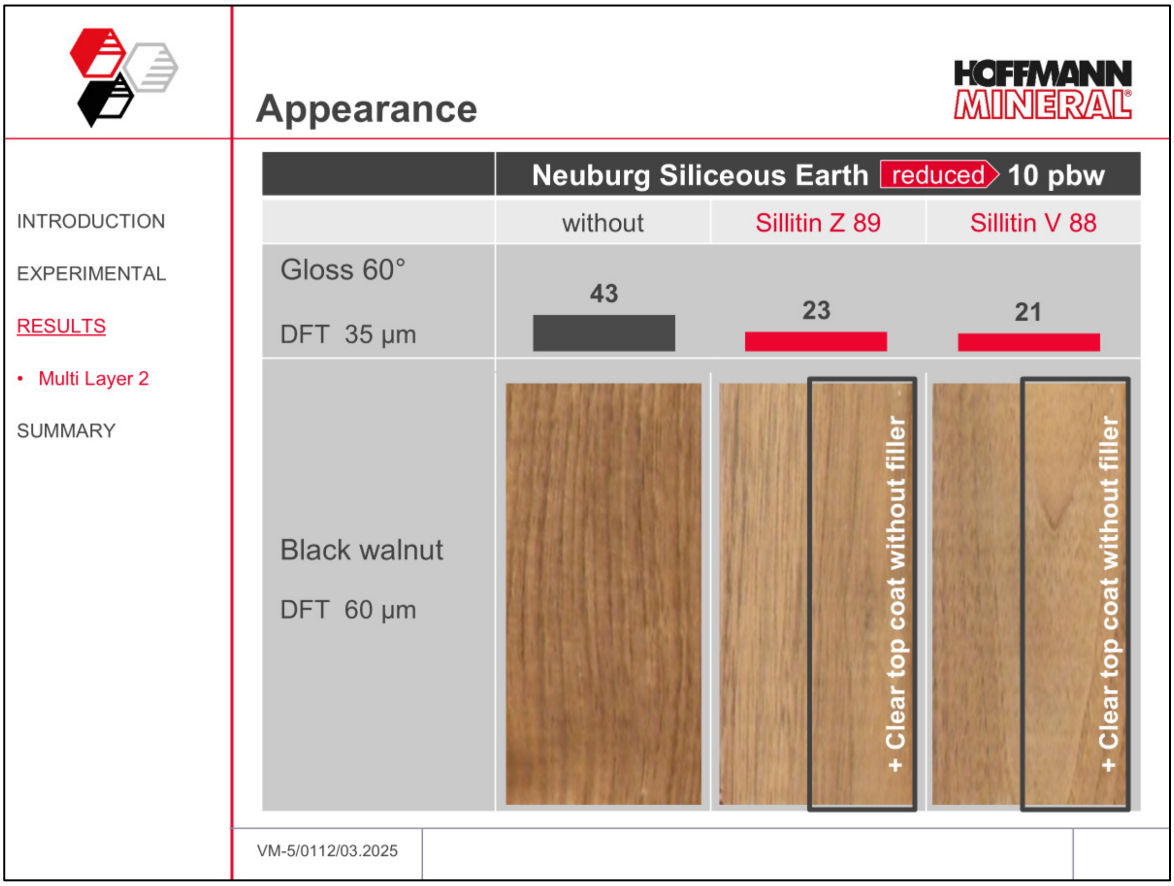

Fig. 17

3.3.3 Abrasion resistance

For the assessment of the mechanical resistance of coated wood and furniture surfaces when exposed to two-dimensional wear, the Taber abrasion test was carried out with CS-17 abrasive wheels under a weight load of 1 kg. As can be seen in *Fig. 18*, Neuburg Siliceous Earth gives rise to a significantly improved wear resistance along with a removal of abraded material lower by 30 % by volume, and this already at low loadings.

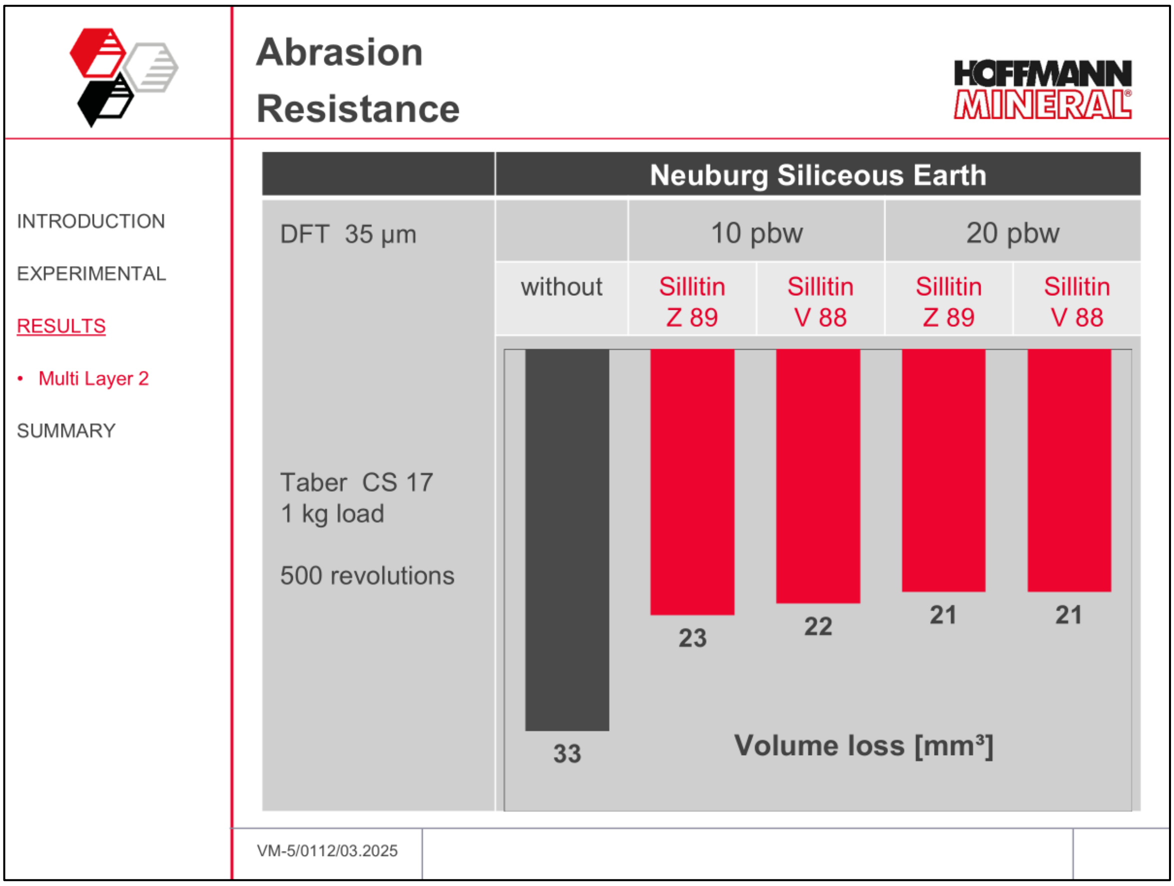

Fig. 18

3.3.4 Water, Chemical and Stain resistance

Compared with Multi-layer 1, the present study shows a higher sensibility of the coatings towards water or aqueous test liquids as well as ink. In *Fig. 19*, the slightly brightened or colored areas at high filler loadings optically point to first losses in protective effects. The better results on films with 10 pbw Neuburg Siliceous Earth can be seen as an indication towards more favorable film formation conditions and thus a higher barrier effect of the coating.

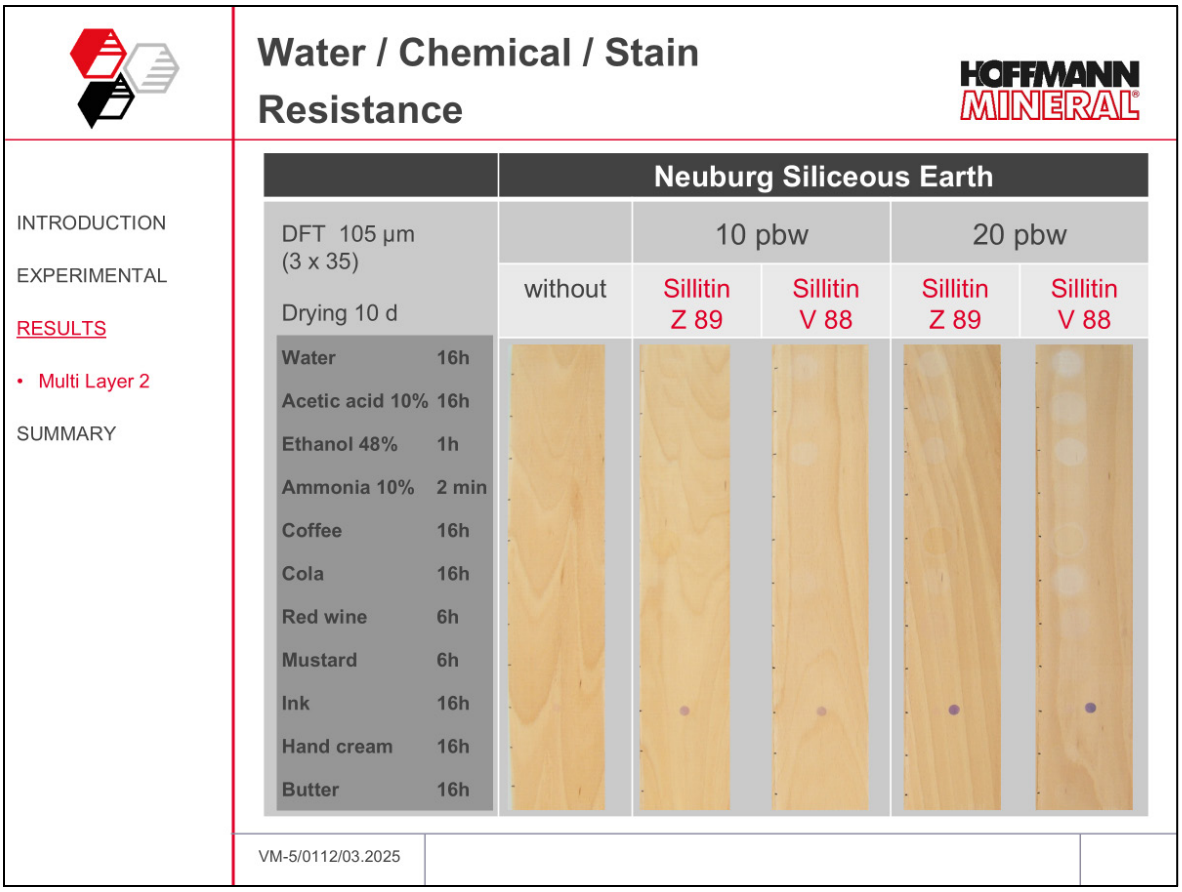

Fig. 19

Further studies were carried out with the objective of optimizing the performance at higher filler loadings. Really favorable results were obtained with the aqueous matting agent product Gloxil WW SL, which gives rise to high surface resistance properties already at early stage exposure.

Fig. 20 represents the results for coatings on American walnut or beech wood after 28 days of drying upon exposure to water and ink. The direct comparison with the coating with Sillitin Z 89 clearly gives evidence of the advantages obtained when using Gloxil WW SL. After 16 hours of exposure a resulting virtually flawless coating surface can be seen as a result of excellent resistance against water and ink.

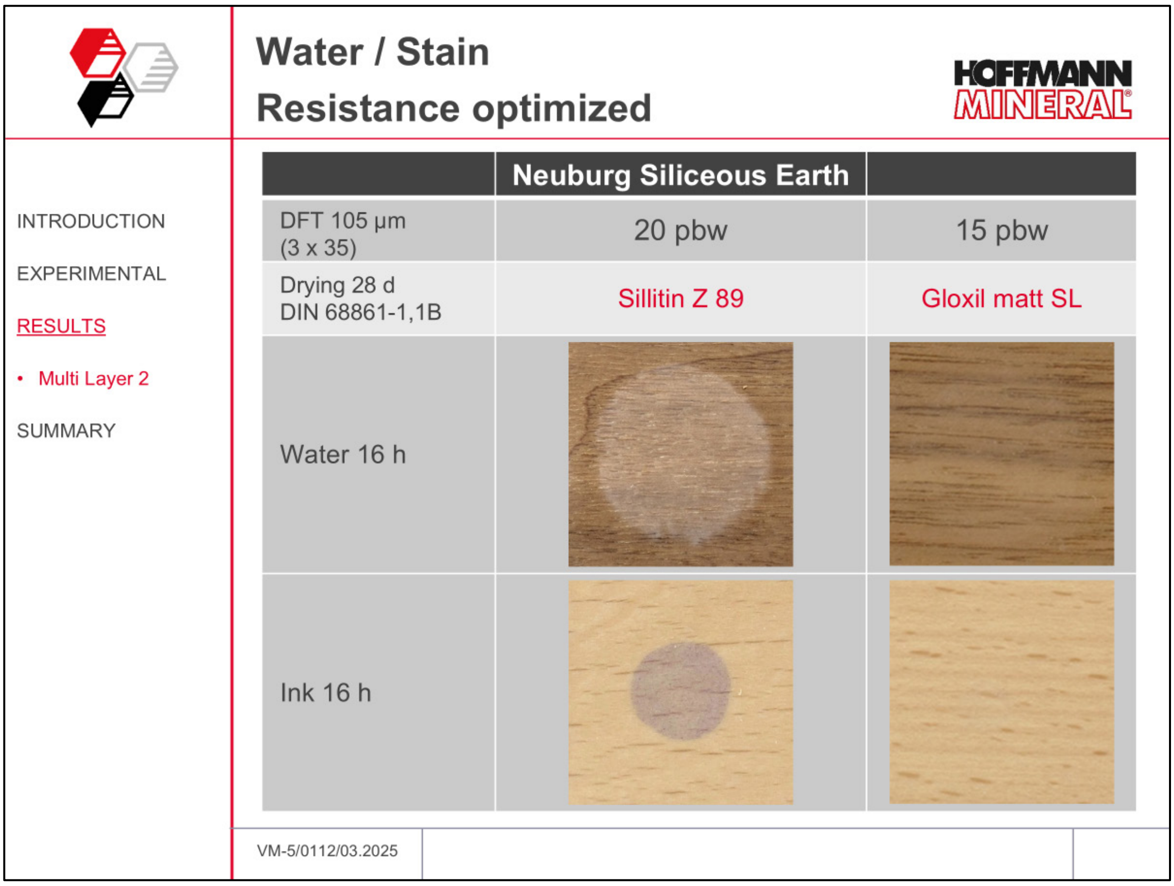

Fig. 20

Compared with Sillitin Z 89 and Sillitin V 88, Gloxil WW SL through very high transparency furthermore leads to an improved optical appearance on wood substrates, as can be seen in *Fig. 21*.

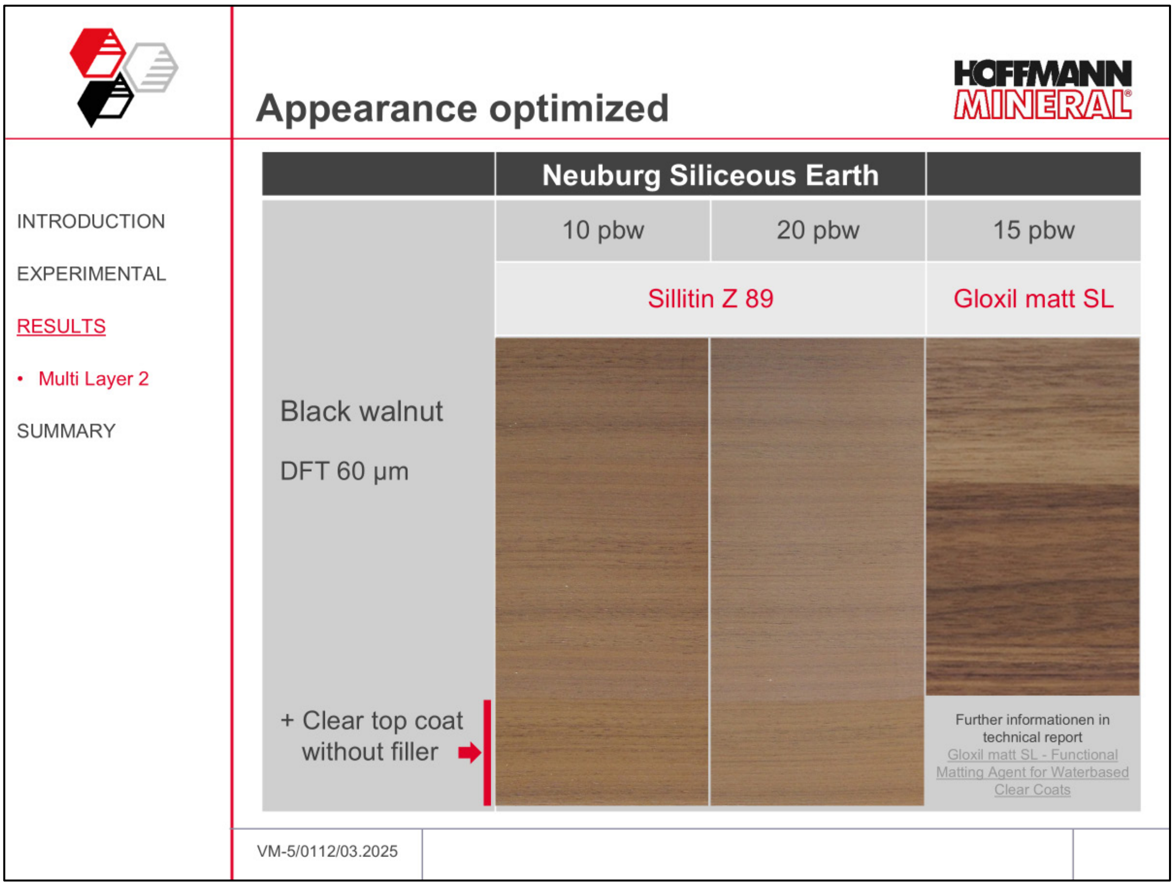

Fig. 21

In a direct comparison of the untreated siliceous earth grades, the relevant optical film properties are strongly dependent on the loading used. The basically good transparency of the coating film with Sillitin Z 89 (*Fig. 21* lower left, 10 pbw) at higher loadings is modified by the slight yellowish color of the filler, which makes the coating on American walnut look brighter. For higher loadings on dark wood substrates, therefore, the more color neutral Sillitin V 88 or Gloxil WW SL should be preferred.

3.3.5 Overall Performance

Figs. 22/23 summarize the performance profile of the tested grades of Neuburg Siliceous Earth in comparison with the filler-free control batch. Aside from the matting effect, the primarily more rapid drying and earlier sandability as well as improved blocking and abrasion resistance come out as real benefits. Slight disadvantages in transparency, stain, water and chemical resistance compared to the unfilled control compound can clearly be optimized by working with Gloxil WW SL (not dispayed here).

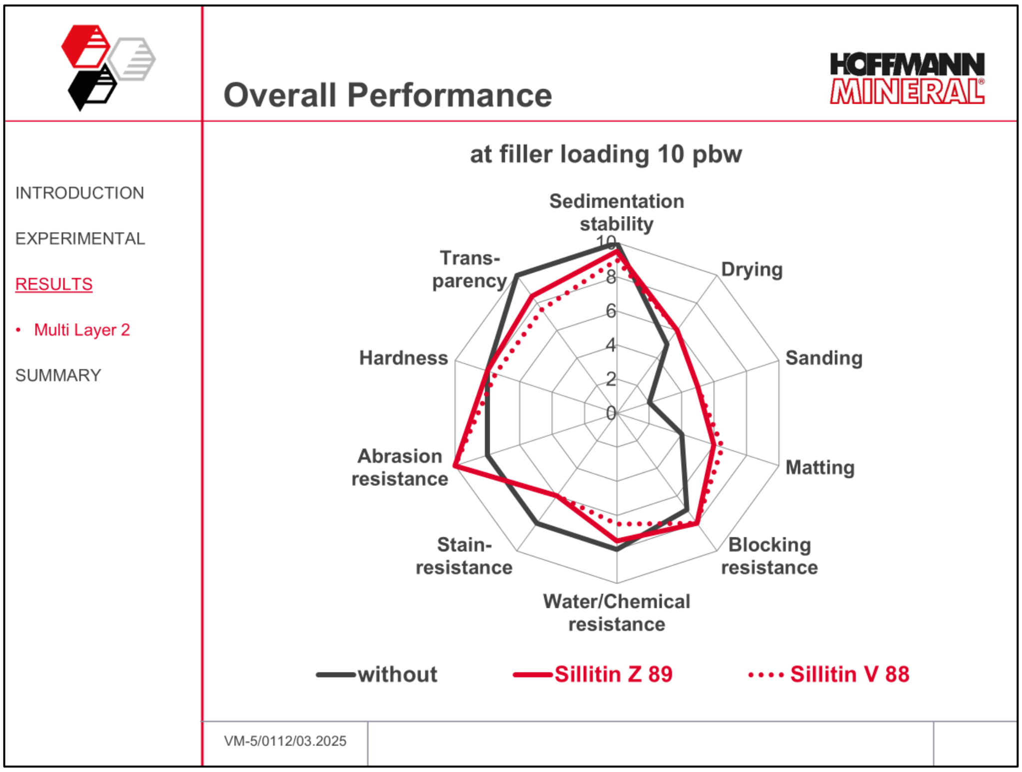

Fig. 22

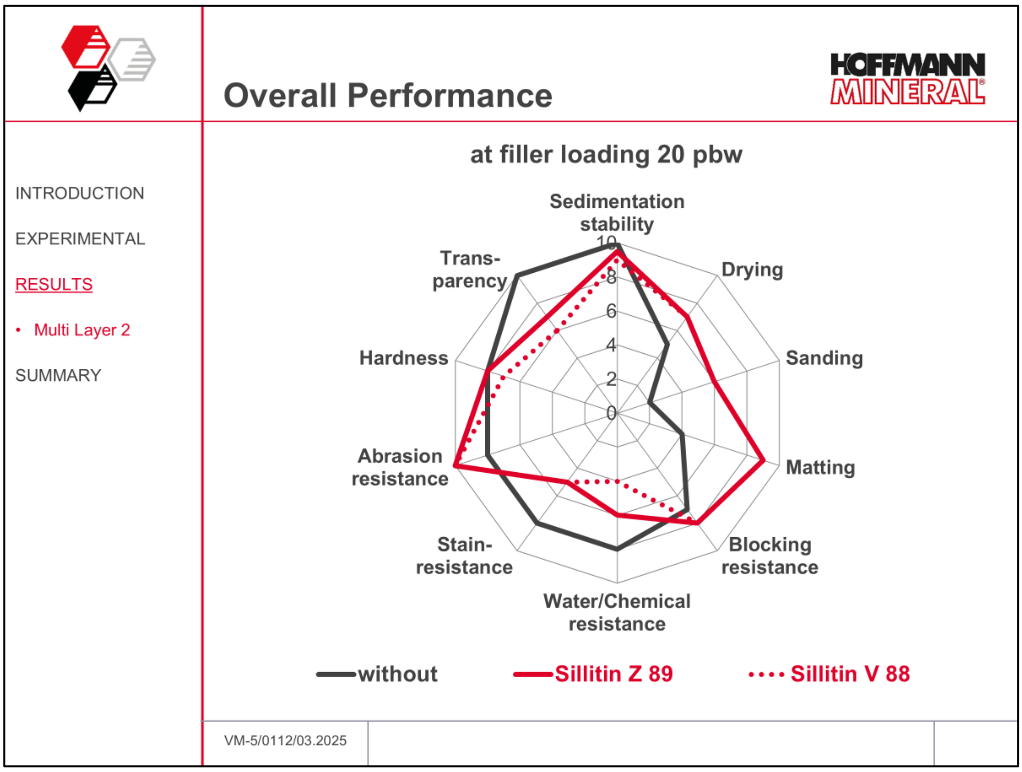

Fig. 23

4 Summary and outlook

The base formulation and in particular the selected binder heavily influence the performance level of the coating system and decidedly affect the extent of the relevant filler effect. With suitable selection and loading of Neuburg Siliceous Earth grades the performance profile of water-based acrylic clear coats for wood can further be improved.

In general terms, Neuburg Siliceous Earth offers the chance to formulate clear wood coatings with

- higher solids content
- reduced drying time
- markedly improved sandability
- outstanding matting effect

In particular, the following products call for attention:

Sillitin Z 89

- universal use
- moderate matting effect
- good overall performance profile at low loadings
- no sedimentation tendency

Sillitin V 88

- strong matting effect
- high transparency
- good blocking resistance

Aktisil MAM

- strong matting effect
- high abrasion resistance
- high stain resistance

As an additional product in Hoffmann Mineral´s product portfolio Gloxil WW SL provides

- subsequent matting level adjustment without laborious dispersion process
- very high water, chemical and stain resistance
- very high transparency, also at higher loadings

In particular, the special properties of Gloxil WW SL can be used with benefits in those cases where classical silica-based matting additives give rise to difficult film formation and reduced resistance properties of the coated films. Related test results in this respect can be found in the Technical Report „Gloxil WW SL - Functional Matting Agent for Water-based Clear Coats".