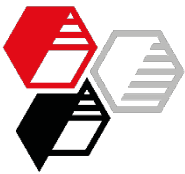


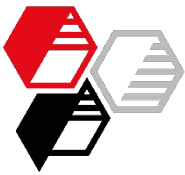
Calcined Neuburg Siliceous Earth in powder coatings (hybrid-based, white)

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



Contents

- Introduction
- Experimental
- Results
 - Color
 - Hiding power / opacity
 - Gloss and Haze
 - Leveling
 - Flexibility (impact test and cupping test)
 - Mechanical resistance (scratch test)
 - Density / spreading rate
 - Cost index
- Summary
- Appendix



Status Quo

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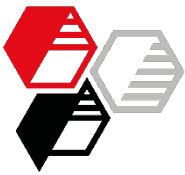
RESULTS

SUMMARY

An earlier study of a hybrid powder coating focused 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

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Thus the question comes up if **Calcined Neuburg Siliceous Earth** is capable of partially substituting titanium dioxide while maintaining mechanical and optical properties (particularly opacity).

The main filler remains in the formulation:

- natural barium sulfate

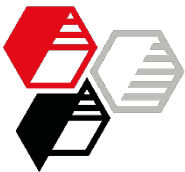
as example for low cost formulation with standard requirements

or

- precipitated barium sulfate grade

special grade for powder coatings with high quality optical properties

In addition, cost aspects were also taken into account.



Filler Characteristics

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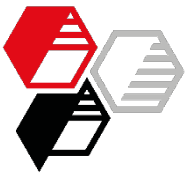
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		Barium sulfate		Calcined Neuburg Siliceous Earth
		natural	ppt. special grade for powder coatings	Silfit Z 91
Morphology		corpuscular		corpuscular / lamellar aggregated
Density	[g/cm ³]	4.4	4.4	2.6
Particle size d ₅₀	[μm]	2.9	1.6	2.0
Particle size d ₉₇	[μm]	14	5	10
Oil absorption	[g/100g]	14	22	60
Specific surface area BET	[m ² /g]	0.8	2.6	7.5



Filler Characteristics

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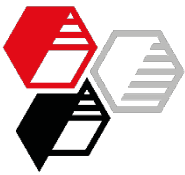
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Color	Barium sulfate		Calcined Neuburg Siliceous Earth
	natural	ppt. special grade for powder coatings	Silfit Z 91
L*	95	97	95
a*	- 0.3	- 0.5	- 0.2
b*	0.2	0.5	1.2



What is Neuburg Siliceous Earth?

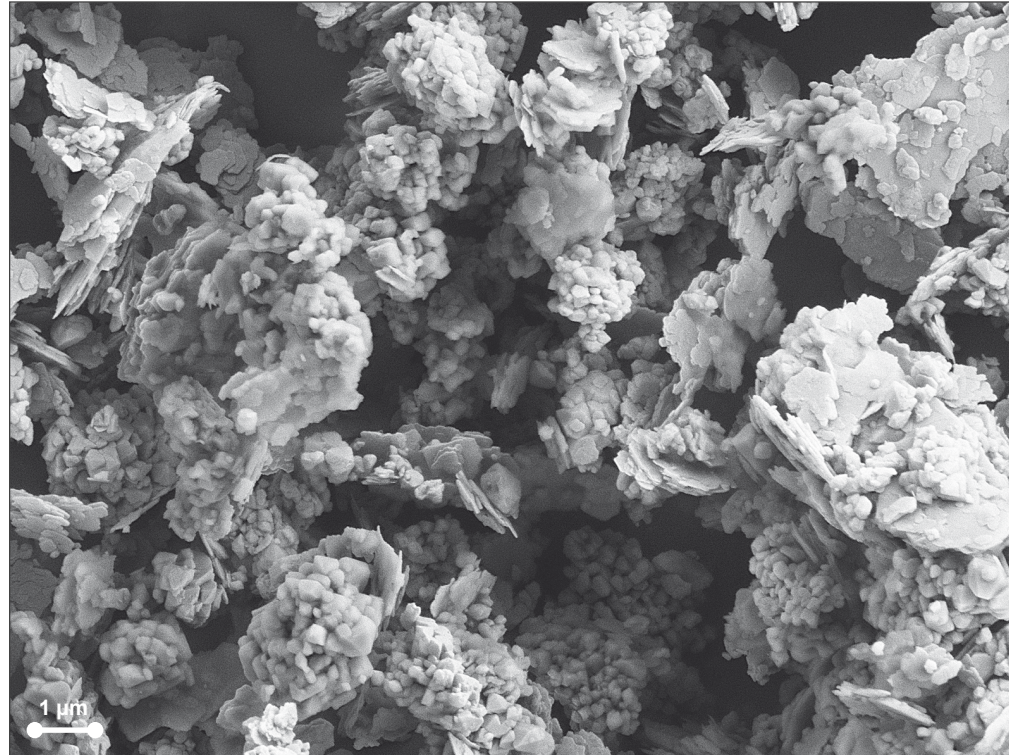
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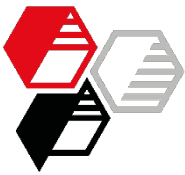
RESULTS

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

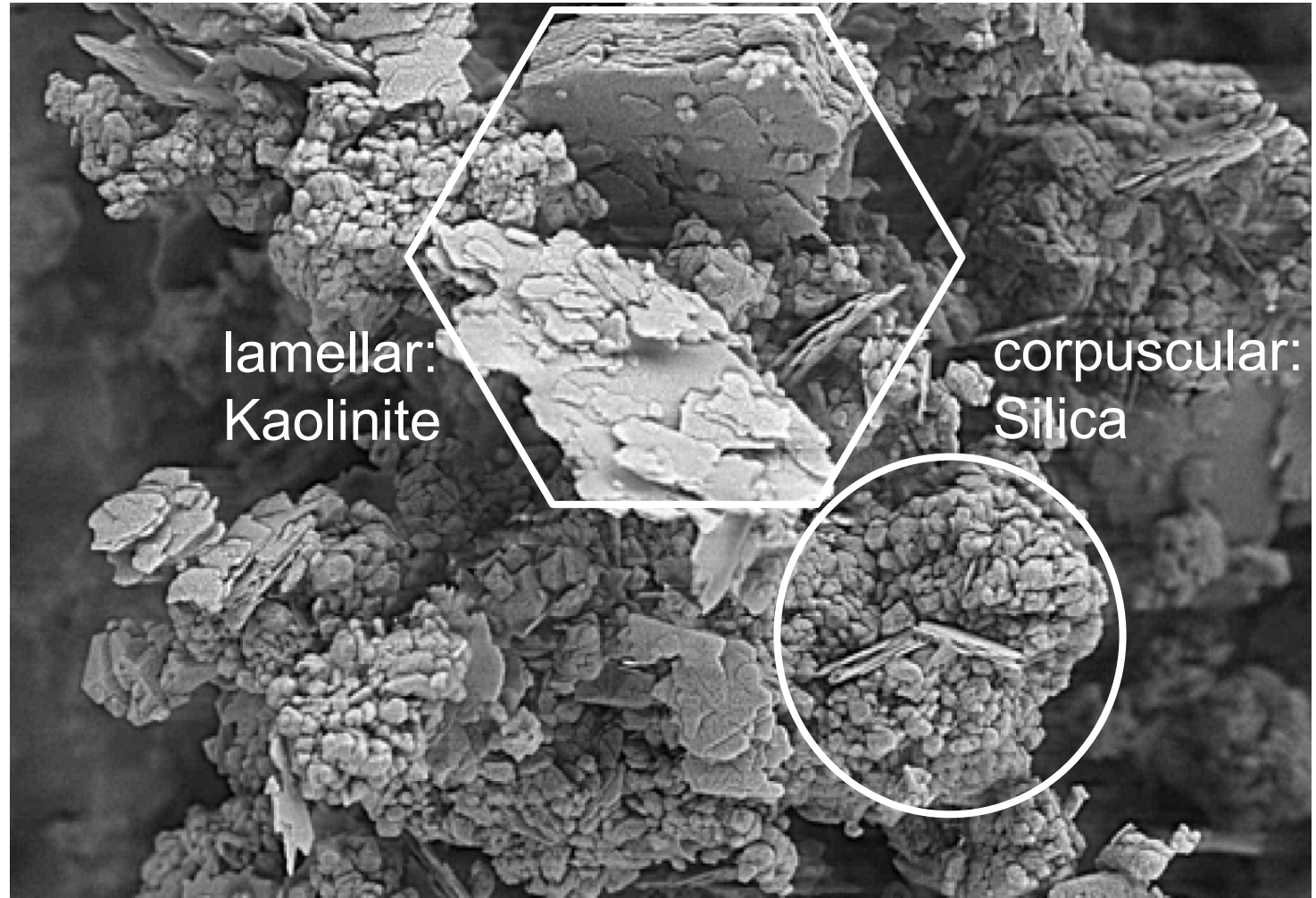
The silica portion exhibits a round grain shape and consists of aggregated primary particles of about 200 nm diameter.



Morphology of Neuburg Siliceous Earth

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Magnification 10.000x

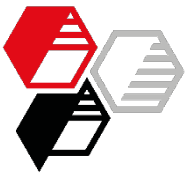


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Calcined Neuburg Siliceous Earth

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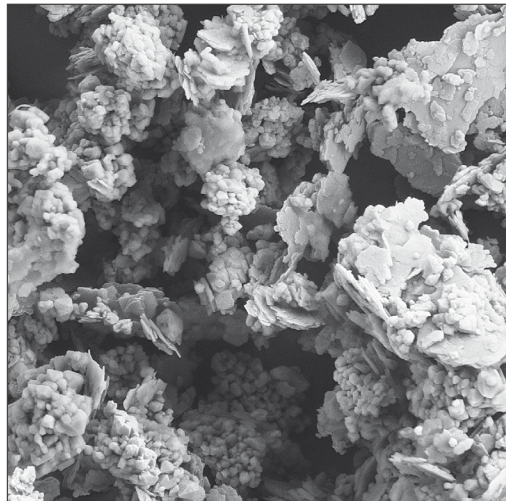
A downstream thermal process leads to the calcined products **SILFIT** and **AKTIFIT**, based on SILLITIN Z 86.

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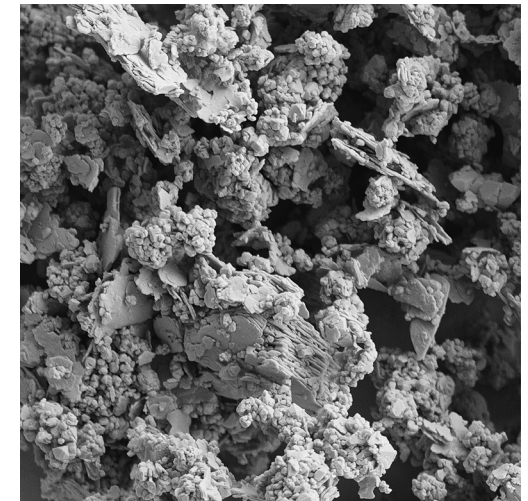
RESULTS

SUMMARY



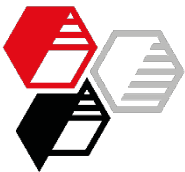
Neuburg Siliceous Earth

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.

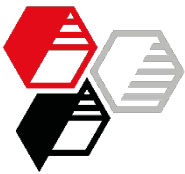


Base Formulation

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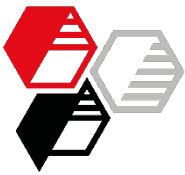
Parts per weight = %

	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



Preparation

Premix	Mixaco Mixer LAB CM 3, 2 min, 1000 rpm
Extruder	Coperion ZSK 18, twin screw, shaft speed 350 rpm, heating zone 50°C/100°C/100°C/100°C/100°C
Micronizing	Retsch ZM 100, 0.5 sieve, 18000 rpm
Sieving	Fritsch Analysette 3 PRO, 5-8 min, mesh size 100 µm; Amplitude 2.5 mm
Application	Powder gun GEMA Corona, Type PG 1-B, 80 kV, 2 bar, dry film thickness ~ 70 µm
Substrate	Q-Panels: Aluminum A 36 and A 48
Curing	15 min at 180 °C oven temperature, corresponds to approx. 10 min PMT 180 °C



What kind of barium sulfate do you use?

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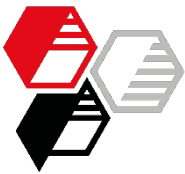
- Barium sulfate natural



or

- Barium sulfate precipitated



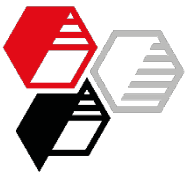


Formulations

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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 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



Formulations

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Parts per cent (%)

INTRODUCTION

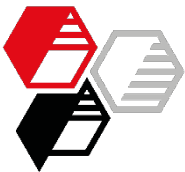
EXPERIMENTAL

• BaSO₄ natural

RESULTS

SUMMARY

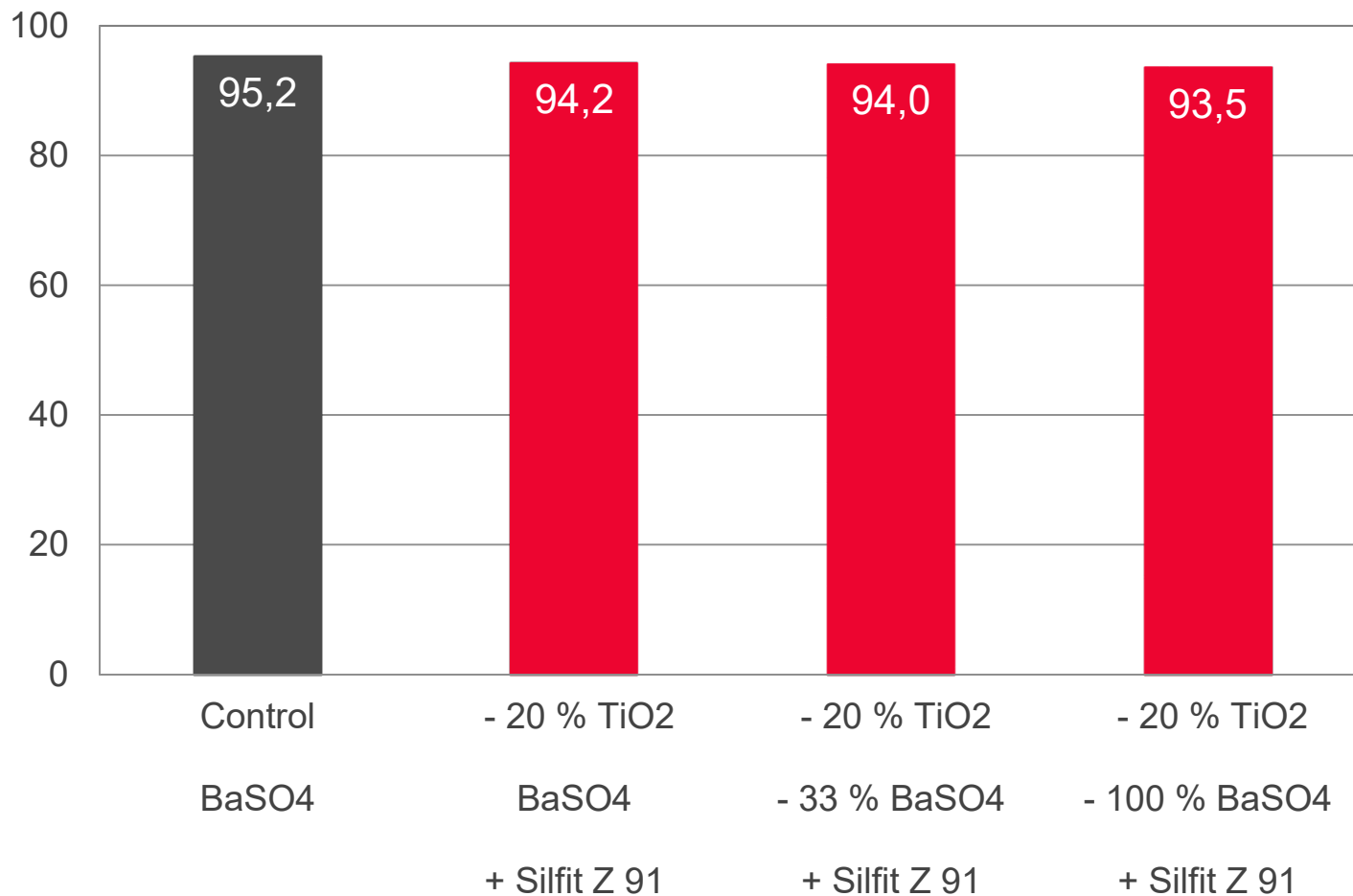
	Control BaSO ₄	- 20 % TiO ₂ BaSO ₄ + Silfit Z 91	- 20 % TiO ₂ - 33 % BaSO ₄ + Silfit Z 91	- 20 % TiO ₂ - 100 % BaSO ₄ + Silfit Z 91
Crylcoat 1771-3	39.0	38.9	40.0	42.4
Epikote 1003	18.0	18.0	18.5	19.6
Additol P 896	3.0	3.2	3.3	3.5
Titanium dioxide	19.5	15.6	16.0	16.9
BaSO ₄ natural	20.0	20.0	13.7	-
Silfit 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

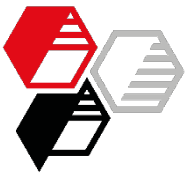


Color Brightness L*

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CIE L*



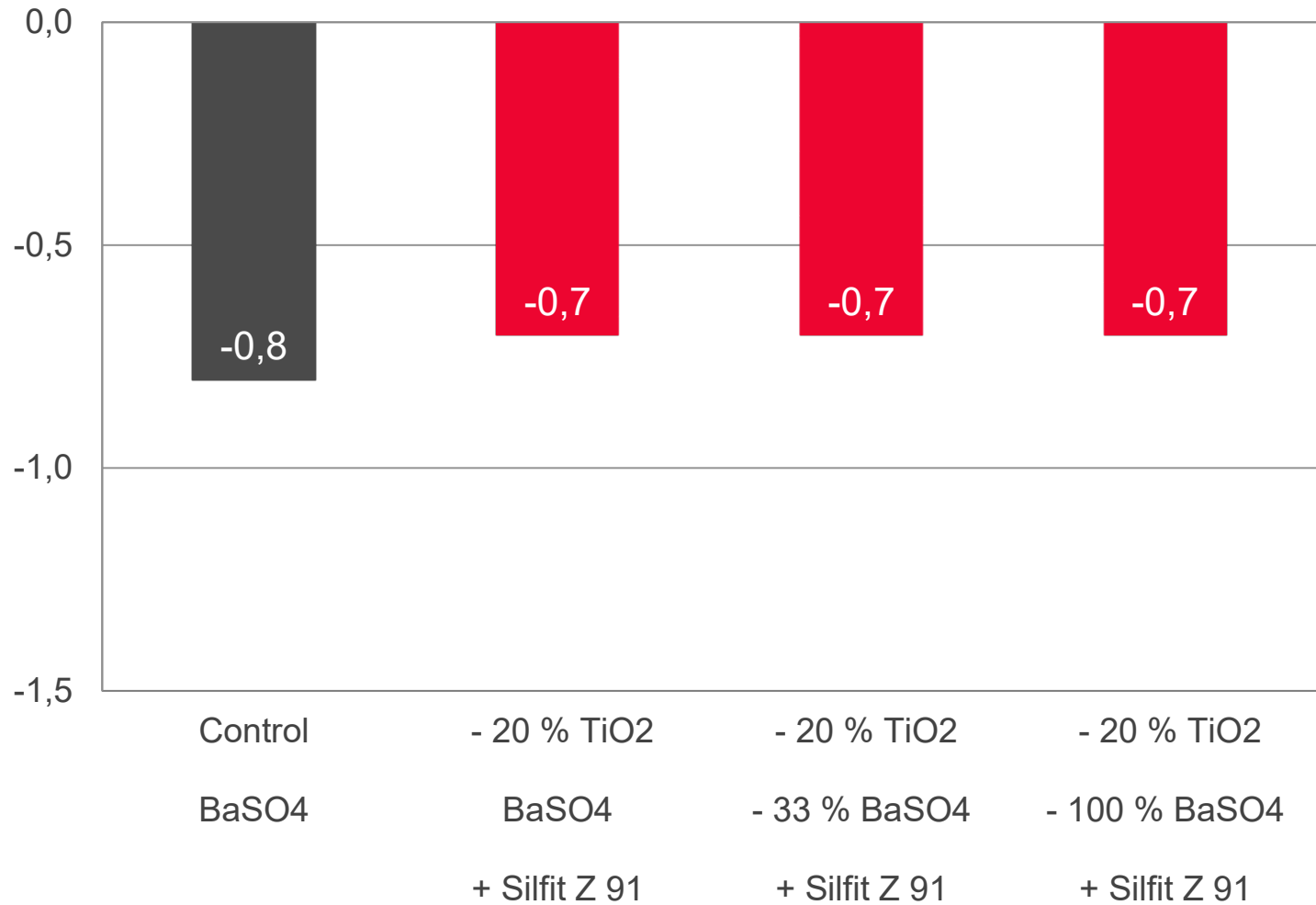


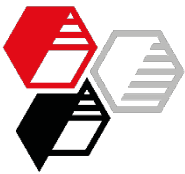
Color

Red/green-ratio a^*

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CIE a^*



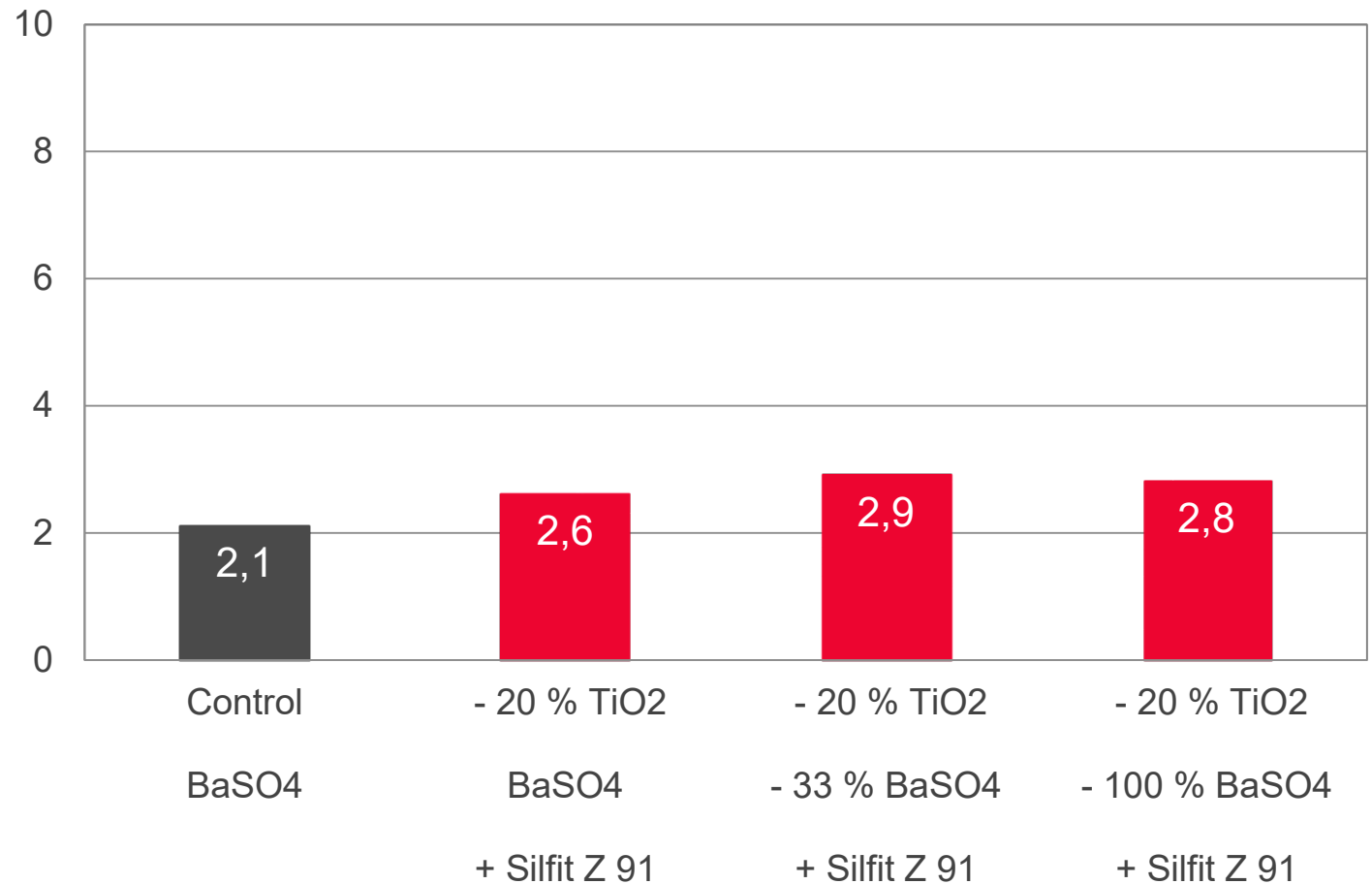


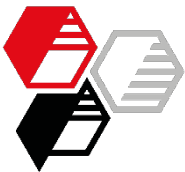
Color

Yellow/blue-ratio b^*

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CIE b^*

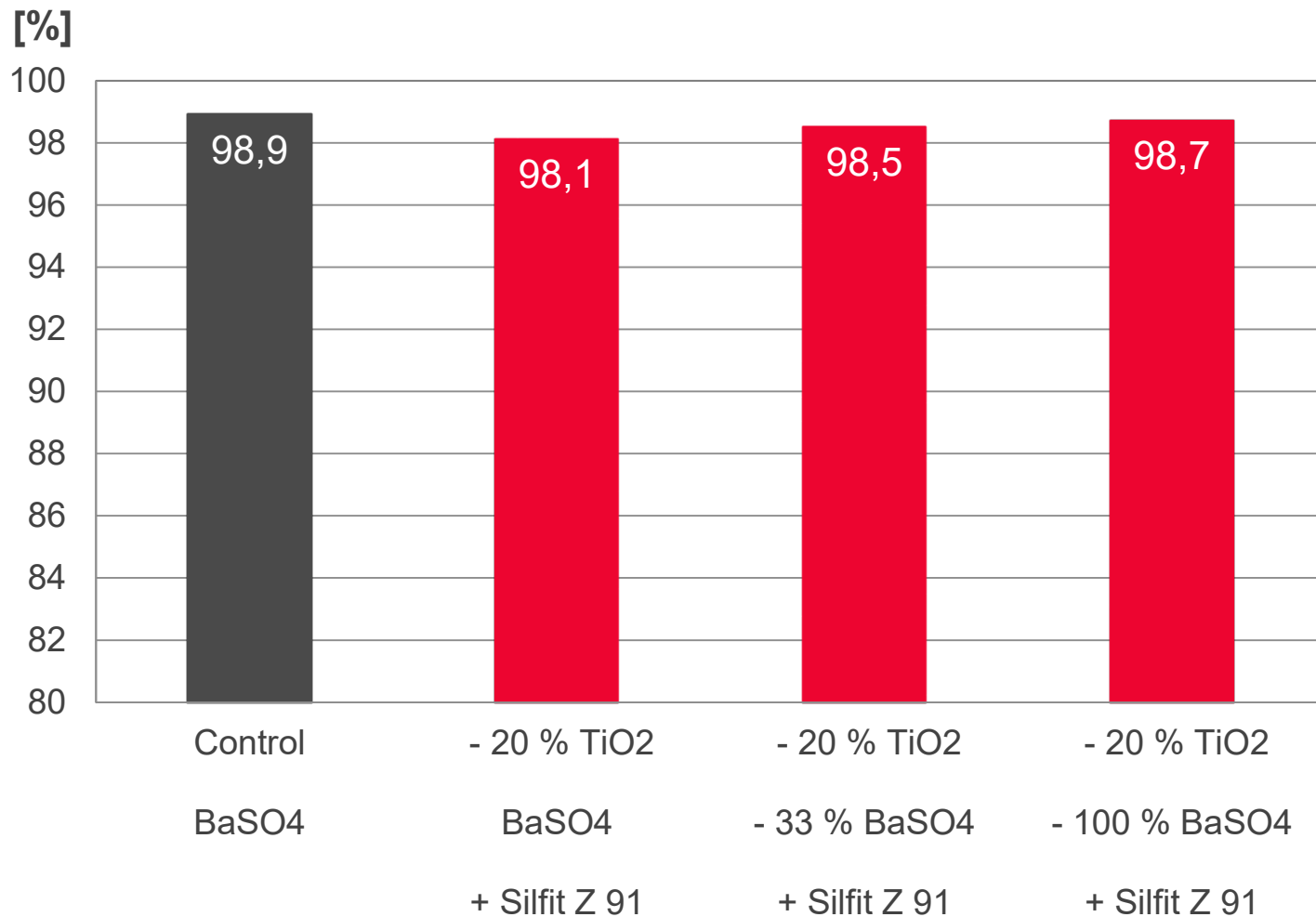


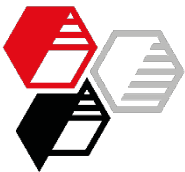


Hiding Power / Opacity

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Contrast ratio at a dry film thickness ~ 70 µm





Gloss 20°

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INTRODUCTION

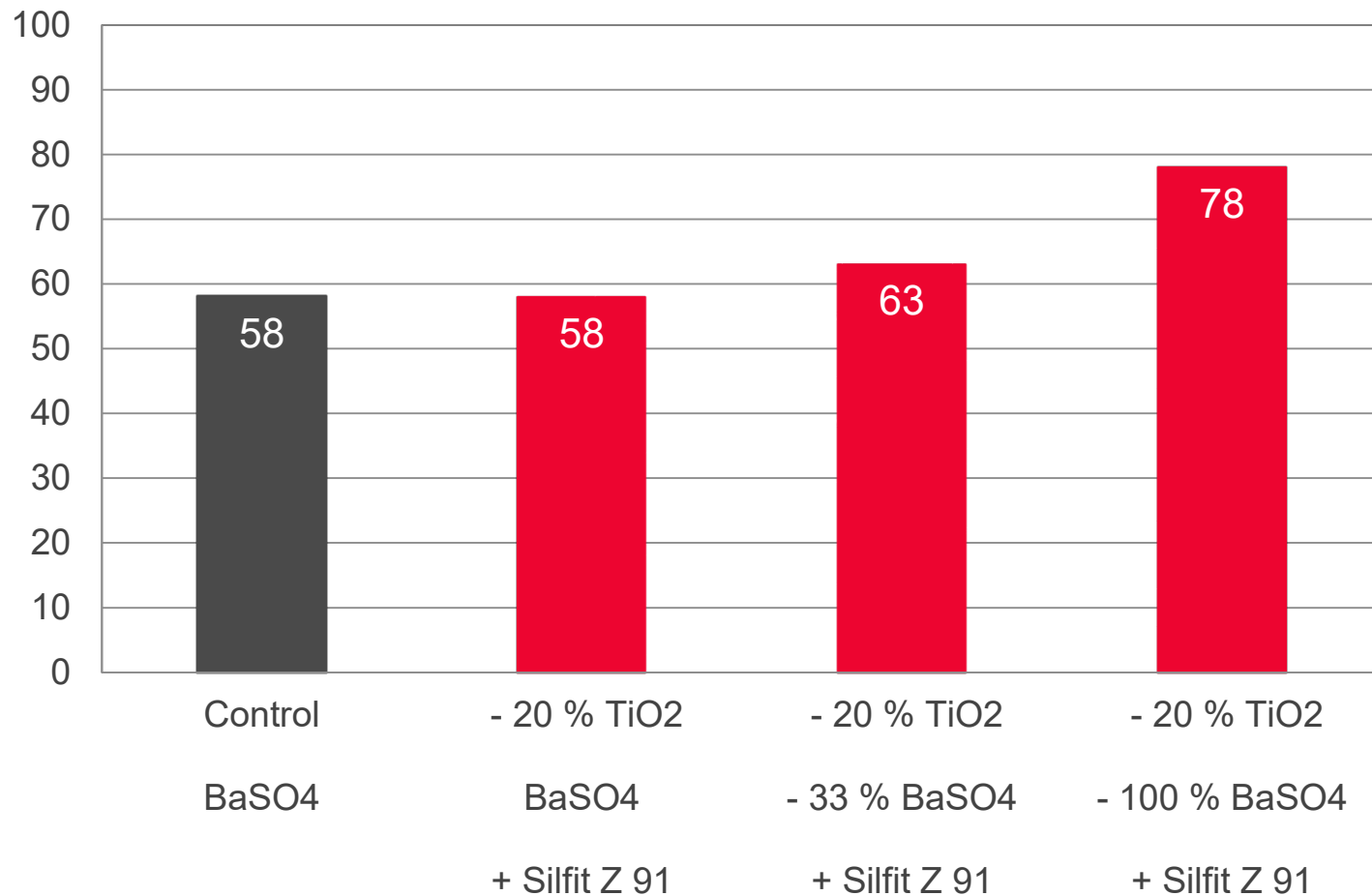
EXPERIMENTAL

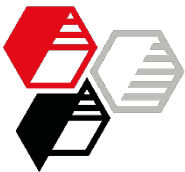
RESULTS

• BaSO₄ natural

SUMMARY

[units]





Haze

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INTRODUCTION

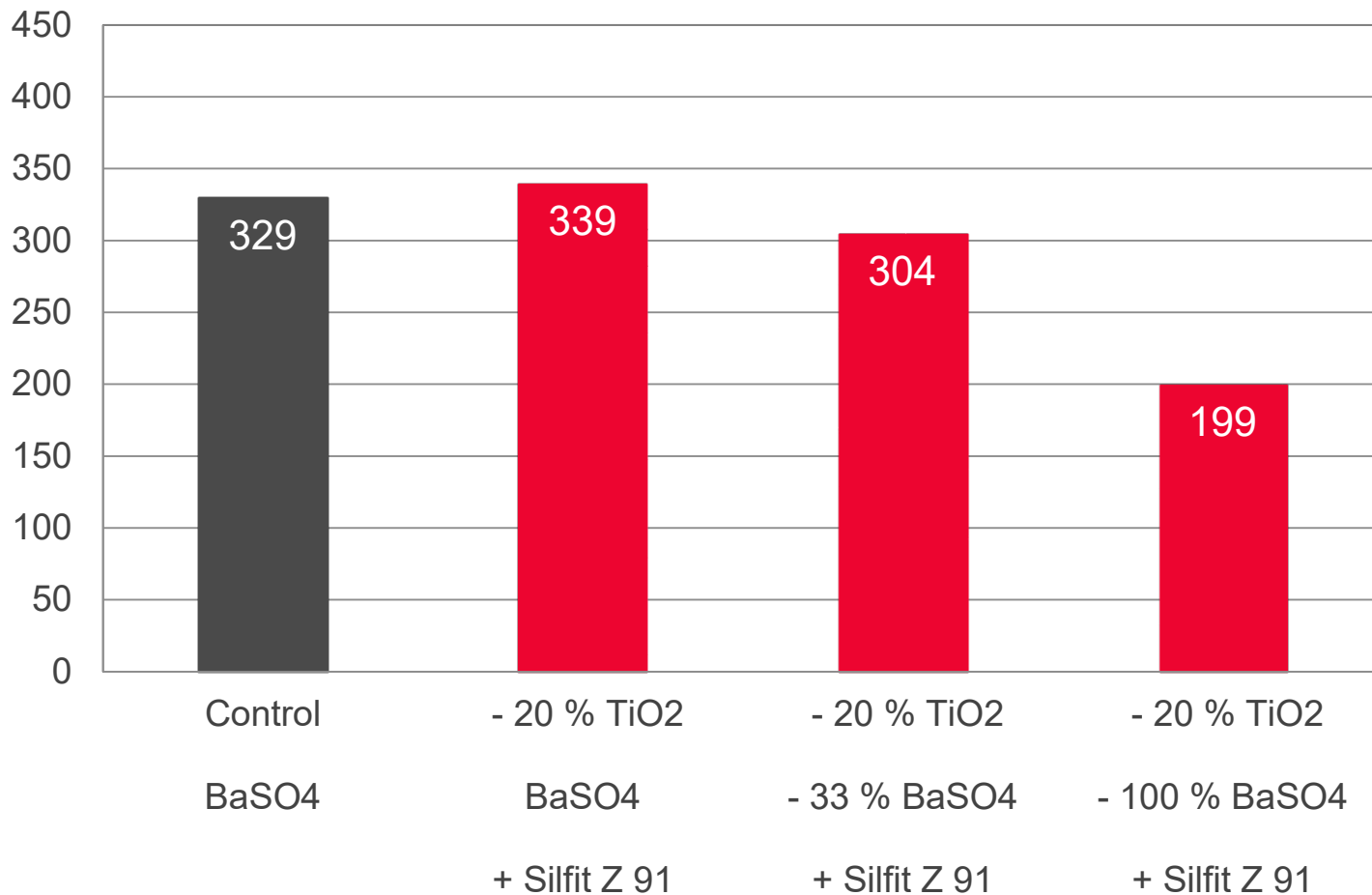
EXPERIMENTAL

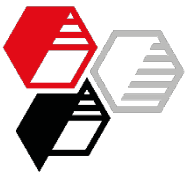
RESULTS

• BaSO₄ natural

SUMMARY

[units]





Gloss 60°

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INTRODUCTION

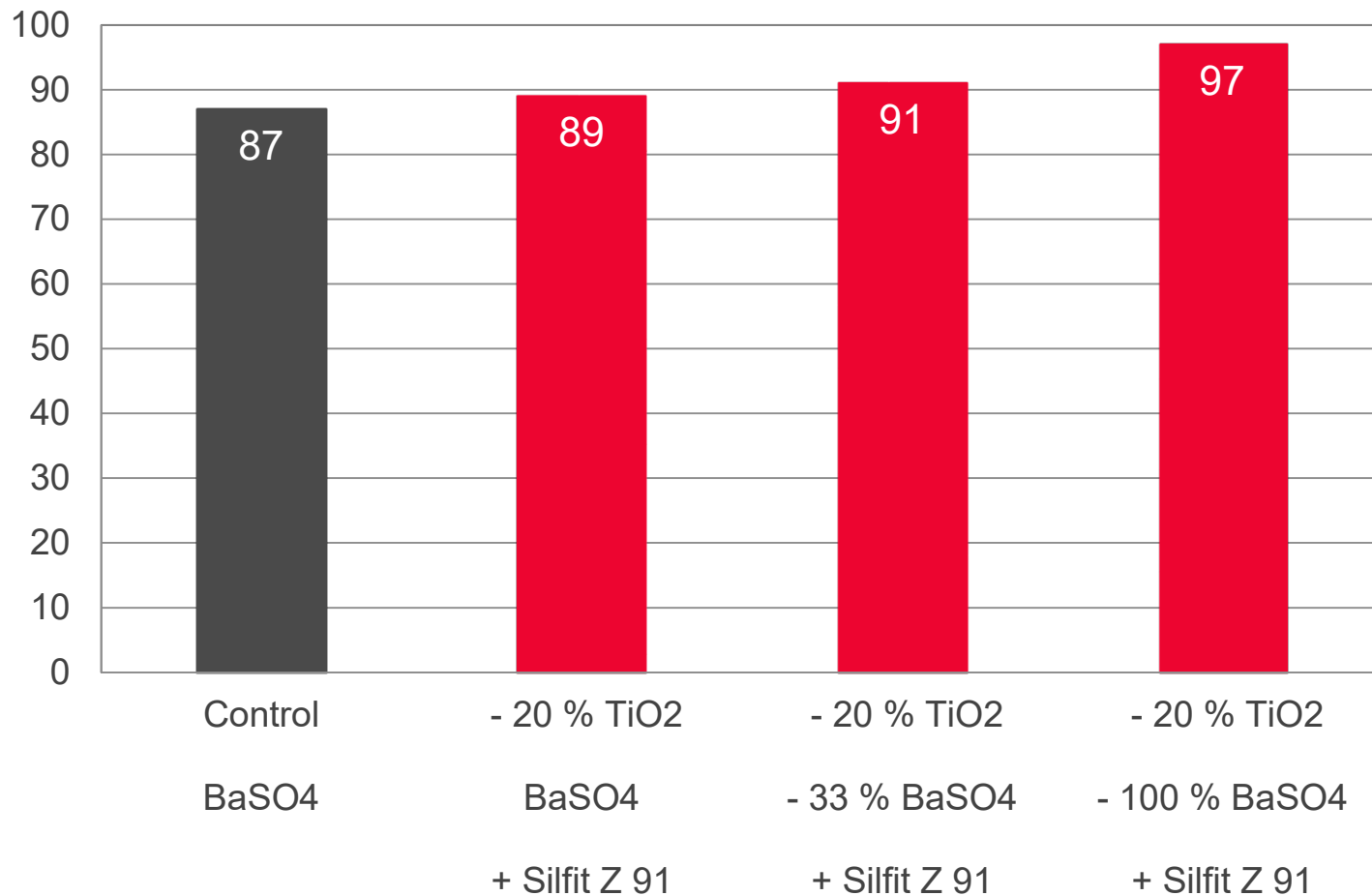
EXPERIMENTAL

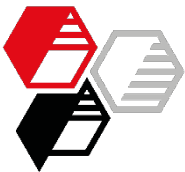
RESULTS

• BaSO₄ natural

SUMMARY

[units]





Leveling

Appearance of surface (visual assessment)

Substrate: aluminum A 48

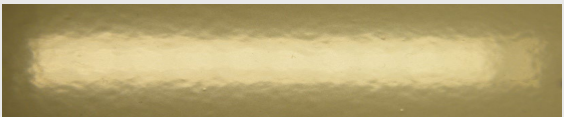
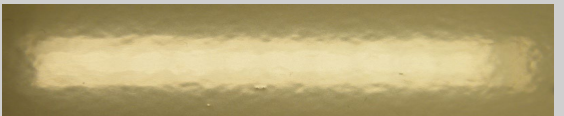
INTRODUCTION

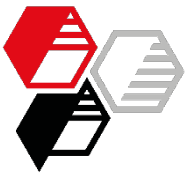
EXPERIMENTAL

RESULTS

• BaSO₄ natural

SUMMARY

	Reflection of overhead light
Control BaSO ₄	 0
- 20 % TiO ₂ BaSO ₄ + Silfit Z 91	 0
- 20 % TiO ₂ - 33 % BaSO ₄ + Silfit Z 91	 0+
- 20 % TiO ₂ - 100 % BaSO ₄ + Silfit Z 91	 +



Flexibility

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

INTRODUCTION

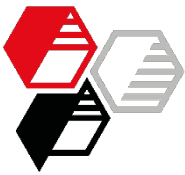
EXPERIMENTAL

RESULTS

• BaSO₄ natural

SUMMARY

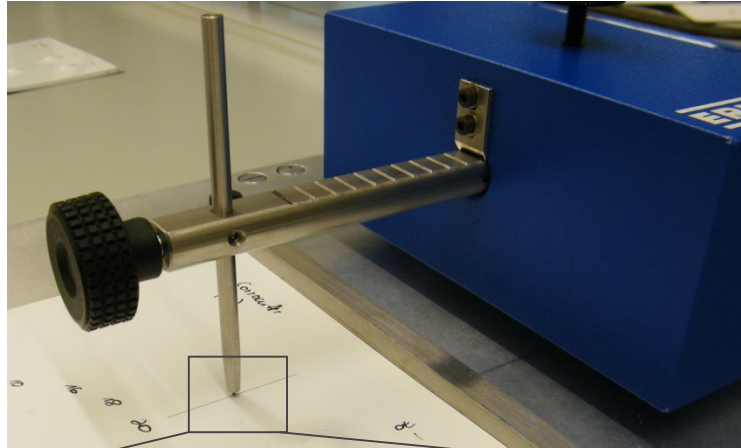
	Reverse Impact Test [inch pounds]	Cupping Test [mm]
Control BaSO ₄	18	6.9
- 20 % TiO ₂ BaSO ₄ + Silfit Z 91	14	6.4
- 20 % TiO ₂ - 33 % BaSO ₄ + Silfit Z 91	12	6.7
- 20 % TiO ₂ - 100 % BaSO ₄ + Silfit Z 91	28	6.4



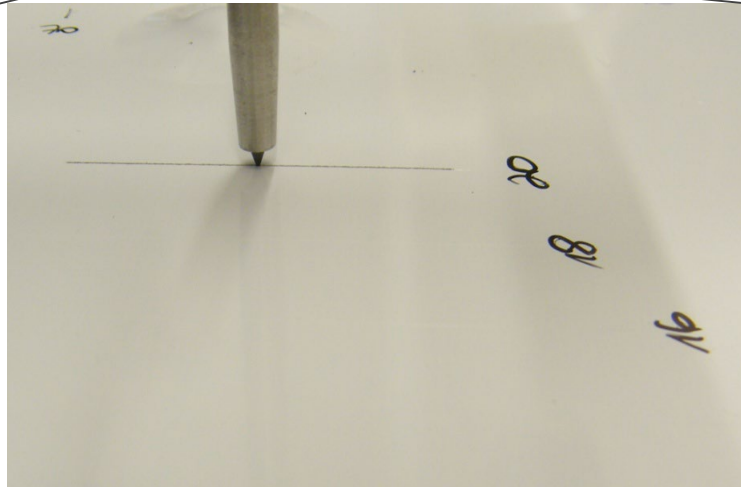
Mechanical Resistance

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Mechanical resistance is tested by scratching the coating down to the substrate with a hard metal tip (substrate: aluminum A 48)



Testing equipment (Erichsen Corrocutter, model 639) with loaded weight, force from 2 – 20 Newton



Round hard metal tip
(van Laar, diameter 0.5 mm)

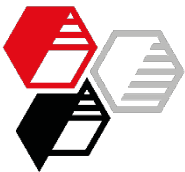
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• BaSO₄ natural

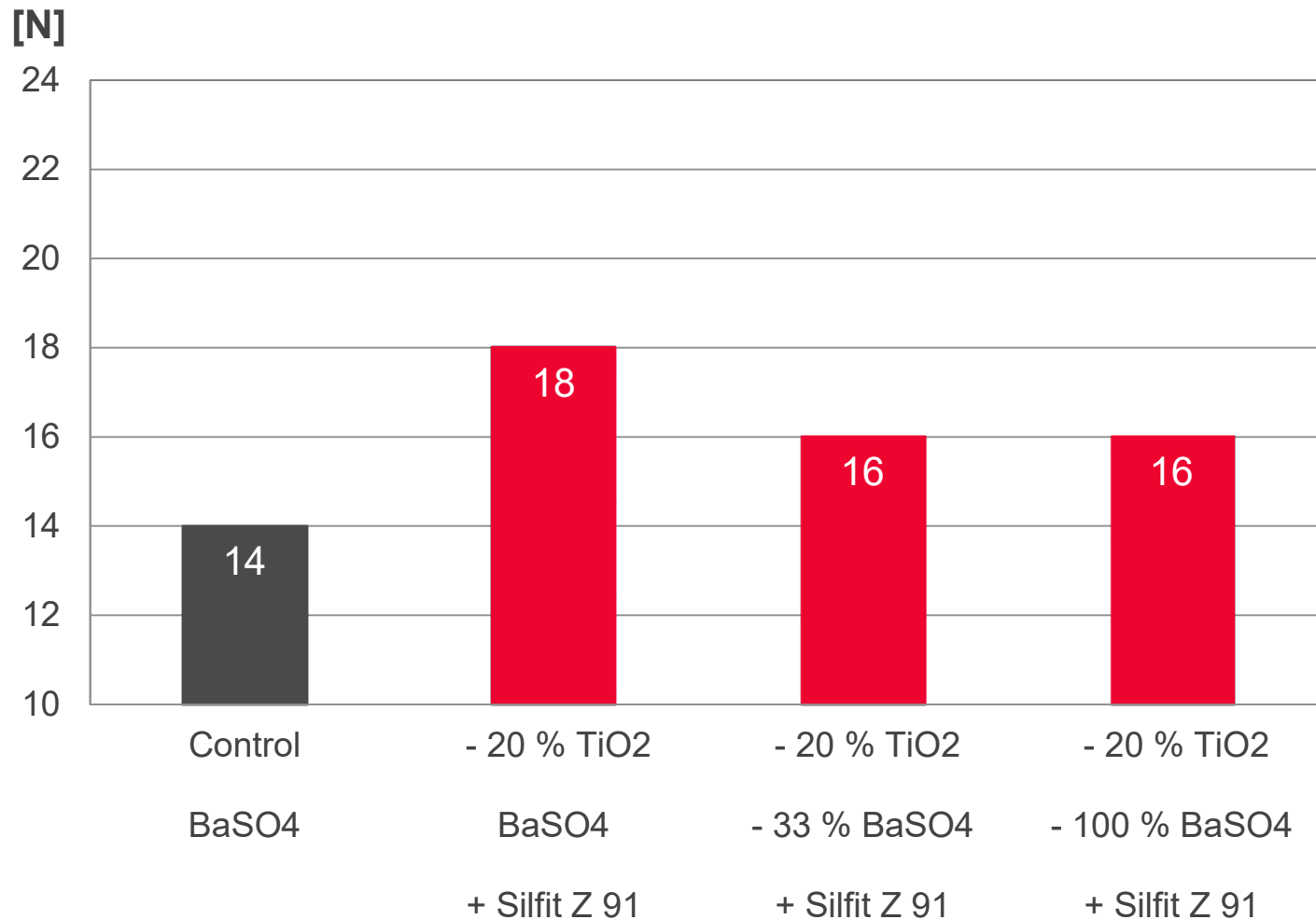
SUMMARY

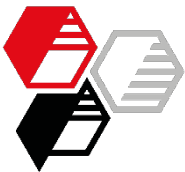


Mechanical Resistance

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Scratch test with Corrocutter



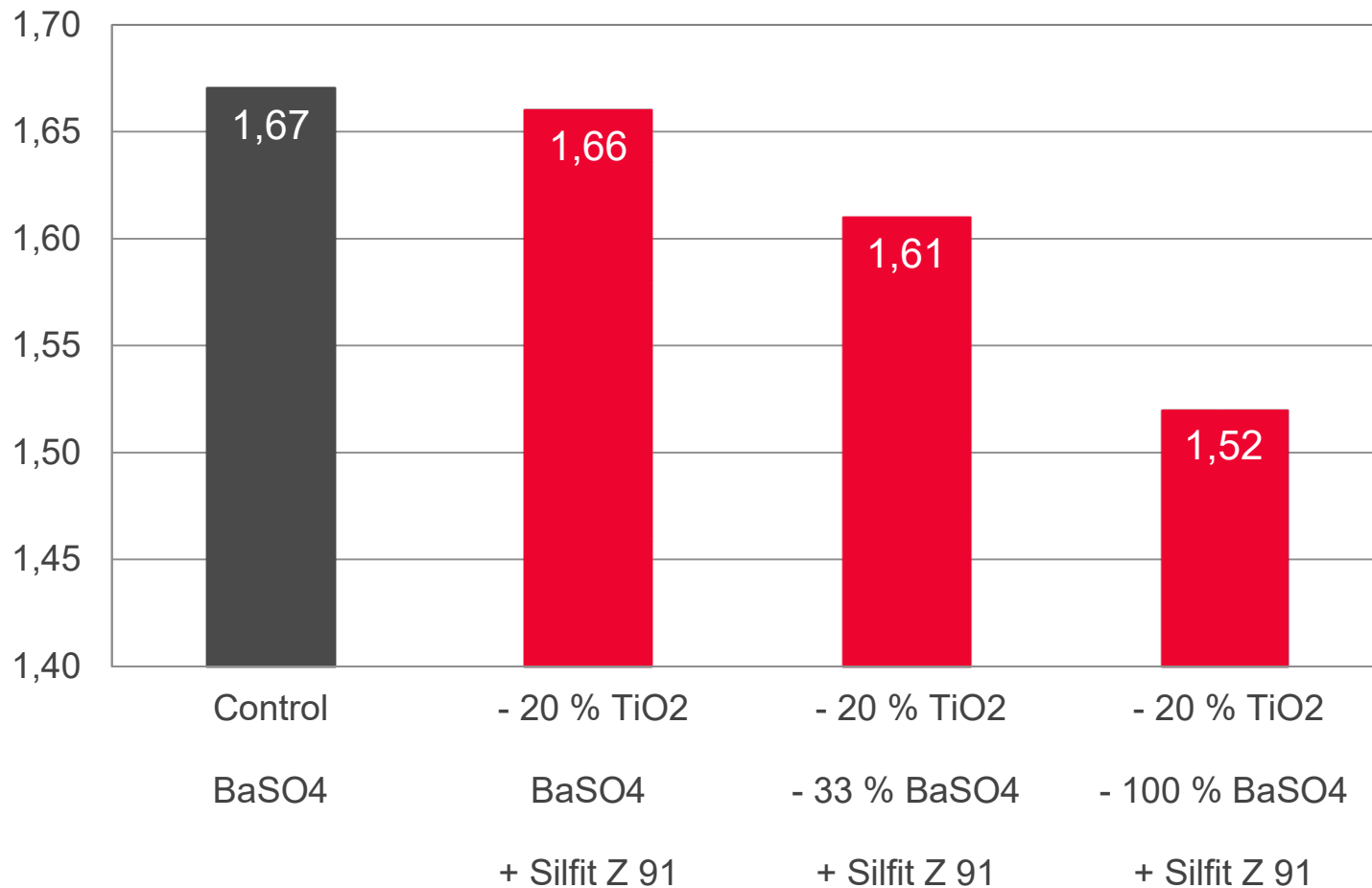


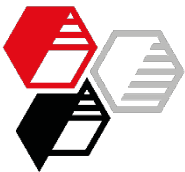
Density

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calculated

[g/cm³]

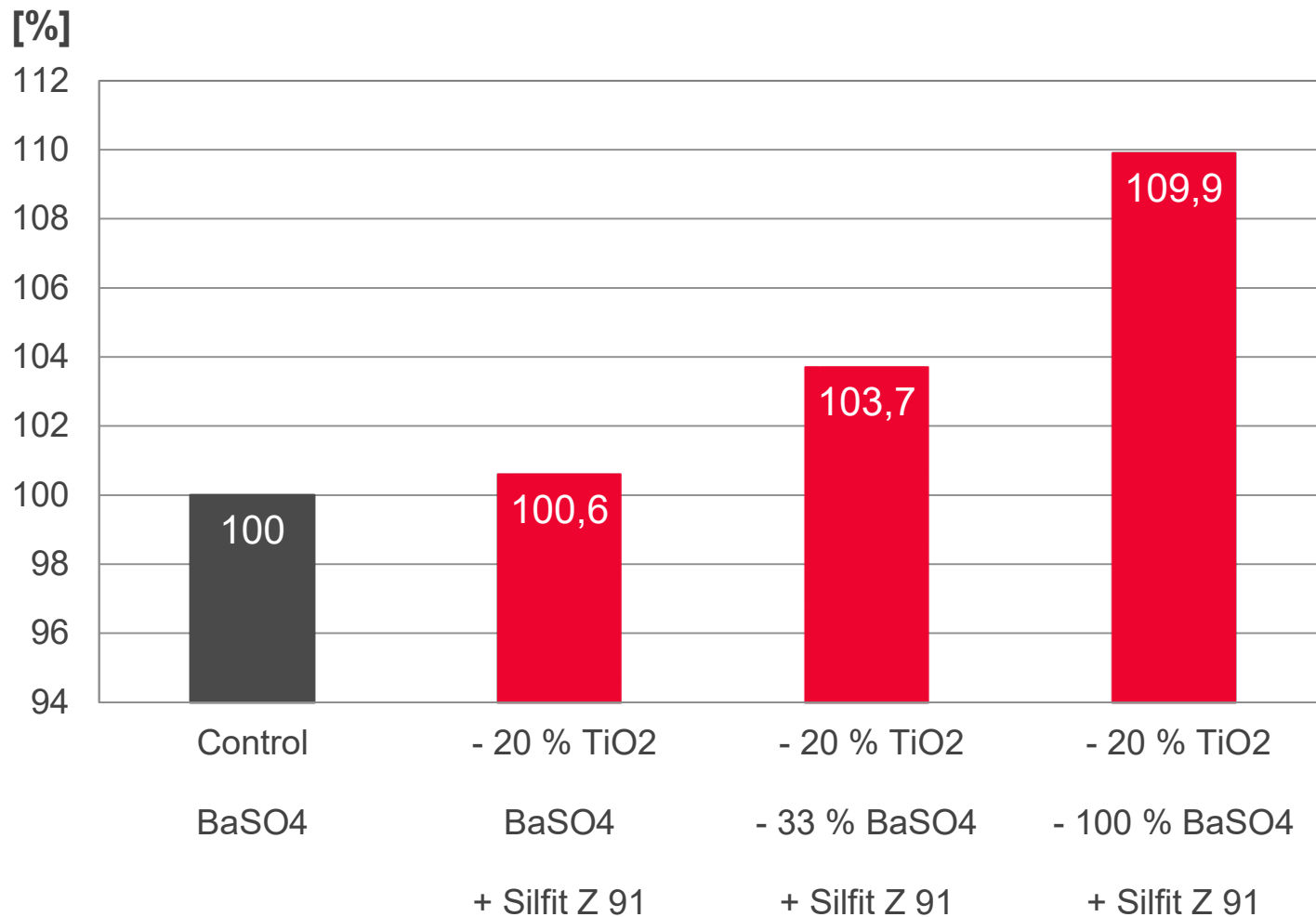


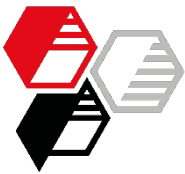


Spreading Rate

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

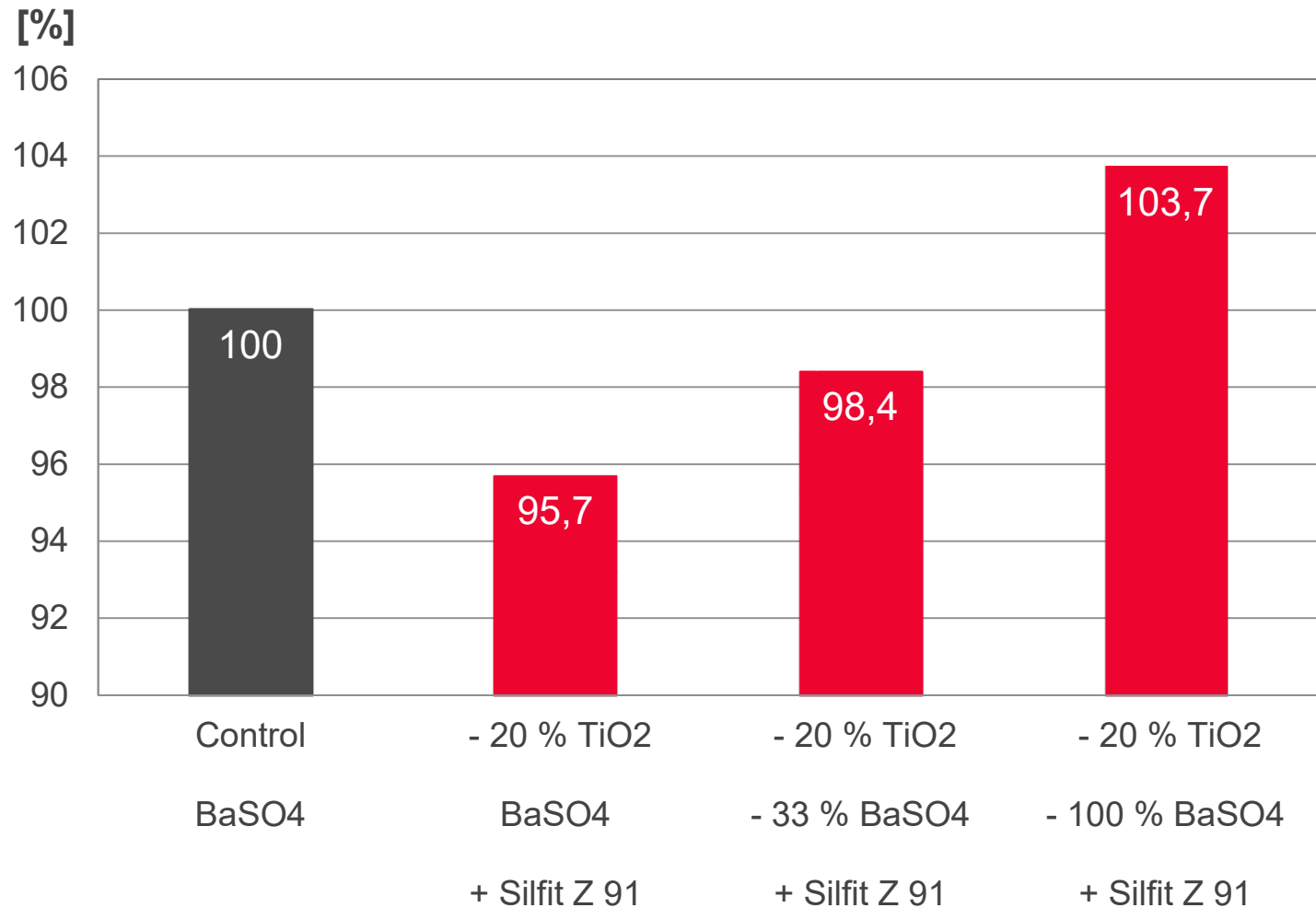


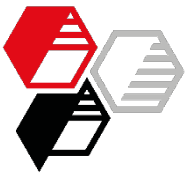


Cost Index Based on Weight

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Control = 100 % (Base: Germany 2011)

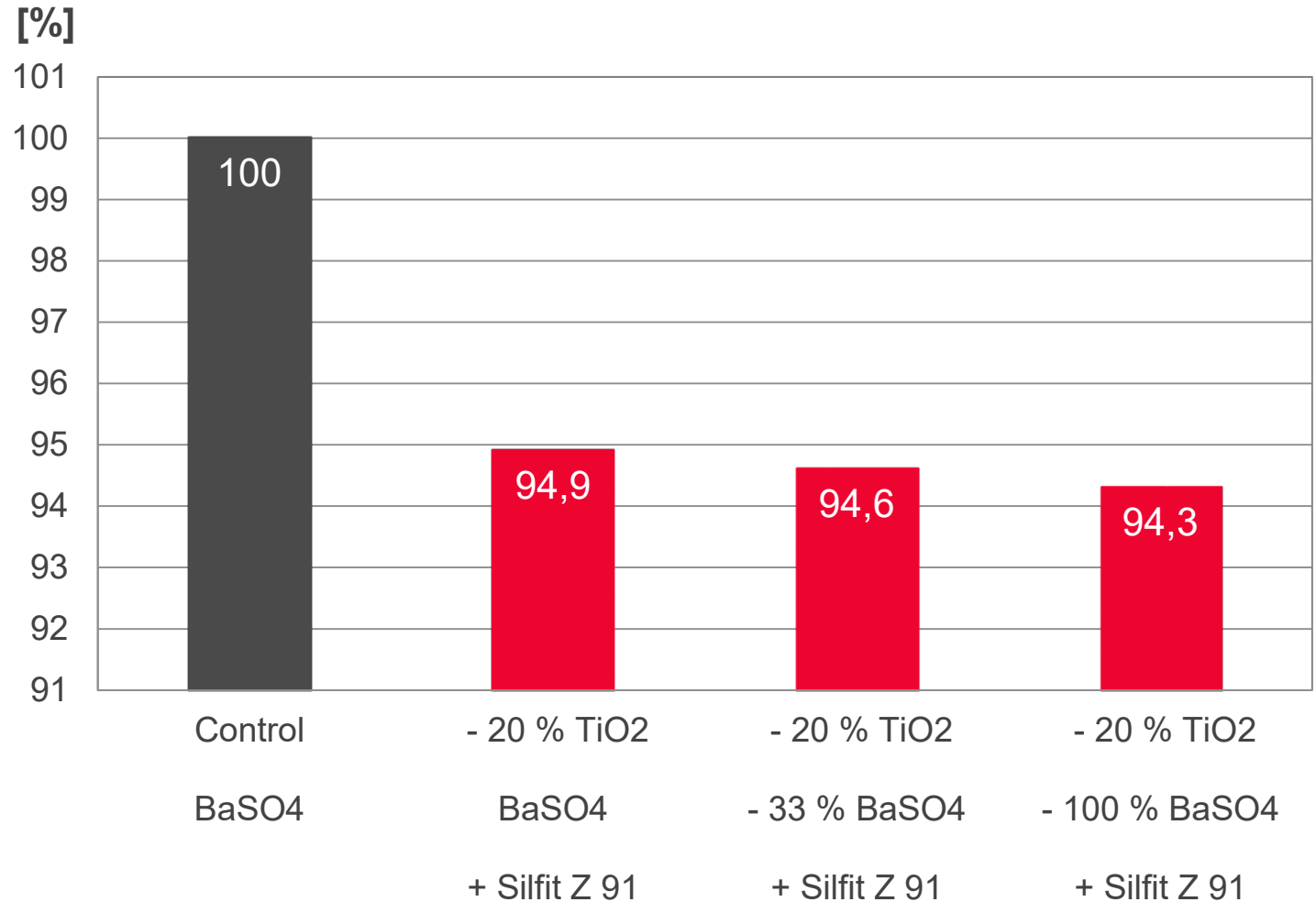


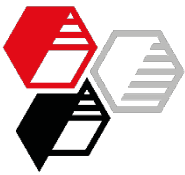


Cost Index Based on Volume

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Control = 100 % (Base: Germany 2011)





Summary

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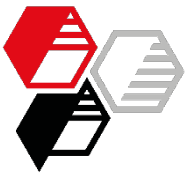
• BaSO₄ natural

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

- similar optical properties and flexibility
- + improved scratch resistance
- + cost reduction potential

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

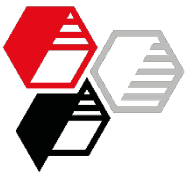


We supply material for good ideas!

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Formulations

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Parts per weight

INTRODUCTION

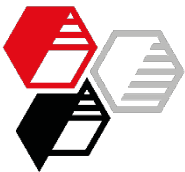
EXPERIMENTAL

• BaSO₄ ppt

RESULTS

SUMMARY

	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



Formulations

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Parts per cent (%)

INTRODUCTION

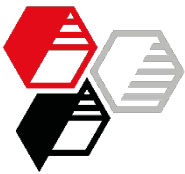
EXPERIMENTAL

• BaSO₄ ppt

RESULTS

SUMMARY

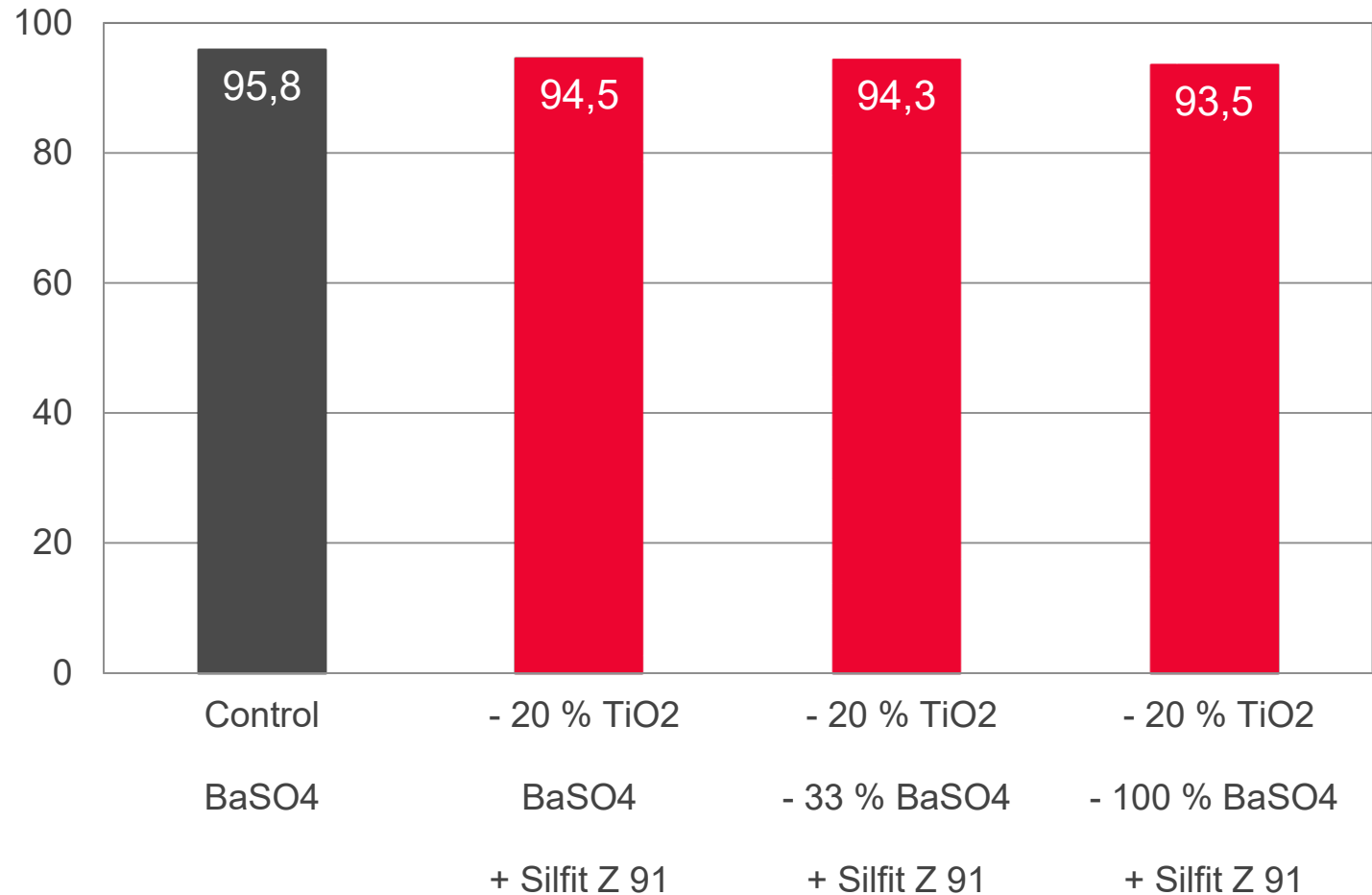
	Control BaSO ₄	- 20 % TiO ₂ BaSO ₄ + Silfit Z 91	- 20 % TiO ₂ - 33 % BaSO ₄ + Silfit Z 91	- 20 % TiO ₂ - 100 % BaSO ₄ + Silfit Z 91
Crylcoat 1771-3	39.0	38.9	40.0	42.4
Epikote 1003	18.0	18.0	18.5	19.6
Additol P 896	3.0	3.2	3.3	3.5
Titanium dioxide	19.5	15.6	16.0	16.9
BaSO ₄ ppt	20.0	20.0	13.7	-
Silfit 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

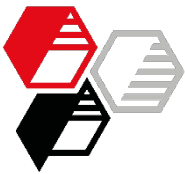


Color Brightness L*

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CIE L*



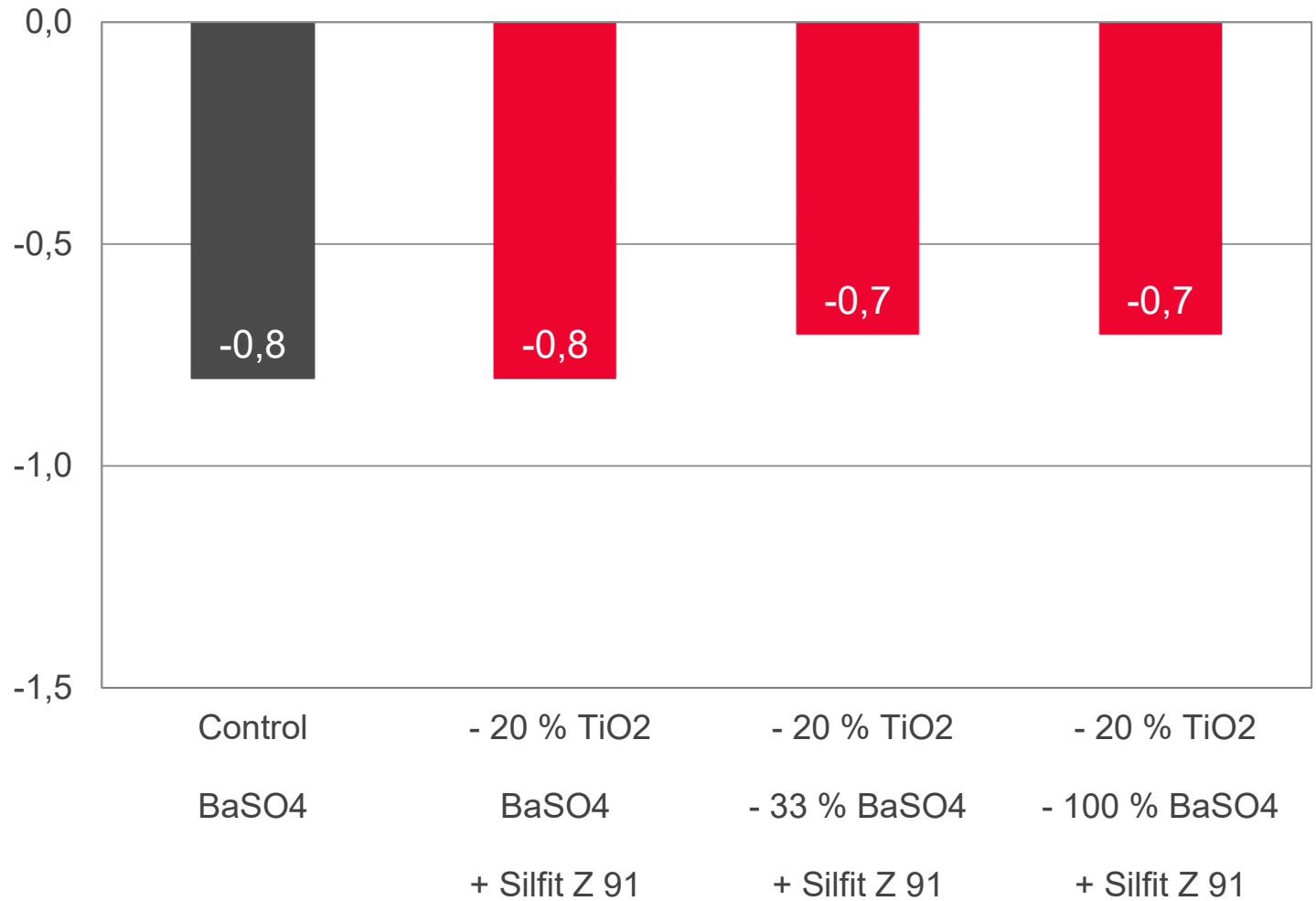


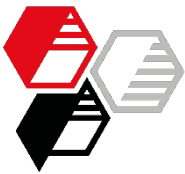
Color

Red/green-ratio a^*

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CIE a^*



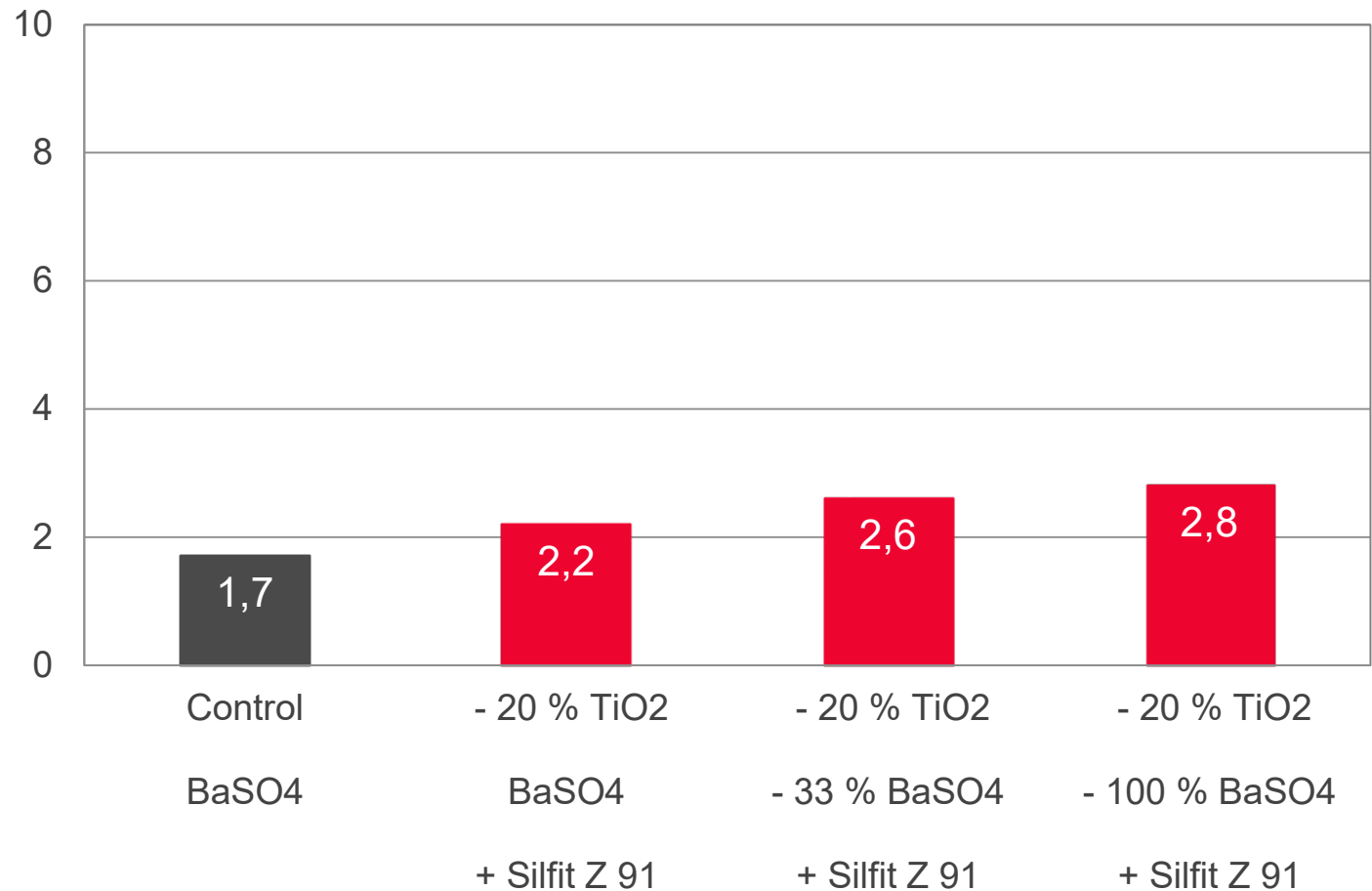


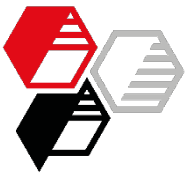
Color

Yellow/blue-ratio b^*

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CIE b^*

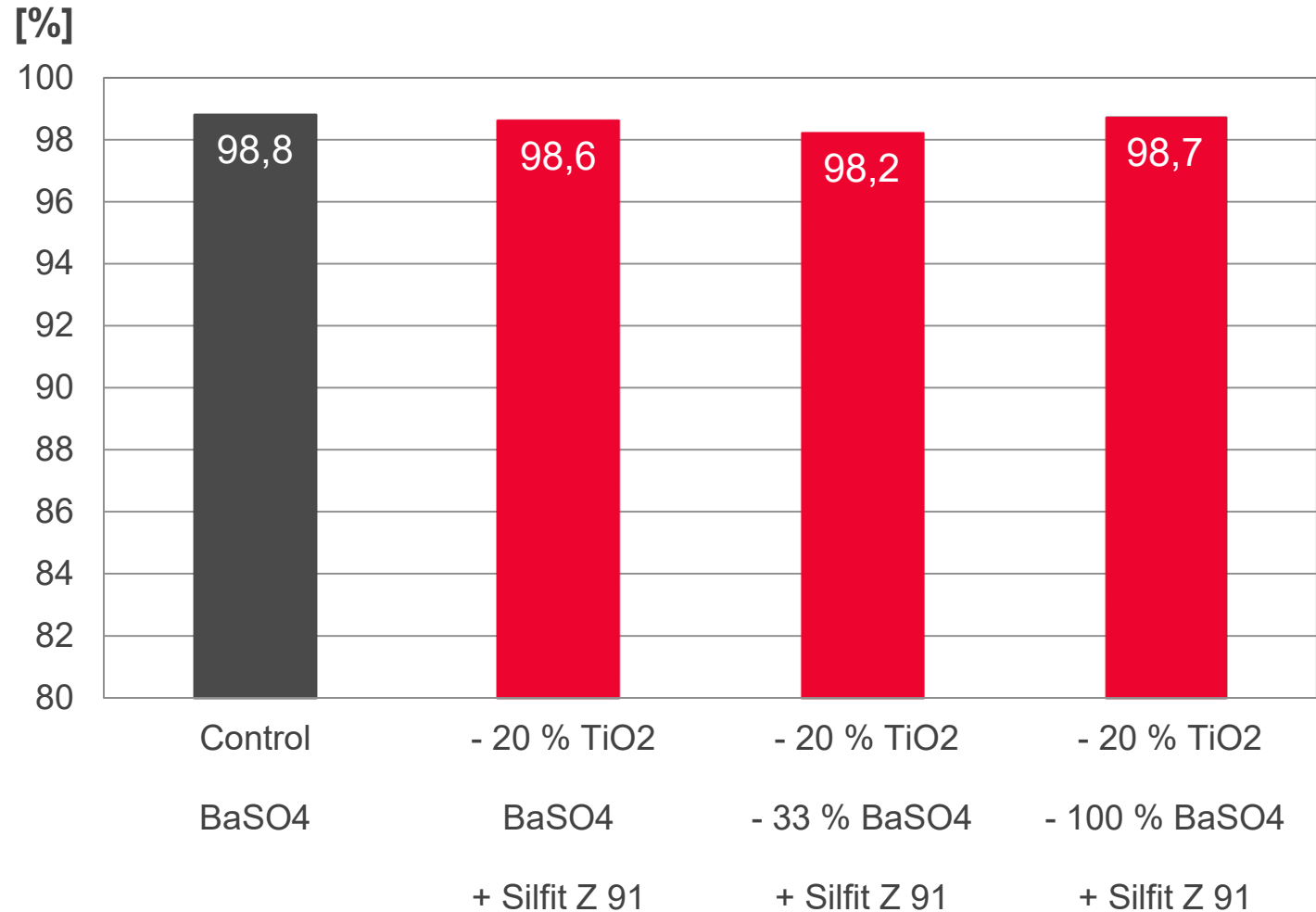


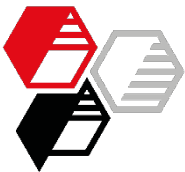


Hiding Power / Opacity

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Contrast ratio at a dry film thickness ~ 70 µm





Gloss 20°

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INTRODUCTION

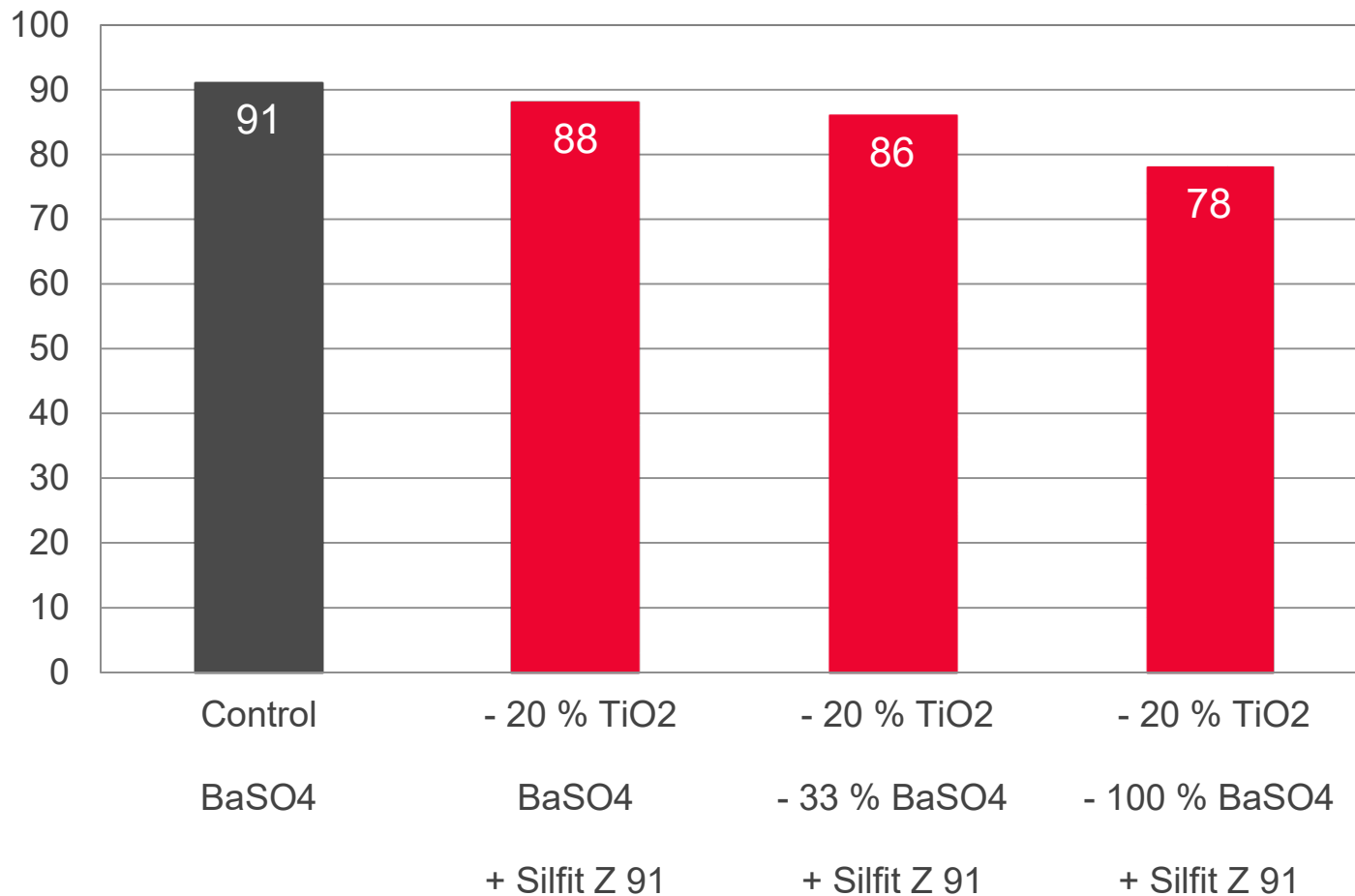
EXPERIMENTAL

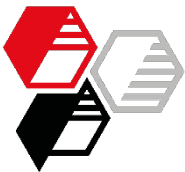
RESULTS

• BaSO₄ ppt

SUMMARY

[units]





Haze

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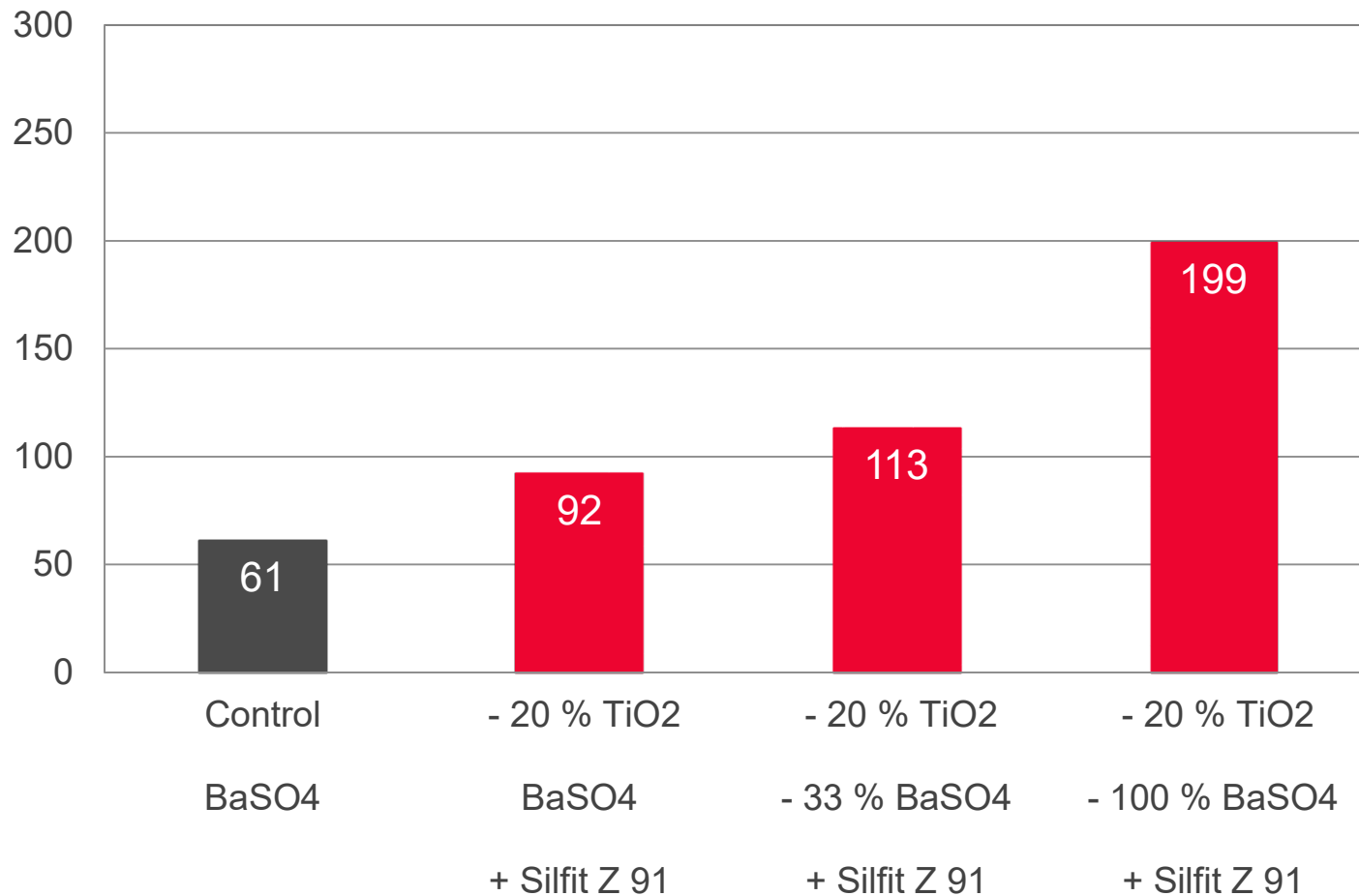
EXPERIMENTAL

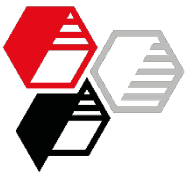
RESULTS

• BaSO₄ ppt

SUMMARY

[units]





Gloss 60°

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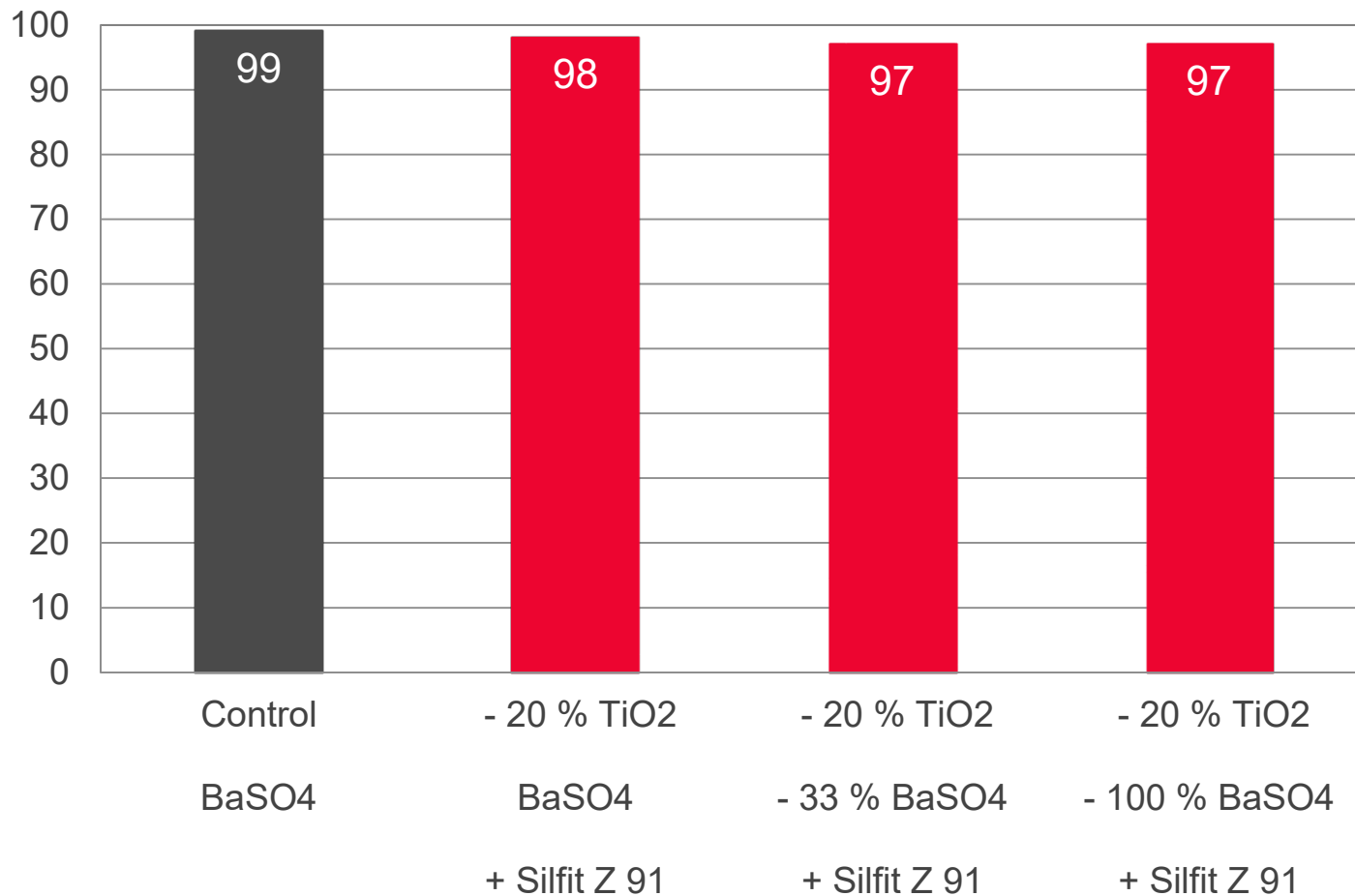
EXPERIMENTAL

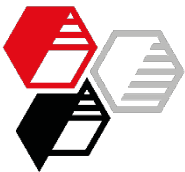
RESULTS

• BaSO₄ ppt

SUMMARY

[units]





Leveling

Appearance of surface (visual assessment)

Substrate: aluminum A 48

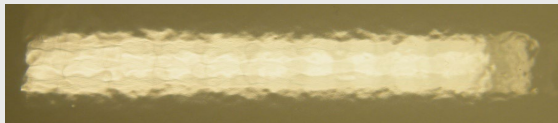
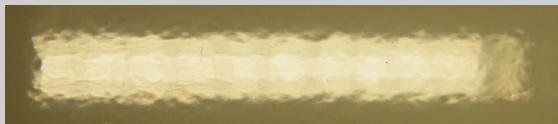
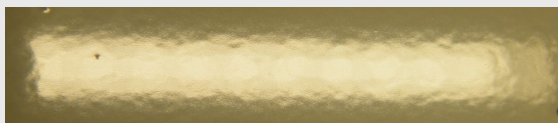
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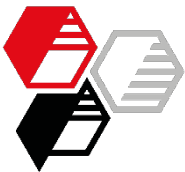
EXPERIMENTAL

RESULTS

• BaSO₄ ppt

SUMMARY

	Reflection of overhead light
Control BaSO ₄	 0
- 20 % TiO ₂ BaSO ₄ + Silfit Z 91	 0
- 20 % TiO ₂ - 33 % BaSO ₄ + Silfit Z 91	 0
- 20 % TiO ₂ - 100 % BaSO ₄ + Silfit Z 91	 0-



Flexibility

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

INTRODUCTION

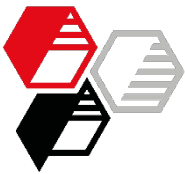
EXPERIMENTAL

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• BaSO₄ ppt

SUMMARY

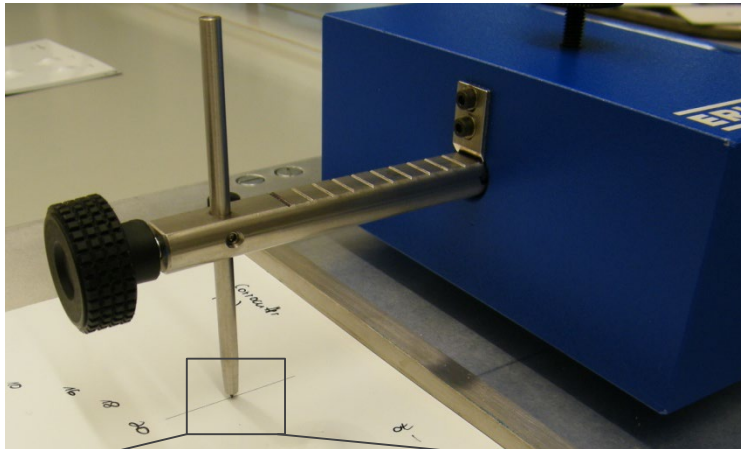
	Reverse Impact Test [inch pounds]	Cupping Test [mm]
Control BaSO ₄	16	6.8
- 20 % TiO ₂ BaSO ₄ + Silfit Z 91	10	7.0
- 20 % TiO ₂ - 33 % BaSO ₄ + Silfit Z 91	14	6.6
- 20 % TiO ₂ - 100 % BaSO ₄ + Silfit Z 91	28	6.4



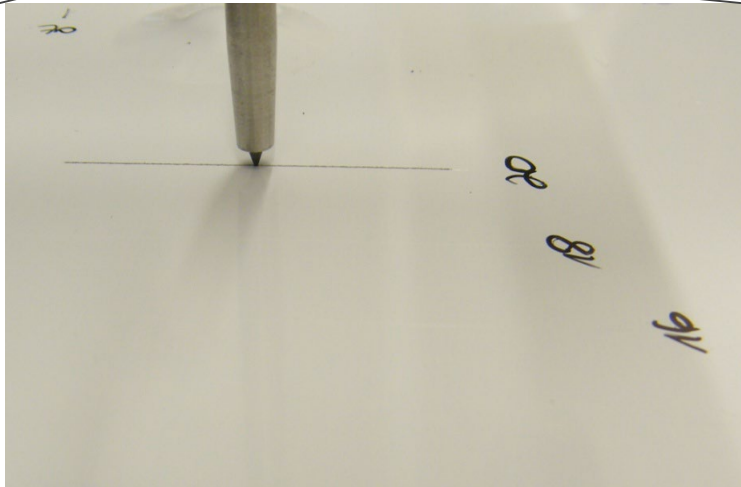
Mechanical Resistance

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Mechanical resistance is tested by scratching the coating down to the substrate with a hard metal tip (substrate: aluminum A 48)



Testing equipment (Erichsen Corrocutter, model 639) with loaded weight, force from 2 – 20 Newton



Round hard metal tip
(van Laar, diameter 0.5 mm)

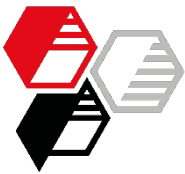
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• BaSO₄ ppt

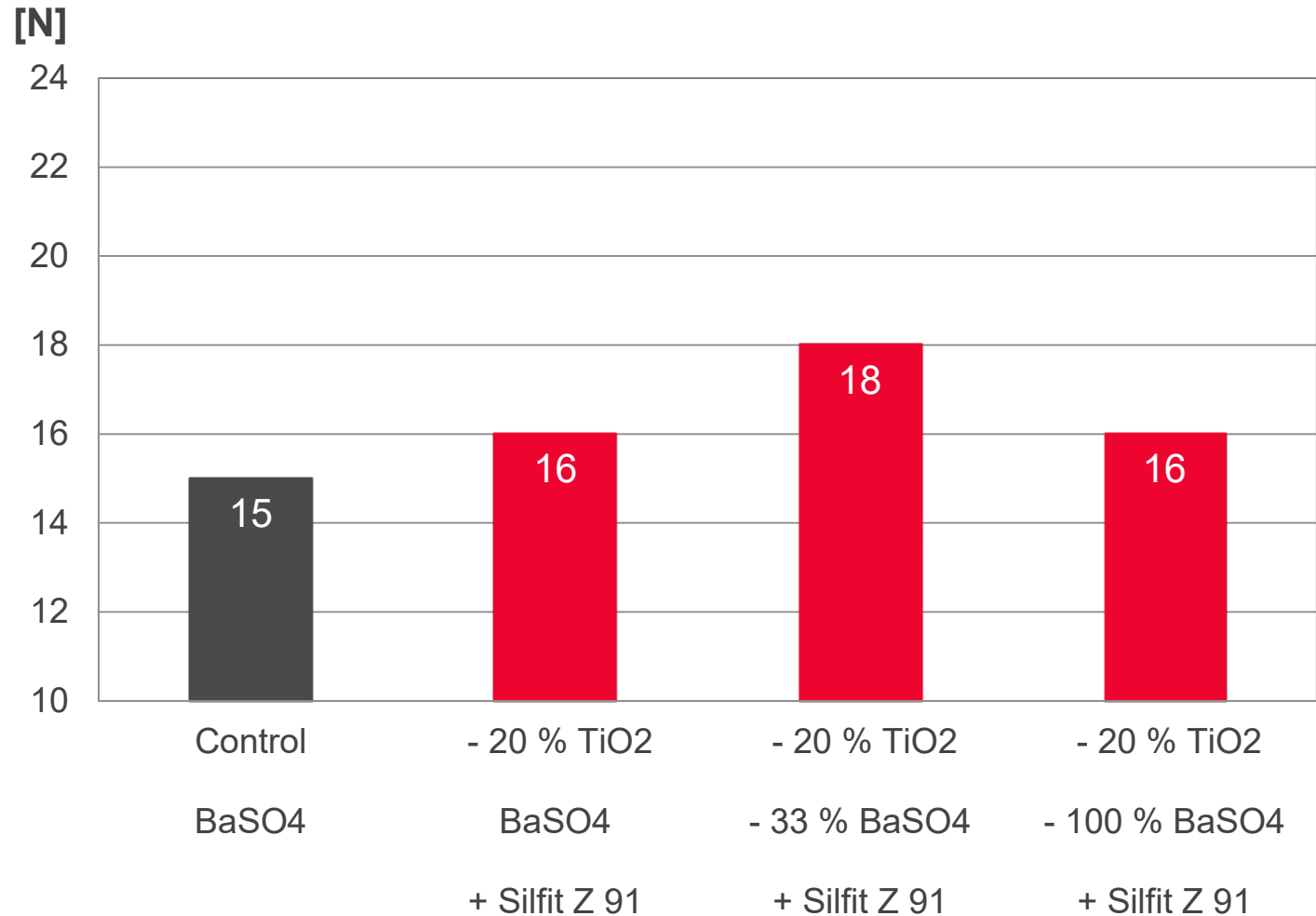
SUMMARY

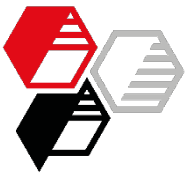


Mechanical Resistance

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Scratch test with Corrocutter



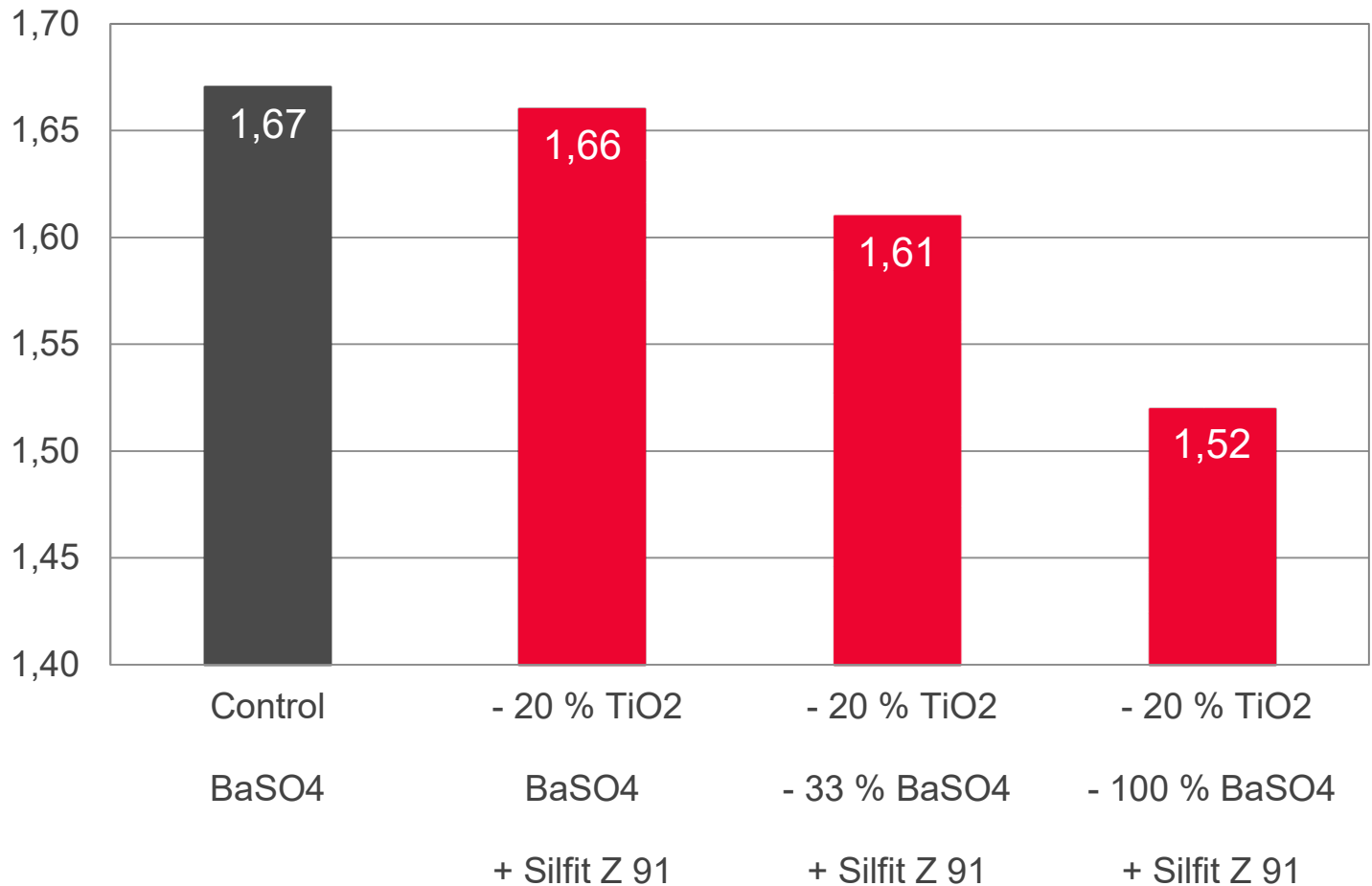


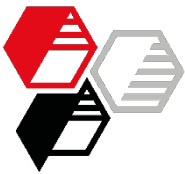
Density

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calculated

[g/cm³]

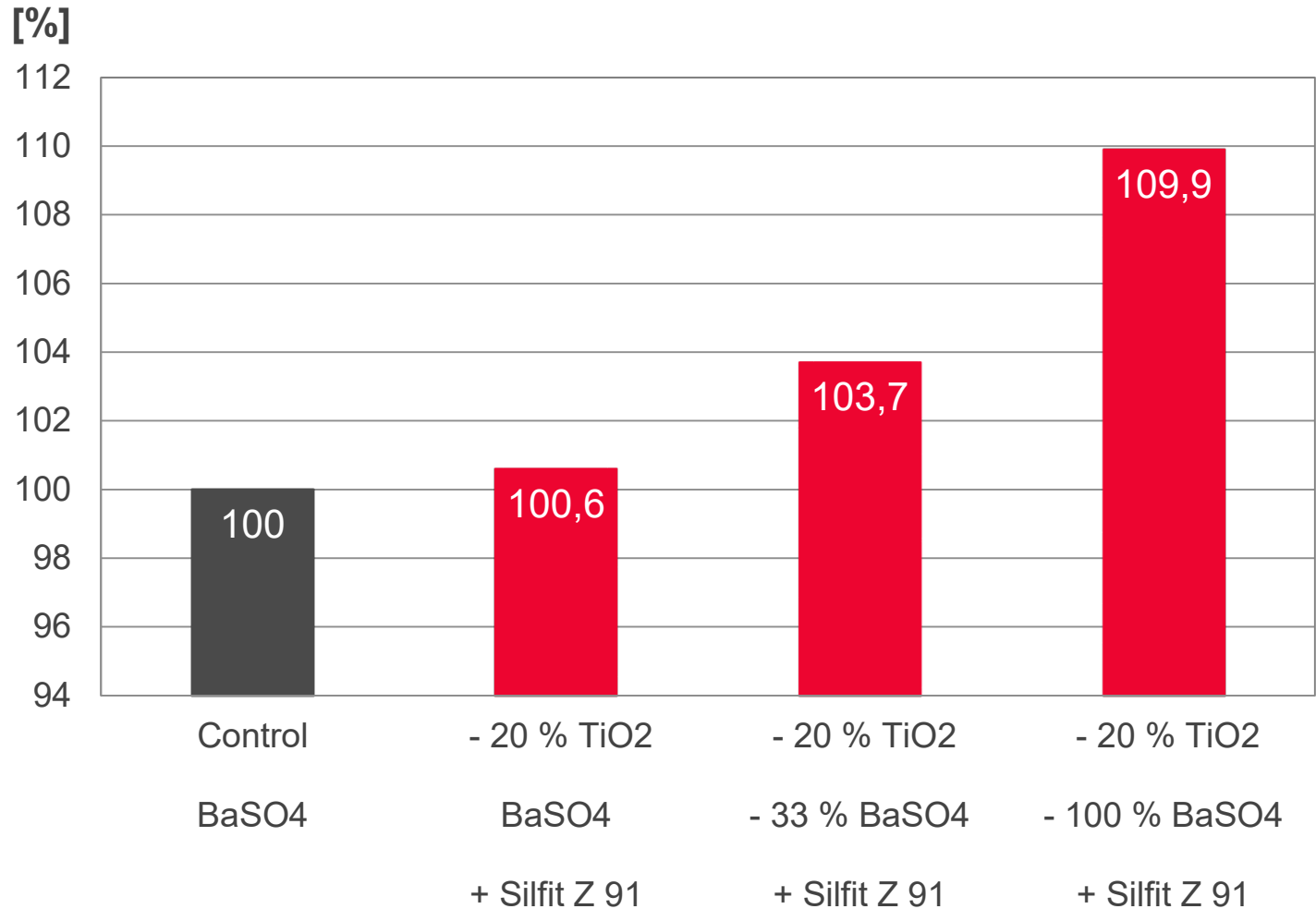


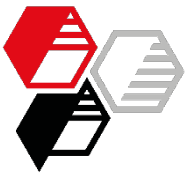


Spreading Rate

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

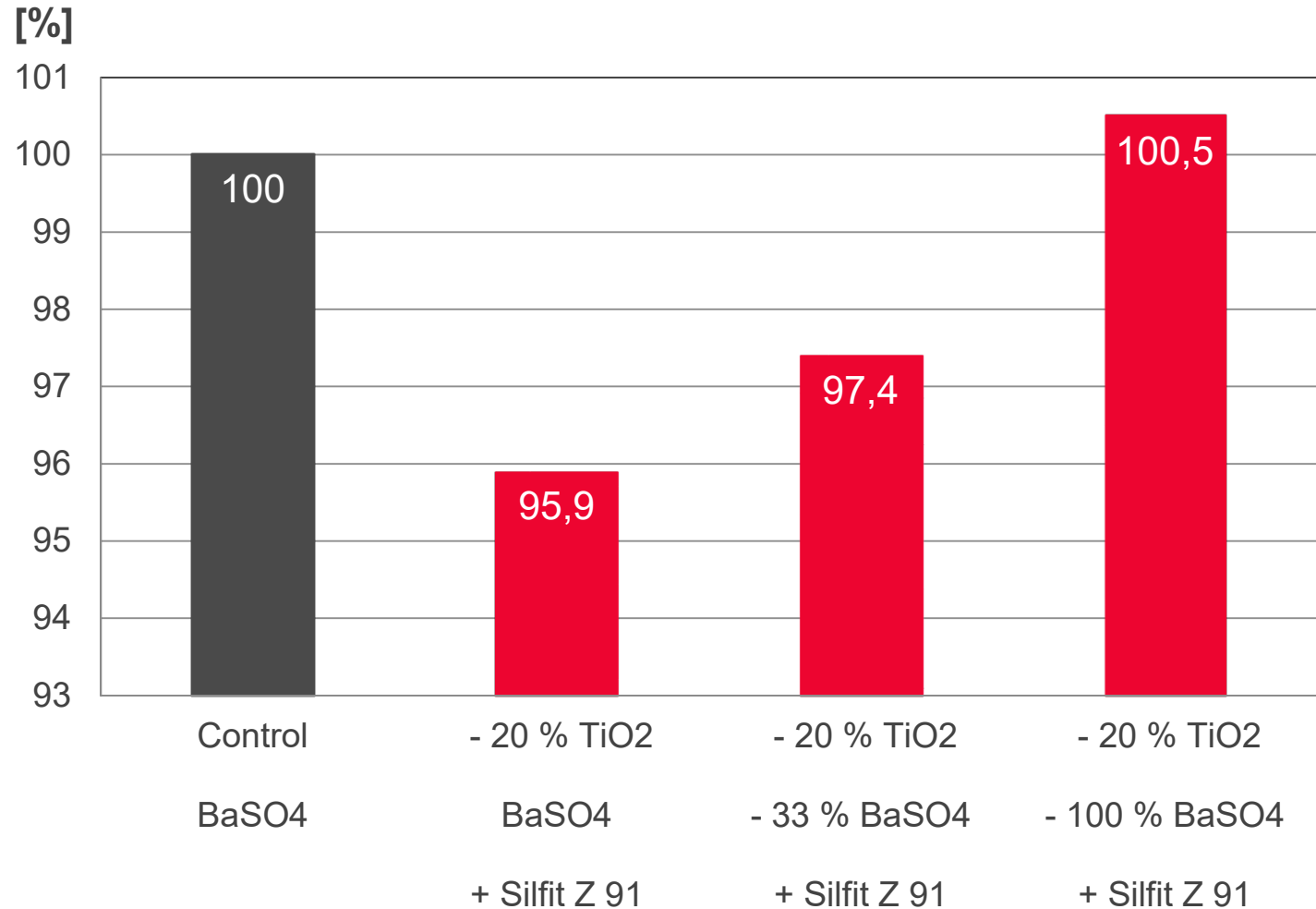


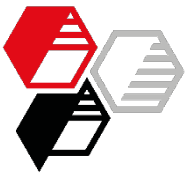


Cost Index Based on Weight

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Control = 100 % (Base: Germany 2011)

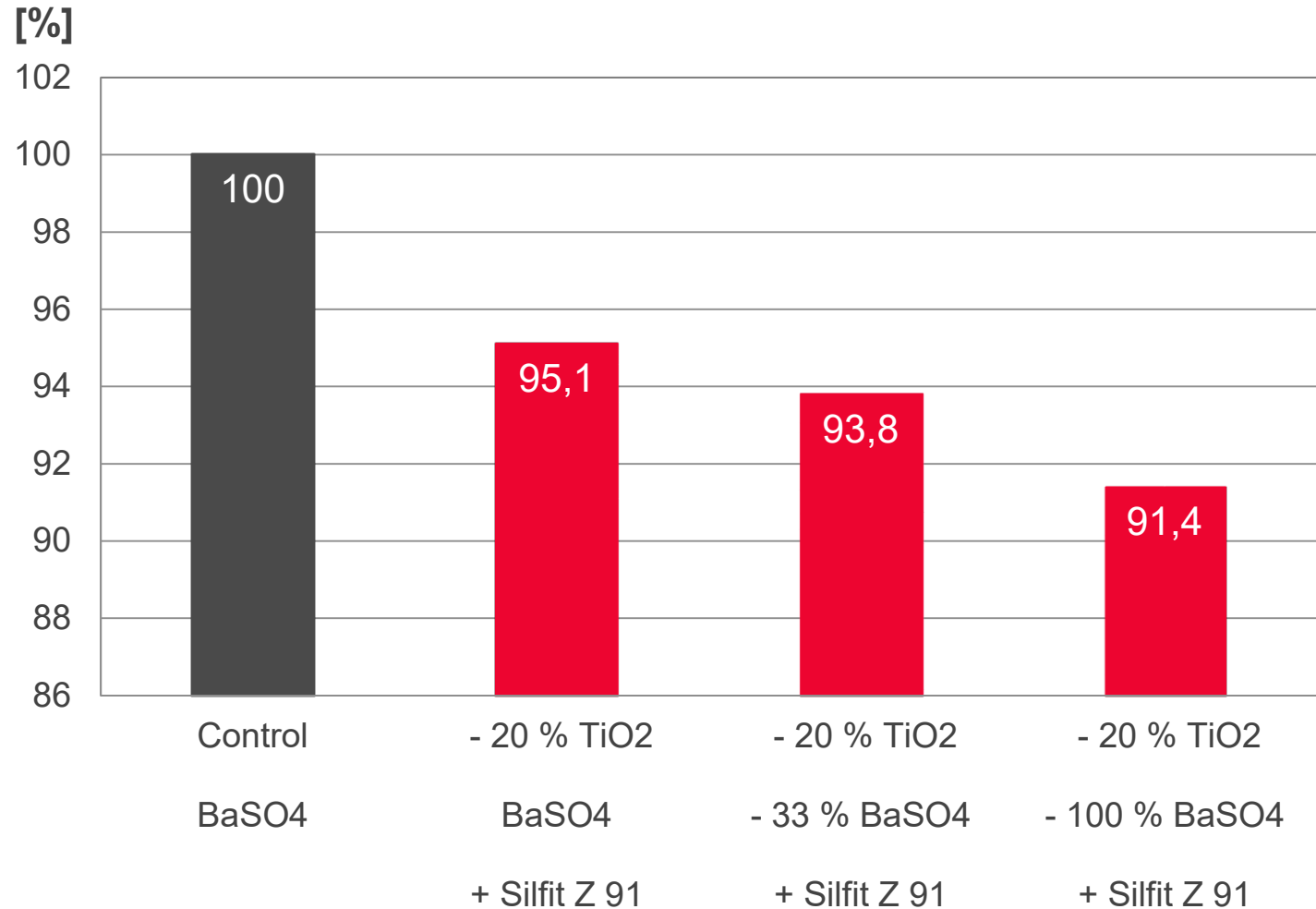


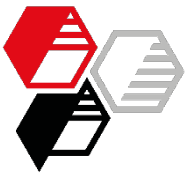


Cost Index Based on Volume

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Control = 100 % (Base: Germany 2011)





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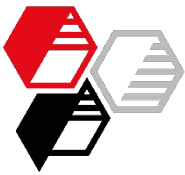
• BaSO₄ ppt

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 and flexibility
- + slightly improved scratch resistance
- + cost reduction potential

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

- + higher scratch resistance
- + higher spreading rate (lower density of powder coating)
- + cost reduction potential

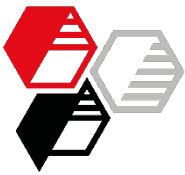


Conclusion

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 losing hiding power. The scratch resistance is enhanced and the costs lowered.

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

Additional partial substitution (33 %) of the precipitated barium sulfate by **Silfit Z 91** improved furthermore the scratch resistance, 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 Abrasiveity [mg]	51	54	54	54	62

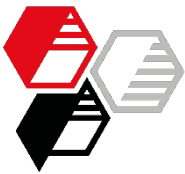
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Abrasivity Einlehner-Test

**HOFFMANN
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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 Abrasiveity [mg]	51	54	54	54	62

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