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Unternehmensgruppe Hoffmann

Matt UV clear coat, topcoat:

Matting constancy and

cost reduction by using

Neuburg Siliceous Earth

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1 Introduction

The matting of 100 % UV coatings is generally more difficult than in solvent-based formulations. Because of the fast curing and the absence of volatile solvents, volume shrinkage is prevented, so the micro-roughness which is responsible for the matting can hardly develop. Nevertheless, there are several ways to mat solvent-free UV coatings. Newer formulation methods include surface structuring or internal light scattering. Excimer laser curing is another procedural variant.

The common used and also most effective method is, however, the conventional, classic variant that uses wax or a matting agent such as silica. Depending on the used amount, wax and silica can achieve sufficient matting with excellent transparency.
The stronger the matting should be on a surface, the more matting agent is required. Strong matting quickly becomes a question of cost, since large quantities of matting agent also mean higher raw material costs, which in turn results in higher formulation costs. Furthermore, a high wax or silica concentration can cause a strong viscosity increase in the liquid varnish. All of these factors make it hard to find a suitable and also economically interesting solution.

Therefore this study presents the matting efficiency and constancy of Neuburg Siliceous Earth compared to commercially available matting agents.
The target is to maintain or even improve the properties of the best matting agent and reduce the formulation costs at the same time.

Commercially available matting agents

None of the matting agents used in 100 % UV varnish can induce matting regardless of thickness. A large number of matting agents such as diverse types of silica gel silicas as well as different types of wax and compounds with organic polymers were tested; the best results were achieved by the four products shown in the following chart. As shown in *Figure 1,* Silica C offers good matting together with relatively good constancy at varying dry film thicknesses (DFT). Silica C is therefore used as control in the following and in blends with the different Neuburg Siliceous Earth products.

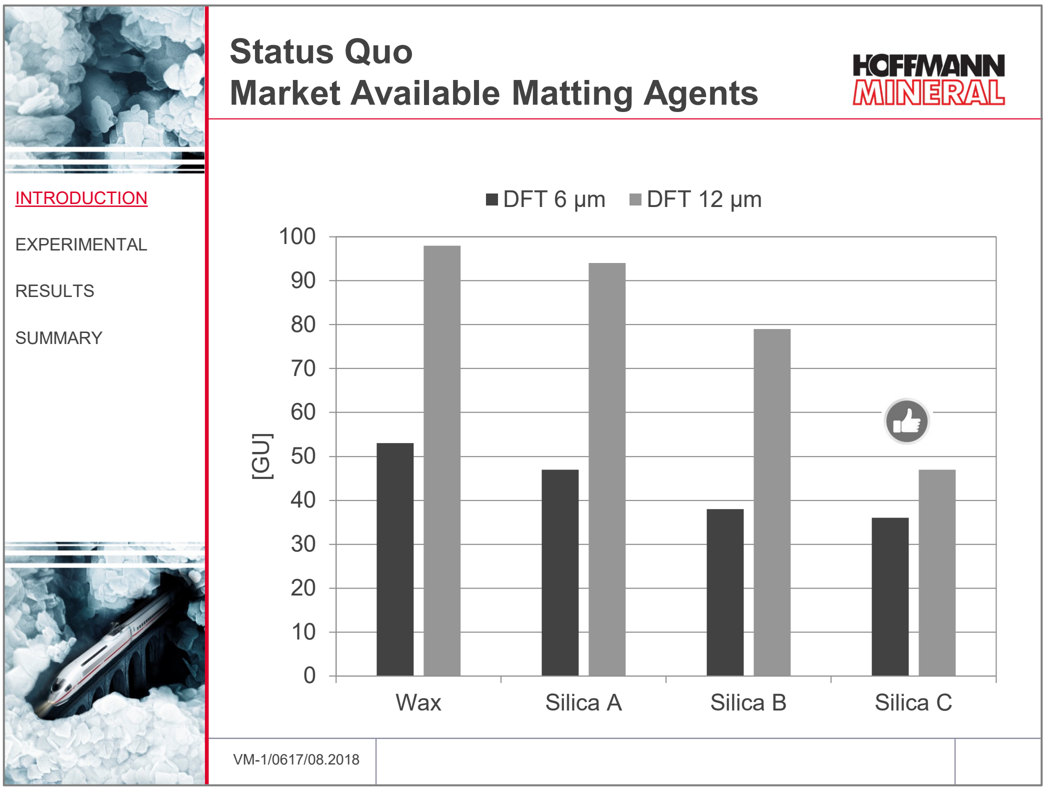
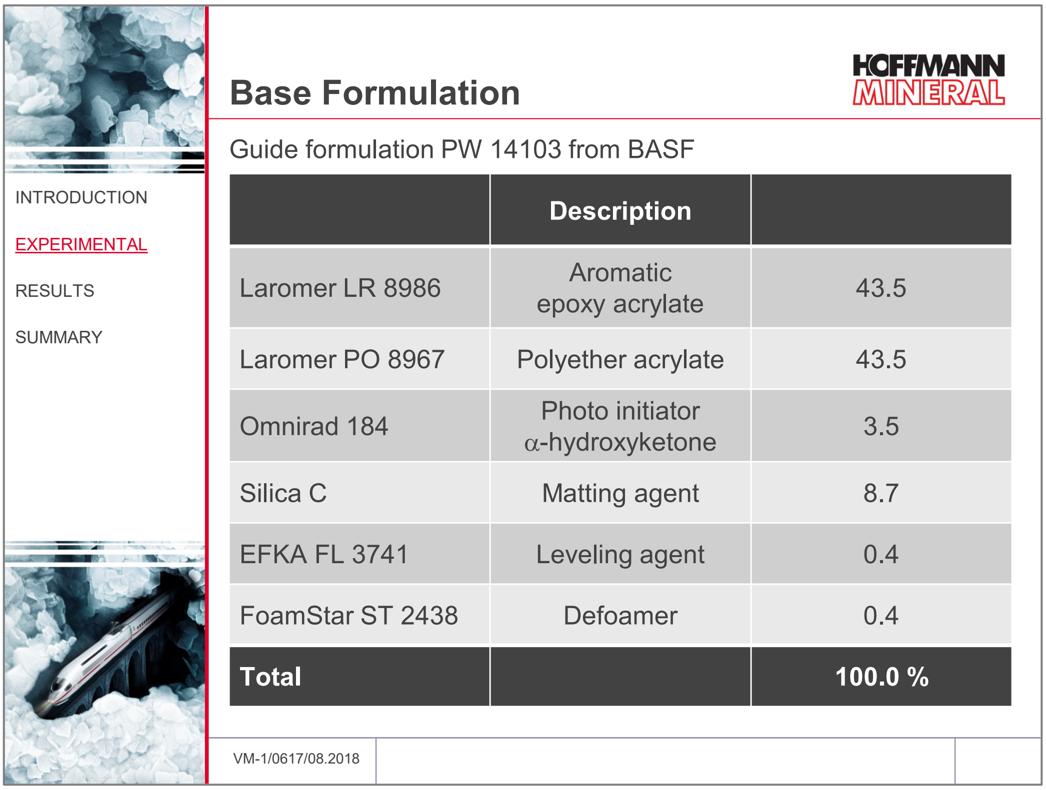


Figure 1

2 Experimental

2.1 Base formulation

A guide formulation from the BASF Company was used as base formulation (*Figure 2*). The binder system consists of a combination of an aromatic epoxy acrylate (Laromer LR 8986) and a polyether acrylate (Laromer PO 8967). In addition to a photo initiator, leveling agent and defoamer, the formulation contains 8.7 % Silica C as single matting agent.



	Description	
Laromer LR 8986	Aromatic epoxy acrylate	43.5
Laromer PO 8967	Polyether acrylate	43.5
Omnirad 184	Photo initiator α-hydroxyketone	3.5
Silica C	Matting agent	8.7
EFKA FL 3741	Leveling agent	0.4
FoamStar ST 2438	Defoamer	0.4
Total		**100.0 %**

Figure 2

2.2 Partial replacement of the matting agent and formulation variants

The guide formulation (control) contains 8.7 % Silica C. This amount was reduced and replaced through Neuburg Siliceous Earth in preliminary tests so that a gloss level similar to the control was reached again. So the quantity of Silica C can be reduced by 50 % through Neuburg Siliceous Earth in the proportion of 1 to 4. As seen in Figure 3, the new concentrations of 4.35% Silica C and 17.4 % Neuburg Siliceous Earth are depicted.

The procedure of replacing half of the contained silica by the fourfold quantity of Neuburger Siliceous Earth can also be used in other UV coatings for initial tests.

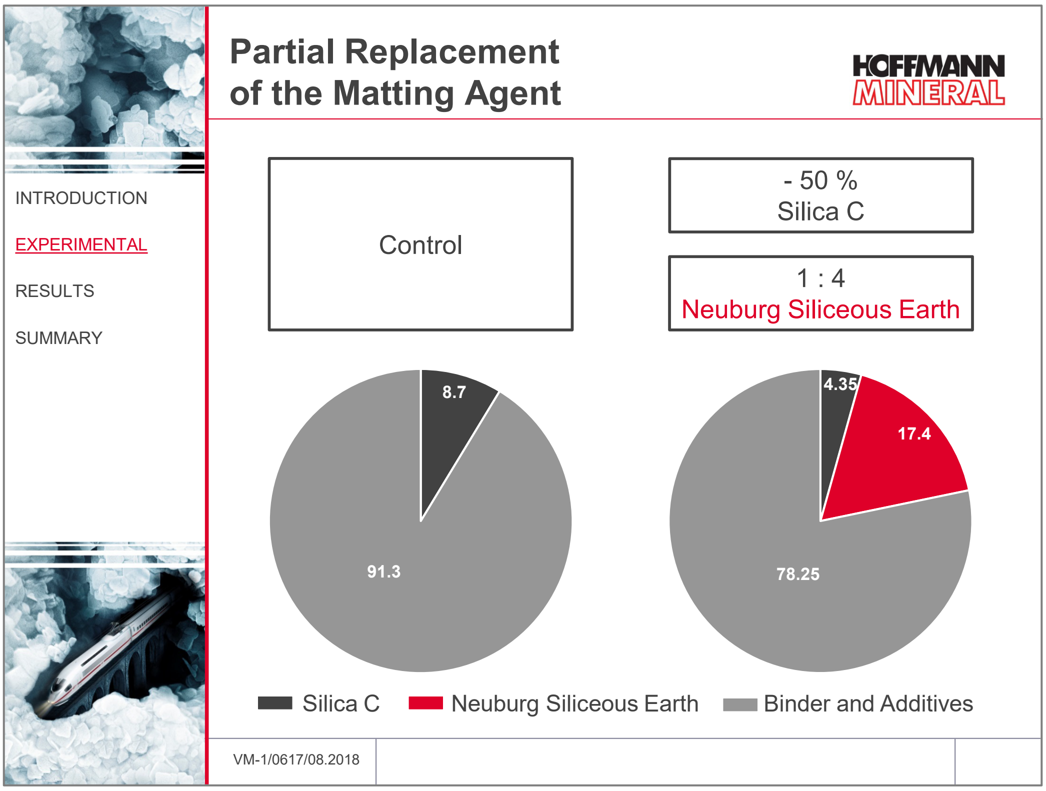
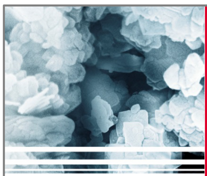


Figure 3

The partial replacement of the matting agent described above, results in the following formulation variants shown in *Figure 4*. The products Sillitin V 88 and Aktisil MAM, which are suitable for matting, were tested. The PVC of the control was 5.2 %, but due to the higher filler dosage, it is increased to 11.9 % by the partial replacement.



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Formulation Variants

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Percent

	Control	- Silica + Sillitin V 88	- Silica + Aktisil MAM
Laromer LR 8986	43.50	37.28	37.28
Laromer PO 8967	43.50	37.28	37.28
Omnirad 184	3.50	3.00	3.00
Silica C	8.70	4.35	4.35
Sillitin V 88		17.40	
Aktisil MAM			17.40
EFKA FL 3741	0.40	0.34	0.34
FoamStar ST 2438	0.40	0.34	0.34
Total	**100.0**	**100.0**	**100.0**
PVC	5.2	11.9	11.9

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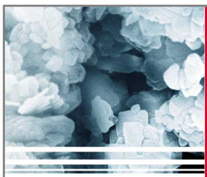
Figure 4

2.3 Fillers and characteristics

Figure 5 provides an overview of the main characteristics of the employed fillers. Silica C is organically modified silica whose average particle size d_{50} is 11 µm.
With a d_{50} of approximately 4.5 µm, Silitin V 88 is much finer than Silica C. Aktisil MAM is a variant of Sillitin V 88 whose surface was treated with methacrylic silane and has the same d_{50} as the basic product. The oil absorption, representing the binder demand, is very high with 300 g/100 g in the case of silica. Whereas the two grades of Neuburg Siliceous Earth are significantly lower than silica, absorptionlevel is only about one sixth. This effect becomes positively noticeable when adding the filler into the liquid binder. The incorporation is obviously faster with less dust formation.



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Filler Characteristics

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	Silica C	Sillitin V 88	Aktisil MAM
Color X	86.0	80.1	79.9
Color Y	90.6	84.5	84.2
Color Z	95.7	86.4	85.1
Particle size d_{50} [µm]	11	4.5	4.4
Particle size d_{97} [µm]	27	15	15
Oil absorption [g/100g]	300	45	45
Surface Treatment	organic	none	methacrylic silane

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Figure 5

2.4 Preparation, application and curing

All of the UV varnishes were simply prepared by dispersion with a toothed disk at a low peripheral speed of 4.2 m/s.

To obtain films with a sufficiently low film thickness of 6 to 12 µm, the varnishes were applied onto a glass plate using a hand roller. For medium film thicknesses of 20 to 40 µm, films were applied onto contrast cardboard charts by using a doctor blade.

The films were cured with the Aktiprint L UV-curing machine using a mercury lamp with an intensity of 120 W/cm and a belt speed of 10 m/min. It took six cycles through the UV-curing machine to achieve a fully cured layer of the varnish. The required number of cycles was determined using a potassium permanganate test.
For the test, an aqueous 1% potassium permanganate solution was prepared. The solution was applied to the varnish films one day after curing on the white section of the contrast cardboard. After waiting for one minute, the solution was first soaked up with a cloth before being wiped away with a damp cloth. The resulting discoloration of the film, representing residual double bonds, was measured with a spectrophotometer one hour later. The delta E of the discoloration was calculated from the colour values of the affected and unaffected film. A graph was drawn up to show the dose plotted against the delta E according to the number of cycles. After six cycles, the delta E remained largely constant, so that maximum conversion and thus complete hardening can be assumed.

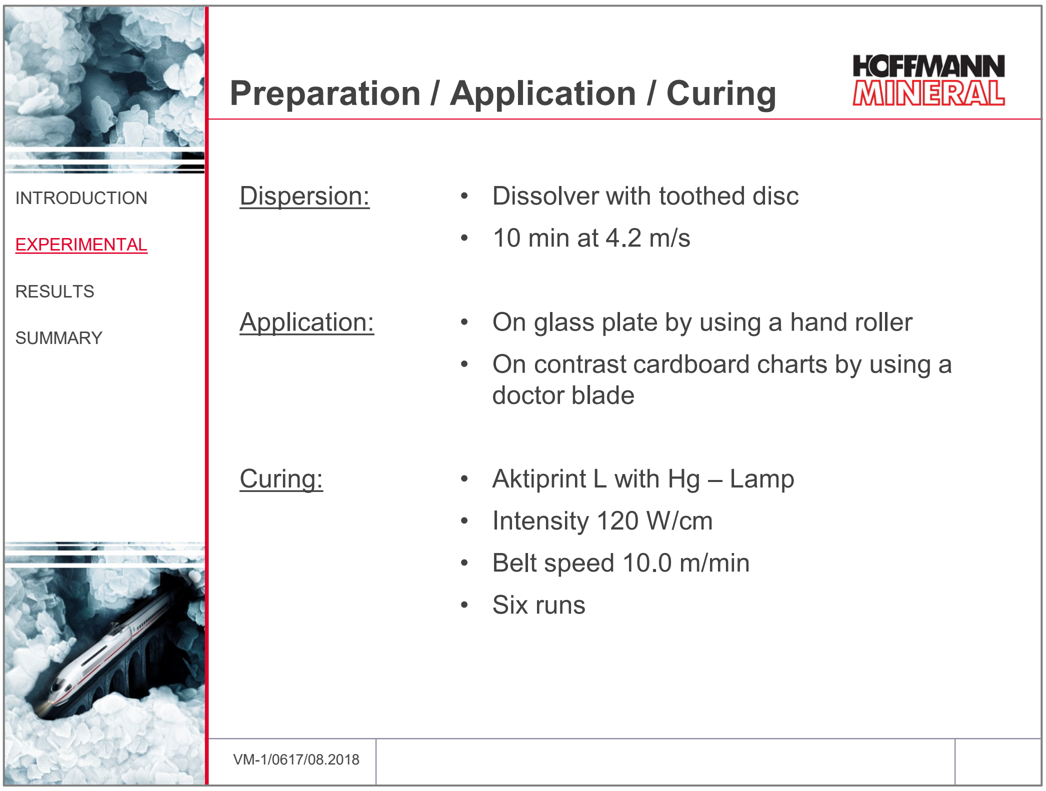

Figure 6

3 Results

3.1 Rheology

Viscosity was determined in a plate-plate rheometer via a logarithmic shear rate sweep. A measuring system with a diameter of 50 mm was used, the gap distance was 0.5 mm. In place of the low shear range, viscosity was analyzed at a shear speed of 0.1 s^{-1} (*Fig. 7*). All formulations result in similar viscosities, only the variant with Aktisil MAM is slightly higher.

Very similar results were obtained also at 500 s^{-1}.

Thus all variants with Neuburg Siliceous Earth achieve a flow behavior similar to the control with Silica C and can also be processed just as easily.

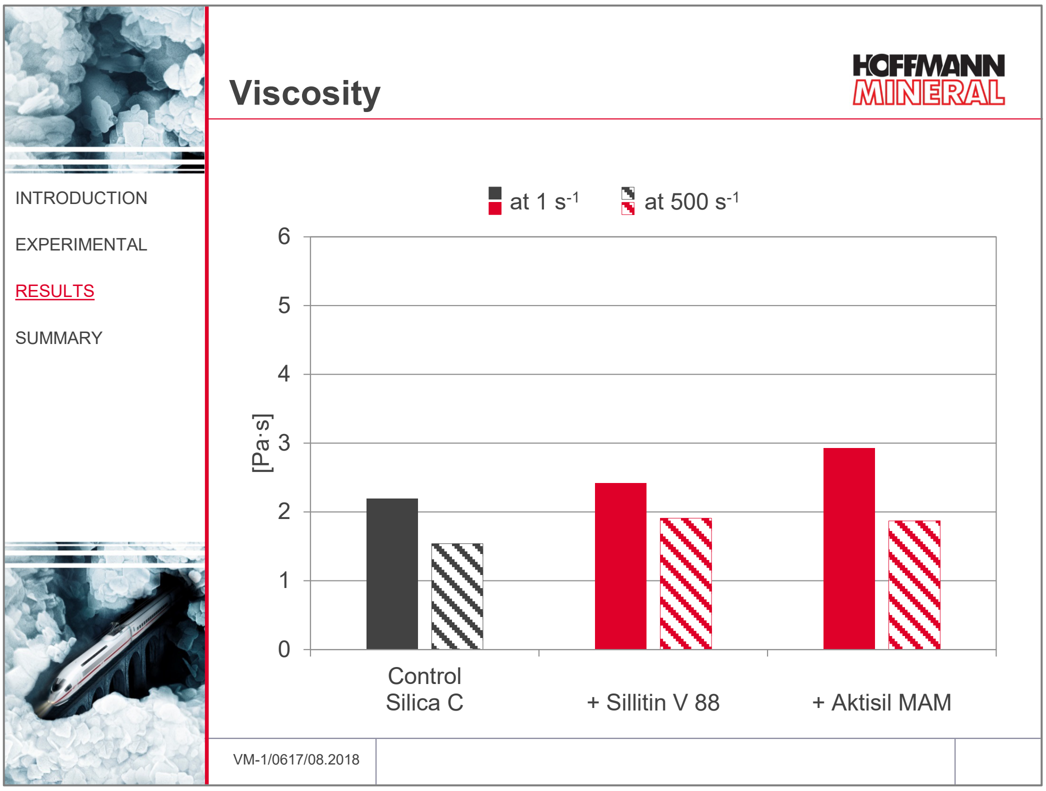

Figure 7

3.2 Gloss and gloss change absolute

Basically the target is to achieve a preferably low gloss value which is ideally independent of the film thickness. The gloss were examined in the low (6 µm to 12 µm) and medium (25 µm to 40 µm) film thickness range. These two ranges were selected, because there are situations which require a higher film thickness, even though no optical difference in gloss should be detectable, or the matted varnish should be suitable for several applications with different film thicknesses.

3.2.1 Low film thickness

Figure 8 shows the gloss results at low film thickness. With Sillitin V 88 and Aktisil MAM, the same matting is achieved at 6 µm thickness as with Silica C. When thickness is doubled, Sillitin V 88 and Aktisil MAM achieve similarly good matting as the control.

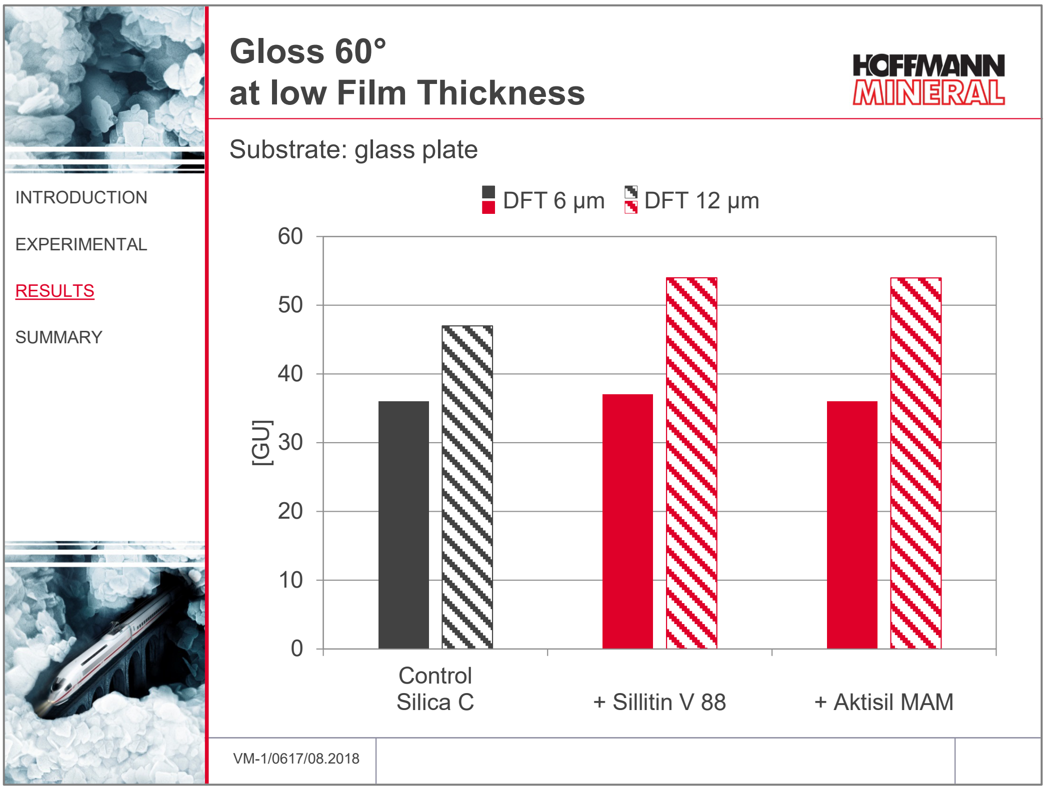

Figure 8

3.2.2 Medium film thickness

Looking at gloss at the higher film thickness range from 25 µm to 40 µm, no difference in matting performance can be observed between the control and Sillitin V 88 or Aktisil MAM (Figure *9*).

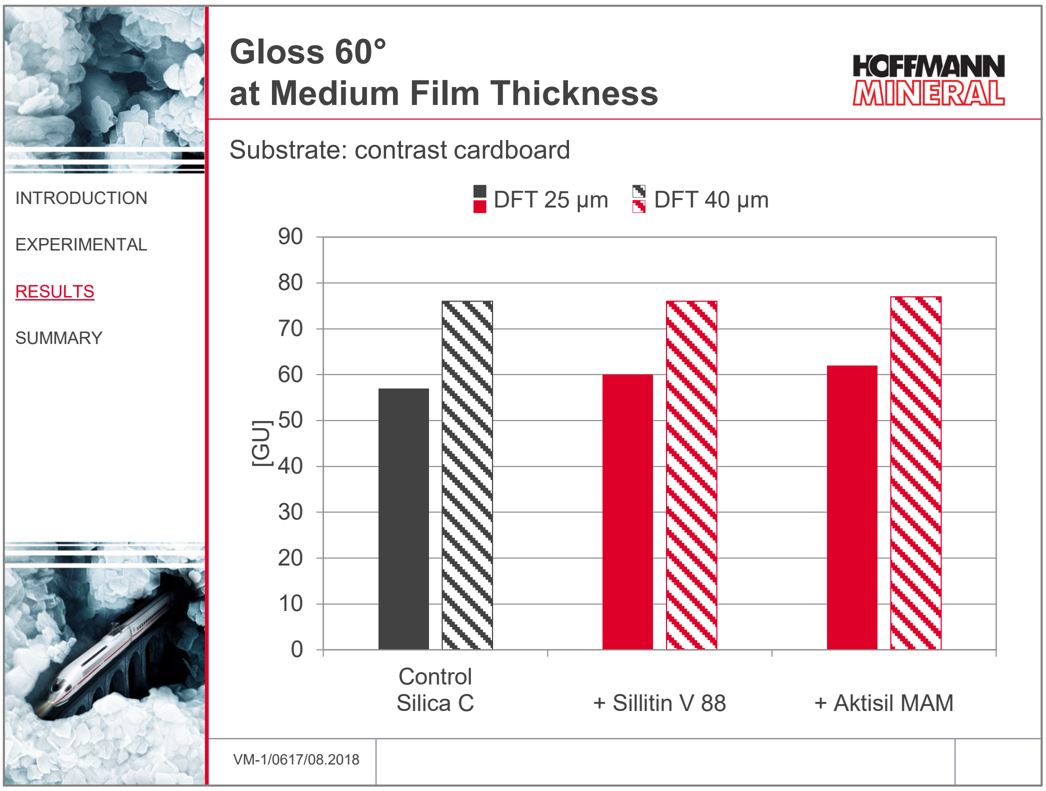

Figure 9

3.3 Transparency

Transparency was determined at 20 µm and 40 µm on the black fields of a contrast cardboard chart by color measurement with a spectral photometer. The middle film thickness range was chosen because it is more critical than the low range. The basic formulation that did not contain any matting agent served as a reference value. Its brightness values L* were set as a reference and the ΔL* for all the other formulations was calculated from it.
The results shown in *Figure 10* show that very good transparency is achieved with the control as well as with Sillitin V 88 and Aktisil MAM.

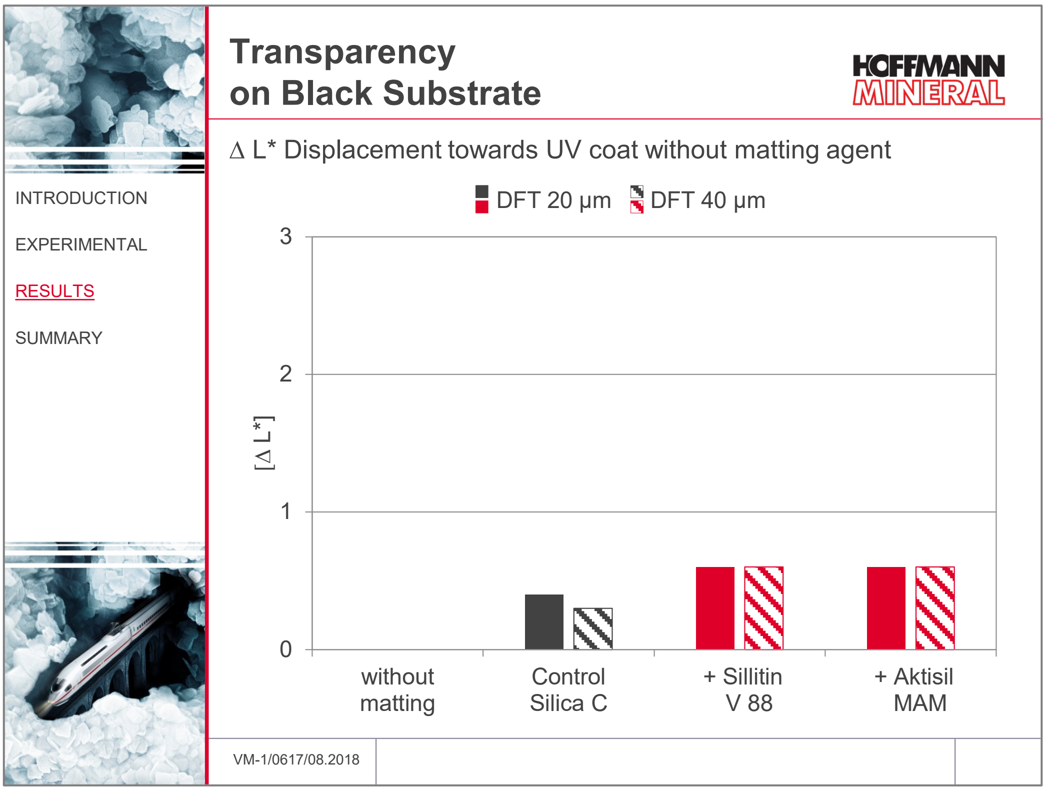

Figure 10

3.4 Abrasion resistance

Abrasion loss was determined according to two different methods using a Taber Abrader, under rather moderate conditions according to ASTM D 4060 with CS17 abrading wheels and a load of 1 kg after 1000 revolutions, and also more critically according to DIN 53754 with S42 sand paper strips and a load of 5.4 N after 100 revolutions. For both standards, a rotation speed 55 min^{-1} was adjusted on the Taber instrument.
The abrasion of the basic formulation without matting agent, identical formulation as for transparency, was also determined.

Since the coatings differ in their density, abrasion is shown in *Figure 11* as volume abrasion in cubic millimeter according to ASTM D 4060, and not in mass as required by the standard.
The formulation without matting agent has the highest volume abrasion loss. Silica C as well as the tested Hoffmann Mineral products distinctly improve abrasion resistance compared to the unfilled formulation.

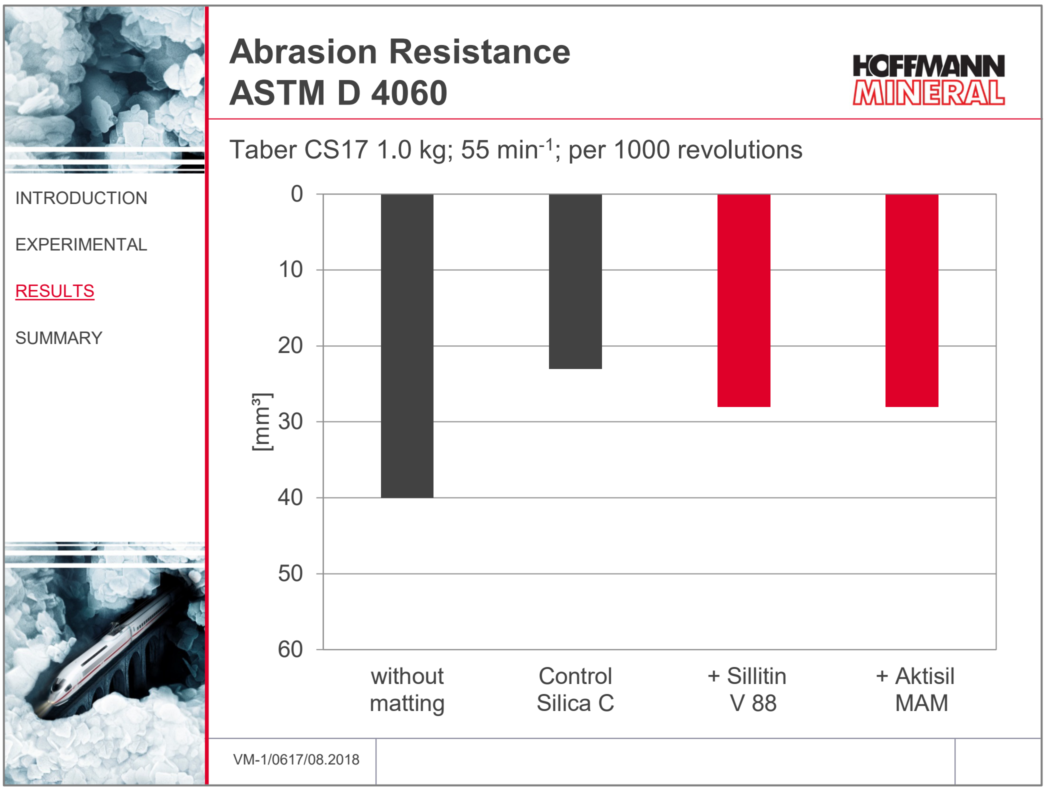

Figure 11

The abrasion results when using the more aggressive and therefore more critical S 42 abrasive paper strips show positive effects for the variants with Neuburg Siliceous Earth (*Figure 12)*. While the same abrasion resistance is achieved with Sillitin V 88 as with Silica C, abrasion resistance is improved again with Aktisil MAM.

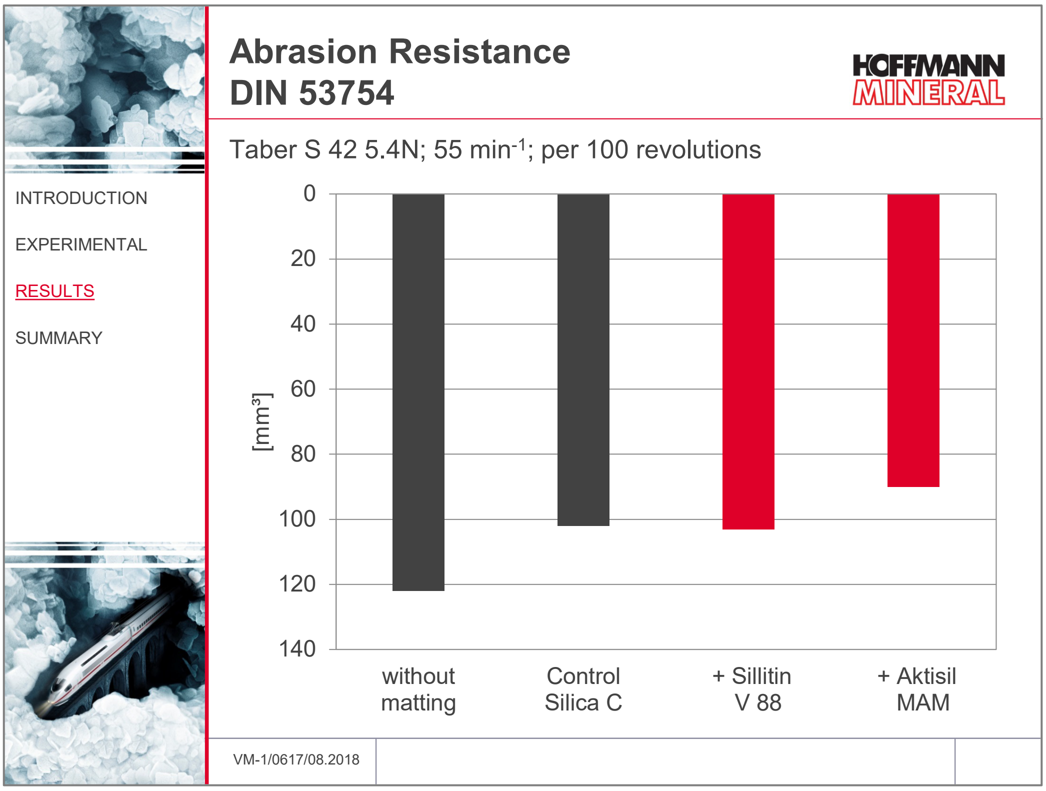

Figure 12

3.5 Formulation cost reduction potential

Figure 13 provides an overview of the possible reduction of formulation costs based on raw material prices in Germany in 2018. With reference to the control with only Silica C, up to 4.2 % of the costs can be saved by using Aktisil MAM. If Sillitin V 88 is used, formulation costs can even be reduced by up to 8.5 %.

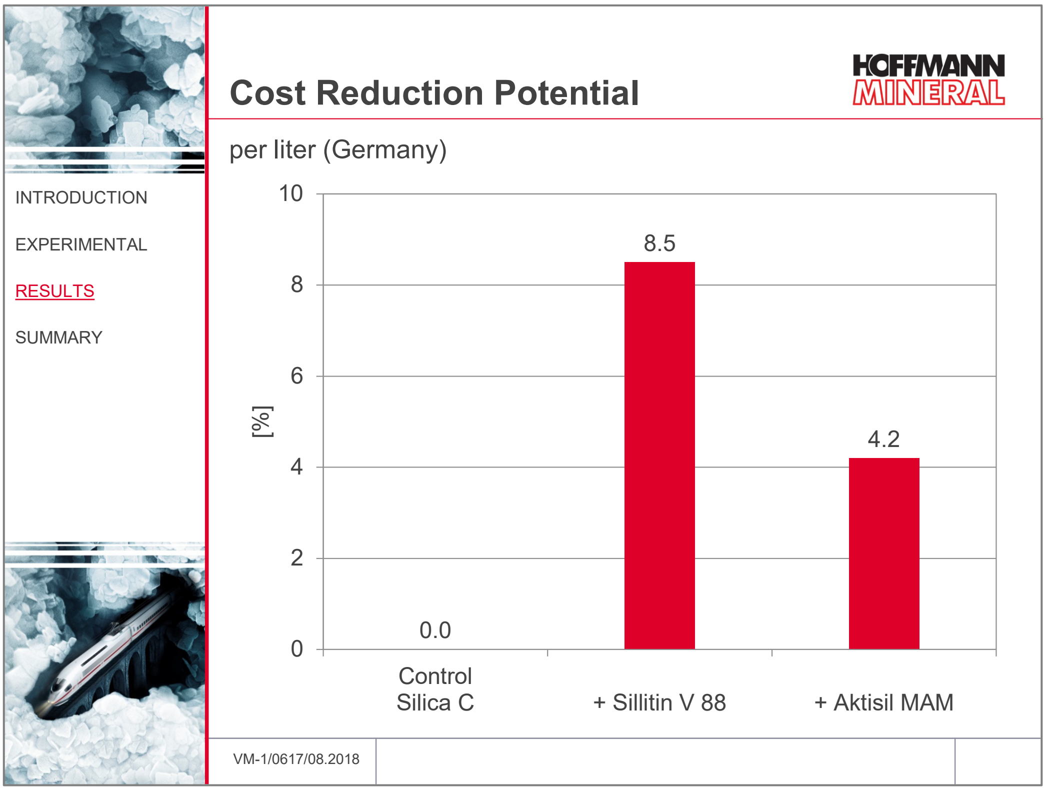

Figure 13

4 Summary

In 100 % UV clear coats, formulation costs can be significantly reduced through partial replacement of the matting agent by Neuburg Siliceous Earth. The presented types can at least maintain or even improve the property profile of the control.

With the selected replacement ratio of 1 to 4 of silica to Neuburg Siliceous Earth, the viscosity of the solvent-free UV varnish is not increased and the good transparency remains unchanged.
Furthermore, excellent matting is achieved with outstanding matting constancy and abrasion resistance is additionally improved.

Product recommendations:

Sillitin V 88

- Good transparency
- Good matting

Aktisil MAM

- Good transparency
- Good matting
- Improved abrasion resistance