Calcined Neuburg Siliceous Earth in adhesives with high strength based on silane terminated polyurethane (STP-U)

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Status Quo

- Apart from the widely introduced silicone and polyurethane systems, also hybrid prepolymers based on silane terminated polyurethanes (STP-U) offer themselves for the preparation of sealants and adhesives.
- They combine the benefits of a polyurethane base structure with a silane based curing mechanism.
- The formulations prepared are non-hazardous with respect to health and environment and are distinguished by outstanding mechanical properties along with excellent adhesion characteristics.
- The standard filler here is calcium carbonate, and for highly demanding adhesives in preference precipitated calcium carbonate (PCC) with a higher specific surface area.

Objective

This study will present Calcined Neuburg Siliceous Earth grades as functional fillers for high strength adhesives based on silane terminated polyurethanes.

The objective was to improve the strength of the adhesive and take advantage of this effect for upgrading traditional compounds formulated with the established filler calcium carbonate.

Formulation

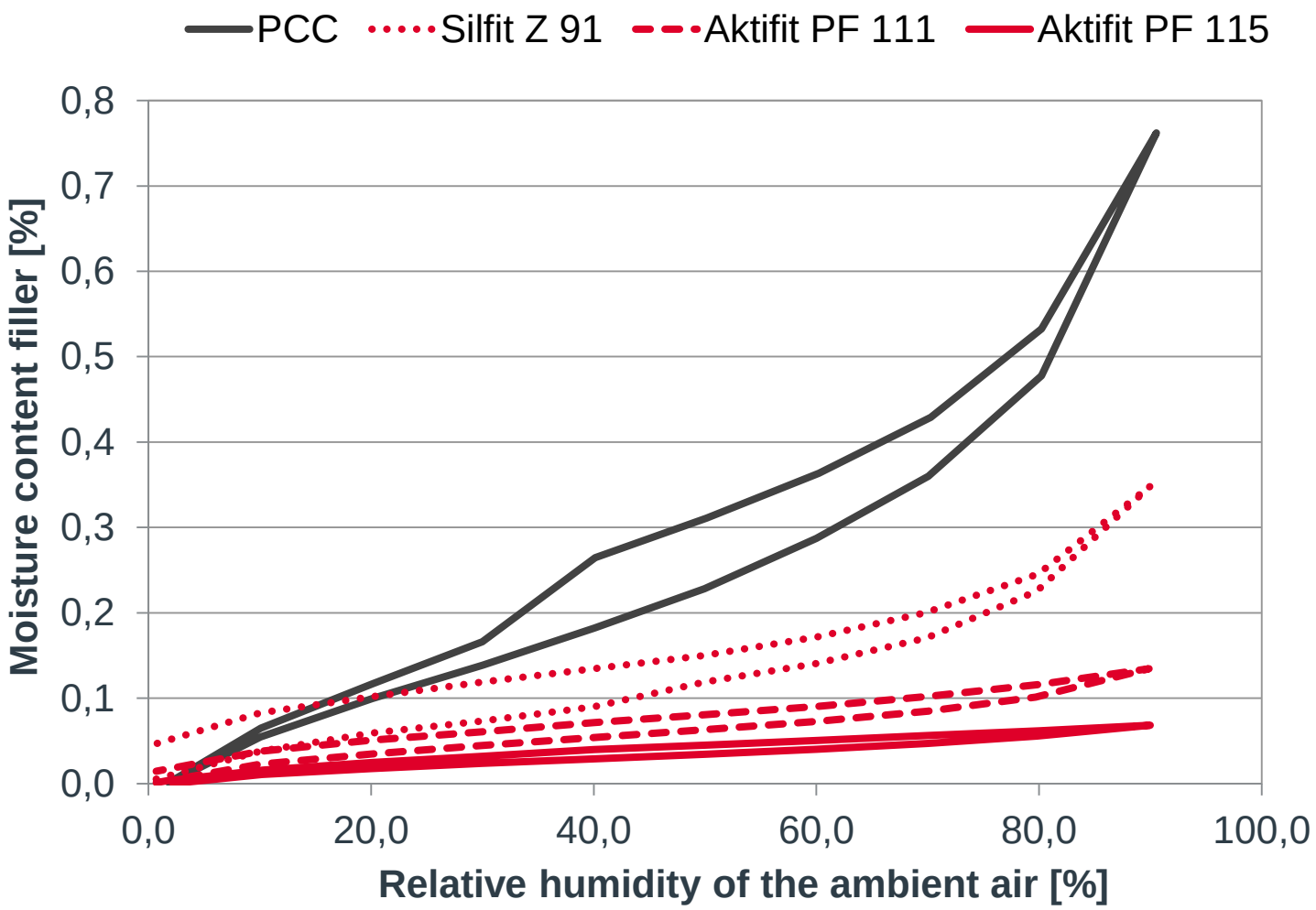
Basis: Guide formulation BBB 7507 from Covestro

		parts or % by weight
Desmoseal S XP 2821	Polymer: silane terminated polyurethane	38.88
Irganox 1135	Antioxidant	0.46
Bayferrox Gelb 415	Pigment	0.28
Cab-O-Sil TS 720	Rheological additive: fumed silica	0.95
Filler		**53.71**
Dynasylan VTMO	Drying agent: vinyl silane	2.61
DBU	Catalyst: diazabicycloundecene	0.11
Dynasylan 1146	Adhesion promoter: amino silane	1.50
Dynasylan AMEO	Adhesion promoter: amino silane	1.50
Total		**100.00**

Fillers, Characteristics

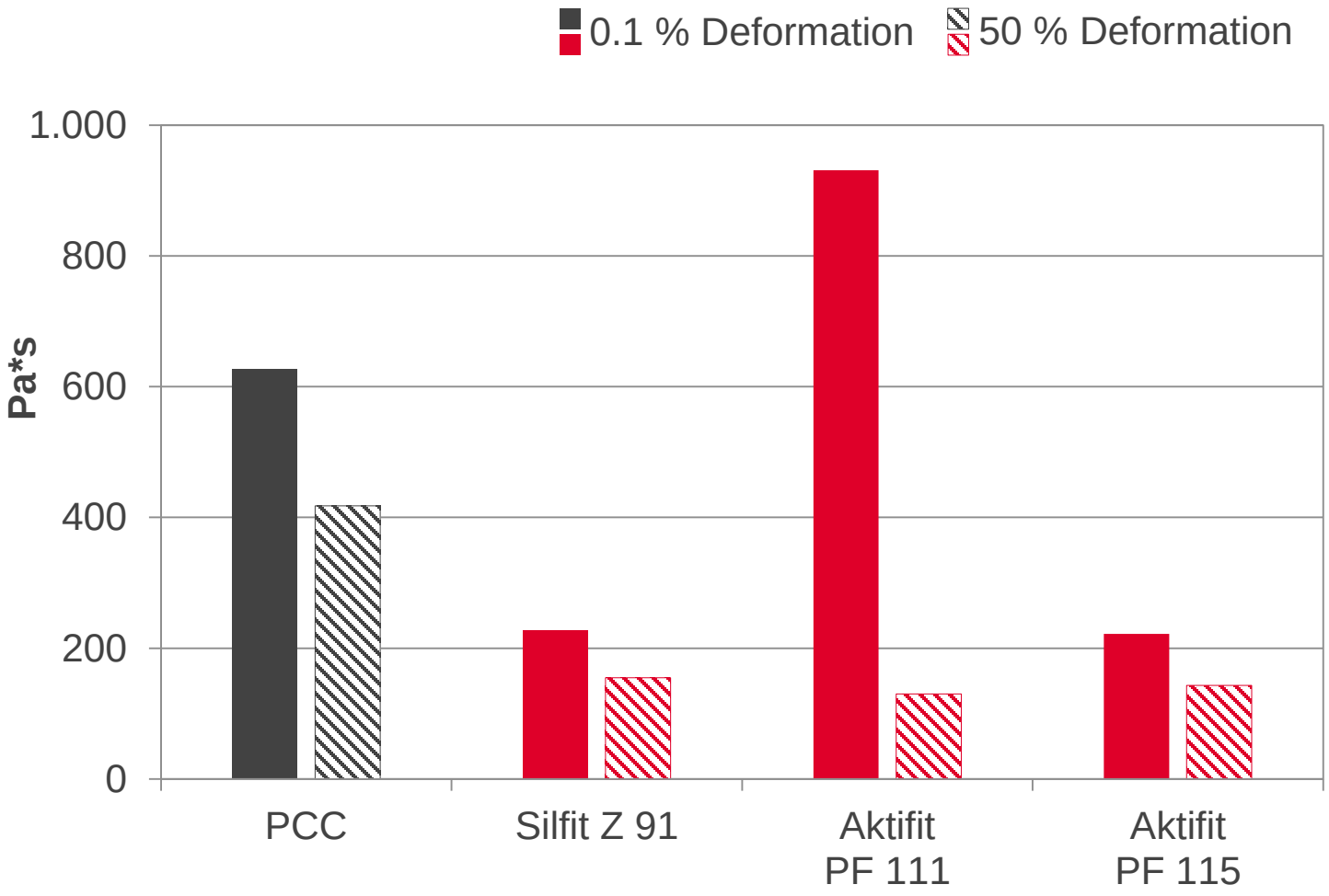
		PCC	Calcined Neuburg Siliceous Earth		
			Silfit Z 91	Aktifit PF 111	Aktifit PF 115
Volatile matter at 105 °C	%	0.5	0.2	0.2	0.1
Oil absorption	g/100g	44	55	49	59
Specific surface area BET	m^2/g	11	8	7	8
Functionalisation		---	---	Alkyl	Special amino
Surface character		hydrophilic	hydrophilic	hydrophobic	hydrophobic

Moisture Content of Fillers vs. Ambient Air Humidity



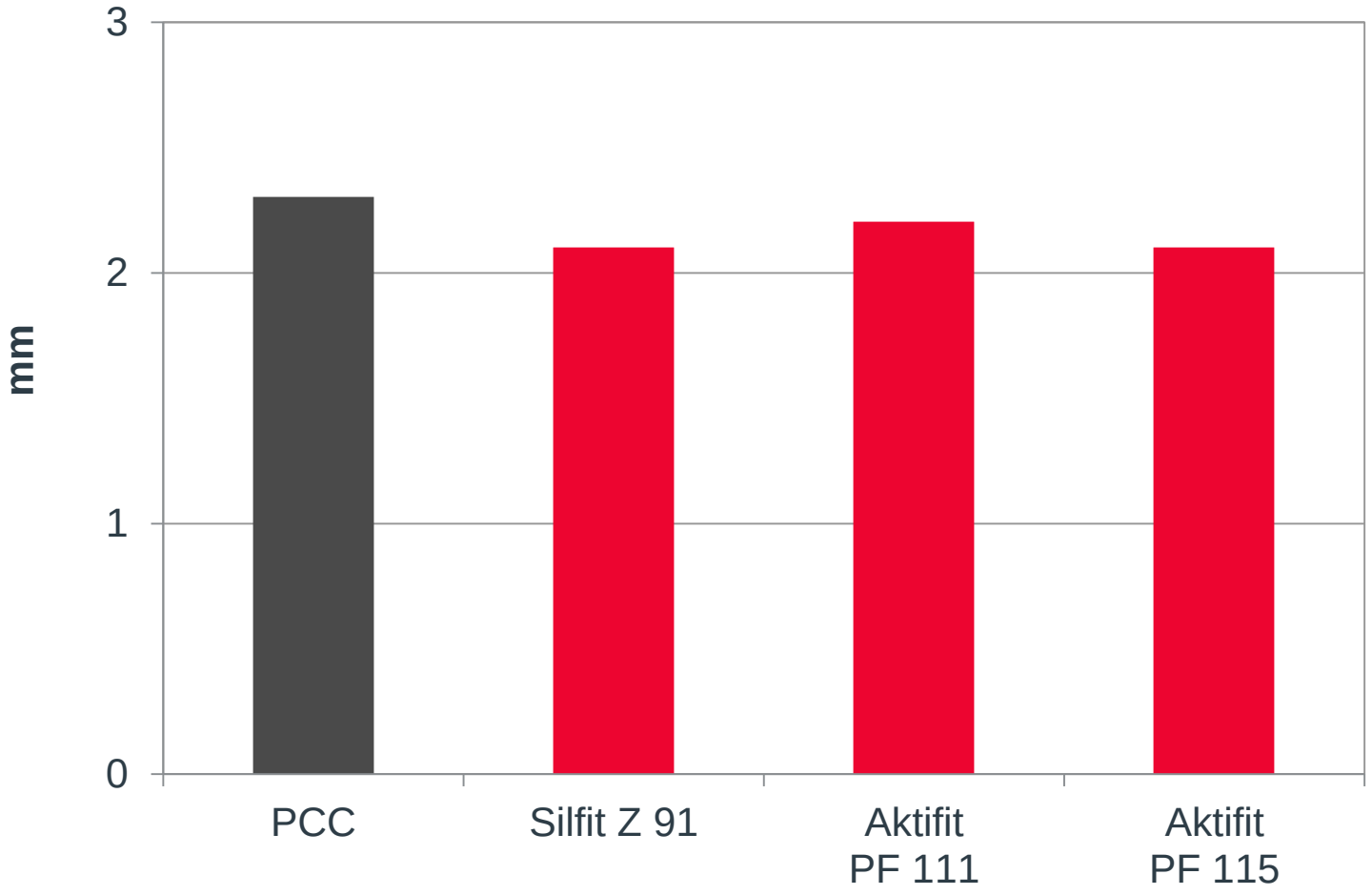
Complex Viscosity

DIN 54458



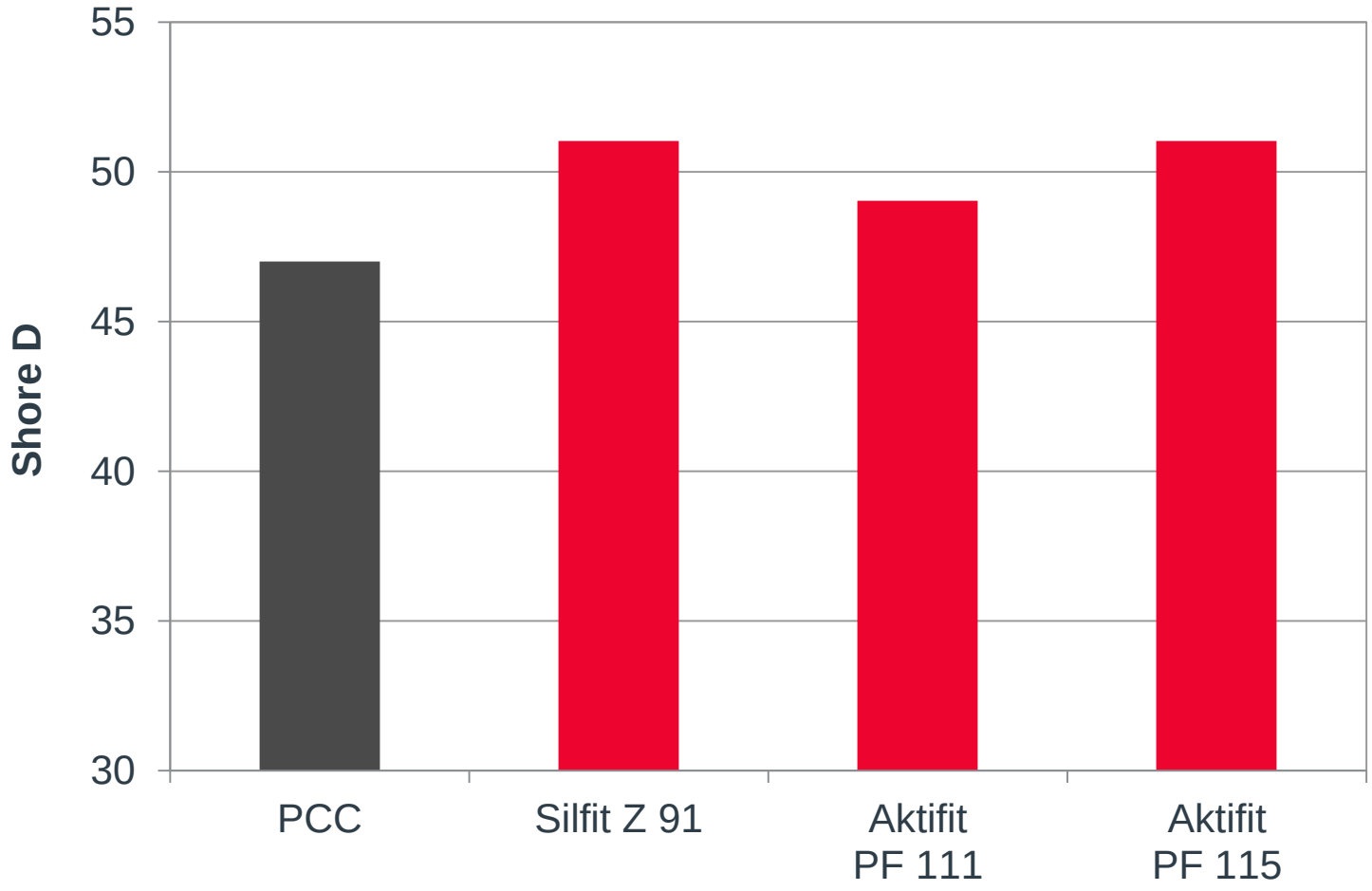
In-depth Cure

Thickness of the reacted layer after 24 h



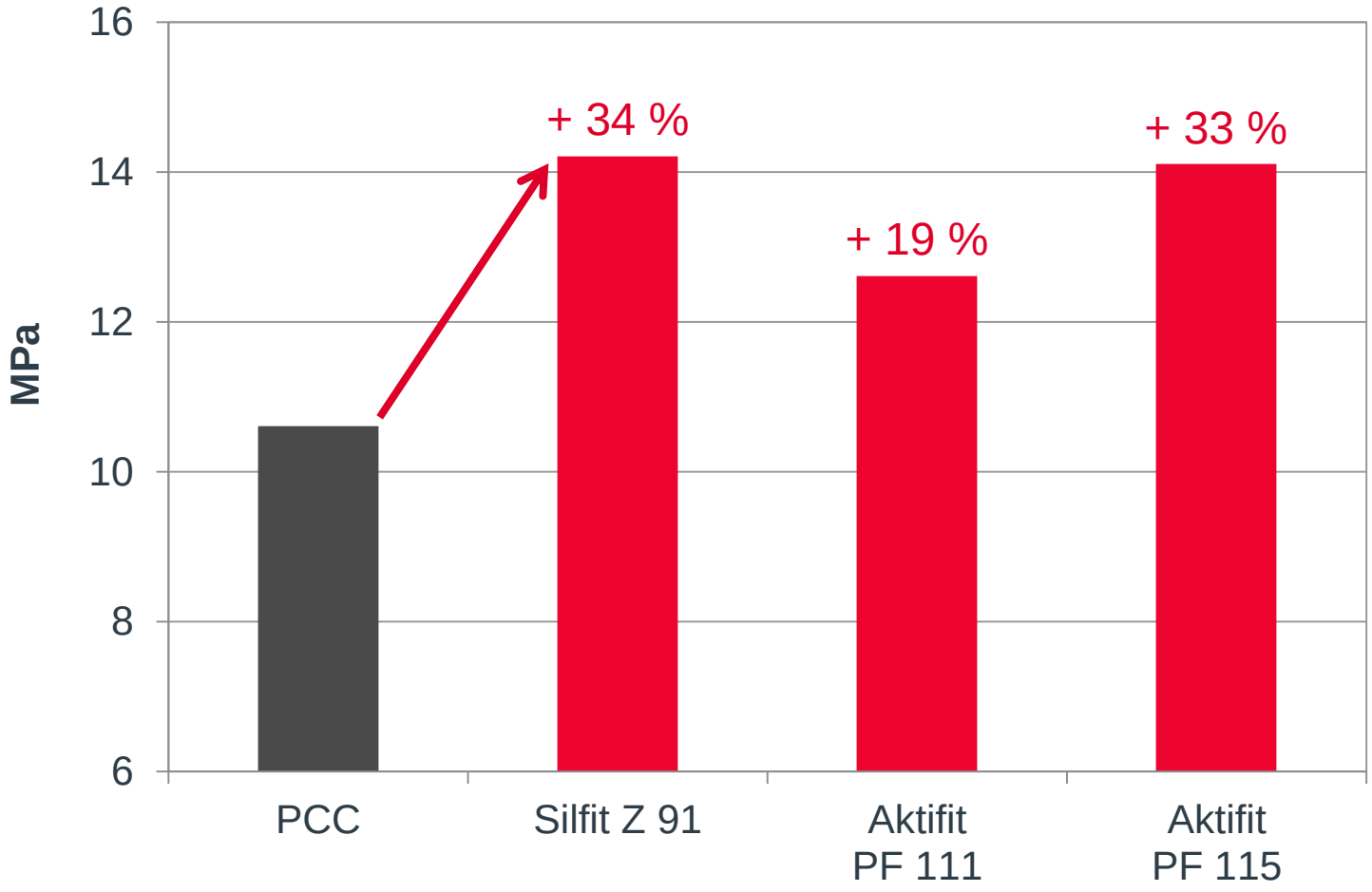
Hardness Shore D

DIN ISO 7619-1



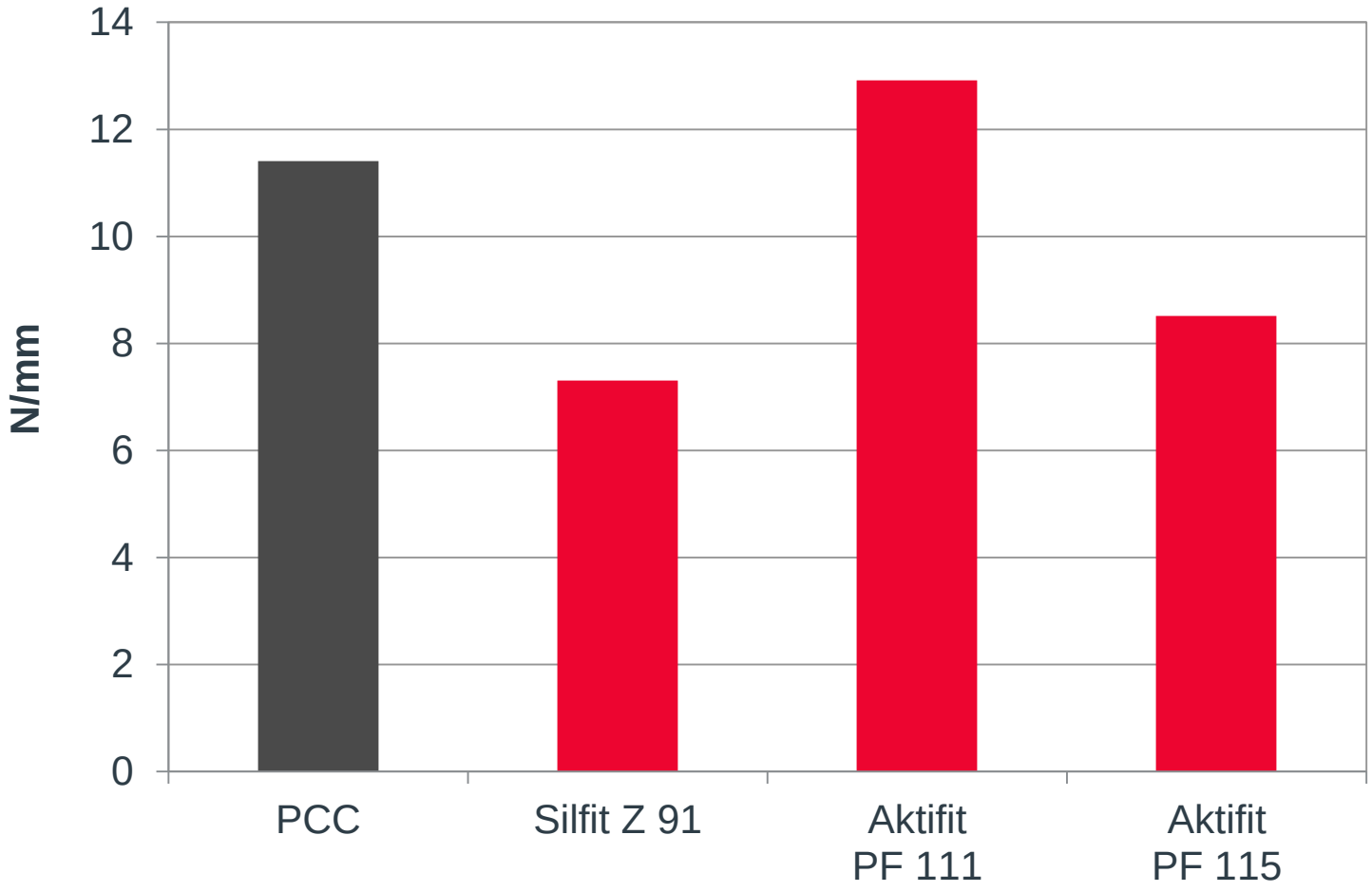
Tensile Strength

DIN 53504, S2



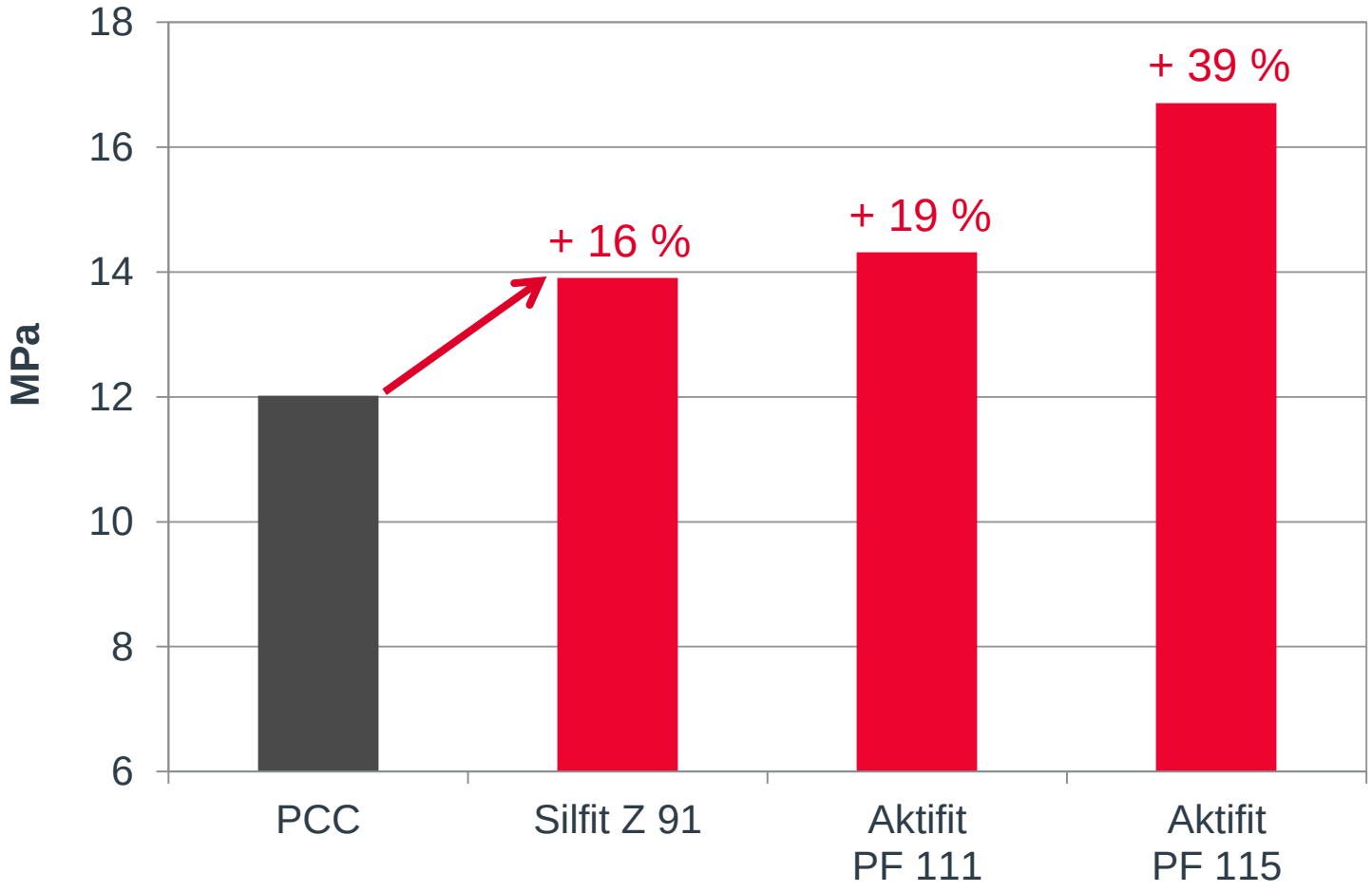
Tear Resistance Graves

DIN ISO 34-1, method B(b)



Lap Shear Strength
Beech Wood

DIN EN 205, after 7 d, layer thickness 0.1 mm



Summary

The PCC grade used for the class of precipitated calcium carbonates already represents the optimum with respect to mechanical properties that can be realized.

In comparison, Calcined Neuburg Siliceous Earth shows:

- Significant lower viscosity at higherer deformation, thereby easier application
- Viscosity at low deformation / yield point variably adjustable
- High hardness
- Increased tensile strength of up to 14 MPa
- Marked increase of lap shear strength, more than 16 MPa are possible

Distinguishing Features of the NSE Grades

- **Silfit Z 91** — Low moisture content, white and color-neutral, cost effective, good mechanical properties
- **Aktifit PF 115** — Very low moisture content and extremely low moisture absorption even under humid conditions, white and color-neutral, for highest requirements on tensile strength and lap shear strength
- **Aktifit PF 111** — Very low moisture content and very low moisture absorption even under humid conditions, white and color-neutral, rheology control along with high strength, high elongation at break and high tear resistance

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Preparation of Batches

- **Planetary mixer**
- **Dissolver disc, bar blade and scraper**
- **Batch size approx. 500 ml**

- Feed polymer, pigment and antioxidant
- Stir in rheological additive
- Stir in (undried) filler
- Add drying agent and catalyst

Disperse under vacuum:
- 5 min at 3000 rpm and 600 rpm
- 10 min at 1000 rpm and 300 rpm
- 5 min at 800 rpm and 300 rpm

Cool down to <60 °C

- Add both adhesion promoters

Disperse under vacuum:
- 15 min at 1000 rpm and 300 rpm

- Fill into cartridge

Testing Conditions

Rheology	DIN 54458, MCR 300, PP 25 mm, d: 0.5 mm, Oscillation: deformation 0.01 to 100 %, f = 10 Hz
Hardness Shore D	DIN ISO 7619-1, piled S2 specimens Curing / conditioning: 4 weeks @ standard conditions 23/50
Tensile test	DIN 53504, S2 specimens Curing / conditioning: 4 weeks @ standard conditions 23/50
Tear resistance Graves	DIN ISO 34-1, method B (b) Curing / conditioning: 4 weeks @ standard conditions 23/50
Lap shear test	DIN EN 205 Substrate beech wood Adhesive layer: 0.1 mm Curing: 7 d @ standard conditions 23/50 Crosshead speed: 50 mm/min

Table of Results

		PCC	Calcined Neuburg Siliceous Earth		
			Silfit Z 91	Aktifit PF 111	Aktifit PF 115
Rheology					
Complex viscosity at 0.1 % deformation	Pa*s	626	227	931	222
Complex viscosity at 50 % deformation	Pa*s	418	155	130	143
Curing					
Skin formation	min	25	45	25	35
In-depth cure after 8 h	mm	1.4	1.2	1.3	1.3
In-depth cure after 24 h	mm	2.3	2.1	2.2	2.1
Mechanical properties					
Hardness	Shore D	47	51	49	51
Tensile strength	MPa	10.6	14.2	12.6	14.1
Elongation at break	%	38	20	30	22
Tear resistance Graves	N/mm	11.4	7.3	12.9	8.5
Lap shear strength					
Beech wood, layer 0.1 mm	MPa	12.0	13.9	14.3	16.7