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Unternehmensgruppe Hoffmann

Reduced titanium dioxide content:

Calcined Neuburg Siliceous Earth

in powder coatings

(polyester, TGIC-based, white)

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1 Introduction

Benefits of Neuburg Siliceous Earth have been shown earlier in a study on hybrid-based powder coatings with focus on replacing parts of the titanium dioxide content and the commonly used filler barium sulfate. The optical and mechanical properties could be maintained or even improved.

In previous projects, as for example in a Coil Coating Top Coat, the potential of Calcined Neuburg Siliceous Earth as a partial replacement of titanium dioxide had already been successfully evaluated.

More and more consumers of titanium dioxide are looking for alternatives or partial replacement of this pigment because its price has risen globally.

As a result, the question came up if Calcined Neuburg Siliceous Earth would be able to partially replace titanium dioxide in a TGIC-based[1] powder coating while maintaining the optical properties, above all hiding power as well as corrosion resistance.

The major filler in the formulation:

- Natural barium sulfate (barite), for cost-effective formulations meeting basic requirements

or

- Precipitated barium sulfate (ppt), a special grade with higher quality and better optical properties to meet higher expectations

The objective of the study was to maintain or improve performance characteristics while reducing costs by replacing titanium dioxide with Calcined Neuburg Siliceous Earth.

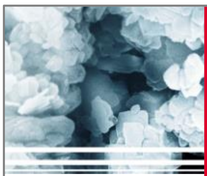
[1] Triglycidyl isocyanurate

2 Experimental

2.1 Base formulation

The base formulation given in *Fig. 1* represented the starting point of the study. Crylcoat 2441-3 is a carboxylated polyester suitable for curing with TGIC. Modaflow P 6000 and benzoin were used as leveling agents. The pigment was rutile (TiO_2 content: >= 92 %), with a surface treatment (aluminum and silicon compounds).

The base formulation's pigment volume concentration (PVC) was 14.4 %.



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Base Formulation

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		Base formulation
Crylcoat 2441-3	Carboxylated polyester , AV: 30-35 mg KOH/g	59
TGIC	Triglycidyl isocyanurate, hardener	4.5
Titanium dioxide	Pigment	20
Barium sulfate	Filler	16.5
Modaflow P 6000	Leveling agent	1
Benzoin	Leveling agent	0.2
Total		101.2
PVC [%]	14.4	

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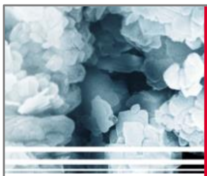
Fig. 1

2.2 Fillers used and their typical properties

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.

The calcination of the Neuburg Siliceous Earth is used to expel the crystalline water in the kaolinite portion and new mineral phases are fomed practically amorphous. The silica portion remains inert under the temperature chosen. Through an integrated air classifier process grain sizes > 15 µm are being removed.

Fig. 2 shows the typical properties of natural barium sulfate (Barite) and precipitated barium sulfate (ppt), both used in this investigation, and Silfit Z 91. Compared with the barium sulfate used in the base formulation, Silfit Z 91 is distinguished by a markedly lower density, higher oil absorption and a larger specific surface area. The natural barium sulfate (barite) displayed a greater and the precipitated type a somewhat smaller medium particle size (d_{50}) as well as lower top cut (d_{97}) than Silfit Z 91.



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		Barium sulfate		Calcined Neuburg Siliceous Earth
		natural	ppt	Silfit Z 91
Morphology		corpuscular		corpuscular / lamellar aggregated
Density	[g/cm³]	4.4	4.4	2.6
Particle size d_{50}	[µm]	2.9	1.2	2.0
Particle size d_{97}	[µm]	14	5	10
Oil absorption	[g/100g]	14	19	60
Specific surface area BET	[m²/g]	0.8	not measured	7.5

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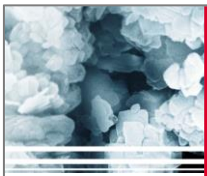
Fig. 2

The color values were determined with a spectral photometer (geometry d/8°, light D 65). The precipitated barium sulfate, with an L* value of 97, showed the highest brightness, followed by barite and Silfit Z 91, both with L* = 95. The a* values of these fillers were in the range of -0.2 to -0.4 while the b* values, indicating yellowish tint, revealed a somewhat higher color neutrality than Silfit Z 91 (*Fig. 3*).



Filler Characteristics

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Color	Barium sulfate		Calcined Neuburg Siliceous Earth
	natural	ppt	Silfit Z 91
L*	95	97	95
a*	- 0.3	- 0.4	- 0.2
b*	0.2	0.4	0.9

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Fig. 3

2.3 Preparation of batches

The premix was mixed for 2 minutes at 2000 rpm in a Mixaco unit and subsequently homogenized in an extruder (OMC Saronno EBPV 19.2, shaft speed 150 rpm, heating zones 110°C / 120°C). The batches were ground in a mill and finally sieved. The powder was applied with a Wagner powder pistol (EPM sprint pistol PEM CG 4 model 360; 90 kV) onto Q-Panel sheets (chromated aluminum AL 48). The coatings were baked at an oven temperature of 200°C for 10 minutes peak metal temperature (PMT). The dry film thickness ran between 80 and 90 µm.

2.4 Test methods

Color values

The color values CIE L*, a*, and b* were determined with a spectral photometer at a measuring geometry d/8° with light D 65.

Hiding power / Opacity

Opacity was determined on black / white panels (checkered pattern) from the Q-Panel company. The hiding power was obtained by measuring the standard color index Y over the black and white substrate. The ratio of Y_{black} to Y_{white}, multiplied by 100, yielded the hiding power in percent. With a hiding power of 98 % or greater, a coating is judged as covering.

Gloss

Gloss was determined with the Micro-Tri-Gloss unit of the BYK company. The measuring angle of 20° represents the range of high gloss, 60° the medium gloss range.

Haze

High quality surfaces are expected to offer a clear, brilliant aspect. Microstructures, which can be introduced by incomplete dispersion or big particles, result in a slight opacity or haze. This effect is named haze and was determined with the Micro-Haze Plus unit of the BYK company.

Leveling

For this test, the surface was evaluated optically: how well the overhead lamp was mirrored on the coating, how well the edges were displayed and how far they were irregular or spread out. The better the leveling properties, the smoother and more uniform the appearance.

Artificial Weathering (QUV-A Test)

These results are from customer feedback. The test was running for 1000 hours with cycles of 4 h UVA light (340 nm) at 50°C and 4 h condensation at 50°C and 100% relative humidity.

Acetic Acid Salt Spray Test AASS

This test according to DIN EN ISO 9227 was running for 2000 hours. Upon completion, the evaluation of blistering (DIN EN ISO 4628/2) and the evaluation of delamination as well as corrosion at the scribe (DIN EN ISO 4628/8) were performed.

Humidity Test CH

This test according to DIN EN ISO 6270-2 was running for 2000 hours. Upon completion, the evaluation of blistering (DIN EN ISO 4628/2) and the evaluation of delamination as well as corrosion at the scribe (DIN EN ISO 4628/8) were performed.

3. Results with natural barium sulfate (barite)

3.1 Formulation variations

Starting from the base formulation (control) with 16.5 parts barite and 20 parts titanium dioxide, in the following variants 20 % of the titanium dioxide were replaced at equal weight (i.e. 4 parts by weight) with Calcined Neuburg Siliceous Earth Silfit Z 91. In the first version, the amount of barium sulfate remained unchanged, while in the second version 33 % of the barite was also replaced by Silfit Z 91 by equal volume. Finally, barite was completely replaced in an analogous manner. The different formulations are shown in *Fig. 4* in parts by weight and in *Fig. 5* in percent.

All formulations had a slightly increased PVC of 15.1 %, compared with the control at 14.4 %, and this because of the replacement of 20 % titanium dioxide by Silfit Z 91 at equal weight instead of equal volume.

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Parts per weight

	Control $BaSO_4$	- 20 % TiO_2 $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 33 % $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 100 % $BaSO_4$ + Silfit Z 91
Crylcoat 2441-3	59	59	59	59
TGIC	4.5	4.5	4.5	4.5
Titanium dioxide	20	16	16	16
$BaSO_4$ natural	16.5	16.5	11	-
Silfit Z 91	-	4	7.25	13.75
Modaflow P 6000	1	1	1	1
Benzoin	0.2	0.2	0.2	0.2
Total	**101.2**	**101.2**	**98.95**	**94.45**
PVC [%]	14.4	15.1	15.1	15.1

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Fig. 4

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Parts per cent (%)

	Control $BaSO_4$	- 20 % TiO_2 $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 33 % $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 100 % $BaSO_4$ + Silfit Z 91
Crylcoat 2441-3	58.13	58.13	59.45	62.27
TGIC	4.43	4.43	4.53	4.75
Titanium dioxide	19.70	15.76	16.12	16.89
$BaSO_4$ natural	16.26	16.26	11.08	-
Silfit Z 91	-	3.94	7.30	14.51
Modaflow P 6000	0.99	0.99	1.01	1.06
Benzoin	0.49	0.49	0.50	0.53
Total	**100**	**100**	**100**	**100**
PVC [%]	14.4	15.1	15.1	15.1

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Fig. 5

3.2 Color values

The a* value, indicating the red/green portions, came off at a level of about - 1.0 for all formulations.
The brightness L* was highest with the control at 95.9. In the variants, the L* value remained comparable at a high level of <= 95 and this, despite the titanium dioxide reduction of 20 % (*Fig. 6*).
The color index b*, which stands for the yellow/blue portions, was in the slightly yellowish region between 0.6 and 1.0 (*Fig. 7*).
So considering the measured results, the L*a*b*color values of all formulations are comparable and situated on the same high level.

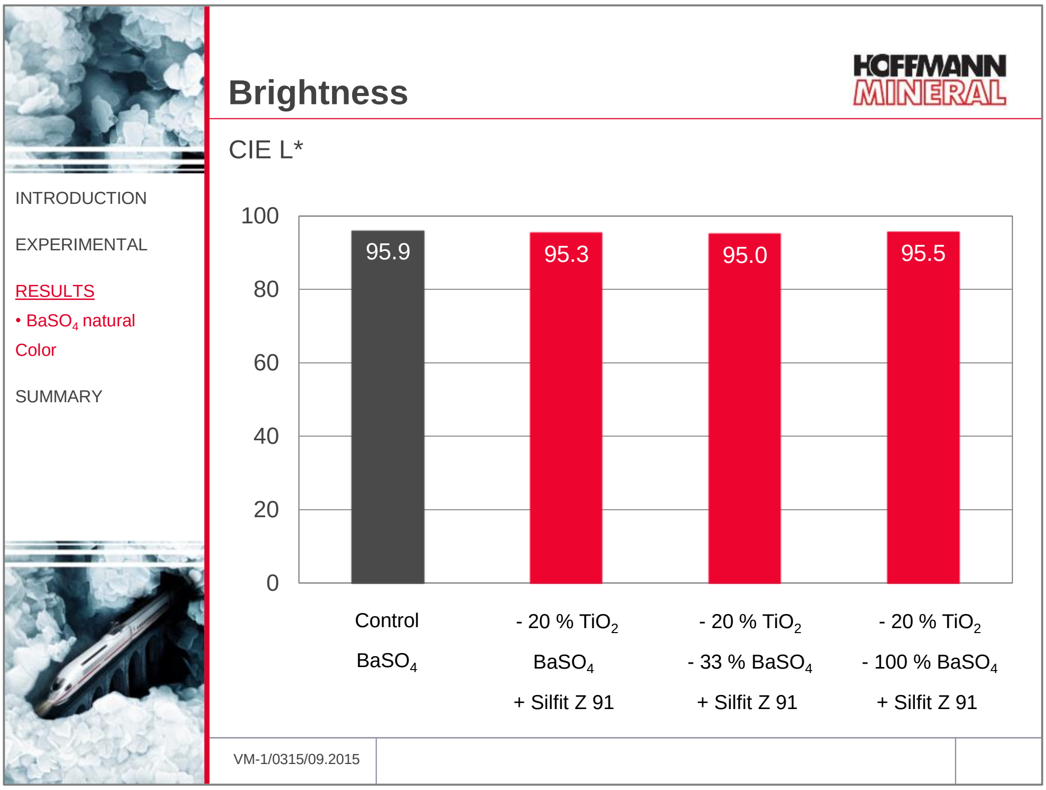


Fig. 6

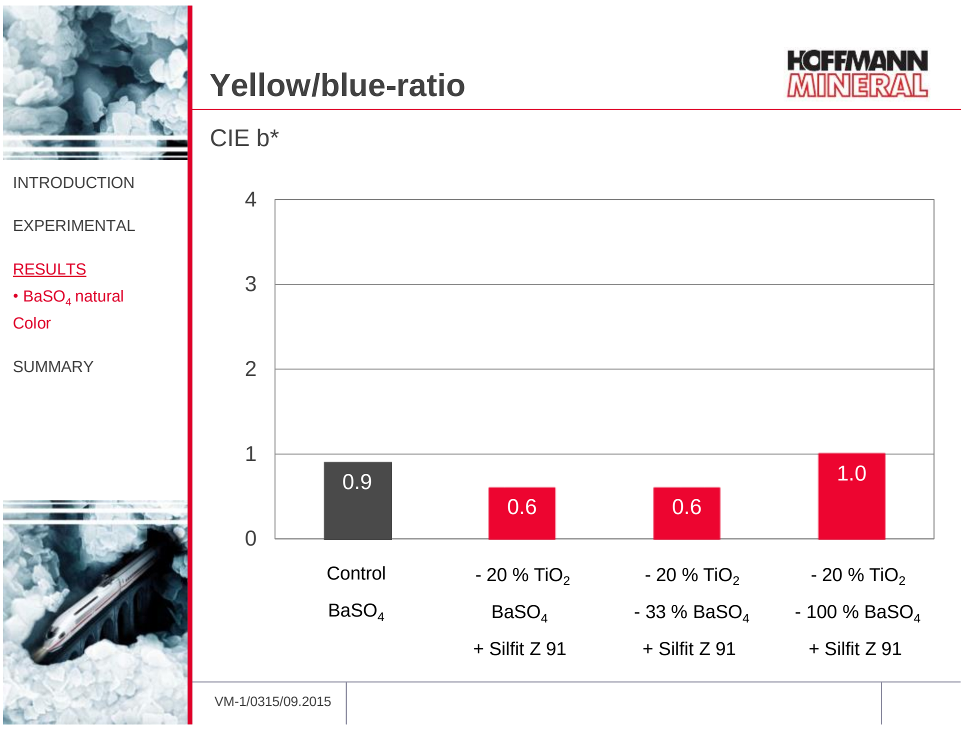


Fig. 7

3.3 Hiding power / Opacity

Fig. 8 shows the opacity results at a film thickness of about 80 µm. Although the titanium dioxide content had been reduced by 20 %, the required hiding power of 98 % was exceeded with every formulation. In view of deviations in film thickness and brightness measurements, the hiding power of all formulations can be judged as equal.

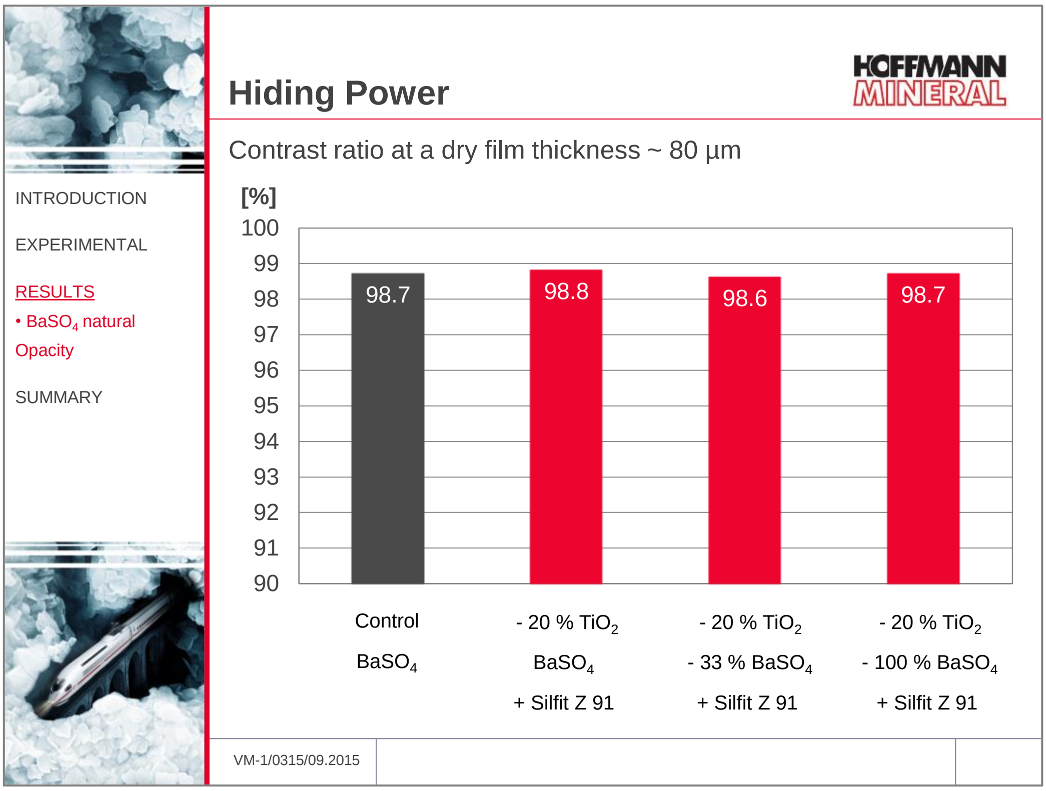


Fig. 8

3.4 Gloss and Haze

The control containing the full loading of titanium dioxide and barite had a gloss of 63 units with the measuring angle of 20°. This was changed to 53 units through the substitution of 20 % titanium dioxide with Silfit Z 91. Replacing the barite completely with Silfit Z 91 reversed this trend; total substitution elevated the gloss even beyond the level reached by the control: 72 units (*Fig. 9*).

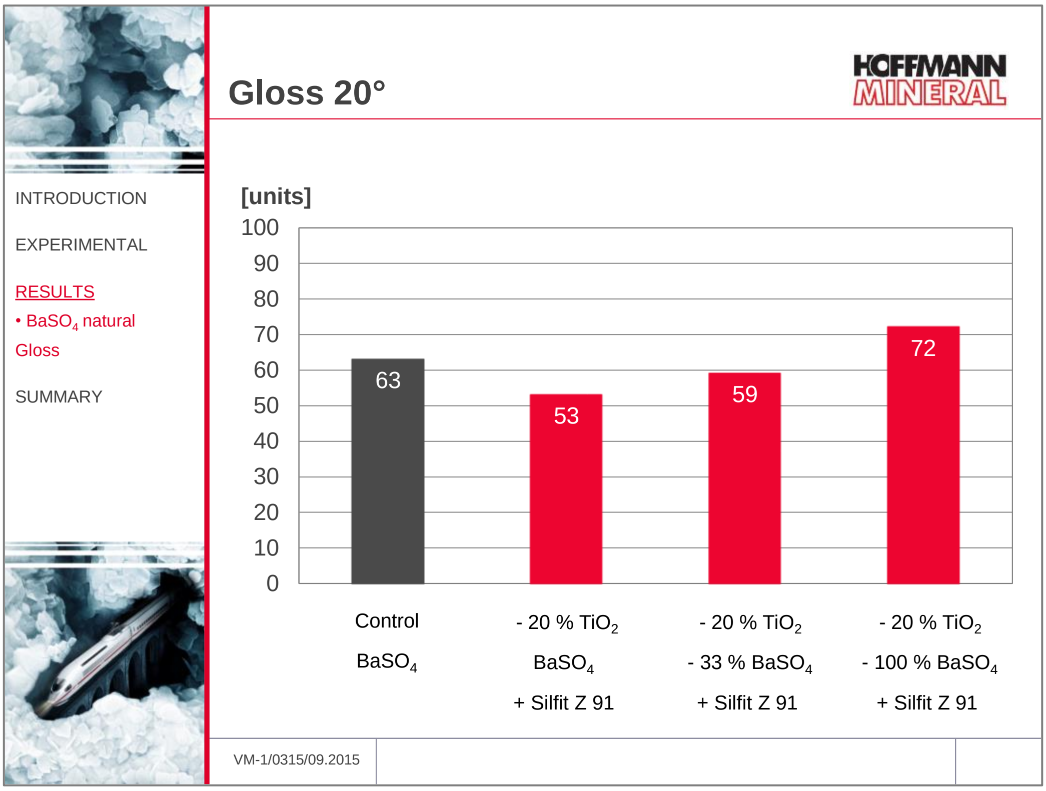


Fig. 9

Fig. 10 illustrates the gloss at a measuring angle of 60°. The gloss increasing effect of Silfit Z 91 here does not come off as high as at 20°, but is at least as high as the gloss of the control.

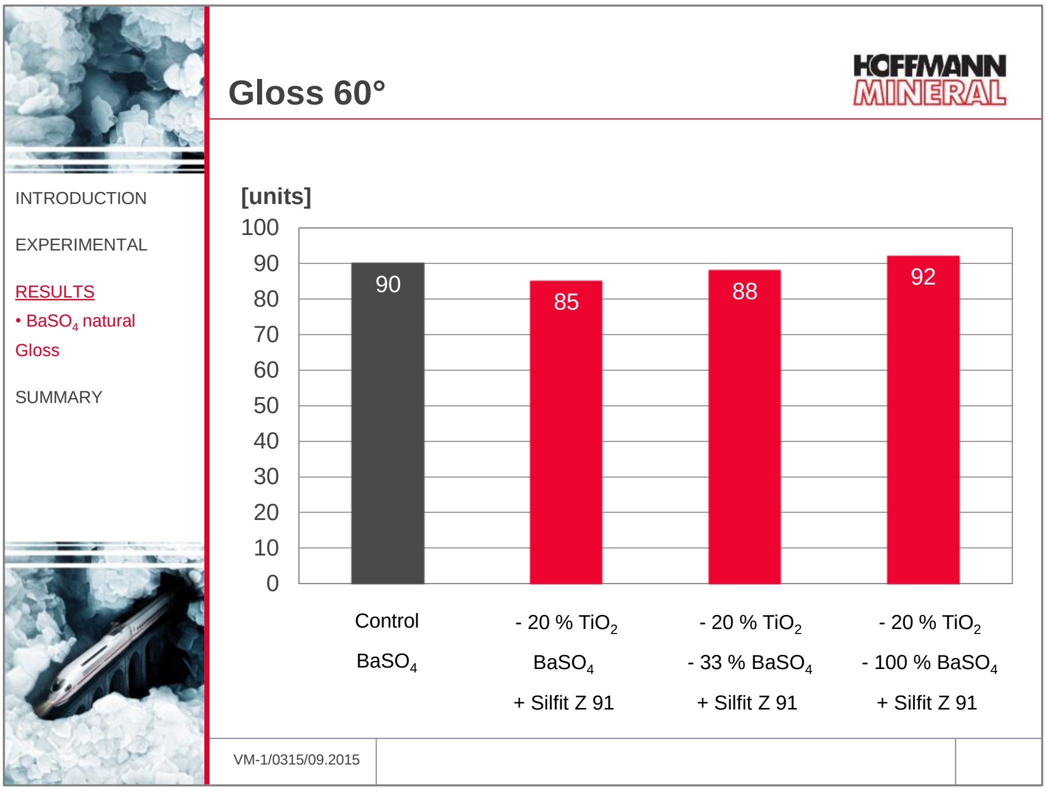


Fig. 10

The control had a haze of 292 units, similar to the first and second variants with 326 to 280 units. By completely replacing the barite with the Calcined Neuburg Siliceous Earth the haze could be reduced down to 204 units. Silfit Z 91 allows obtaining a markedly better optical impression compared to what is achievable with barite (*Fig.11*).

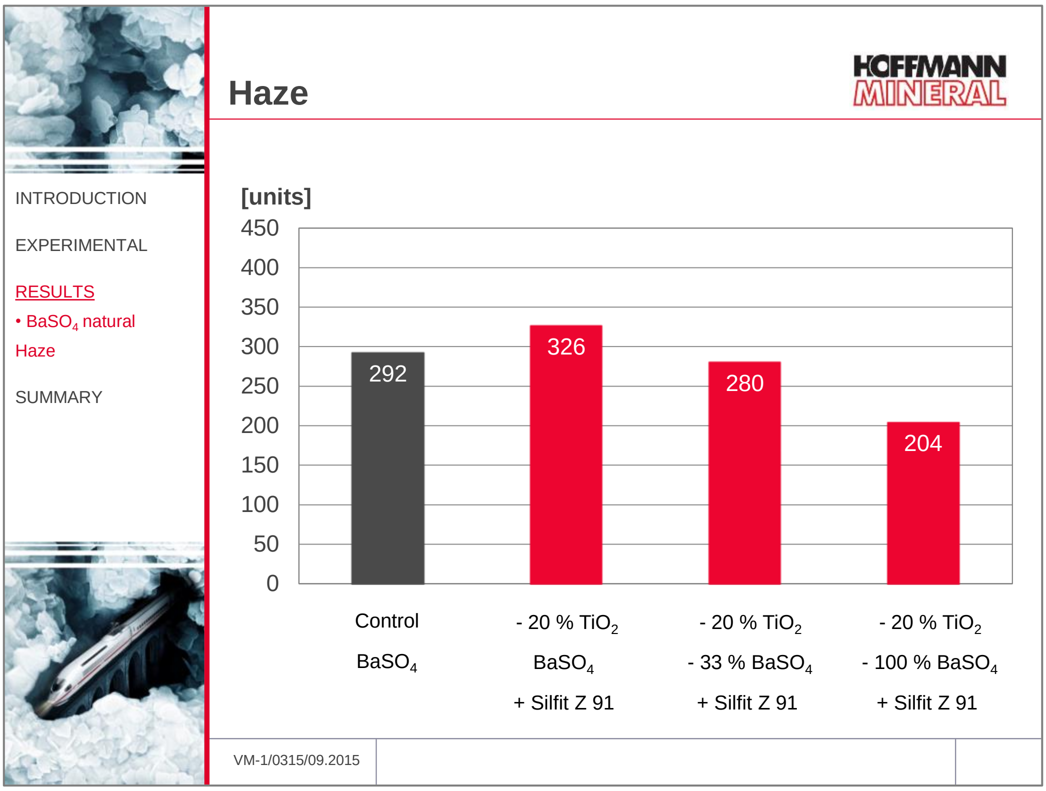


Fig. 11

3.5 Leveling

When 20 % of the titanium dioxide was replaced with Silfit Z 91, the coating's surface structure did not change at all. The more barite was substituted by Silfit Z 91, the better was the appearance, which means less structure became visible. The surface appeared smoother and a better leveling was evident (*Fig.12*).

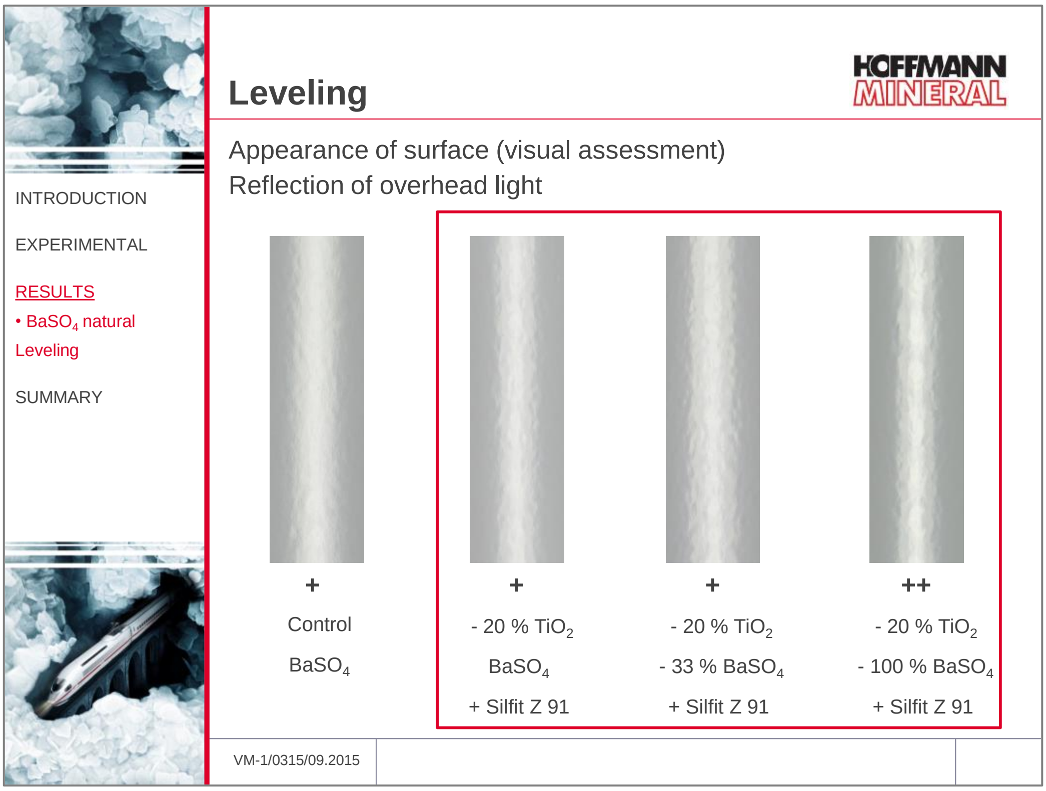
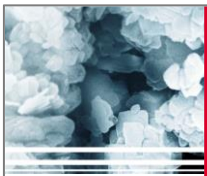


Fig. 12

3.6 Artificial weathering (QUV-A Test)

The specimen were exposed to artificial weathering 1000 hours with cycles of 4 hours UVA light (340 nm) at 50 °C and 4 hours condensation at 100 % relative humidity and 50 °C. The composition of the base recipe was a customer formulation, different from our tested formulations. Beginning with the control formulation containing titanium dioxide, steps with 10 %, 30 % and 50 % titanium dioxide substitution with Silfit Z 91 were evaluated. As depicted in the chart there is, even with the highest substitution level, nearly no change in the Delta E after 1000 hours of exposure. The remaining gloss is almost as high as it was at the beginning. None of the formulations exhibited signs of chalking or white spots. Thus, concluding the results of the artificial weathering a reduction up to the half of titanium dioxide and therefore the introduction of Silfit Z 91 is easily possible *(Fig. 13).*



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Artificial Weathering

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Artificial Weathering 1000h
Customer Feedback

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Cycles of 4 h UVA light (340 nm) at 50°C and
4 h condensation at 50°C and 100% relative humidity

Customer Formulation containing TiO_2 and $BaSO_4$	Control with TiO_2	- 10 % TiO_2 + Silfit Z 91	- 30 % TiO_2 + Silfit Z 91	- 50 % TiO_2 + Silfit Z 91
Delta E	1.2	1.1	1.5	1.3
Remaining Gloss [%]	92	93	93	96

None of the formulations exhibited after exposure signs of chalking or white spots.

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Fig. 13

3.7 Corrosion resistance (Acetic Salt Spray Test and Humidity Test)

Acetic Acid Salt Spray Test DIN EN ISO 9227 / AASS (2000 hours):

1. After exposure the panels were assessed regarding blistering (DIN EN ISO 4628/2):

   In *Fig. 14* the blistering on the surface of the panels is illustrated. The control formulation showed a smattering of defects, approximately 5 % of the upper side surface was covered with blisters 3–3 (S2). All of the variations containing Silfit Z 91 showed no blistering.

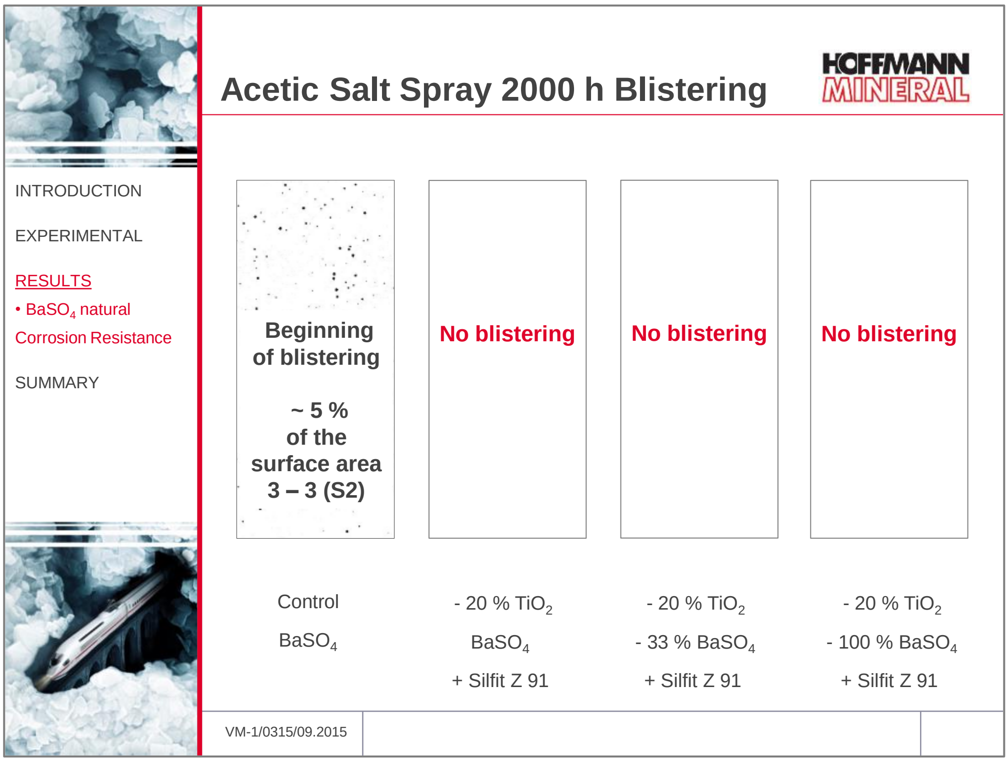

Fig. 14

2. The delamination at the scribe (scratching tool according to Sikkens, right-angled cutting edge, longitudinal section) was also evaluated according to DIN EN ISO 4628/8:

   Fig. 15 illustrates the delamination at the scribe. The control formulation had 0.7 mm delamination at the scribe. The variations containing Silfit Z 91 showed nearly no delamination at the scribe.

   Corrosion at the scribe occurred only in terms of single blisters (point corrosion), however the same with all formulations, no further differentiation possible.

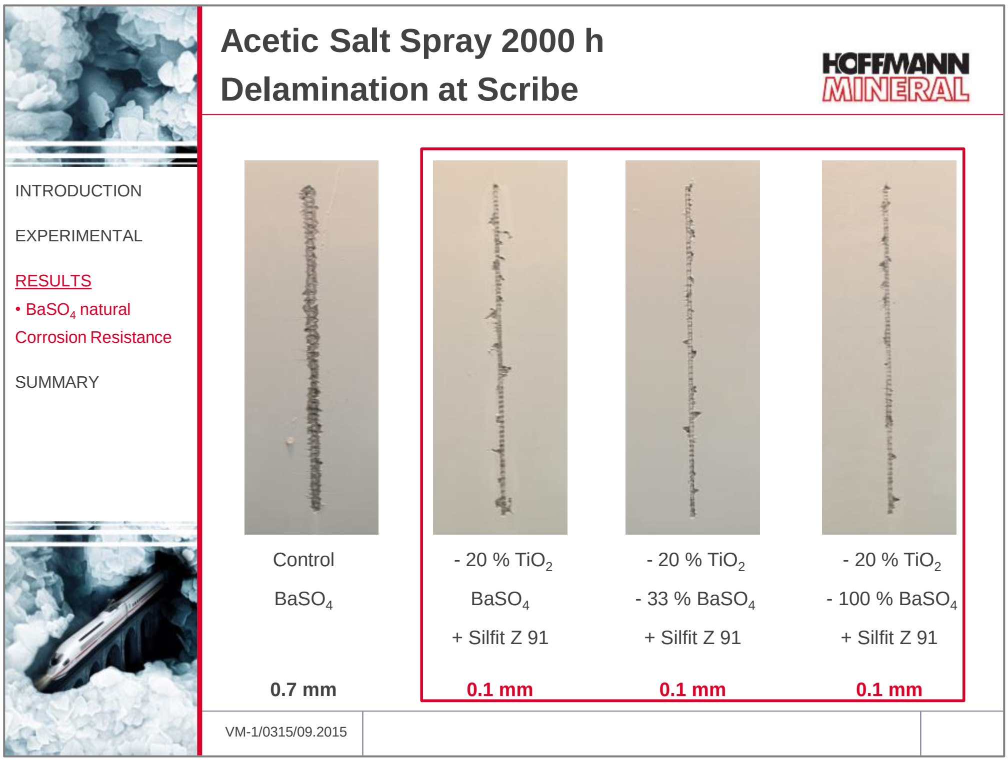

Fig. 15

Humidity Test DIN EN ISO 6270-2 / CH (2000 hours):

1. After exposure the panels were assessed to blistering (DIN EN ISO 4628/2):

   In *Fig. 16* the blistering on the surface of the panels is illustrated. The control formulation showed multiple defects, approximately 30 % of the surface was covered with blisters 2-2 (S2). All of the variations containing Silfit Z 91 showed no blistering.

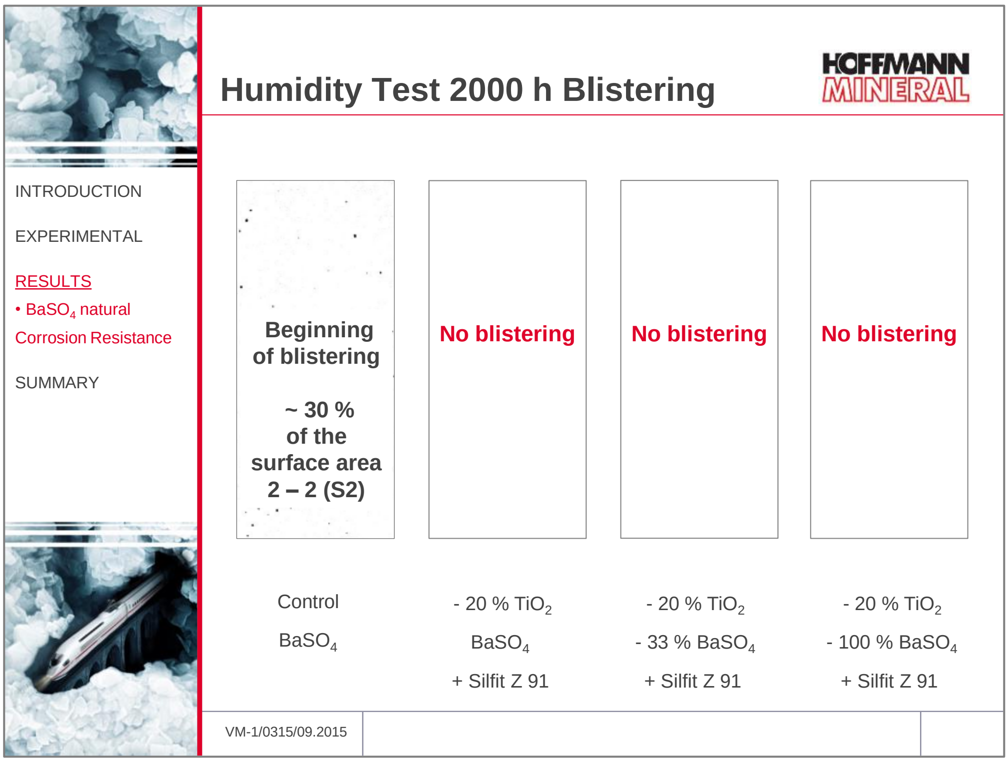

Fig. 16

2. The delamination at the scribe (scratching tool according to Sikkens, right-angled cutting edge, longitudinal section) was also evaluated (DIN EN ISO 4628/8):

   In *Fig. 17* the delamination at the scribe is illustrated. The control formulation had 7 mm delamination distance at the scribe. The variations containing Silfit Z 91 showed no signs of delamination at all. There was no corrosion at the scribe.

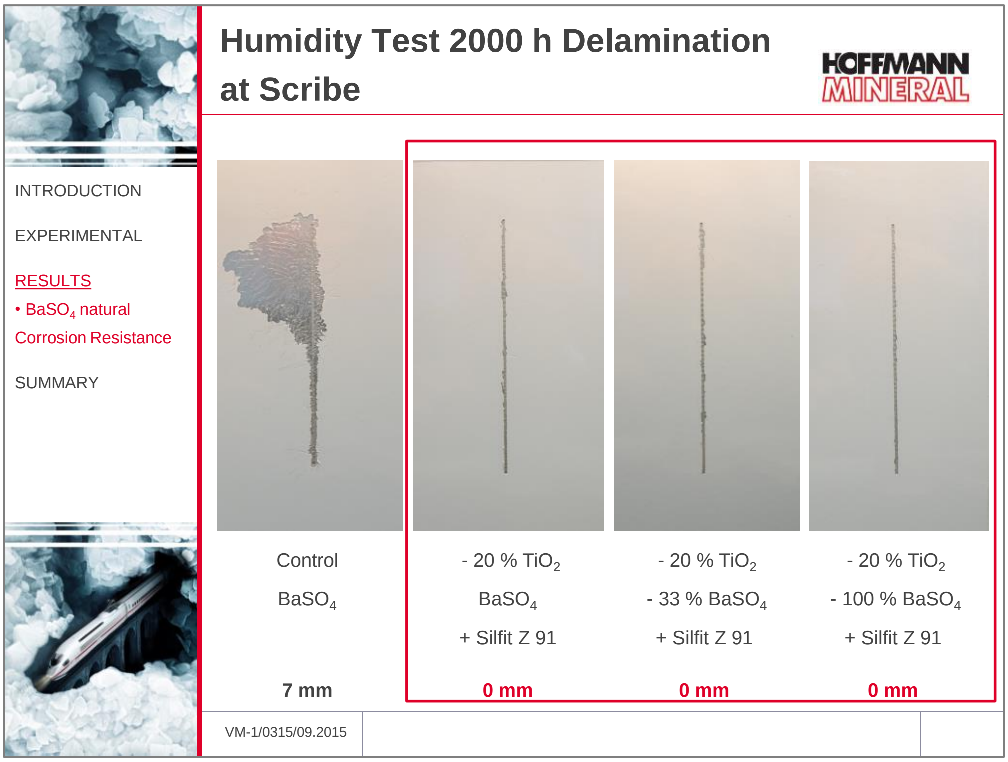

Fig. 17

3.8 Density and Spreading rate

Fig. 18 shows the densities of the formulations. The control exhibited the highest level of 1.61 g/cm³, caused by the density of the straight barite at 4.4 and titanium dioxide at 3.9. The replacement of 20 % titanium dioxide at equal weight, i.e. 4 pbw, by Silfit Z 91 with a density of 2.6 hardly affected the total density at all. However, when replacing 33 resp. 100 % of the barite at equal volume with Silfit Z 91, the density decreased down to 1.49 g/cm³. As shown in the following figure, this change has a positive effect on the spreading rate.

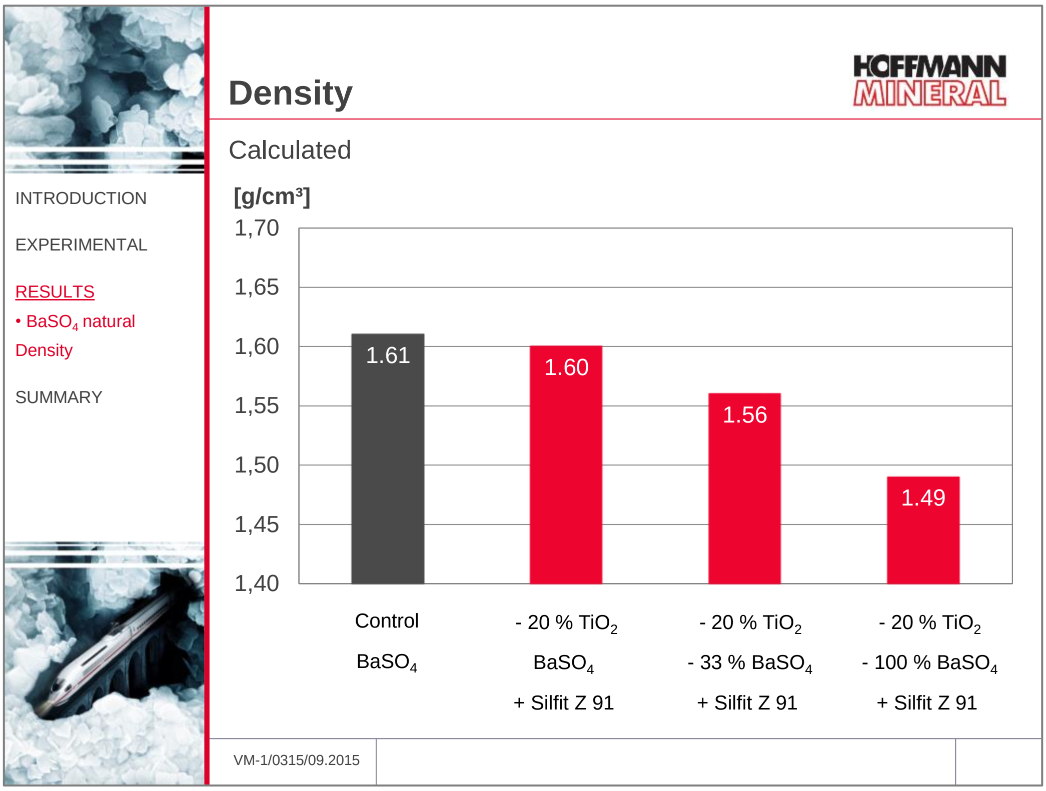

Fig. 18

Fig. 19 illustrates the spreading rate relative to the control as index. It shows how much surface can be coated by a mass unit of powder coating for a similar dry film thickness.

As powder coatings are sold by weight, the spreading rate is considerably improved by using Silfit Z 91!

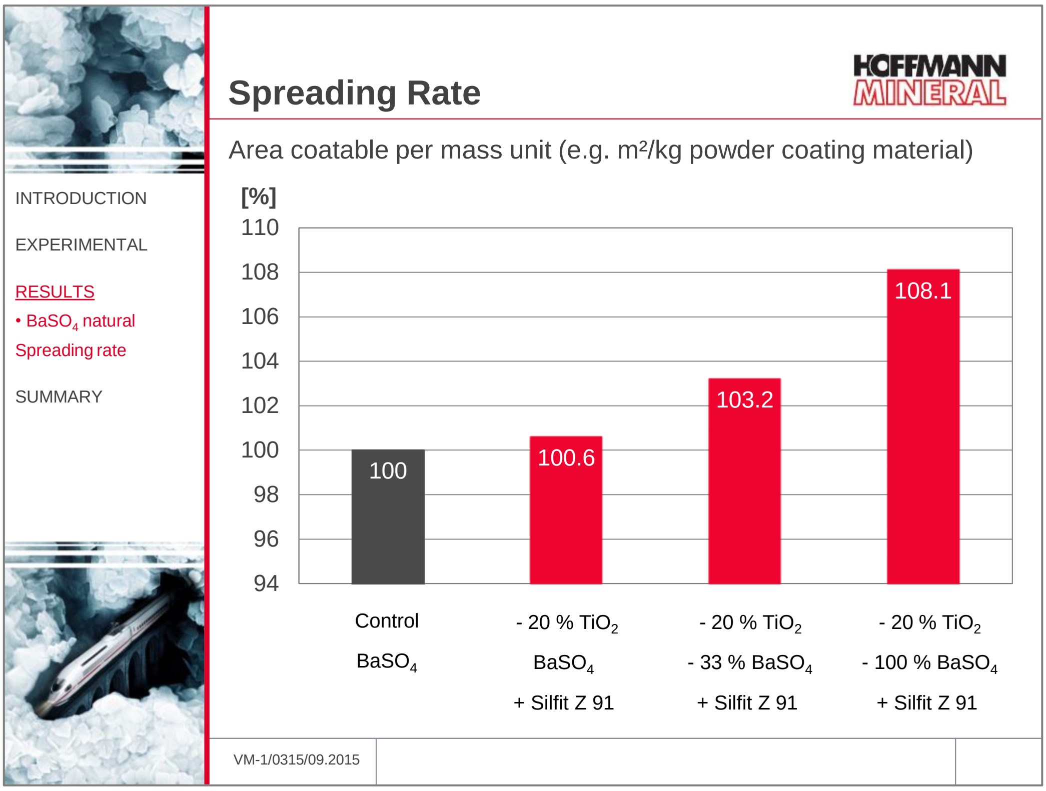

Fig. 19

3.9 Cost index

Fig. 20 gives the weight-related costs based on German prices during the year 2014. The price for titanium dioxide was taken as € 2.40 per kg. The replacement of 20 % titanium dioxide with Silfit Z 91 allowed saving approx. 3 % of the costs. The further partial replacement of barite reduced the cost advantage to 0.5 %. The complete replacement of the barite with Silfit Z 91 caused a cost increase of 4.4 %, which however is more than compensated by the higher spreading rate of 8.1 %.

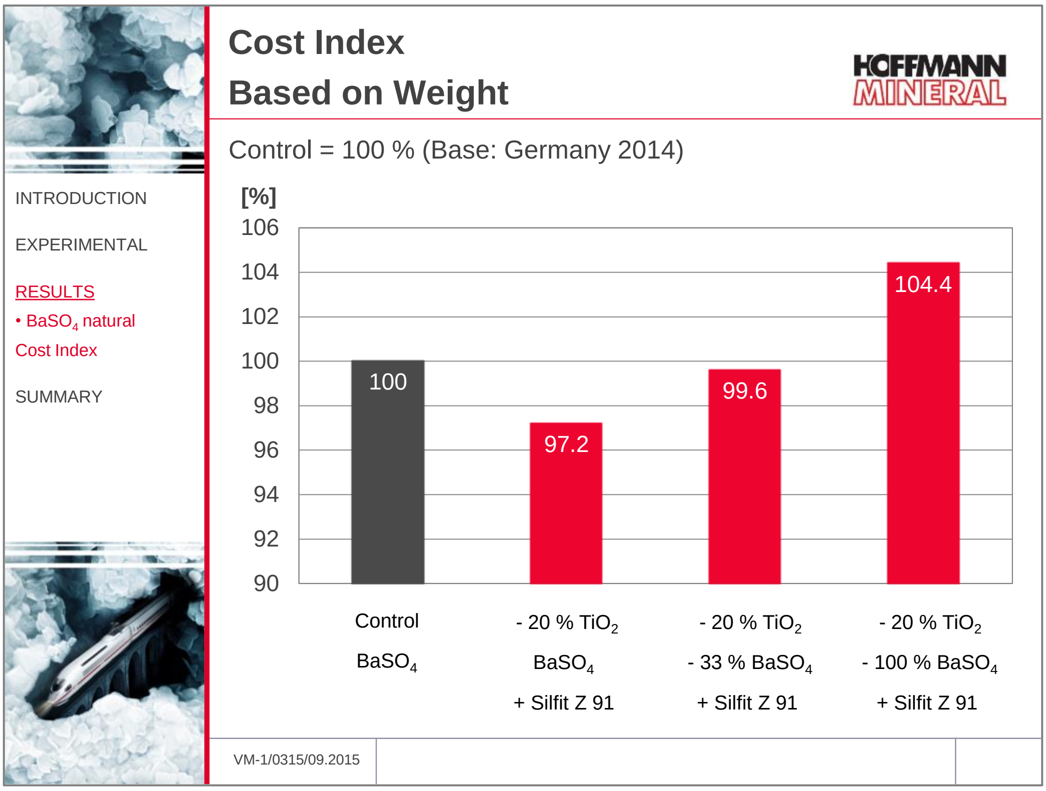

Fig. 20

If the cost index is calculated based on volume, all formulations with Silfit Z 91 gave rise to marked cost savings of about 4 % (*Fig. 21*).

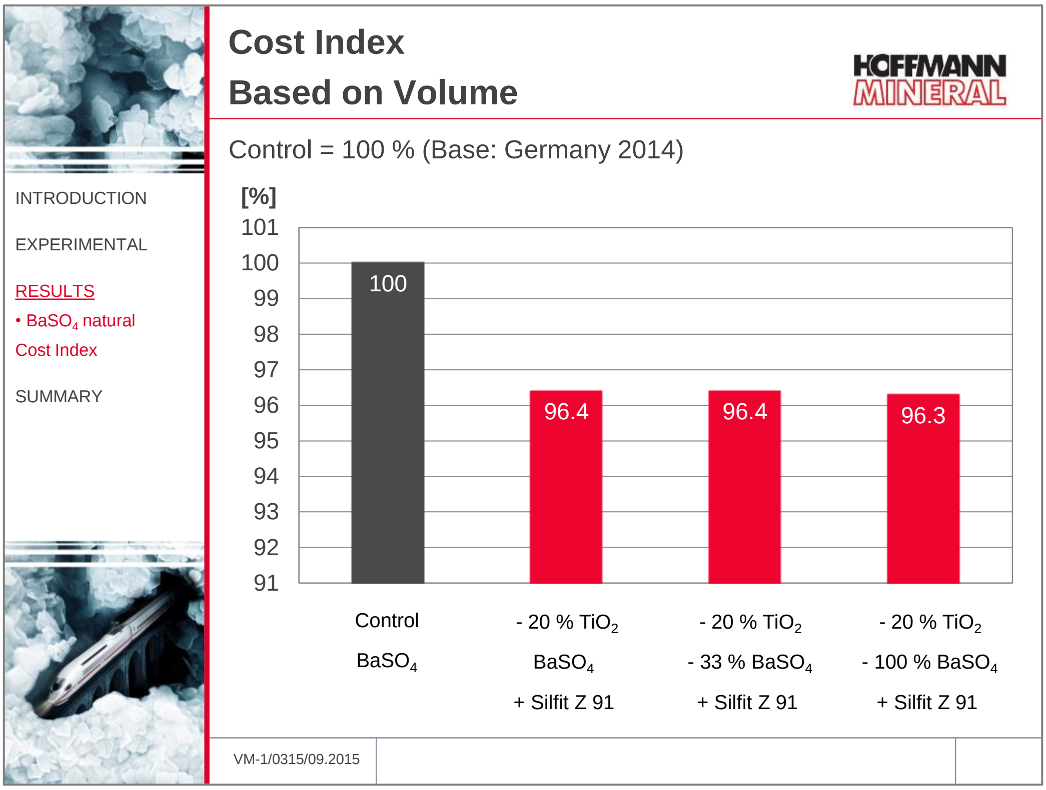

Fig. 21

3.10 Summary of the results with natural barium sulfate (barite)

The replacement of 20 % titanium dioxide with Silfit Z 91 at equal weight led to the following effects:

- similar optical properties
- excellent weatherability (even up to 50 % titanium dioxide substitution)
- + improved corrosion resistance
- + potential for cost savings

The further replacement of barite with Silfit Z 91 at equal volume achieved in addition:

- + higher gloss
- + lower haze
- + better leveling
- + improved spreading rate through lower density of the coating
- + potential for cost savings

4. Results with precipitated barium sulfate

4.1 Formulation variations

Starting from the base formulation (control) with 16.5 parts precipitated barium sulfate and 20 parts titanium dioxide, in the following variants 20 % of the titanium dioxide were replaced at equal weight (i.e. 4 parts by weight) with Calcined Neuburg Siliceous Earth Silfit Z 91. In the first version, the amount of barium sulfate remained unchanged, while in the second version 33 % of the barite was also replaced by Silfit Z 91 by equal volume. Finally, barite was completely replaced in an analogous manner. The different formulations are shown in *Fig. 22* in parts by weight and in *Fig. 23* in percent.

All formulations had slightly increased PVC of 15.1 %, compared with the control at 14.4 %, and this because of the replacement of 20 % titanium dioxide by Silfit Z 91 at equal weight instead of equal volume.

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Parts per weight

	Control $BaSO_4$	- 20 % TiO_2 $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 33 % $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 100 % $BaSO_4$ + Silfit Z 91
Crylcoat 2441-3	59	59	59	59
TGIC	4.5	4.5	4.5	4.5
Titanium dioxide	20	16	16	16
$BaSO_4$ ppt	16.5	16.5	11	-
Silfit Z 91	-	4	7.25	13.75
Modaflow P 6000	1	1	1	1
Benzoin	0.2	0.2	0.2	0.2
Total	**101.2**	**101.2**	**98.95**	**94.45**
PVC [%]	14.4	15.1	15.1	15.1

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Fig. 22

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Formulations HOFFMANN MINERAL

Parts per cent (%)

	Control $BaSO_4$	- 20 % TiO_2 $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 33 % $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 100 % $BaSO_4$ + Silfit Z 91
Crylcoat 2441-3	58.13	58.13	59.45	62.27
TGIC	4.43	4.43	4.53	4.75
Titanium dioxide	19.70	15.76	16.12	16.89
$BaSO_4$ ppt	16.26	16.26	11.08	-
Silfit Z 91	-	3.94	7.30	14.51
Modaflow P 6000	0.99	0.99	1.01	1.06
Benzoin	0.49	0.49	0.50	0.53
Total	**100**	**100**	**100**	**100**
PVC [%]	14.4	15.1	15.1	15.1

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Fig. 23

4.2 Color values

The a* value, representing the red/green portions, for all formulations came out at an equal level of approx. -1.1.
The brightness L* was highest with the control at 96.3. For the variants, the L* value remained at a high level between 95.5 and 95.9, and this despite 20 % less titanium dioxide (*Fig. 24*).
The color index b* which indicates the yellow/blue portions, was in the slightly yellowish region between 0.7 and 1.0 (*Fig. 25*).
So considering the results of the measured color values, the L*a*b* of all formulations stays at the same level.

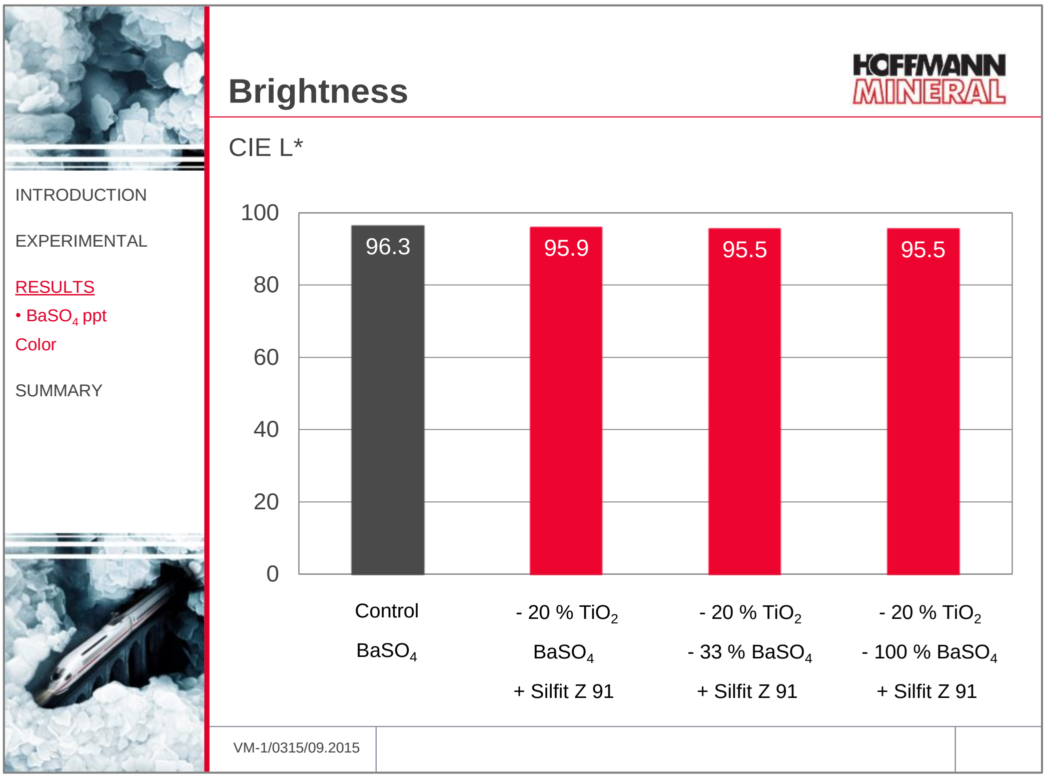


Fig. 24

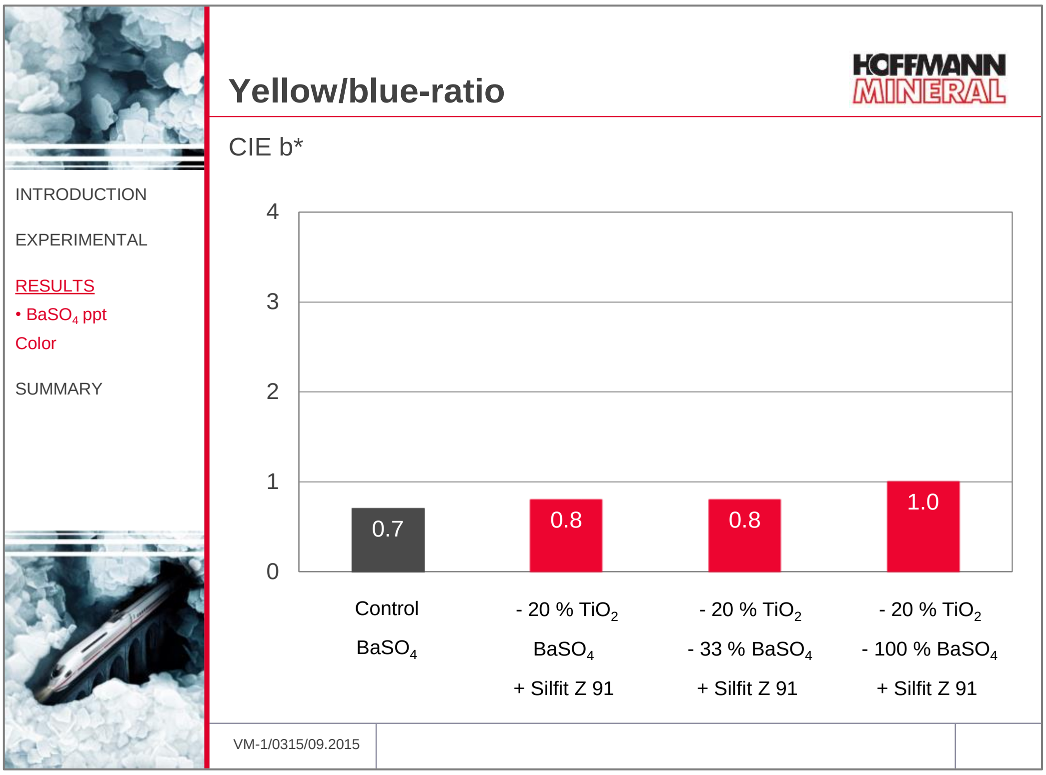


Fig. 25

4.3 Hiding power / Opacity

Fig. 26 shows the opacity results at a film thickness of approx. 80 µm. Despite the lower titanium dioxide content by 20 %, the required hiding power of 98 % or greater has been maintained in the formulations filled with Silfit Z 91. In view of deviations in film thickness and brightness measurements, the hiding power of all formulations can be judged as equal.

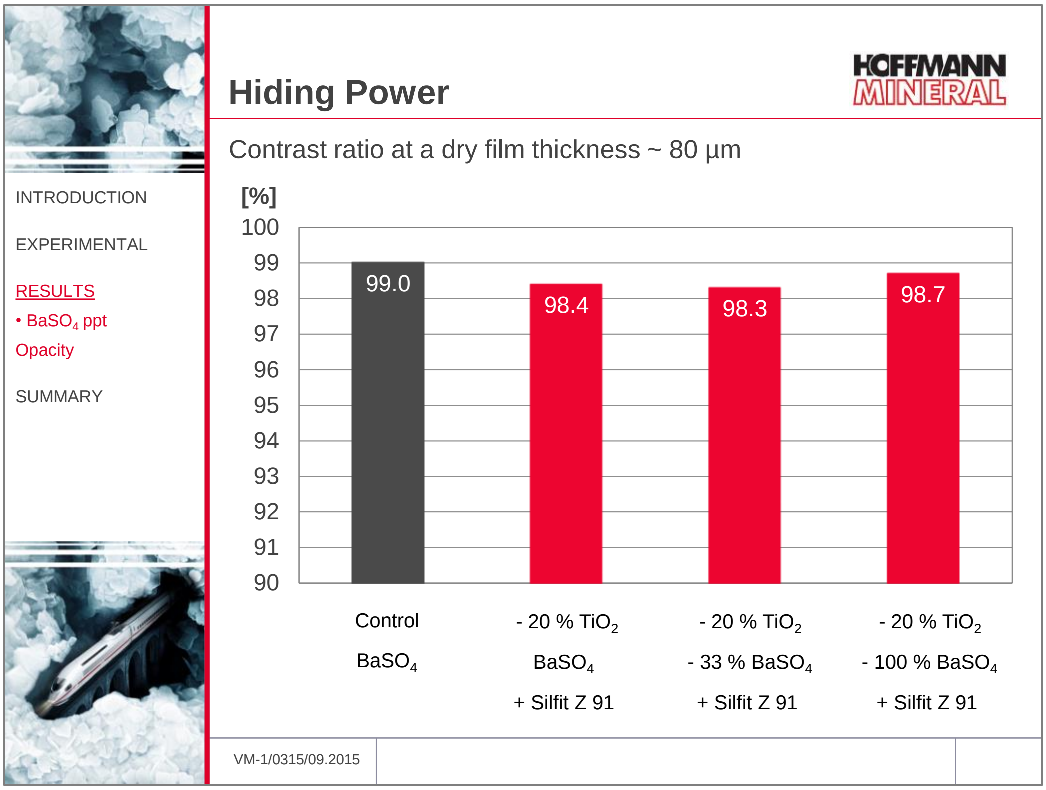


Fig. 26

4.4 Gloss and Haze

With the control loaded with the full portion of titanium dioxide and the precipitated barium sulfate, at a measuring angle of 20° a gloss of 85 units was obtained. The replacement of 20 % titanium dioxide with Silfit Z 91 gave rise to a marginally degraded gloss of 76 units. Even with the additional substitution of 33 % of the precipitated barium sulfate, the gloss of 74 units can be achieved. Replacing the total amount of the precipitated barium sulfate by Silfit Z 91 still leaves to a 20° gloss of 72 units (*Fig. 27*).

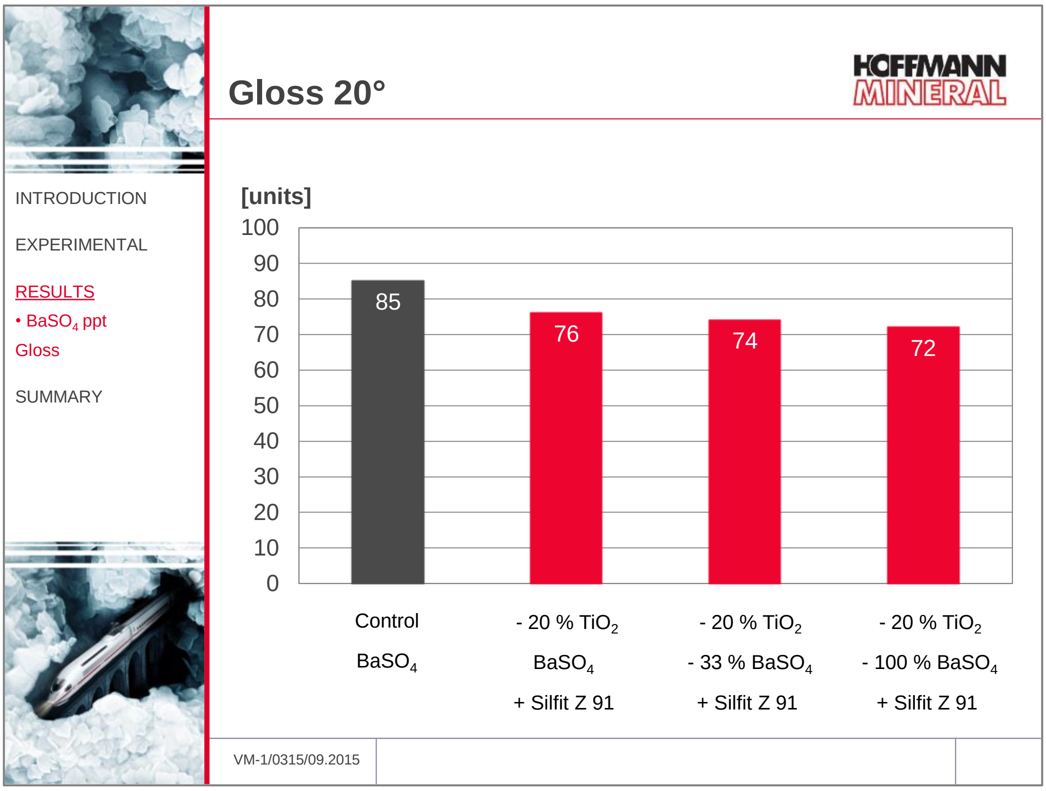


Fig. 27

Fig. 28 shows the gloss at a measuring angle of 60°. Here no further differentiation could be observed, as all formulations came out in the range of 92 to 95 units.

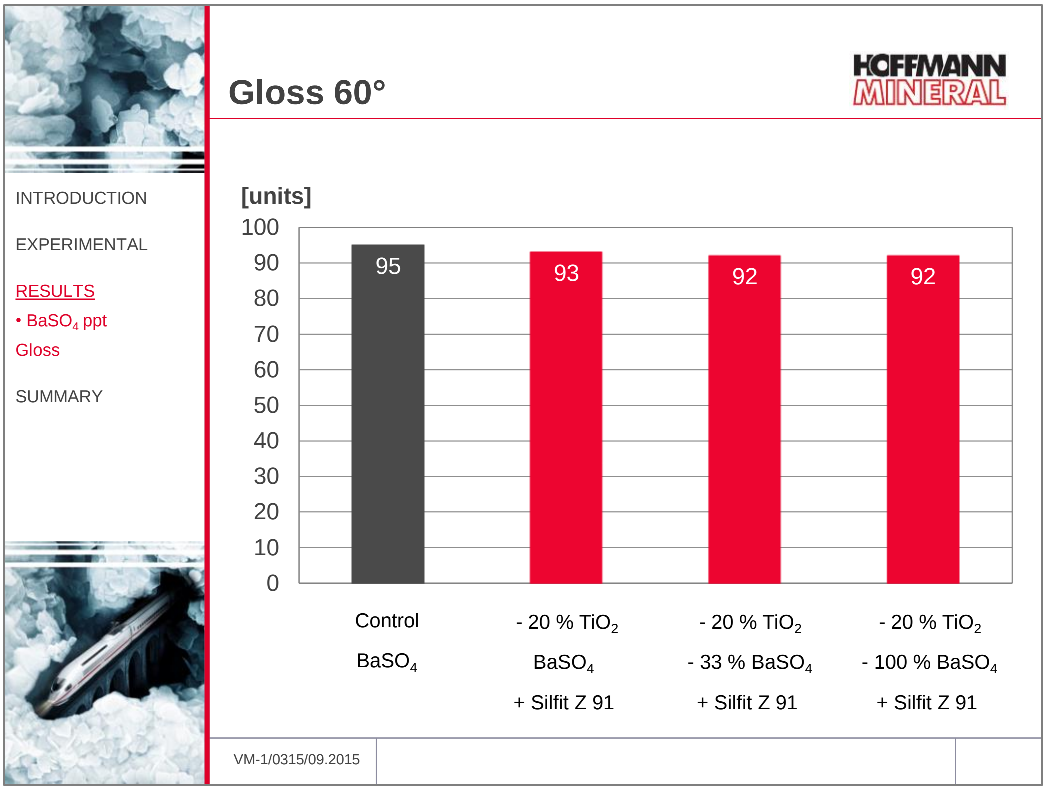


Fig. 28

The control offered a haze of 103 units, followed by the second variant where only titanium dioxide was replaced by Silfit Z 91, with 147 units. The substitution of 33 % precipitated barium sulfate with Silfit Z 91 led to another moderate increase of haze to 166 units. Just for comparison, the total replacement of this special precipitated barium sulfate resulted in 204 units (*Fig. 29*).

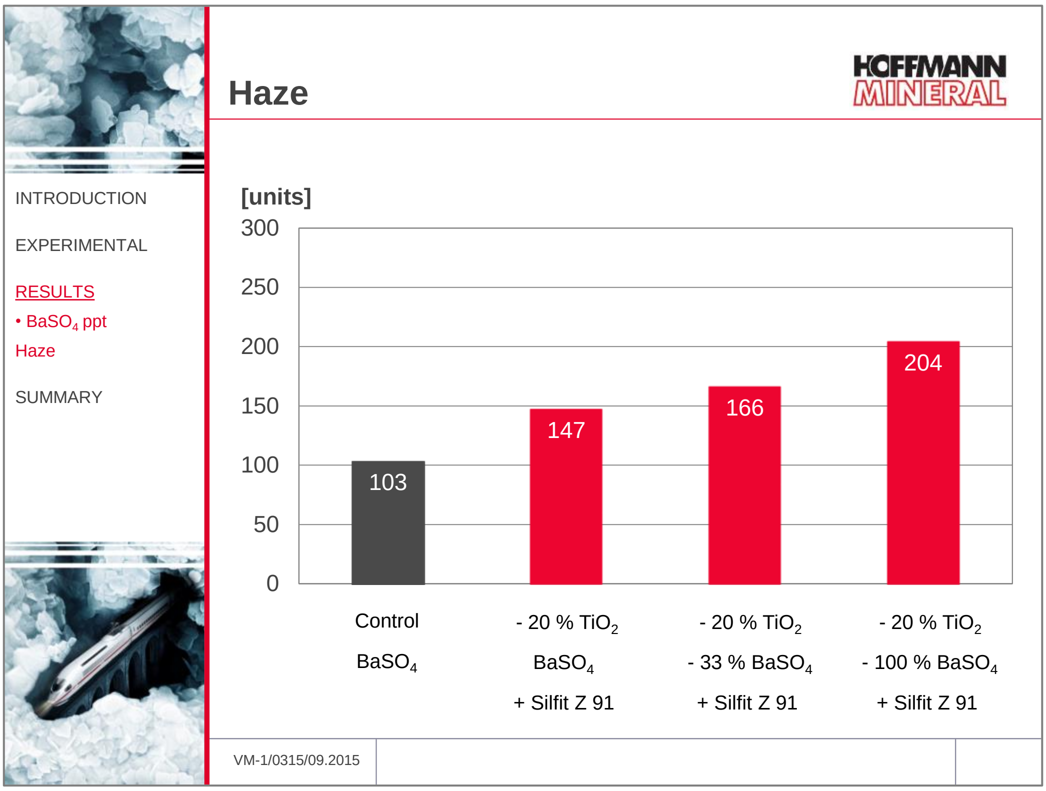


Fig. 29

4.5 Leveling

The replacement of 20 % titanium dioxide and up to 33 % of the precipitated barium sulfate allowed maintaining good leveling properties. The surface appeared smooth, and hardly any structure was visible. Only when replacing the total amount of the precipitated barium sulfate with Silfit Z 91, the appearance became slightly less favorable, i.e. the surface came out not quite so smooth, and some more structure stood out (*Fig. 30*).

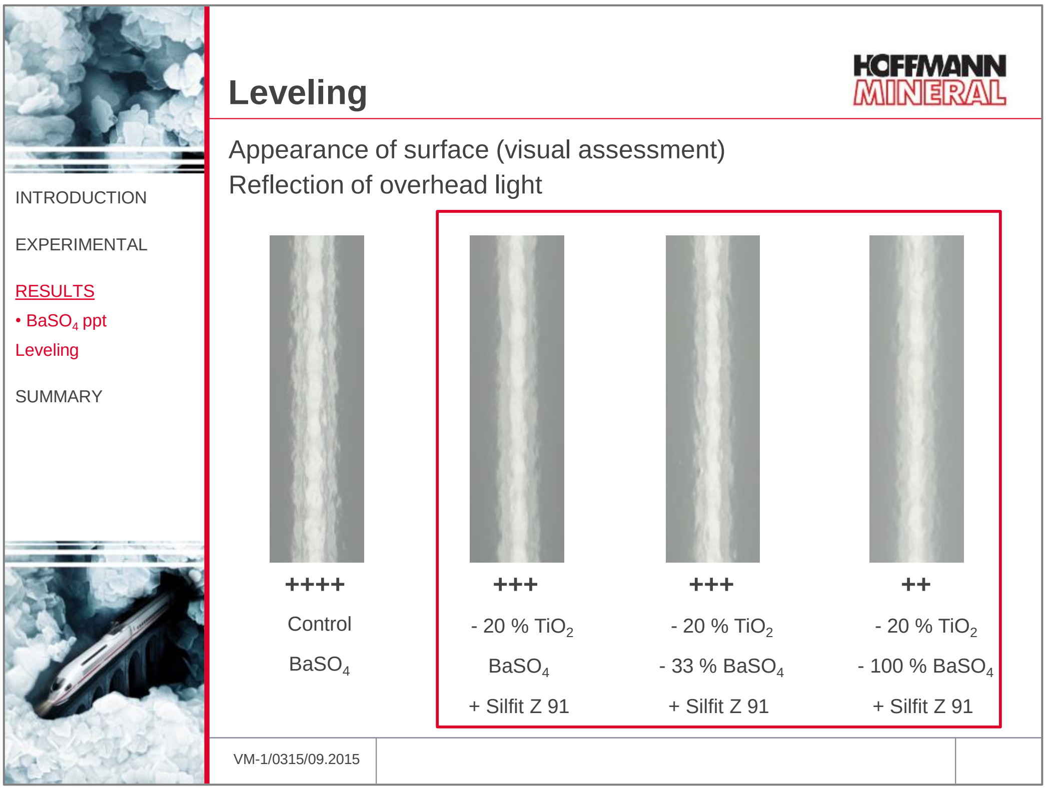
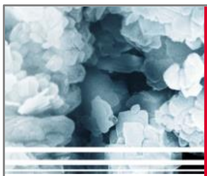


Fig. 30

4.6 Artificial weathering (QUV-A Test))

The artificial weathering was realized for 1000 hours with cycles of 4 hours UVA light at 340 nm at 50 °C and 4 hours condensation at 100 % relative humidity and 50 °C. The composition of the base recipe was a customer formulation and therefore different from our tested formulations. Beginning with the control formulation containing titanium dioxide, different steps with 10 %, 30 % and 50 % titanium dioxide substitution through Silfit Z 91 were evaluated. As depicted in the chart there is, even with the 50 % titanium dioxide substitution, nearly no change in Delta E after 1000 hours of exposure. The remaining gloss is almost as high as at the beginning. None of the formulations exhibited signs of chalking or white spots. Thus, concluding the results of the artificial weathering a reduction up to the half of titanium dioxide and therefore the introduction of Silfit Z 91 is easily possible *(Fig. 31).*



INTRODUCTION

EXPERIMENTAL

RESULTS
Artificial Weathering

SUMMARY

Artificial Weathering 1000h
Customer Feedback

HOFFMANN MINERAL

Cycles of 4 h UVA light (340 nm) at 50°C and
4 h condensation at 50°C and 100% relative humidity

Customer Formulation containing TiO_2 and $BaSO_4$	Control with TiO_2	- 10 % TiO_2 + Silfit Z 91	- 30 % TiO_2 + Silfit Z 91	- 50 % TiO_2 + Silfit Z 91
Delta E	1.2	1.1	1.5	1.3
Remaining Gloss [%]	92	93	93	96

None of the formulations exhibited after exposure signs of chalking or white spots.

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Fig. 31

4.7 Corrosion resistance (Acetic Salt Spray Test and Humidity Test)

Acetic Acid Salt Spray Test DIN EN ISO 9227 / AASS (2000 hours):

1. After exposure the panels were assessed regarding blistering (DIN EN ISO 4628/2): In *Fig. 32* the blistering on the surface of the panels is illustrated. The control formulation showed a smattering of defects, approximately 2 % of the upper right side surface was covered with blisters 3–3 (S2). All of the variations containing Silfit Z 91 showed no blistering.

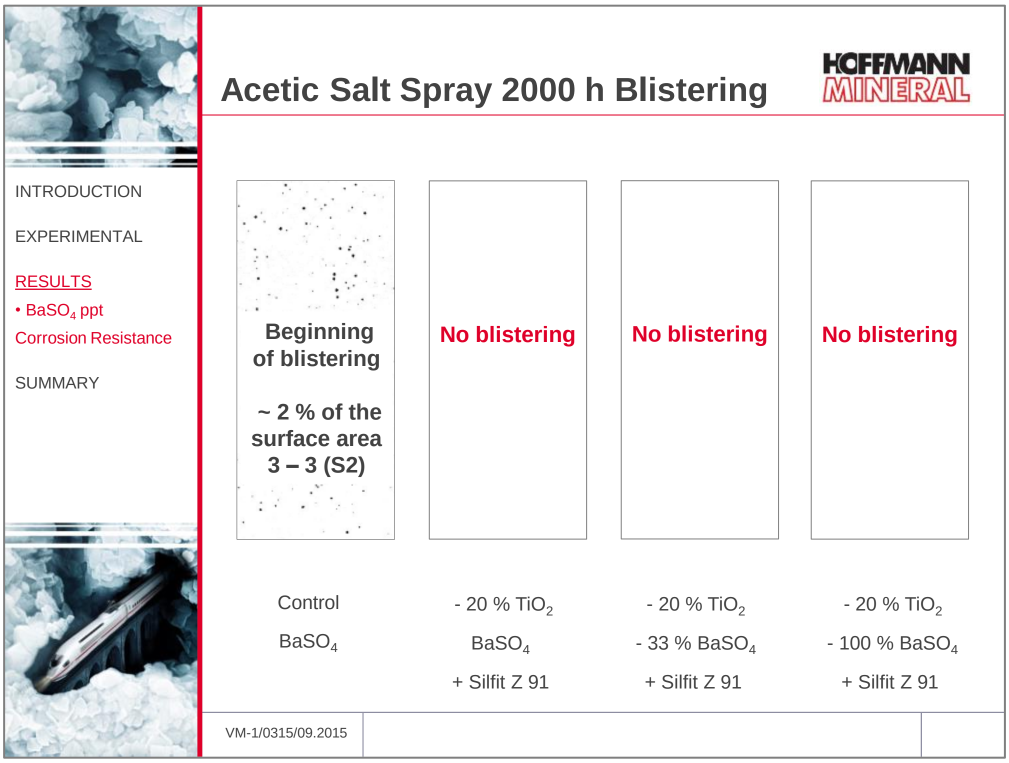

Fig. 32

2. The delamination at the scribe (scratching tool according to Sikkens, right-angled cutting edge, longitudinal section) was also evaluated (DIN EN ISO 4628/8):
   In *Fig. 33* the delamination at the scribe is illustrated. The control formulation had 0.8 mm delamination at the scribe. The variations containing Silfit Z 91 showed no = 0 mm delamination at the scribe.

   Corrosion at the scribe occurred only in terms of single blisters (point corrosion), however the same with all formulations, no further differentiation possible.

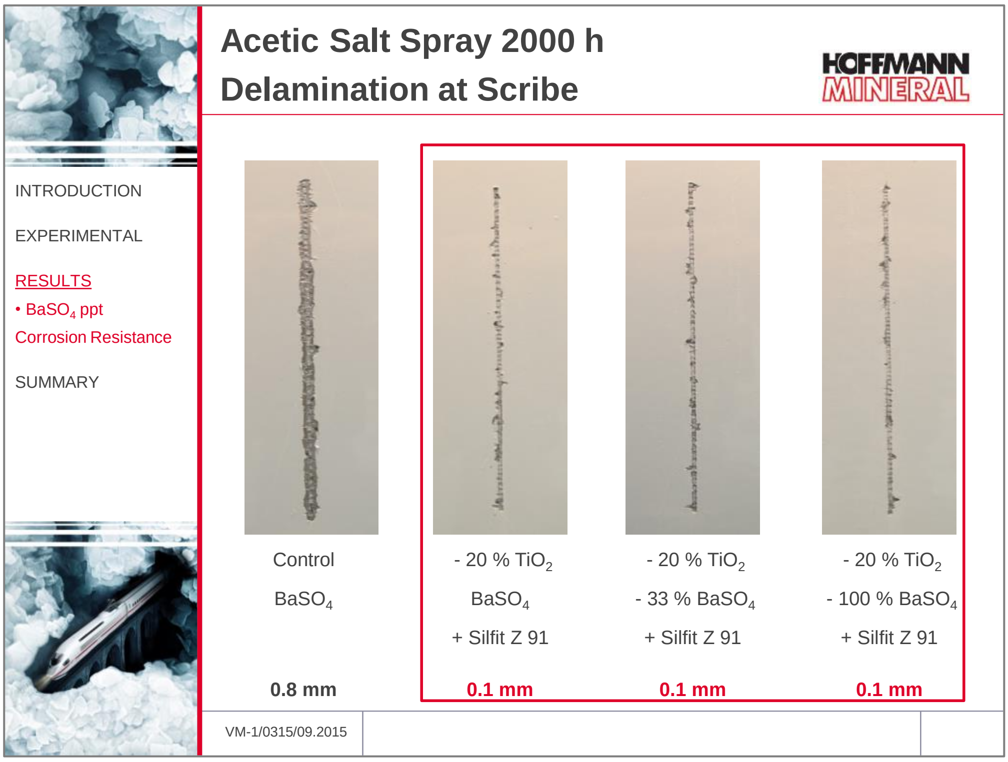

Fig. 33

Humidity Test DIN EN ISO 6270-2 / CH (2000 hours):

1. After exposure the panels were assessed to blistering (DIN EN ISO 4628/2):

   In *Fig. 34* the blistering on the surface of the panels is illustrated. The control formulation showed quite a lot of defects, the surface was covered with blisters 4-4 (S3). All of the variations containing Silfit Z 91 showed no blistering.

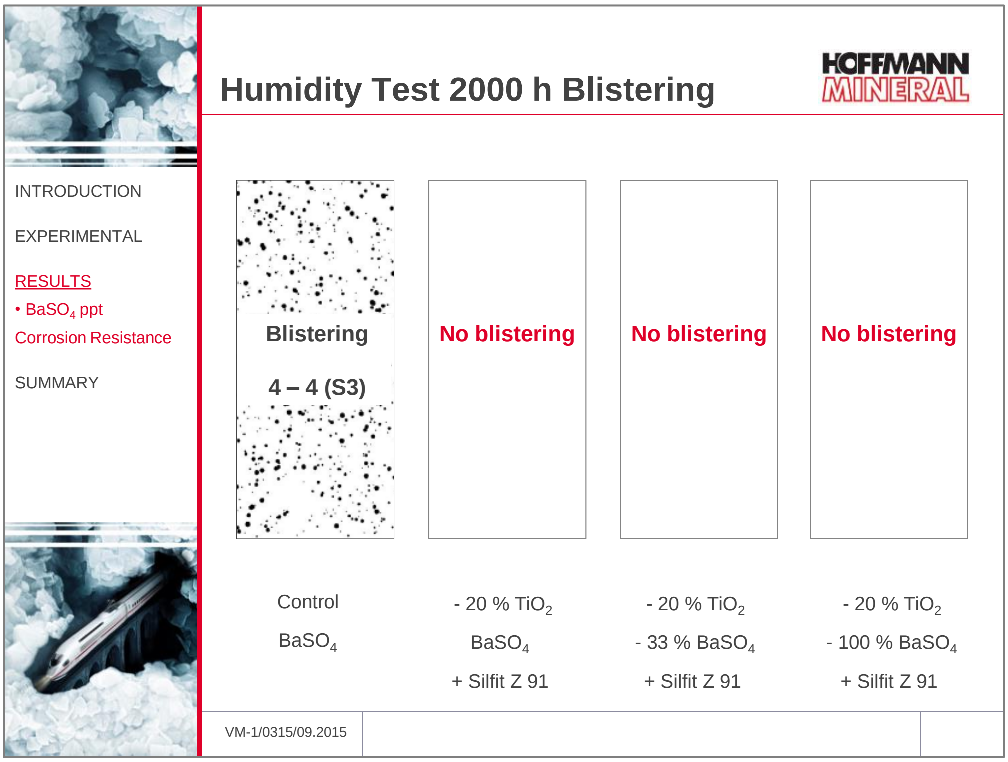

Fig. 34

2. The delamination at the scribe (scratching tool according to Sikkens, right-angled cutting edge, longitudinal section) was also evaluated (DIN EN ISO 4628/8):

   In *Fig. 35* the delamination at the scribe is illustrated. The control formulation had 24 mm delamination distance at the scribe. The variations containing Silfit Z 91 showed no signs of delamination at all. There was no corrosion at the scribe.

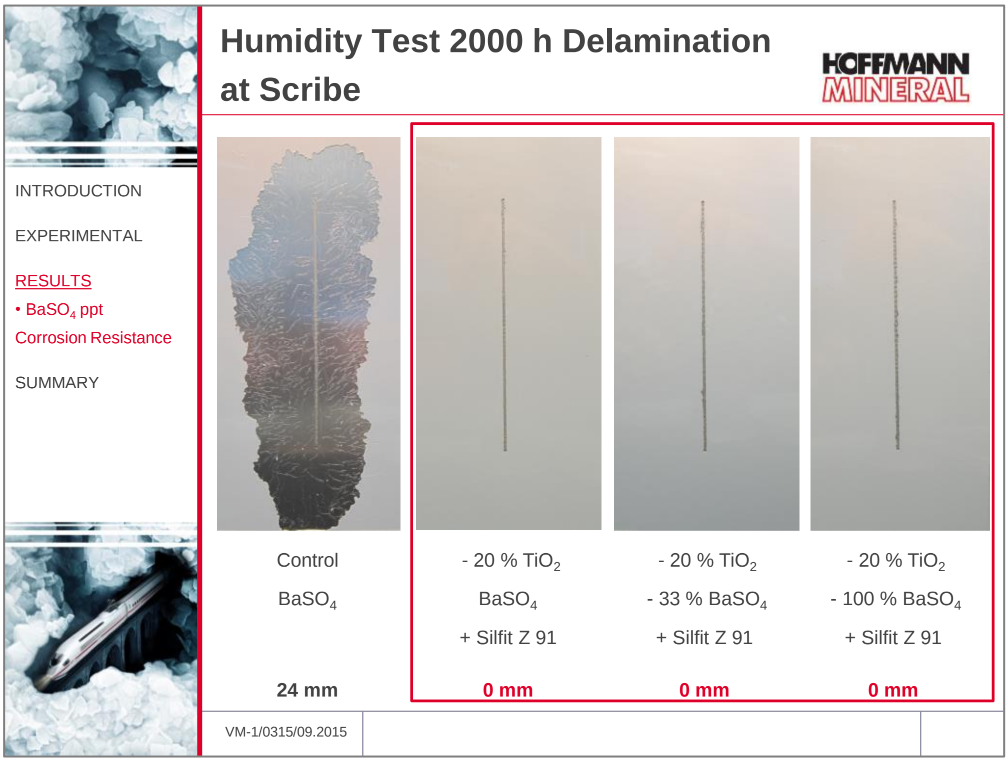

Fig. 35

4.8 Density and Spreading rate

Fig. 36 shows the densities of the formulations. The control exhibited the highest level of 1.61 g/cm³, caused by the density of the straight barite at 4.4 and titanium dioxide at 3.9. The replacement of 20 % titanium dioxide at equal weight, i.e. 4 pbw, by Silfit Z 91 with a density of 2.6 hardly affected the total density at all. However, when replacing 33 resp. 100 % of the barite at equal volume with Silfit Z 91, the density decreased down to 1.49 g/cm³. As shown in the following figure, this change has a positive effect on the spreading rate.

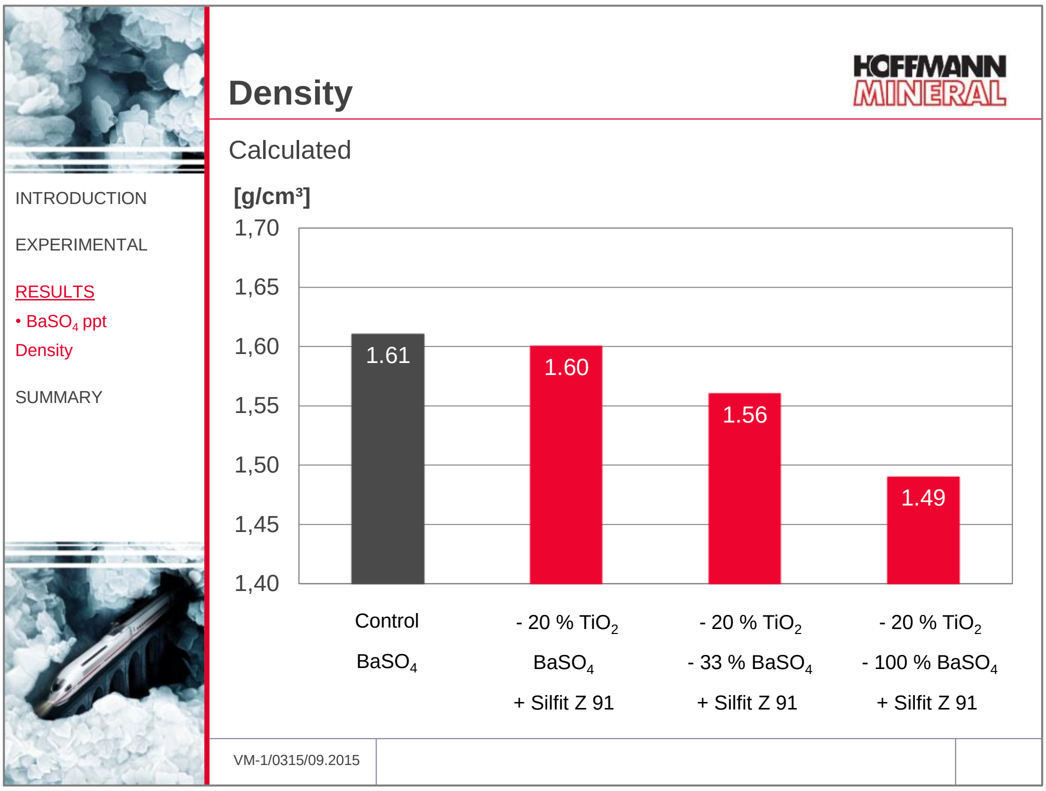

Fig. 36

Fig. 37 illustrates the spreading rate relative to the control as index. It shows how much surface can be coated by a mass unit of powder coating for a similar dry film thickness. As powder coatings are sold by weight, the use of Silfit Z 91 definitely gives rise to an increased spreading rate!

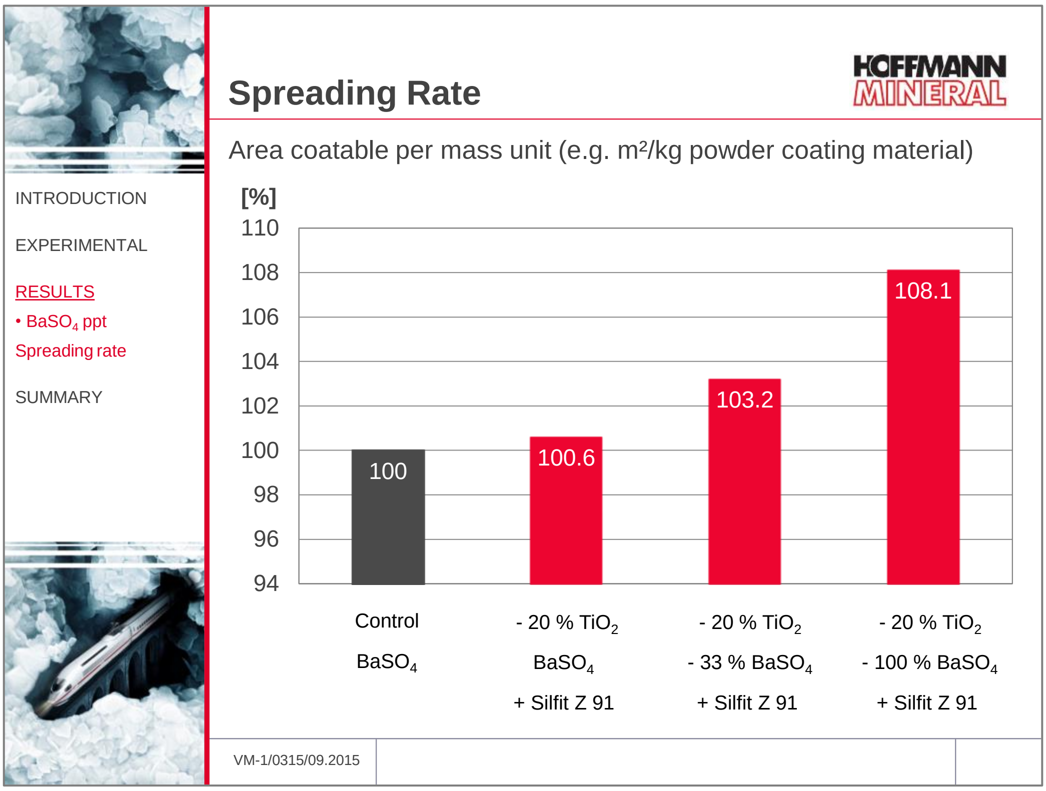

Fig. 37

4.9 Cost index

Fig. 38 summarizes the weight-related costs based on prices in Germany during the year 2014. The price for titanium dioxide was taken as € 2.40 per kg. Via the replacement of 20 % titanium dioxide with Silfit Z 91, cost savings of approx. 3 % could be achieved, through the additional substitution of 33 % of the precipitated barium sulfate almost 1.2 %. Replacing the total amount of precipitated barium sulfate by Silfit Z 91 gave rise to a cost increase of 2.8 %, which however is more than compensated by the almost 8.1 % higher spreading rate.

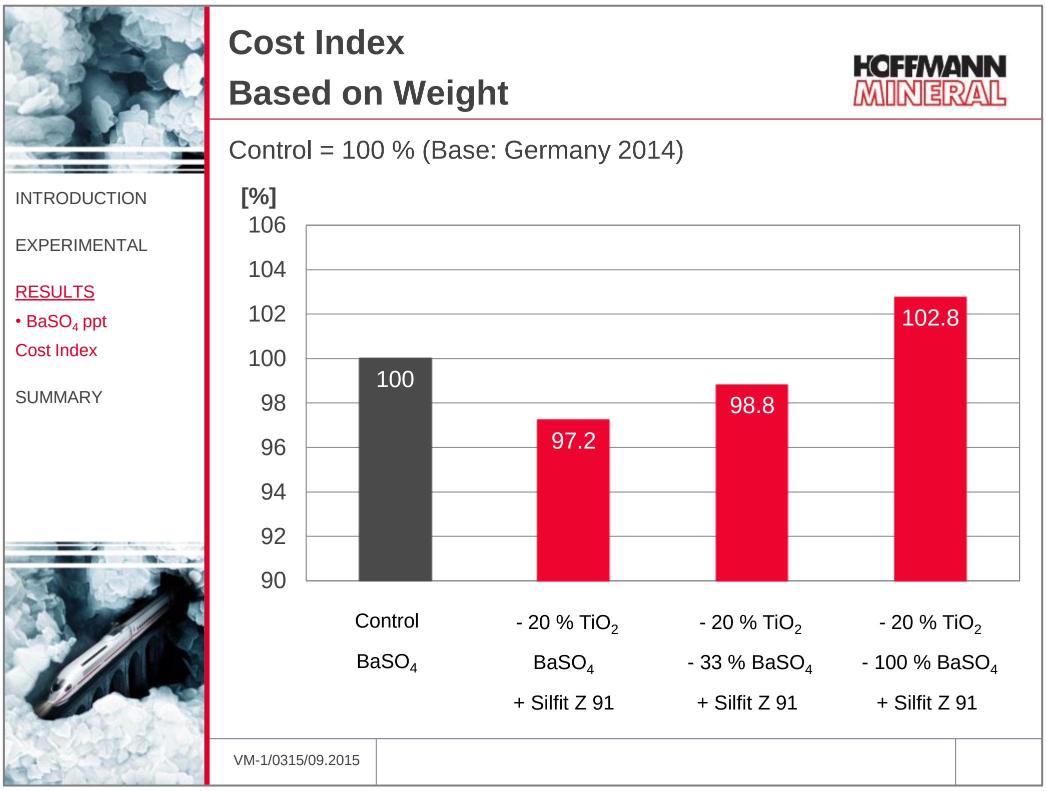

Fig. 38

If the cost index is calculated based on volume, all formulations with Silfit Z 91 gave rise to marked cost savings of 3.4 to nearly 5 % (*Fig. 39*).

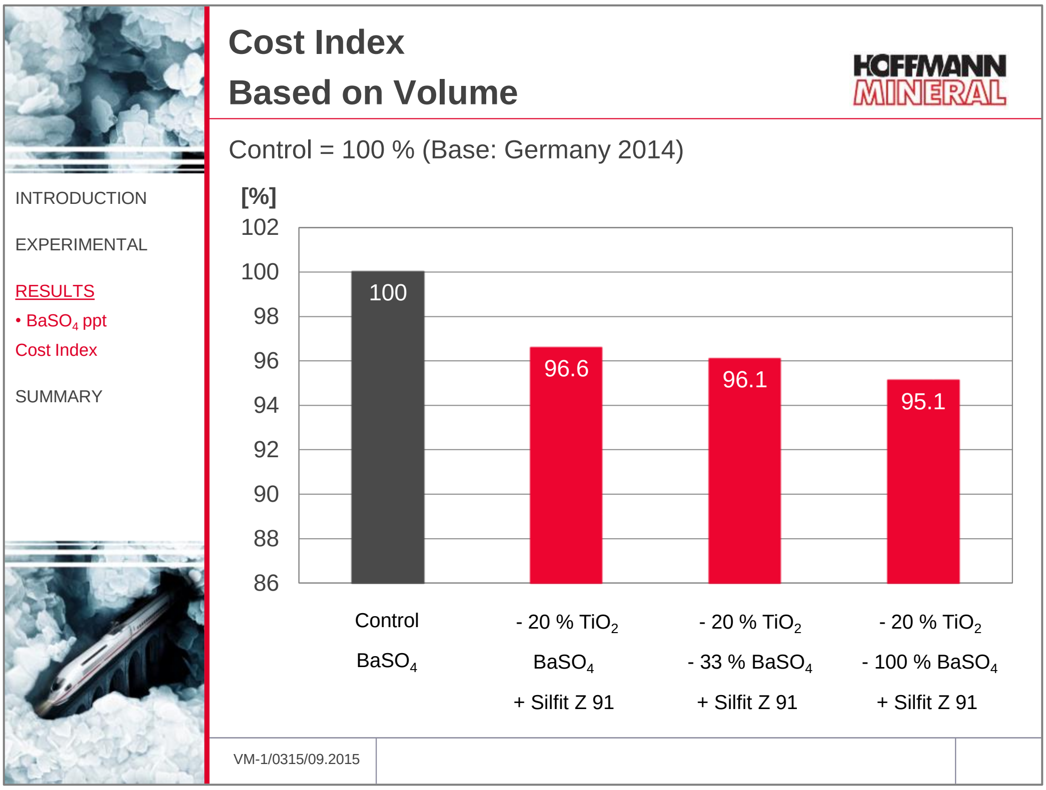

Fig. 39

4.10 Summary of the results with precipitated barium sulfate

The replacement of 20 % titanium dioxide by Silfit Z 91 at equal weight led to the following effects:

- except slightly higher haze, similar optical properties
- excellent weatherability (even up to 50 % titanium dioxide substitution)
- + improved corrosion resistance
- + potential for cost savings

The additional partial (33 %) substitution of the precipitated barium sulfate by Silfit Z 91 at equal volume gave rise to:

- + higher spreading rate through lower density
- + potential for cost savings

5 Overall summary and outlook

Irrespective of whether natural (barite) or precipitated barium sulfate was used, it has been shown possible to replace 20 % of the titanium dioxide loading by Silfit Z 91 at equal weight without losing significant hiding power or weatherability. The corrosion resistance could be considerably improved and it offered the most economizing potential.

The additional substitution at equal volume of barite by Silfit Z 91 improved the optical properties, increased the spreading rate and offered potential for cost savings.

The additional partial replacement at equal volume of the precipitated barium sulfate (33 %) by Silfit Z 91 increased the spreading rate and offered potential for cost savings.