

Neuburg Siliceous Earth

for epoxy pipeline powder coatings

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1 Introduction


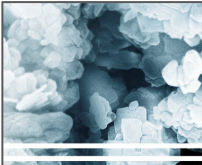
The following study will compare Neuburg Siliceous Earth (Neuburger Kieselerde) with various other fillers in a pipeline powder coating based on epoxy resin. The objective of the work was to obtain an improvement of the property profile of the coatings by changing the nature of the filler.

The project was carried out at the iLF Institute in Magdeburg, Germany, under the supervision of Dipl. Ing. Markus Witter between November 2005 and May 2006. Corrosion and chemical resistance tests (except cathodic disbondment) were run at the Coatings Laboratory of Hoffmann Mineral.

2 Experimental

2.1 Base formulation and variations

The guide formulation in *Fig.1* from the Hexion Company (ex Resolution) served as the starting point of the study. Epikote 1055 is a solid epoxy resin based on Bisphenol A / epichlorohydrine with an epoxide equivalent mass of about 850; Epi-Cure 104, an accelerated dicyanamide, was used as hardener.



Formulation		HOFFMANN MINERAL
Base formulation (from Hexion)		
		Parts per weight
Epikote 1055	Solid epoxy resin	827
Epi-cure Curing Agent P-104	Hardener	33
BYK 368-P	Levelling agent	10
Iron oxide red 3296	Color pigment	15
Barite	Filler	120
Total		1005
Density		1.32
PVC (%)		4.2

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Fig. 1

The fillers were varied as follows. Starting with 120 parts by weight (pbw) of barite, corresponding to PVC of 4.2 %, a formulation was included entirely without filler as a zero control. Sillitin Z 86 was incorporated, maintaining the same PVC as with the barite. In view of existing industrial formulations, the PVC was then increased to 7.6 % with the fillers listed in Fig. 2.

		Formulation										
		HOFFMANN MINERAL										
<p>EXPERIMENTAL</p> <p>RESULTS</p> <ul style="list-style-type: none"> • Application Properties • Optical Properties • Mechanical Properties • Resistance Properties <p>SUMMARY</p>	Filler variations											
	Without filler											
	Barite		120									
	Sillitin Z 86			74								
	Barite				240							
	Sillitin Z 86					149						
	Aktisil AM					149						
	Aktisil MM					149						
	(NCC) Natural Calcium Carbonat						154					
	Wollastonite							163				
	Clay (Kaolin)								149			
	Mica									162		
	Talc										159	
	PVC (%)		0.4	4.2	4.2	7.6	7.6	7.6	7.6	7.6	7.6	7.6
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Fig. 2

2.2 Fillers used and their characteristics

Neuburg Siliceous Earth, extracted in the surrounding of Neuburg (Danube), is a natural combination of corpuscular Neuburg Silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods. As a result of natural formation, the silica portion exhibits a round grain shape and consists of aggregated, cryptocrystalline primary particles of about 200 nm diameter. This structure explains the relatively high surface area and oil number, aside from the rheological activity the reasons for good mechanical properties.

For the Neuburg Siliceous Earth, as base materials were selected Sillitin Z 86 as well as the surface modified grades Aktisil AM (an activated Sillitin Z 86 whose surface was modified with an amino functional group) and an analogous grade Aktisil MM (surface treated with a mercapto functional group). The reference fillers included barite, calcium carbonate, wollastonite, clay, mica and talc.

Fig. 3 lists the medium and higher particle sizes, oil numbers and specific surface areas of the fillers used. Apart from the somewhat coarser wollastonite and mica, the fillers are largely comparable with respect to their particle size. Barite and calcium carbonate are characterized by a very low oil number, all the others range between 47 and 66 g of oil per 100 g of filler. Barite has the smallest surface area with 0.7 m²/g, followed by wollastonite (1.1 m²/g) and calcium carbonate (3.4 m²/g). Mica and talc are located at the same level of about 5 m²/g. Aktisil MM, AM and Sillitin Z 86 exhibit higher BET surface areas between 7.4 and 11.1 m²/g, and clay takes the top with 17.8 m²/g.

	Particle Size		Oil Absorption	Surface Area - BET	Surface Treatment
	d_{50}	d_{97}			
	[μm]	[μm]	[g/100 g]	[m^2/g]	
Barite	4.3	17	12	0.7	none
Sillitin Z 86	1.9	8	47	11.1	none
Aktisil AM	2.2	10	53	9.0	amino functionalized
Aktisil MM	2.3	10	48	7.4	mercapto functionalized
NCC	1.9	7	27	3.4	none
Wollastonite	12.3	77	55	1.1	none
Clay	2.0	11	65	17.8	none
Mica	14.5	53	66	5.7	none
Talc	7.1	20	55	4.5	none

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Fig. 3

2.3 Preparation of the coatings and application

The individual components of the powder coating formulations were weighed out and mixed for 10 min at 500 min^{-1} in an 'Universalmischer' from MIT Mischtechnik GmbH. This premix was then extruded in a twin-screw extruder MP 19 (from APV Baker; $D = 19 \text{ mm}$, $L = 25 D$). In the entry zone, the temperature was controlled at $100 \text{ }^\circ\text{C}$, in the process zone at $120 \text{ }^\circ\text{C}$. Extrusion was done at 300 rpm with a load of 55 to 65 %.

The extrudates were coarsely broken up and ground in an ultracentrifuge mill (Pulverisette 14 from Fritsch) at 16.000 min^{-1} and finally sieved through a mesh of $100 \mu\text{m}$ size.

According to the formulations, application followed two routes:

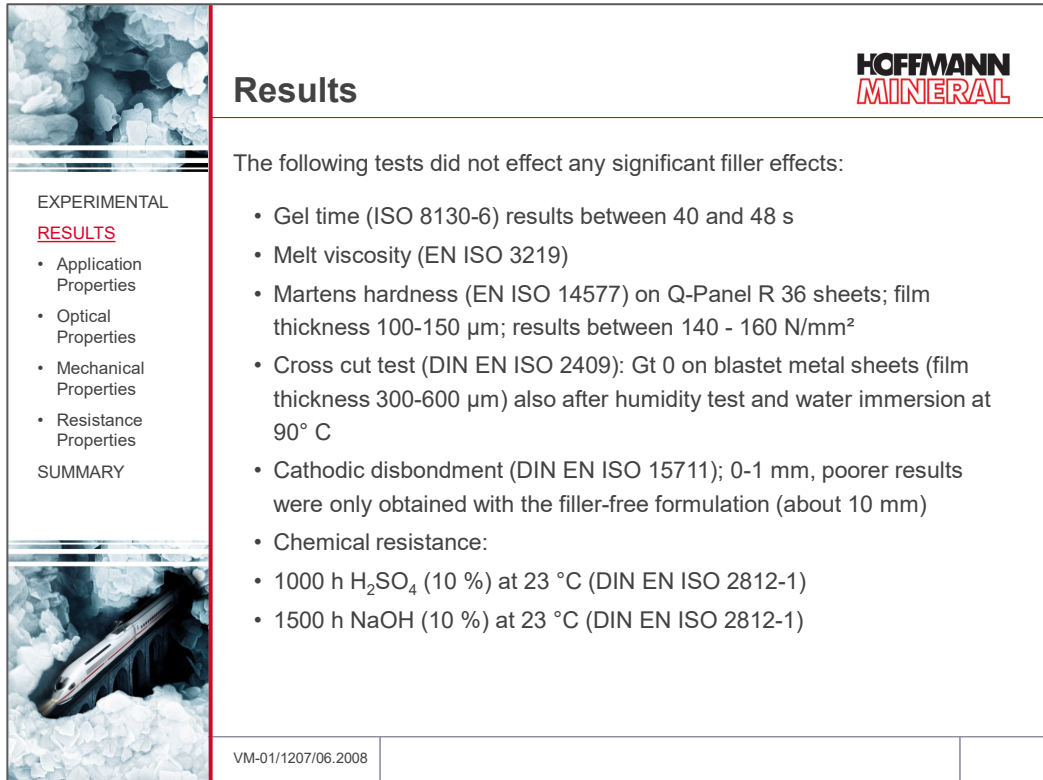
- with a Corona powder gun (70 kV) onto Q-Panel R 36 steel sheets for the optical and Martens hardness tests, film thickness $100 - 150 \mu\text{m}$
- via tribodeposition on sandblasted steel sheets (surface roughness $50 - 70 \mu\text{m}$) pre-heated to $200 \text{ }^\circ\text{C}$ in order to ensure the high film thicknesses of $300 - 500 \mu\text{m}$, for the mechanical tests except Martens hardness

Hardening was carried out at an object temperature of $200 \text{ }^\circ\text{C}$ for 10 min.

3 Results

3.1 Tests without significant filler effects

The tests listed in *Fig. 4* were carried out, but they will not be discussed here in detail, as no significant effects of the individual fillers could be observed. The cathodic disbondment was tested in 3 % NaCl (potential: -1050 mV) for 38 days at 23 °C, but no delamination was seen. The test severity was then increased to a higher temperature, and after 38 days at 60 °C all fillers showed a delamination of 0 - 1 mm, only the control without filler was higher at about 10 mm.



Results

**HOFFMANN
MINERAL**

The following tests did not effect any significant filler effects:

- Gel time (ISO 8130-6) results between 40 and 48 s
- Melt viscosity (EN ISO 3219)
- Martens hardness (EN ISO 14577) on Q-Panel R 36 sheets; film thickness 100-150 µm; results between 140 - 160 N/mm²
- Cross cut test (DIN EN ISO 2409): Gt 0 on blastet metal sheets (film thickness 300-600 µm) also after humidity test and water immersion at 90° C
- Cathodic disbondment (DIN EN ISO 15711); 0-1 mm, poorer results were only obtained with the filler-free formulation (about 10 mm)
- Chemical resistance:
 - 1000 h H₂SO₄ (10 %) at 23 °C (DIN EN ISO 2812-1)
 - 1500 h NaOH (10 %) at 23 °C (DIN EN ISO 2812-1)

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Fig. 4

3.2 Fluidizability

Fluidizability was tested according to DIN ISO 8130-8 in a Fluidimeter AS 100 (from Sames). The Fluidimeter is filled with 250 g of the coating powder, and clean dry air is introduced through the porous bottom with a throughput sufficient for optimum fluidizing of the sample. The sample will be stirred by hand with a spatula for such a time until the height of the fluid bed remains constant. The height of the fluidized coating powder is measured and noted as "h1". After stopping the air supply and settling of the powder, the height of the settled powder is measured as „h0“. Again, the powder is fluidized with the same air throughput as before. After obtaining a constant height of the fluid bed, the amount „m“ of coating powder is determined which discharges through an opening in the container. The flow rate „R“ is calculated as $m \times (h1/h0)$.

Of course, the tests were carried out without the addition of free-flow agents.

Good fluidizability was found for the control without filler, with Sillitin Z 86, Aktisil AM, Aktisil MM and the clay. By contrast, the powders with calcium carbonate and talc are very difficult to fluidize (Fig. 5).

		Fluidizability ISO 8130-8		HOFFMANN MINERAL	
			Assessment of Fluidizability		
EXPERIMENTAL RESULTS <ul style="list-style-type: none"> • Application Properties • Optical Properties • Mechanical Properties • Resistance Properties SUMMARY	PVK 0.4	Without filler	+		
	PVK 4.2	Barite	0		
		Sillitin Z 86	+		
	PVK 7.6	Barite	0		
		Sillitin Z 86	+		
		Aktisil AM	+		
		Aktisil MM	+		
		NCC	-		
		Wollastonite	0		
		Clay	+		
		Mica	0		
		Talc	-		
		VM-01/1207/06.2008			

Fig. 5

3.3 Deposition efficiency

The deposition efficiency characterizes the mass amount of sprayed powder coating that is deposited on a standard object. In this case, the standard object was built up from five steel tubes wrapped in aluminium foil (outer diameter 25 mm, length 500 mm). The foil used for the central tube will be weighed before the test. The powder coating is sprayed onto the objects from a constant distance for 60 seconds. The tubes are placed into the furnace, and after melting the residual (cooled-down) amount of coating is weighed out.

The deposition efficiency «E» (as a mass percentage) is calculated as $E = (m \times 60 \times 100) / (P_f \times t)$, with m = the mass of powder coating deposited onto the foil in grams, t = spray time in seconds, and P_f = the throughput rate of the powder in grams per minute.

The results given in Fig. 6 do not represent absolute figures, but are differentiated according to the application profile. All the same, the fillers can be characterized as follows: a good deposition efficiency is offered by talc. The filler-free control, Sillitin Z 86 (at lower PVC), calcium carbonate, wollastonite and mica do not quite reach the same optimum level. Barite, clay and the Neuburg Siliceous Earth grades at higher PVC do not influence the deposition efficiency compared to the control with barite.

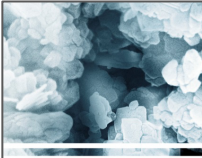

		Deposition efficiency ISO 8130-10		HOFFMANN MINERAL	
		Assessment of Deposition Efficiency	Results in %		
 <p>EXPERIMENTAL</p> <p>RESULTS</p> <ul style="list-style-type: none"> • Application Properties • Optical Properties • Mechanical Properties • Resistance Properties <p>SUMMARY</p> 	PVK 0.4	Without filler	0-	7.5	
	PVK 4.2	Barite	0	9.3	
		Sillitin Z 86	-	5.6	
	PVK 7.6	Barite	0	9.0	
		Sillitin Z 86	0	8.9	
		Aktisil AM	0	8.8	
		Aktisil MM	0	9.7	
	PVK 7.6	NCC	-	6.9	
		Wollastonite	0-	7.8	
		Clay	0	8.3	
		Mica	0-	7.3	
		Talc	+	13.6	
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Fig. 6

3.4 Flow tendency (run-off)

In order to obtain the high film thicknesses, the metal sheets were pre-heated. During the application, therefore, the powder coating started to melt. The assessment of the run-off tendency did show only minor differences between the fillers; the high run-off stability of the talc remains noteworthy, but also the wollastonite and the Neuburg Siliceous Earth grades came up with a reduced tendency to sag (Fig. 7).

		Run-off		HOFFMANN MINERAL	
		Run-off tendency during Stoving			
EXPERIMENTAL RESULTS <ul style="list-style-type: none"> • Application Properties • Optical Properties • Mechanical Properties • Resistance Properties SUMMARY	PVK 0.4	Without filler	-		
	PVK 4.2	Barite	0		
		Sillitin Z 86	-	0 moderate	
	PVK 7.6	Barite	0	- high	
		Sillitin Z 86	+	+ marginal	
		Aktisil AM	+	++ no	
		Aktisil MM	+		
		NCC	0		
		Wollastonite	+		
		Clay	-		
	Mica	0			
	Talc	++			
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Fig. 7

3.5 Gloss at 60°

Gloss was determined on smooth Q-panel sheets with a dry film thickness of about 130 μm (Fig. 8). Most of the fillers imparted a gloss above 90 units, just wollastonite and mica were somewhat lower at 80 to 85 units. Talc gave the lowest gloss with only 70 units; this result looks surprising in view of the rather low PVC.

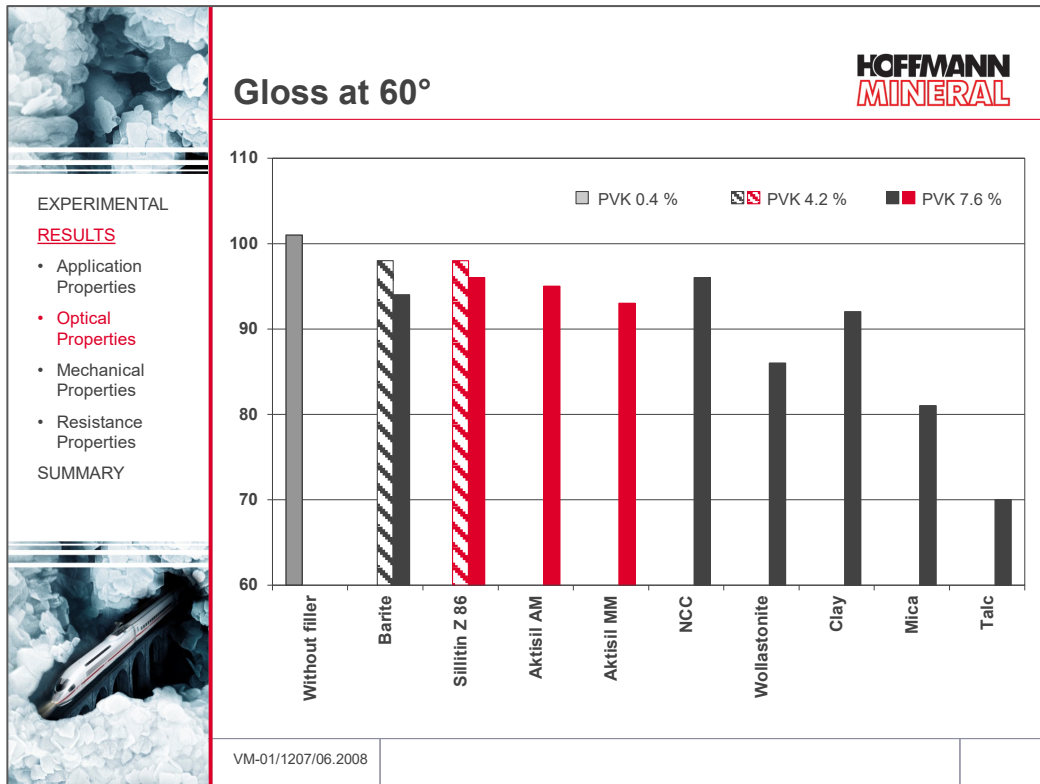


Fig. 8

3.6 Visual assessment of surface appearance

The visual assessment took into account the waviness of the surface and the surface structure (precision of depicting the ceiling lamp in reflexion, following DOI = Distinctness of image), at a film thickness of about 450 µm. The control formulation without any filler came up with the best leveling, with the talc on the lower end (Fig. 9). All others can be rated as moderate.

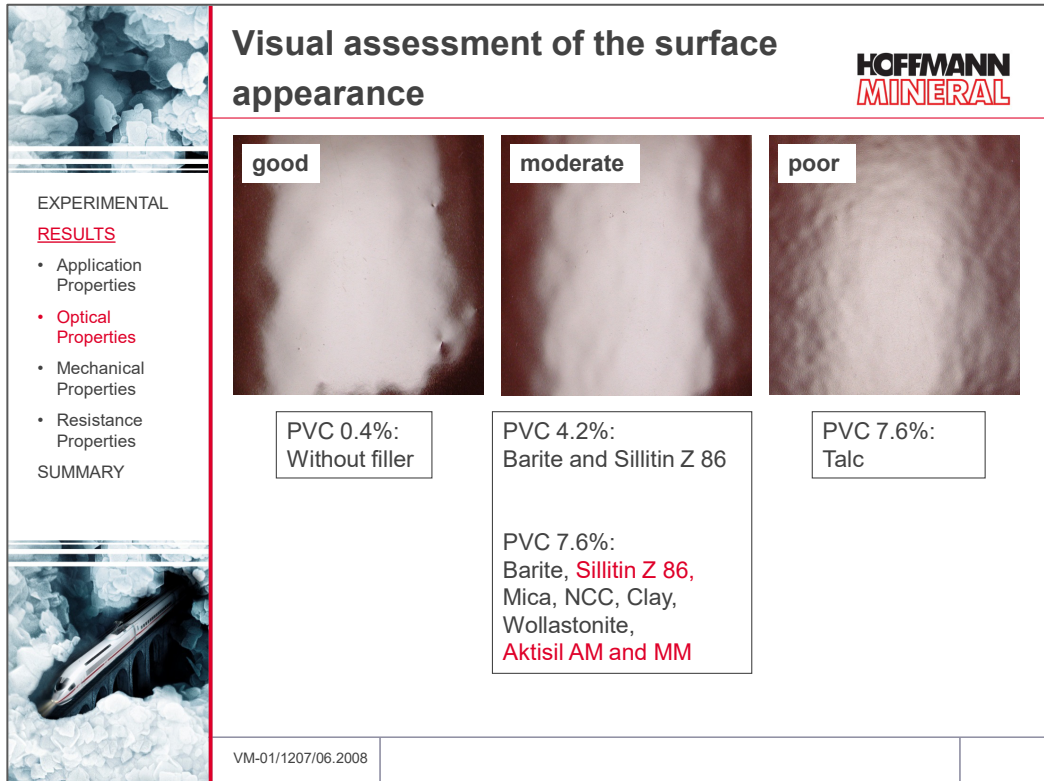


Fig. 9

3.7 Impact Test

In order to test the response to rapid deformation by a falling weight, the Impact Test resp. the Reverse Impact Test was carried out according to DIN EN ISO 6272. The film thickness was between 300 and 500 µm on sandblasted steel sheets. In the Impact Test, a 2 kg weight is dropped onto the coated side, in the Reverse Impact Test onto the uncoated side. The hatched areas of the bars represent the range of measured data.

On the coated side, the individual fillers showed no differentiation, because all results were higher than 200 kg x cm. When the test was run on the backside, the control formulation, Sillitin Z 86 at low PVC and Aktisil MM even in higher concentration gave rise to the best Reverse Impact results above 100 kg x cm.

The fillers used at PVC 7.6 showed the following ranking: Aktisil AM (about 70 kg x cm) > Sillitin Z 86 (about 50 kg x cm) > barite and wollastonite (about 35 kg x cm), clay and mica (about 15 kg x cm) > talc (<10 kg x cm). Neuburg Siliceous Earth offers benefits because of its special structure, and the effects are further increased by the reactive surface treatment of the Aktisils. (Fig. 10)

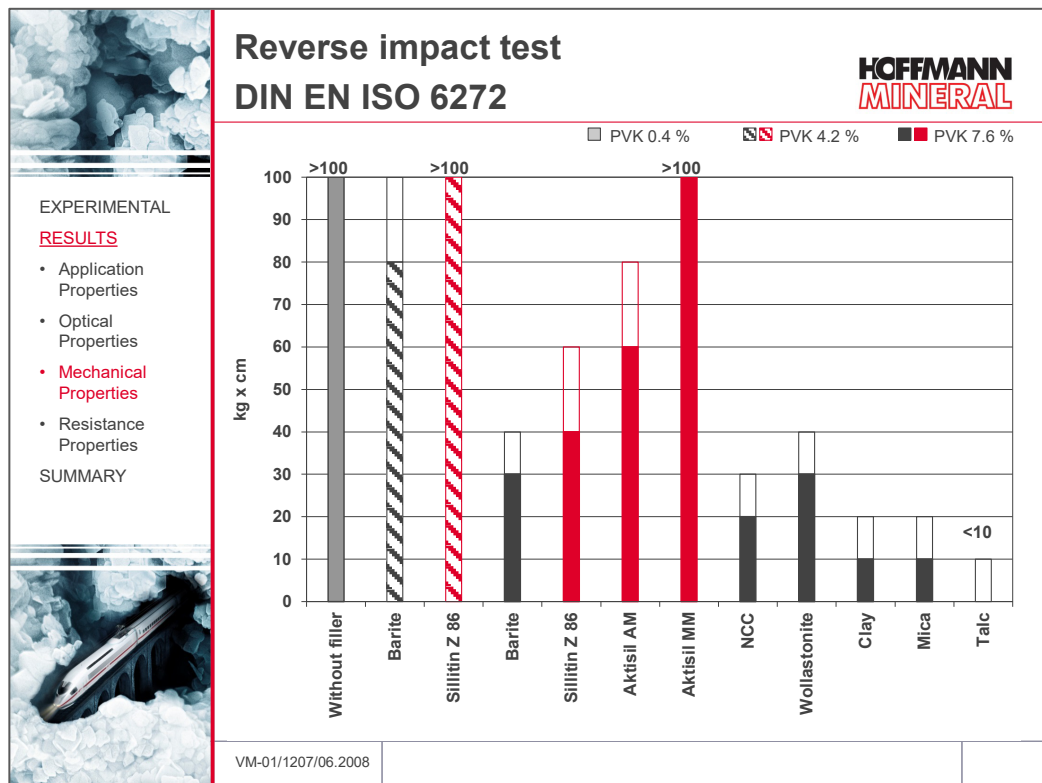


Fig. 10

3.8 Cupping test

In order to check the response to deformation at low speed, the Erichsen Cupping test was run according to DIN EN ISO 1520 (Fig. 11). The film thickness again was between 300 and 500 µm on sandblasted steel sheets. The coatings based on the formulations with low PVC, the filler-free control and all Neuburg Siliceous Earth grades can be deformed to a high degree. Also, calcium carbonate and wollastonite, with 8 - 9 mm, give evidence of rather good flexibility. Clay and barite at the high PVC show cracks already at 6 mm. Early crack formation at about 1 mm due to high rigidity is caused by mica and talc. This way, the results confirm the ranking found in the Reverse Impact test: Sillitin and the Aktisils impart excellent properties.

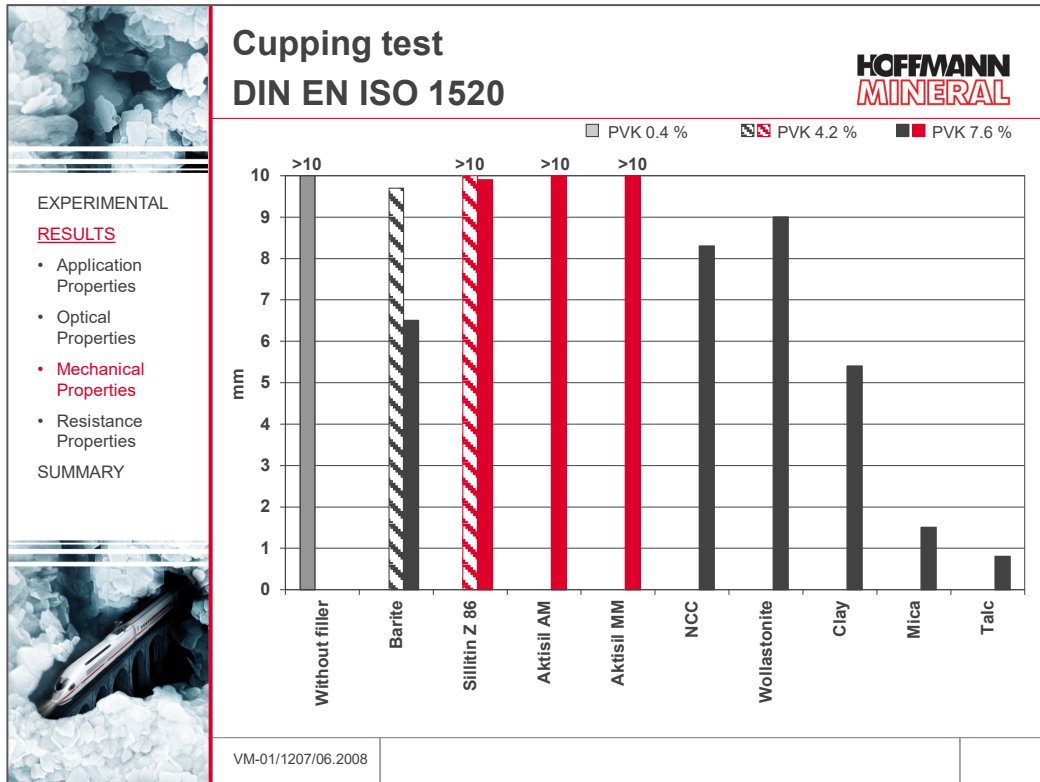


Fig. 11

3.9 Mandrel bending test

A further mechanical test relative to deformation resistance was the Mandrel bending test according to DIN EN ISO 1519. A coated metal sheet with a dry film thickness of 300 to 500 µm is bent around a mandrel with a diameter of 32 mm. Mica and talc flaked off immediately. Barite at the high PVC and calcium carbonate showed medium to large cracks. All other formulations exhibited better flexibility, as only small cracks became visible (Fig. 12).

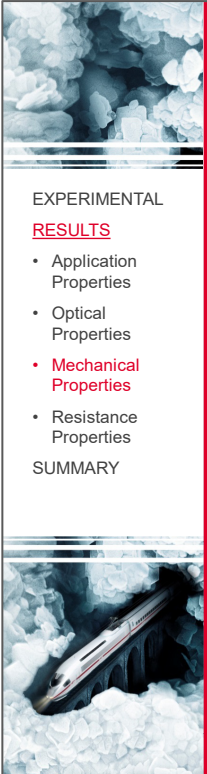
		Mandrel bending test DIN EN ISO 1519		HOFFMANN MINERAL	
				Mandrel Bending Test [mandrel = 32 mm]	
EXPERIMENTAL RESULTS <ul style="list-style-type: none"> • Application Properties • Optical Properties • Mechanical Properties • Resistance Properties SUMMARY		PVK 0.4	Without filler	fine cracks	
		PVK 4.2	Barite	fine cracks	
			Sillitin Z 86	fine cracks	
		PVK 7.6	Barite	large cracks	
			Sillitin Z 86	fine cracks	
			Aktisil AM	fine cracks	
			Aktisil MM	fine cracks	
			NCC	moderate cracks	
			Wollastonite	fine cracks	
			Clay	fine cracks	
			Mica	flake-off	
		Talc	flake-off		
		VM-01/1207/06.2008			

Fig. 12

Summing up the flexibility tests, mica and talc throughout show the least satisfactory results. The Neuburg Siliceous Earth formulations with Sillitin Z 86, Aktisil AM and Aktisil MM are distinguished by outstanding flexibility.

3.10 Abrasion resistance (Taber) with abrader wheels CS 17 and S 42 abrasive paper strips

Abrasion tests were carried out following two different standards:

ASTM D 4060: with abrader wheels CS 17 (moderately abrasive), load 1 kg. Fig. 13 shows the weight loss in milligrams after 1000 revolutions.

DIN 53754: with S 42 abrasive paper strips (markedly more abrasive than CS 17), load 5.4 N. Fig. 14 summarizes the average weight losses in milligrams after 100 revolutions.

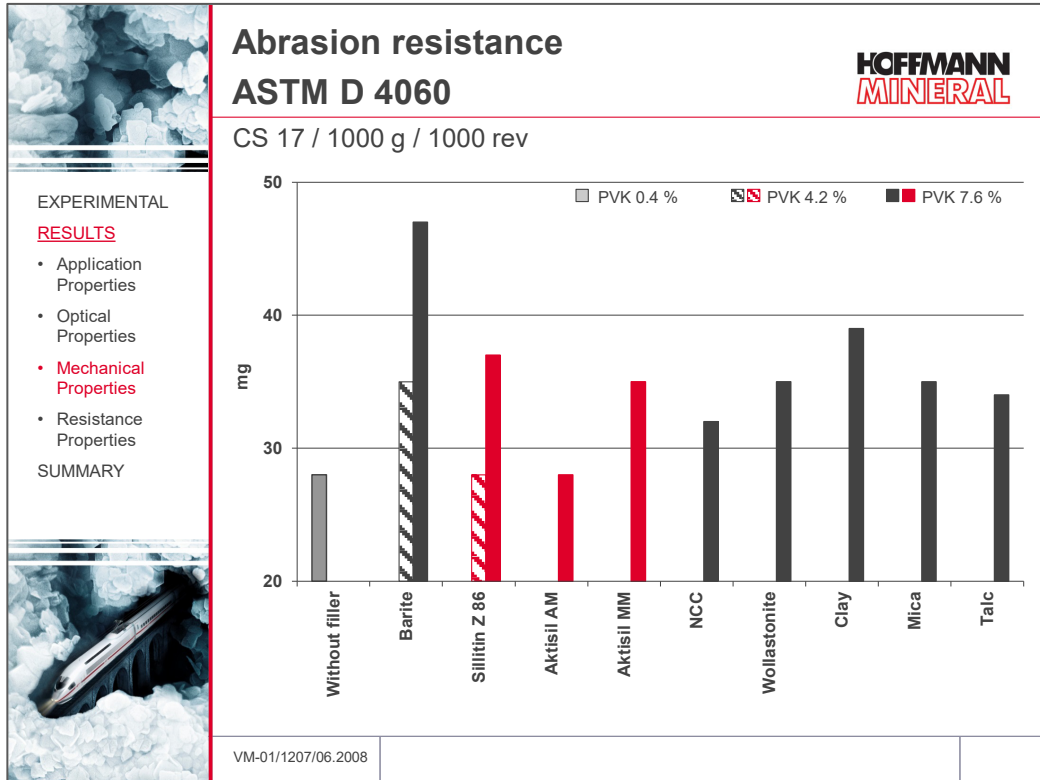


Fig. 13

In the test with the slightly abrasive CS 17 abrader wheels, in particular Sillitin Z 86 at low PVC and Aktisil AM at high PVC stand out with a good abrasion resistance comparable to the control formulation without filler. All other products result in increased abrasion loss, above all the barite.

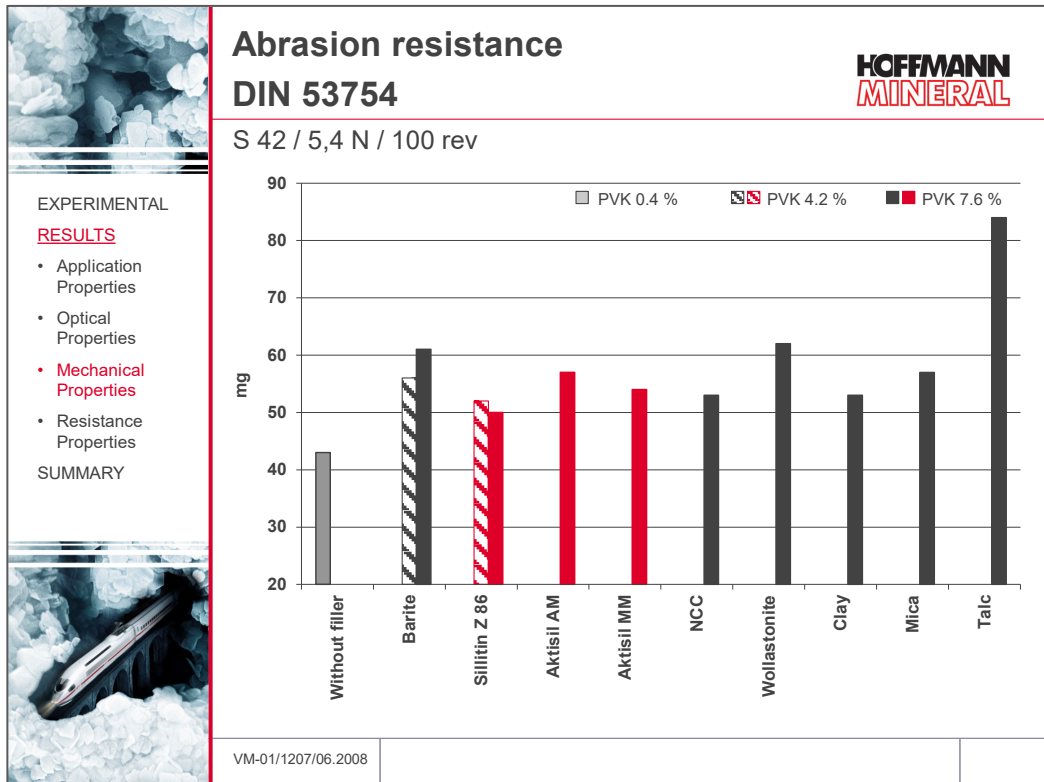


Fig. 14

When tested with the markedly more highly abrasive S 42 strips which do not only abrade the layers near the surface, Sillitn Z 86 at the same PVC gives rise to better results than barite or the other fillers. In particular, talc stands out with a very high abrasion loss which will be due to its low mineralogical hardness.

3.11 Salt spray test

For each formulation, two sandblasted steel sample sheets with a film thickness of 300 - 500 µm were scratched with a scribe stylus according to van der Laar and then subjected for 2000 hours to the salt spray test following DIN EN ISO 9227. The evaluation, as the average of the two sample sheets, in accordance with DIN EN ISO 4628-8, considered the delamination in millimeters (i.e. the average delamination width at the scribe minus the scribe width divided by 2) (Fig. 15).

On the surface as well at the scribe, no blister formation could be observed (i.e., all formulations m 0 / g 0).

Some small degree of delamination could be seen with all Siliceous Earth grades as well as with calcium carbonate, clay, mica and talc. Barite, wollastonite and the control formulation without fillers on average gave twice as severe a delamination.

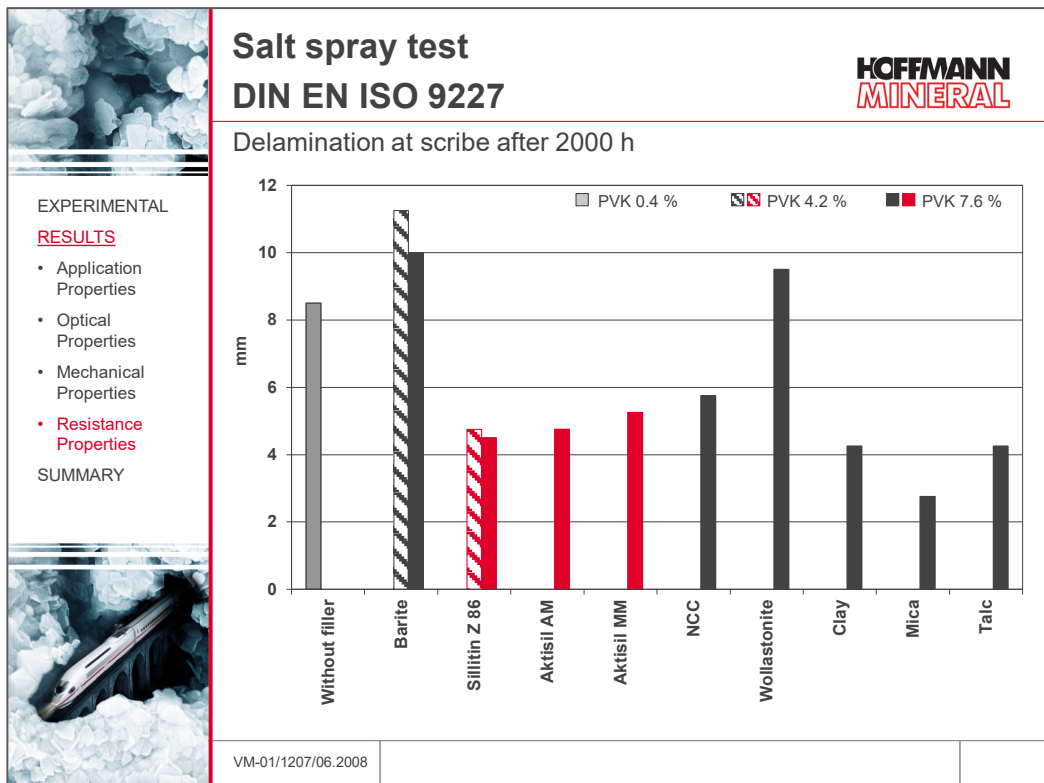


Fig. 15

Fig. 16 illustrates the evaluation as the average of the two sample sheets according to DIN EN ISO 4628-8 via the degree of corrosion (i.e. the average width of rusting at the scribe minus the scribe width divided by 2). A low degree of corrosion is obtained with the control formulation, barite, calcium carbonate, wollastonite and all Neuburg Siliceous Earth grades. Clay, mica and talc give rise to markedly more corrosion at the scribe.

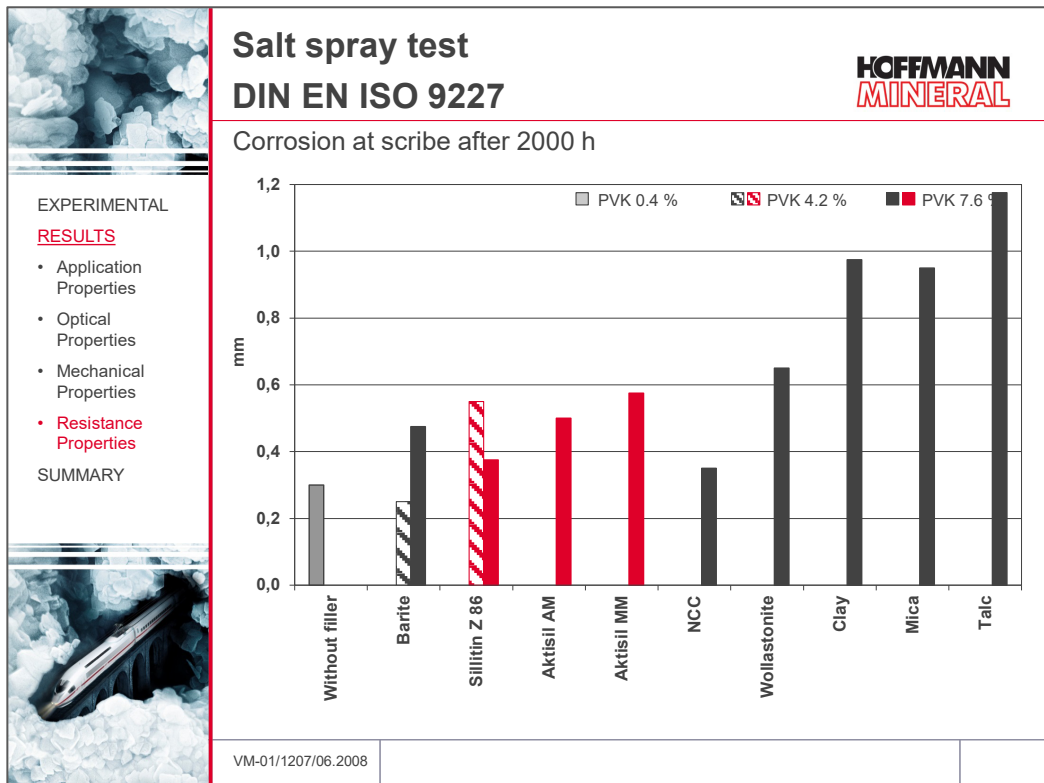


Fig. 16

3.12 Humidity test

Sandblasted metal sheets with a coating film thickness of 300 to 500 µm were scratched with a scribe stylus according to van der Laar, and exposed for 4000 hours to a constant condensation water environment CH (DIN EN ISO 6270-2: 40 °C and 100 % relative humidity). The evaluation again followed DIN EN ISO 4628-8 in defining the degree of delamination in millimeters (i.e., the average width of delamination at the scribe minus the scribe width divided by 2).

On the coating surface, none of the formulations showed blisters. *Fig. 17* visualizes the scribes and the delamination, *Fig. 18* shows the corresponding average delamination in millimeters. Barite and talc give rise to very high, Sillitín Z 86, Aktisil AM, calcium carbonate and mica to very little delamination. The control formulation without filler, wollastonite, clay and Aktisil MM came off without any delamination.

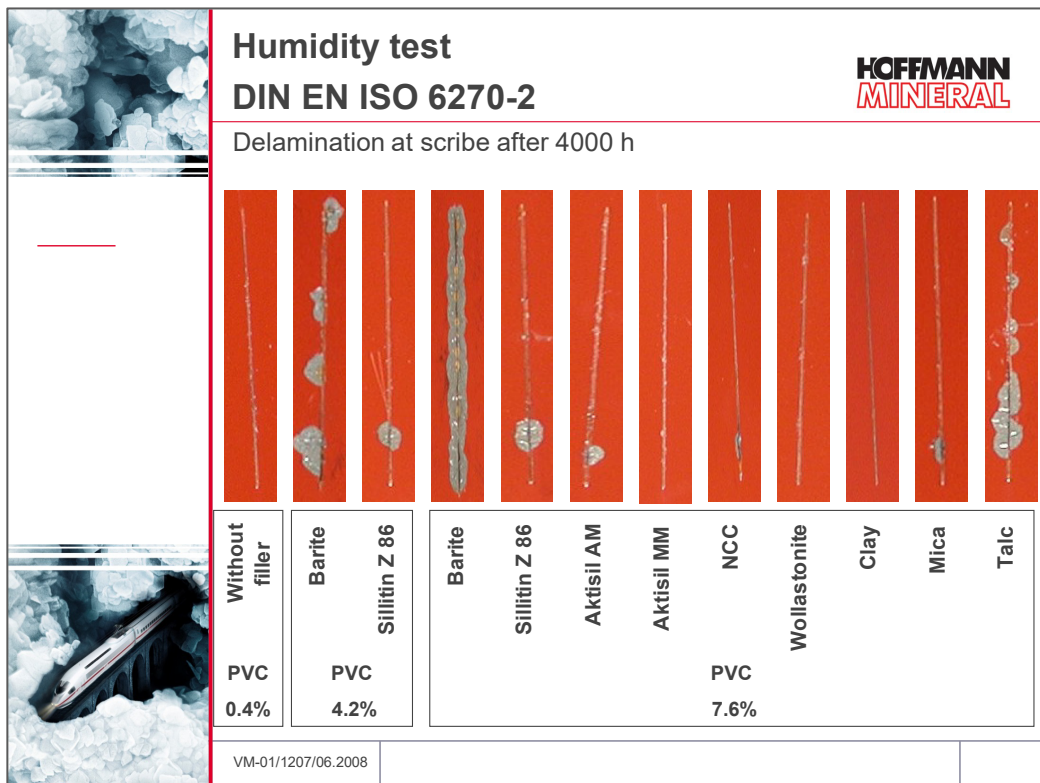


Fig. 17

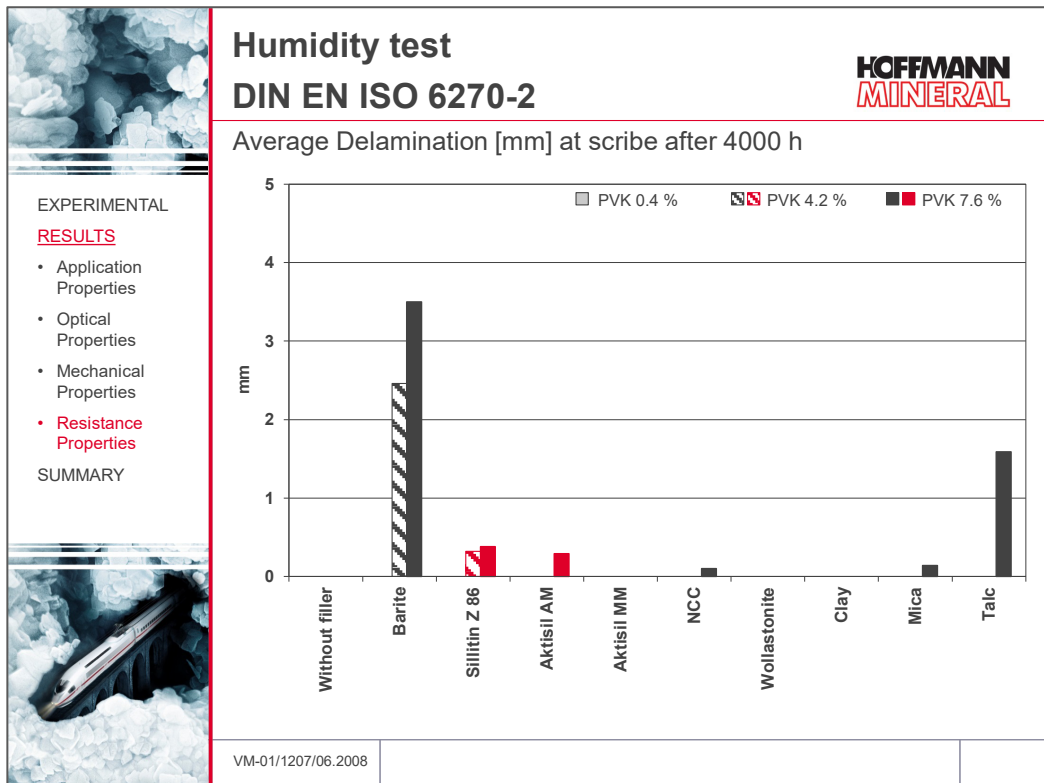


Fig. 18

On unscratched metal sample sheets, after 4000 hours of exposure to the humidity test, the cupping test was run according to DIN EN ISO 1520. The results came out similar to the tests without exposure, apart from barite which turned out better, and wollastonite and clay which turned out poorer (Fig. 19).

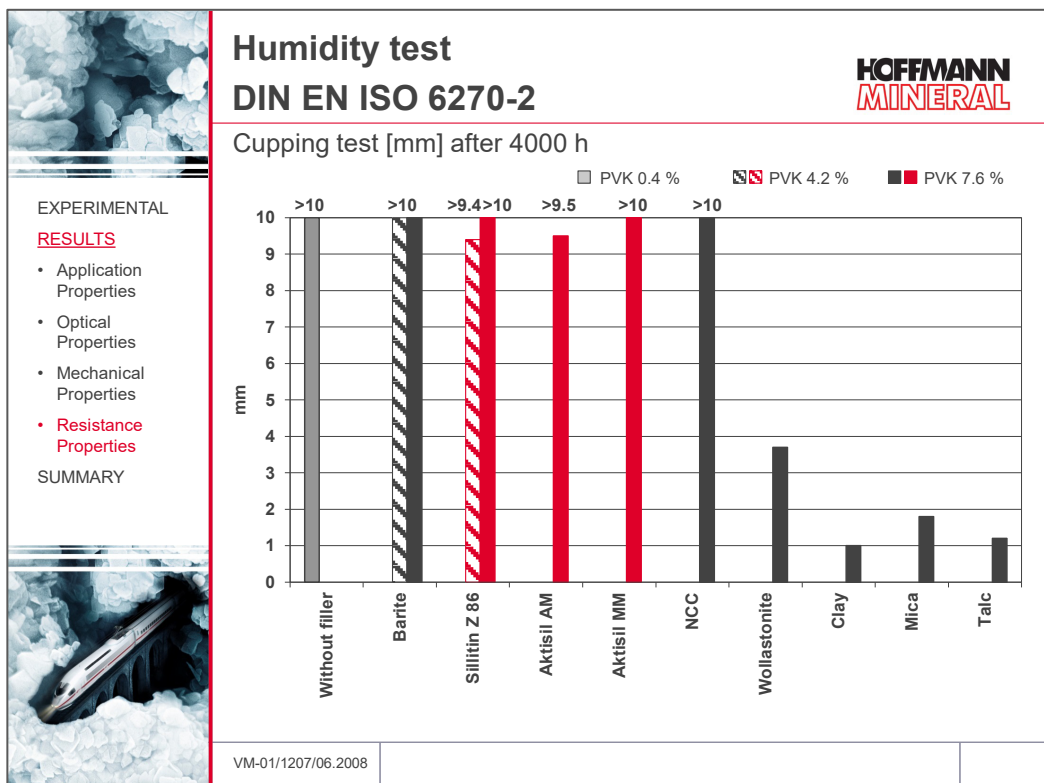


Fig. 19

3.13 Chemical resistance in hot water at 90 °C

Sandblasted steel sample sheets after coating with a film thickness of 300 to 500 µm were immersed for 1700 hours in distilled water at 90 °C. Fig. 20 shows the degree of fading for the individual formulations, Fig. 21 illustrates the corresponding color change in ΔE . The strongest fading is caused by calcium carbonate and wollastonite. Hardly a change is seen with Sillitin Z 86 and Aktisil AM, which consequently offer the best hot water resistance.

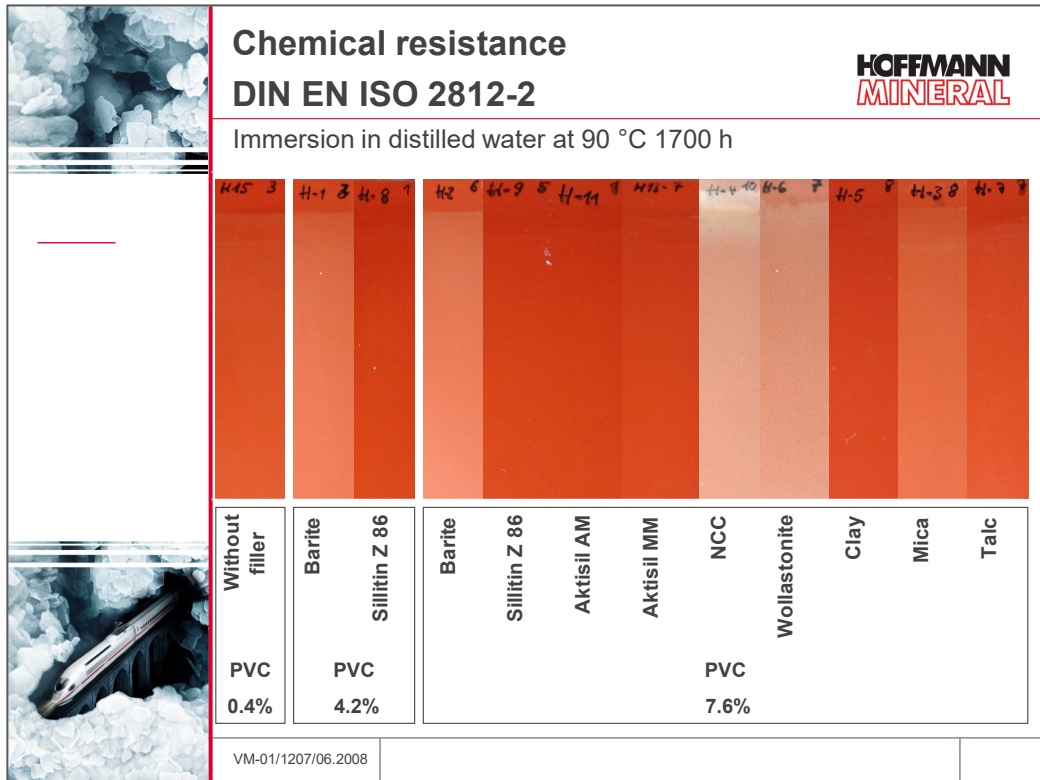


Fig. 20

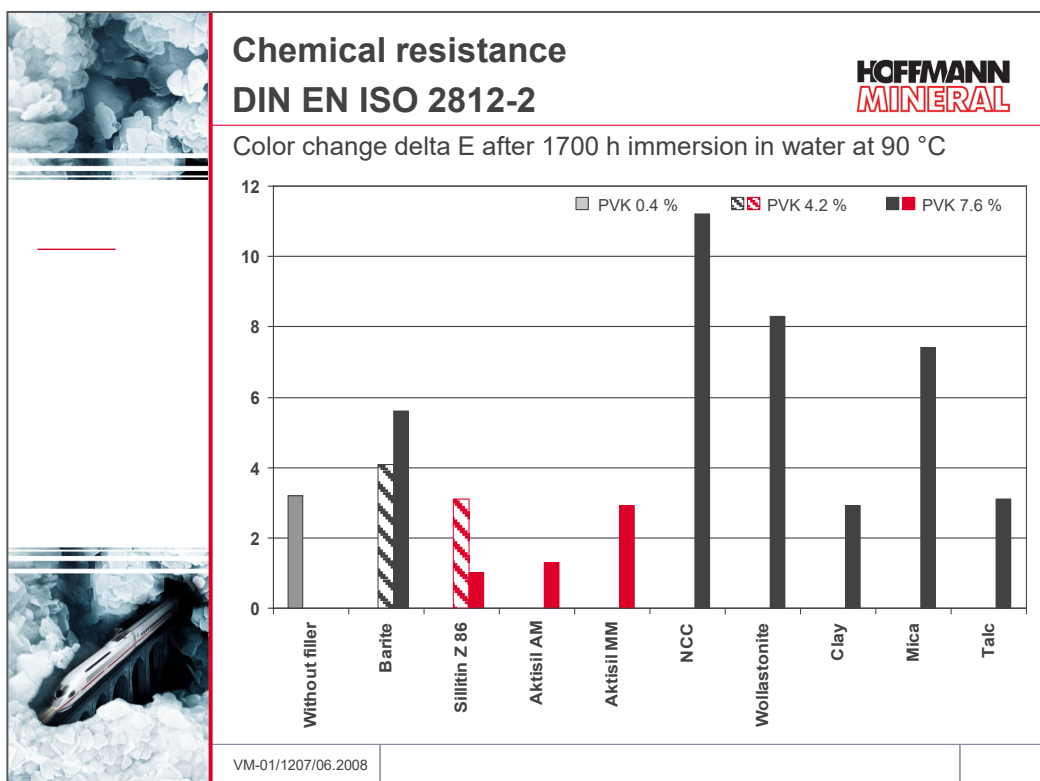


Fig. 21

The remaining relative pendulum hardness after the hot water immersion is summarized in Fig. 22. Here again, calcium carbonate and wollastonite show the poorest results, which means that these formulations have undergone the strongest softening during the exposure.

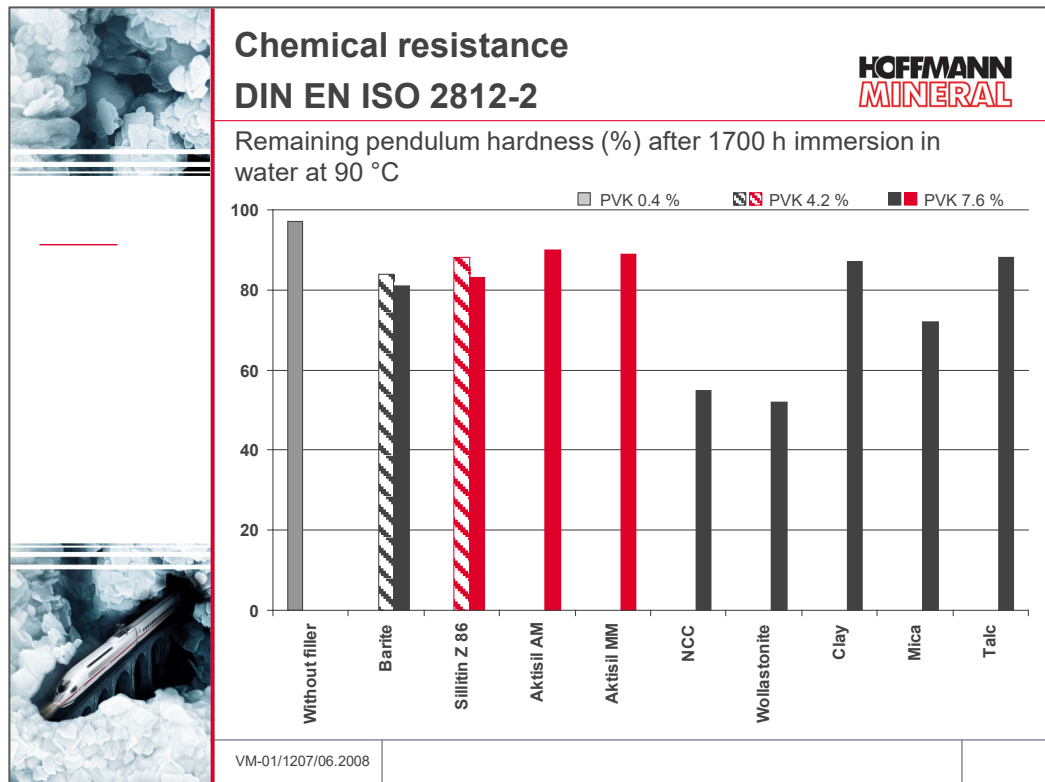


Fig. 22

The remaining relative gloss at 60° after hot water immersion shows a similar ranking of the fillers as the pendulum hardness and the color changes: in particular, calcium carbonate and wollastonite have turned to a dull appearance. Mica during the exposure loses almost half of its gloss, Sillitin Z 86 comes out better than the barite. Very good gloss retention with almost no change during the exposure is offered by the formulations without filler or with Sillitin Z 86 (at PVC 4.2), with clay and talc, and with the two Aktisil grades (Fig. 23).

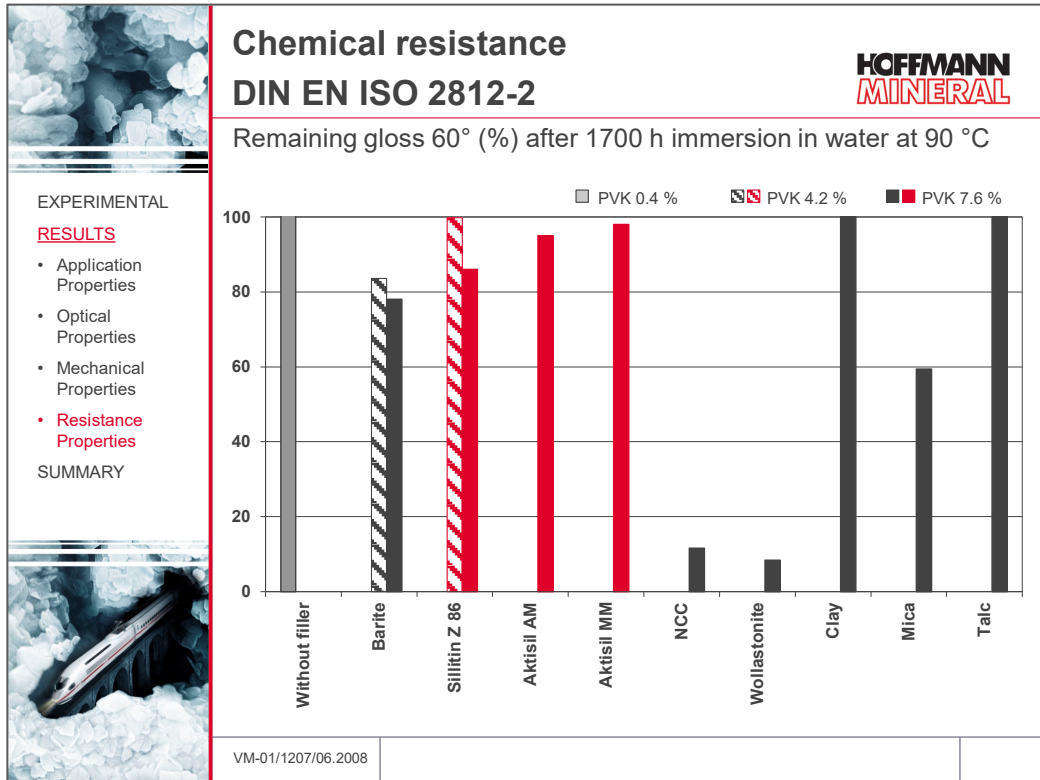


Fig. 23

Summing up the humidity, salt spray and hot water tests, only the Neuburg Siliceous Earth grades Sillitin Z 86, Aktisil AM and Aktisil MM come off with good to very good resistance results throughout.

4. Summary and outlook

Fig. 24 gives a summary of the fillers tested and their advantages and disadvantages in the tests as discussed, reported versus the barite formulation at 4.2 % PVC as reference.

		Summary														Compared to the standard barite PVC 4.2:			
		Fluidizability	Deposition efficiency	Run-off during application (flow tendency)	Gloss at 60°	Visual assessment of surface appearance	Impact test	Reverse impact test	Cupping test	Mandrel bending test	Abrasion loss (CS17)	Abrasion loss (S 42)	Salt spray test (delamination)	Salt spray test (corrosion)	Humidity test (delamination)		Humidity test (cupping test after immersion)	Water immersion at 90 °C (color change)	Water immersion at 90 °C (remaining gloss at 60°)
Reference: standard (with barite PVC 4.2)																			
Without filler (PVC 0.4)		+	0-	-	0	+	0	+	+	0	+	+	+	0	++	0	0	0	+
Sillitin Z 86 (PVC 4.2)		+	-	-	0	0	0	+	+	0	+	+	++	0	+	0	0	0	+
Barite (PVC 7.6)		0	0	0	0	0	0	-	-	-	-	-	0	0	0	0	0	0	0
Sillitin Z 86 (PVC 7.6)		+	0	+	0	0	0	0	0	0	0	0	+	+	0	+	0	+	0
Aktisil AM (PVC 7.6)		+	0	+	0	0	0	0	+	0	+	0	++	0	+	0	+	+	0
Aktisil MM (PVC 7.6)		+	0	+	0	0	0	+	+	0	0	0	++	0	++	0	0	+	0
NCC (PVC 7.6)		-	-	0	0	0	0	-	0	0	0	++	0	+	0	-	-	-	+
Wollastonite (PVC 7.6)		0	0-	+	-	0	0	-	0	0	-	0	0	++	-	-	-	-	-
Clay (PVC 7.6)		+	0	-	0	0	0	-	0-	0	0	0	++	-	++	-	0	+	0
Mica (PVC 7.6)		0	0-	0	-	0	0	-	-	-	-	0	0	++	-	+	-	0	-
Talc (PVC 7.6)		-	+	++	-	-	0	-	-	-	0	-	++	-	0	-	0	+	0

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Fig. 24

The use of Neuburg Siliceous Earth offers the following benefits:

- improved fluidizability
- optical properties maintained at high level
- mechanical properties (flexibility, impact resistance) markedly improved
- improved abrasion resistance
- delamination markedly improved, low corrosion at scribe
- hot water resistance increased

These positive effects are also evident with the high PVC formulations.

Sillitin Z 86 recommends itself because of a favorable price/performance ratio.

Aktisil AM gives the best results in the resistance tests.

Aktisil MM imparts low delamination and better mechanical properties.

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.