Neuburg Siliceous Earth: Functional filler for epoxy powder pipeline coatings

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Introduction

EXPERIMENTAL

RESULTS

- Application Properties
- Optical Properties
- Mechanical Properties
- Resistance Properties

SUMMARY

The project was carried out at the iLF in Magdeburg, Germany under the supervision of Dipl. Ing. Markus Witter, between November 2005 and May 2006.

The objective of the study was to evaluate the property profile of Neuburg Siliceous Earth in comparison with other functional fillers which are usually incorporated in powder coatings.

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Experimental

- Base formulation
- Fillers and their characteristics
- Preparation of the coatings and application

Results

- Fluidizability (ISO 8130-8)
- Deposition efficiency (ISO 8130-10)
- Run-off
- Gloss at 60°

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Tests on sandblasted steel (Surface roughness 50-70 µm), film thickness 300-600 µm

- visual assessment of the surface appearance
- Impact / reverse impact strength (DIN EN ISO 6272)
- cupping test (DIN EN ISO 1520)
- mandrel bending test (DIN EN ISO 1519)
- abrasion loss (Taber) - ASTM D 4060 (abrader wheels CS 17) and DIN 53754 (abrasive strips S-42)
- salt spray test (DIN EN ISO 9227)
- humidity test (DIN EN ISO 6270-2)
- chemical resistance
- 1700 h water immersion at 90 °C (DIN EN ISO 2812-2)

Formulation

Base formulation (from Hexion)

		Parts per weight
Epikote 1055	Solid epoxy resin	827
Epi-cure Curing Agent P-104	Hardener	33
BYK 368-P	Levelling agent	10
Iron oxide red 3296	Color pigment	15
Barite	Filler	120
Total		1005

Density		1.32
PVC (%)		4.2

Formulation

Filler variations

EXPERIMENTAL

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SUMMARY

Without filler										
Barite		120								
Sillitin Z 86			74							
Barite				240						
Sillitin Z 86					149					
Aktisil AM					149					
Aktisil MM					149					
(NCC) Natural Calcium Carbonat						154				
Wollastonite							163			
Clay (Kaolin)								149		
Mica									162	
Talc										159
PVC (%)	0.4	4.2	4.2	7.6	7.6	7.6	7.6	7.6	7.6	7.6

Filler characteristics

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SUMMARY

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

Test design

Preparation:

- Premix and extrusion in a twin screw extruder (MP 19, APV Baker)
- D = 19 mm; L = 25 D
- Screw speed 300 min^{-1}
- Grind at 16000 min^{-1} and screening through 100 µm mesh

Application:

- Onto Q-Panel R-36 sheets with Corona powder spray gun (70 kV) for optical assessments
- Onto blasted metal sheets (50-70 µm surface roughness; before application pre-heated to 200 °C) via tribo application for mechanical tests (except Martens hardness)

Curing at 200 °C subject temperature for 10 min

Results

The following tests did not effect any significant filler effects:

- Gel time (ISO 8130-6) results between 40 and 48 s
- Melt viscosity (EN ISO 3219)
- Martens hardness (EN ISO 14577) on Q-Panel R 36 sheets; film thickness 100-150 µm; results between 140 - 160 N/mm²
- Cross cut test (DIN EN ISO 2409): Gt 0 on blastet metal sheets (film thickness 300-600 µm) also after humidity test and water immersion at 90° C
- Cathodic disbondment (DIN EN ISO 15711); 0-1 mm, poorer results were only obtained with the filler-free formulation (about 10 mm)
- Chemical resistance:
- 1000 h H_2SO_4 (10 %) at 23 °C (DIN EN ISO 2812-1)
- 1500 h NaOH (10 %) at 23 °C (DIN EN ISO 2812-1)

Fluidizability ISO 8130-8

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SUMMARY

		Assessment of Fluidizability
PVC 0.4	Without filler	+
PVC 4.2	Barite	0
	Sillitin Z 86	+
PVC 7.6	Barite	0
	Sillitin Z 86	+
	Aktisil AM	+
	Aktisil MM	+
	NCC	-
	Wollastonite	0
	Clay	+
	Mica	0
	Talc	-

0 moderate (80 – 120)
– poor (< 80)
+ good (120 – 140)

Deposition efficiency
ISO 8130-10

EXPERIMENTAL

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- Application Properties
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SUMMARY

		Assessment of Deposition Efficiency	Results in %
PVC 0.4	Without filler	0-	7.5
PVC 4.2	Barite	0	9.3
	Sillitin Z 86	-	5.6
PVC 7.6	Barite	0	9.0
	Sillitin Z 86	0	8.9
	Aktisil AM	0	8.8
	Aktisil MM	0	9.7
	NCC	-	6.9
	Wollastonite	0-	7.8
	Clay	0	8.3
	Mica	0-	7.3
	Talc	+	13.6

Run-off

EXPERIMENTAL

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- Application Properties
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- Resistance Properties

SUMMARY

		Run-off tendency during Stoving
PVC 0.4	Without filler	-
PVC 4.2	Barite	0
	Sillitin Z 86	-
PVC 7.6	Barite	0
	Sillitin Z 86	+
	Aktisil AM	+
	Aktisil MM	+
	NCC	0
	Wollastonite	+
	Clay	-
	Mica	0
	Talc	++

0	moderate
–	high
+	marginal
++	no

Gloss at 60°

EXPERIMENTAL

RESULTS

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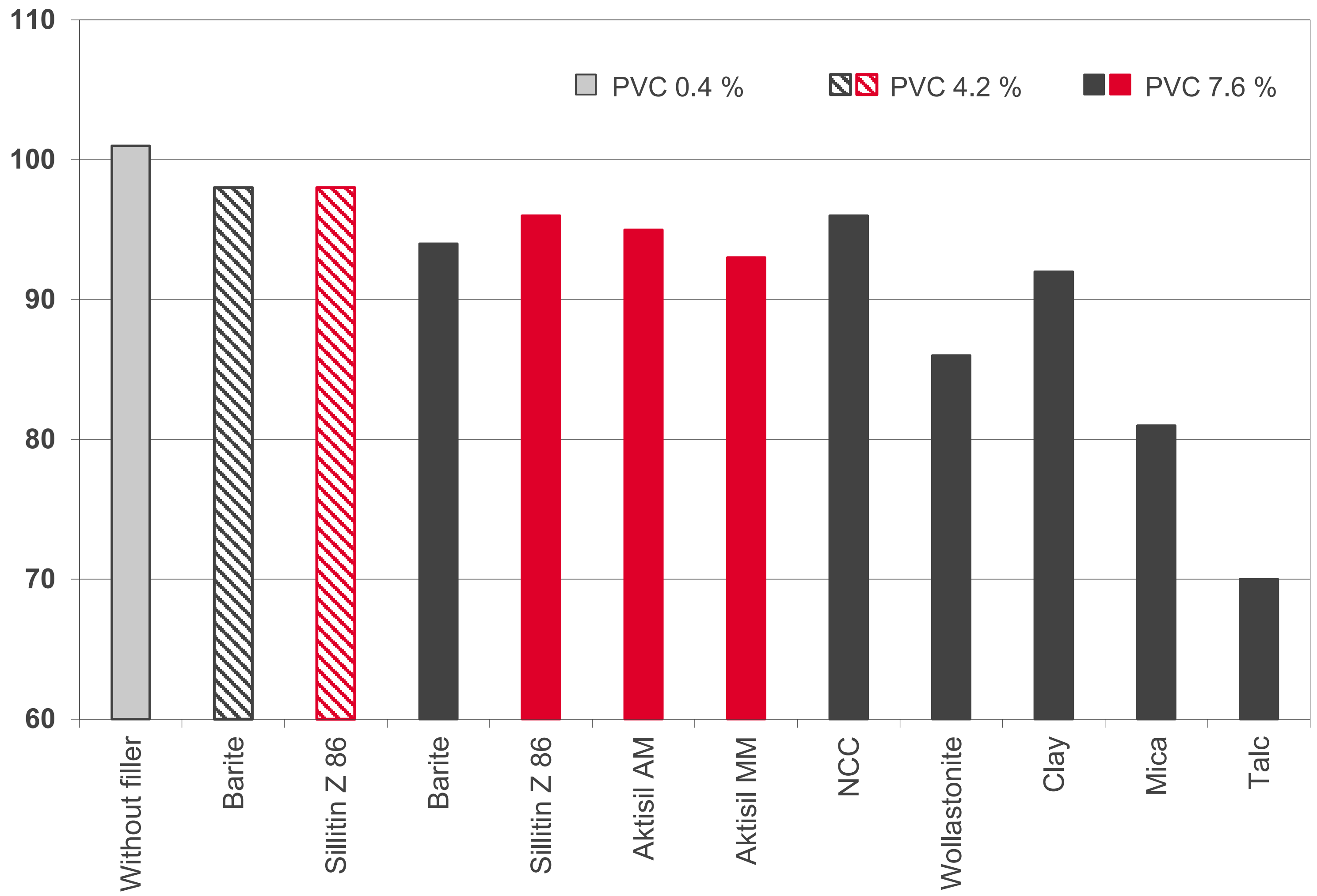
SUMMARY



Visual assessment of the surface appearance

PVC 0.4%:
Without filler

PVC 4.2%:
Barite and Sillitin Z 86

PVC 7.6%:
Barite, Sillitin Z 86, Mica, NCC, Clay, Wollastonite, Aktisil AM and MM

PVC 7.6%:
Talc

Impact test
DIN EN ISO 6272

EXPERIMENTAL

RESULTS

- Application Properties
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SUMMARY

		Impact [kg x cm]
PVC 0.4	Without filler	> 200
PVC 4.2	Barite	
	Sillitin Z 86	
PVC 7.6	Barite	
	Sillitin Z 86	
	Aktisil AM	
	Aktisil MM	
	NCC	
	Wollastonite	
	Clay	
	Mica	
	Talc	

Reverse impact test
DIN EN ISO 6272

EXPERIMENTAL

RESULTS

- Application Properties
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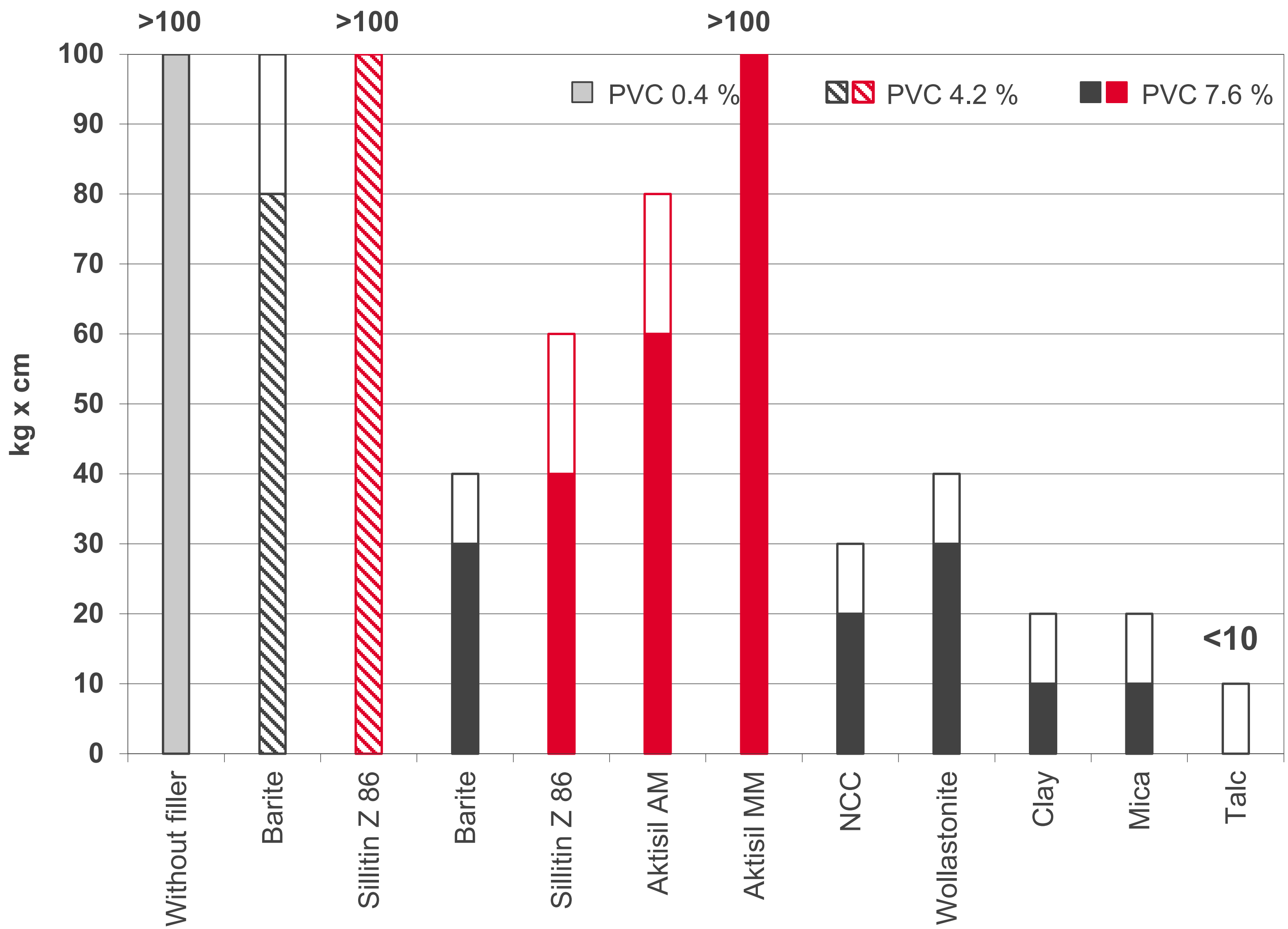
SUMMARY



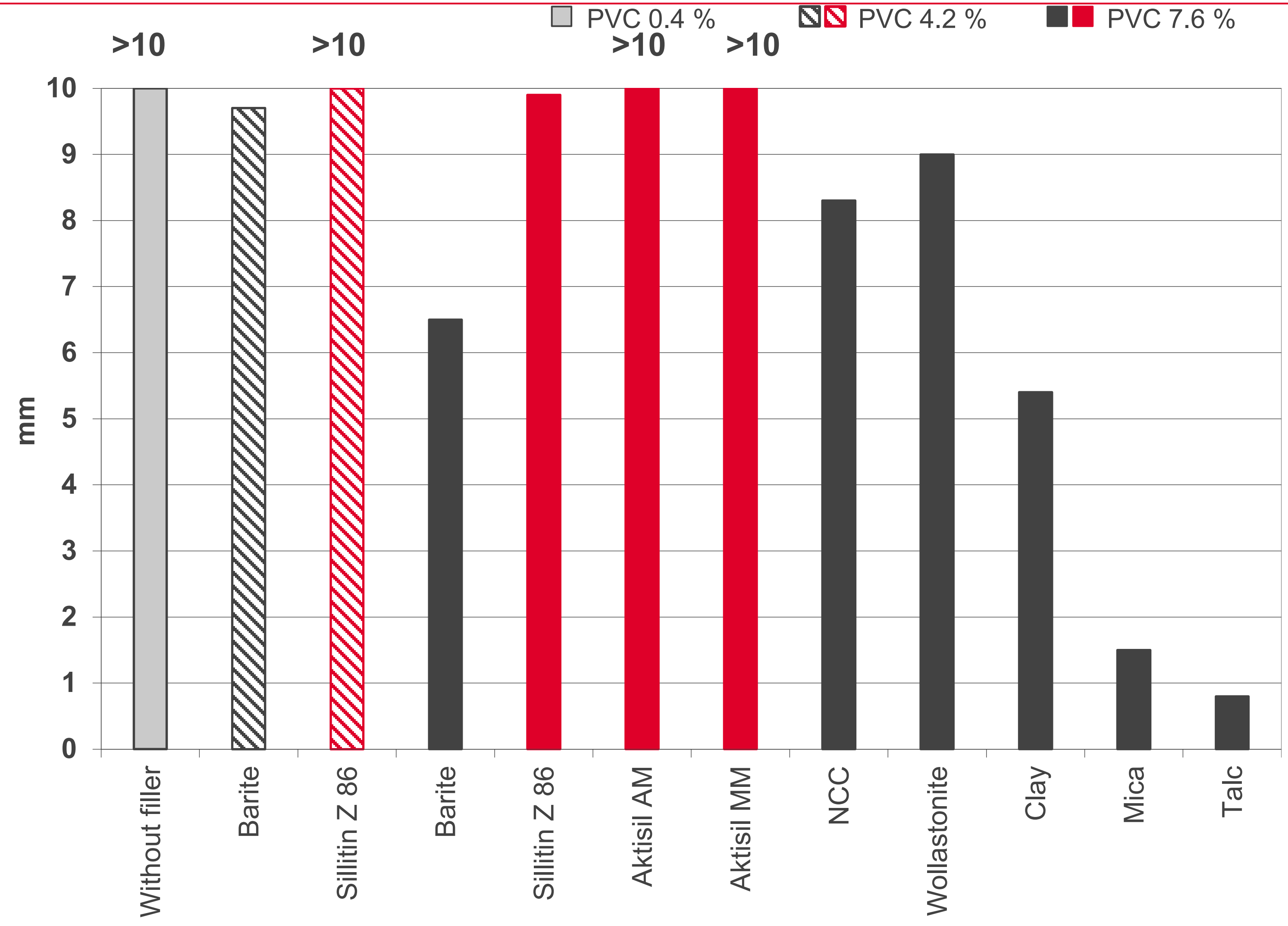
Cupping test
DIN EN ISO 1520

EXPERIMENTAL

RESULTS

- Application Properties
- Optical Properties
- Mechanical Properties
- Resistance Properties

SUMMARY



Mandrel bending test
DIN EN ISO 1519

EXPERIMENTAL

RESULTS

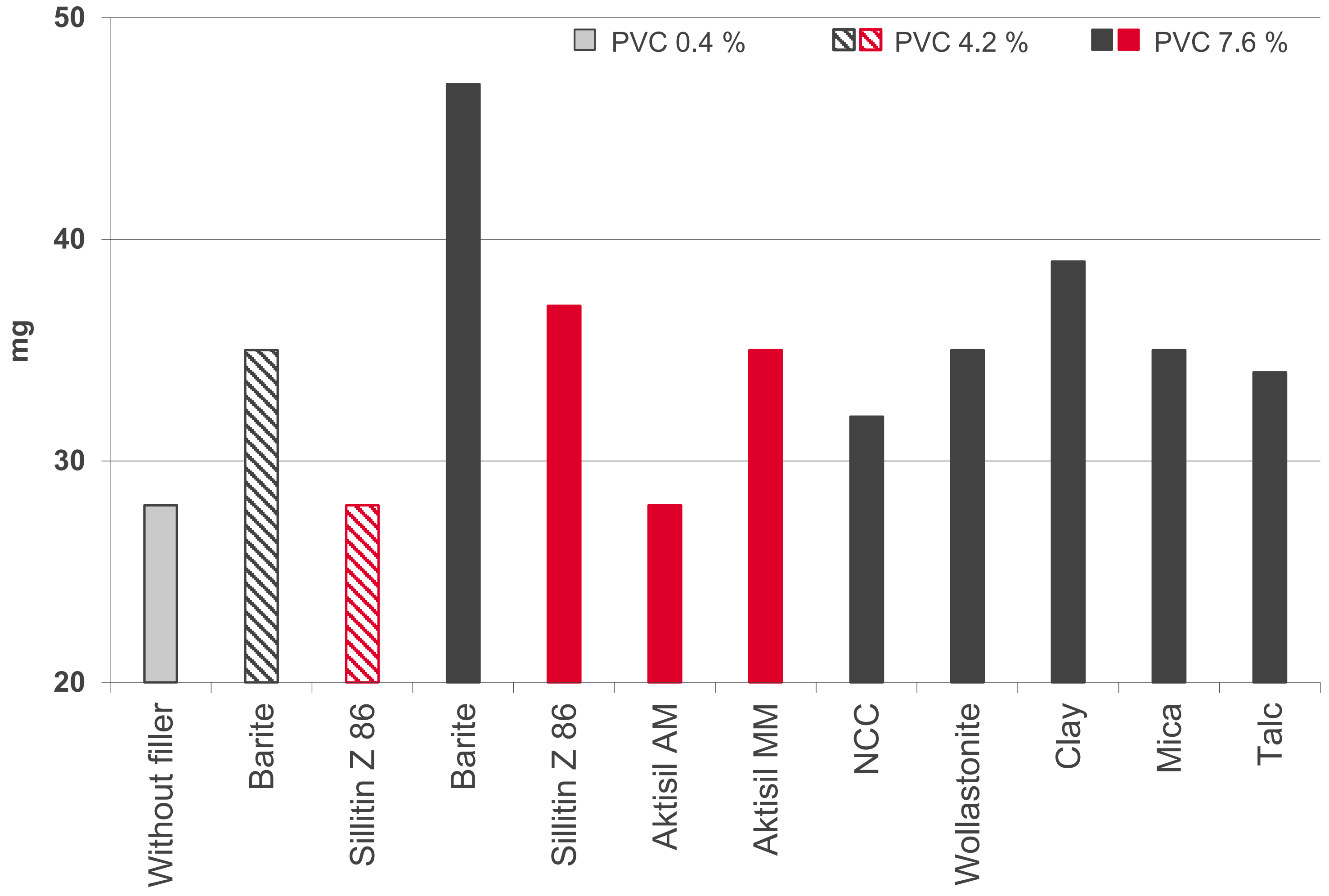
- Application Properties
- Optical Properties
- Mechanical Properties
- Resistance Properties

SUMMARY

		Mandrel Bending Test [mandrel = 32 mm]
PVC 0.4	Without filler	fine cracks
PVC 4.2	Barite	fine cracks
	Sillitin Z 86	fine cracks
PVC 7.6	Barite	large cracks
	Sillitin Z 86	fine cracks
	Aktisil AM	fine cracks
	Aktisil MM	fine cracks
	NCC	moderate cracks
	Wollastonite	fine cracks
	Clay	fine cracks
	Mica	flake-off
	Talc	flake-off

Abrasion resistance
ASTM D 4060

CS 17 / 1000 g / 1000 rev



EXPERIMENTAL

RESULTS

- Application Properties
- Optical Properties
- Mechanical Properties
- Resistance Properties

SUMMARY

Abrasion resistance
DIN 53754

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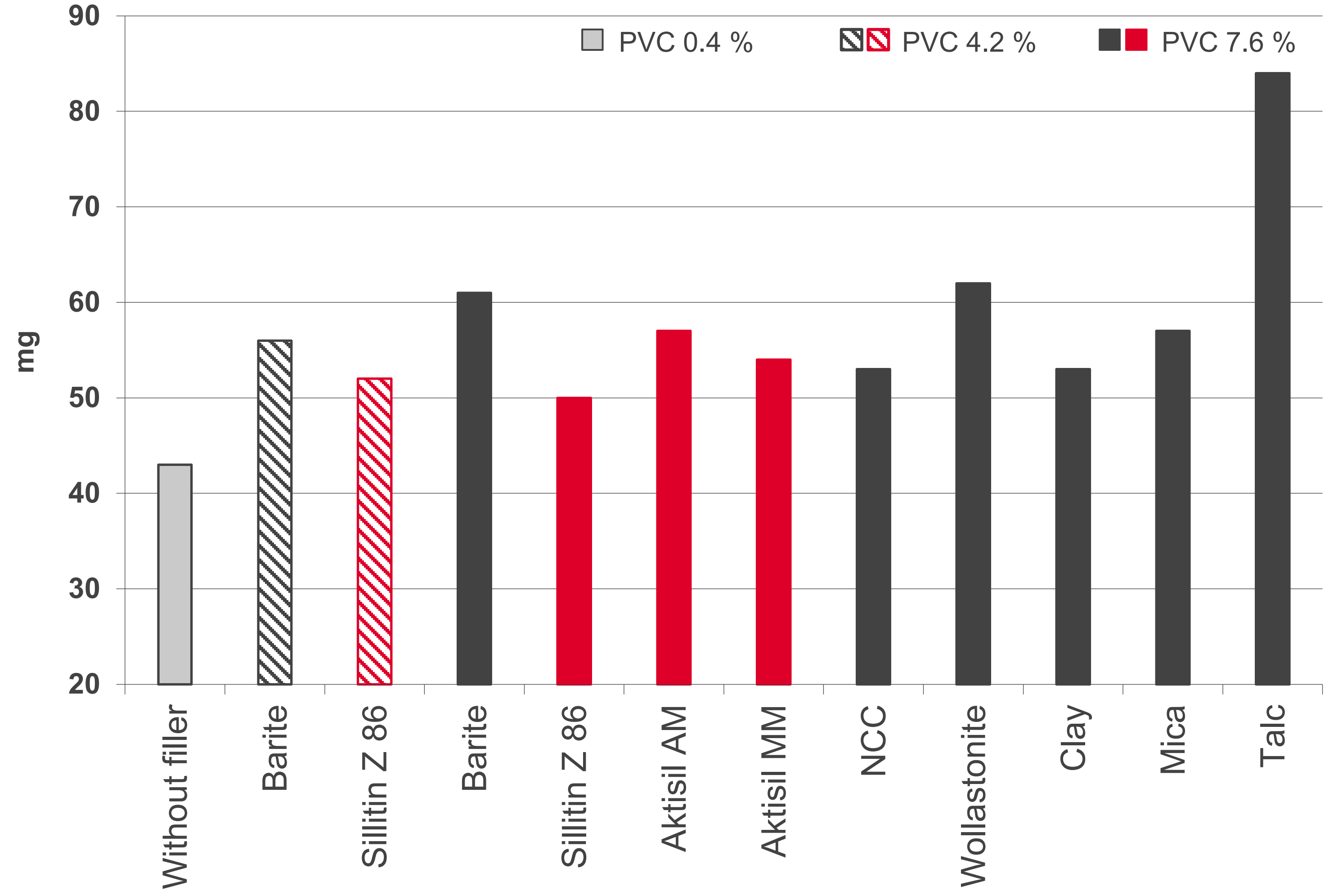
S 42 / 5,4 N / 100 rev

EXPERIMENTAL

RESULTS

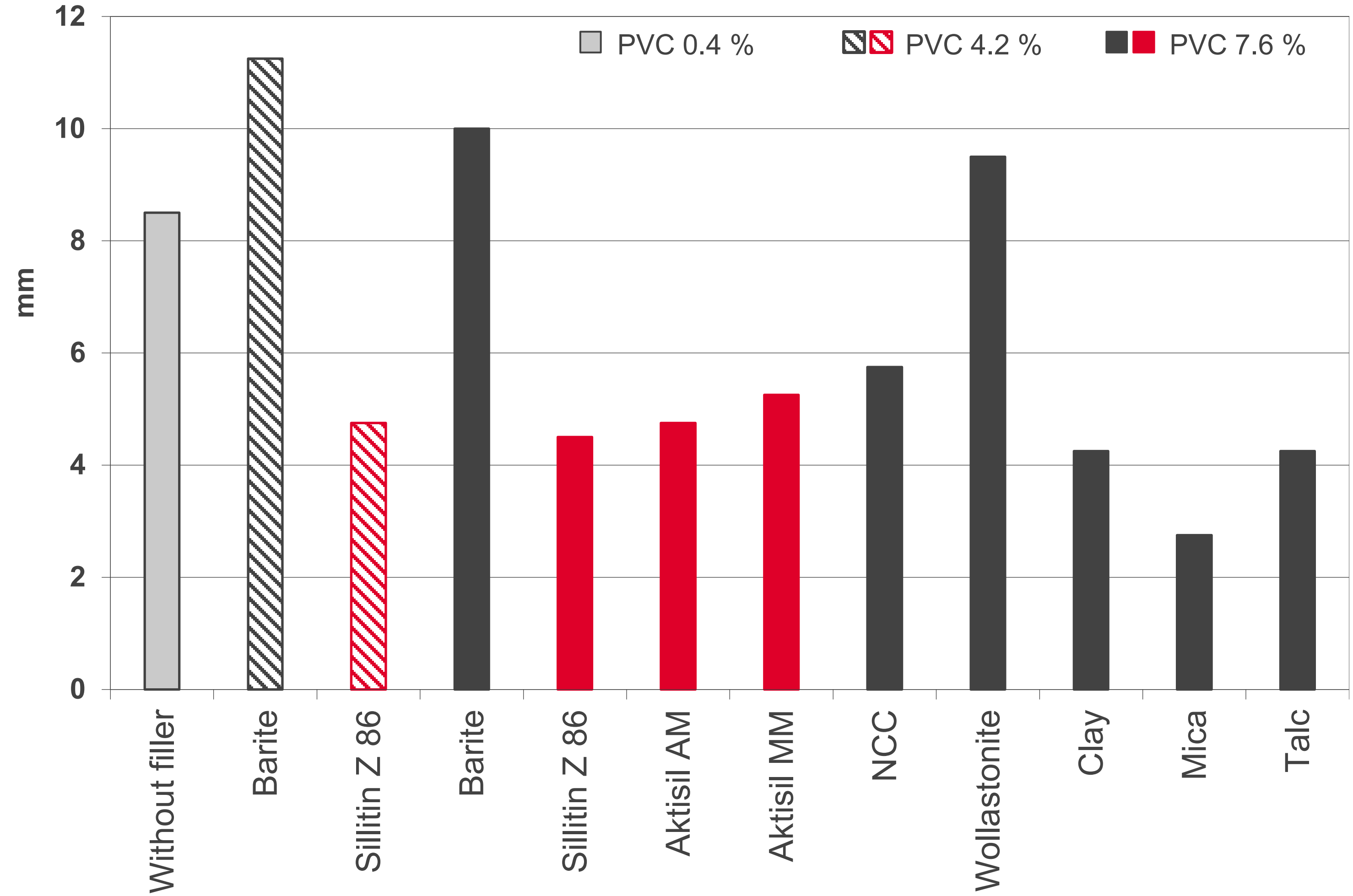
- Application Properties
- Optical Properties
- Mechanical Properties
- Resistance Properties

SUMMARY



Salt spray test
DIN EN ISO 9227

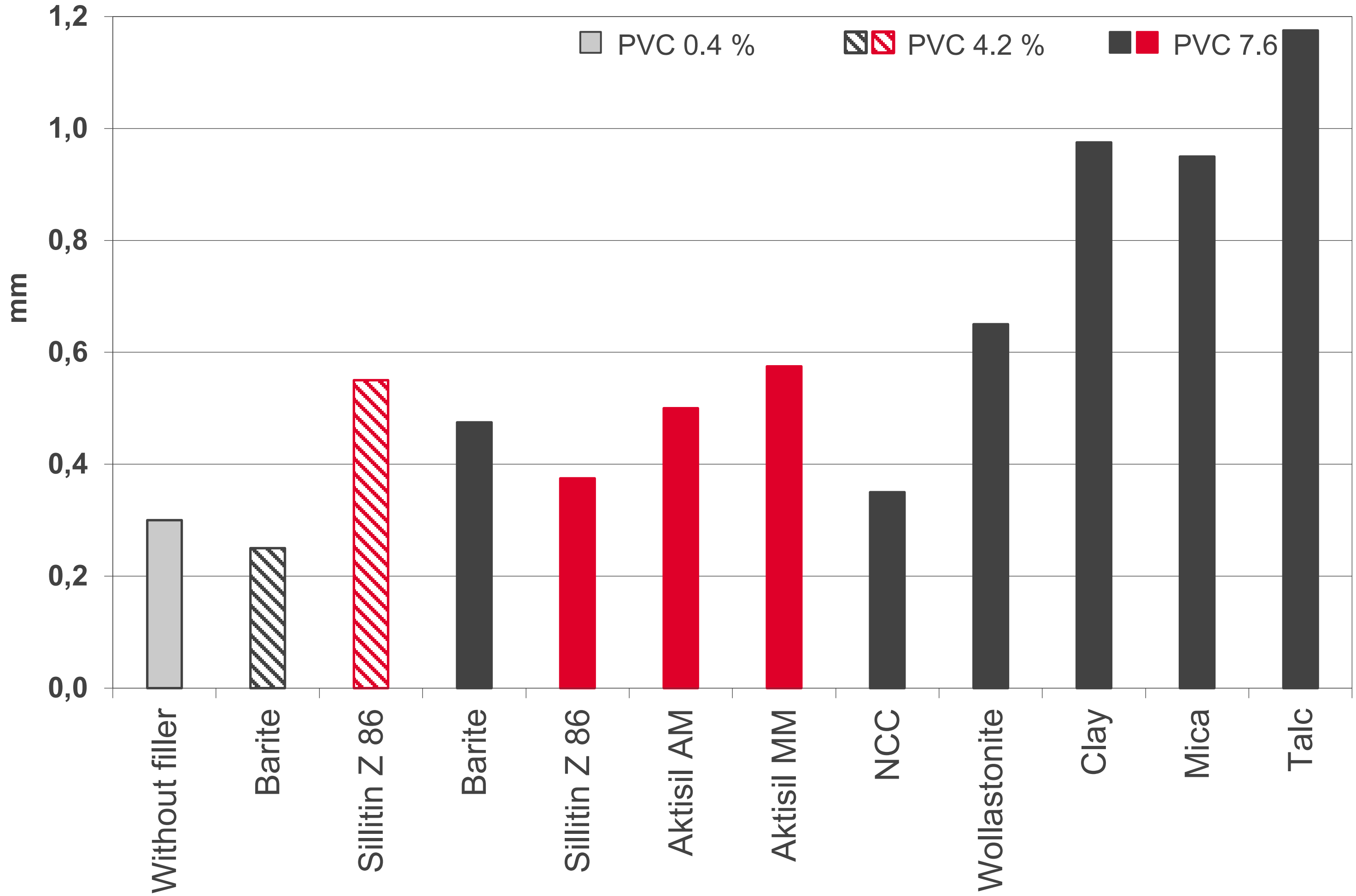
Delamination at scribe after 2000 h



Salt spray test

DIN EN ISO 9227

Corrosion at scribe after 2000 h



EXPERIMENTAL

RESULTS

- Application Properties
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SUMMARY

Humidity test
DIN EN ISO 6270-2

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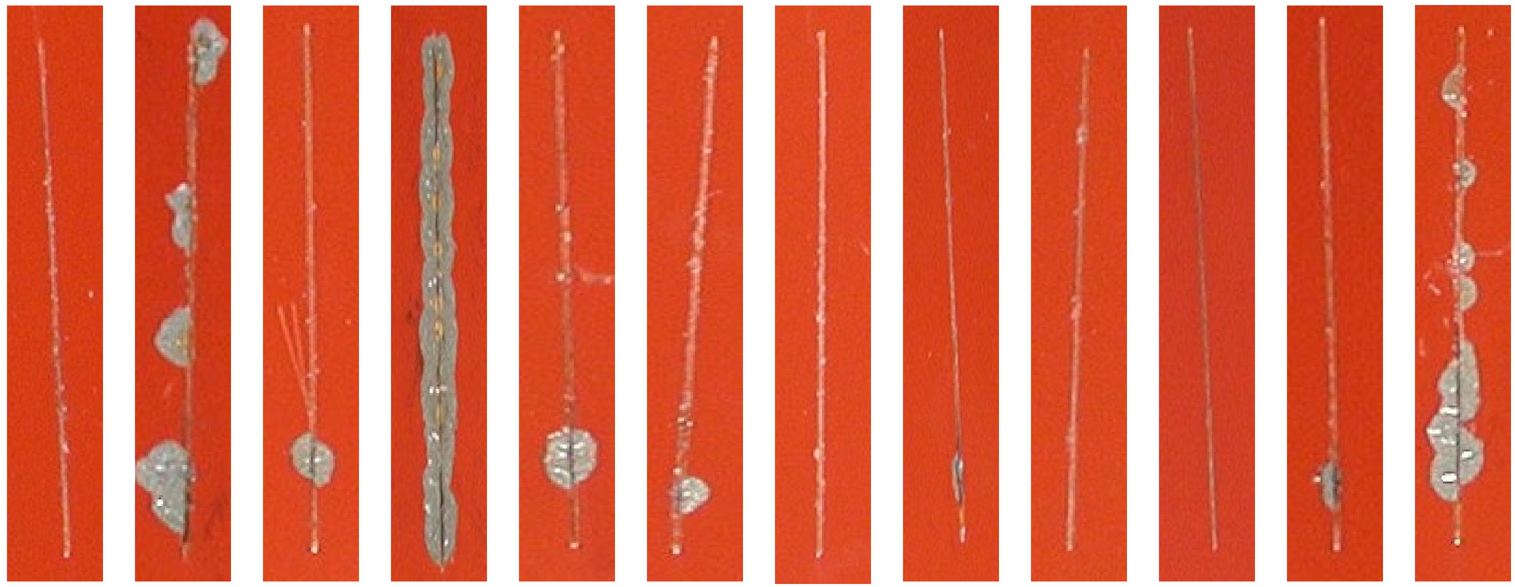
Delamination at scribe after 4000 h

EXPERIMENTAL

RESULTS

- Application Properties
- Optical Properties
- Mechanical Properties
- Resistance Properties

SUMMARY



Filler	PVC
Without filler	0.4%
Barite	4.2%
Sillitin Z 86	4.2%
Barite	7.6%
Sillitin Z 86	7.6%
Aktisil AM	7.6%
Aktisil MM	7.6%
NCC	7.6%
Wollastonite	7.6%
Clay	7.6%
Mica	7.6%
Talc	7.6%

Humidity test
DIN EN ISO 6270-2

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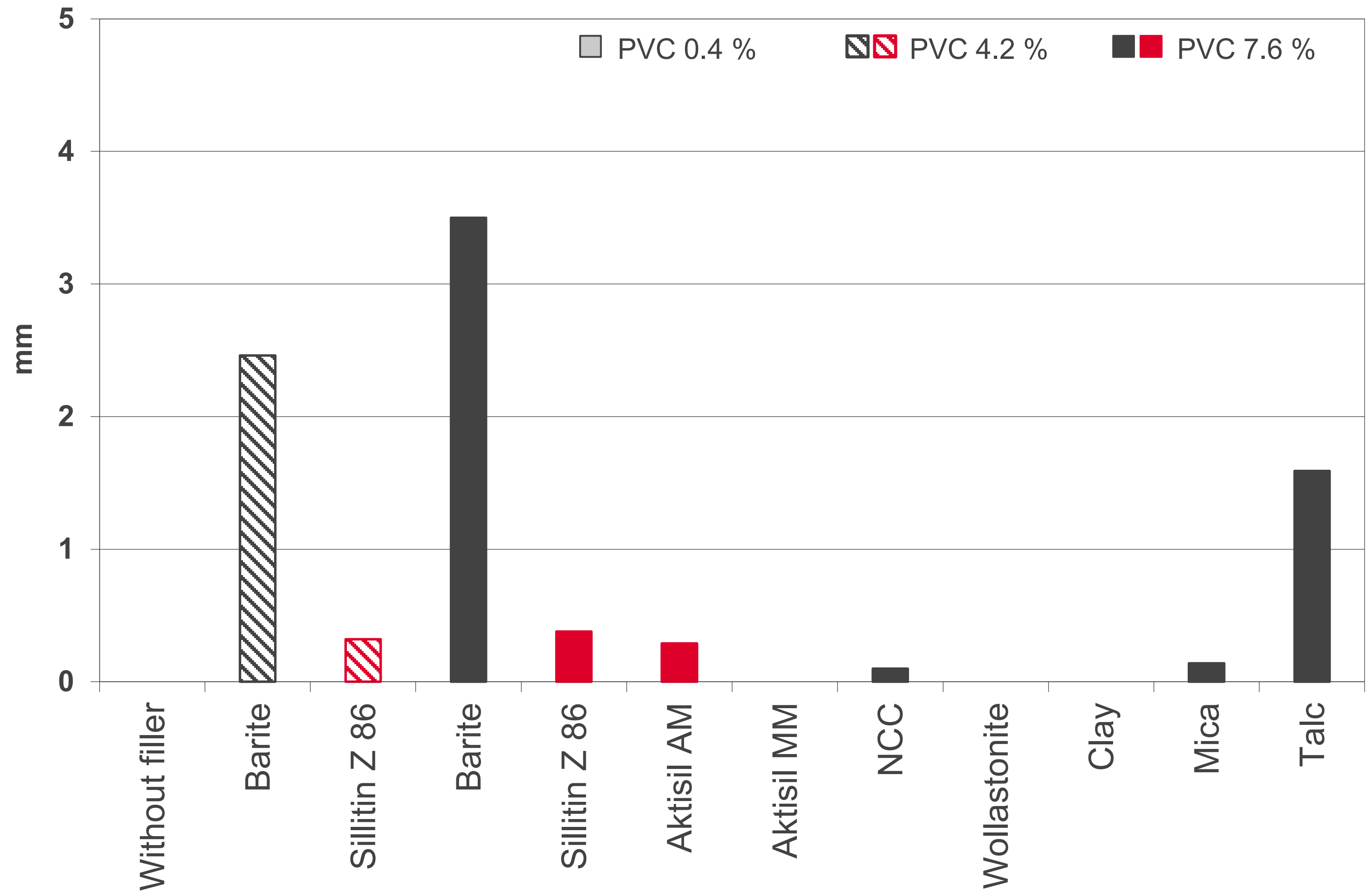
Average Delamination [mm] at scribe after 4000 h



Humidity test
DIN EN ISO 6270-2

EXPERIMENTAL

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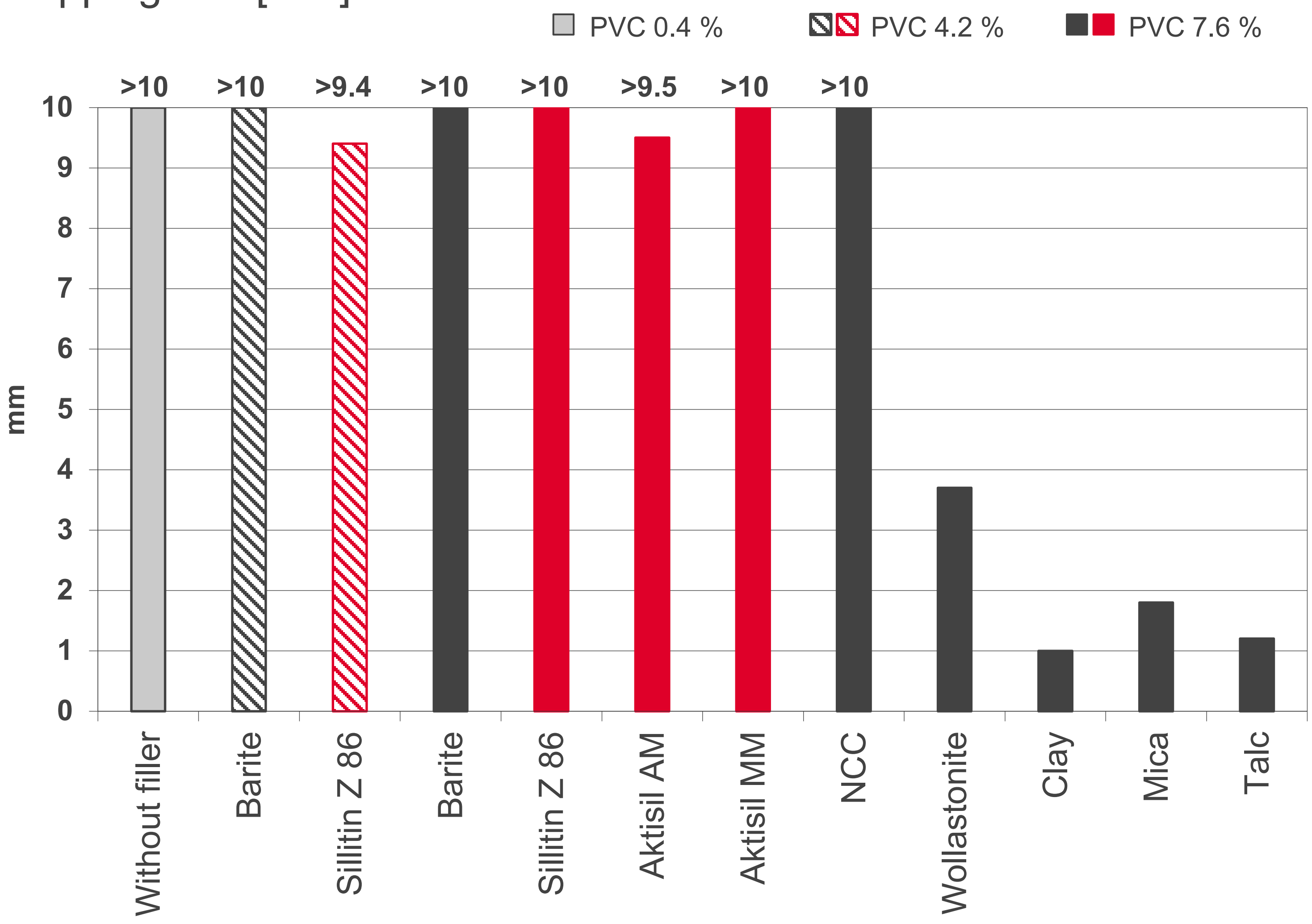
SUMMARY

Cupping test [mm] after 4000 h



Chemical resistance
DIN EN ISO 2812-2

Immersion in distilled water at 90 °C 1700 h

EXPERIMENTAL

RESULTS

- Application Properties
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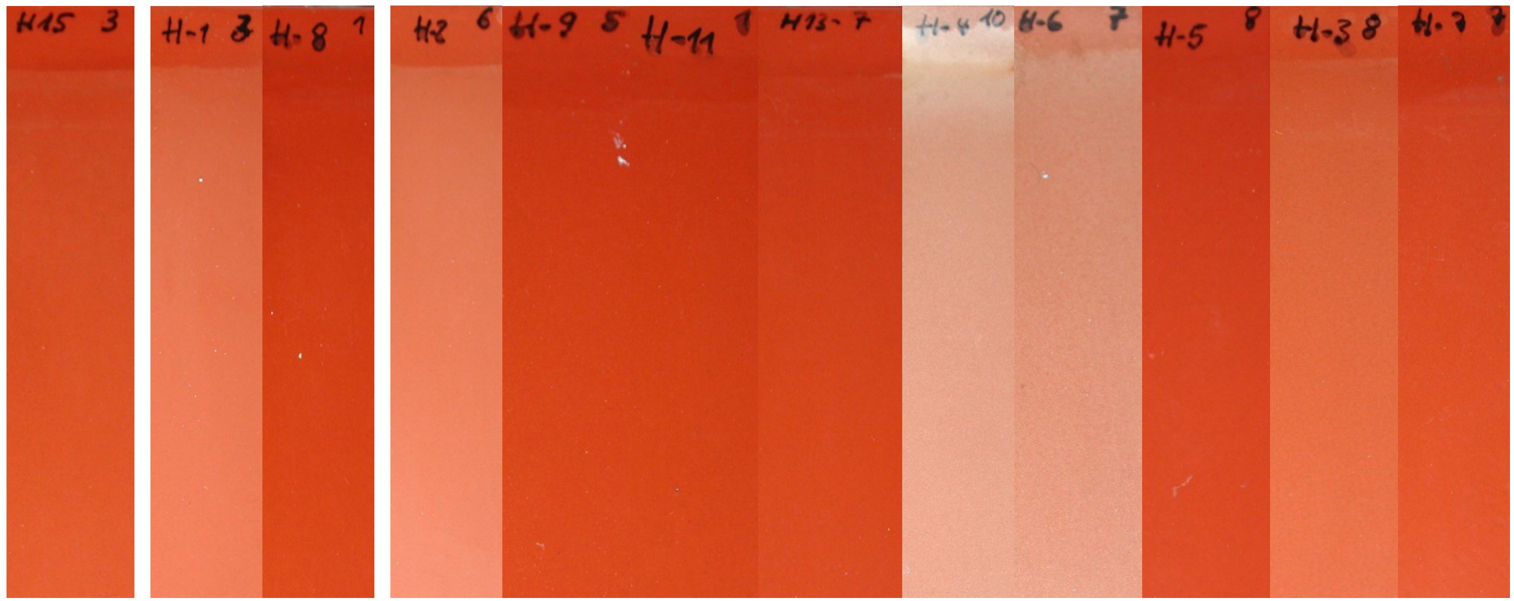
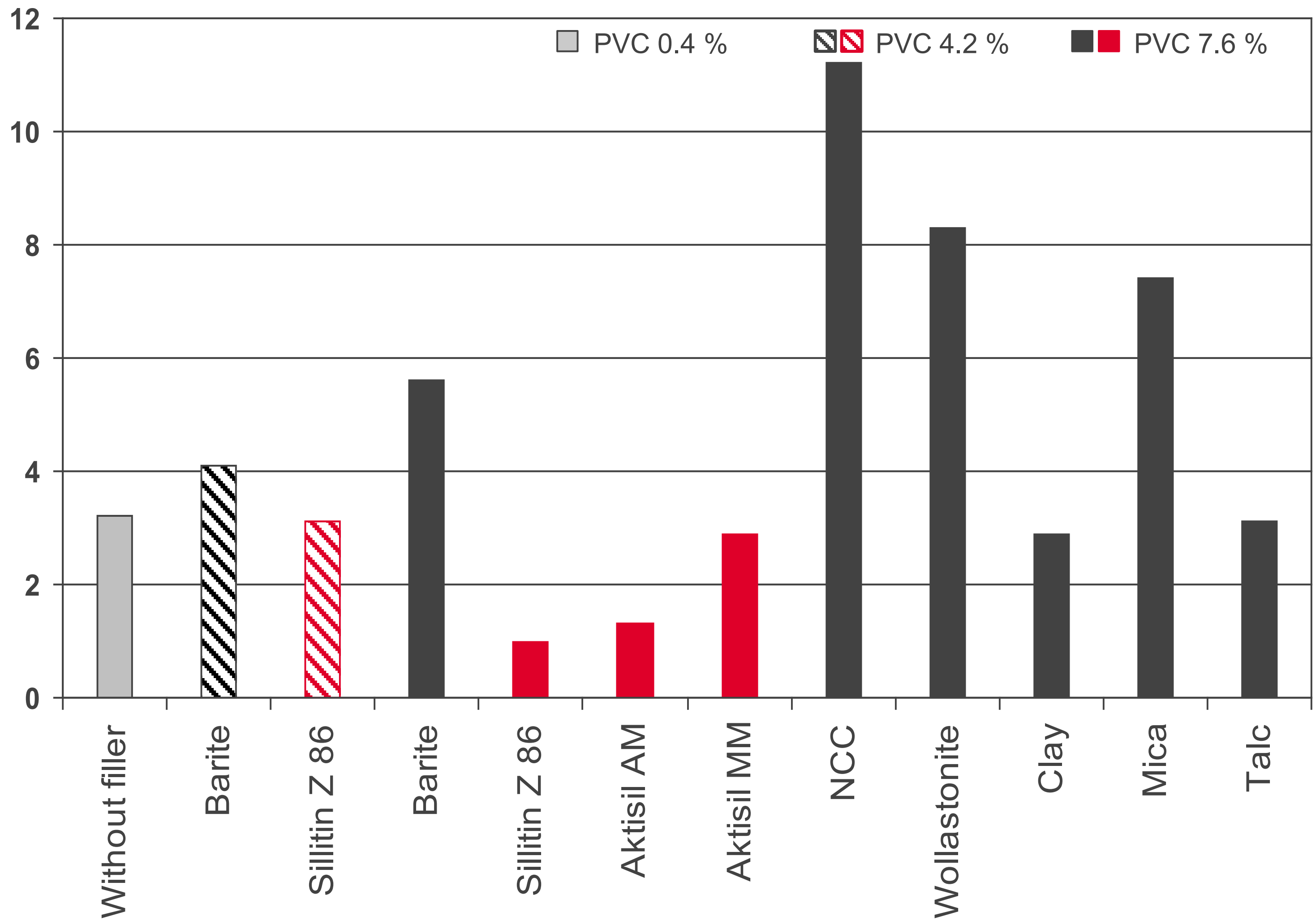
SUMMARY



Without filler	Barite	Sillitin Z 86	Barite	Sillitin Z 86	Aktisil AM	Aktisil MM	NCC	Wollastonite	Clay	Mica	Talc
PVC 0.4%	PVC 4.2%		PVC 7.6%								

Chemical resistance
DIN EN ISO 2812-2

Color change delta E after 1700 h immersion in water at 90 °C



EXPERIMENTAL

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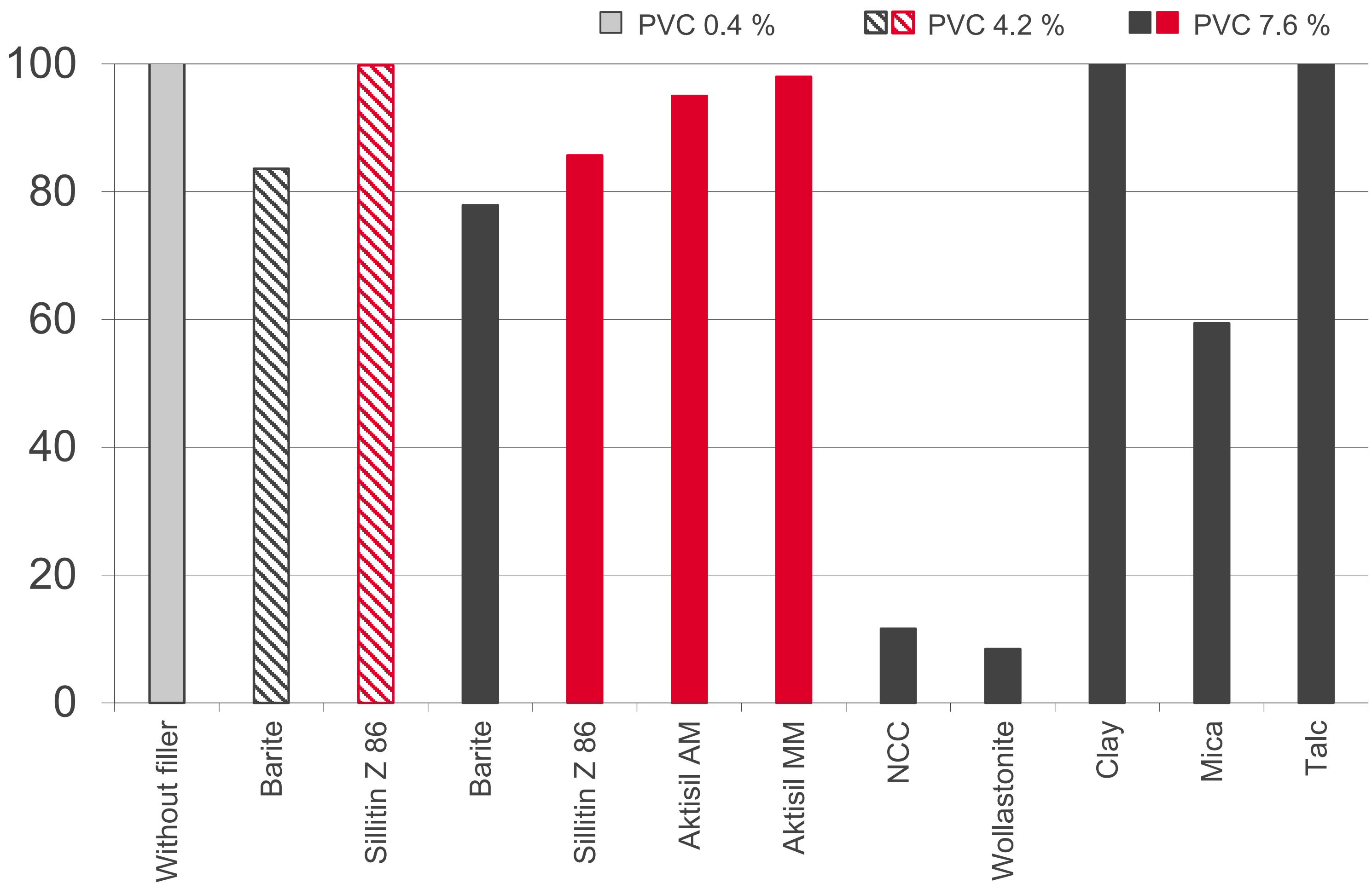
Chemical resistance
DIN EN ISO 2812-2

Remaining gloss 60° (%) after 1700 h immersion in water at 90 °C



EXPERIMENTAL

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Chemical resistance
DIN EN ISO 2812-2

EXPERIMENTAL

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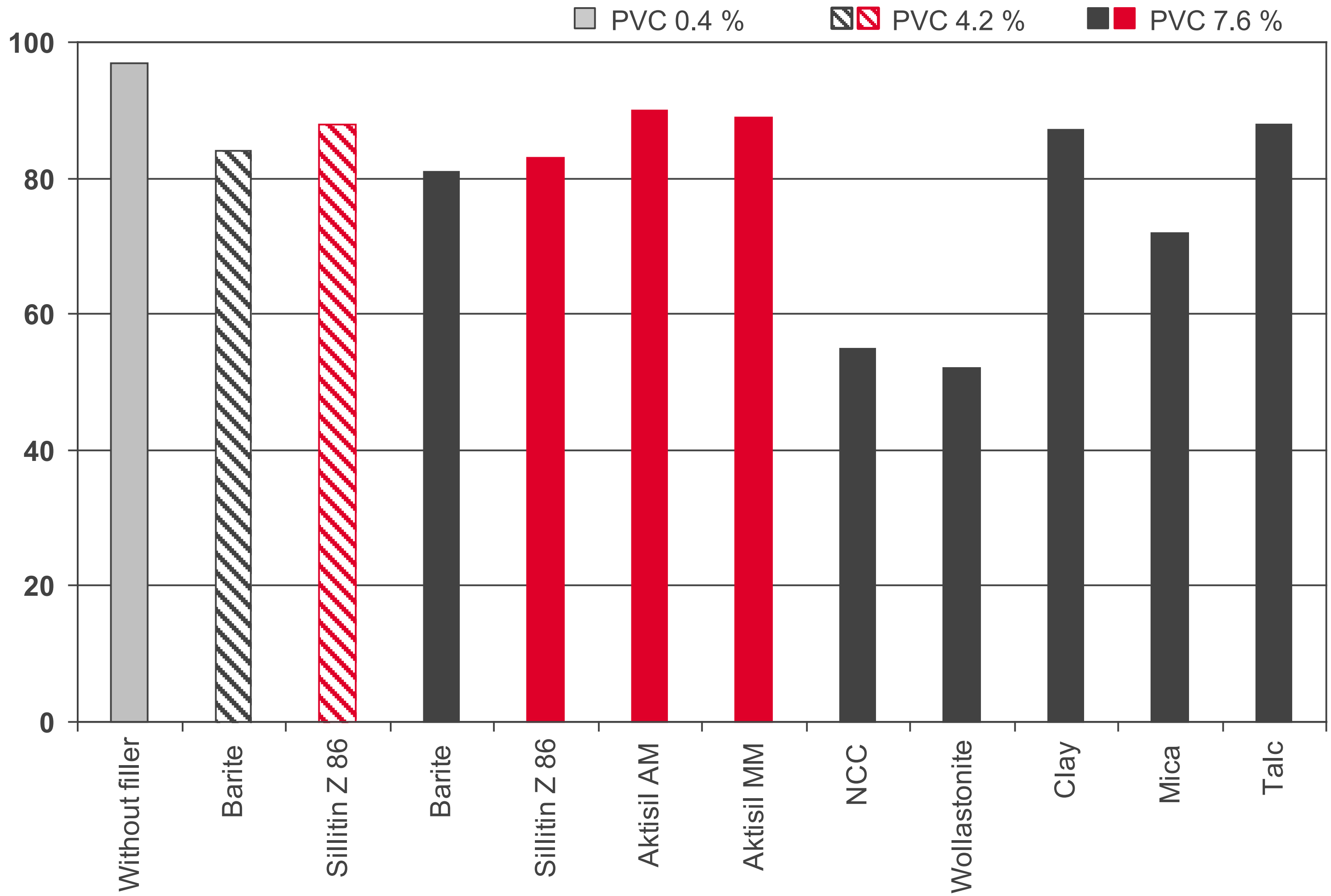
Remaining pendulum hardness (%) after 1700 h immersion in water at 90 °C



Summary

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SUMMARY

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

Compared to the standard barite PVC 4.2:

+	Advantage
++	Significant advantage
-	Disadvantage
--	Significant disadvantage
0	No significant effect

Summary

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SUMMARY

Working with Neuburg Siliceous Earth offers the following benefits:

- Better fluidizability
- Optical properties remain on a high level
- Markedly improved mechanical properties (cupping test, reverse impact test)
- Increased abrasion resistance
- Markedly reduced delamination, low corrosion at scribe
- Increased hot water resistance

These positive effects were also widely received in the PVC raised variation.

Summary

Sillitin Z 86 offers an attractive price/performance ratio.

Aktisil AM allows to obtain the best resistance properties.

Aktisil MM shows lower delamination and better mechanical properties.

We supply material for good ideas!

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