Cleanable, low-gloss interior emulsion paints: Optimization of stain resistance through Neuburg Siliceous Earth

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1 Introduction

The ability to achieve a decorative appearance is one of the most sought-after and demanded characteristics of modern interior paints. Conventional, mostly low-gloss interior emulsion paints often have the disadvantage of having insufficient resistance to staining, since liquid stains are easily absorbed into the surface due to the high and often supercritical pigment volume concentration, and are therefore difficult to remove.

As a result, reduced liquid and stain absorbency in the coating is a desirable quality for cleanable paints. This can be achieved by setting a suitably subcritical pigment volume concentration which enables more homogeneous film formation and sealed, less absorbent coating films. In addition, the sensitivity to staining can be reduced by using hydrophobic or oleophobic additive components in the formulation.

Low-gloss, cleanable coatings must also have sufficient durability and wet-scrub resistance in order to ensure residue-free stain removal without removing the paint as well. In these kinds of paints, coarser and relatively hard fillers can help achieve better wear resistance in addition to the matting effect. Depending on the extent of mechanical cleaning, this is intended to counteract excessive paint removal, while also preventing changes to the surface's visual appearance such as streaking or polishing.

This study builds on the principles of protection that have already been outlined. However, its primary aim is not just to achieve mechanical stability in the surface coating, but also to imbue the surface with improved resistance to staining in the first place through the use of suitable fillers in order to keep the amount of cleaning required to a minimum.

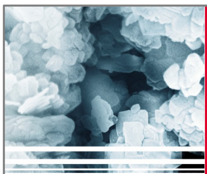
With this in mind, is further optimization possible with selected functional fillers such as Neuburg Siliceous Earth in order to achieve surfaces that are easier to clean and can repel dirt with greater resistance to staining?

The report examines this question in detail and takes into account other important characteristics of decorative interior coatings which have also been tested as part of the investigation.

2 Experimental

2.1 Basic formulation and variations

This guide formulation of a low-gloss interior emulsion paint combines an environmentally friendly, low-VOC formulation with more modern binder technology specifically optimized for cleanability based on the solvent-free pure acrylic binder “Acronal PLUS 6282” from BASF (see *Fig. 1*).



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Formulation

Guide formulation from BASF

		parts by weight
Water deionized	-	148.4
Natrosol 250 HR	Thickener	2.2
Dispex CX 4320	Dispersing additive	7.0
Acticide MV	In-can preservative	1.0
Silres BS 16, 20 % in water	Water repellent	3.4
Foamaster MO 2150	Defoamer	2.0
Tronox CR 828	White Pigment TiO_2	257.0
Filler	varied	166.0
Acronal PLUS 6282	Emulsion binder, pure acrylic	407.0
Rheovis HS 1212	Rheological additive	2.0
Foamaster MO 2150	Defoamer	4.0
Total		**1000.0**
Dilution with 5 % deionized water		
PVC ~ 41 % Solids content w/w ~ 60 %		

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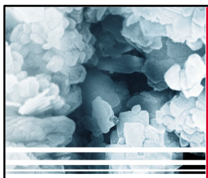
Fig. 1

The reduced pigment volume concentration relative to conventional paints is approx. 41%, and the solids content of the formulation with 5% water dilution is at around 60%. In addition to a high level of white pigment, the formulation contains 16.6% filler, the composition of which is varied as described on the next page.

The guide formulation (control) contains a three-part combination of fillers as indicated in *Fig. 2*. The diatomaceous earth is preferably used as a very coarse matting agent in order to enable luster in the dull matt area. Both types of syenite, which are present in varying proportions by weight, exhibit incrementally greater fineness as well as significantly lower oil absorptions than the diatomaceous earth.



Fillers

Characteristics

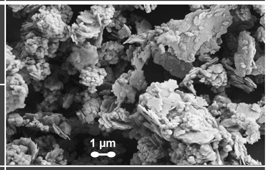
		Control			Neuburg Siliceous Earth
Diatomaceous Earth coarse, flux calcined		**33** pbw			
Nepheline Syenite coarse			**100** pbw		
Nepheline Syenite fine				**33** pbw	**166** pbw **Aktisil MAM**
Particle size	d_{50} [µm]	17	12	4.5	4
	d_{97} [µm]	40	42	19	18
Oil absorption	[g/100g]	130	21	27	45
		Ø 44 of package			
Surface treatment					methacrylic functionalized

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Fig. 2

In the following formulation variations, the entire filler package, with an average oil absorption of 44 g/100 g, is replaced with the Neuburg Siliceous Earth variant Aktisil MAM with approximately equivalent oil consumption:

Aktisil MAM exhibits a fineness and particle size similar to the fine nepheline syenite, as well as an additional surface treatment with a methacrylic functional group.

2.2 Preparation, application, and testing

The formulations were prepared according to the sequence of raw materials indicated in the formulation using the laboratory dissolver under cooling with water. White pigment and fillers were mixed in advance and added to the preparation before being dispersed for 20 minutes at a peripheral speed of the toothed disc of 15 m/s. After adding the binder and the other additives, a maturation time of 12 hours was observed.
The coatings were then diluted with 5% deionized water before normally being applied to black Leneta film and contrast cardboard with a doctor blade on an automated film applicator. The drying and conditioning of the paint films and the testing after 28 days were performed in an air-conditioned laboratory at 23°C and 50% air humidity. For more detailed information, see *Fig. 3/4*.

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Preparation & Testing (1)

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Paint Preparation	
Mixing and dispersing	With dissolver, in sequence of mentioning in the formulation Peripheral speed of toothed disc (Cowles blade) 15 m/s for 20 min, water cooling with T max. = 50°C Subjective assessment of filler incorporation and foam formation
Let Down	With binder and further additives
Maturation	Over night
Dilution	Deionized water, 5 %
Wet Paint	
Fineness of grind	Grindometer 0 – 50 µm
Viscosity	1d after preparation, Rheometer 23°C, Searle system
Storage stability	Diluted in 375 ml-metal can, 23°C / 90 d or 38°C / 42 d
Application	With doctor blade on automated film applicator, speed 12 mm/s

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Fig. 3

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Preparation & Testing (2)

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Dry Paint	
Conditioning	Drying conditions before / during tests: 23°C / 50% rel. humidity Drying time before testing: 28 days
Application with gap 300 µm on black leneta film, DFT* ~ 80 µm	
Cleanability	Color shift delta E* of soiled surface after wiping off with damp cloth and after additional wet-scrub
Wet-scrub	Weight loss after 200 Cycles on wet-scrub resistance tester according ISO 11998, Classification along with DIN EN 13300
Application with gap 100 - 225 µm gradually on cardboard, DFT ~ 35 - 80 µm	
Color / Gloss	L*, a*, b* over white, 85°- Gloss (Sheen) at full hiding film with DFT 80 µm
Dry Burnish	Gloss increase after 200 cycles with dry cloth on automated wet-scrub resistance tester derived from ISO 11998, DFT ~ 80 µm
Hiding Power	Contrast ratio over black/white depending on dry film thickness. Calculation of minimum dry film thickness to comply with DIN EN 13300 classifications and resulting spreading rates.

* Dry film thickness

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Fig.4

3 Results

3.1 Processing properties and storage stability

During formulation preparation, Aktisil MAM achieves proportionately fast and foam-free incorporation similar to the control thanks to generally good dispersion behavior in aqueous media.

The grain size of the complete interior emulsion paints reflects the varying fineness of the fillers used. According to grindometer measurements, the grain size in the control was determined at 35–40 µm and already noticeably reduced at less than 10 µm with the Aktisil MAM formulation.

The rheology profile exhibits shear thinning which is typical of interior emulsion paints, whereas the significantly reduced viscosity from 0.20 to 0.34 Pa s under higher shear stress (1000 s^{-1}) enables easy workability and spreadability. High viscosity values from 167 to 193 Pa s at low shear stress (0.1 s^{-1}) cause low sagging tendency after application and provide the film layer thicknesses required for good surface coverage.

Both formulations exhibit very good storage stability properties after 3 months at 23°C or after 42 days at 38°C, without phase separation or deposit characteristics.

3.2 Cleanability

3.2.1 Testing method

To determine cleanability, the formulation variants were applied to black Leneta film with a sufficient layer thickness using a film applicator frame, and conditioned for 28 days.

As the example in *Fig. 5* shows, the staining defined in terms of quantity and area was then caused by colored and predominantly fluid substances that are common in households. Once the varying exposure time had elapsed, the film surface was simply wiped down repeatedly with a damp cloth as an initial cleaning step.

In the second step, the standardized mechanical cleaning process was performed using an abrasive pad soaked in cleaning agent on an abrasion tester in order to remove further staining. Finally, the wiped-off exterior and the additionally scrubbed middle area were assessed colorimetrically after 24 hours of drying.

The color shift delta E* compared with the unsoiled surface describes the level of staining left behind quantitatively. The lower the values, the better the cleanability.

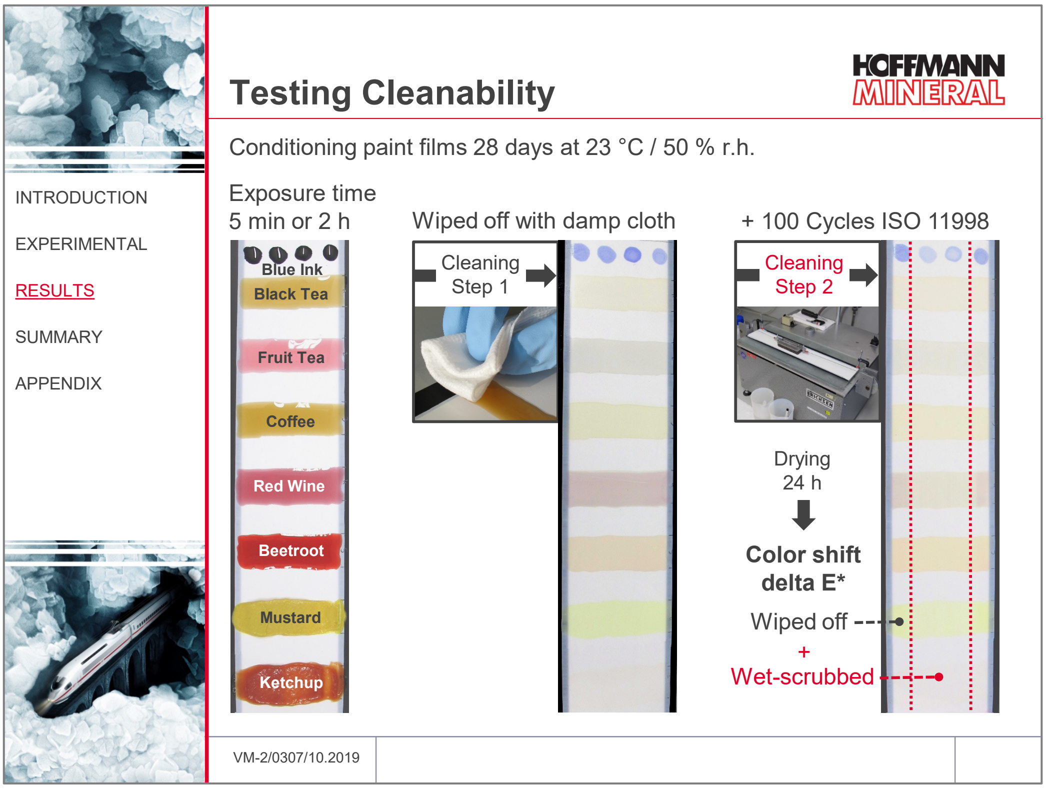


Fig. 5

3.2.2 Cleanability after short exposure time

The following models contain the quantitative results of the colorimetric analysis. Each test substance specifically tends towards different levels of discoloration, which can be observed after just 5 minutes of exposure and wiping off with a damp cloth. In order to account for these circumstances as a whole, the color shifts of all test substances in *Fig. 6* are each consolidated into an average value for an overall assessment.

The improved resistance of the formulation with Aktisil MAM to stain formation manifests in considerably lower delta E* values when compared to the control, whereas the staining can be reduced by half in the average value.

Through the more intense cleaning by means of abrasion with cleaning agent, the staining can be reduced further in both formulation variations, which results in disproportionately positive results in the formulation with Aktisil MAM. Through the use of Aktisil MAM, even critical staining from black tea, coffee or beetroot juice, for example, can be removed almost completely provided cleaning is performed promptly.

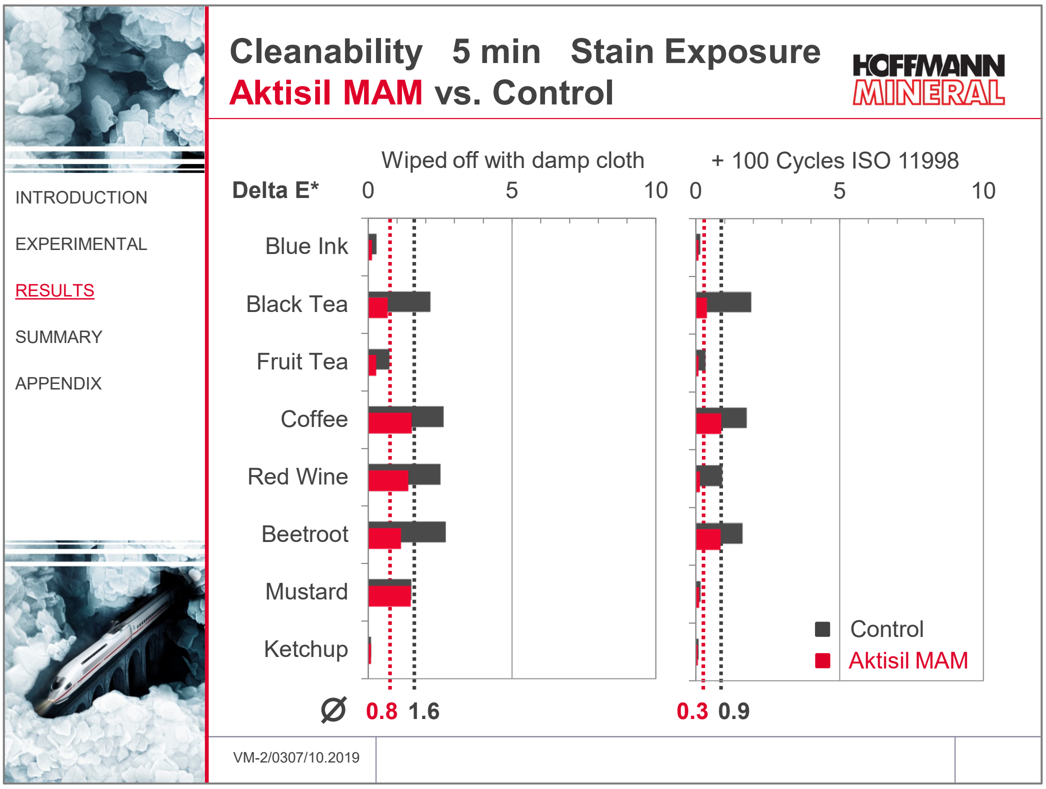


Fig. 6

3.2.3 Cleanability after prolonged exposure time

Prolonged exposure to the staining media generally has a very adverse effect on subsequent cleaning attempts (*Fig. 8*).

In the control formulation with diatomaceous earth and syenite, removal of the discolored stain is only minimal. The color shifts measured are therefore at a very high level, with the exception of the test involving ketchup.

The high performance values of Aktisil MAM meant that it is visually the more effective solution. In view of the results produced when the exposure time of stains was only brief, the protective effect is even clearer when compared to the control. Even as early as the damp cloth wipe-off stage, it becomes clear from the significantly reduced delta E* values that the staining resistance can be noticeably increased by varying the filler and using Neuburg Siliceous Earth alone.

One particular result to note is the lower average color shift achieved with Aktisil MAM, bordered in red. Similarly positive results were nowhere near being achieved with the control fillers, even when using cleaning agent and an abrasive pad. The color values in the right-hand diagram in *Fig. 7* indicate even higher average residual staining, even after the more intense cleaning. The disadvantages for competitor fillers and, as it were, the advantages for the Neuburg Siliceous Earth become even clearer when the formulation with Aktisil MAM is also subjected to a similar cleaning step.

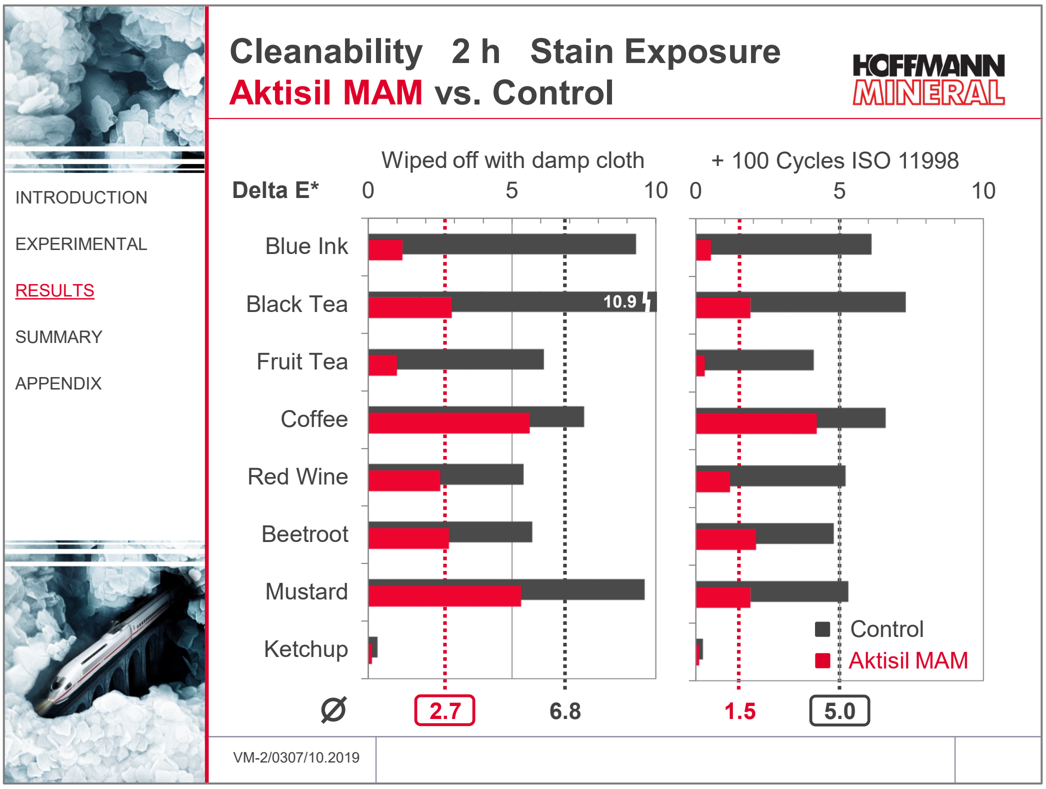


Fig. 7

The results demonstrate that Aktisil MAM actively counteracts penetration of the stain into the paint layer thanks to its special properties and that subsequent cleaning processes can be performed not only more effectively, but also can be carried out under milder conditions. Even with more intense cleaning, residual staining can be eliminated more easily and 70% more effectively relative to the control, as indicated by the very low delta E* values of the wet-scrubbed sample.

3.2.4 Visual appearance of cleaned coatings

Fig. 8 focuses on the abraded middle section of the coating films, which is shown here separately and visually spread out.

The diagrams underscore the cleanable nature of this emulsion paint. The overwhelming majority of applied stains can be removed relatively easily and effectively, provided cleaning attempts are made within the first few minutes. But even in this early stage, a cleaner coating surface underlines the effectiveness of Aktisil MAM with almost complete removal of stains relative to the control.

Prolonging the stain exposure time, however, will have a noticeably negative impact on cleaning options. Only ketchup is easy to remove in all formulation variations. With regard to the control formulation, all other test media lead to visible stain formation. Replacing the competitor fillers with Aktisil MAM improves its characteristics. Stain formation is visibly reduced and cleanability is optimized. Significantly reduced stains demonstrate a high level of resistance to soiling, even to those test substances which are seen as the most drastic stains, such as black tea and coffee.

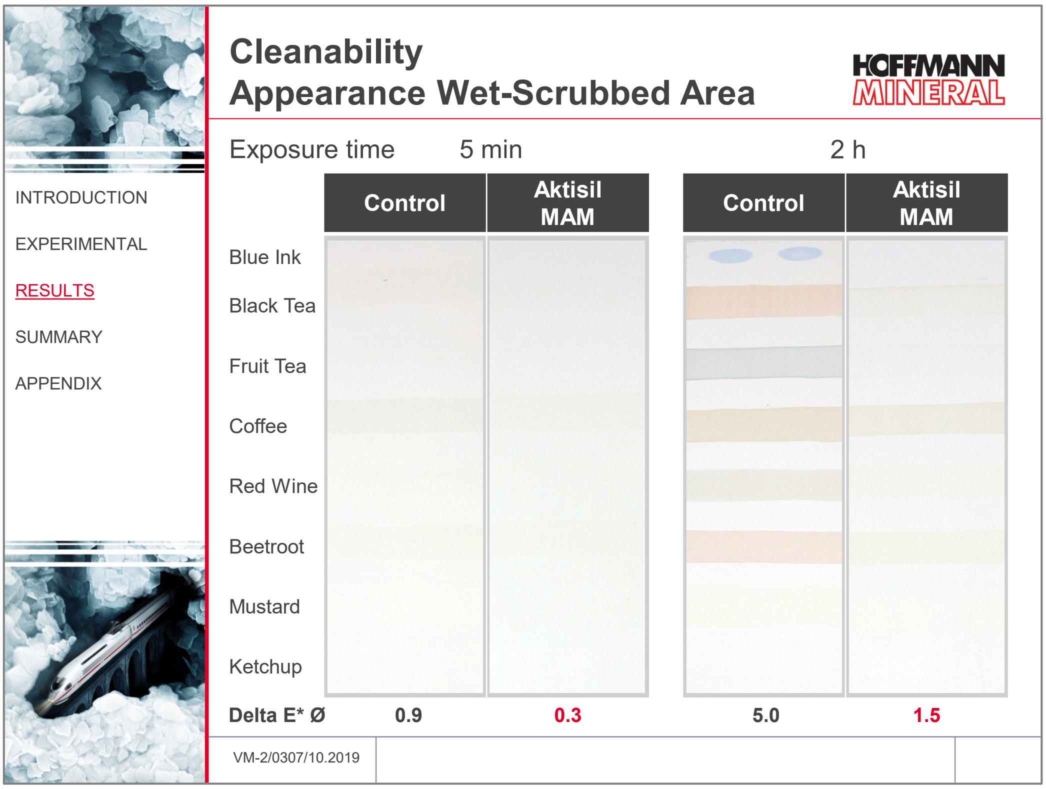


Fig. 8

In *Fig. 9* there is also visual confirmation that coatings with Aktisil MAM can be cleaned more effectively by means of simply wiping off with a damp cloth than is possible with the control formulation, even when subjected to intense abrasion with cleaning agent. When using Aktisil MAM, annoying stains can be reduced or even removed more effectively and gently without the need for wet scrubbing the surface.

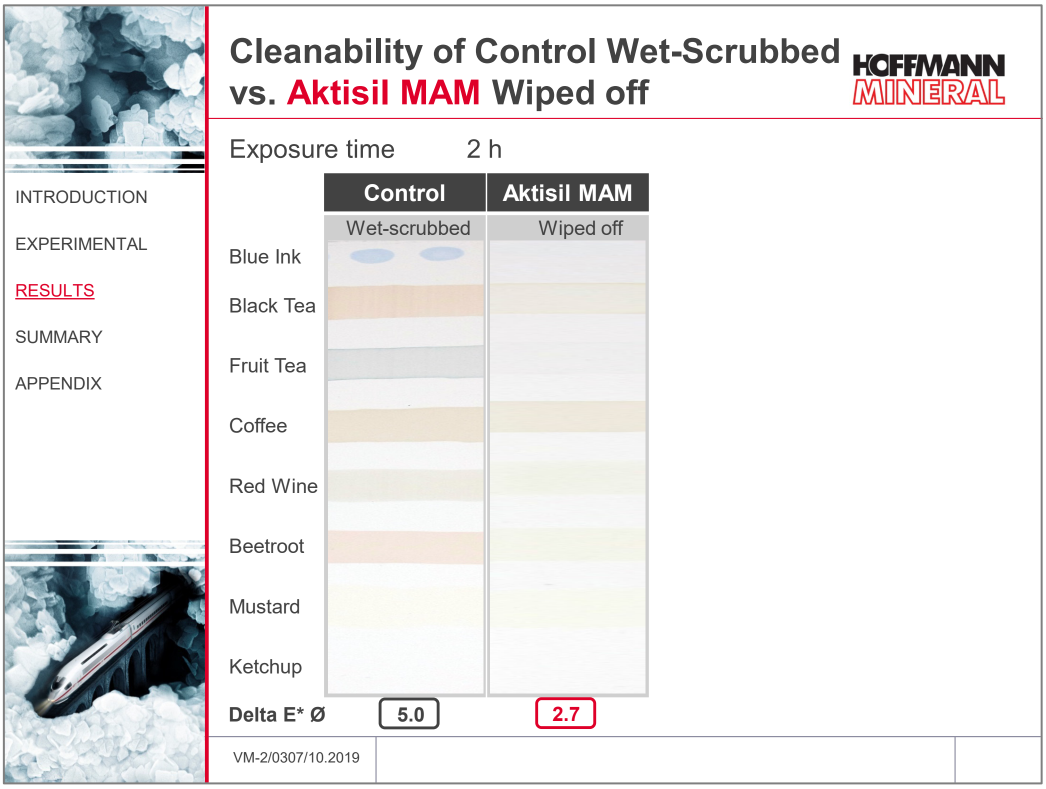


Fig. 9

3.3 Wet-scrub resistance

Assessing the effects of fillers on cleanability requires identical mechanical resistance of the coating surfaces during the cleaning process. If this condition is not met, good properties can theoretically only be simulated by means of excessive removal of material and staining during wet scrubbing (see, for example, film application in *Fig. 10* with heavily reduced discoloration from red wine after abrasion of a film with relatively poor wet-scrub resistance).

The test was performed on the abrasion tester in accordance with ISO 11998 using unstained coating strips, after which the effective layer thickness loss was measured after 200 cycles. The test produced values that are still in the range of the best wet-scrub class 1, in accordance with EN 13300, for the control formulation with 4 to 5 µm removal.

Through the use of Neuburg Siliceous Earth, good mechanical strength can be optimized further with Aktisil MAM and can achieve the lowest wet-scrub abrasion with 3 µm and the highest durability level.

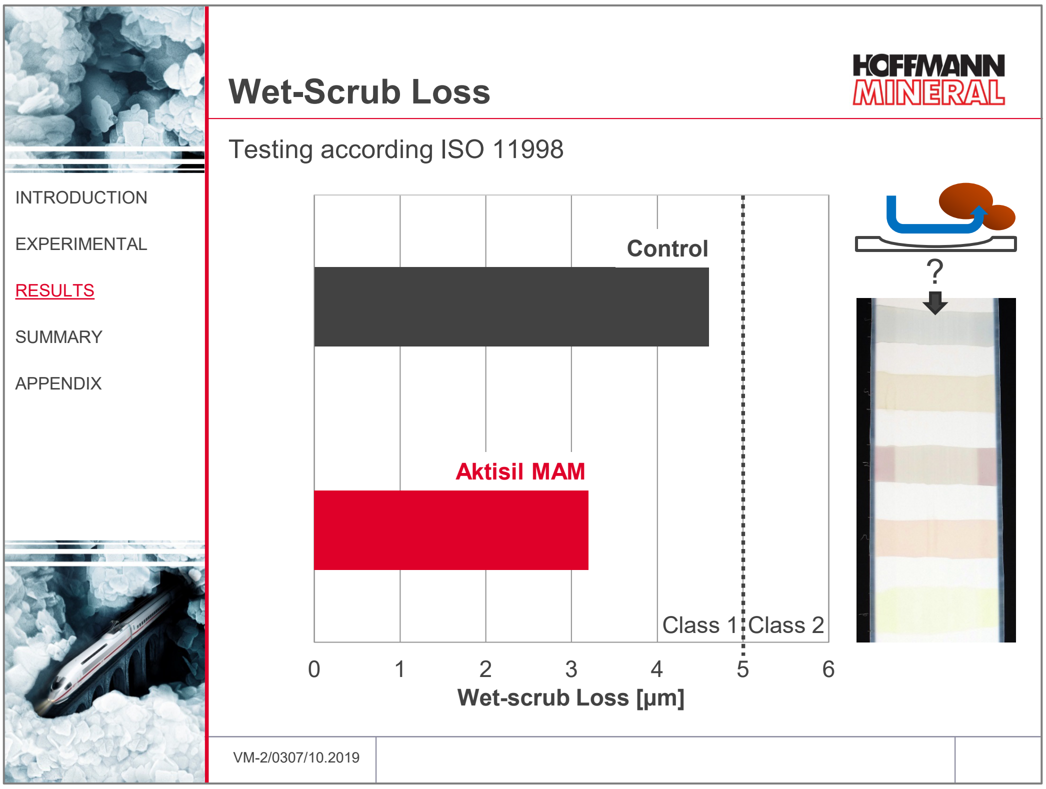


Fig. 10

3.4 Combined cleanability / wet-scrub resistance rating

Once the wet-scrub resistances have been determined, the performance of the tested filler variations can be evaluated objectively and graphically represented in accordance with *Fig. 11*.

The point of origin represents the goal of optimal cleanability, with complete removal of the stain without compromising the layer thickness.

After prolonged exposure to the test substances, the control formulation exhibits noticeable staining and high delta E* values. At the same time, the mechanical resistance permits classification still within the best wet-scrub class 1.

The Neuburg Siliceous Earth grade has significant advantages with regard to both testing criteria. The gain in terms of cleanability is primarily the significantly lower residual soiling, owing to its higher staining resistance.

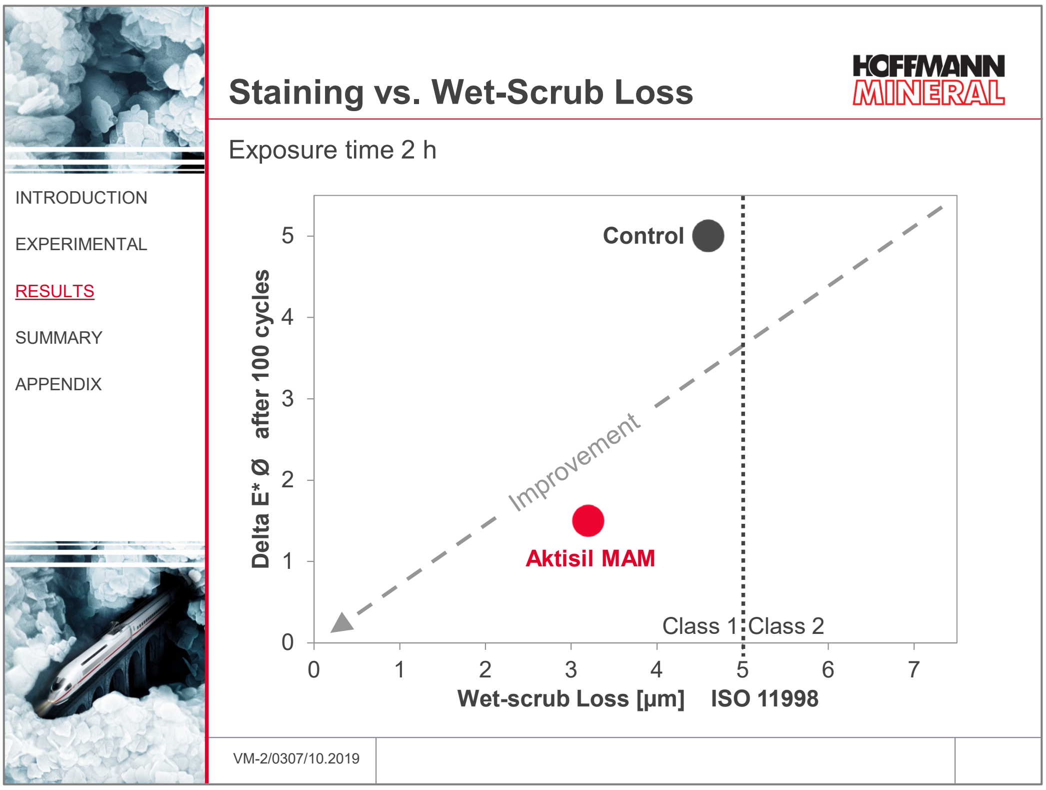


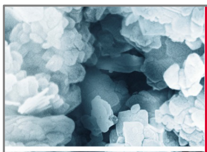
Fig. 11

Using Aktisil MAM causes a considerable improvement in effectiveness overall. Staining resistance and cleanability of the present formulation are optimized significantly. Stubborn stains as a result of prolonged exposure or highly critical substances can be reduced or even avoided completely. In the case of more intense mechanical cleaning, the Neuburg Siliceous Earth variant meets the most pertinent requirements thanks to improved wet-scrub resistance.

The initial question on whether there were further options for optimization of cleanability can therefore be answered with a clear yes if Aktisil MAM is used.

3.5 Other characteristics for the overall evaluation

In order to assess the potential of the Neuburg Siliceous Earth more exactly, other properties were examined which are important for the visual appearance of cleanable emulsion paints. The results are presented with previous data in *Fig. 12* in the form of a summarized comparison.



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Performance Overview

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		Control	Neuburg Siliceous Earth
Diatomaceous Earth		33 pbw	166 pbw
Nepheline Syenite, coarse		100 pbw	
Nepheline Syenite, fine		33 pbw	
			Aktisil MAM
Key features			
Delta E* Ø wiped off	5 min	1.6	0.8
+ wet-scrubbed	5 min	0.9	0.3
Delta E* Ø wiped off	2 h	6.8	2.7
+ wet-scrubbed	2 h	5.0	1.5
Wet-scrub loss ISO 11998	[µm]	4.6	3.2
Further properties			
Gloss 85°	[GU]	3.7	9.7
Dry Burnish	+ [GU]	0.7	1.4
Brightness L*		96.3	96.4
Spreading rate at contrast ratio 98 %	[m²/l]	9.5	11.8

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Fig. 12

With regard to the gloss, Aktisil MAM produces a slightly higher level with matt gloss gradations in comparison to competitor fillers."Dry burnish" refers to the visual polishing by means of dry, mechanical friction of the coating surface. Owing to frequent friction with textiles or other objects, some areas may appear glossier than others, thus compromising the homogeneous look of the wall surface. In both variants, only a low absolute gloss increase was found during testing, and this level is practically unnoticeable in terms of overall appearance. Use of Aktisil MAM leads to an almost similar level of resistance to polishing compared to the control formulation: A gloss increase being slightly higher, but also starting from a higher value.

Aktisil MAM offers high brightness values and extraordinarily good hiding power with regard to visual requirements. This improves the spreading rate of the formulation with Aktisil MAM by almost 25%.

3.6 Cleanability when varying the binder

The effects of Neuburg Siliceous Earth can also be proven in other binders. To this end, the original binder was replaced by “Acronal ECO 6270” from BASF, which exhibits the following properties:

- Environmentally friendly pure acrylic binder
- Widely used in high-quality interior and exterior paints
- Similar physical characteristics
- Component of interior paints that have already been internally tested and that have used the calcined Neuburg Siliceous Earth for partial TiO_2 substitution to positive effect

The binder was replaced by equal weight approximately maintaining the same level of pigment volume concentration and solids content. All other components remain unchanged in the formulation. *Fig. 13* illustrates the results of the cleanability evaluation.

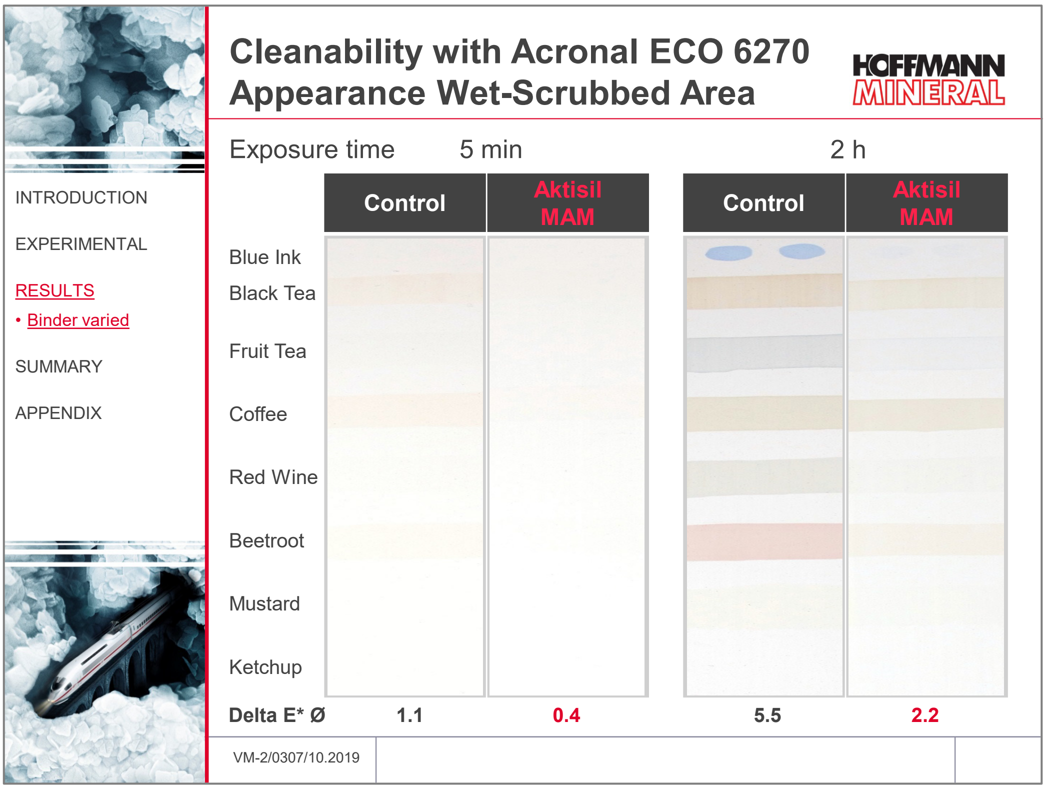


Fig. 13

By varying the binder used, the good cleanability of the formulation is only marginally reduced. In these results as well, black tea, coffee, and red beetroot are found to be critical staining media which can nonetheless still be removed easily, provided the exposure time was short.

In both short and prolonged exposure to the test substance, Aktisil MAM confirms the previous results showing very good staining resistance and optimized cleanability. Further investigations into wet scrubbing and visual characteristics attest to the particular effectiveness of Aktisil MAM and confirm the transferability of the positive effects to other binders as well.

4 Summary

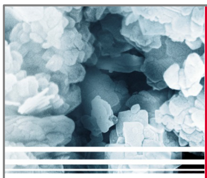
Compared to the diatomaceous earth and nepheline syenite control fillers, Aktisil MAM exhibits better performance for cleanable, low-gloss interior emulsion paints, preserving processing properties and storage stability:

- Superior staining resistance and significantly lower soiling
- Easier and gentle stain removal even with just wiping off with a damp cloth
- Optimized wet-scrub resistance
- Good dry burnish resistance
- Low gloss
- Improved hiding power and optimized spreading rate at high brightness
- Formulation with just one single filler; can nonetheless be combined freely with other fillers
- Transferability of effectiveness to other binders or formulations

This characteristics profile offers the following advantages for the user:

- Reduced soiling, lower cleaning requirements, and very good mechanical durability for the preservation of clean surfaces with a longer service life
- Adjustable low gloss level, combination with matting agents for dull matt surfaces when required
- More cost-effective formulation thanks to visibly higher spreading rates at low consumption
- Improved and highly balanced property profile based on a single filler

A recommendation for the formulation with Neuburg Siliceous Earth can be found in *Fig. 14* on the next page.



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Starting Formulations

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Easy-to-clean interior dispersion paint without co-solvent cleanable, highly wet-scrub resistant, dry burnish resistant, matt, top hiding	
Water deionized	148.4
Natrosol 250 HR	2.2
Dispex CX 4320	7.0
Acticide MV	1.0
Silres BS 16, 20 % in water	3.4
Foamaster MO 2150	2.0
Tronox CR 828	257.0
Aktisil MAM	166.0
Acronal PLUS 6282	407.0
Rheovis HS 1212	2.0
Foamaster MO 2150	4.0
Total Dilution with 5 % deionized water	**1000.0**
PVC [%]	41.4
Solids content w/w [%]	60.1

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Fig. 14

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