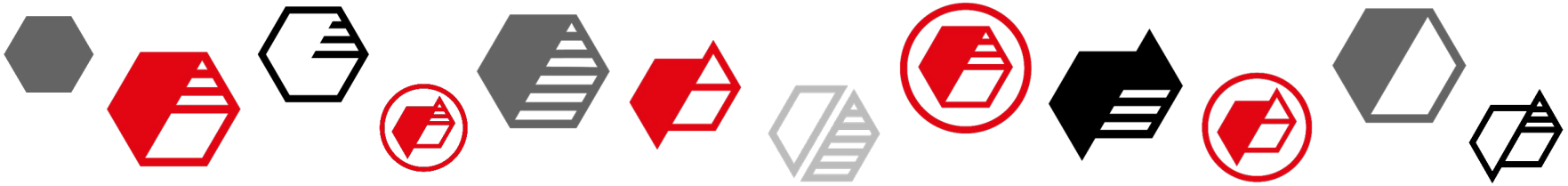

Calcined Neuburg Siliceous Earth in powder coatings (polyester, TGIC-based, white)

Reduced titanium dioxide content – What can Calcined Neuburg Siliceous Earth offer?

Author: Susanne Reiter

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We supply material for good ideas

Contents

- Introduction
- Experimental
- Results
  - Color
  - Hiding power / opacity
  - Gloss and Haze
  - Leveling
  - Artificial weathering (QUV-A test)
  - Corrosion resistance (acetic salt spray test and humidity test)
  - Density / spreading rate
  - Cost index
- Summary
- Appendix (Abrasivity)

Status Quo

Earlier studies exhibit Neuburg Siliceous Earth as suitable filler in powder coatings. The focus was on replacing the common used filler barium sulfate with grades of Neuburg Siliceous Earth. Positive effects were found: optical and mechanical properties were maintained or even improved.

Studies in other coatings like Coil Coating Top Coat proved Calcined Neuburg Siliceous Earth´s potential for partially replacing titanium dioxide.

Titanium dioxide prices have risen considerably and continue to do so. Thus, the coating producers are looking for measures to compensate for these cost increases.

Objective

Thus the question comes up if Calcined Neuburg Siliceous Earth is capable of partially substituting titanium dioxide while maintaining optical properties (particularly opacity) as well as corrosion resistance.

The main filler in the formulation:

- natural barium sulfate

as example for low cost formulation with standard requirements

or

- precipitated barium sulfate grade (ppt)

special grade with higher quality and optical properties

In addition, cost aspects were also taken into account.

Filler Characteristics

		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

Filler Characteristics

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

What is Neuburg Siliceous Earth?

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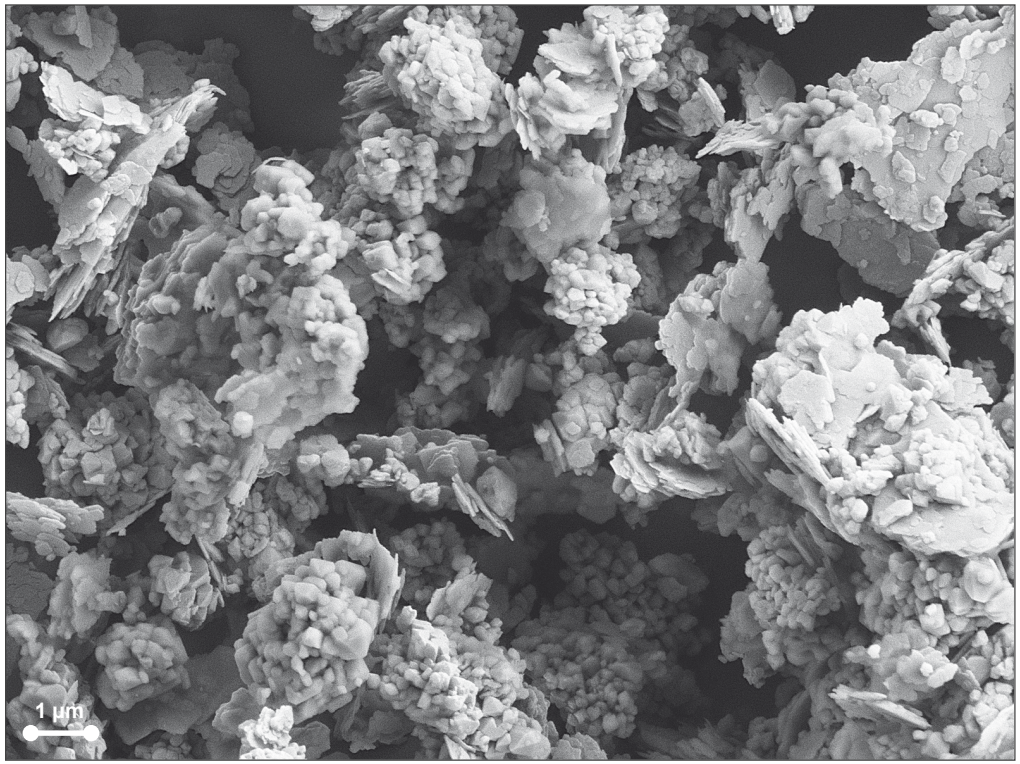

A natural combination of corpuscular Neuburg silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods.

The silica portion exhibits a round grain shape and consists of aggregated primary particles of about 200 nm diameter.

Morphology of Neuburg Siliceous Earth

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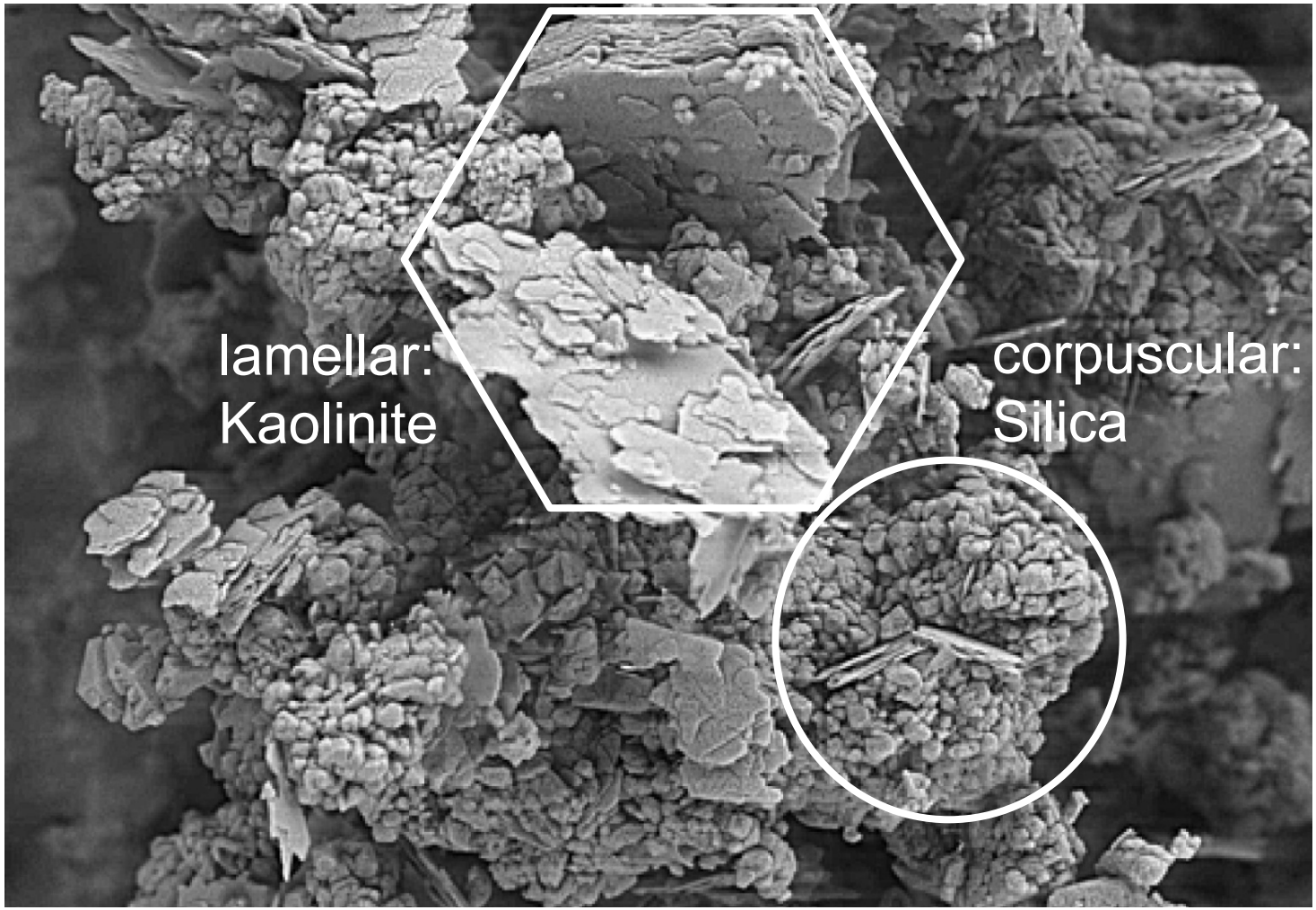
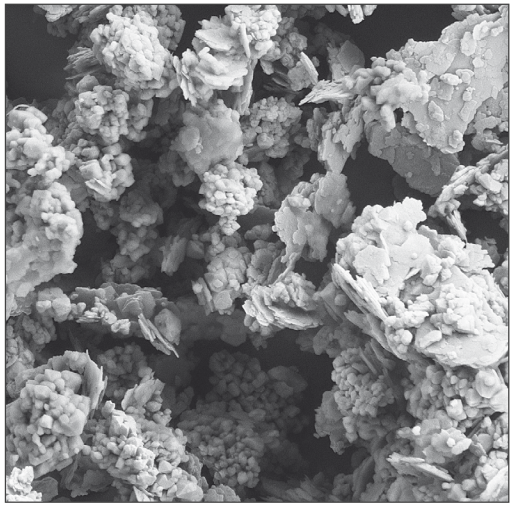
Magnification 10.000x



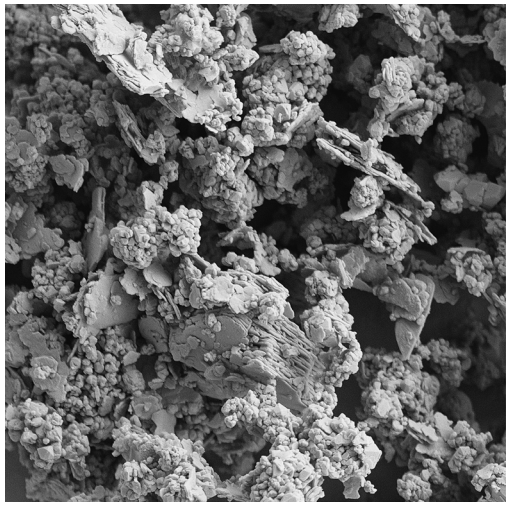
Calcined Neuburg Siliceous Earth

A downstream thermal process leads to the calcined products SILFIT and AKTIFIT, based on SILLITIN Z 86.



Neuburg Siliceous Earth

Calcination Process



Calcined Neuburg Siliceous Earth

Additional application benefits, as well as the removing of crystal water included in the kaolinite. The silica part remains inert.

Base Formulation

		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	

What kind of barium sulfate do you use?

- Barium sulfate natural 

or

- Barium sulfate precipitated (ppt) 

Formulations

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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

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
$BaSO_4$ 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

Brightness

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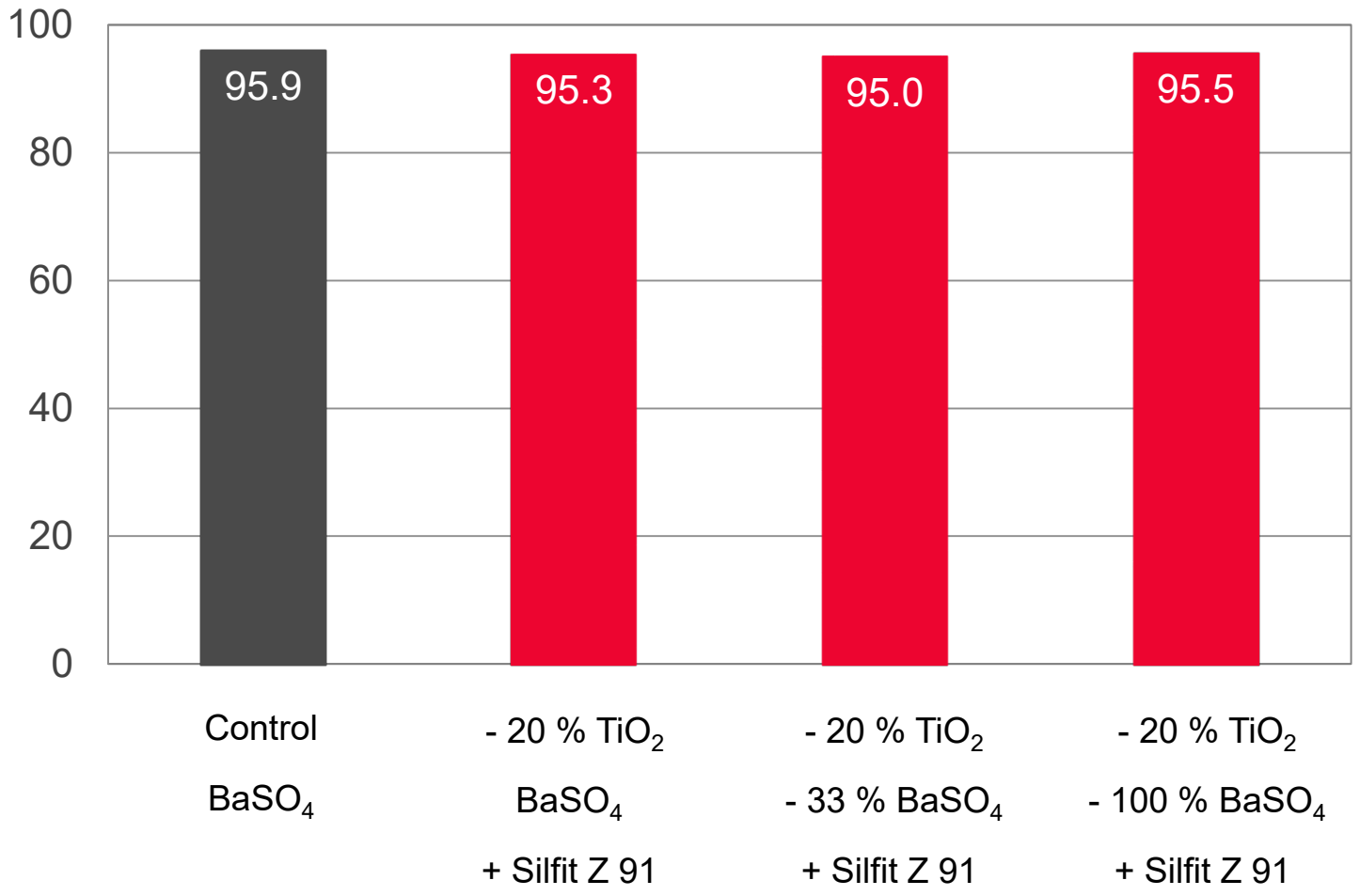
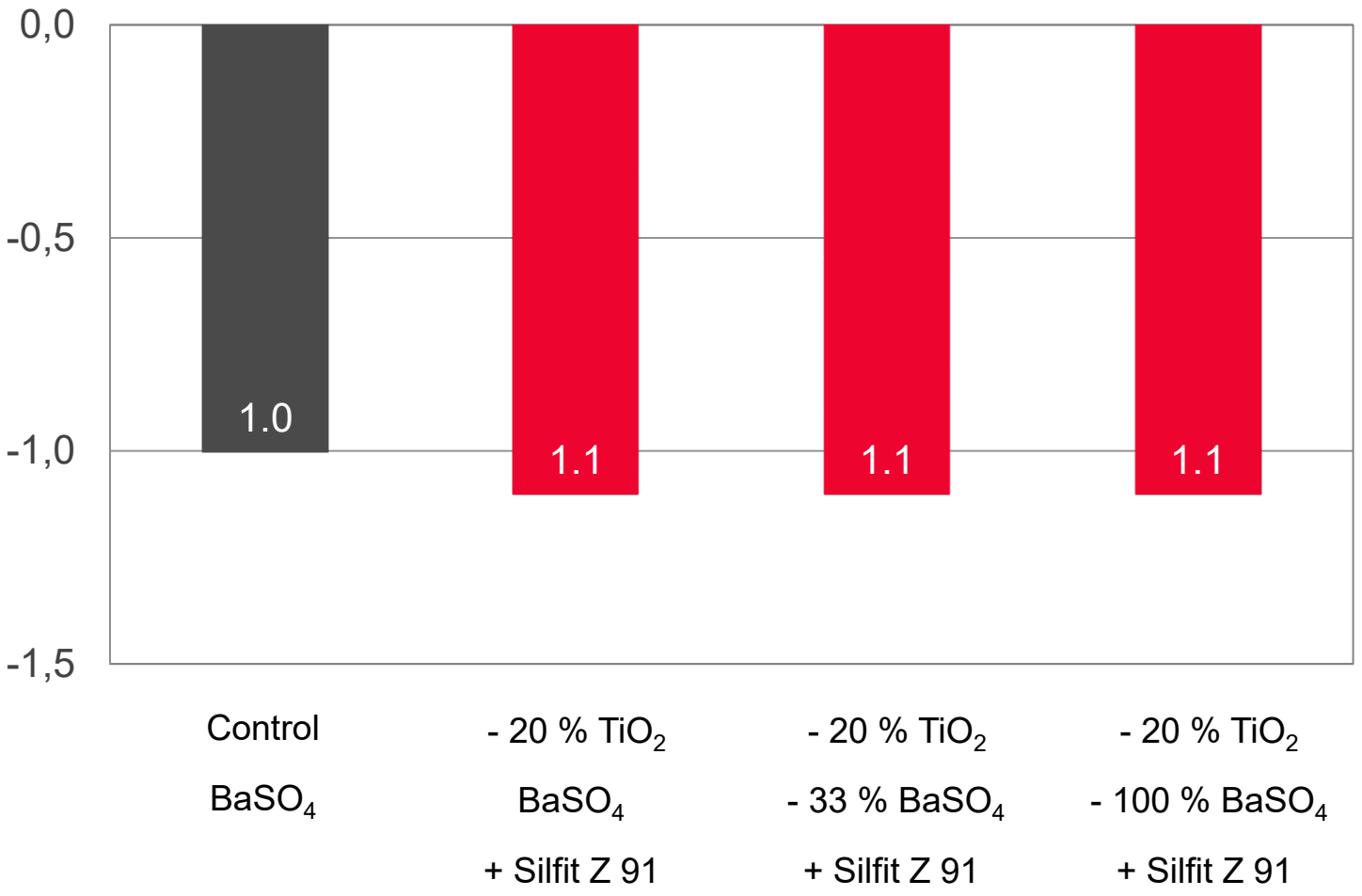
CIE L*



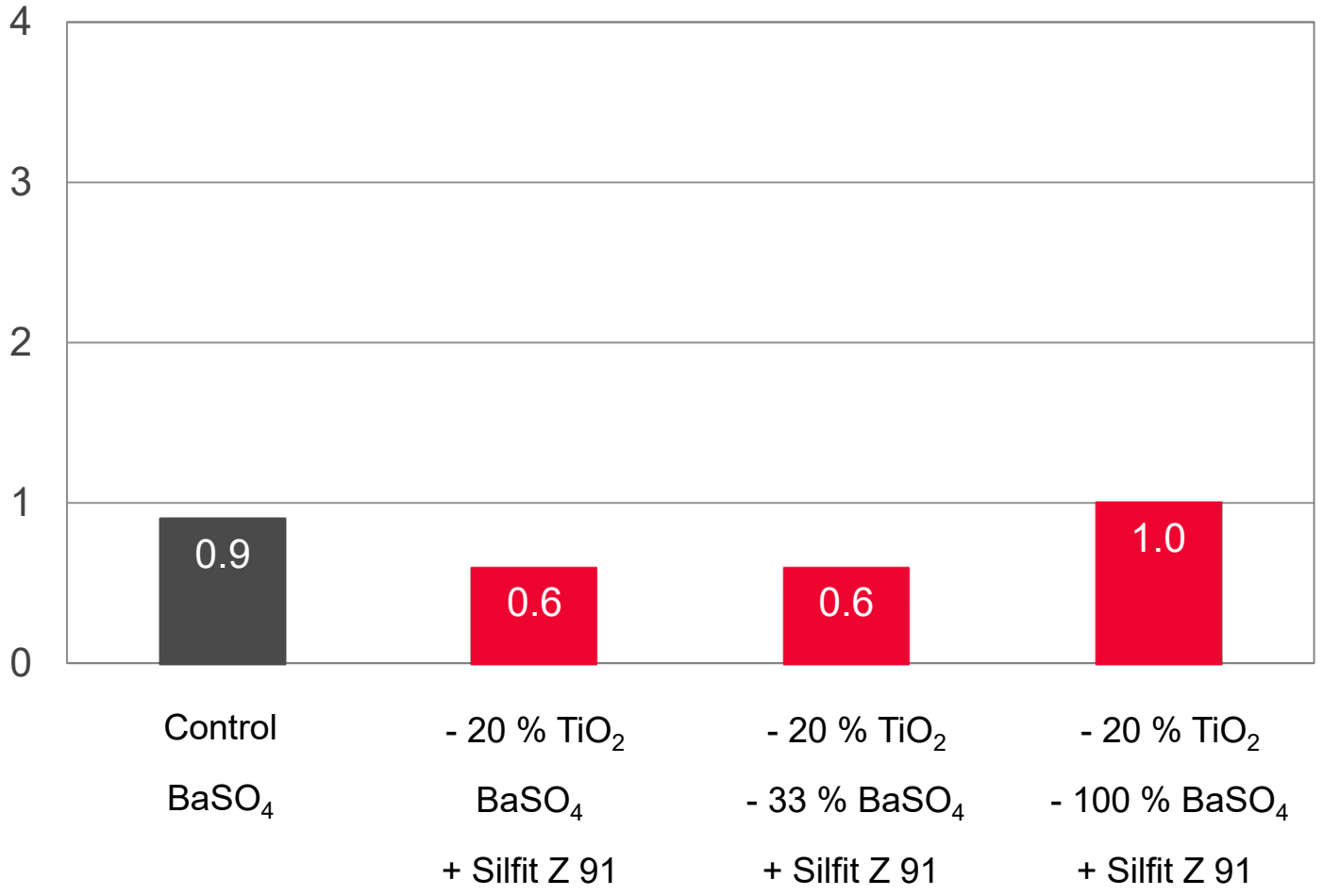
Red/green-ratio

CIE a*



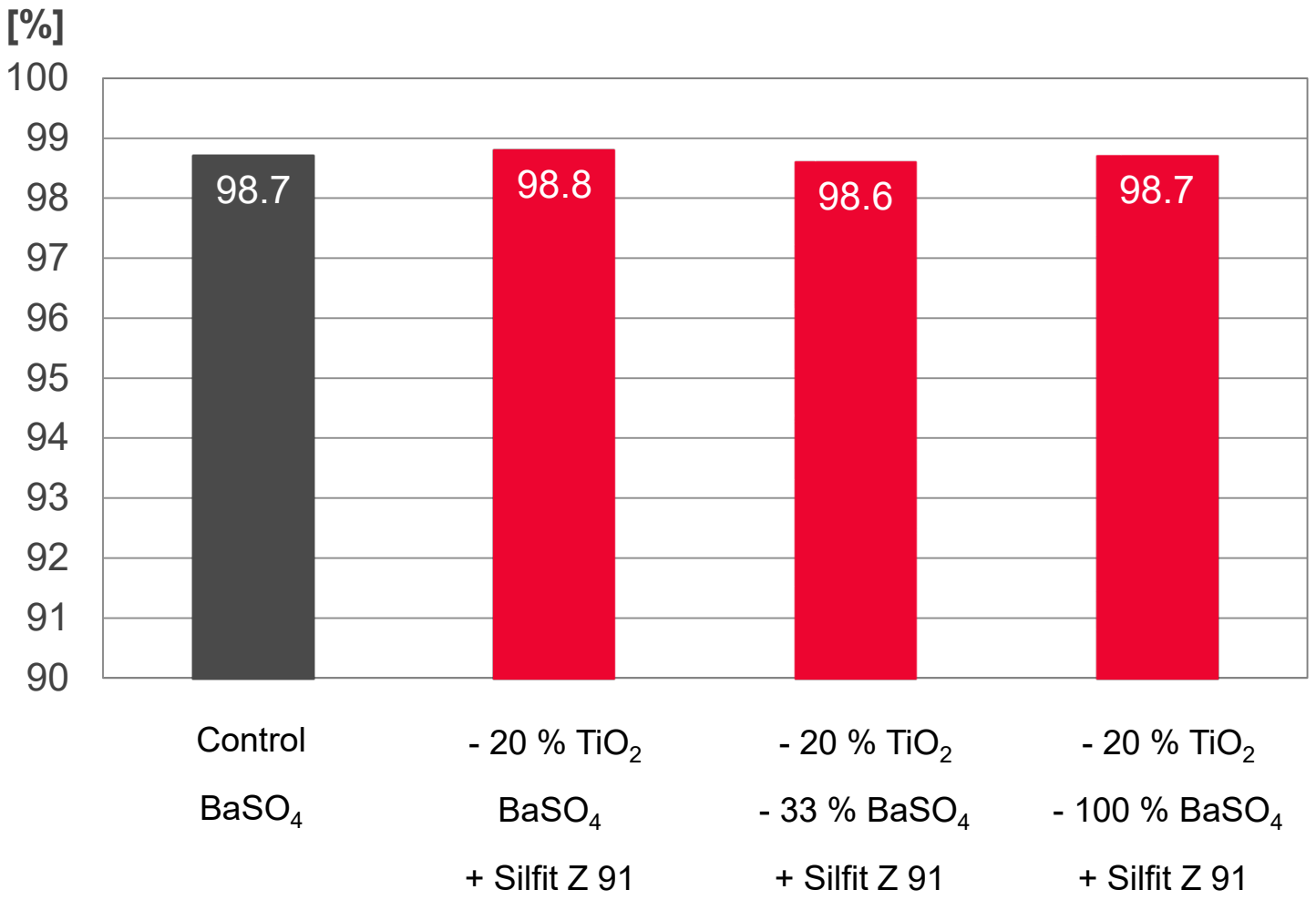
Yellow/blue-ratio

CIE b*

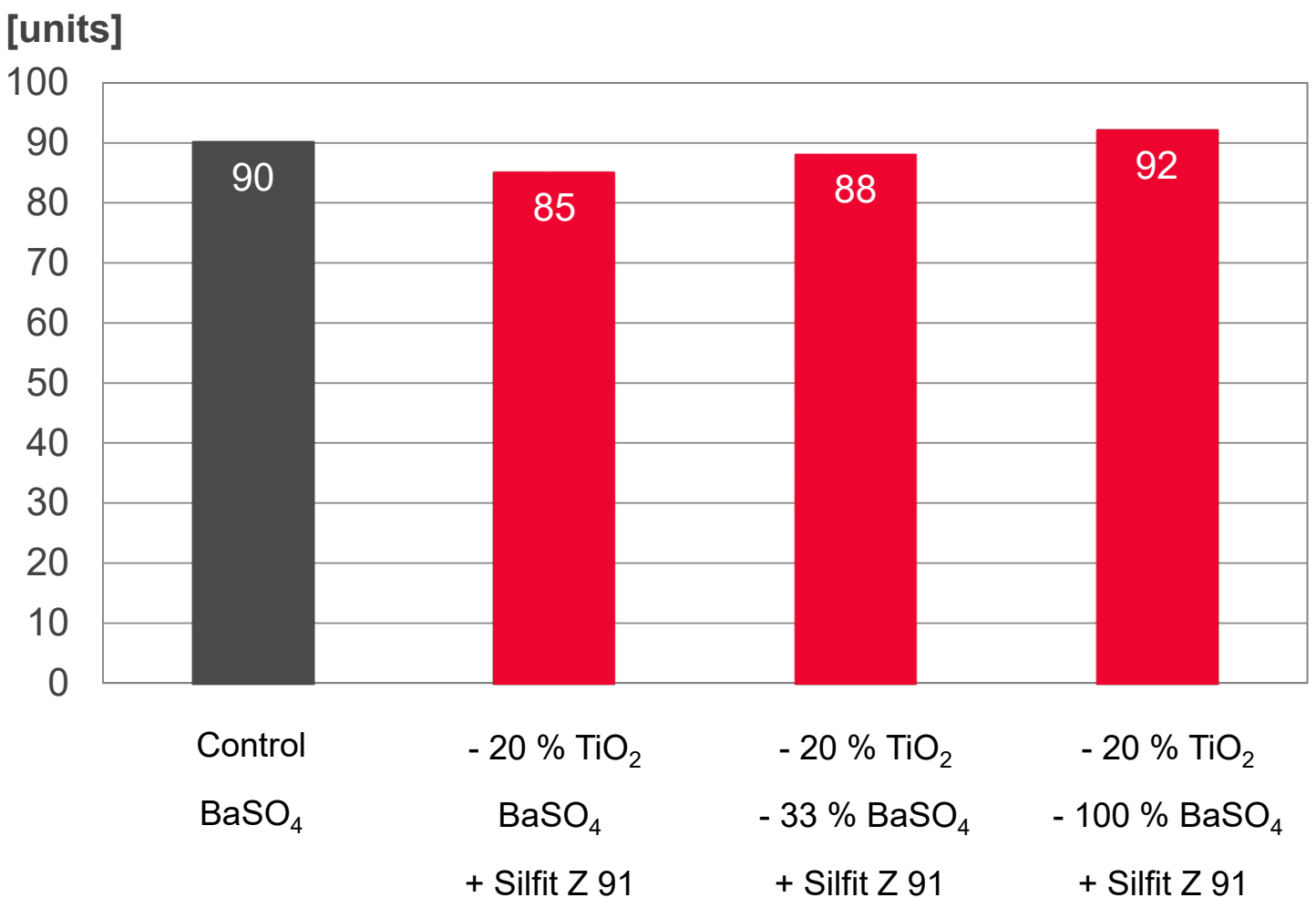


Hiding Power

Contrast ratio at a dry film thickness ~ 80 µm



Gloss 60°



Gloss 20°

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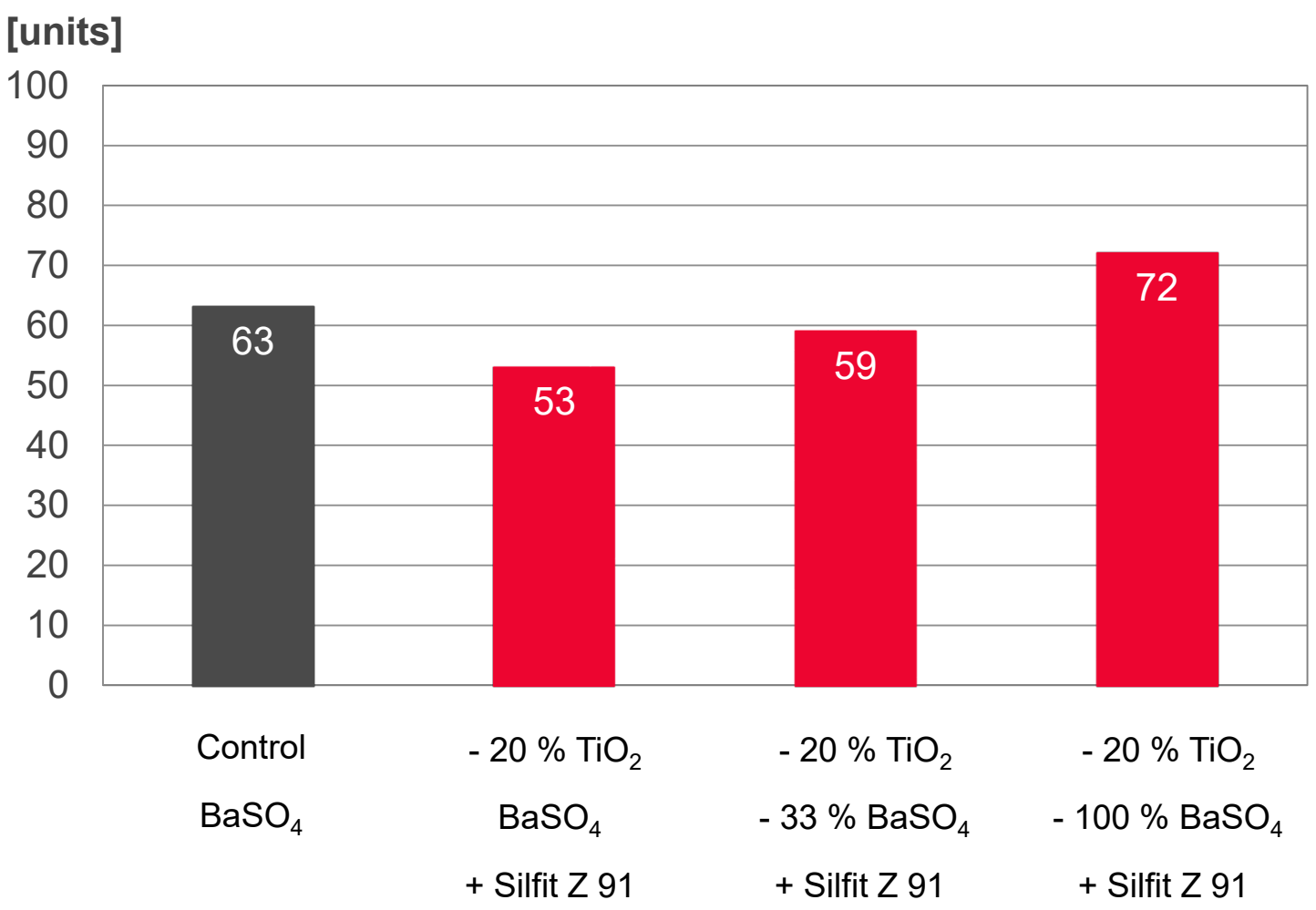
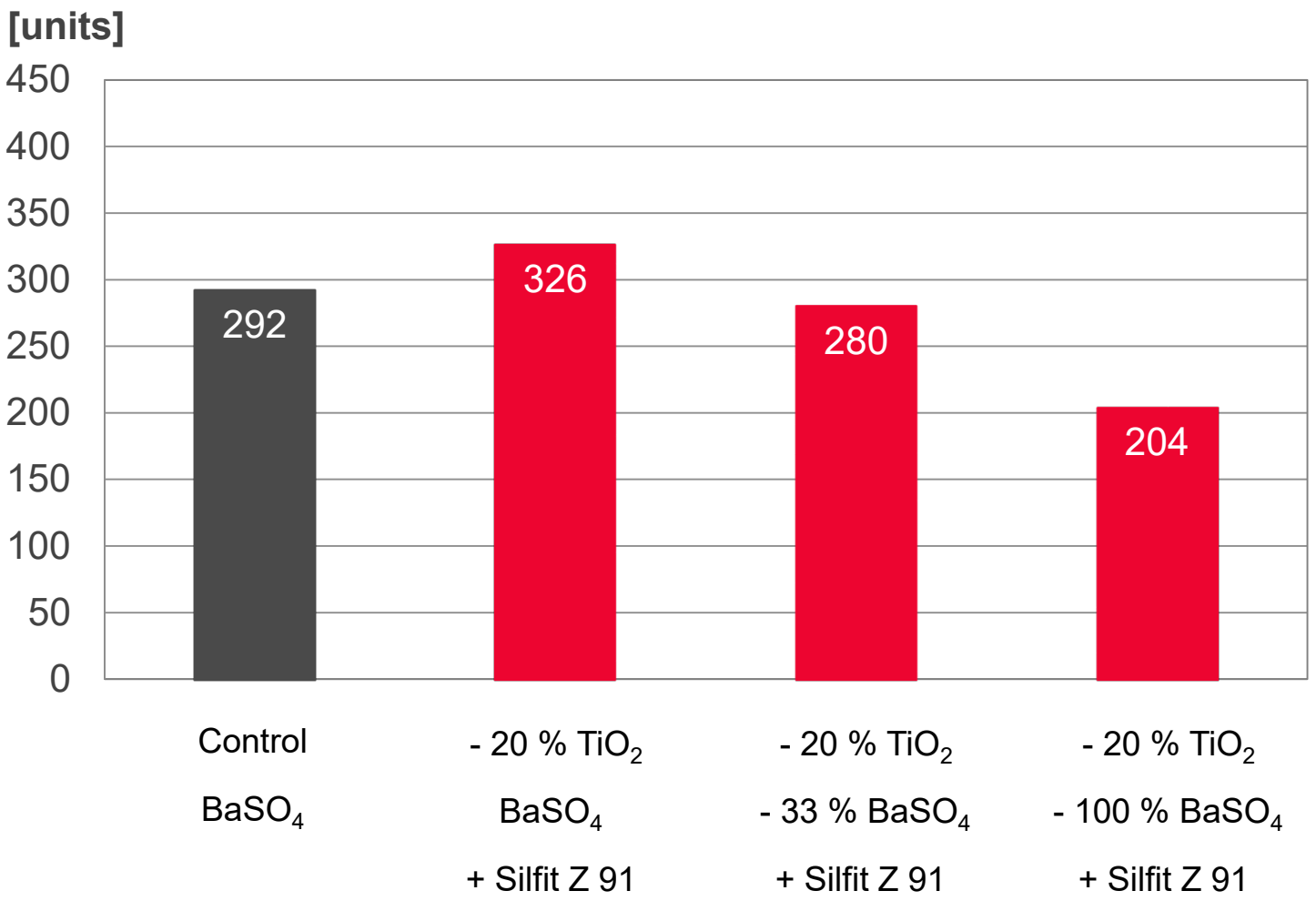


Haze



Leveling

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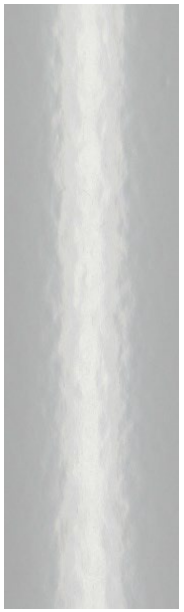
Appearance of surface (visual assessment)

Reflection of overhead light

+

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

Artificial Weathering 1000h
Customer Feedback

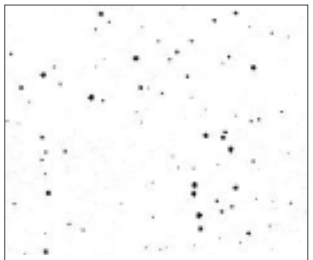
HOFFMANN MINERAL

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.

Acetic Salt Spray 2000 h Blistering

 **Beginning of blistering** **~ 5 % of the surface area 3 – 3 (S2)**	**No blistering**	**No blistering**	**No blistering**
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

Acetic Salt Spray 2000 h
Delamination at Scribe

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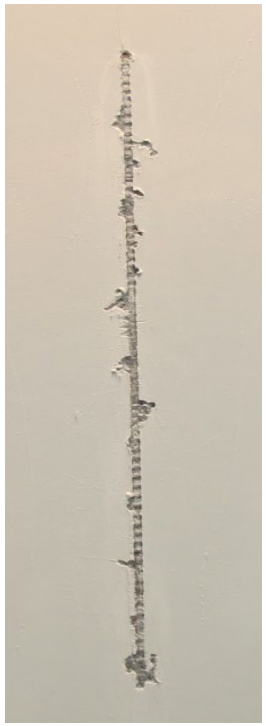
Control

$BaSO_4$

0.7 mm



- 20 % TiO_2

$BaSO_4$

+ Silfit Z 91

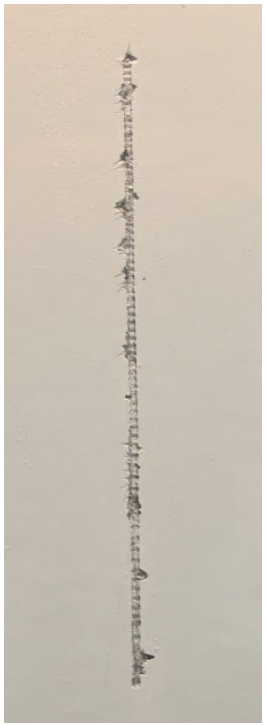
0.1 mm

- 20 % TiO_2

- 33 % $BaSO_4$

+ Silfit Z 91

0.1 mm



- 20 % TiO_2

- 100 % $BaSO_4$

+ Silfit Z 91

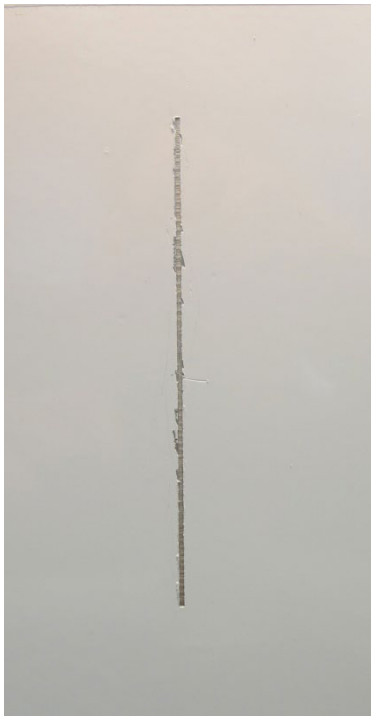
0.1 mm

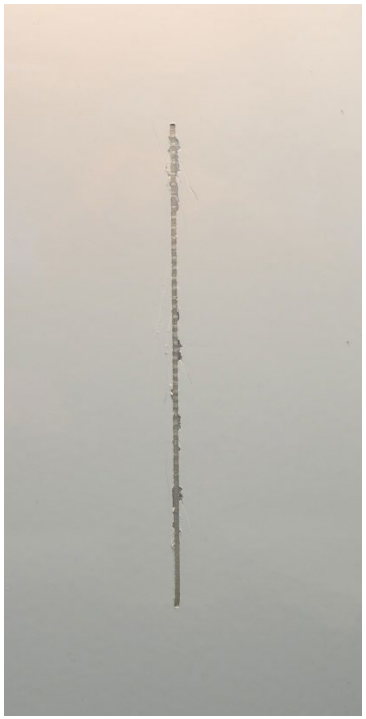
Humidity Test 2000 h Blistering

 **Beginning of blistering** **~ 30 % of the surface area 2 – 2 (S2)**	**No blistering**	**No blistering**	**No blistering**
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

Humidity Test 2000 h Delamination at Scribe

HOFFMANN MINERAL

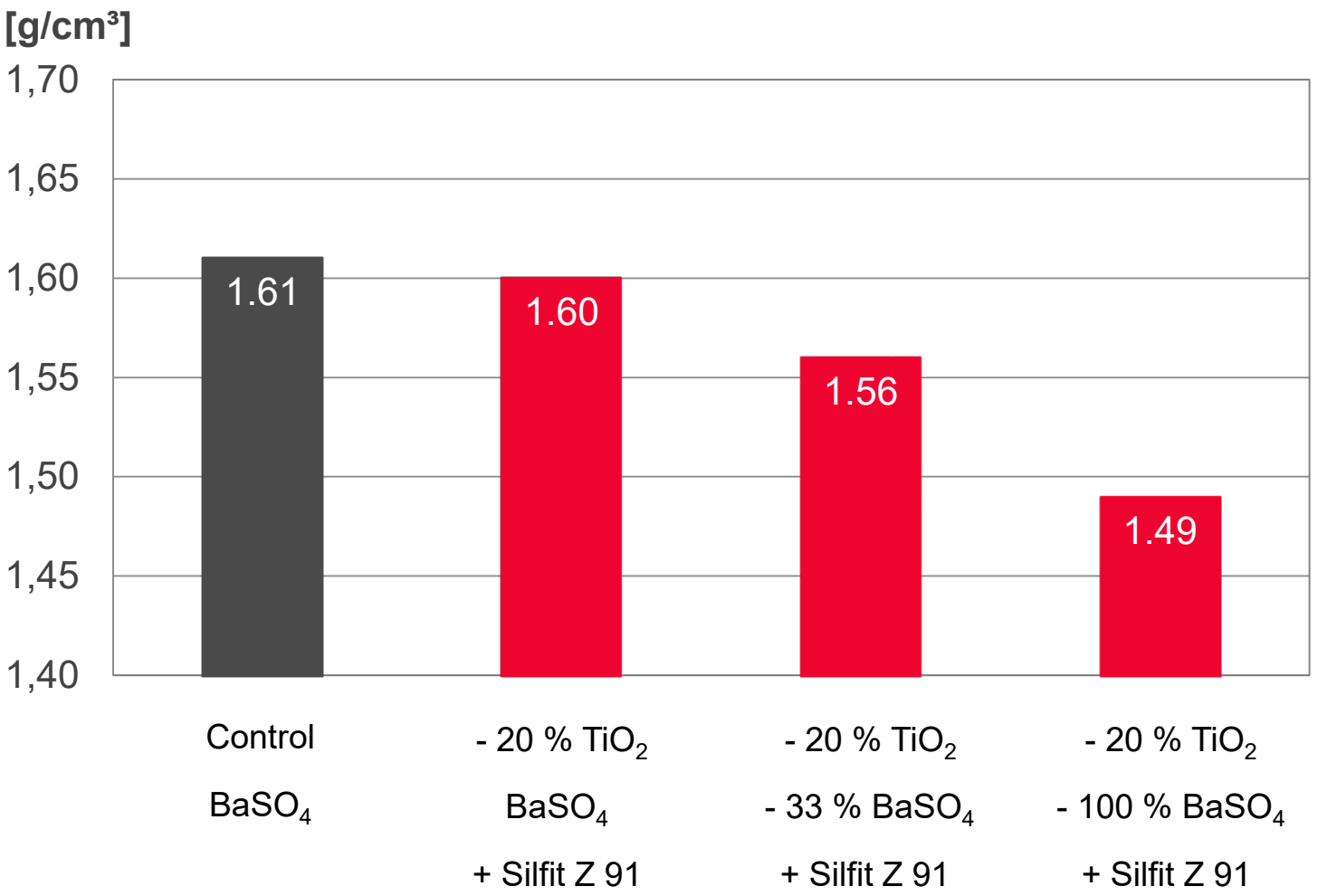




Control $BaSO_4$	- 20 % TiO_2 $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 33 % $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 100 % $BaSO_4$ + Silfit Z 91
7 mm	**0 mm**	**0 mm**	**0 mm**

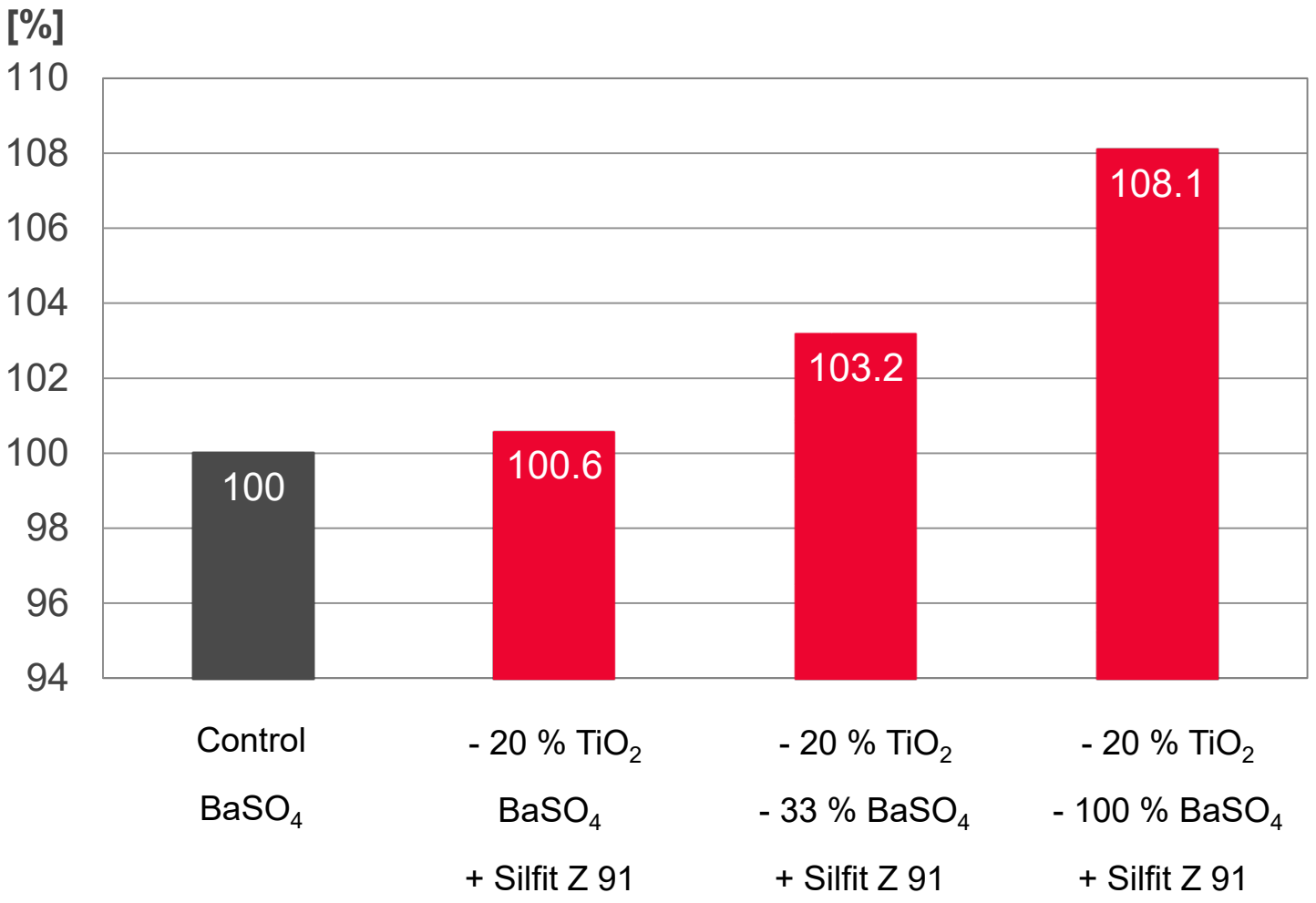
Density

Calculated



Spreading Rate

Area coatable per mass unit (e.g. m²/kg powder coating material)



Cost Index
Based on Weight

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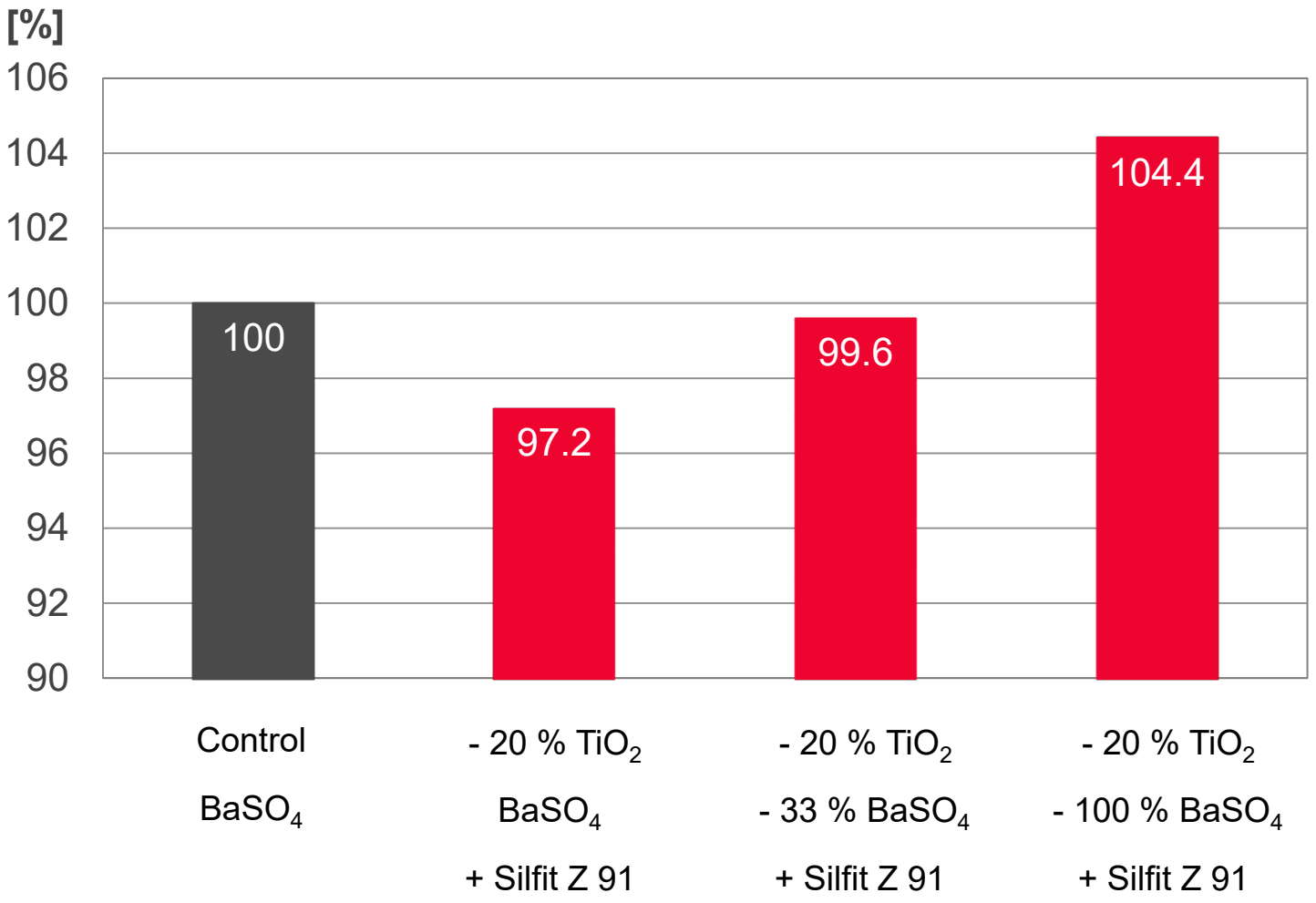
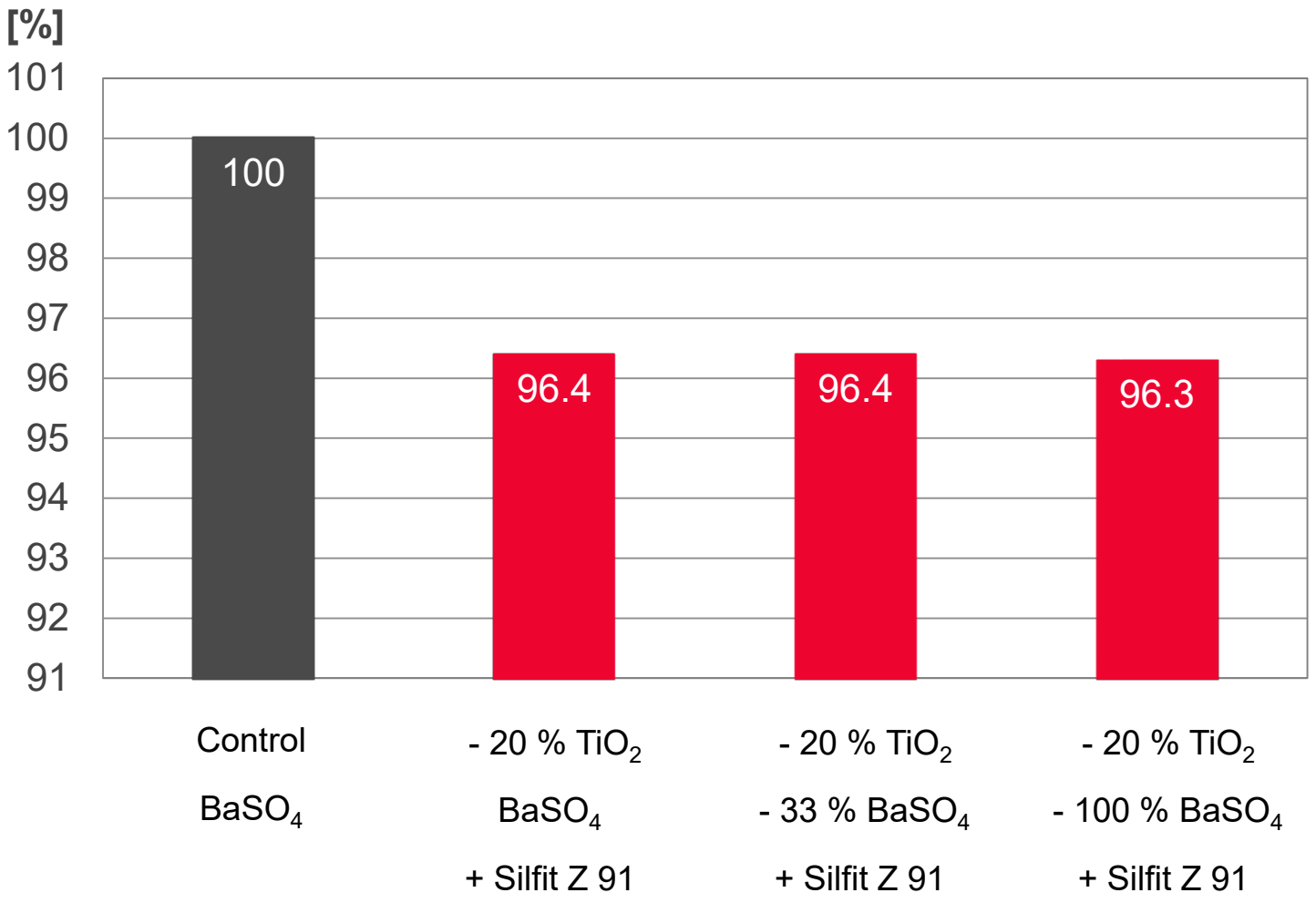
Control = 100 % (Base: Germany 2014)



Cost Index
Based on Volume

HOFFMANN MINERAL

Control = 100 % (Base: Germany 2014)



Summary

Replacement of 20 % titanium dioxide at equal weight with Silfit Z 91 gave rise to the following effects :

- similar optical properties
- excellent weatherability (even up to 50 % titanium dioxide substitution)

+ improved corrosion resistance

+ cost reduction potential

Summary

Additional substitution of the natural barium sulfate by Silfit Z 91 improved furthermore:

+ higher gloss

+ lower haze

+ better leveling

+ higher spreading rate (lower density of powder coating)

+ cost reduction potential

Thank you very much for your attention!

For more information please visit our website:

www.hoffmann-mineral.com

HOFFMANN MINERAL GmbH • P.O.Box 14 60 • D-86619 Neuburg (Donau)
Phone +49 (0) 8431-53-0 • Fax +49 (0) 8431-53-3 30
Internet: www.hoffmann-mineral.com • e-Mail: info@hoffmann-mineral.com

Formulations

HOFFMANN MINERAL

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

Formulations

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

Brightness

INTRODUCTION

EXPERIMENTAL

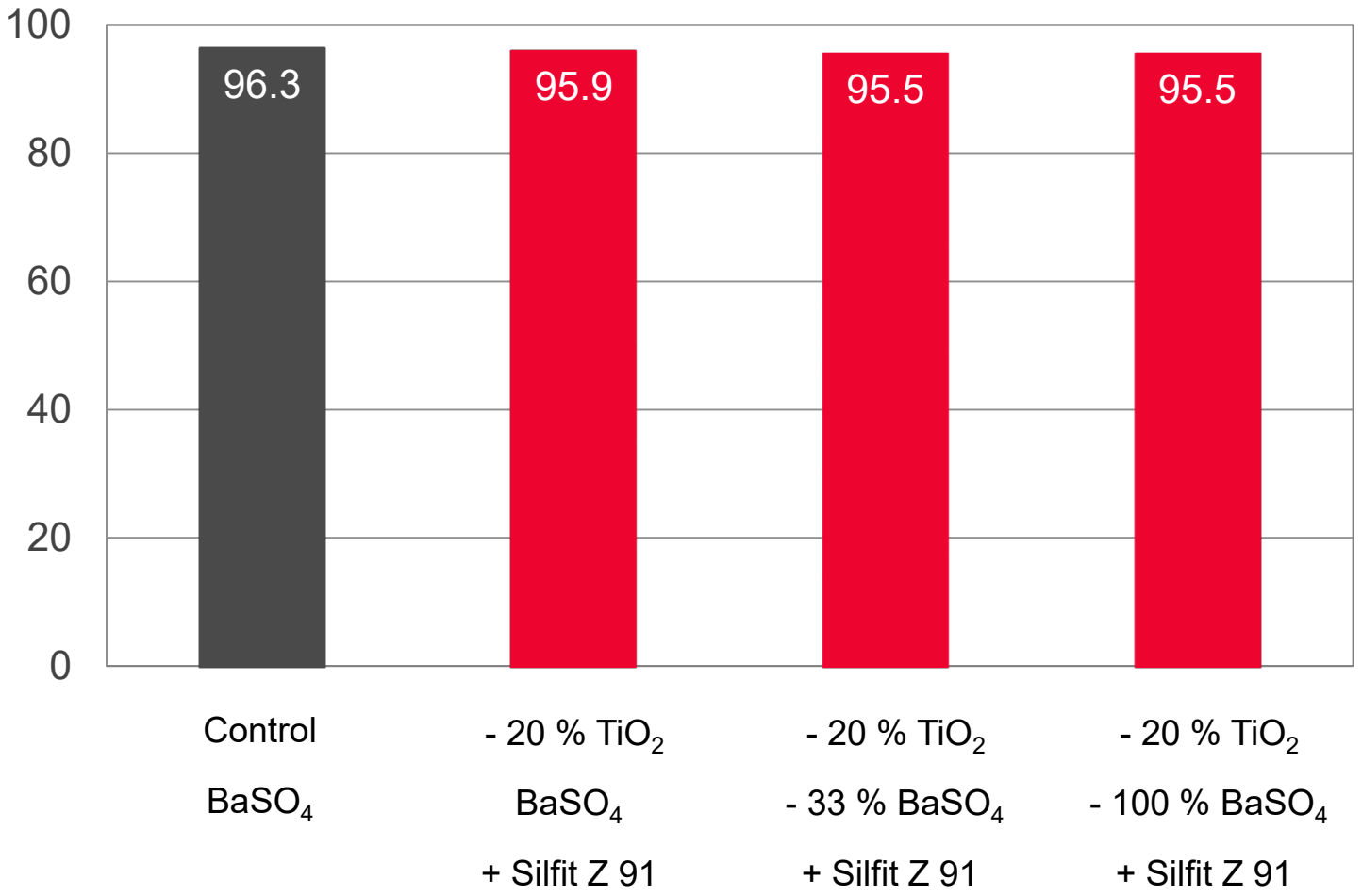
RESULTS

- $BaSO_4$ ppt

Color

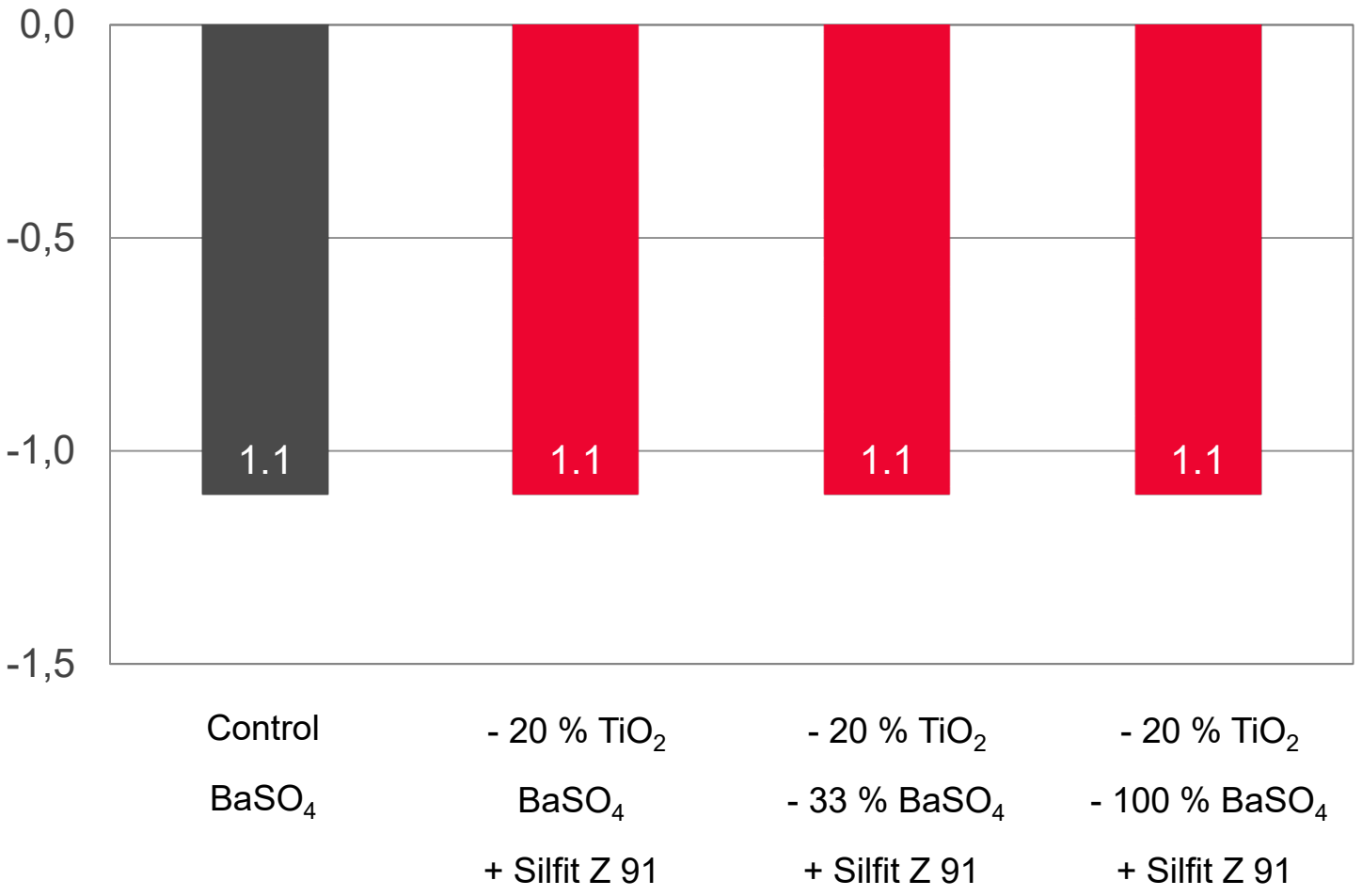
SUMMARY

CIE L*



Red/green-ratio

CIE a*



Yellow/blue-ratio

INTRODUCTION

EXPERIMENTAL

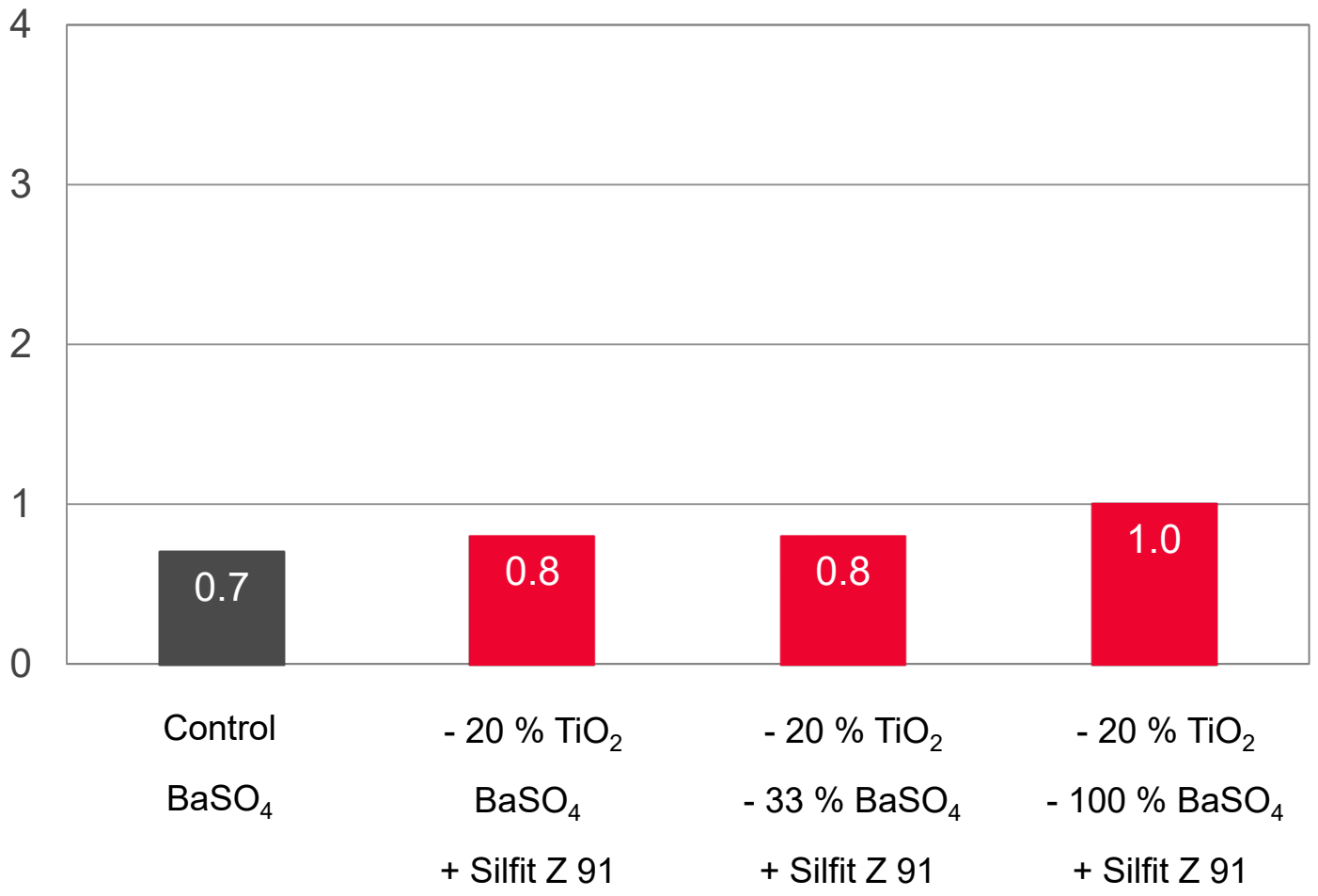
RESULTS

- $BaSO_4$ ppt

Color

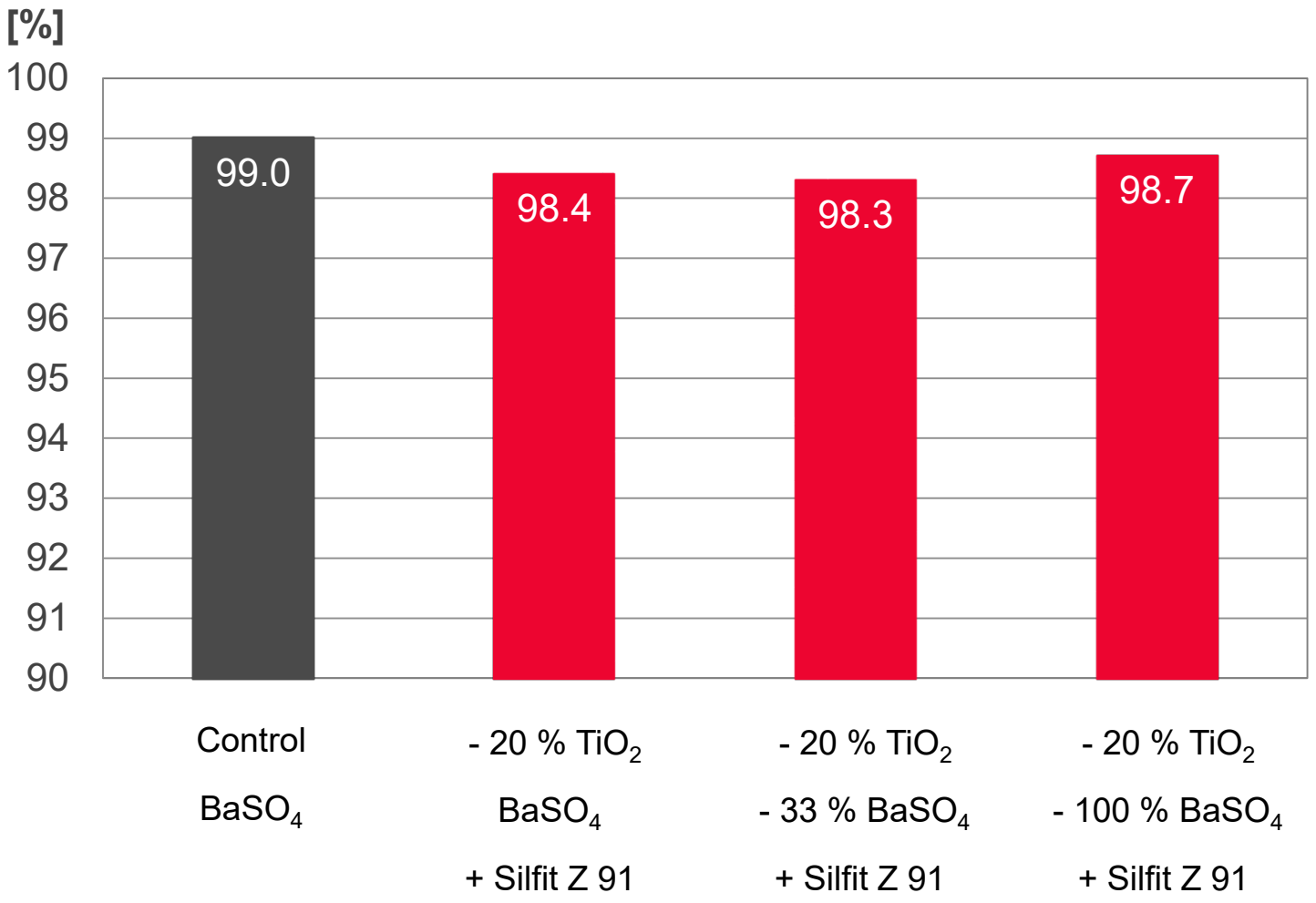
SUMMARY

CIE b*

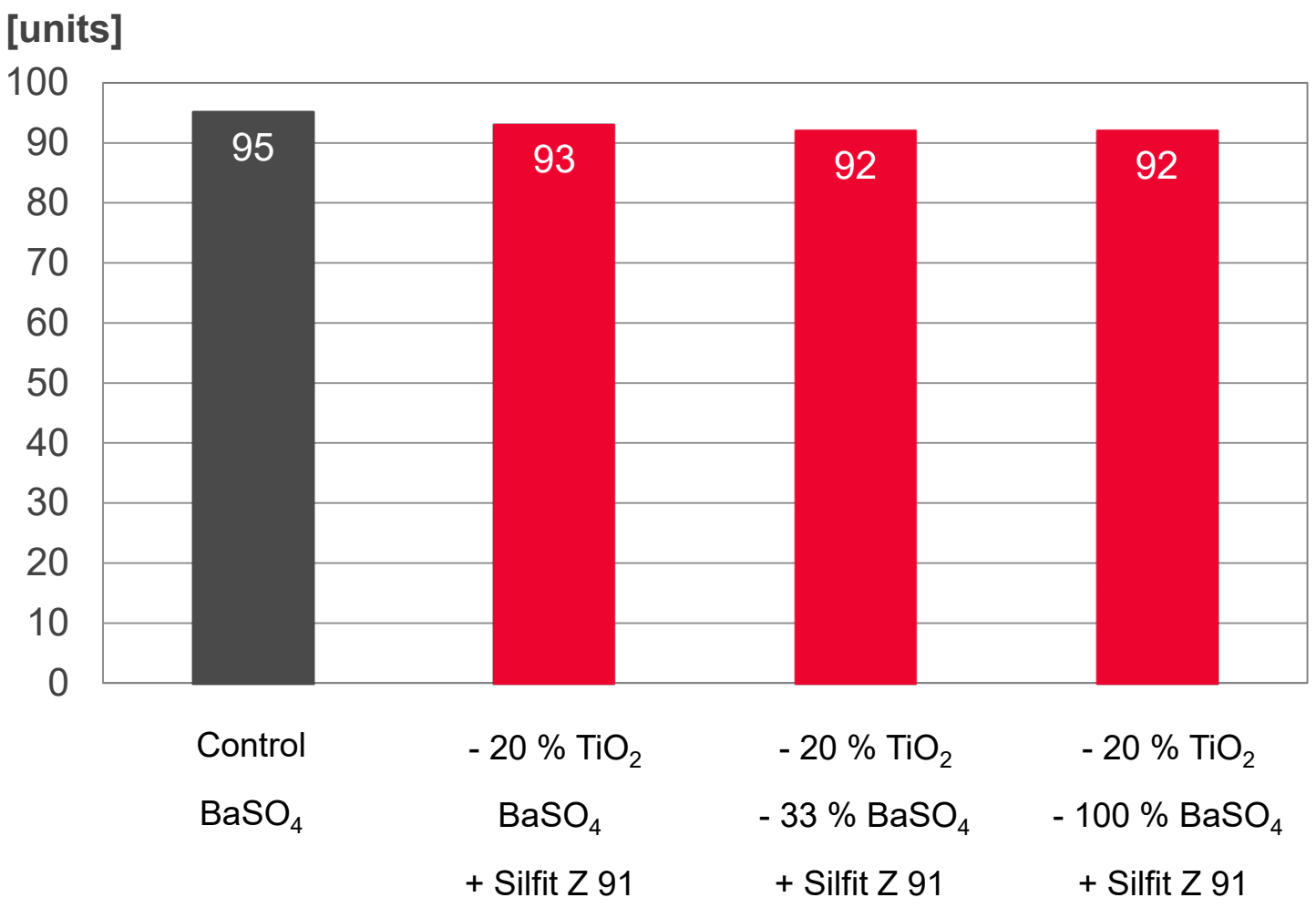


Hiding Power

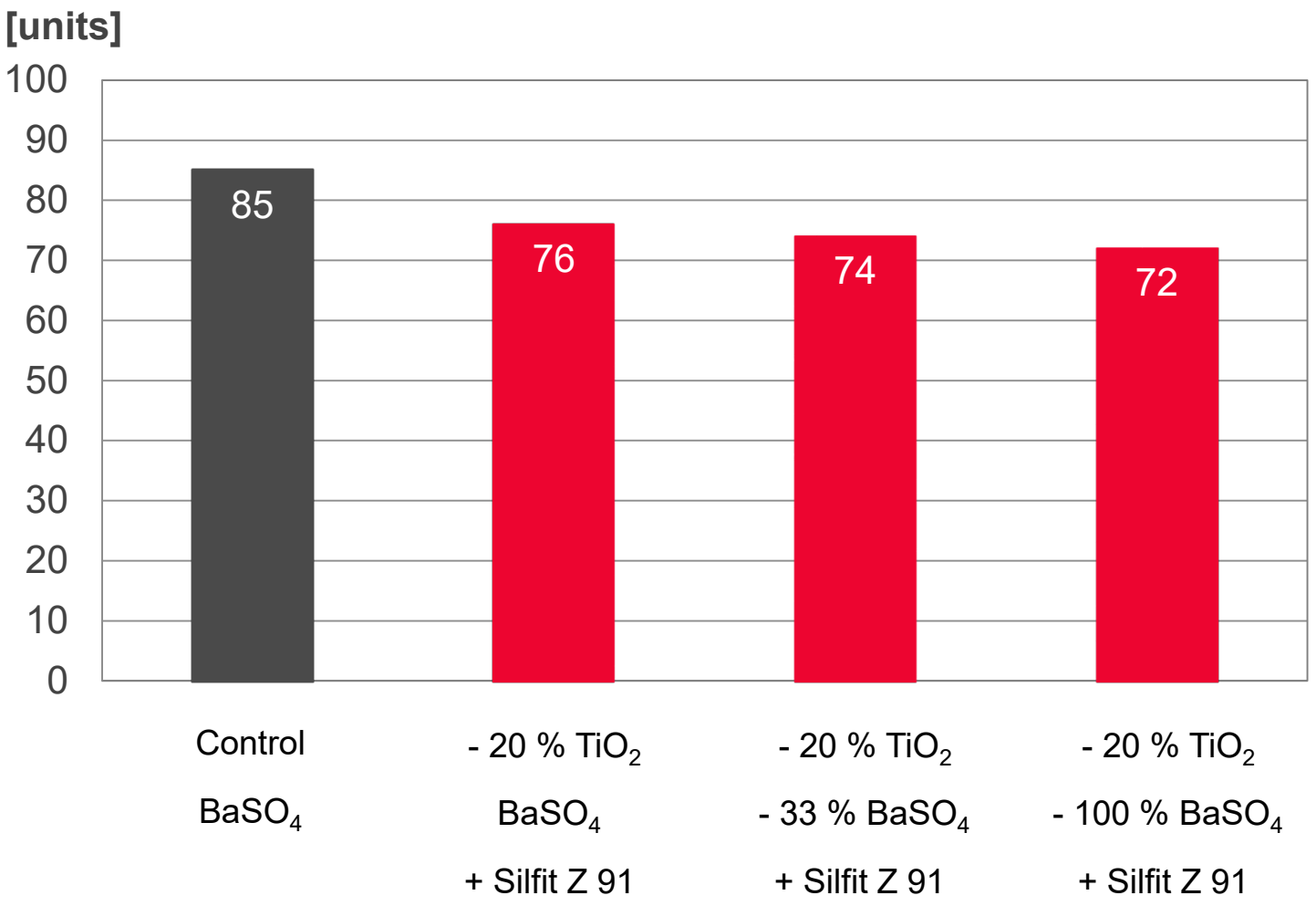
Contrast ratio at a dry film thickness ~ 80 µm



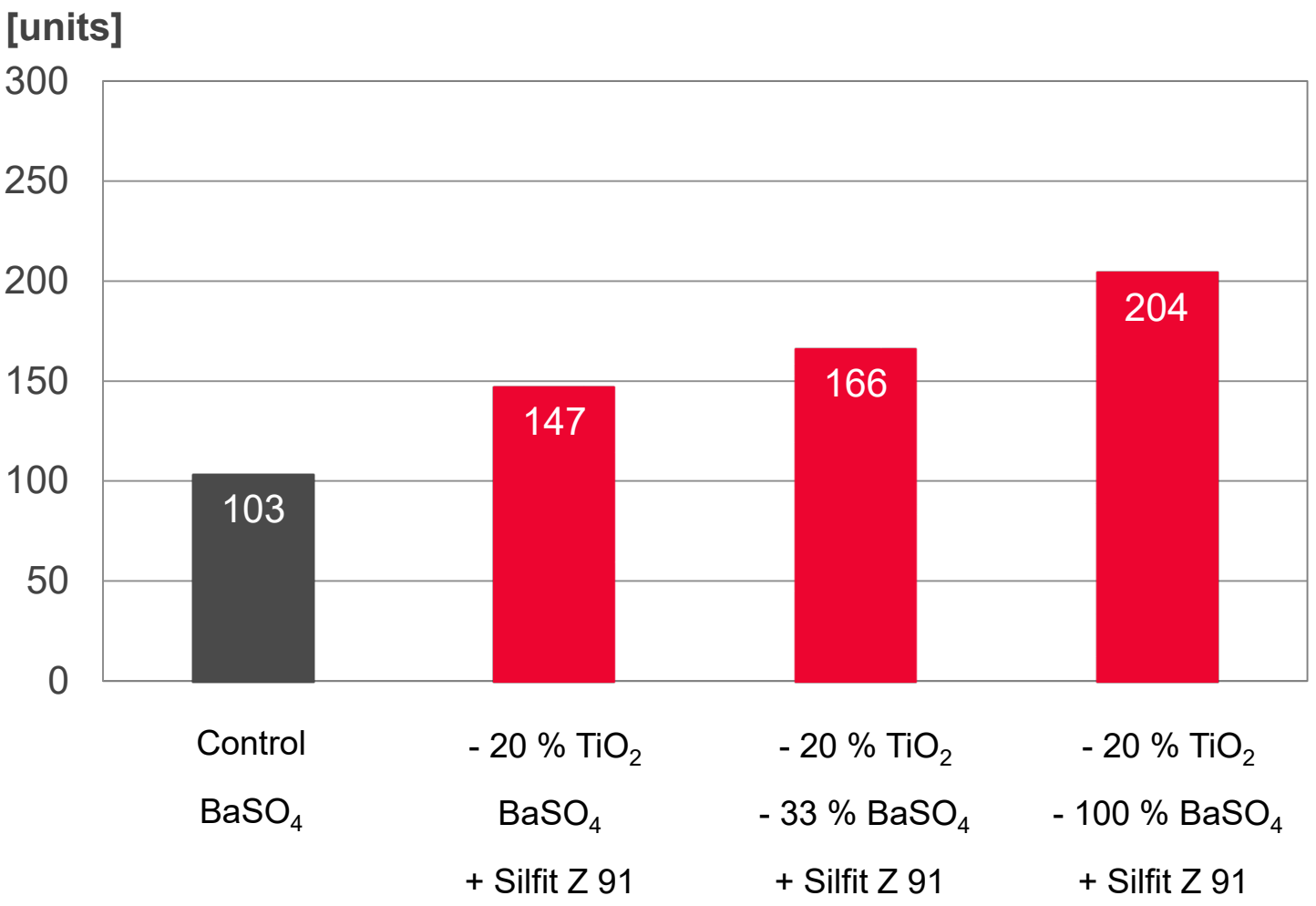
Gloss 60°



Gloss 20°



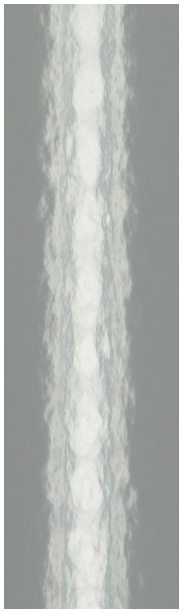
Haze



Leveling

Appearance of surface (visual assessment)

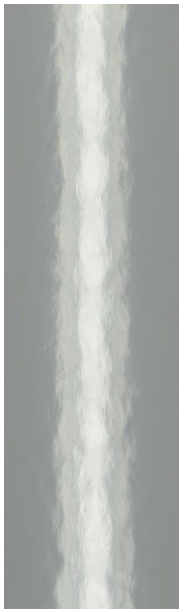
Reflection of overhead light



++++

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

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.

Acetic Salt Spray 2000 h Blistering

HOFFMANN MINERAL

 **Beginning of blistering** **~ 2 % of the surface area 3 – 3 (S2)**	**No blistering**	**No blistering**	**No blistering**
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

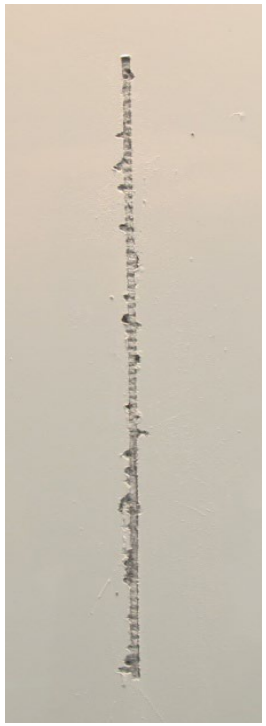
Acetic Salt Spray 2000 h
Delamination at Scribe

HOFFMANN MINERAL

Control

$BaSO_4$

0.8 mm

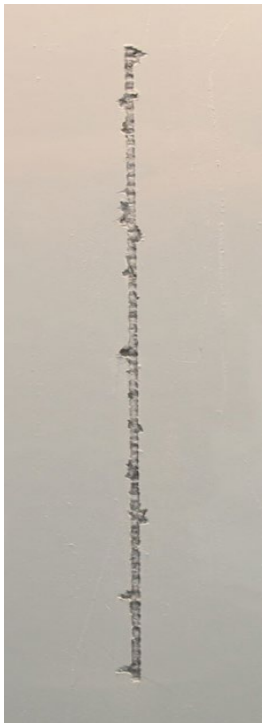


- 20 % TiO_2

$BaSO_4$

+ Silfit Z 91

0.1 mm

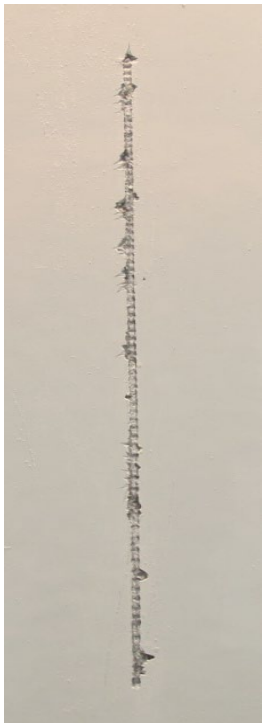


- 20 % TiO_2

- 33 % $BaSO_4$

+ Silfit Z 91

0.1 mm



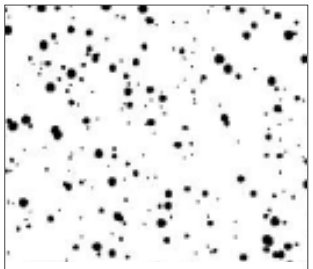
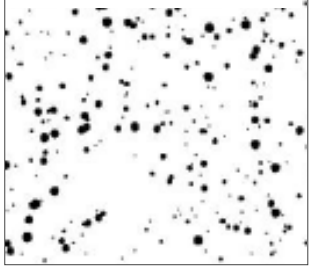
- 20 % TiO_2

- 100 % $BaSO_4$

+ Silfit Z 91

0.1 mm

Humidity Test 2000 h Blistering

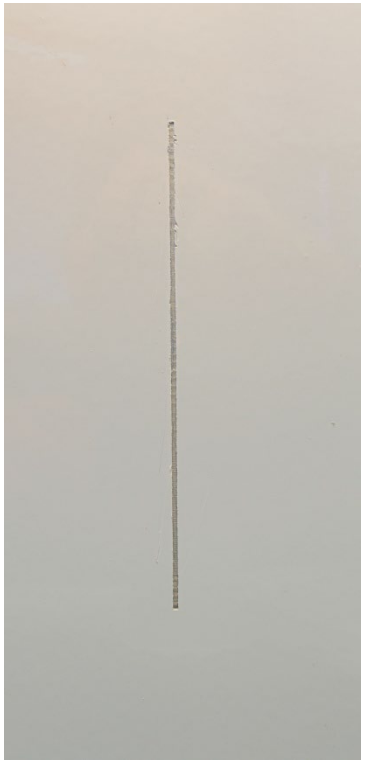
 **Blistering** **4 – 4 (S3)** 	**No blistering**	**No blistering**	**No blistering**
Control $BaSO_4$	- 20 % TiO_2 $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 33 % $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 100 % $BaSO_4$ + Silfit Z 91

Humidity Test 2000 h Delamination at Scribe

Control

$BaSO_4$

24 mm

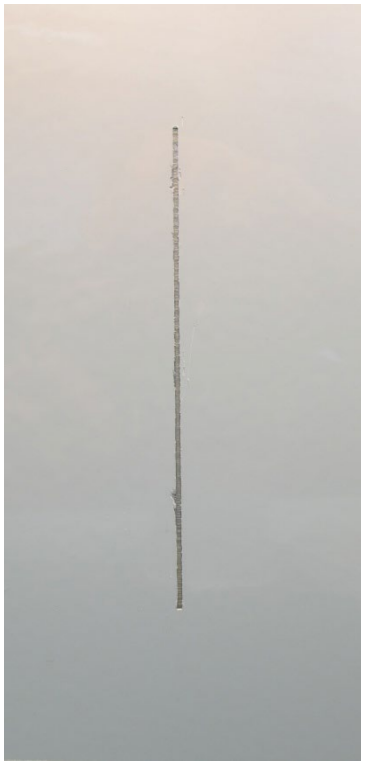


- 20 % TiO_2

$BaSO_4$

+ Silfit Z 91

0 mm

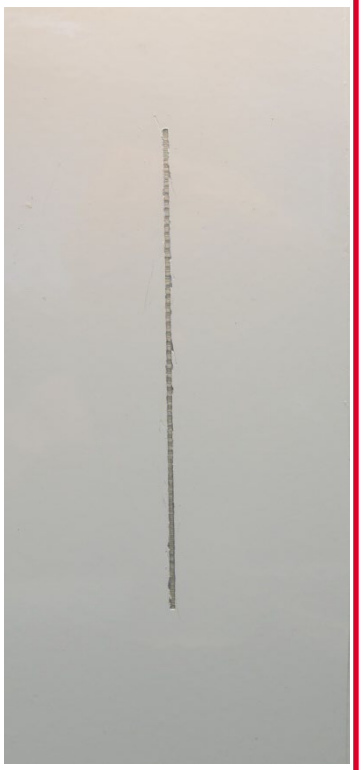


- 20 % TiO_2

- 33 % $BaSO_4$

+ Silfit Z 91

0 mm



- 20 % TiO_2

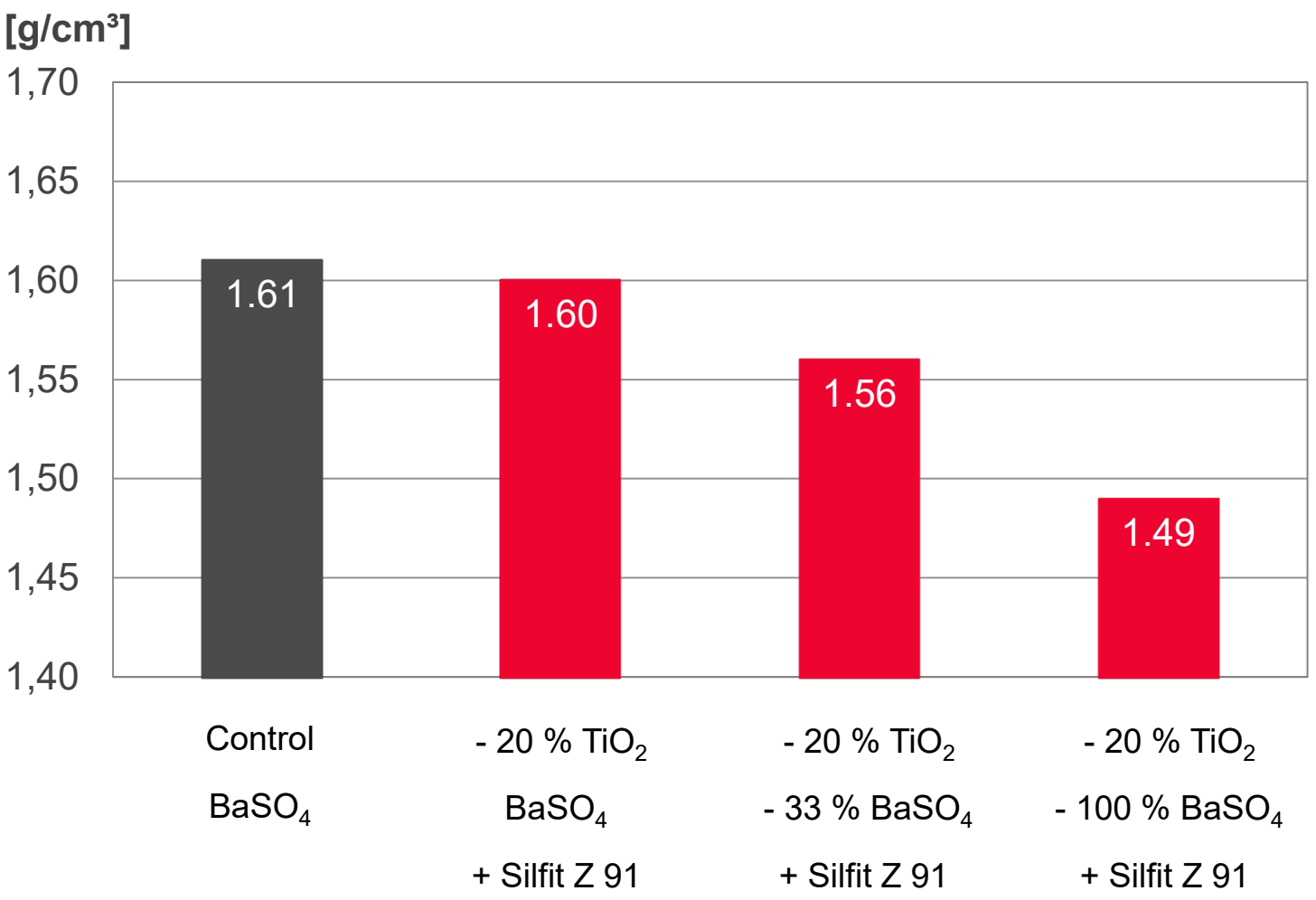
- 100 % $BaSO_4$

+ Silfit Z 91

0 mm

Density

Calculated



Spreading Rate

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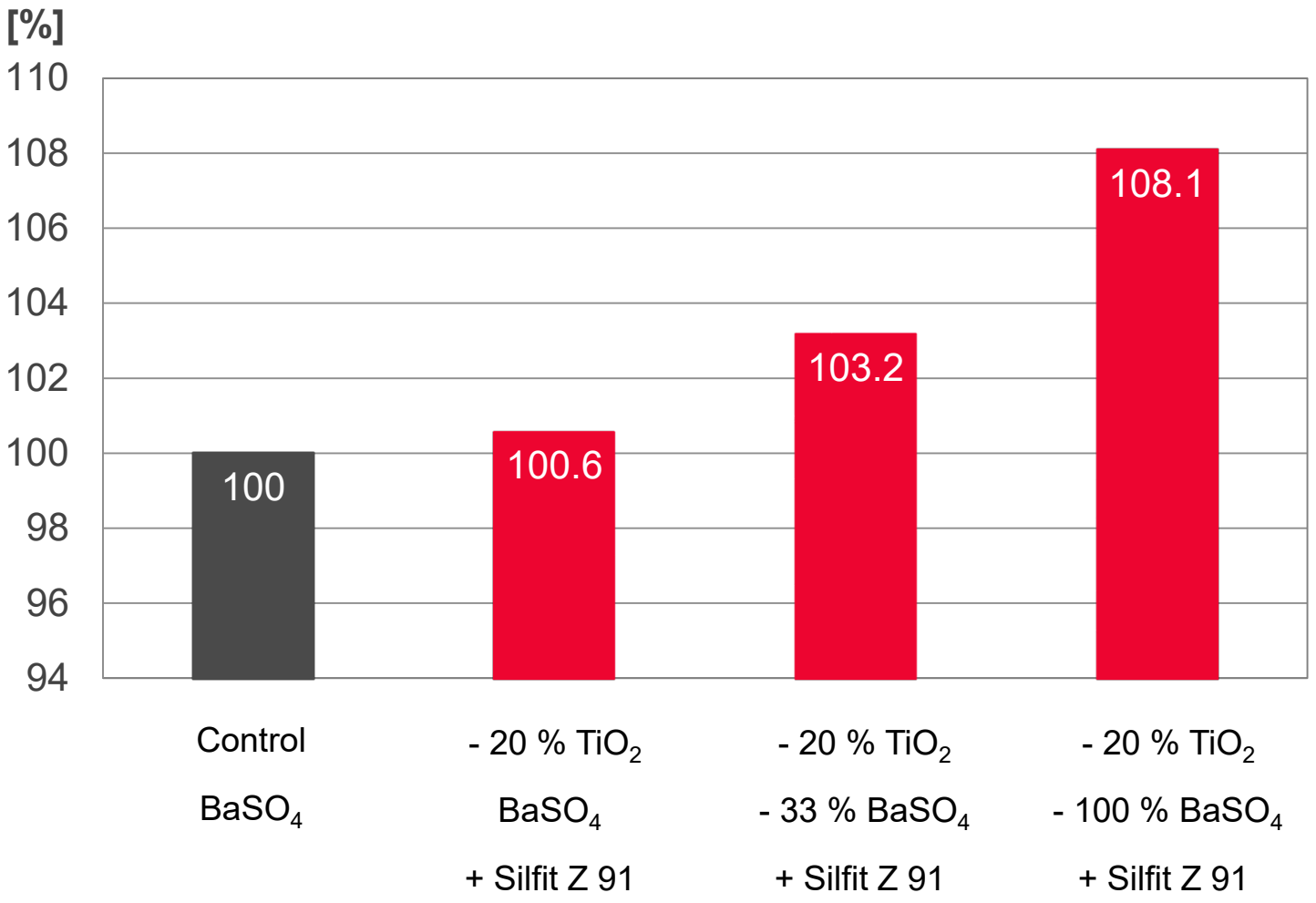
Area coatable per mass unit (e.g. m²/kg powder coating material)



Cost Index
Based on Weight

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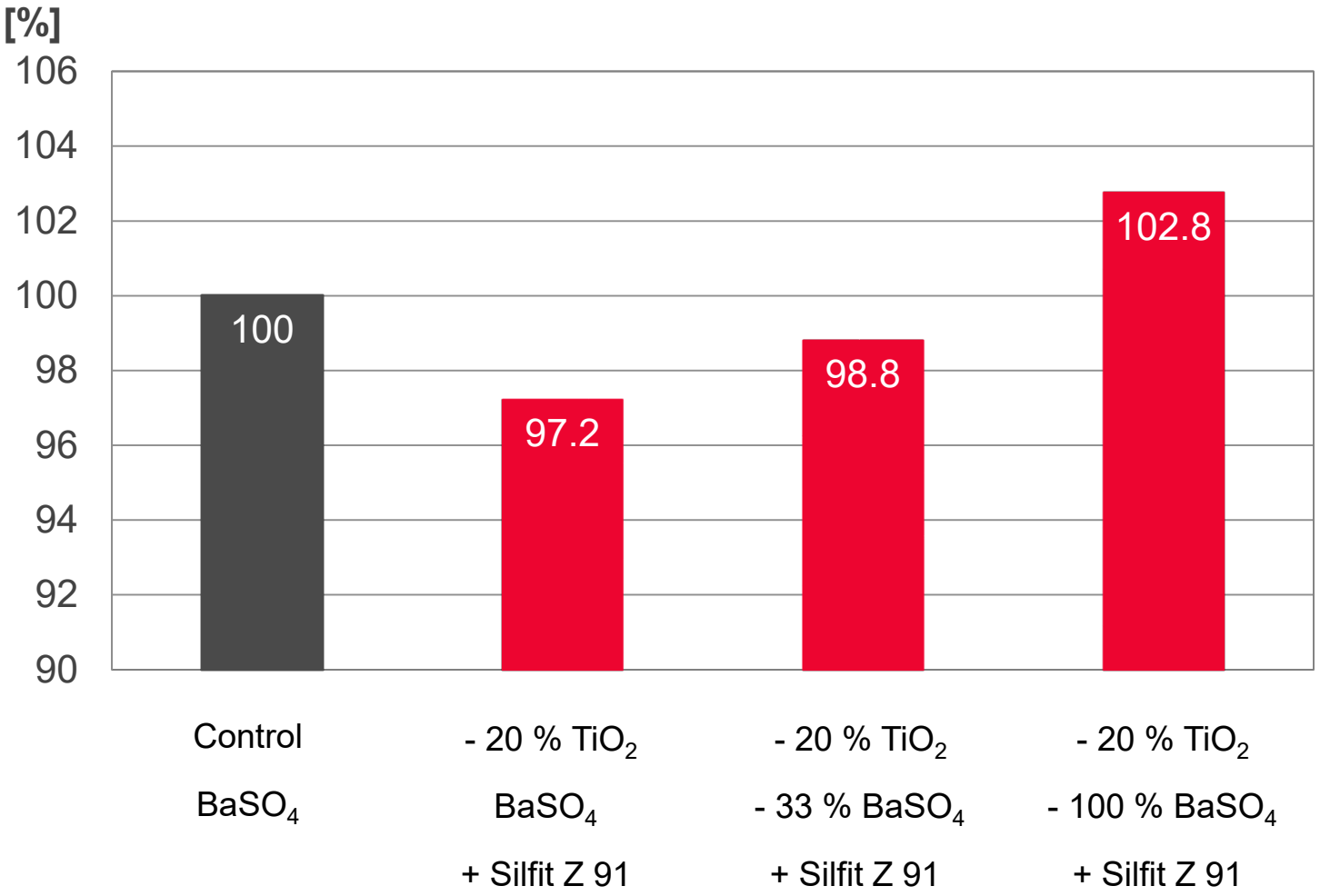
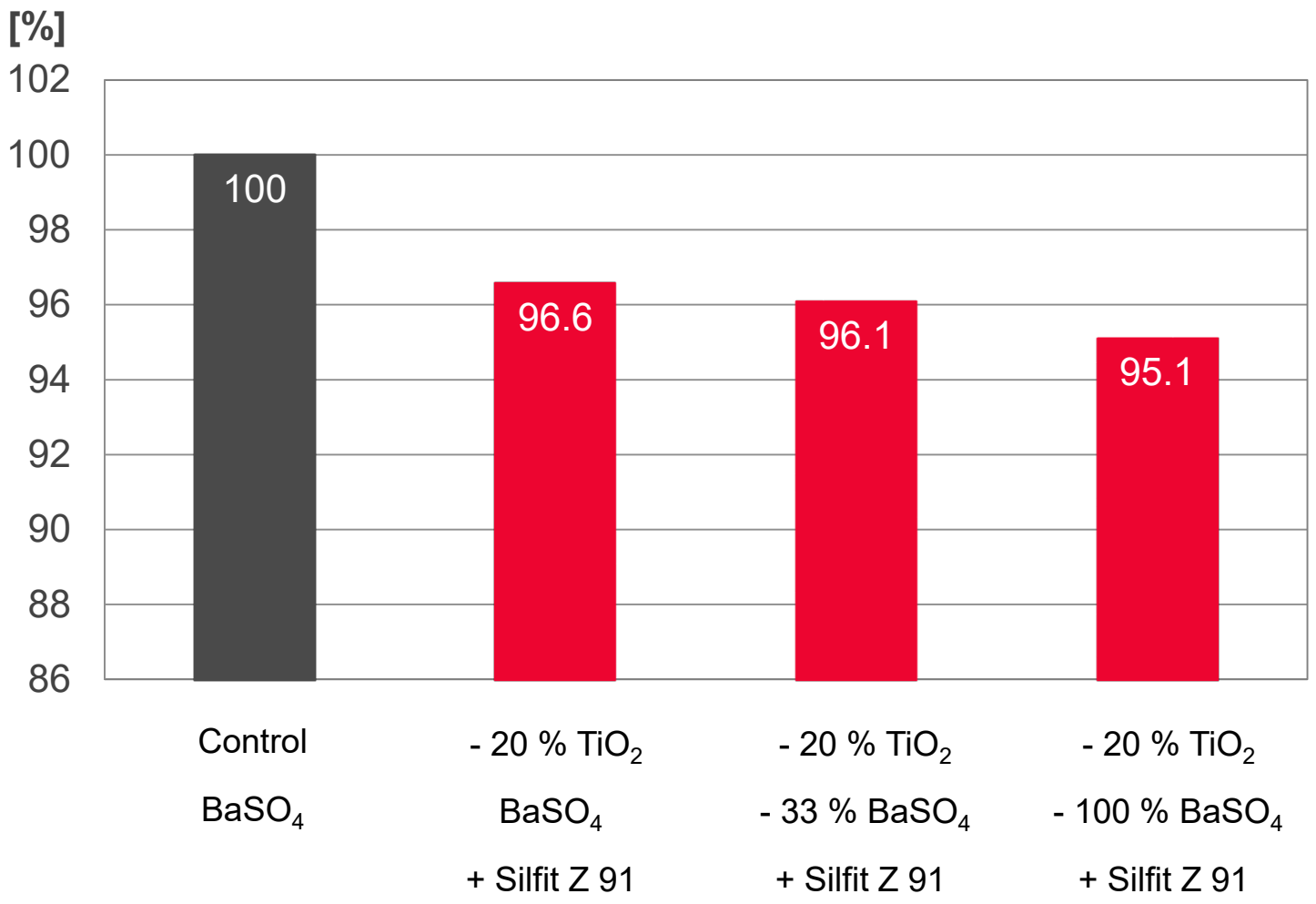
Control = 100 % (Base: Germany 2014)



Cost Index
Based on Volume

HOFFMANN MINERAL

Control = 100 % (Base: Germany 2014)



Summary

Replacement of 20 % titanium dioxide at equal weight with Silfit Z 91 gave rise to the following effects :

- except slightly higher haze, similar optical properties
- excellent weatherability (even up to 50 % titanium dioxide substitution)

+ improved corrosion resistance

+ cost reduction potential

Summary

Additionally partial substitution (33 %) of the precipitated barium sulfate by Silfit Z 91 improved furthermore:

+ higher spreading rate (lower density of powder coating)

+ cost reduction potential

Conclusion

Independent from the type of barium sulfate, natural or precipitated, it is possible to replace 20 % of titanium dioxide at equal weight with Silfit Z 91 without loosing significant hiding power or weatherability. The corrosion resistance can be considerably improved and it offers the most economizing potential.

Additional (partial) substitution of the natural barium sulfate by Silfit Z 91 improved the optical properties, increases the spreading rate and offers cost reduction potential.

Additional partial substitution (33 %) of the precipitated barium sulfate by Silfit Z 91 increases the spreading rate and offers cost reduction potential.

Abrasivity
Einlehner-Test

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Variations of titanium dioxide / filler (parts per weight in formulation)

	Control $BaSO_4$	- 20 % TiO_2 $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 33 % $BaSO_4$ + Silfit Z 91	- 40 % TiO_2 + Silfit Z 91	TiO_2
				in addition only for Einlehner test	
Titanium dioxide	19.5	15.6	15.6	11.7	39.5
$BaSO_4$ natural	20	20	13.4	20	0
Silfit Z 91	-	3.9	7.8	7.8	0
Einlehner Abrasivity [mg]	51	54	54	54	62

Abrasivity
Einlehner-Test

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Variations of titanium dioxide / filler (parts per cent)

	Control $BaSO_4$	- 20 % TiO_2 $BaSO_4$ + Silfit Z 91	- 20 % TiO_2 - 33 % $BaSO_4$ + Silfit Z 91	- 40 % TiO_2 + Silfit Z 91	TiO_2
				in addition only for Einlehner test	
Titanium dioxide	49.4	39.5	42.4	29.6	100
$BaSO_4$ natural	50.6	50.6	36.4	50.6	0
Silfit Z 91	-	9.9	21.2	19.7	0
Einlehner Abrasivity [mg]	51	54	54	54	62

We supply material for good ideas!

HOFFMANN MINERAL GmbH
Muenchener Straße 75
DE-86633 Neuburg (Donau)

Phone: +49 8431 53-0
Internet: www.hoffmann-mineral.de
E-mail: info@hoffmann-mineral.com