Neuburg Siliceous Earth

in UV-curing wood coatings:

Surfacer and top coat

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This study was carried out in cooperation with UCB Chemie (now Allnex). Thank you for the support provided.

1 Surfacer based on epoxy acrylate

1.1 Introduction

The objective of this study was to show how different fillers affect important properties of a surfacer for parquet floorings.

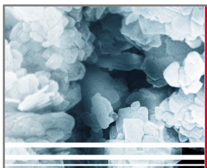
For this purpose a mineral filler should provide:

- high amounts in formulation
- viscosity increase, especially associated with an yield point (important for processing behaviour on a roller coater)
- good transparency
- high abrasion resistance
- cost reduction

As the most frequently used fillers for this product sector, two different types of talc, one precipitated barium sulfate and, for comparison, three products based on Neuburg Siliceous Earth were selected: Sillitin V 88, Sillitin Z 89 und Aktisil MAM.

1.2 Filler characteristics

The selection was based on particle shape, particle size and surface treatment.
The table summarizes the filler characteristics. The particle shape is determined by the type of mineral, where the two talcs differ in structure (intense lamellar to lamellar stacked) as well as in their chemical composition. With the exception of Talc 1 and Sillitin Z 89, the particle sizes are comparable: Talc 1 is coarser, and Sillitin Z 89 slightly finer. As a surface treated material Aktisil MAM was included, a grade based on Sillitin V 88 coated with methacrylic silane.



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Filler Characteristics HOFFMANN MINERAL

	Talc 1	Talc 2	Blanc Fixe	Sillitin Z 89	Sillitin V 88	Aktisil MAM
Mineral Description	Talc + others	Talc + others	prec. Barium-sulfate	Silica/ Kaolinite	Silica/ Kaolinite	Silica/ Kaolinite
Chemical Composition	55 % SiO_2 32 % MgO 5 % Al_2O_3	32 % SiO_2 31 % MgO 22 % Al_2O_3	99 % $BaSO_4$	82 % SiO_2 12 % Al_2O_3	88 % SiO_2 8 % Al_2O_3	88 % SiO_2 8 % Al_2O_3
Particle Shape	lamellar	lamellar, more stacks	rhombo-hedral	corpuscular aggregates and lamellar	corpuscular aggregates and lamellar	corpuscular aggregates and lamellar
Particle Size d_{50} [µm]	7.8	3.2	4.4	2	4	4
Particle Size d_{97} [µm]	27	17	16	9	18	18
Oil Absorption [g/100 g]	47	42	17	55	45	45
Density [g/cm³]	2.8	2.8	4.4	2.6	2.6	2.6
Surface Treatment	none	none	none	none	none	Methacrylic silane

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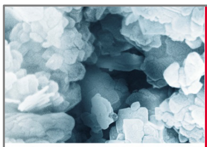
1.3 Base formulation

The base formulation was a guide formulation from UCB Chemie (now Allnex).



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	Parts by weight
Ebecryl 6040	42.4
OTA 480	50.8
Benzophenone	3.4
Omnirad BDK	3.4
Filler	variable X
Total	100 + X

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The filler was added additively to 100 parts by weight of the base formulation.

Due to the very different density of the fillers used, the fillers are given below by volume in order to ensure better comparability.

1.4 Application, tests and parameters

Viscosity

Rotation viscosimeter MC 1 (Anton Paar), 23 °C; recording of flow curves with increasing shear rate from 0 to 100 s^{-1}. The figure indicated represents the measured viscosity at 100 s^{-1}.

Yield point

Rotation viscosimeter MC 1 (Anton Paar), 23 °C; determined via continually increasing the shear stress up to a continuous flow movement of the material.

UV-curing surfacer

Mercury UV lamp at 80 W/cm; speed 15 m/min; two passes

Color shift

The color value b* (CIELAB Color system) of the film was measured on a white substrate with a Luci 100 color meter; dry film thickness 40 µm.
The color shift Δb* is the difference between the measured value and the color value b* of the formulation without filler; the higher the number the more yellowish.

Transparency loss

The color value Brightness L* (CIELAB Color system) of the film was measured on a black substrate with a Luci 100 color meter; dry film thickness 40 µm.
The transparency loss ΔL* is the difference between the measurement of the black substrate without coating and the measurement with applied film; the lower the number, the better the transparency.

Abrasion resistance

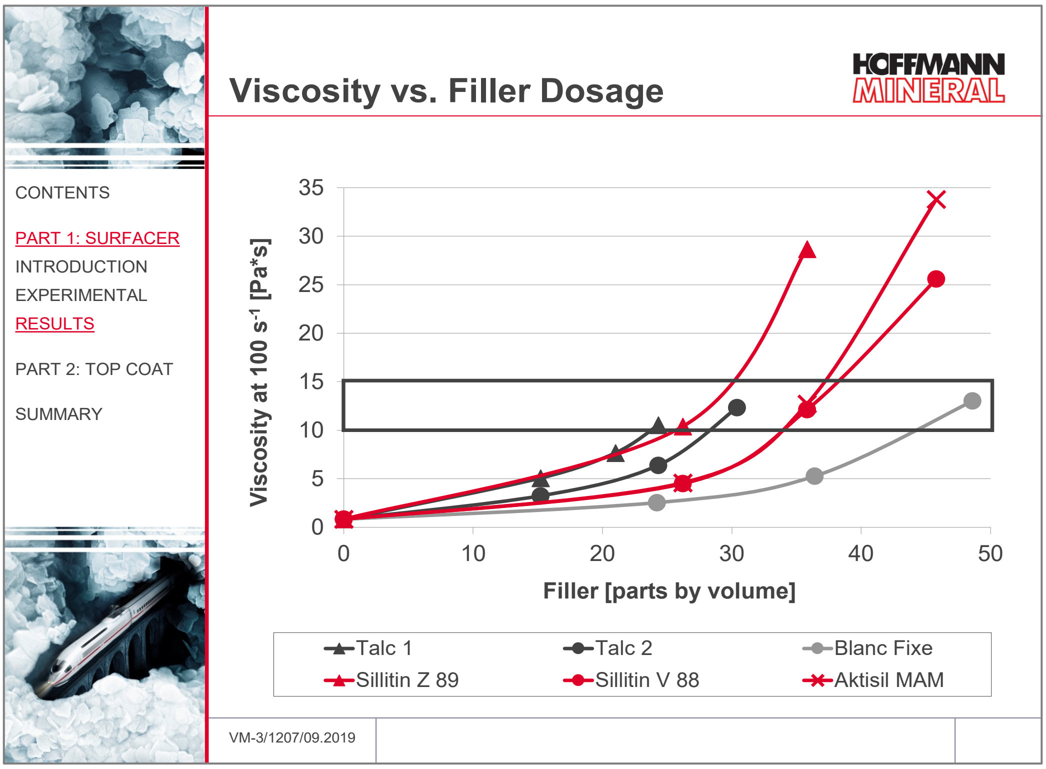
Tested with a Taber Abraser with S 42 sanding strips at a load of 0.5 kg. The abrasion loss was determined after 100 revolutions at a speed of 55 rpm by weighing the specimen and calculating the volume loss (considering the different densities). MDF sheets were used as substrate.

1.5 Results surfacer

1.5.1 Viscosity vs. filler dosage

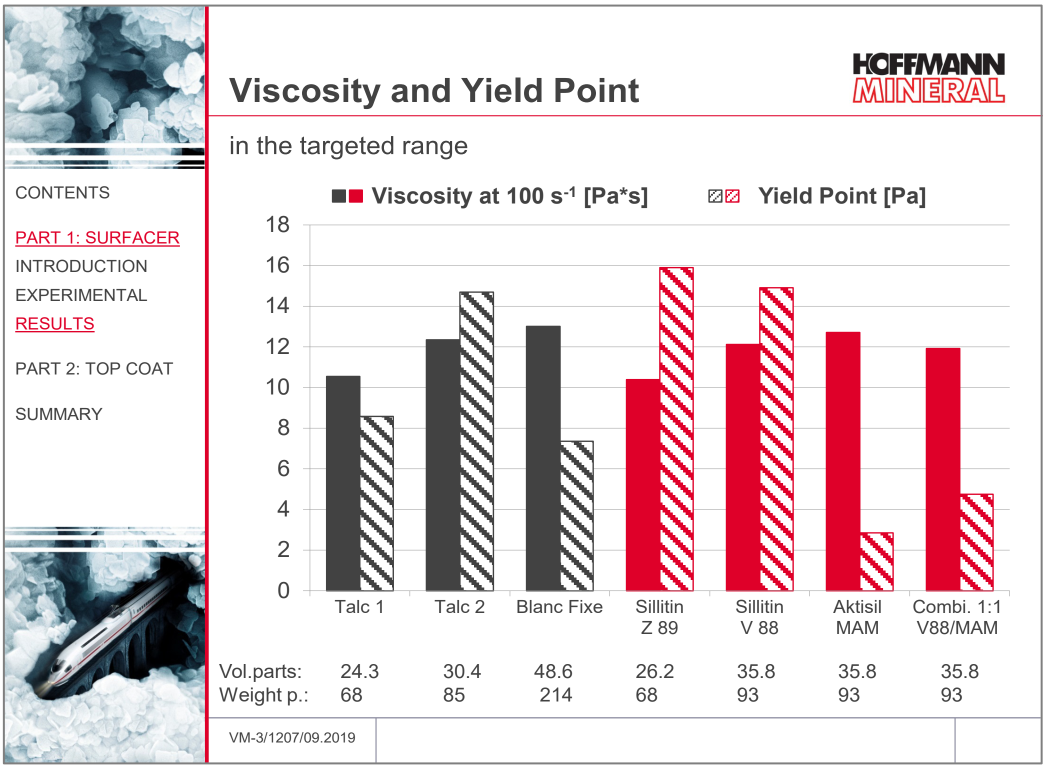
The fillers differ both in the highest applicable loadings and in their thickening effect. As for the application under study mainly the viscosity range of 10 to 15 Pa*s is of interest (the black rectangle), the dosages to be used are quite different.

Talc 1 already with about 24 parts by volume comes to the limits of processability, while 30 parts by volume are possible with Talc 2. Blanc fixe can be dosed the highest of all, but only brings about a minor viscosity increase. The Neuburg Siliceous Earth fillers allow to work with high dosages and obtain high viscosity levels. For the following tests, only filler concentrations from inside the black rectangle will be considered.



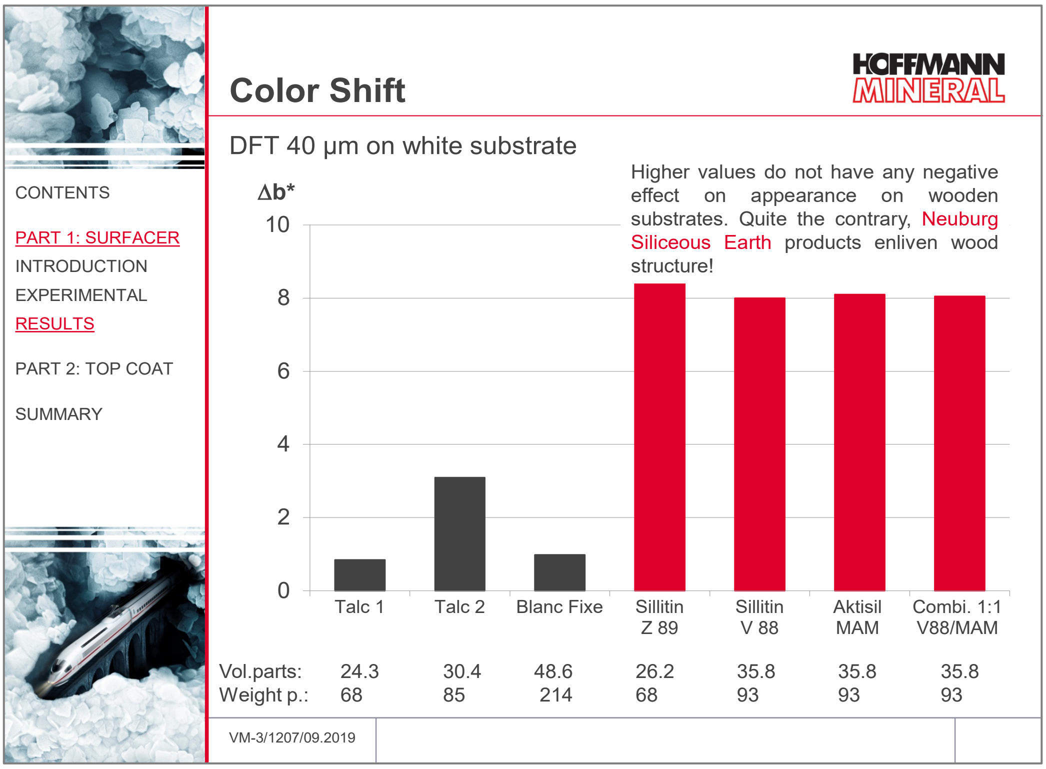
1.5.2 Viscosity and yield point

The filled bars show the viscosity at 100 s^{-1} for the corresponding volume of filler. The yield point - represented as hatched bars - reflects the subjective impression of the "paste consistency" of a formulation as a measured value. The results vary between 3 and 16 Pa, with Sillitin Z 89 at the high and Aktisil MAM at the low end. The strongly differing yield point of Sillitin V 88 and Aktisil MAM at equal viscosity appears of particular interest. Therefore, also the 1:1 blend of these fillers was included in the tests, because with such combinations it looks possible to adjust the yield point without affecting the viscosity.



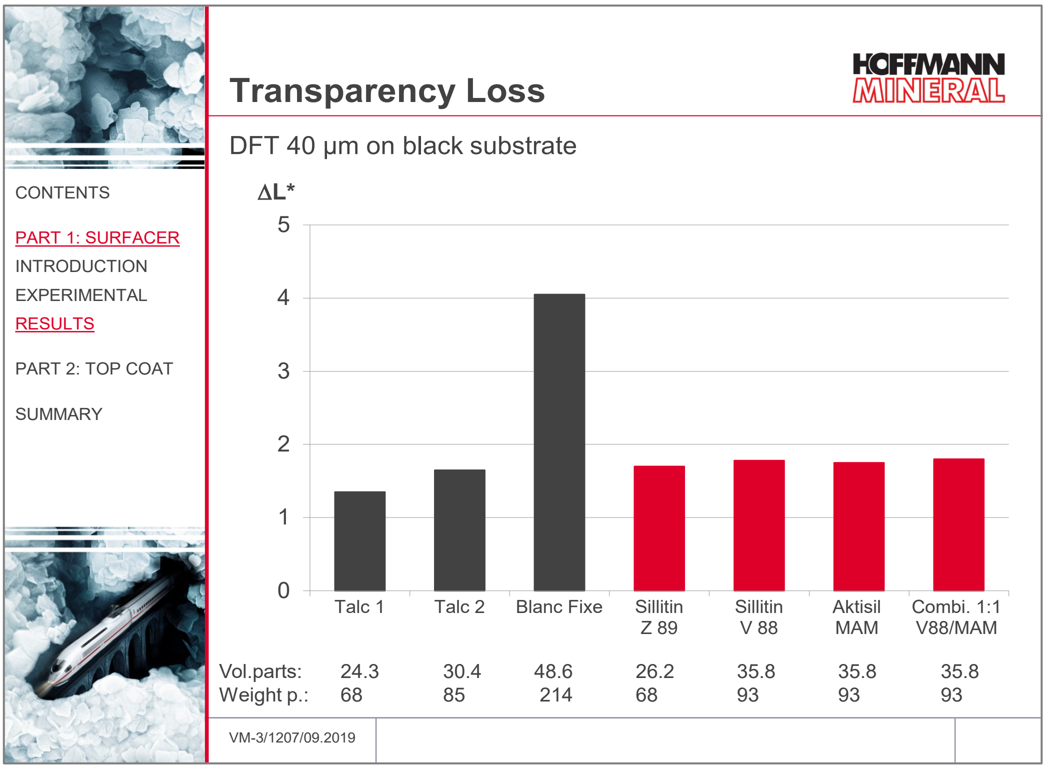
1.5.3 Color shift

All fillers give rise to a color change vs. the base formulation, with essentially the yellow tint increasing. This is evident from the increase of the b* value (CIE-LAB system) compared to the filler free formulation, as indicated in the graph via Δb*.
All the Neuburg Siliceous Earth grades come out with higher figures compared to the other fillers.
This yellow tint does not create a negative impression on beechwood, rather an "enlivening" effect is observed with the Neuburg Siliceous Earth products.



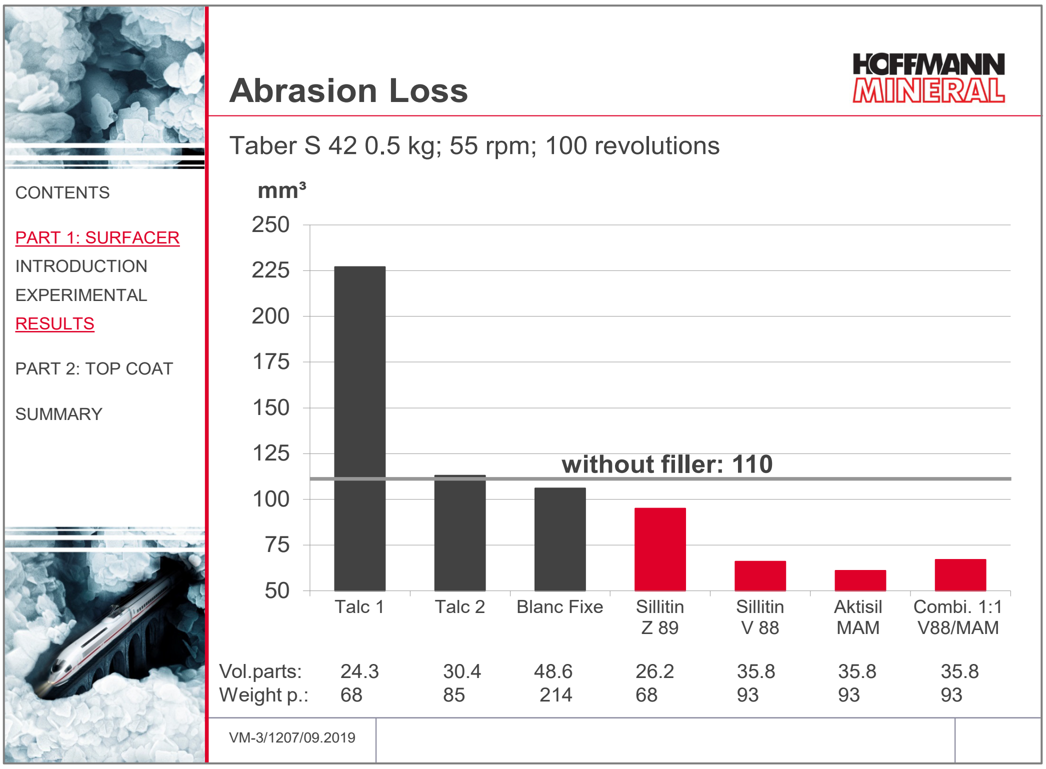
1.5.4 Transparency loss

The graph shows the brightness change via the ΔL*. All fillers give rise to a shift of the L*, with Blanc fixe offering the highest transparency loss because of the higher refraction index and the high dosage. The talcs and the Neuburg Siliceous Earth grades all show a smaller change in the L* value and thus better transparency.

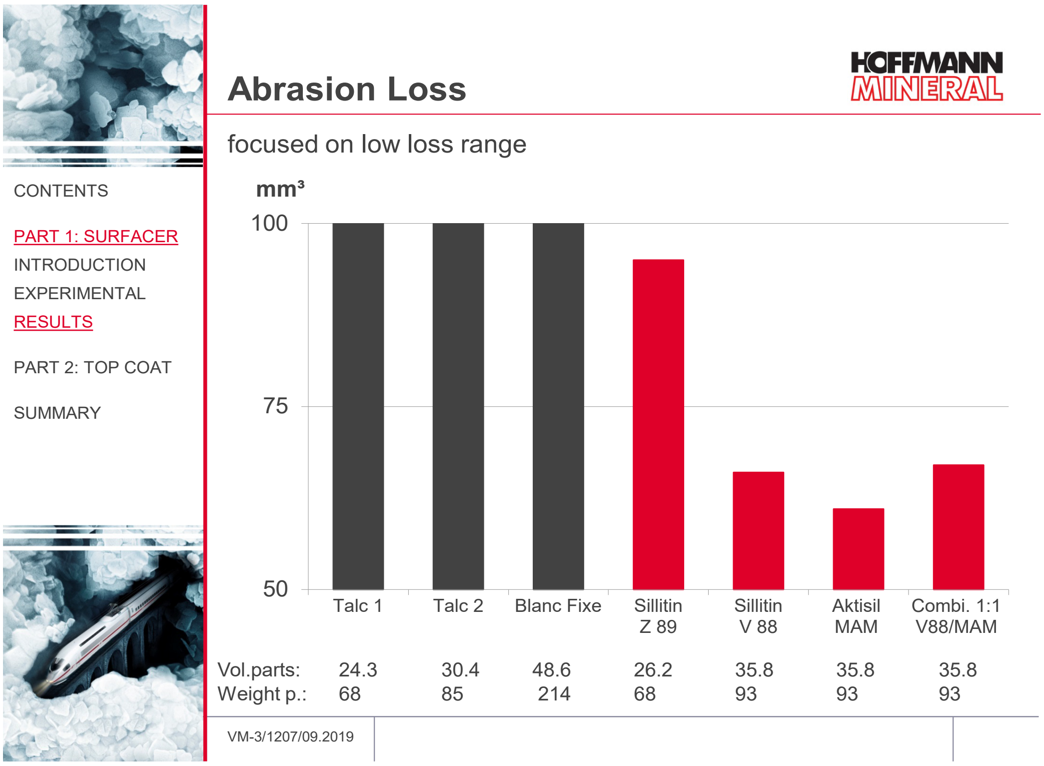


1.5.5 Abrasion resistance

As the coatings differ in their density, the graph shows the volume abrasion in mm³. The filler free base formulation gives an abrasion loss of 110 mm³. The talcs come out with widely differing results, with Talc 1 at the highest end of the comparison. Talc 2 and the barium sulfate place themselves at about the same level in the medium range. The best results are obtained with the Neuburg Siliceous Earth grades at 60 to 70 mm³.

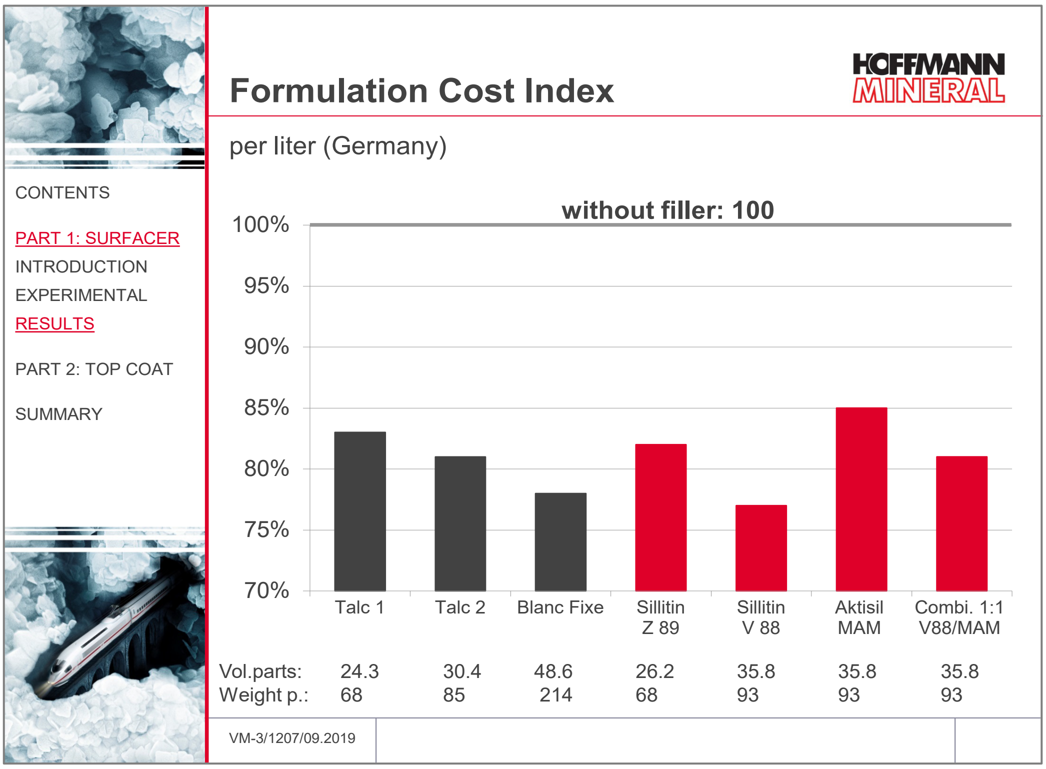


The next graph illustrates the abrasion in a more differentiating way. Aktisil MAM allows to obtain the lowest figures, followed by Sillitin V 88 and their blend. Sillitin Z 89 comes out markedly weaker, which may partly be explained by the lower loading.
The sandability by machine of the formulations with Neuburg Siliceous Earth has been judged “good”, with lower wear loss compared with the other fillers



1.5.6 Formulation costs

The graph gives the cost index for the formulation per liter in Germany. The reference line at 100 % represents the formulation without filler. The lowest material costs can be realized with Sillitin V 88, while Aktisil MAM comes out approximately at the level of Talc 1. Of interest looks the blend of Sillitin V 88 with Aktisil MAM which places itself somewhat above the Blanc fixe, but lower than the two talcs.



2 Top Coat based on aliphatic urethane acrylate

2.1 Introduction

UV-cured coatings, as 100 % systems without any volatile matter, are very difficult to mat. Even classical matting agents like silica gel based products achieve only moderate matting effect, whereas the viscosity increase is dramatic.

The aim was to achieve a good matting with the lowest possible increase in viscosity and, in addition, to minimize abrasion as much as possible.
The influence of a commercially available matting agent based on silica gel or Aktisil MAM and combinations of both materials is examined below.

2.2 Filler characteristics

The matting agent chosen was a silica gel recommended for 100 % UV systems. Apart from composition and grain shape, the mineral additives are different also in the surface area BET and surface treatment. The particle size distributions, however are comparable.

Filler Characteristics

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	Matting Agent	Aktisil MAM
Mineral Description	Silica Gel	Neuburg Siliceous Earth
Composition (approx.)	SiO_2	88 % SiO_2 8 % Al_2O_3
Grain Shape	corpuscular aggregates, highly porous	corpuscular aggregates and lamellar
Particle Size d_{50} [µm]	4	4
Particle Size d_{97} [µm]	12	18
BET Surface [m²/g]	400	11
Surface Treatment	none	Methacrylic silane

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2.3 Base formulation

The base formulation followed a recommendation from UCB Chemie (today Allnex).

The matting agent resp. Aktisil MAM were added to 100.3 parts by weight to the base formulation.

Base Formulation

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	Parts by weight
Ebecryl 294	65.0
HDDA	30.0
Benzophenone	3.0
Omnirad 1173	2.0
Byk 088	0.3
Matting Agent	variable X
Aktisil MAM	variable Y
Total	100.3 + X + Y

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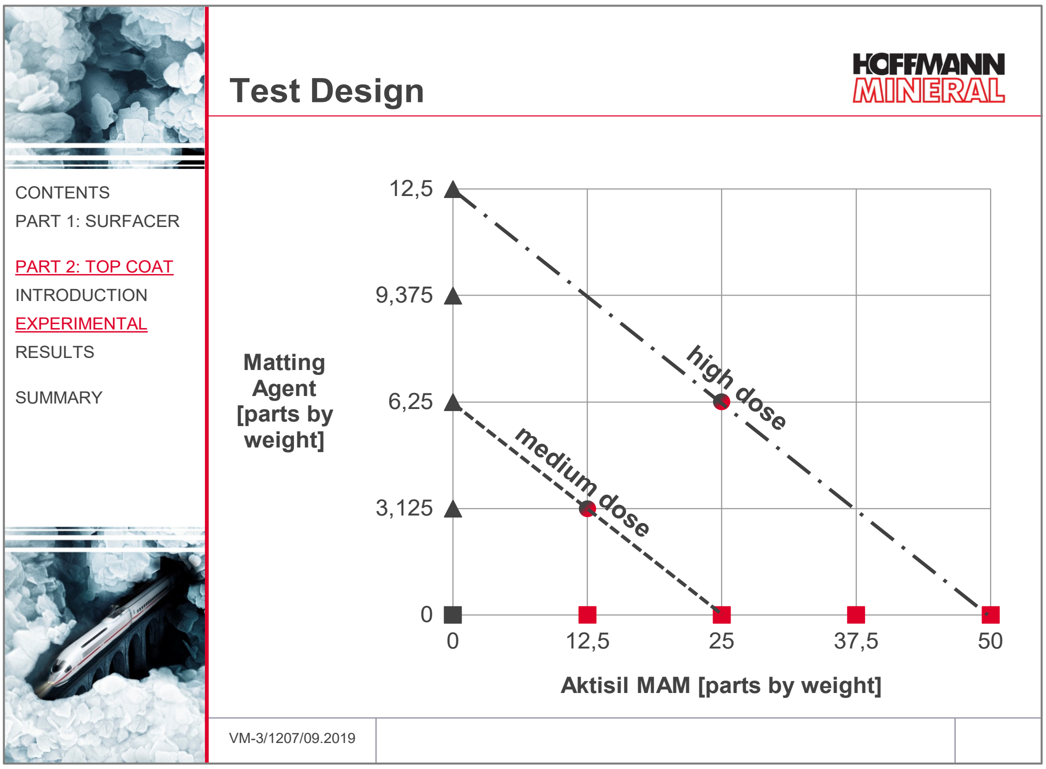
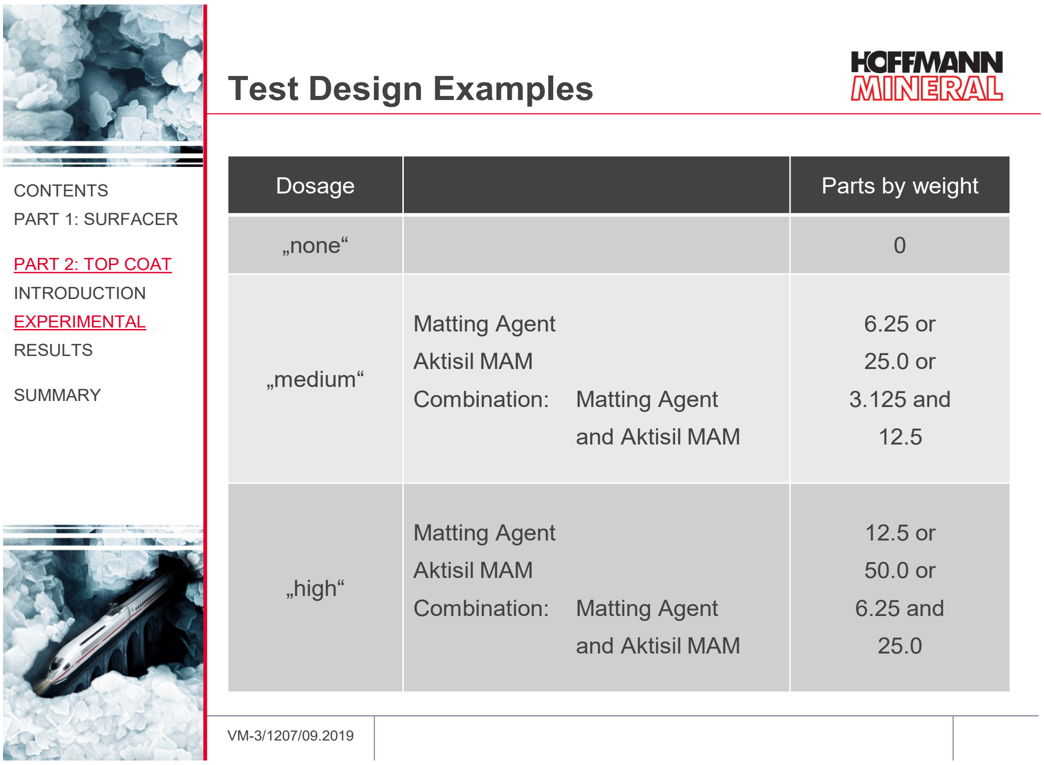
Note: Ebecryl 294 (aliphatic urethane triacrylate, 85% solution in HDDA) is no longer available. The new delivery form is called Ebecryl 294/25 and now contains 25% HDDA.

2.4 Test design

In order to cover the whole test spectrum, a statistical test design looked appropriate. This is illustrated in the graph, with each square indicating a different formulation



Examples of the test design in tabular form:



Dosage		Parts by weight
„none“		0
„medium“	Matting Agent Aktisil MAM Combination: Matting Agent and Aktisil MAM	6.25 or 25.0 or 3.125 and 12.5
„high“	Matting Agent Aktisil MAM Combination: Matting Agent and Aktisil MAM	12.5 or 50.0 or 6.25 and 25.0

In the following the indications "none", "medium" and "high" will be used for specifying the dosages of the two materials resp. their blend.

2.5 Application, tests and parameters

Viscosity

Rotation viscosimeter MC 1 (Anton Paar), 23 °C; recording of flow curves with increasing shear rate from 0 to 100 s^{-1}. The figure indicated represents the measured viscosity at 500 s^{-1}.

UV-curing Top Coat

Mercury UV lamp at 80 W/cm; speed 15 m/min; two passes

Glanz

Determination of the gloss of the coating on a white substrate with Erichsen Mini-Glossmaster (60° angle); dry film thickness 40 µm.

Color shift

The color value b* (CIELAB Color system) of the film was measured on a white substrate with a Luci 100 color meter; dry film thickness 40 µm.
The color shift Δb^* is the difference between the measured value and the color value b* of the formulation without filler; the higher the number the more yellowish.

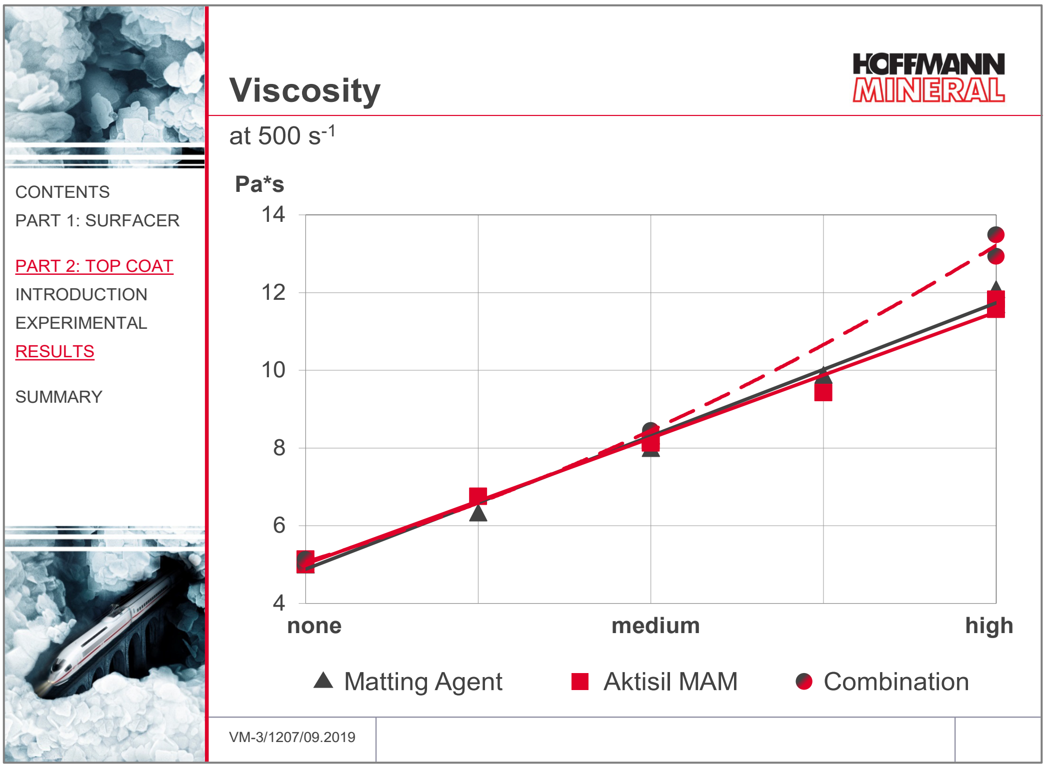
Abrasion resistance

Tested with a Taber Abraser with S 42 sanding strips at a load of 0.5 kg. The abrasion loss was determined after 100 revolutions at a speed of 55 rpm by weighing the specimen and calculating the volume loss (considering the different densities). Beech plywood sheets were used as substrates.

2.6 Results Top Coat

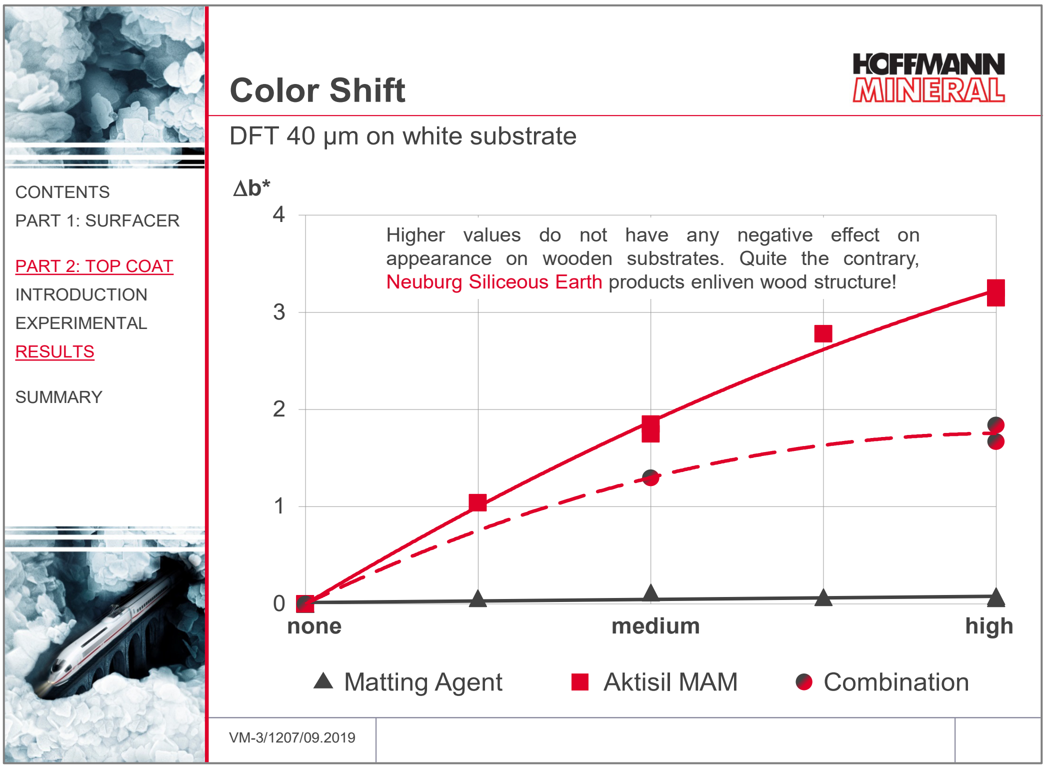
2.6.1 Viscosity

The viscosity shown in the graph can be considered practically the same for the pure materials. It should be noted, however, that Aktisil MAM has always been dosed 4 times higher than the matting agent.
The blend of the two materials shows some deviation from the pure agents. Starting at medium dosage, the viscosity increases in a more pronounced way. This phenomenon might be explained by the packing density.



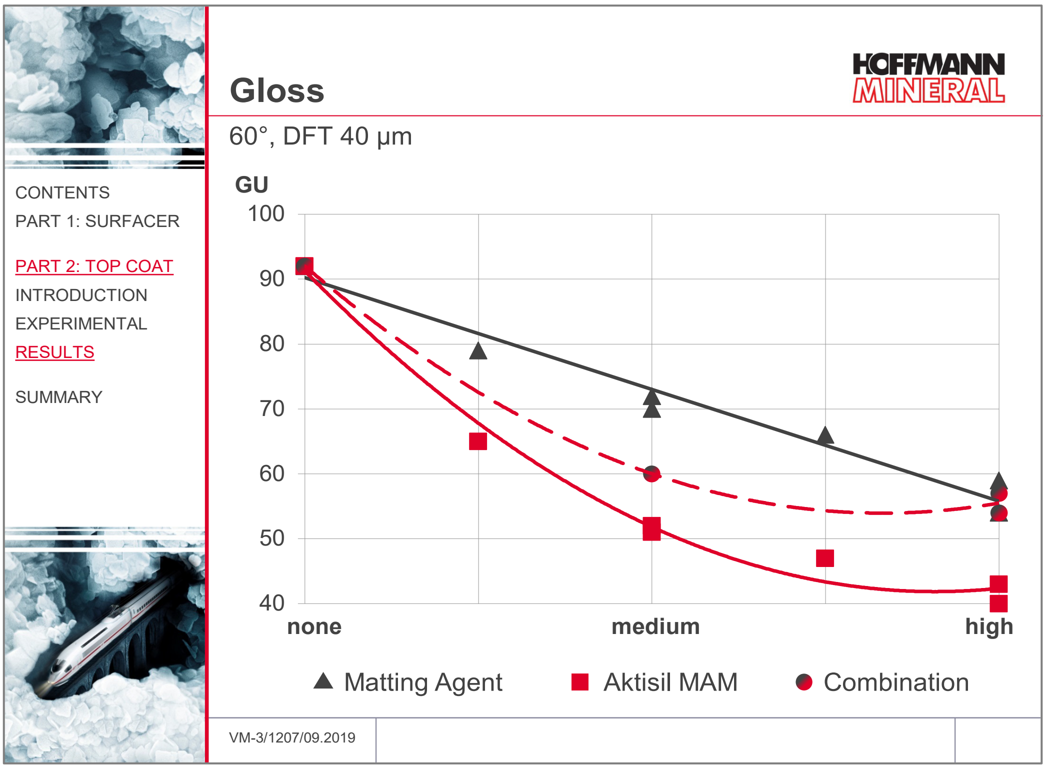
2.6.2 Color shift

The yellow tint as indicated by the Δb* value over the base formulation, increases with Aktisil MAM, whereas the matting agent has no significant influence. The blend with low dosage tends towards the behavior of Aktisil MAM, at higher dosages there is a leveling-out effect.
This yellow tint does not create a negative impression on beechwood, rather an "enlivening" effect is observed with the Neuburg Siliceous Earth products.



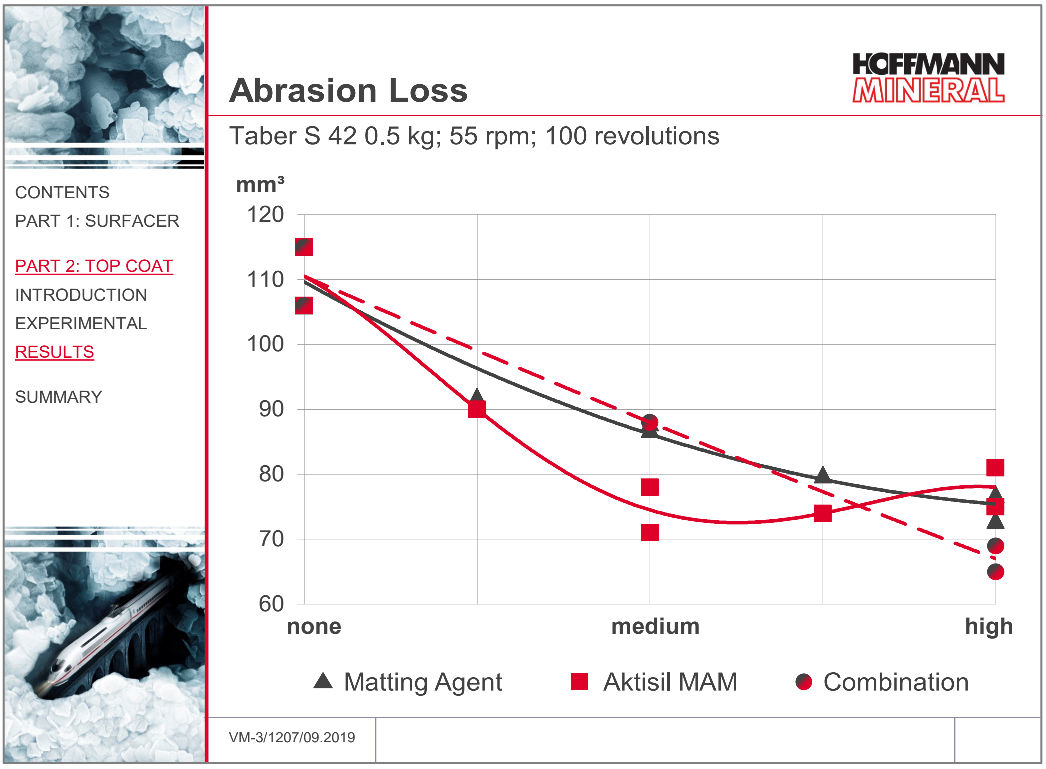
2.6.3 Gloss 60°

The addition of matting agent or Aktisil MAM gives rise to a significant decrease of the gloss values. In the case of the matting agent, this effect comes out as a linear function of dosage and the maximum attainable matting has to be judged just moderate. Aktisil MAM allows to arrive at markedly lower levels, with some attenuation of the effect between medium and higher loadings. The blend of the two products at low dosage is close to Aktisil MAM, against at higher loadings it comes closer to the pure matting agent.



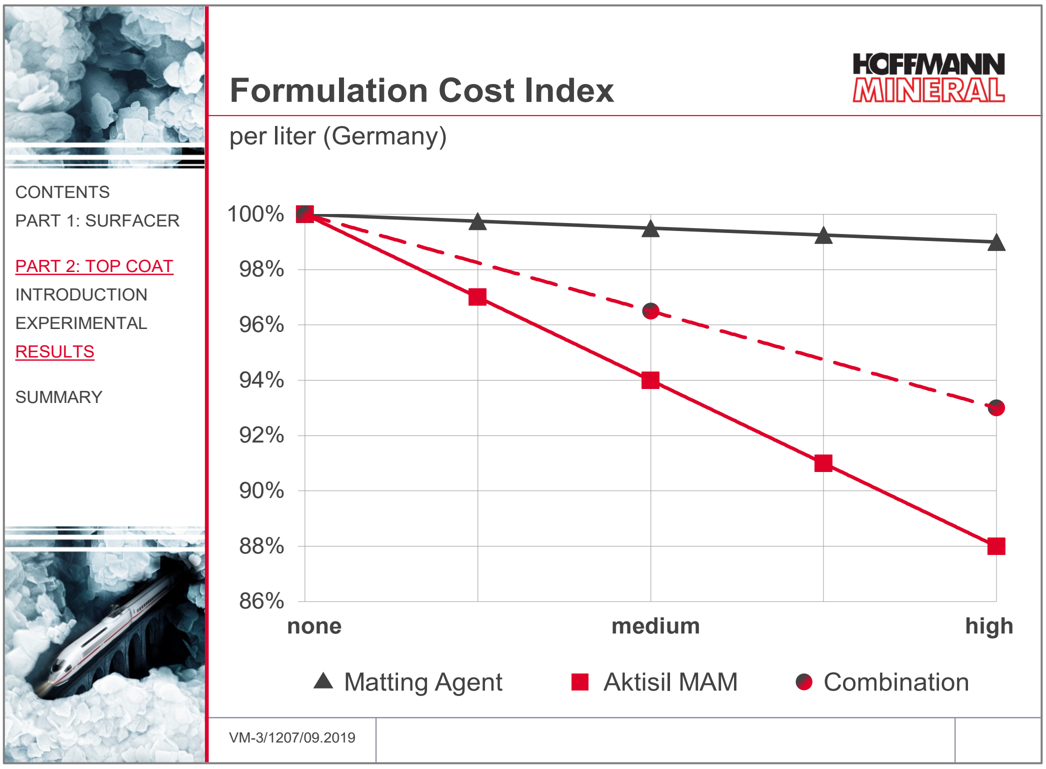
2.6.4 Abrasion resistance

Shown is the volume abrasion in mm³. Both products reduce the abrasion loss, with the matting agent as attaining the level of Aktisil MAM only at higher dosage. The latter already at low dosage shows a strong effect, and reaches a plateau at medium loadings. The blend of the two largely tends towards the behavior of the matting agent, but at high loading it offers the lowest abrasion loss.



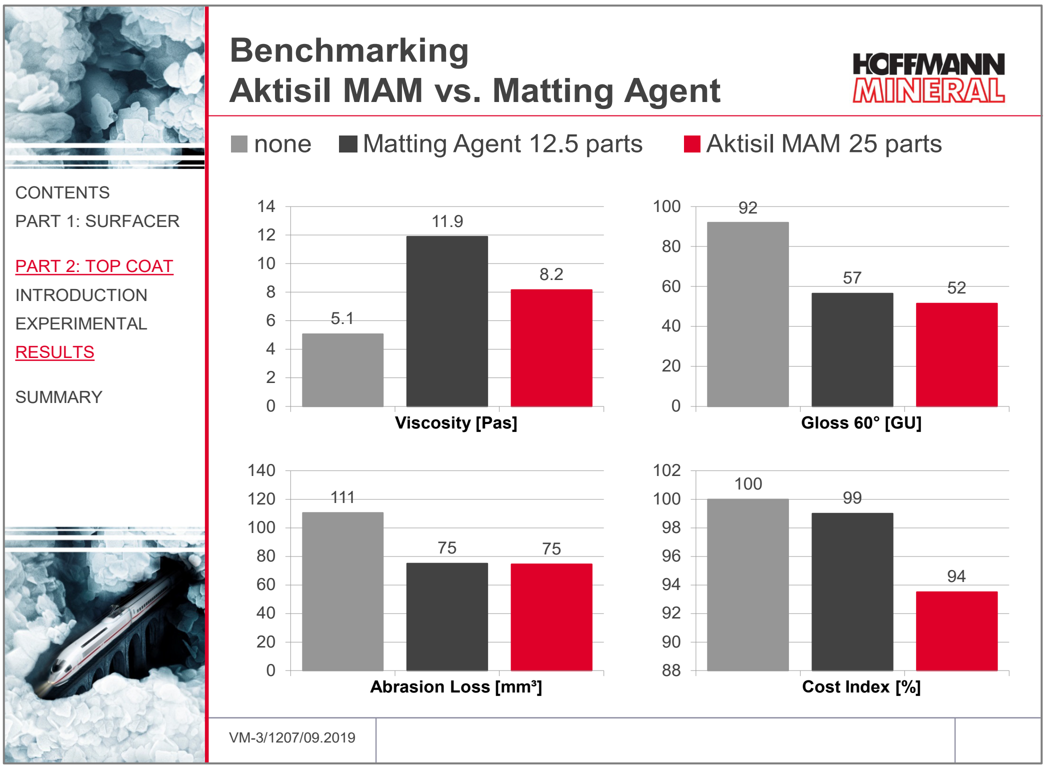
2.6.5 Formulation costs

As the graph shows, the matting agent (silica gel) does hardly affect the cost index. By contrast, Aktisil MAM gives rise to a marked cost reduction of over 10 %. The blend of the two products places itself in between.



2.7 Benchmarking

The graphs compare the formulations without filler, with 12.5 parts by weight matting agent and with 25 parts by weight Aktisil MAM with respect to viscosity, gloss, abrasion loss and formulation costs. The matting effect and the abrasion resistance of Aktisil MAM are widely comparable with the matting agent, however, Aktisil MAM causes a much lower viscosity increase. Likewise, Aktisil MAM allows to markedly reduce the costs of the formulation.



3 Summary

The Neuburg Siliceous Earth grades Sillitin V 88, Sillitin Z 89 and the surface treated Aktisil MAM show excellent optical and mechanical properties in UV-curing wood coating systems.

Surfacer:

Neuburg Siliceous Earth fillers result in very low abrasion loss and do not impair mechanical sanding characteristics.

Rheological behaviour (yield point) can be controlled precisely by combining Sillitin V 88 with Aktisil MAM.

Top Coat:

Aktisil MAM achieves good matting with only a moderate increase in viscosity and low abrasion loss.

The excellent cost-performance ratio complements the overall result.