Calcined Neuburg Siliceous Earth in adhesives with high strength based on Kaneka MS Polymer™

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Status Quo

- Apart from the widely introduced silicone and polyurethane systems, also the class of Kaneka MS Polymer™ offers itself for the preparation of sealants and adhesives.
- For some time now, close-mesh crosslinked polymers are available, which are suitable to formulate high-strength adhesives.
- 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 and surface treated calcium carbonate 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 Kaneka MS Polymer™.

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 precipitated and surface treated calcium carbonate.

Base Formulation

		parts by weight [pbw]
Kaneka Silyl™ SAX750	MS Polymer™, high strength	42.0
Filler PCC s.tr.	Precipitated calcium carbonate, surface treated	52.7
Vinyl silane VTMO	Drying agent	2.3
Amino silane AMMO	Adhesion promoter	1.5
Neostann S1	Catalyst	1.5
Total		**100.0**

Test Design

Weight-equal exchange of PCC s.tr.
by Calcined Neuburg Siliceous Earth

PCC s.tr.	Silfit Z 91	Aktifit VM	Aktifit AM	Aktifit PF 115
	+ 1 pbw rheological additive Cab-O-Sil TS 720			

Aktifit AM combined with varied / increased adhesion promoter

1.5 pbw amino silane AMMO	1.5 pbw amino silane AMEO
	1.5 pbw Dynasylan 1146

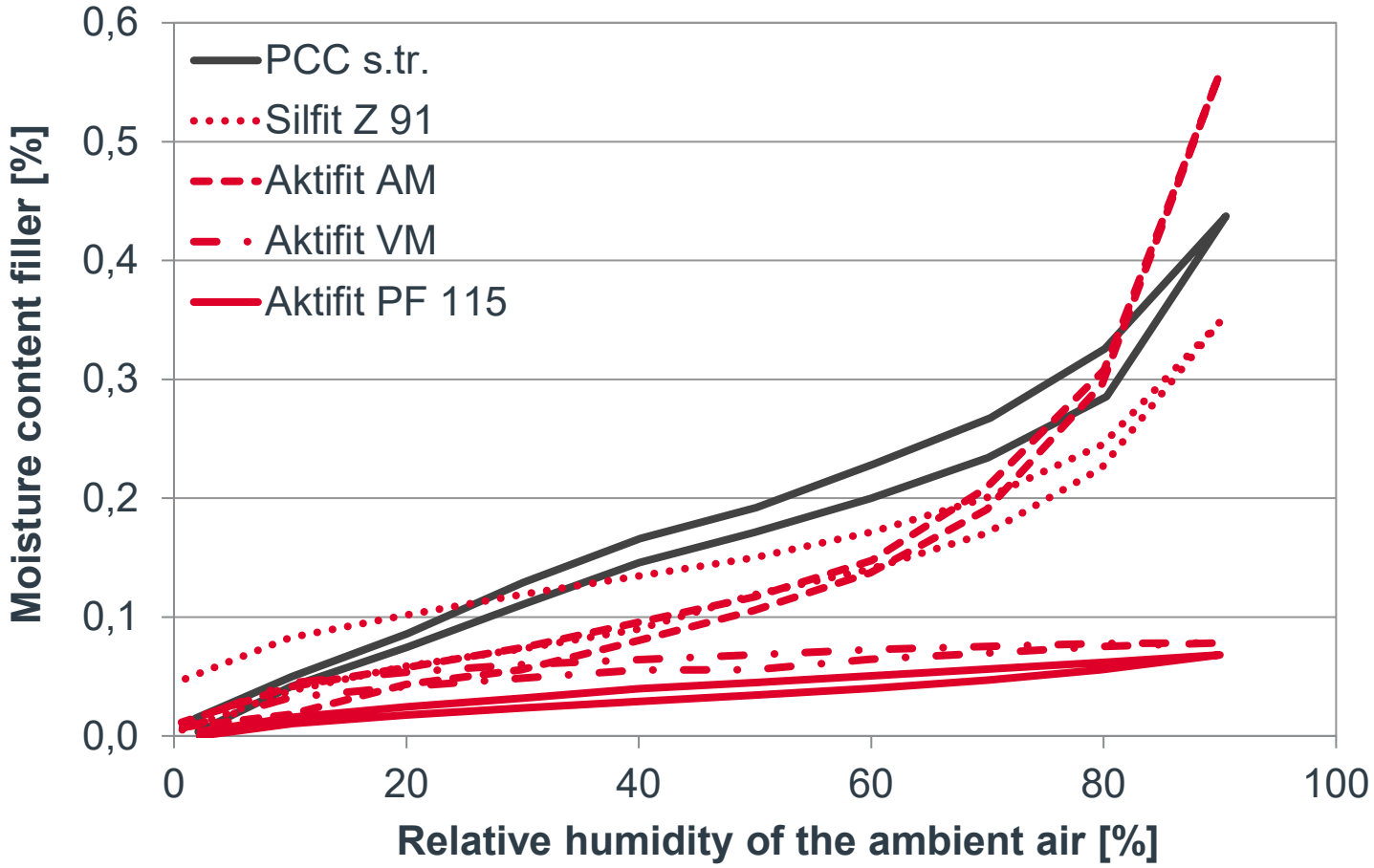
Formulations

	PCC	Calcined Neuburg Siliceous Earth				
	PCC s.tr.	Silfit Z 91	Aktifit VM	Aktifit AM	Aktifit PF 115	Aktifit AM variation
MS Polymer™	42.0	42.0	42.0	42.0	42.0	42.0
Rheological additive	---	1.0	1.0	1.0	1.0	1.0
Filler	52.7	52.7	52.7	52.7	52.7	52.7
Drying agent	2.3	2.3	2.3	2.3	2.3	2.3
Adhesion promoter AMMO	1.5	1.5	1.5	1.5	1.5	---
Adhesion promoter AMEO	---	---	---	---	---	1.5
Adhesion promoter Dynasylan 1146	---	---	---	---	---	1.5
Catalyst	1.5	1.5	1.5	1.5	1.5	1.5
Total	**100.0**	**101.0**	**101.0**	**101.0**	**101.0**	**102.5**

Fillers, Characteristics

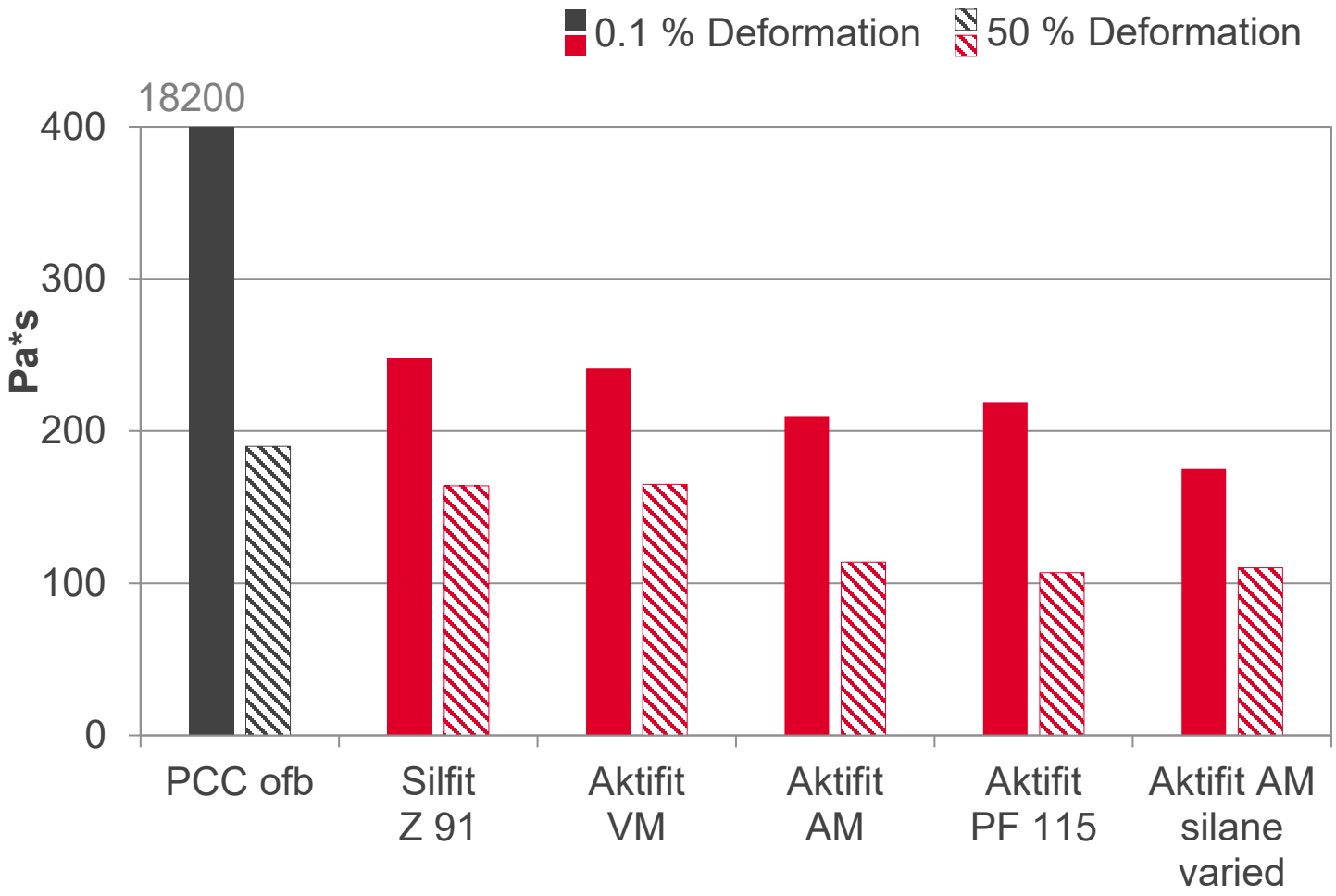
		PCC	Calcined Neuburg Siliceous Earth			
		PCC s.tr.	Silfit Z 91	Aktifit VM	Aktifit AM	Aktifit PF 115
Volatile matter At 105 °C	%	0.3	0.2	0.1	0.2	0.1
Oil absorption	g/100g	34	55	55	55	59
Specific surface area BET	m²/g	16	8	7	7	8
Functionalization		Fatty acids	---	Vinyl	Amino	Special amino
Surface character		hydrophobic	hydrophilic	hydrophobic	hydrophilic	hydrophobic

Moisture Content of Fillers vs. Ambient Air Humidity



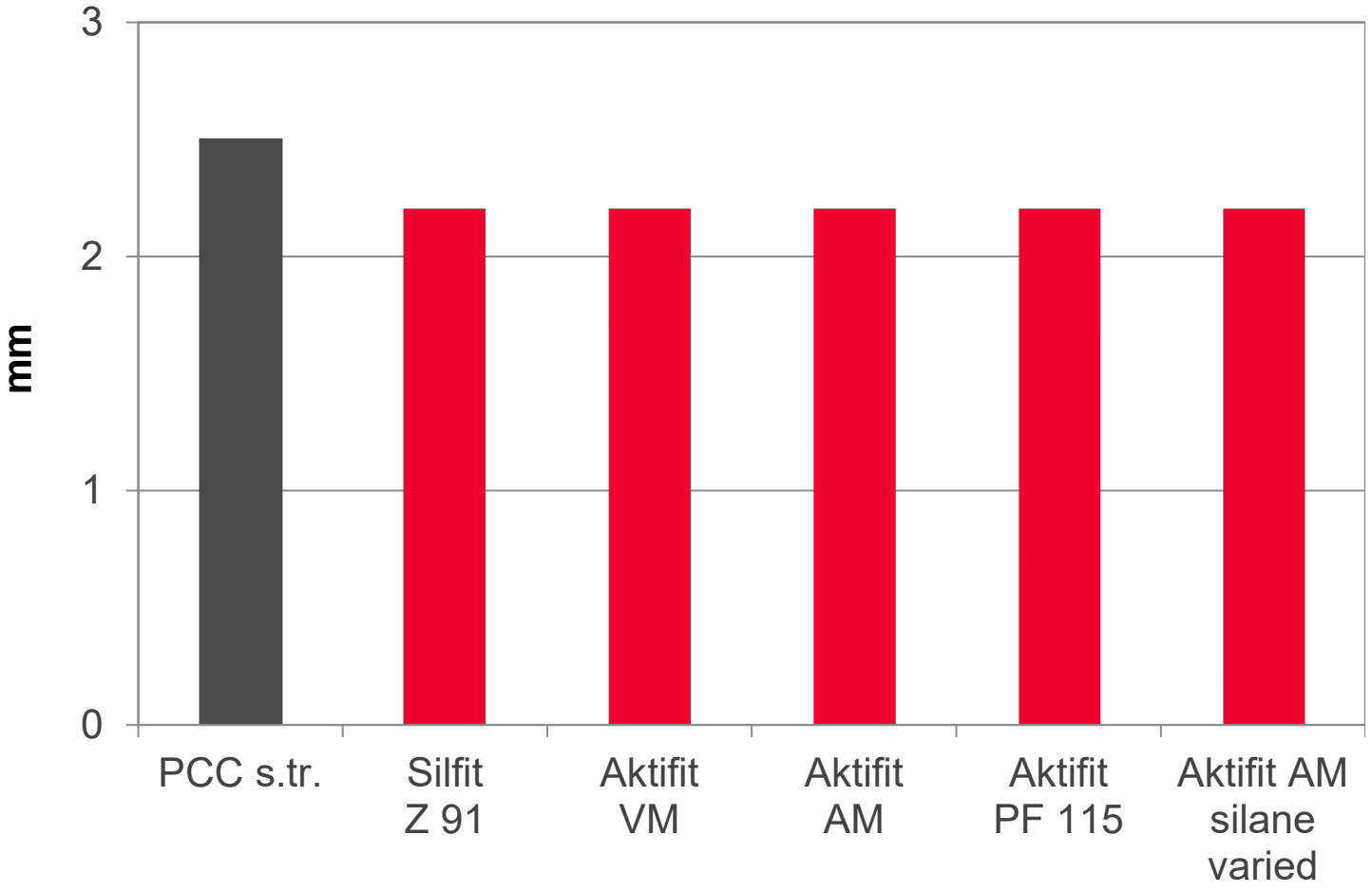
Complex Viscosity

DIN 54458



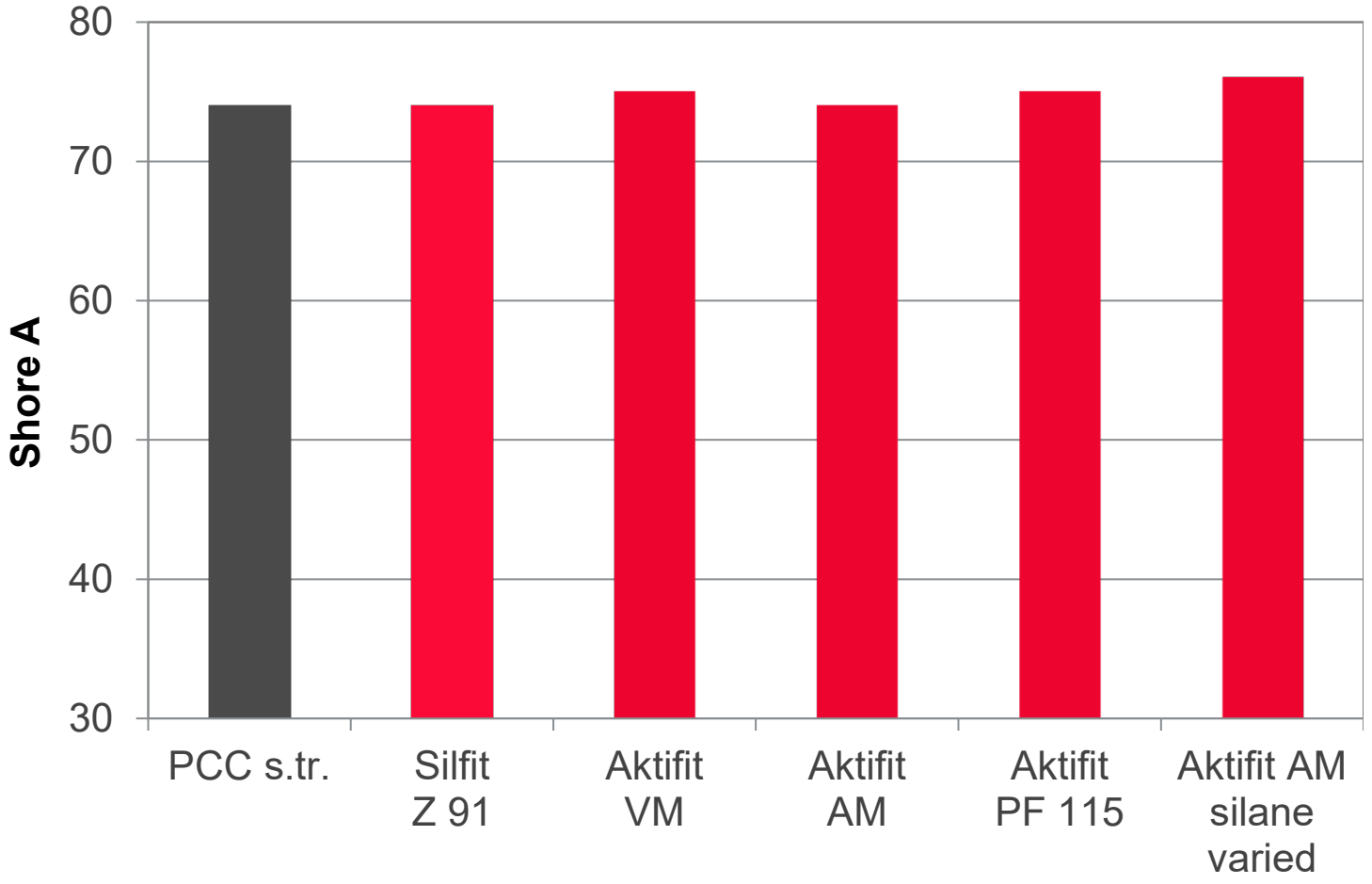
In-depth Cure

Thickness of the reacted layer after 24 h



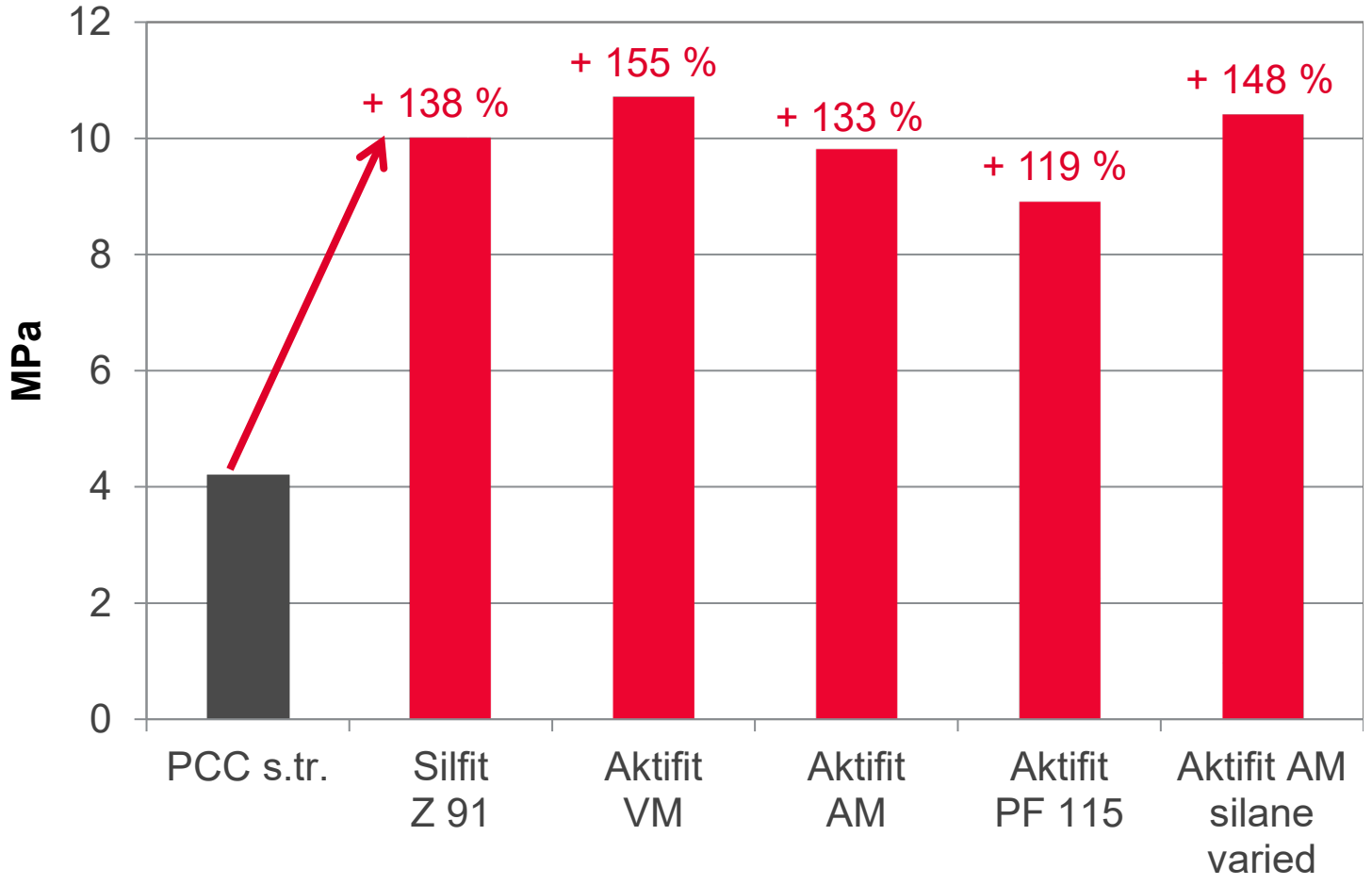
Hardness Shore A

DIN ISO 7619-1



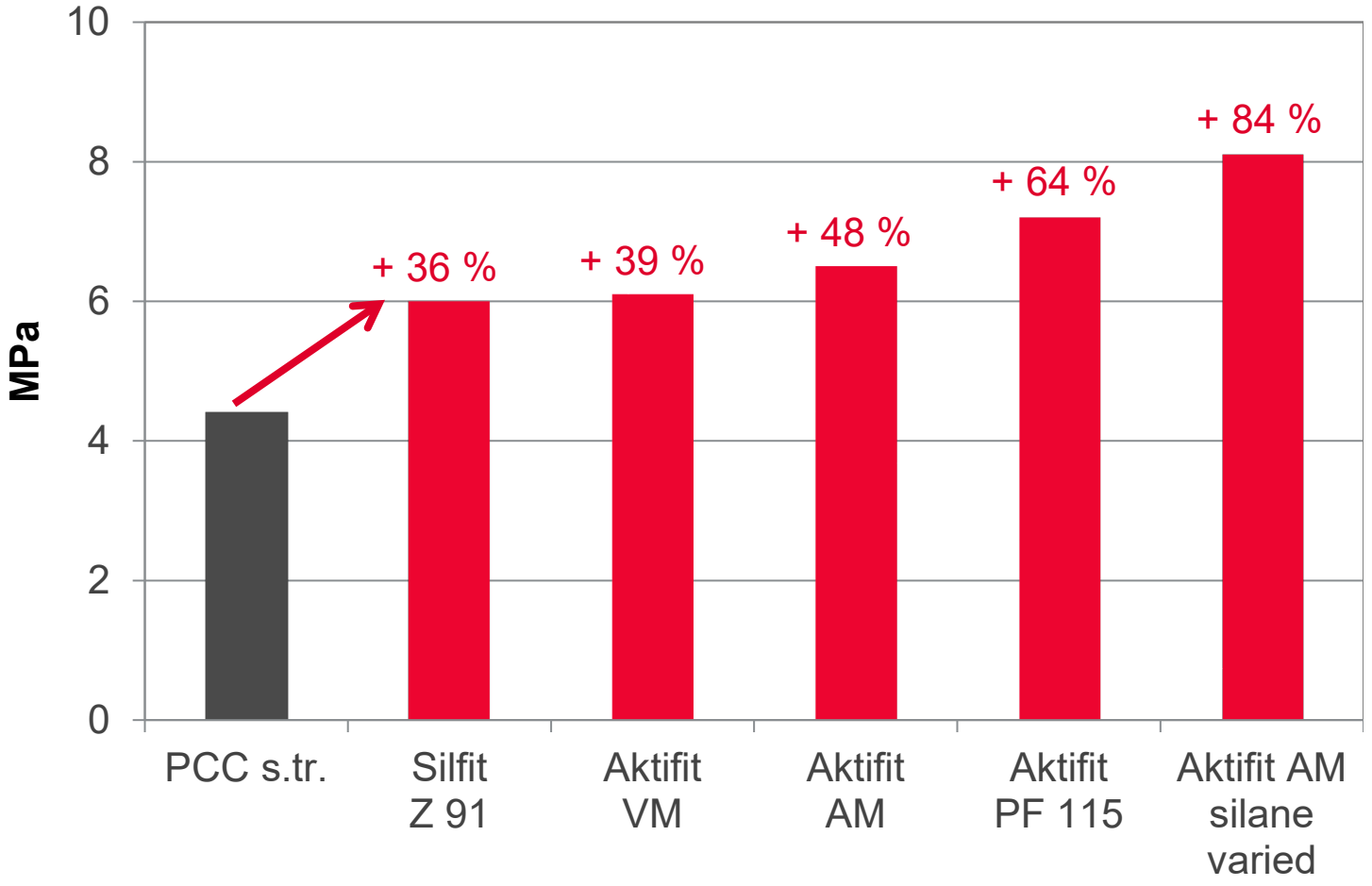
Tensile Strength

DIN 53504, S2



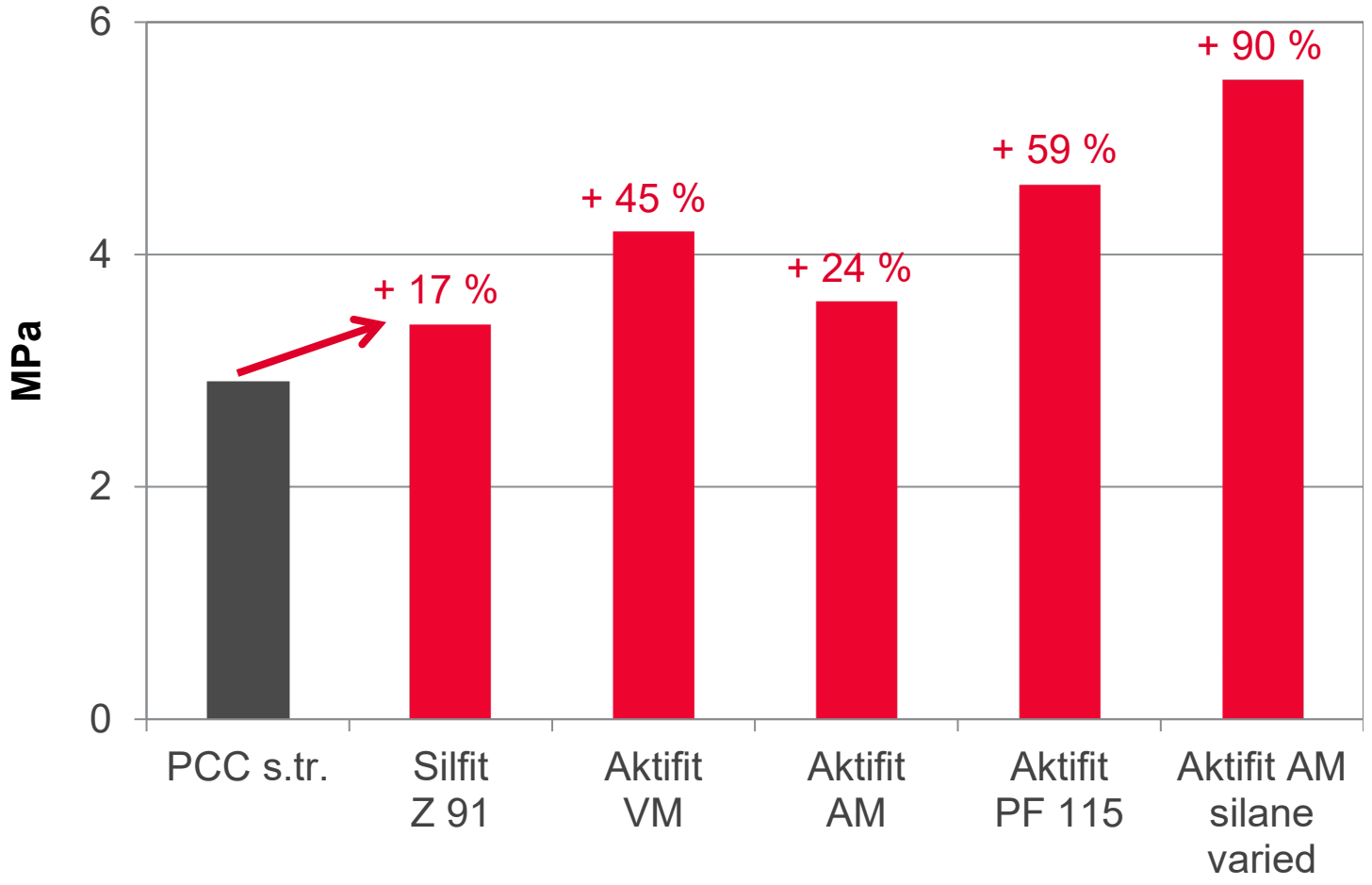
Lap Shear Strength
Beech Wood

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



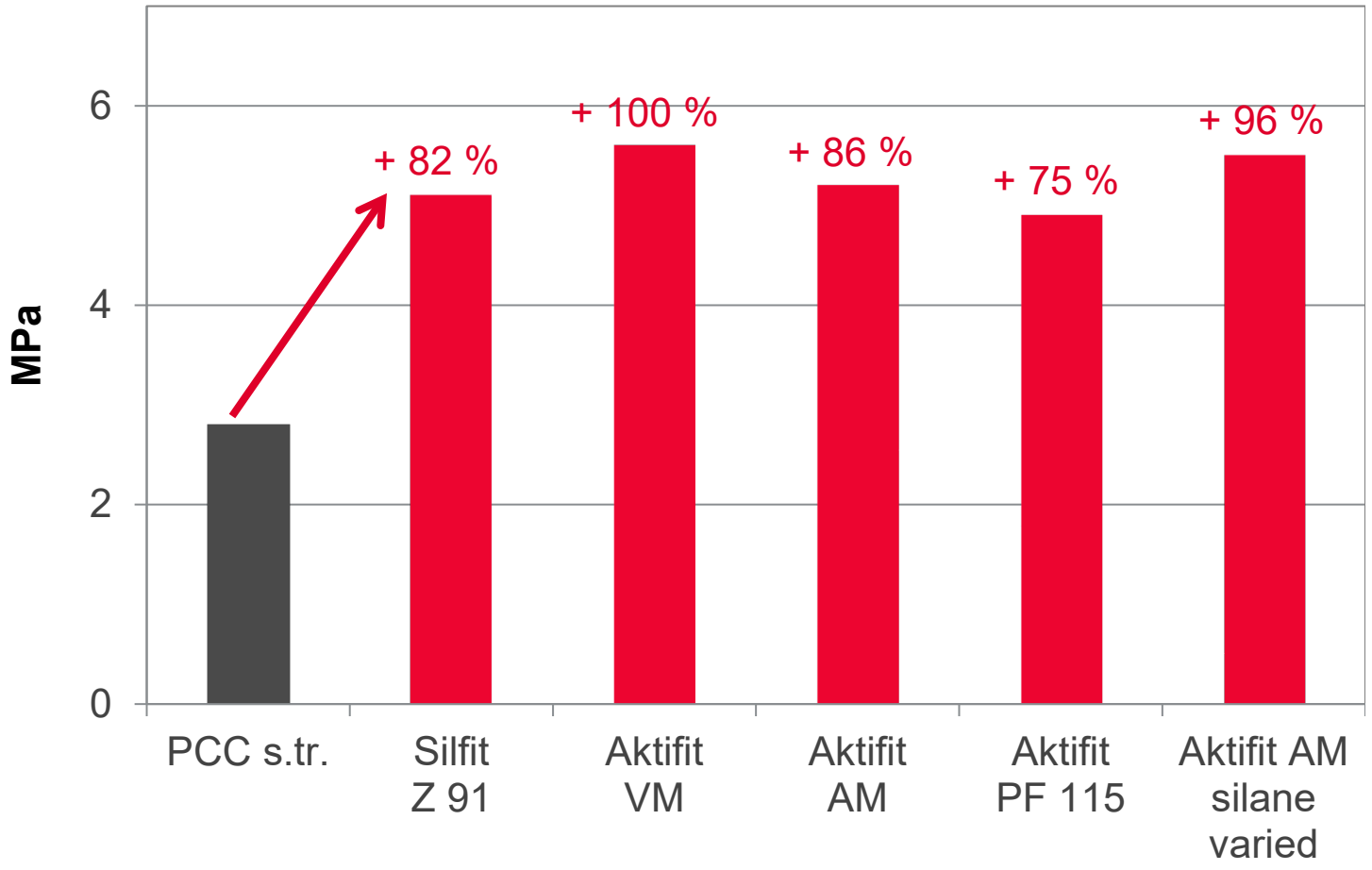
Lap Shear Strength
Beech Wood

DIN EN 205, after 28 d, layer thickness 1 mm



Lap Shear Strength Aluminum

DIN EN 1465, after 28 d, layer thickness 1 mm



Summary

Compared to the precipitated and surface treated calcium carbonate with high specific surface, Calcined Neuburg Siliceous Earth shows:

- lower viscosity at higher deformation, thereby easier application.
- viscosity at low deformation / yield point variably adjustable.
- increased tensile strength of up to 11 MPa or improvement of up to 150 %.
- marked increase of lap shear strength, more than 8 MPa or improvement of up to 100 % possible.

Distinguishing Features of the NSE Grades

- **Silfit Z 91** — Low moisture content, white and color-neutral, cost effective, good mechanical properties.
- **Aktifit VM** — Very low moisture content and extremely low moisture absorption even under humid conditions, white and color-neutral, especially for thick joints in wood and metal.
- **Aktifit AM** — Low moisture content, white and color-neutral, low and thereby adjustable viscosity, especially for thin joints in wood.
- **Aktifit PF 115** — Very low moisture content and extremely low moisture absorption even under humid conditions, white and color-neutral, low and thereby adjustable viscosity, for highest requirements on thin joints in wood.
- **Aktifit AM** + adhesion promoter — Lap shear strength on wood can be optimized with a combination of Aktifit AM and varied/increased adhesion promoter.

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Preparation of Batches

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

- **Feed polymer**
- **Stir in rheological additive and (undried) filler**
  - 10 min at 3000 rpm and 500 rpm, vacuum
  - 20 min at 1500 rpm and 300 rpm, vacuum
  - Cool down to <50 °C
- **Add vinyl silane (drying agent)**
  - 5 min at 1000 rpm and 200 rpm
- **Add amino silane (adhesion promoter)**
  - 5 min at 1000 rpm and 200 rpm
- **Add catalyst**
  - 5 min at 1000 rpm and 200 rpm
- **Deaerate**
  - 5 min at 500 rpm and 150 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 Testing after 28 d
Hardness Shore A	DIN ISO 7619-1, piled S2 specimens Curing / conditioning: 28 d @ standard conditions 23/50
Tensile test	DIN 53504, S2 specimens Curing / conditioning: 28 d @ standard conditions 23/50
Lap shear test Wood	DIN EN 204/205 Substrate: beech wood Adhesive layer: 0.1 mm and 1 mm Curing: 7 resp. 28 d @ standard conditions 23/50 Crosshead speed: 50 mm/min
Lap shear test Aluminum	DIN EN 1465 Substrate: Aluminum 99.5 % Adhesive layer: 1 mm Curing: 28 d @ standard conditions 23/50 Crosshead speed: 10 mm/min

Table of Results

		PCC	Calcined Neuburg Siliceous Earth				
		PCC s.tr.	Silfit Z 91	Aktifit VM	Aktifit AM	Aktifit PF 115	Aktifit AM silane varied
Rheology							
Complex viscosity							
at 0.1 % deformation	Pa*s	18200	248	241	210	219	175
at 50 % deformation	Pa*s	190	164	165	114	107	110
Curing							
Skin formation	min	5	5	5	5	5	5
In-depth cure after 8 h	mm	1.4	1.3	1.3	1.3	1.3	1.3
In-depth cure after 24 h	mm	2.5	2.2	2.2	2.2	2.2	2.2
Mechanical properties							
Hardness	Shore A	74	74	75	74	75	76
Tensile strength	MPa	4.2	10.0	10.7	9.8	8.9	10.4
Elongation at break	%	271	136	148	161	156	155
Lap shear strength							
Beech wood, layer 0.1 mm	MPa	4.4	6.0	6.1	6.5	7.2	8.1
Beech wood, layer 1 mm	MPa	2.9	3.4	4.2	3.6	4.6	5.5
Aluminum, layer 1 mm	MPa	2.8	5.1	5.6	5.2	4.9	5.5