# Reduced titanium dioxide content:

# Calcined Neuburg Siliceous Earth

# in hybrid powder coatings

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**Contents**

# 1 Introduction

Benefits of Neuburg Siliceous Earth have been shown earlier in study of hybrid based powder coatings with a focus on replacing barium sulfate. The optical and mechanical properties could be maintained or even improved.

In other projects, as for example in a Coil Coating Top Coat, the potential of Calcined Neuburg Siliceous Earth has been evaluated with success for a partial replacement of titanium dioxide.

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 the titanium dioxide in a hybrid powder coating while maintaining the mechanical and optical properties, above all the hiding power?

The major filler will remain in the formulation:

➢ Natural barium sulfate (barite), for cost-effective formulations to fulfill basic requirements

or

➢ Precipitated barium sulfate, a grade specially recommended for powder coatings that answer high optical demands

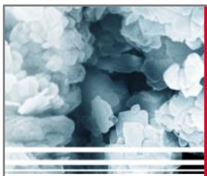
The objective of the study was to maintain or improve the performance characteristics while reducing the costs via replacing titanium dioxide with Calcined Neuburg Siliceous Earth.

# 2 Experimental

## 2.1 Base formulation

The base formulation given in *Fig. 1* represented the starting point of the study. Crylcoat 1771-3 is a carboxyl functional, medium reactive polyester for combination with epoxides, in this case Epikote 1003 based on Bisphenol A / epichlorohydrine. The mix ratio of polyester to epoxide in the powder coating was adjusted to 70 : 30. Additol P 896 and benzoin were used as leveling agents. The titanium dioxide was a micronized rutile pigment ($TiO_2$ content: 93 – 94 %), which had been treated with organic aluminum and silcon compounds.

The pigment volume concentration (PVC) of the base formulation was 16.3 %.



INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

**Base Formulation**

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Parts per weight = %

| | Base formulation |
|---|---|
| Crylcoat 1771-3 | 39.0 |
| Epikote 1003 | 18.0 |
| Additol P 896 | 3.0 |
| Titanium dioxide | 19.5 |
| Barium sulfate | 20.0 |
| Benzoin | 0.5 |
| Total | 100.0 |
| PVC [%] | 16.3 |

VM-1/0811/07.2013

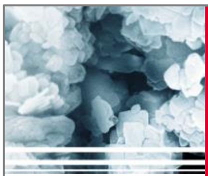
*Fig. 1*

## 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 aging, 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 formed practically amorphous. The silica portion remains inert at the temperature used. Through an integrated air classifier process grain sizes > 15 µm are being removed.

*Fig. 2* shows the typical properties of the natural and precipitated barium sulfate and the Calcined Neuburg Siliceous Earth, 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 higher specific surface area. The natural barium sulfate (barite) displayed a greater and the precipitated one a somewhat smaller medium particle size as well as top cut $d_{97}$ than Silfit Z 91.



**Filler Characteristics**

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INTRODUCTION

EXPERIMENTAL

RESULTS

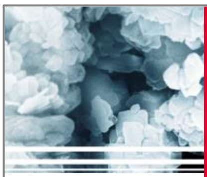
SUMMARY

| | | Barium sulfate | | Calcined Neuburg Siliceous Earth |
|---|---|---|---|---|
| | | natural | ppt. special grade for powder coatings | Silfit Z 91 |
| Morphology | | corpuscular | | corpuscular / lamellar aggregated |
| Density | [g/cm³] | 4.4 | 4.4 | 2.6 |
| Particle size $d_{50}$ | [µm] | 2.9 | 1.6 | 2.0 |
| Particle size $d_{97}$ | [µm] | 14 | 5 | 10 |
| Oil absorption | [g/100g] | 14 | 22 | 60 |
| Specific surface area BET | [m²/g] | 0.8 | 2.6 | 7.5 |

VM-1/0811/07.2013

*Fig. 2*

The color values were determined in a spectral photometer with geometry d/8° and light D 65. The precipitated barium sulfate, with an L* value of 97, showed the highest brightness, followed by the natural barium sulfate (barite) and Silfit Z 91 with L* = 95. The a* value of all fillers came out comparable at – 0.2 to – 0.5. For the b* value, which indicates the yellowish tint, a small difference between the fillers was evident: the barium sulfates with 0.2 to 0.5 revealed themselves somewhat more color neutral than Silfit Z 91 with b* = 1.2 (however, this sample came from a pilot production run, today´s figures typically run lower) (*Fig. 3*).



**Filler Characteristics**

HOFFMANN MINERAL

INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

| Color | Barium sulfate | | Calcined Neuburg Siliceous Earth |
|---|---|---|---|
| | natural | ppt. special grade for powder coatings | Silfit Z 91 |
| L* | 95 | 97 | 95 |
| a* | - 0.3 | - 0.5 | - 0.2 |
| b* | 0.2 | 0.5 | 1.2 |

VM-1/0811/07.2013

*Fig. 3*

## 2.3 Preparation of batches

The premix was mixed for 2 minutes at 1000 rpm in a Mixaco unit and subsequently homogenized in an extruder (Coperion ZSK 18, twin screw, shaft speed 350 rpm, heating zones 50°C / 100°C / 100°C / 100°C / 100°C). The batches were ground in a mill and finally sieved. Application was carried out with a GEMA powder pistol Corona with 80 kV and 2 bar onto Q-Panel sheets (aluminum A 36 and A 48). The coatings were baked for 15 minutes at an oven temperature of 180°C, which corresponds to a peak metal temperature (PMT) of about 180°C for 10 minutes. The dry film thickness came out at about 70 µm.

## 2.4 Test methods

Color values

The color values CIE L* a* and b* were determined in 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 company BYK. 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 unsatisfactory 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 company BYK.

Leveling

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

Flexibility (Reverse impact test)

For the impact test according to ASTM D 2794, a weight of 2 lbs (ball diameter 12.7 mm) is dropped from different heights (10 inches = 25 cm) onto the uncoated backside. The coating on the front side will then be judged for cracks. This study indicates the results in inch-pounds where just no more cracks could be observed.

Flexibility (Cupping test)

In the cupping test according to DIN ISO 1520, a hemisphere is pressed with constant slow speed from the backside into the coating, which then will be checked for developed cracks. The present study will indicate the maximum possible cupping depth in millimeters where just no cracks could be observed.

Mechanical resistance (scribe / scratch resistance)

The mechanical resistance was tested via the scribe or scratch resistance by scratching the coating with a weight-loaded metal tip until the substrate became visible. The test device, as shown in *Fig. 4*, was the Corrocutter of the Erichsen Company (model 639). The rounded hard metal tip according to van Laar had a diameter of 0.5 mm and was pulled over the powder coating layer with a stepwise increased load from 2 to 20 Newton. The test result was expressed as the force necessary to scratch the coating through to the substrate.

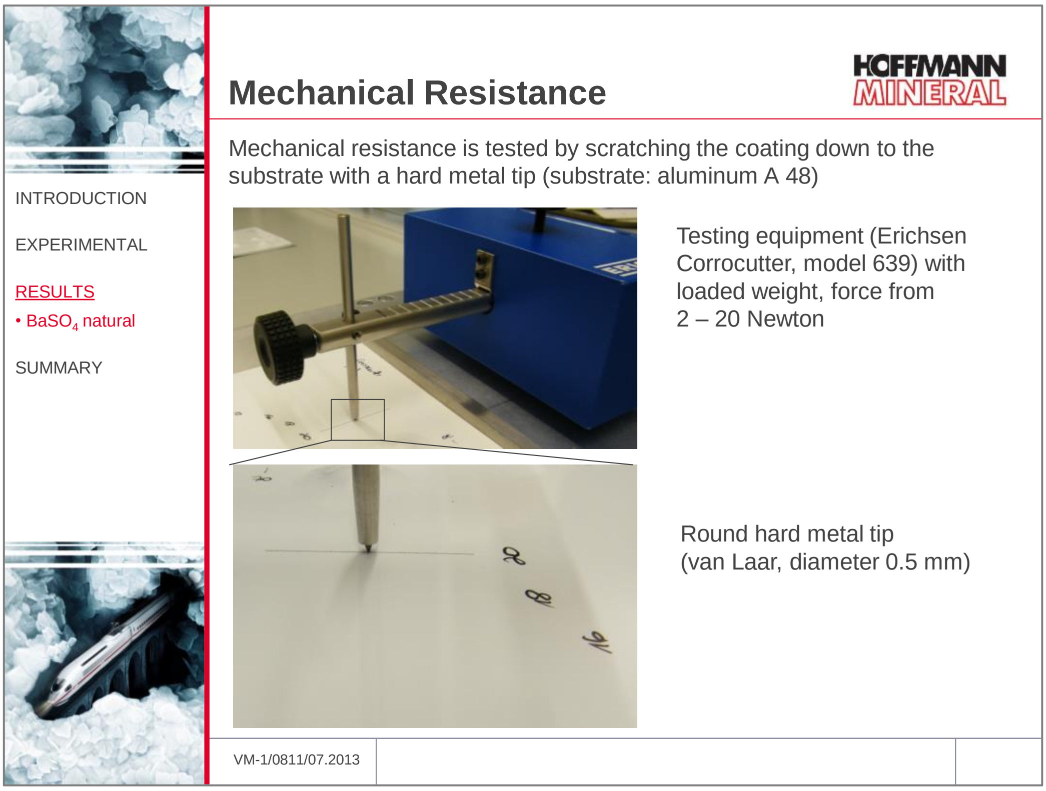


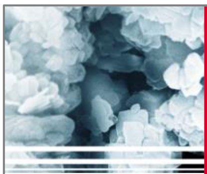

*Fig. 4*

# 3. Results with natural barium sulfate (barite)

## 3.1 Formulation variations

Starting from the base formulation (control) with 20 % barite and 19.5 % titanium dioxide, in the following variants 20 % of the titanium dioxide were replaced at equal weight (i.e. 3.9 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 additional 33 % of the barite and in the third and last version the total amount of barite was replaced at equal volume with Silfit Z 91. The different formulations are shown in *Fig. 5* in parts by weight and in *Fig. 6* in percent.

All formulations had slightly increased PVC of 17.1 %, compared with the control at 16.3 %, 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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INTRODUCTION

EXPERIMENTAL
• $BaSO_4$ natural

RESULTS

SUMMARY

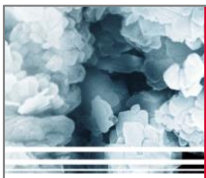
**Formulations**

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 1771-3 | 39.0 | 39.0 | 39.0 | 39.0 |
| Epikote 1003 | 18.0 | 18.0 | 18.0 | 18.0 |
| Additol P 896 | 3.0 | 3.2 | 3.2 | 3.2 |
| Titanium dioxide | 19.5 | 15.6 | 15.6 | 15.6 |
| $BaSO_4$ natural | 20.0 | 20.0 | 13.4 | - |
| Silfit Z 91 | - | 3.9 | 7.8 | 15.7 |
| Benzoin | 0.5 | 0.5 | 0.5 | 0.5 |
| **Total** | **100.0** | **100.2** | **97.5** | **92.0** |
| PVC [%] | 16.3 | 17.1 | 17.1 | 17.1 |

VM-1/0811/07.2013

*Fig. 5*



INTRODUCTION

EXPERIMENTAL

• $BaSO_4$ natural

RESULTS

SUMMARY

## Formulations

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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 1771-3 | 39.0 | 38.9 | 40.0 | 42.4 |
| Epikote 1003 | 18.0 | 18.0 | 18.5 | 19.6 |
| Additol P 896 | 3.0 | 3.2 | 3.3 | 3.5 |
| Titanium dioxide | 19.5 | 15.6 | 16.0 | 16.9 |
| $BaSO_4$ natural | 20.0 | 20.0 | 13.7 | - |
| Silfit Z 91 | - | 3.9 | 8.0 | 17.1 |
| Benzoin | 0.5 | 0.5 | 0.5 | 0.5 |
| **Total** | **100** | **100** | **100** | **100** |
| PVC [%] | 16.3 | 17.1 | 17.1 | 17.1 |

VM-1/0811/07.2013

*Fig. 6*

## 3.2 Color values

The a* value, which indicates the red/green portions, for all formulations came off at a level of - 0.7 to - 0.8. The brightness L* was highest with the control at 95.2. In the variants, the L* value remained at a high level of 94, despite the titanium dioxide reduction of 20 % (*Fig. 7*). The color index b*, which stands for the yellow/blue portions, increased only little. This rise will be further attenuated with the lower b* values of Silfit Z 91 from series production (*Fig. 8*).

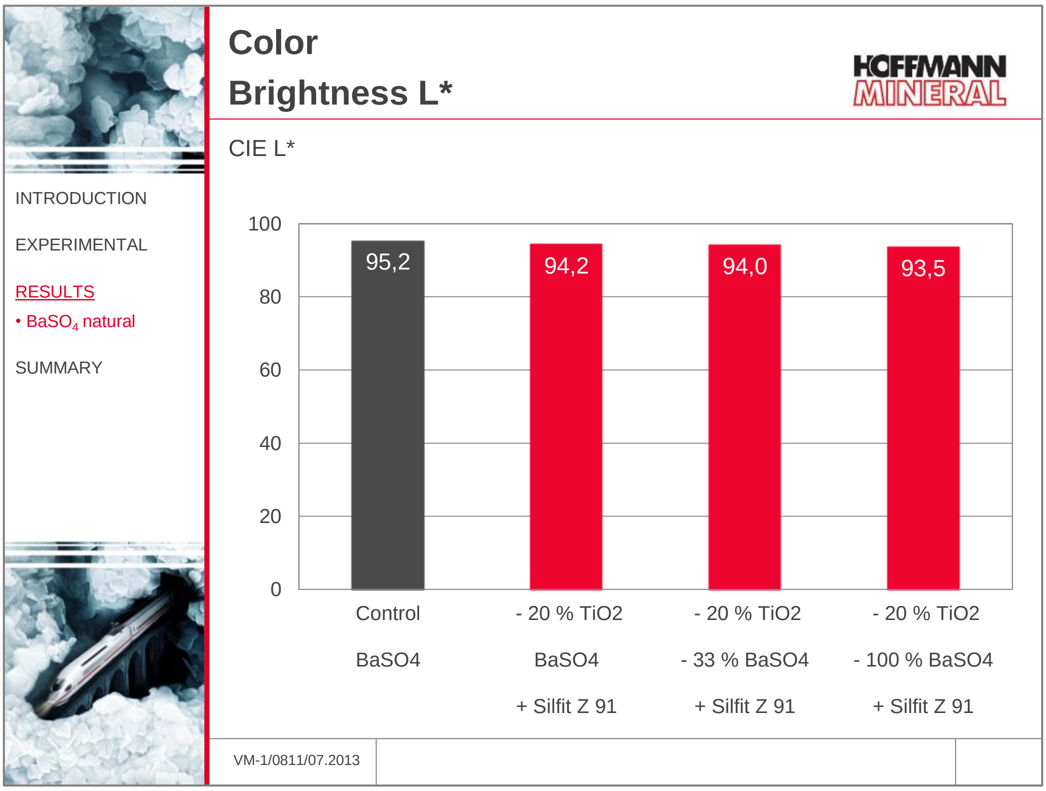

Color
Brightness L*
HOFFMANN MINERAL
CIE L*
INTRODUCTION
EXPERIMENTAL
RESULTS
• $BaSO_4$ natural
SUMMARY
100
80
60
40
20
0
95,2
94,2
94,0
93,5
Control
BaSO4
- 20 % TiO2
BaSO4
+ Silfit Z 91
- 20 % TiO2
- 33 % BaSO4
+ Silfit Z 91
- 20 % TiO2
- 100 % BaSO4
+ Silfit Z 91
VM-1/0811/07.2013

*Fig. 7*

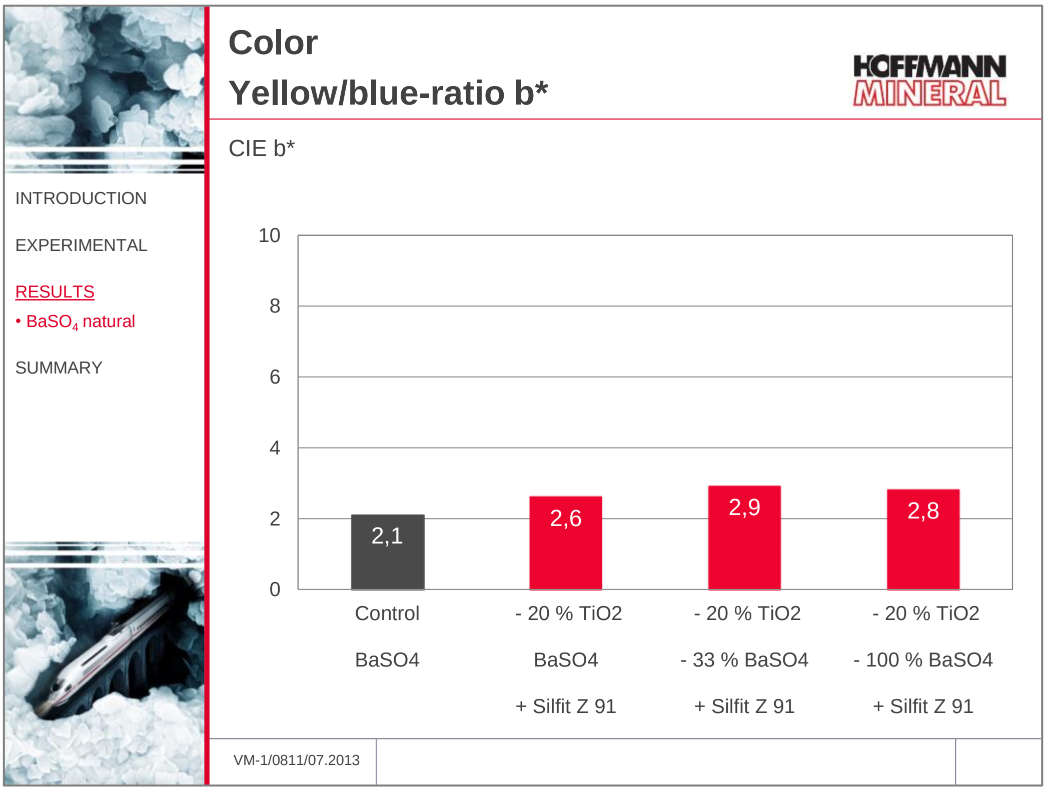

Color
Yellow/blue-ratio b*
HOFFMANN MINERAL
CIE b*
INTRODUCTION
EXPERIMENTAL
RESULTS
• $BaSO_4$ natural
SUMMARY
10
8
6
4
2
0
2,1
2,6
2,9
2,8
Control
BaSO4
- 20 % TiO2
BaSO4
+ Silfit Z 91
- 20 % TiO2
- 33 % BaSO4
+ Silfit Z 91
- 20 % TiO2
- 100 % BaSO4
+ Silfit Z 91
VM-1/0811/07.2013

*Fig. 8*

## 3.3 Hiding power / Opacity

*Fig. 9* shows the opacity results at dry film thickness of about 70 µm. Despite 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. The additional replacement of barite even brought about a trend towards increase, with the result that the formulation exclusively filled with Silfit Z 91 came up to the level of the control. In view of deviations in film thickness and brightness measurements, the hiding power of all formulations can be judged as equal.

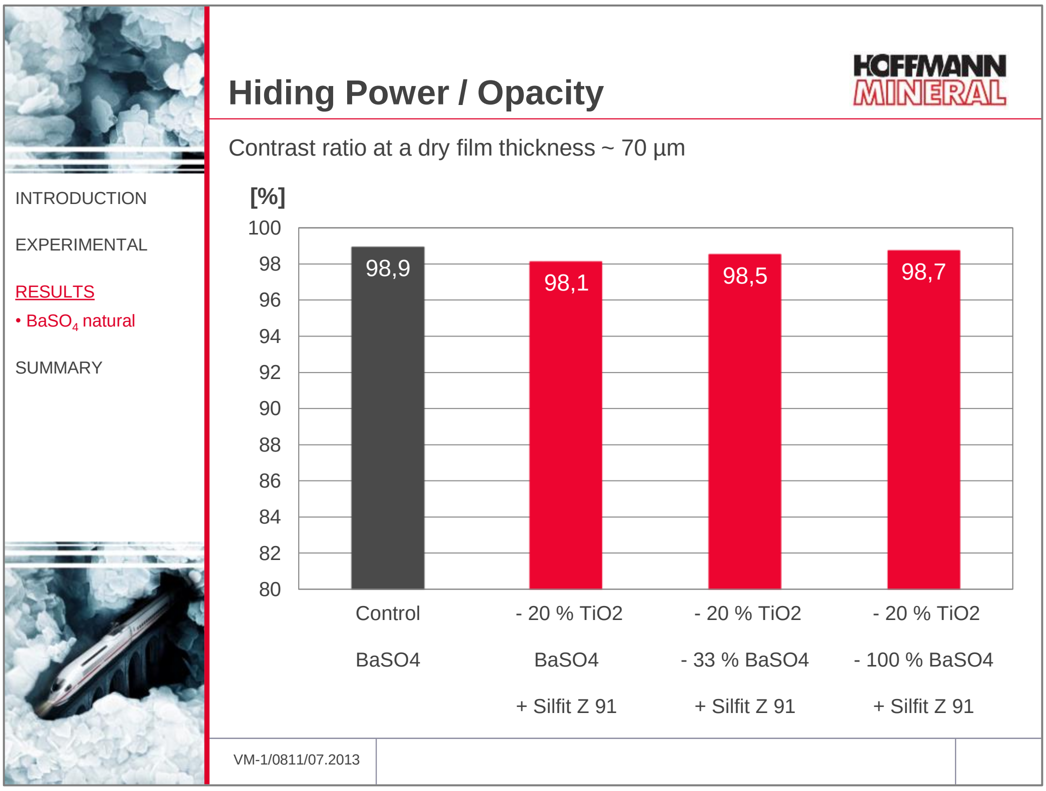



*Fig. 9*

## 3.4 Gloss and Haze

The control, which contained the full loading of titanium dioxide and barite, had a gloss of 58 units with the measuring angle of 20°. The substitution of 20 % titanium dioxide with Silfit Z 91 did not change this result. Through replacing, aside from the titanium dioxide, also the barite with Silfit Z 91, the gloss could be increased, in case of the total substitution even up to 78 units (*Fig. 10*).

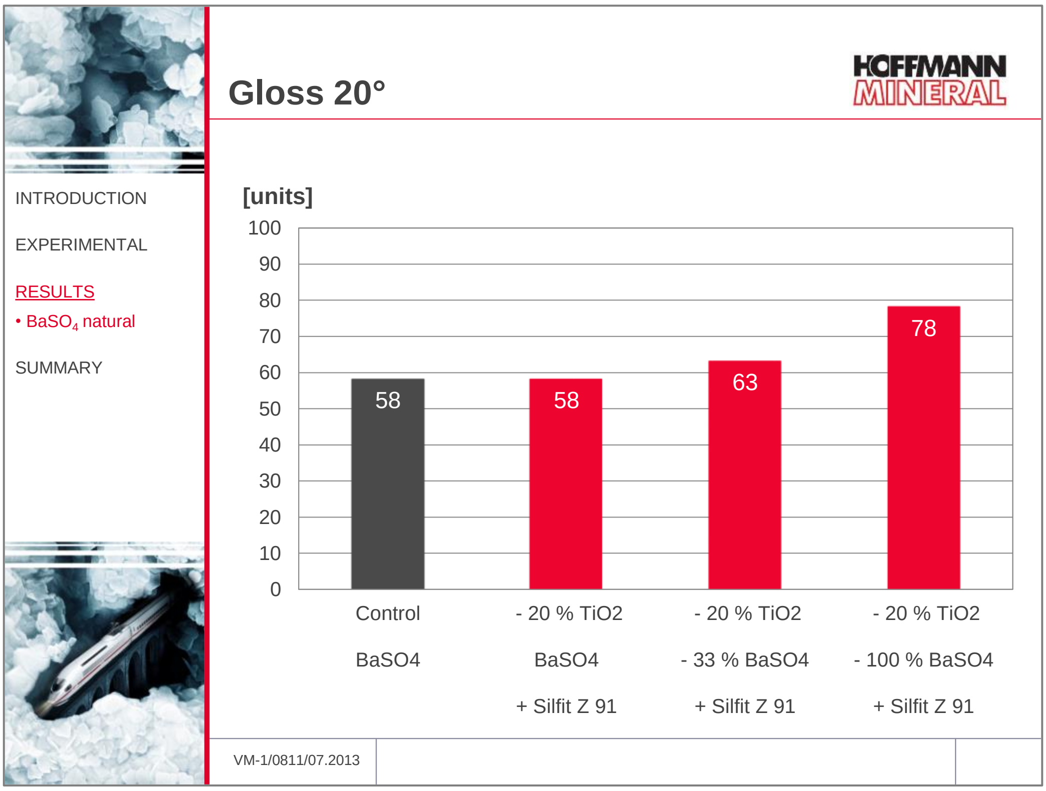



*Fig. 10*

*Fig. 11* illustrates the gloss at a measuring angle of 60°. The gloss-increasing effect of Silfit Z 91 here does not came out as pronounced as at 20°, but remains clearly visible at the total barite replacement with 97 units.

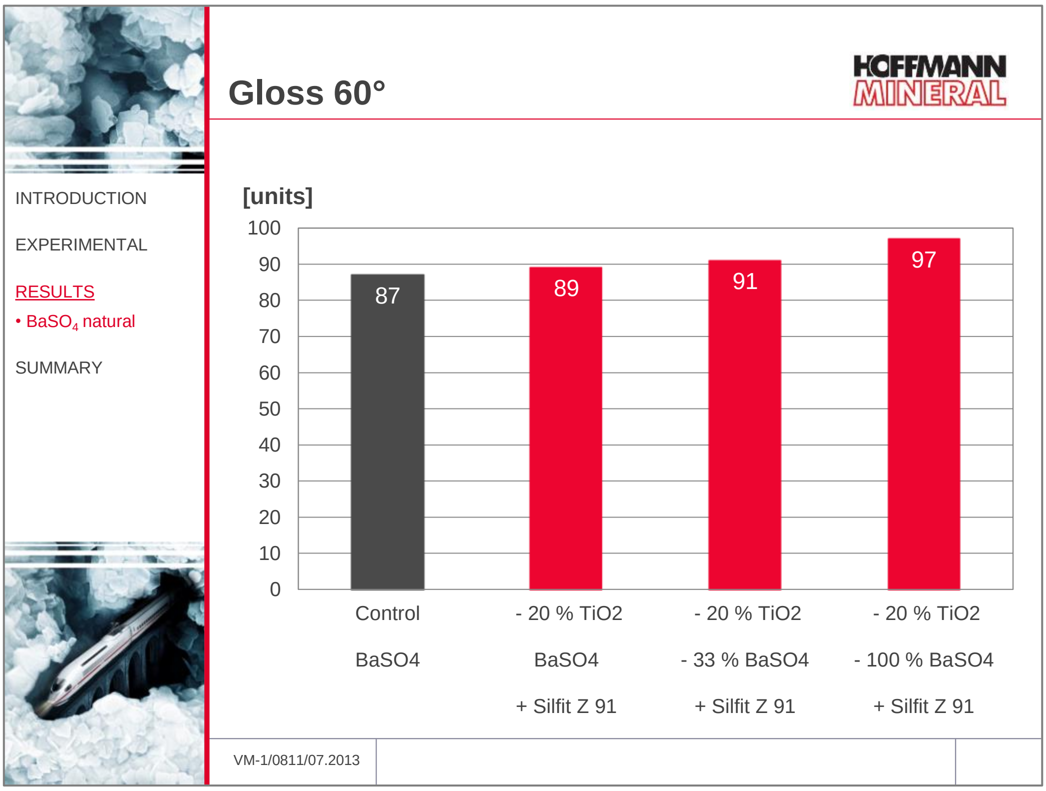



*Fig. 11*

The control had a haze of 329 units, comparable with the first variant, where only titanium dioxide was substituted with Silfit Z 91. The haze could be reduced down to 199 units by completely replacing the barite with the Calcined Neuburg Siliceous Earth. Silfit Z 91 allows obtaining a markedly better optical impression than achievable with barite (*Fig.12*).

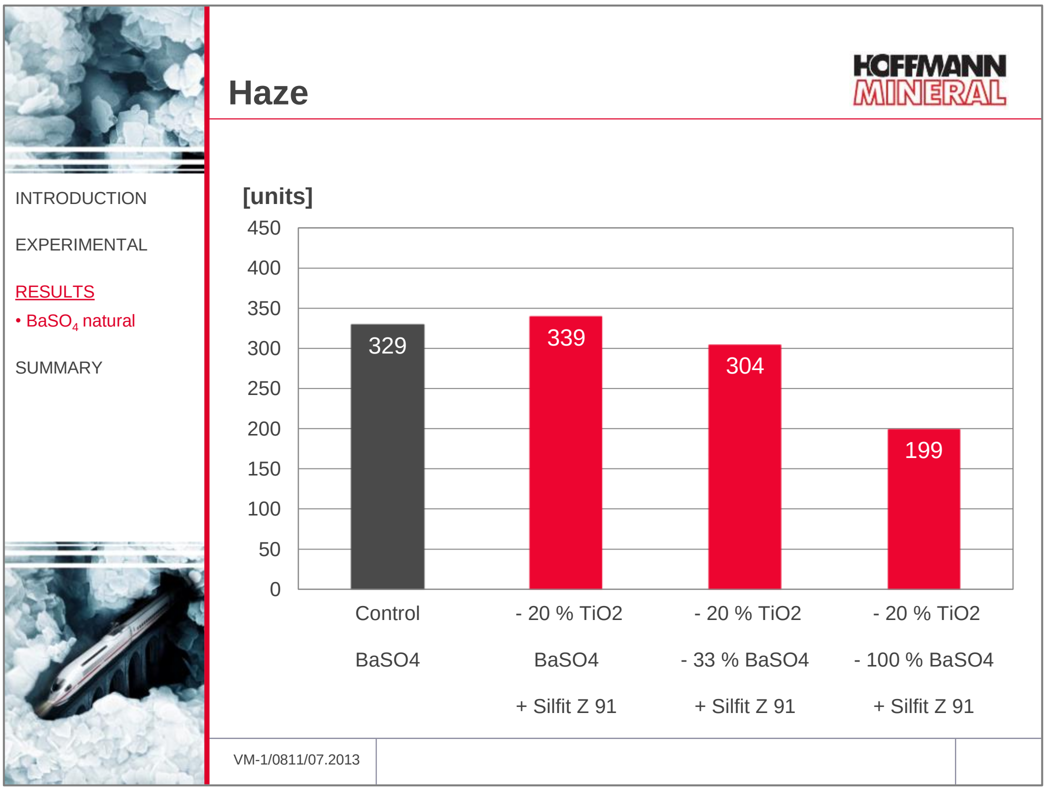
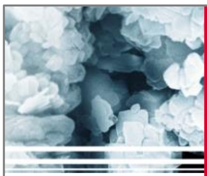



*Fig. 12*

## 3.5 Leveling

When 20 % of the titanium dioxide was replaced with Silfit Z 91, surface structure did not change at all. The more barite was substituted by Silfit Z 91, the better resulted the appearance, which means less structure became visible. The surface appeared smoother and a better leveling was evident (*Fig.13*).



INTRODUCTION

EXPERIMENTAL

RESULTS

• $BaSO_4$ natural

SUMMARY

**Leveling**

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Appearance of surface (visual assessment)

Substrate: aluminum A 48

| | Reflection of overhead light | |
|---|---|---|
| Control<br>$BaSO_4$ |  | 0 |
| - 20 % $TiO_2$<br>$BaSO_4$<br>+ Silfit Z 91 |  | 0 |
| - 20 % $TiO_2$<br>- 33 % $BaSO_4$<br>+ Silfit Z 91 |  | 0+ |
| - 20 % $TiO_2$<br>- 100 % $BaSO_4$<br>+ Silfit Z 91 |  | + |

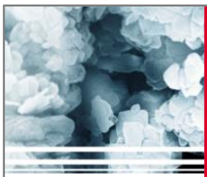
VM-1/0811/07.2013

*Fig. 13*

## 3.6 Flexibility (Impact test and cupping)

The control, the pure replacement of titanium dioxide and the - 33 % barite formulation all gave evidence of a comparable flexibility in the Reverse Impact Test with results between 12 and 18 inch-pounds. Only when no barite at all, i.e. exclusively Silfit Z 91 was used, an improvement up to 28 inch-pounds could be obtained *(Fig. 14).*

The cupping test results came out comparably well with 6-7 mm. A further differentiation could not be observed *(Fig. 14).*



INTRODUCTION

EXPERIMENTAL

RESULTS

• $BaSO_4$ natural

SUMMARY

**Flexibility**

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Impact Test ASTM D 2794 (weight: 2 lbs); no visible cracks
Cupping Test DIN ISO 1520
Substrate: aluminum A 36

| | Reverse Impact Test [inch pounds] | Cupping Test [mm] |
|---|---|---|
| Control $BaSO_4$ | 18 | 6.9 |
| - 20 % $TiO_2$ $BaSO_4$ + Silfit Z 91 | 14 | 6.4 |
| - 20 % $TiO_2$ - 33 % $BaSO_4$ + Silfit Z 91 | 12 | 6.7 |
| - 20 % $TiO_2$ - 100 % $BaSO_4$ + Silfit Z 91 | 28 | 6.4 |

VM-1/0811/07.2013

*Fig. 14*

## 3.7 Mechanical resistance (scribe/scratch resistance)

Silfit Z 91 here showed positive effects. Already at low loadings, just replacing part of the titanium dioxide, with 18 Newton it gave a better scratch resistance compared with the control at only 14 Newton. A further improvement of the scratch resistance by replacing the barite with Silfit Z 91 proved not possible (*Fig. 15*).

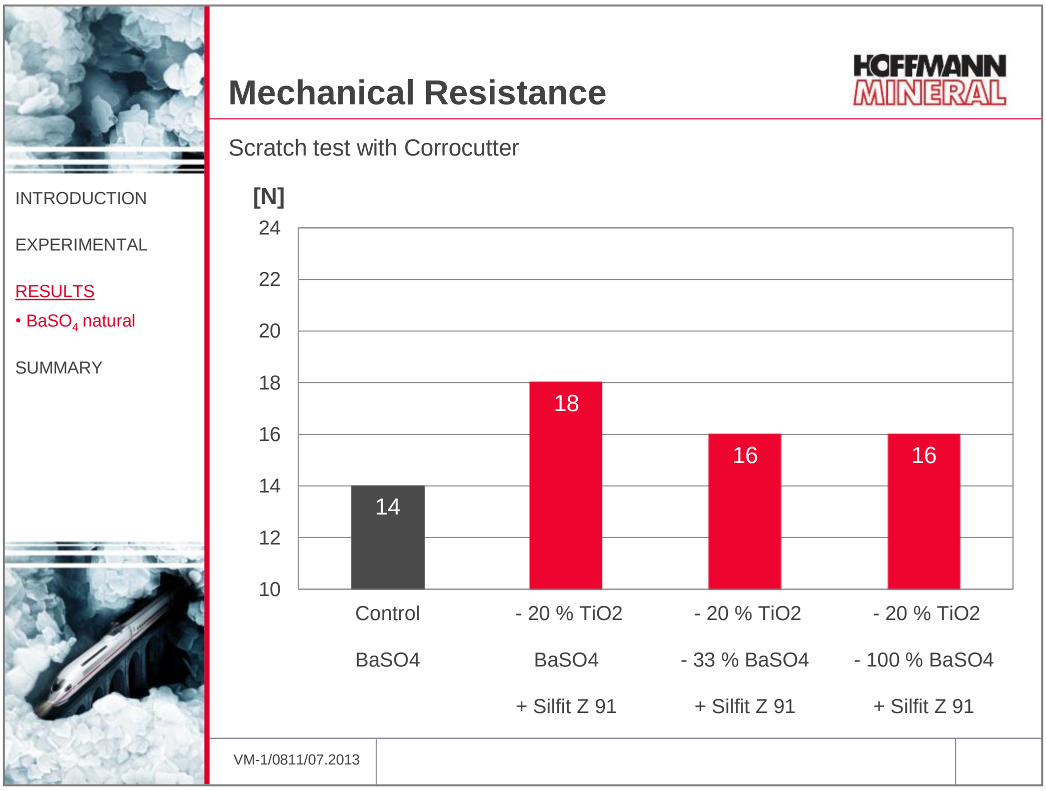



*Fig. 15*

## 3.8 Density and Spreading rate

*Fig. 16* shows the densities of the formulations. The control exhibited the highest level of 1.67 g/cm³, caused by the density of the straight barite at 4.4 and titanium dioxide at 4.1. The replacement of 20 % titanium dioxide at equal weight, i.e. 3.9 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 1.52 g/cm³. As shown in the following figure, this change has a positive effect on the spreading rate.

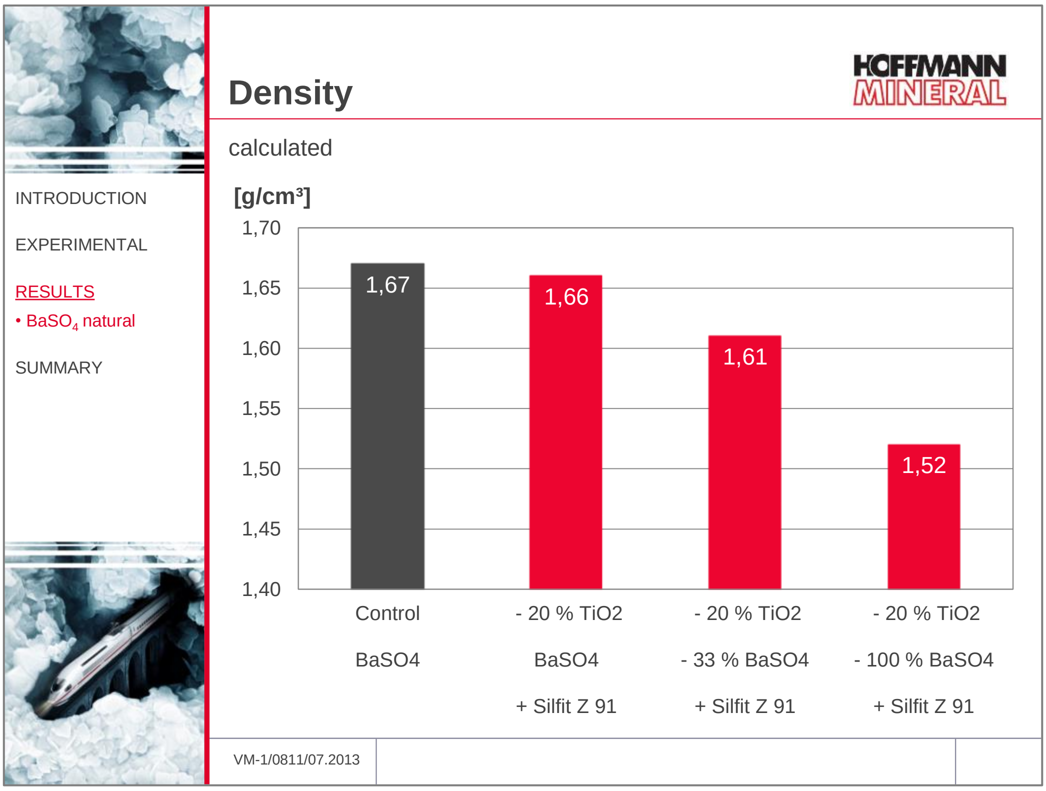



*Fig. 16*

*Fig. 17* 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!

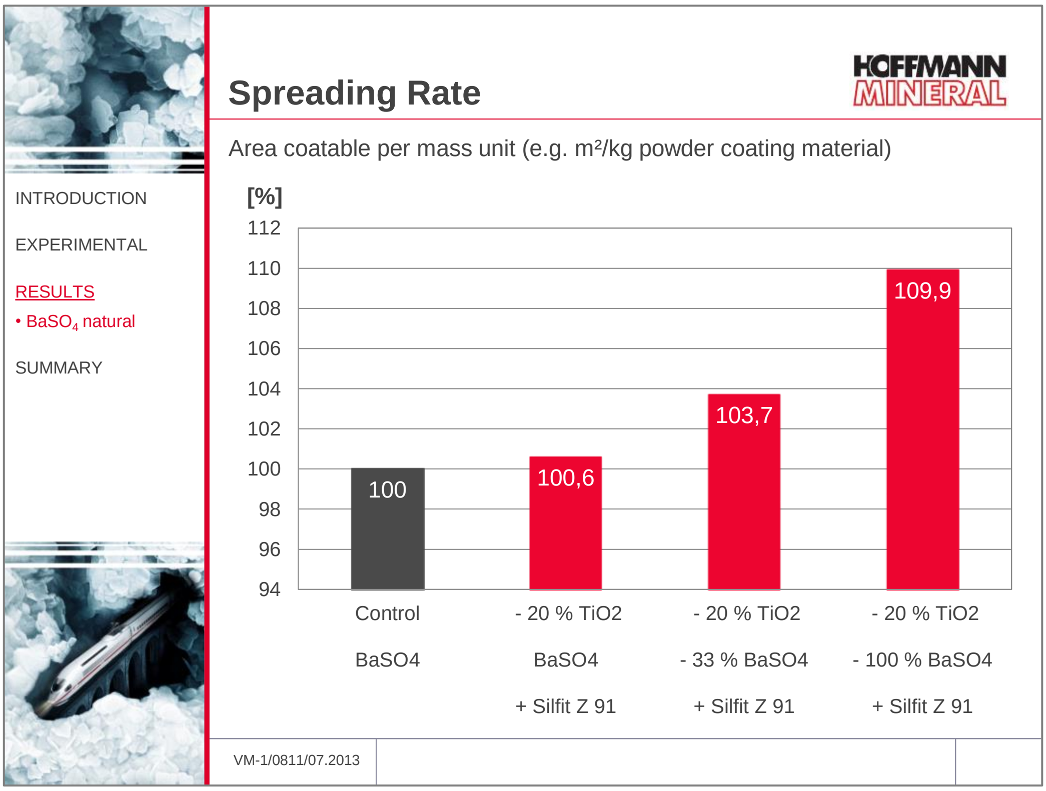



*Fig. 17*

## 3.9 Cost index

*Fig. 18* gives the weight-related costs based on German prices during the year 2011. The price for titanium dioxide was taken as € 2.65 per kg. The replacement of 20 % titanium dioxide with Silfit Z 91 allowed saving approx. 4 % of the costs. The further partial replacement of barite reduced the cost advantage to 1.5 %. The complete replacement of the barite with Silfit Z 91 caused a cost increase of 3.7 %, which however is more than compensated by the almost 10 % higher spreading rate.

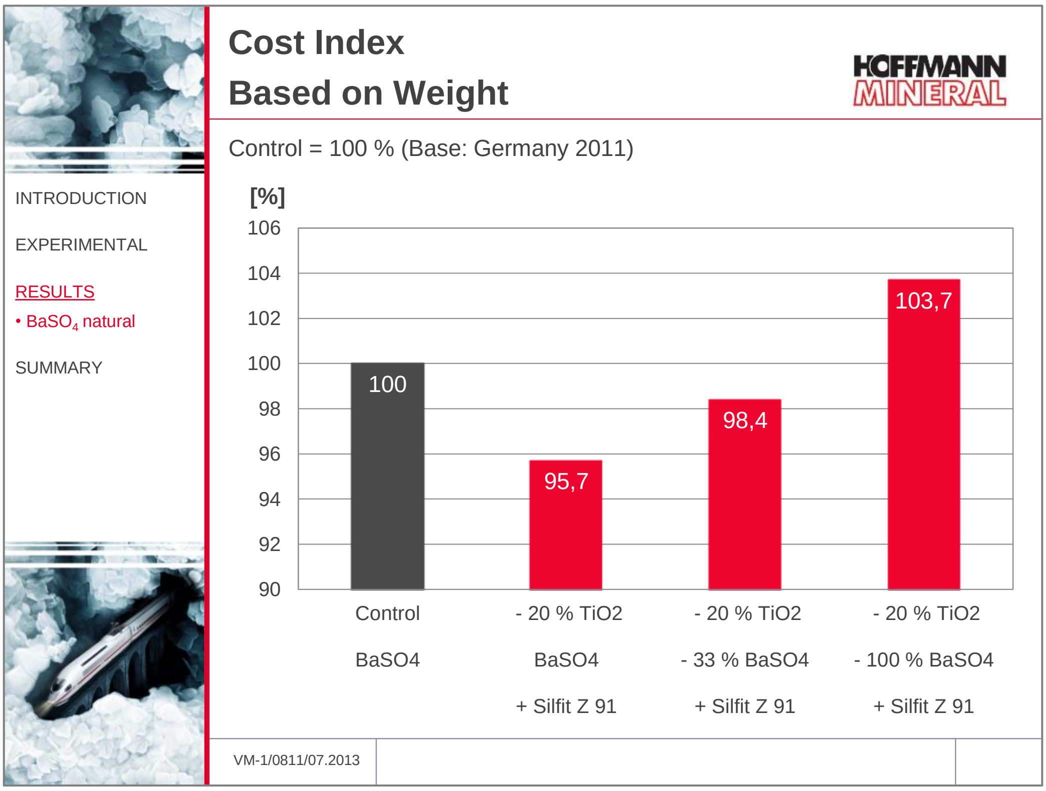



*Fig. 18*

If the cost index is calculated based on volume, all formulations with Silfit Z 91 gave rise to marked cost savings of 5 to 6 % (*Fig. 19*).

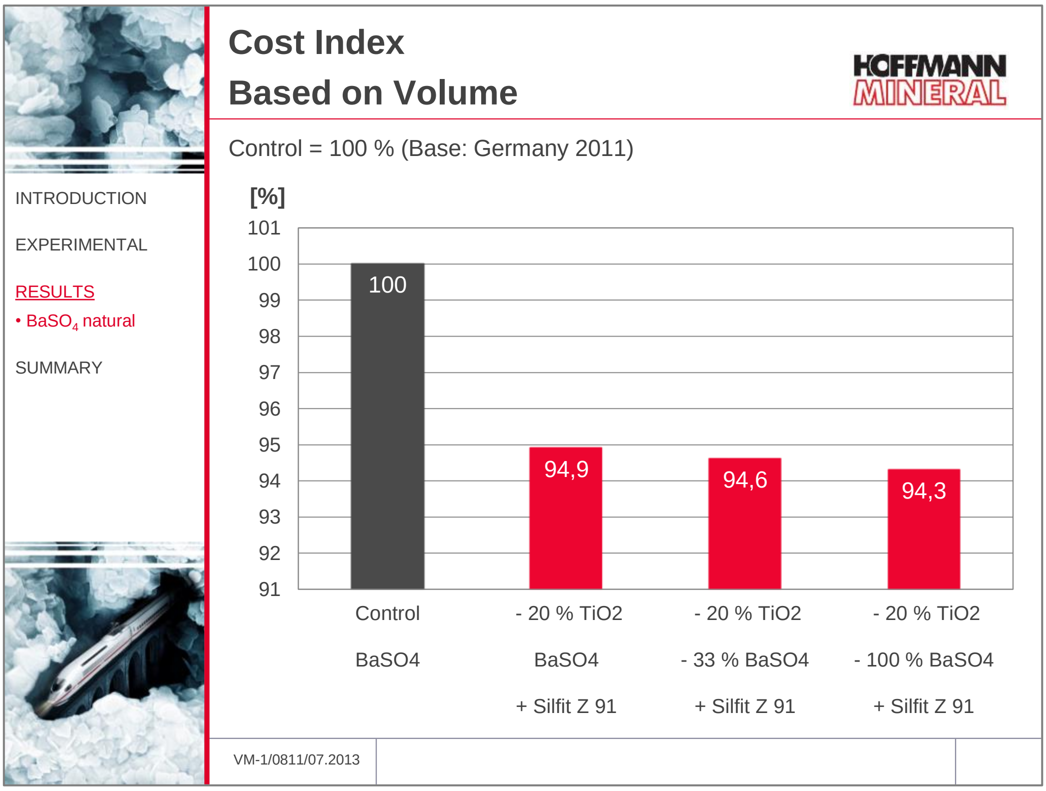



*Fig. 19*

## 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:

- ✓ comparable optical properties and flexibility
- ✓ improved scribe / scratch resistance
- ✓ potential for cost savings

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

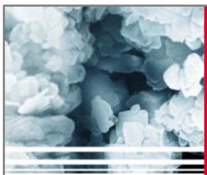
- ✓ higher gloss
- ✓ lower haze
- ✓ better leveling
- ✓ improved spreading rate through lower density of the coating
- ✓ potential for cost savings

## 4. Results with precipitated barium sulfate

### 4.1 Formulation variations

Starting from the base formulation (control) with 20 % precipitated barium sulfate and 19.5 % titanium dioxide, in the following variants 20 % of the titanium dioxide were replaced at equal weight (i.e. 3.9 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 precipitated barium sulfate and in the third and last version the total amount of precipitated barium sulfate was replaced at equal volume with Silfit Z 91. The corresponding formulations are listed in *Fig. 20* in parts by weight and in *Fig. 21* in percent.

All variants had slightly increased PVC of 17.1 %, compared with the control at 16.3 %, and this because of the replacement of 20 % titanium dioxide by Silfit Z 91 at equal weight instead of equal volume.



INTRODUCTION

EXPERIMENTAL

• $BaSO_4$ ppt

RESULTS

SUMMARY

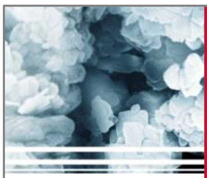
**Formulations**

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 1771-3 | 39.0 | 39.0 | 39.0 | 39.0 |
| Epikote 1003 | 18.0 | 18.0 | 18.0 | 18.0 |
| Additol P 896 | 3.0 | 3.2 | 3.2 | 3.2 |
| Titanium dioxide | 19.5 | 15.6 | 15.6 | 15.6 |
| $BaSO_4$ ppt | 20.0 | 20.0 | 13.4 | - |
| Silfit Z 91 | - | 3.9 | 7.8 | 15.7 |
| Benzoin | 0.5 | 0.5 | 0.5 | 0.5 |
| **Total** | **100.0** | **100.2** | **97.5** | **92.0** |
| | | | | |
| PVC [%] | 16.3 | 17.1 | 17.1 | 17.1 |

VM-1/0811/07.2013

*Fig. 20*



INTRODUCTION

EXPERIMENTAL

• $BaSO_4$ ppt

RESULTS

SUMMARY

## 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 1771-3 | 39.0 | 38.9 | 40.0 | 42.4 |
| Epikote 1003 | 18.0 | 18.0 | 18.5 | 19.6 |
| Additol P 896 | 3.0 | 3.2 | 3.3 | 3.5 |
| Titanium dioxide | 19.5 | 15.6 | 16.0 | 16.9 |
| $BaSO_4$ ppt | 20.0 | 20.0 | 13.7 | - |
| Silfit Z 91 | - | 3.9 | 8.0 | 17.1 |
| Benzoin | 0.5 | 0.5 | 0.5 | 0.5 |
| **Total** | **100** | **100** | **100** | **100** |
| PVC [%] | 16.3 | 17.1 | 17.1 | 17.1 |

VM-1/0811/07.2013

*Fig. 21*

## 4.2 Color values

The a* value, representing the red/green portions, for all formulations came out at an equal level of approx. - 0.7. The brightness L* was highest with the control at 95.8. For the variants, the L* value remained at a high level of 94, and this despite 20 % less titanium dioxide (*Fig. 22*). The color index b* which indicates the yellow/blue portions, increased only little. This rise will be further attenuated with the lower b* values of Silfit Z 91 from series production (*Fig. 23*).

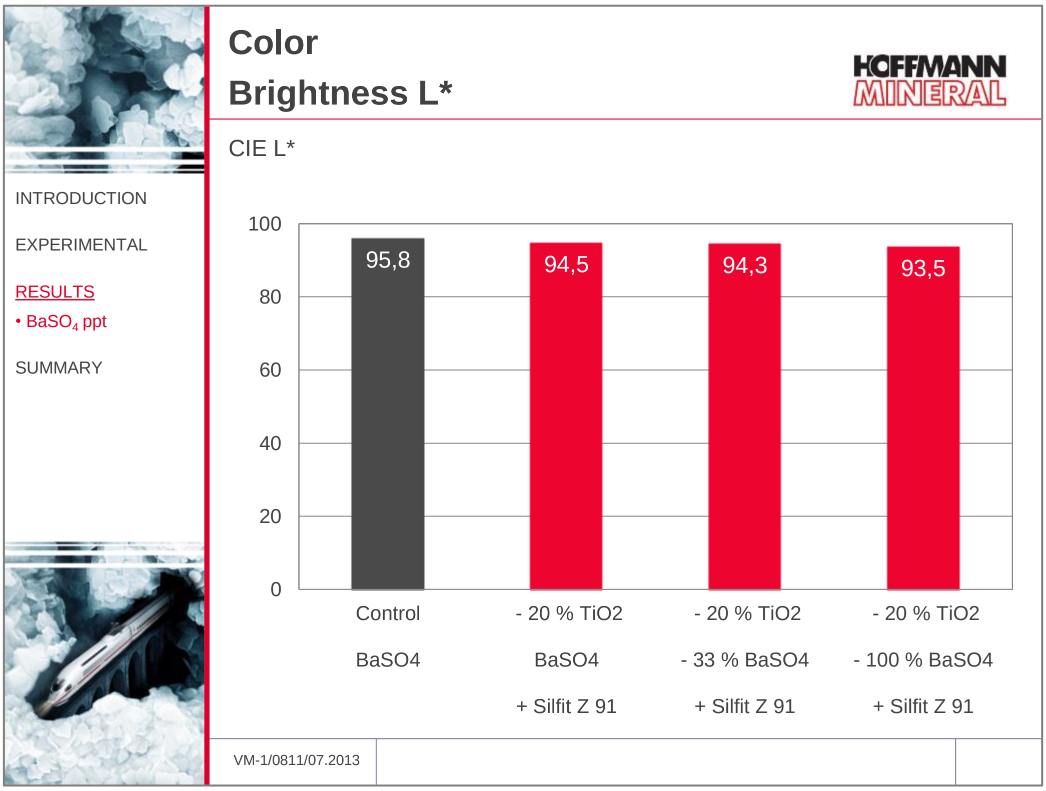

Color
Brightness L*
HOFFMANN MINERAL
CIE L*
INTRODUCTION
EXPERIMENTAL
RESULTS
• BaSO4 ppt
SUMMARY
100
80
60
40
20
0
95,8
94,5
94,3
93,5
Control BaSO4
- 20 % TiO2 BaSO4 + Silfit Z 91
- 20 % TiO2 - 33 % BaSO4 + Silfit Z 91
- 20 % TiO2 - 100 % BaSO4 + Silfit Z 91
VM-1/0811/07.2013

*Fig. 22*

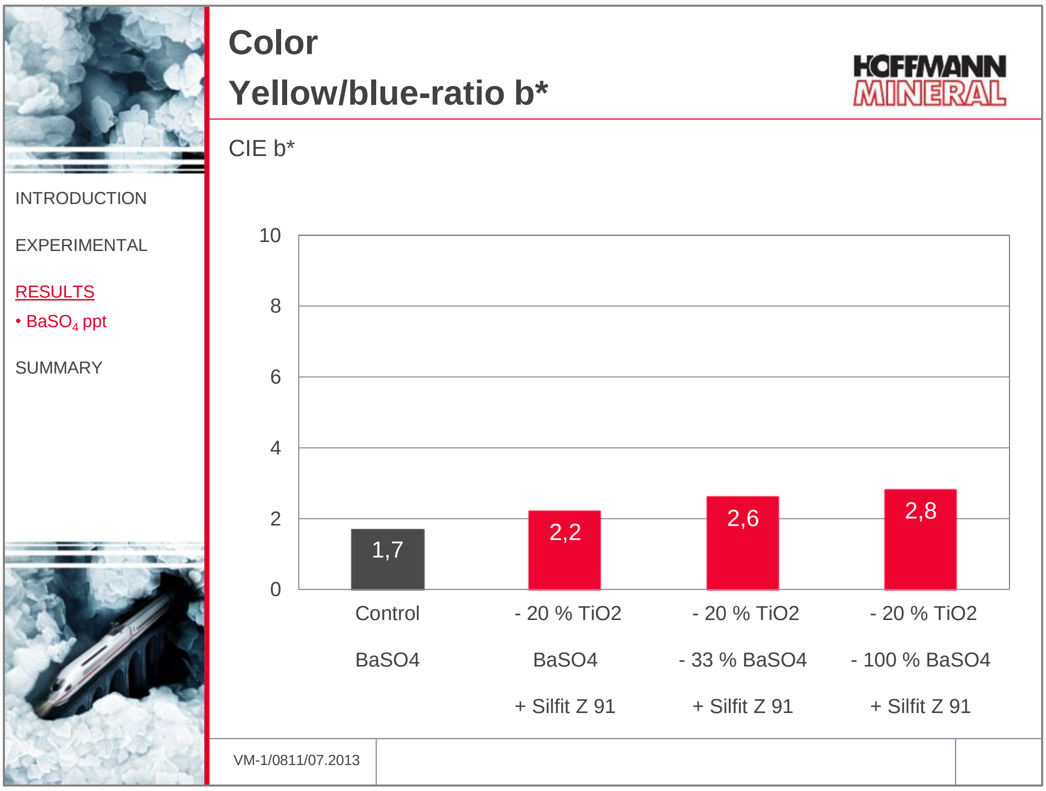

Color
Yellow/blue-ratio b*
HOFFMANN MINERAL
CIE b*
INTRODUCTION
EXPERIMENTAL
RESULTS
• BaSO4 ppt
SUMMARY
10
8
6
4
2
0
1,7
2,2
2,6
2,8
Control BaSO4
- 20 % TiO2 BaSO4 + Silfit Z 91
- 20 % TiO2 - 33 % BaSO4 + Silfit Z 91
- 20 % TiO2 - 100 % BaSO4 + Silfit Z 91
VM-1/0811/07.2013

*Fig. 23*

## 4.3 Hiding power / Opacity

*Fig. 24* shows the opacity results at a dry film thickness of approx. 70 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.

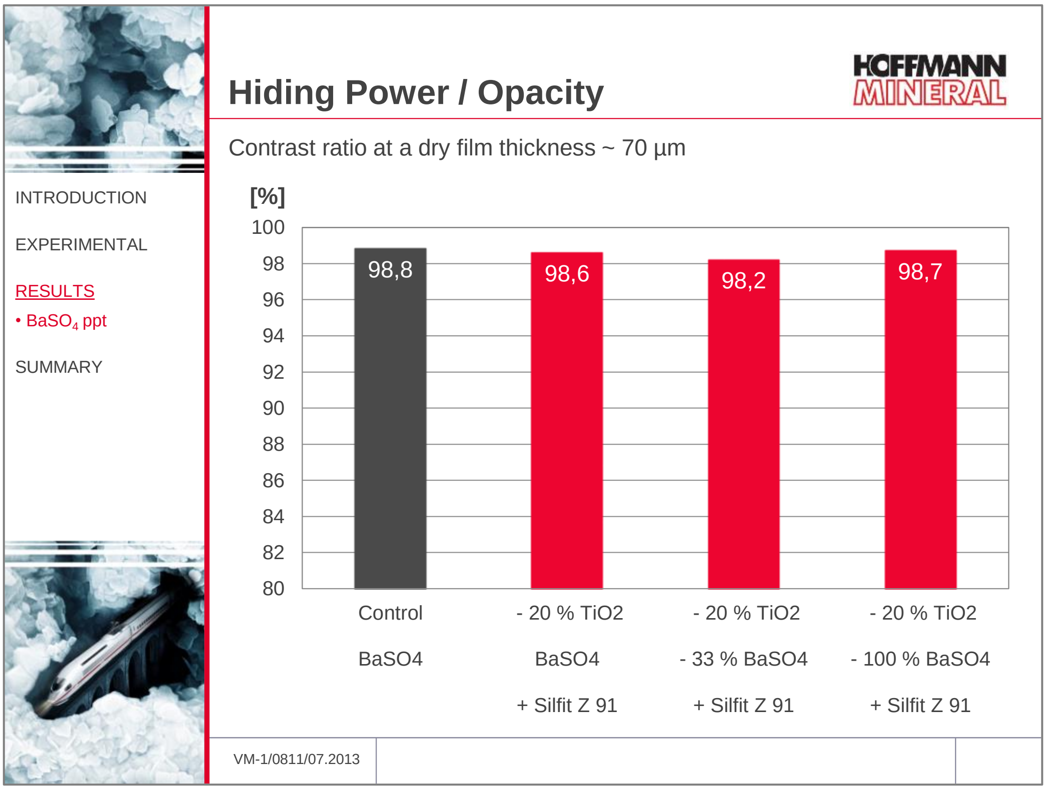



*Fig. 24*

## 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 91 units was obtained. The replacement of 20 % titanium dioxide with Silfit Z 91 gave rise to a comparably favorable result of 88 units. Even with the additional substitution of 33 % of the precipitated barium sulfate, a high gloss of 86 units was measured. Replacing the total amount of the precipitated barium sulfate by Silfit Z 91 still leaves to a 20° gloss of 78 units (*Fig. 25*).

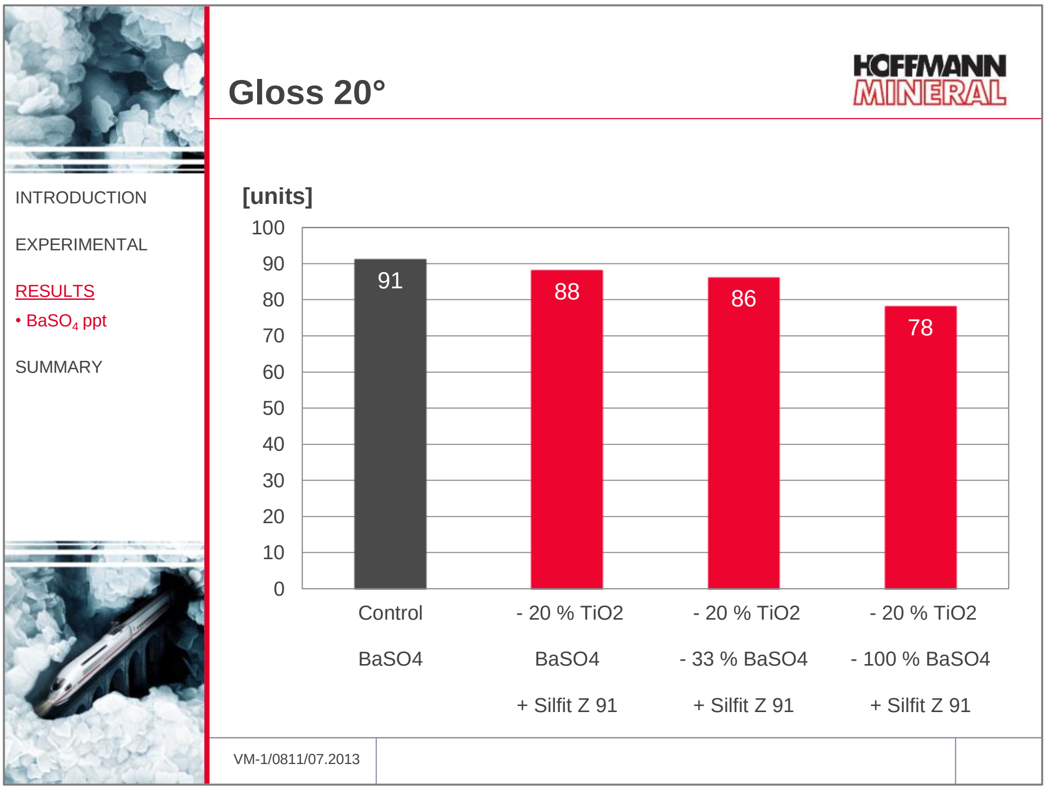



*Fig. 25*

*Fig. 26* 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 97 to 99 units.

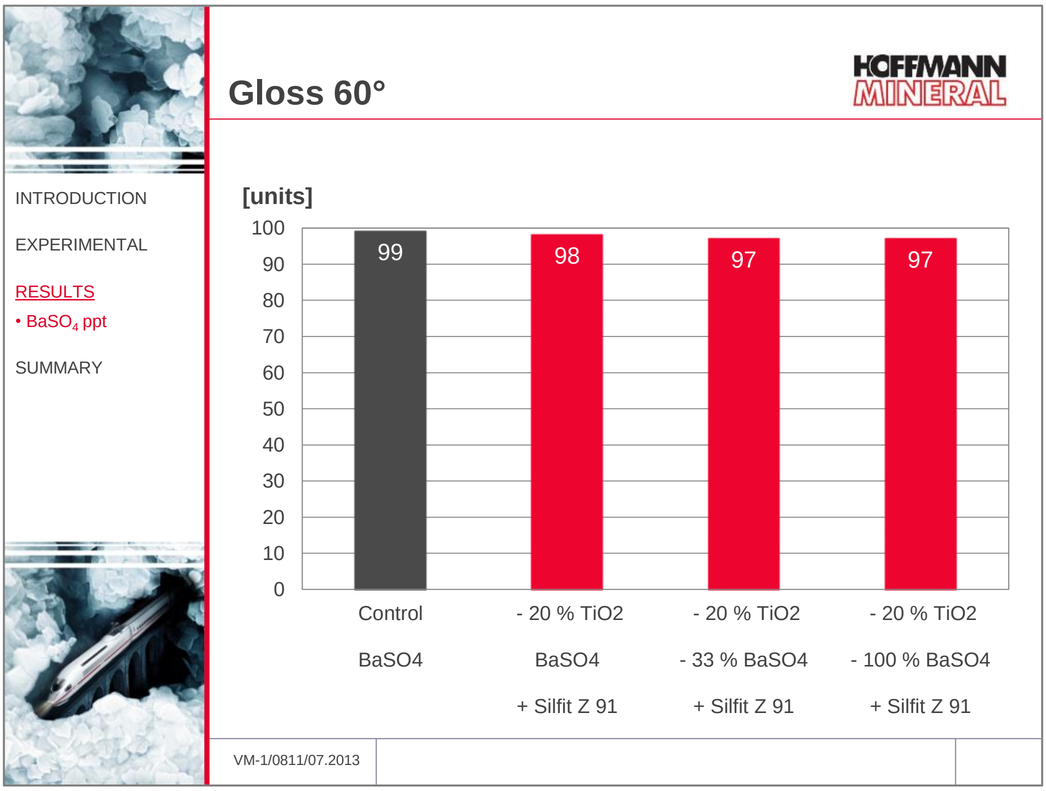



*Fig. 26*

The control offered a haze of 61 units, followed by the second variant where only titanium dioxide was replaced by Silfit Z 91, with 92 units. The substitution of 33 % precipitated barium sulfate with Silfit Z 91 led to a moderate increase of haze to 113 units. Just for comparison, the total replacement of this special precipitated barium sulfate resulted in 199 units (*Fig. 27*).

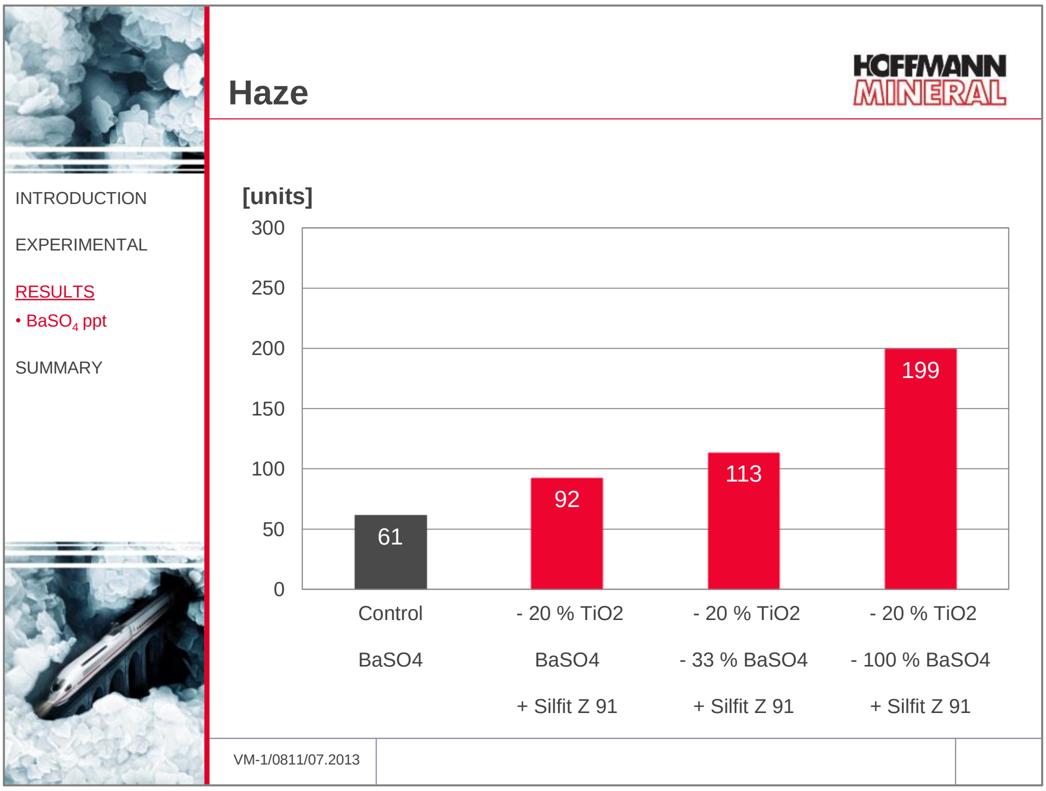



*Fig. 27*

## 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. 28*).

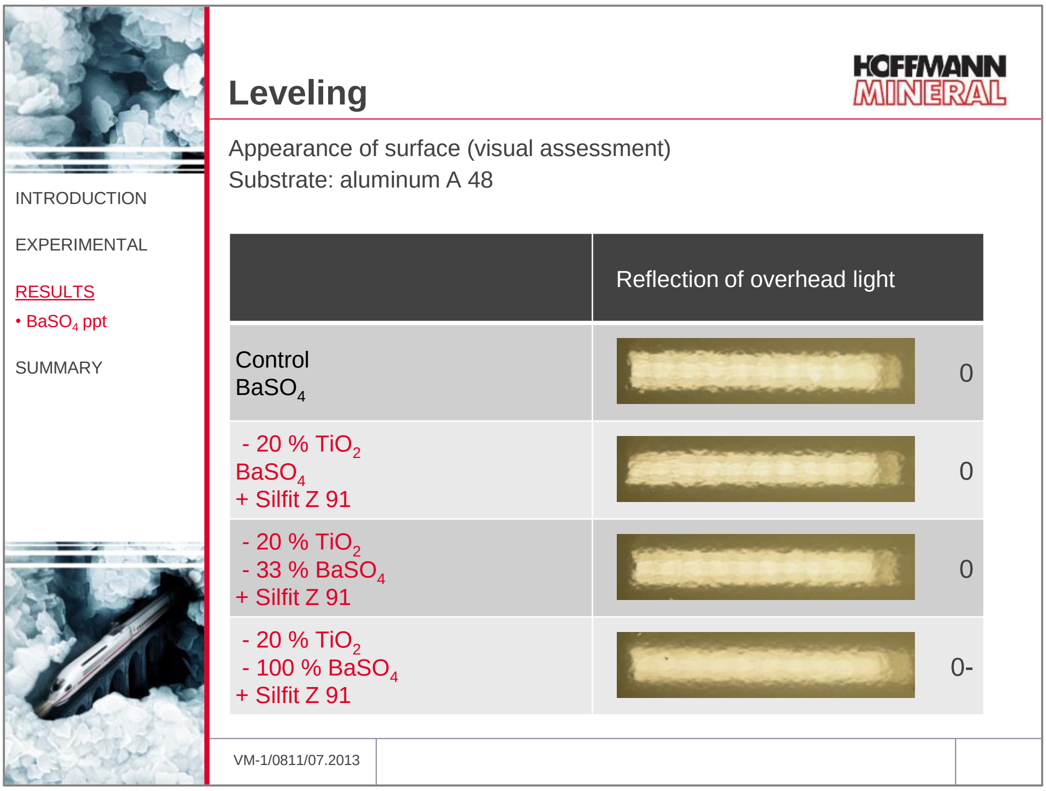
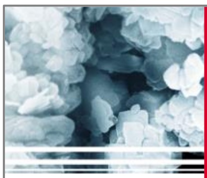



*Fig. 28*

## 4.6 Flexibility (Impact test and cupping)

The control, the pure titanium dioxide replacement and the - 33 % barium sulfate all showed a comparable flexibility in the Reverse Impact Test between 10 and 16 inch-pounds. Only when working no more with any precipitated barium sulfate, just with Silfit Z 91, an improvement up to 28 inch-pounds could be attained (*Fig. 29*)

The cupping test results for all formulations were with 6 to 7 mm comparably good, a further differentiation could not be obtained (*Fig. 29*).



INTRODUCTION

EXPERIMENTAL

RESULTS

• $BaSO_4$ ppt

SUMMARY

**Flexibility**

HOFFMANN MINERAL

Impact Test ASTM D 2794 (weight: 2 lbs); no visible cracks
Cupping Test DIN ISO 1520
Substrate: aluminum A 36

| | Reverse Impact Test [inch pounds] | Cupping Test [mm] |
|---|---|---|
| Control $BaSO_4$ | 16 | 6.8 |
| - 20 % $TiO_2$ $BaSO_4$ + Silfit Z 91 | 10 | 7.0 |
| - 20 % $TiO_2$ - 33 % $BaSO_4$ + Silfit Z 91 | 14 | 6.6 |
| - 20 % $TiO_2$ - 100 % $BaSO_4$ + Silfit Z 91 | 28 | 6.4 |

VM-1/0811/07.2013

*Fig. 29*

## 4.7 Mechanical resistance (scribe/scratch resistance)

With 16 Newton, Silfit Z 91 showed a somewhat better scribe resistance compared with the control with full titanium dioxide content at 15 Newton. Still better scratch resistance at 18 Newton could be obtained by replacing 33 % of the precipitated barium sulfate with Silfit Z 91 (*Fig. 30*).

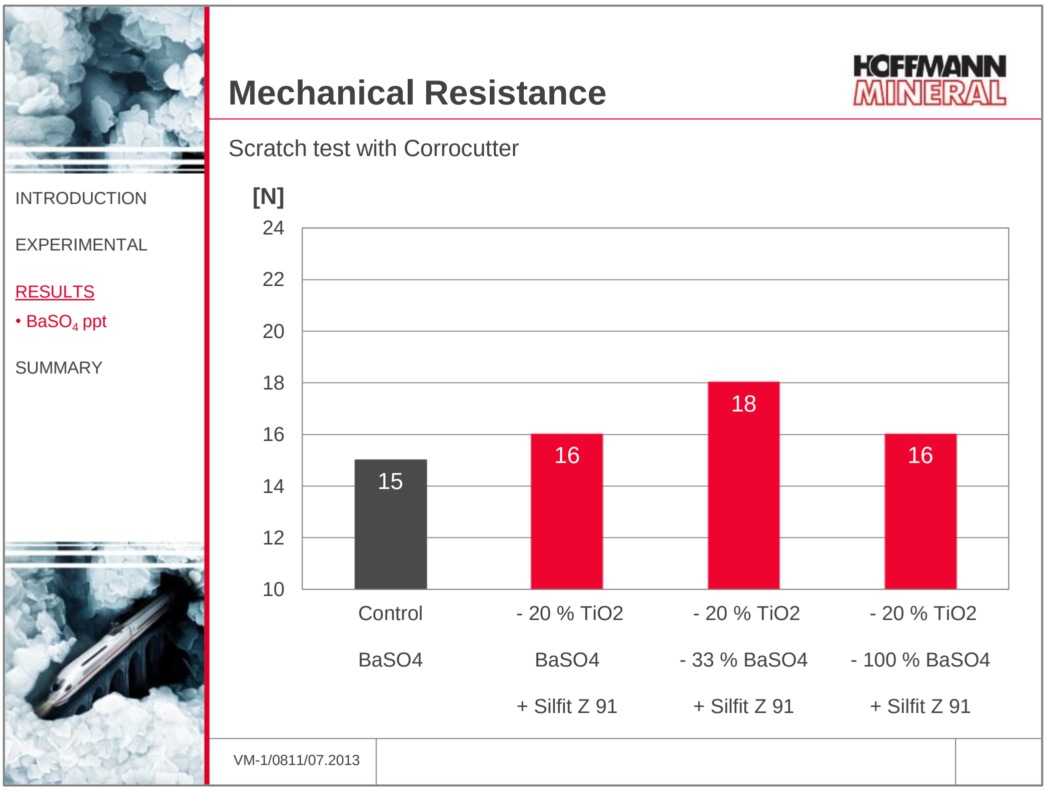



*Fig. 30*

## 4.8 Density and Spreading rate

The densities of the formulations are shown in *Fig. 31*. The control exhibited the highest level of 1.67 g/cm³, caused by the density of the pure precipitated barium sulfate at 4.4 and titanium dioxide at 4.1. The replacement of 20 % titanium dioxide at equal weight, i. e. 3.9 pbw, by Silfit Z 91 with a density of 2.6 hardly affected the total density at all. However, when substituting 33 or 100 % of the precipitated barium sulfate at equal volume with Silfit Z 91, the density went down to 1.52 g/cm³. As discussed in the following, this results in positive effects on the spreading rate.

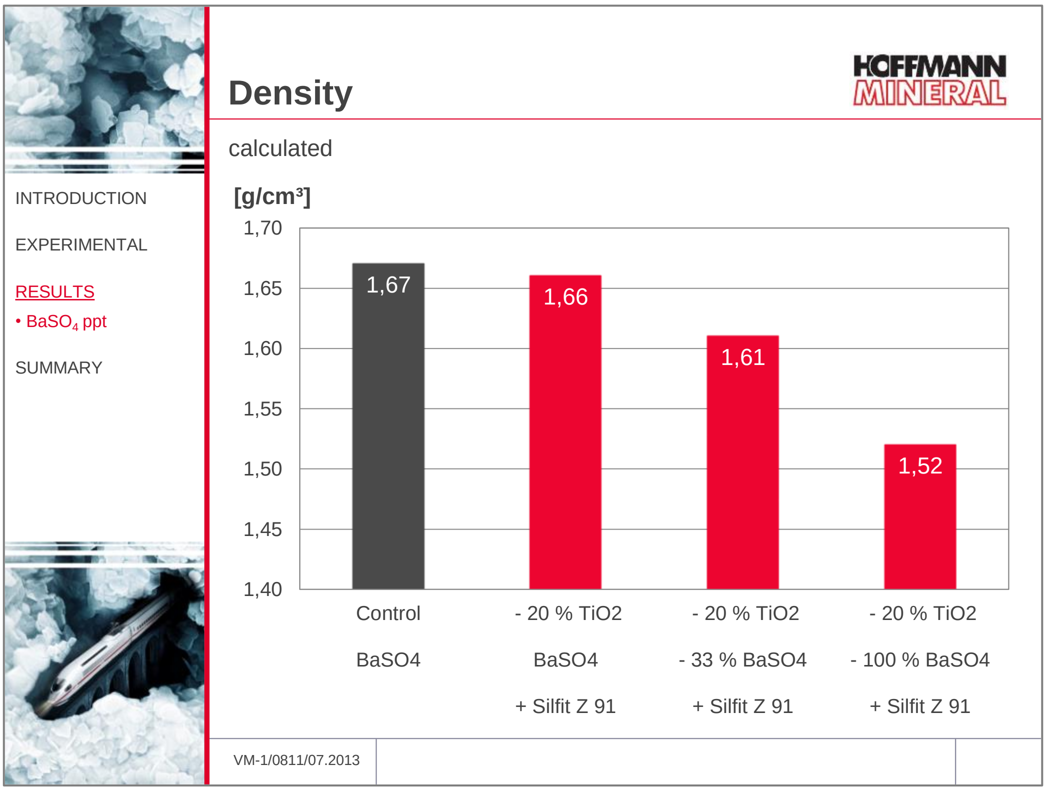



*Fig. 31*

*Fig. 32* 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!

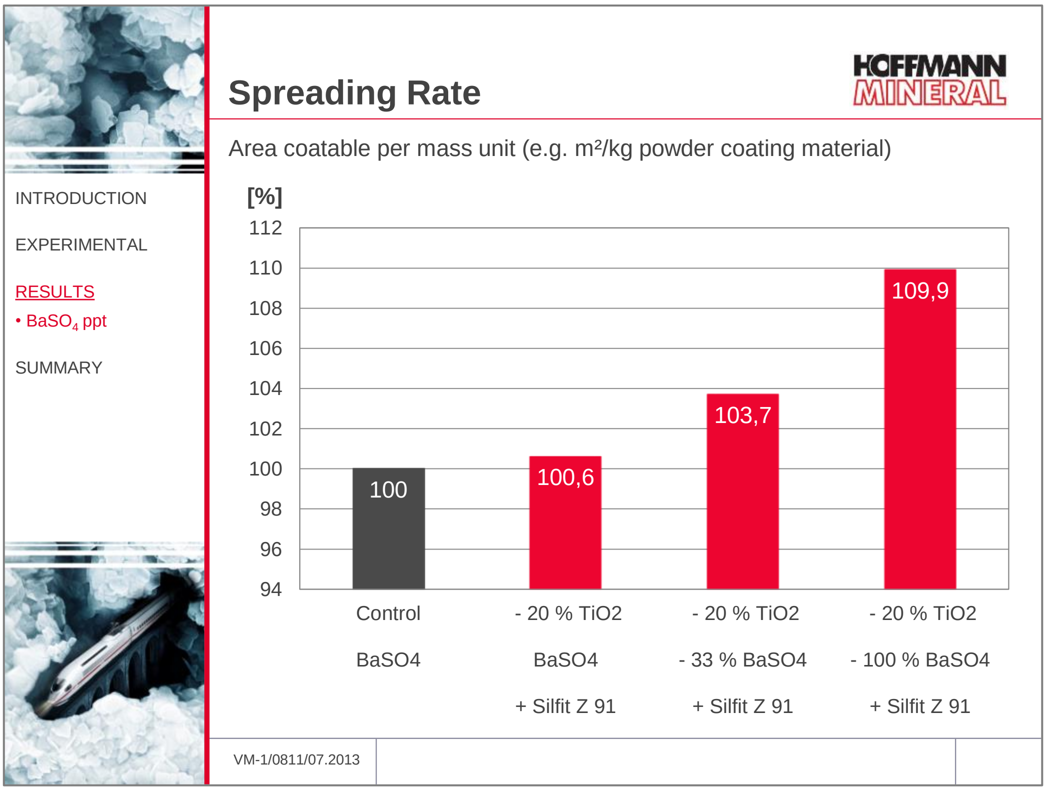


*Fig. 32*

## 4.9 Cost index

*Fig. 33* summarizes the weight-related costs based on prices in Germany during the year 2011. The price for titanium dioxide was taken as € 2.65 per kg. Via the replacement of 20 % titanium dioxide with Silfit Z 91, cost savings of 4 % could be achieved, through the additional substitution of 33 % of the precipitated barium sulfate almost 3 %. Replacing the total amount of precipitated barium sulfate by Silfit Z 91 gave rise to a cost increase of 0.5 %, which however is more than compensated by the almost 10 % higher spreading rate

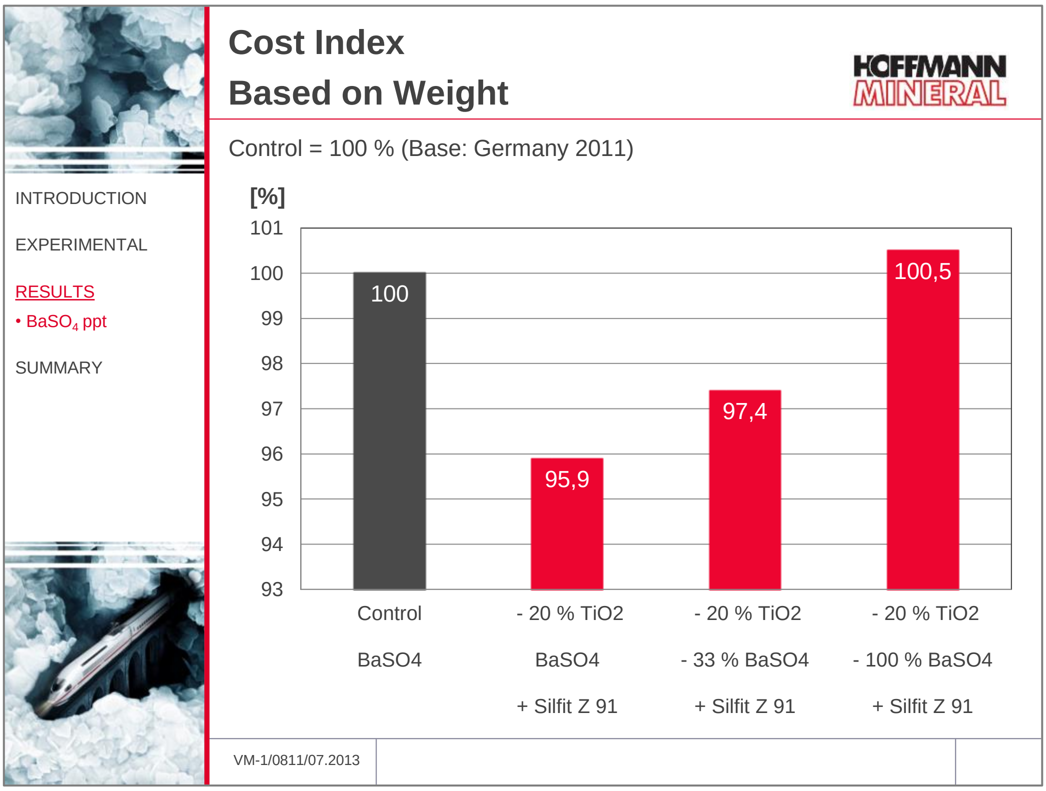



*Fig. 33*

If the cost index is calculated based on volume, all formulations with Silfit Z 91 gave rise to marked cost savings of 5 to 8 % (*Fig. 34*).

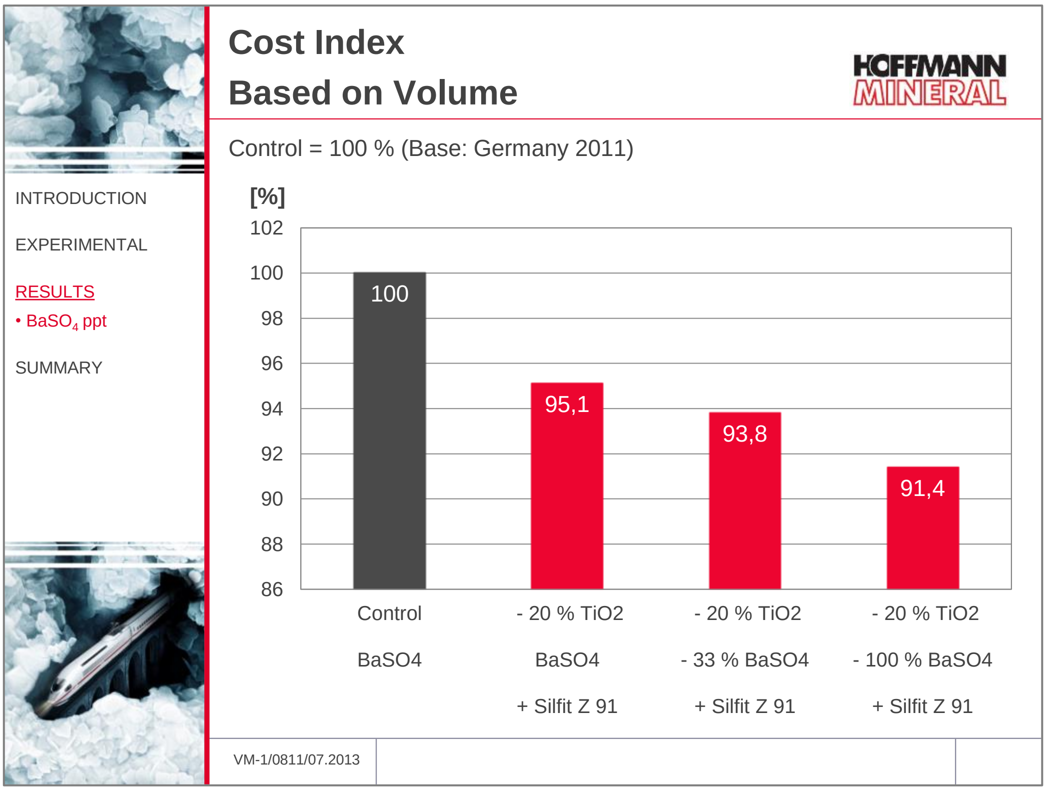



*Fig. 34*

## 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:

- ✓ slightly increased haze along with comparable optical properties and flexibility
- ✓ slightly improved scribe / scratch 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:

- ✓ improved scribe / scratch resistance
- ✓ 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 in hiding power. The scribe / scratch resistance came out better, and the costs were decreased.

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

The additional partial replacement at equal volume of the special precipitated barium sulfate (33 %) by Silfit Z 91 further optimized the scribe / scratch resistance, increased the spreading rate and offered potential for cost savings.