Calcined Neuburg Siliceous Earth in polyamides 6 and 66

Author: Petra Zehnder

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Contents

Status Quo

- Polyamides are used in a multitude of application areas, above all in the automotive and electrical industries.
- Important properties are - apart from easy processing – high surface quality, high stiffness and toughness, low warpage as well as good dimensional stability at elevated temperatures.
- Mineral fillers, also combined with glass fibers, are often added in order to achieve certain properties.
- Calcined clays or wollastonite with block-like grain shape are fillers typically used in parts with low warpage.

Objective

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Improving polyamide compounds by using Aktifit AM in place of conventional competitive fillers with particular focus on

» Flow

» Mechanical properties

- Heat deflection
- Hardness
- Tensile modulus
- Tensile strength
- Tensile strain at break
- Impact Strength

» Graying of black parts

Compounding
Injection Molding

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Composition	60 % Polyamide 40 % Mineral filler
Compounding	Twin screw extruder ZSK 30 no predrying of filler Output: approx. 6 kg/h
Injection molding of test specimen acc. to ISO 1874	Mold acc. to ISO 294 Mold temperature: 80 °C Melt temperature PA 6: 260 °C Melt temperature PA 66: 305 °C Flow front speed: 200 mm/s

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Fillers and Characteristics

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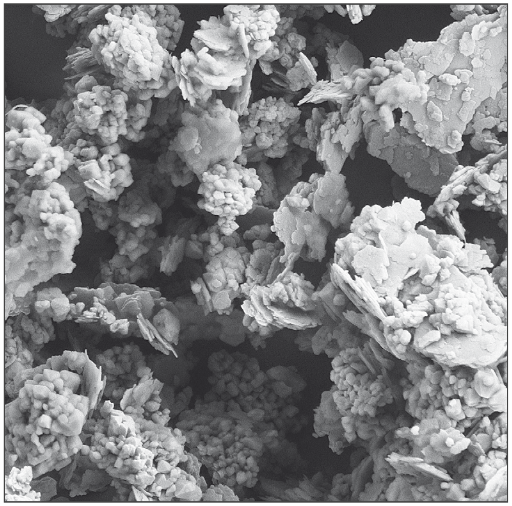
		Calcined clay		Wollastonite	Calcined Neuburg Siliceous Earth	
		Grade 1	Grade 2	block-like particle structure	Silfit Z 91	Aktifit AM
Color value L* (CIELAB)		96.3	96.0	96.7	95.1	95.1
Color value a* (CIELAB)		-0.1	-0.4	-0.2	-0.2	-0.2
Color value b* (CIELAB)		2.5	2.2	1.0	1.0	1.0
Particle size d_{50}	[µm]	3.8	3.4	3.4	1.9	1.9
Particle size d_{97}	[µm]	13	14	14	10	10
Oil absorption	[g/100g]	68	61	33	60	61
Specific surface area BET	[m²/g]	5.8	9.1	3.6	7.4	6.6
Sieve residue > 40 µm	[mg/kg]	180	44	28	8	21
Functionalization		Amino		Amino	none	Amino

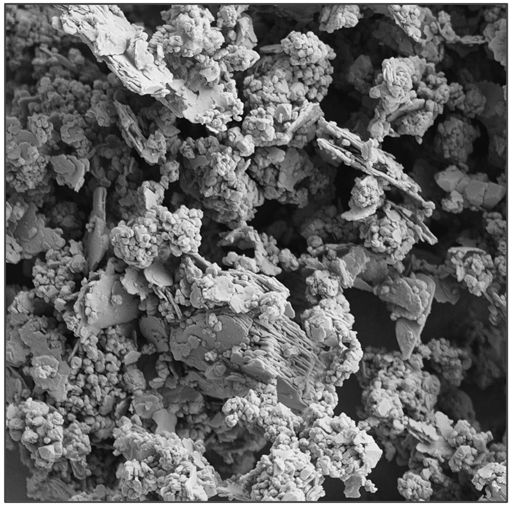
Calcined Neuburg Siliceous Earth

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.

Selection

Polyamide 6	Polyamide 66
• **Durethan® B 31 SK** black-colored ® registered trademark of LANXESS	• **Ultramid® A3K** natural-colored ® registered trademark of BASF
	• **Schulamid® 66 MV 2** natural-colored (only summary tables) ® registered trademark of A. Schulman Inc.

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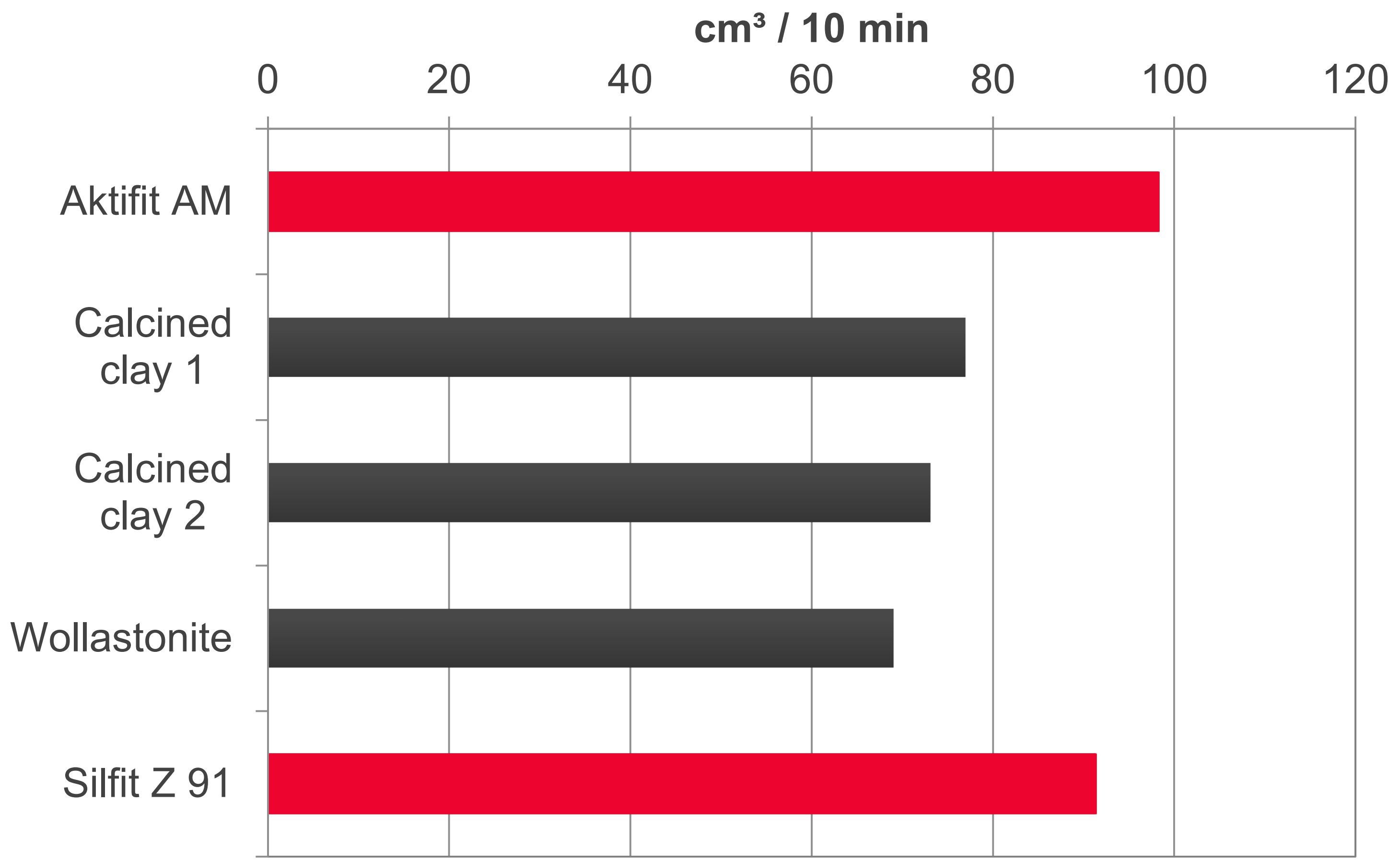
Melt Volume-flow rate

DIN ISO 1133, 275 °C, 5 kg



Heat Deflection
Heat Distortion Temperature

DIN EN ISO 75-1, -2; Method B (0.45 MPa)

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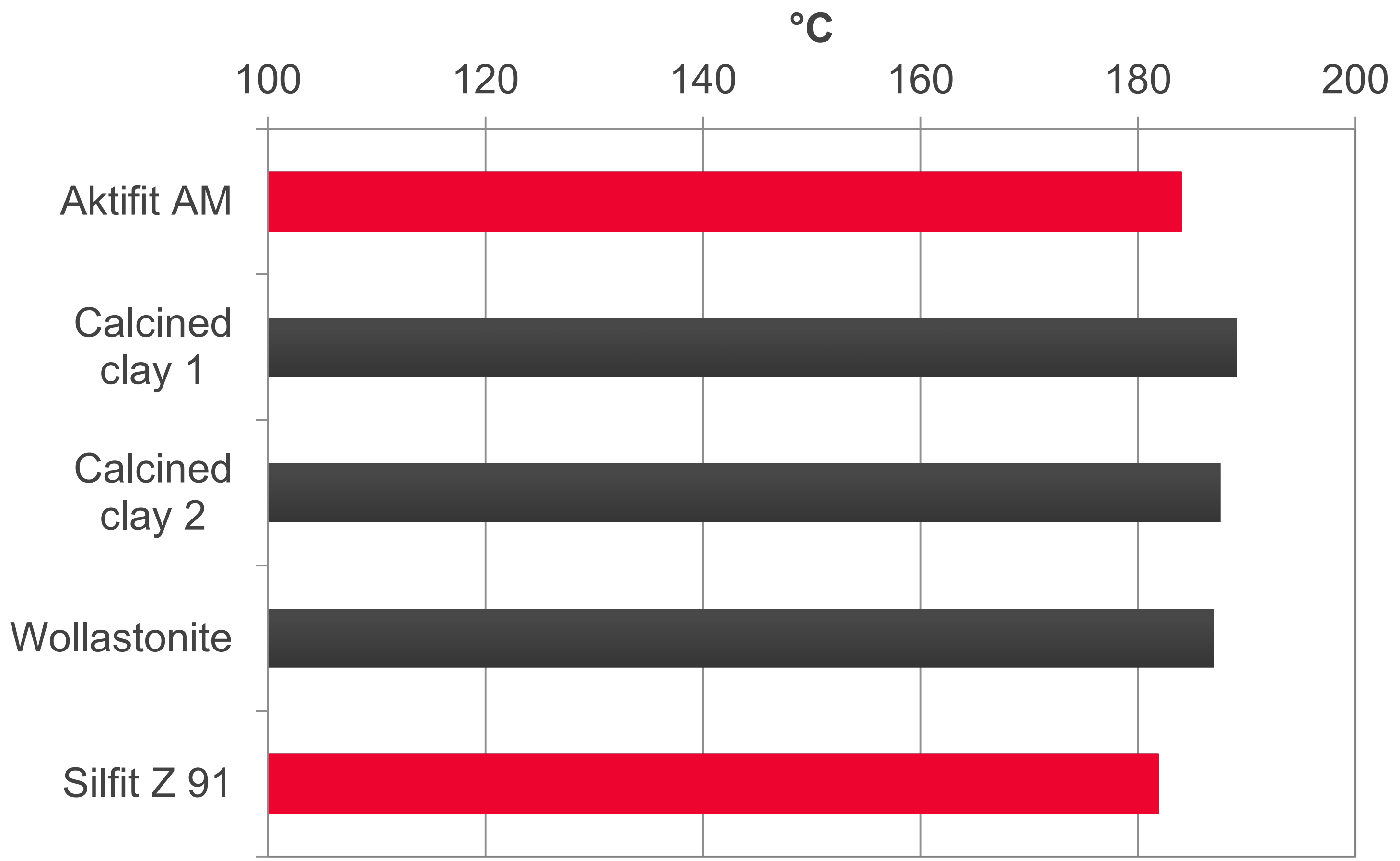

Ball Indentation Hardness

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DIN EN ISO 2039-1; H961N/30s

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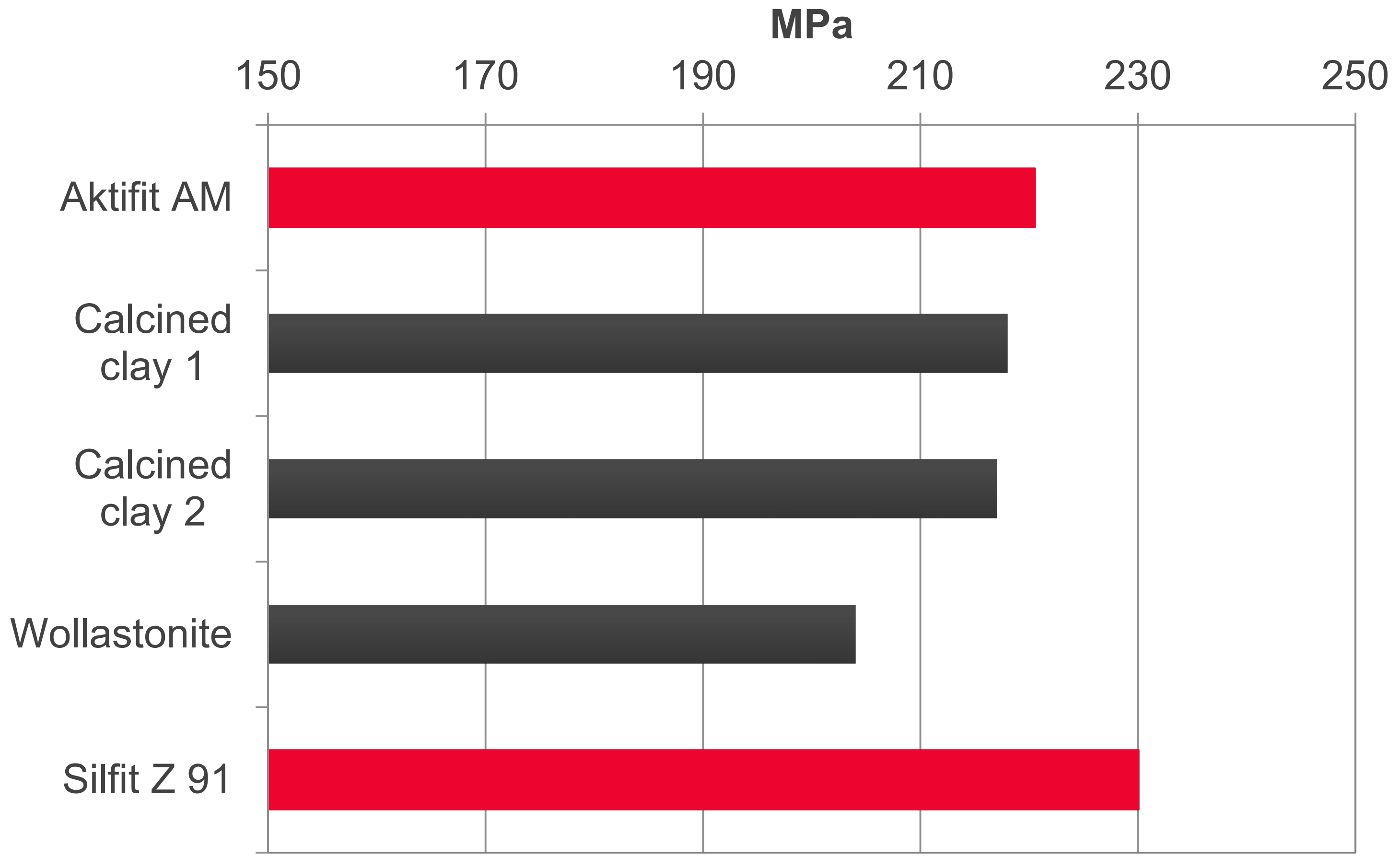

Tensile Modulus

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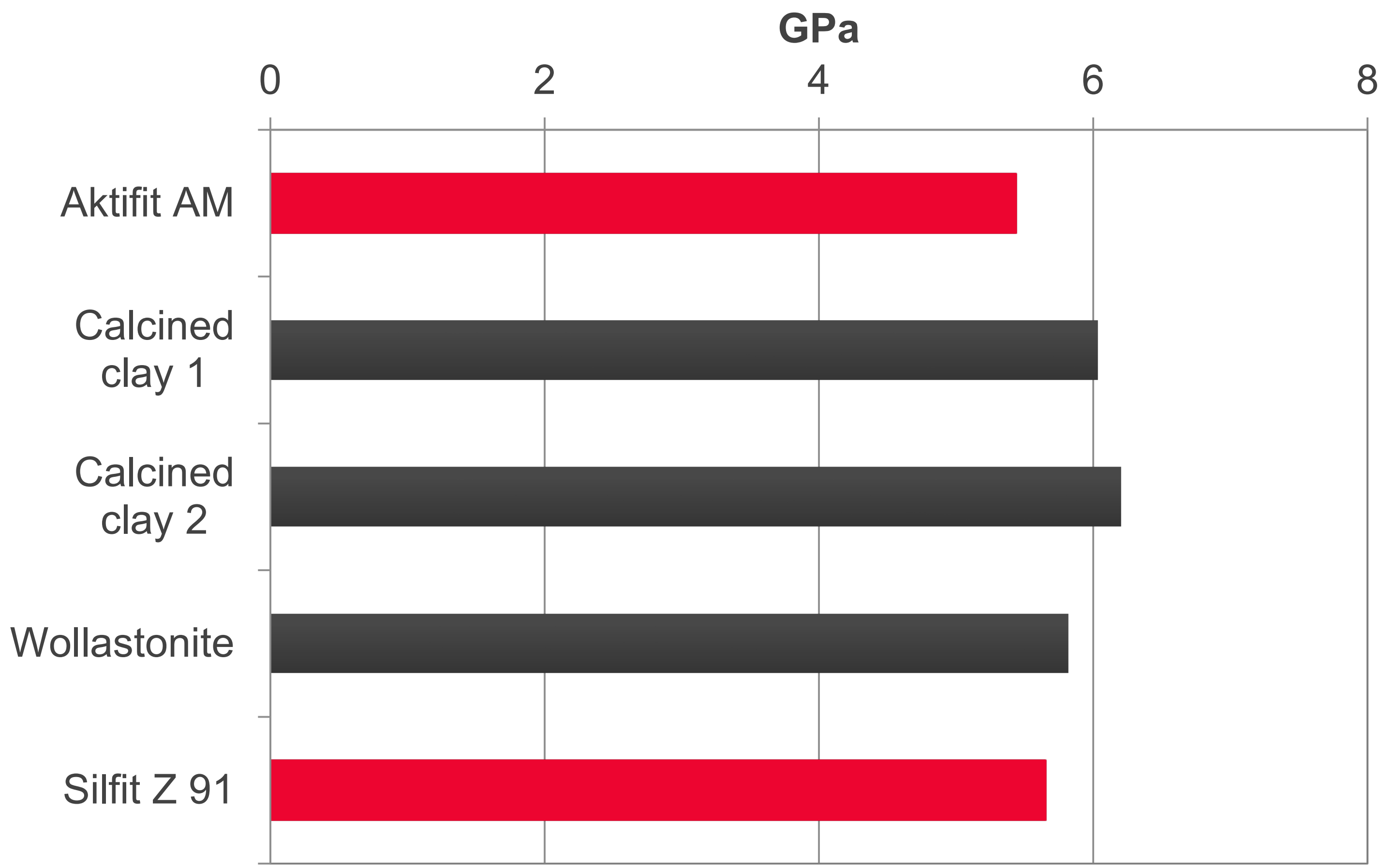
DIN EN ISO 527-1,-2; 0.5 mm/min



Tensile Strength

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DIN EN ISO 527-1,-2; 5 mm/min

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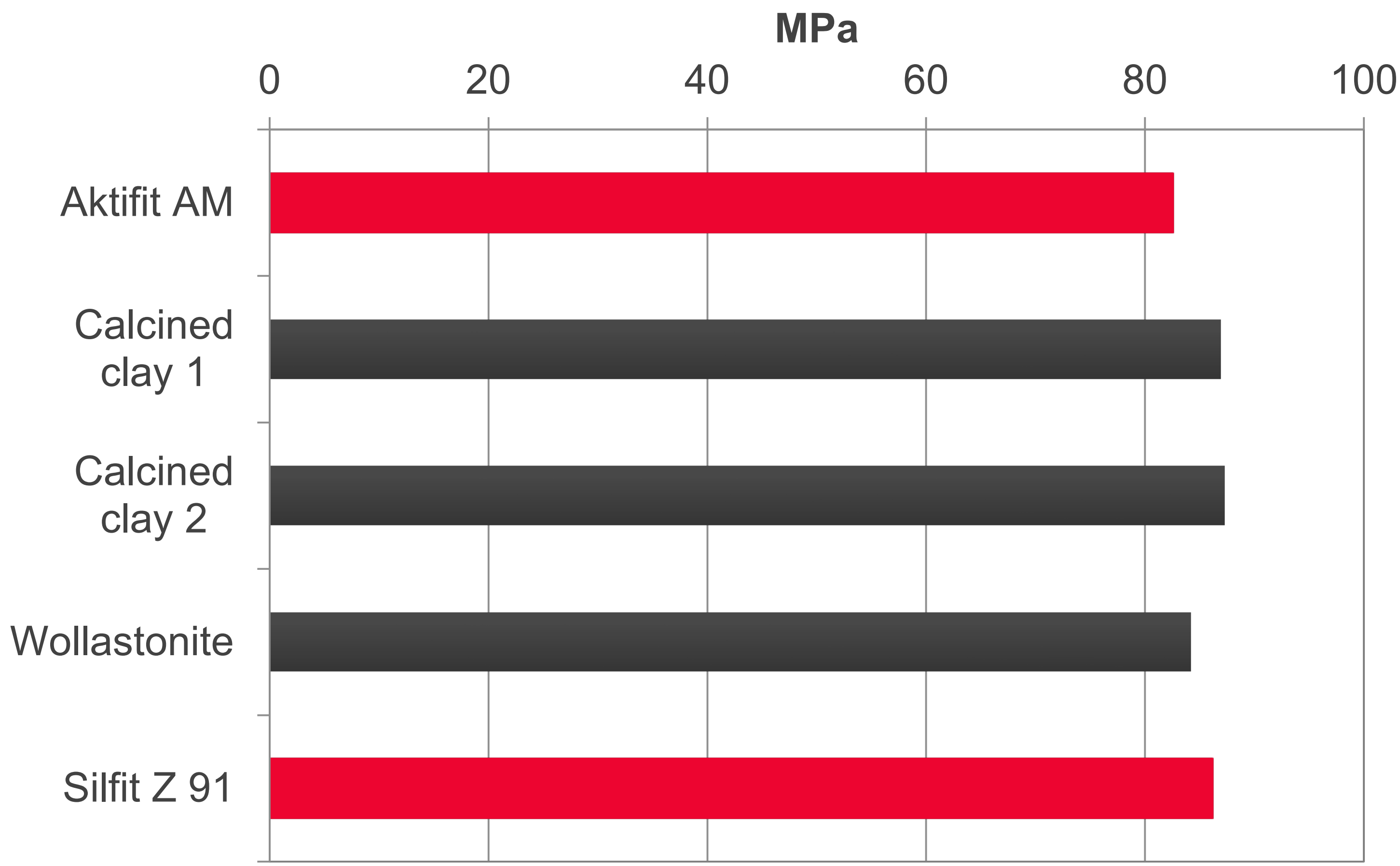

Tensile Strain at Break

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DIN EN ISO 527-1,-2; 5 mm/min

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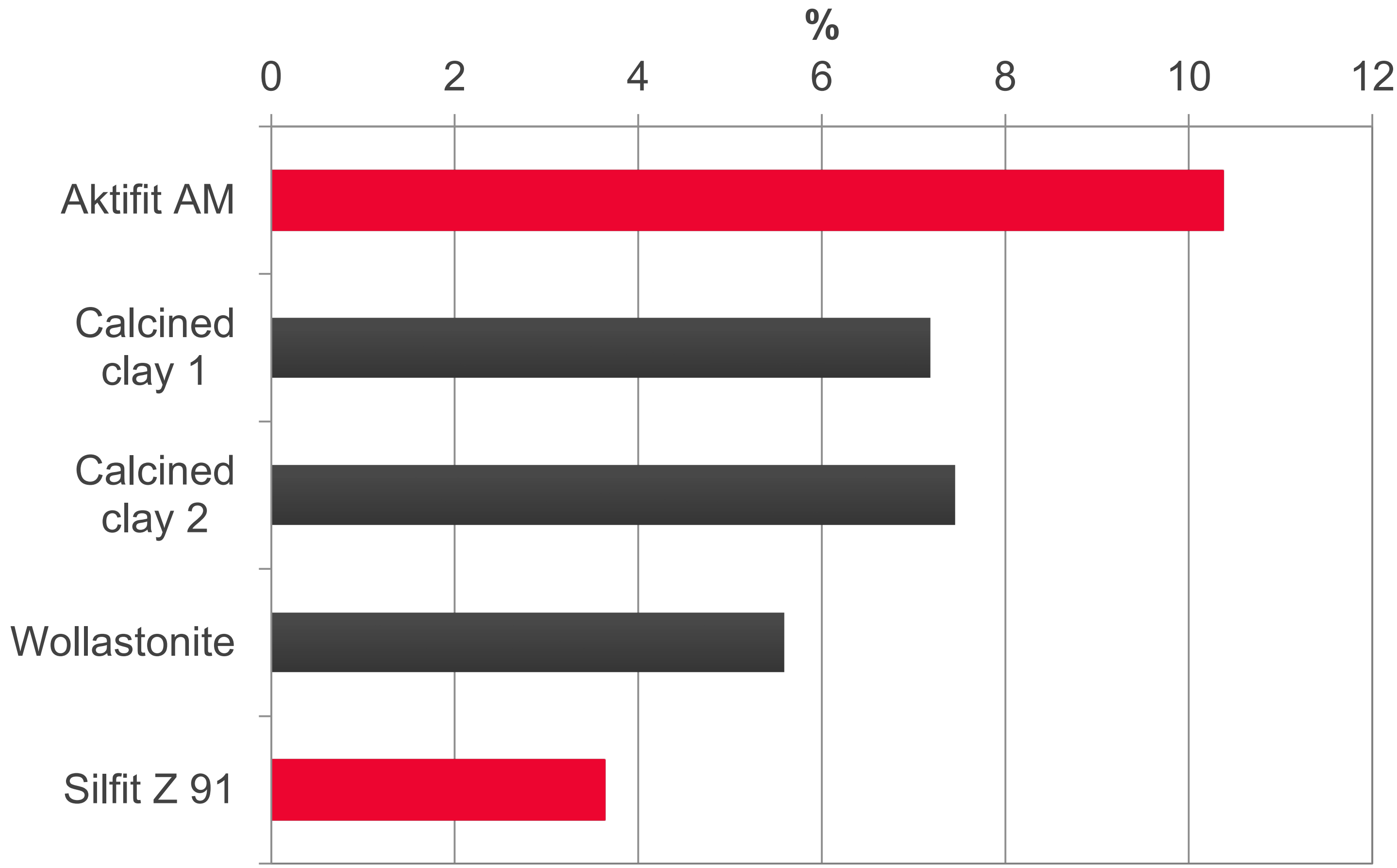
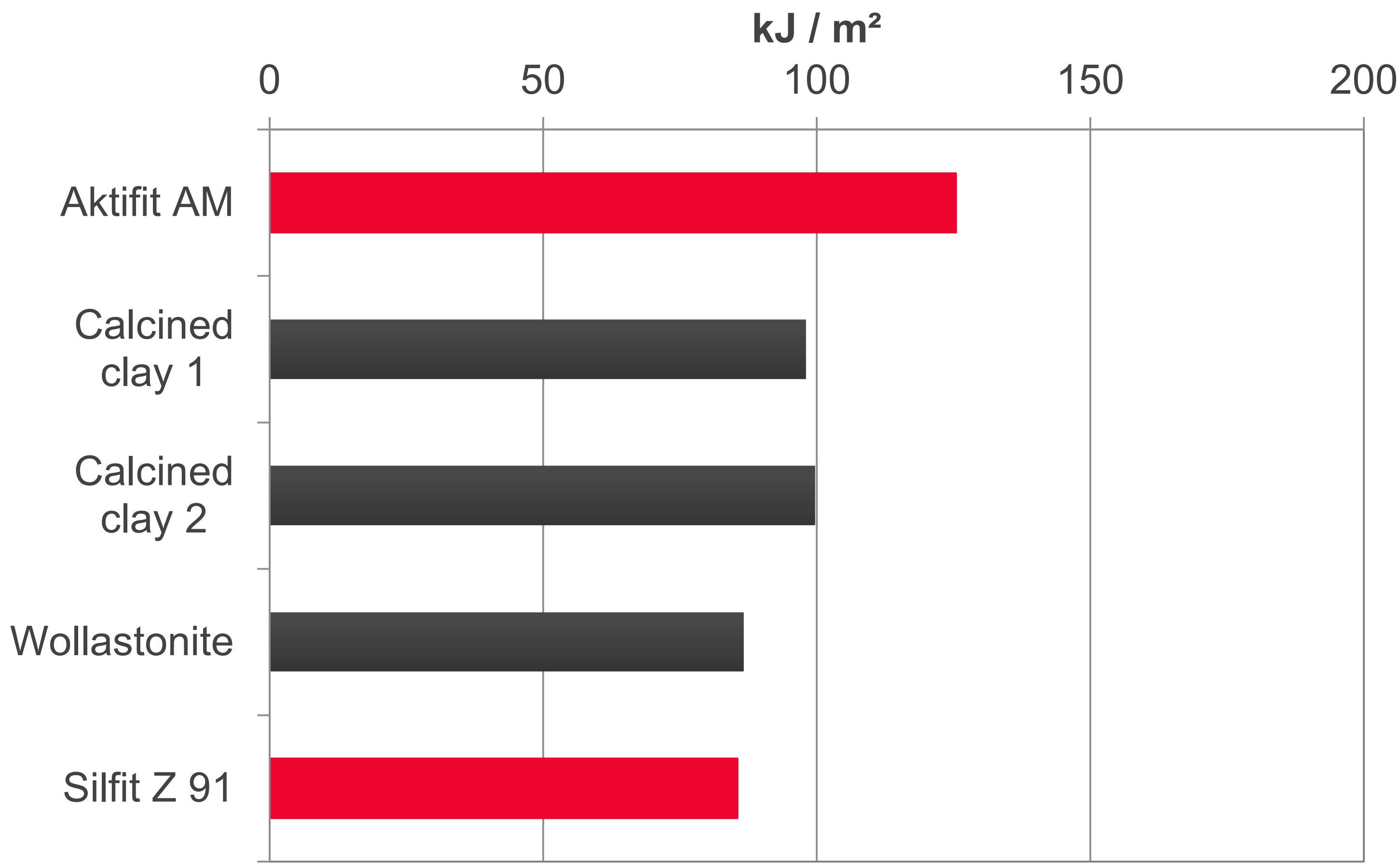


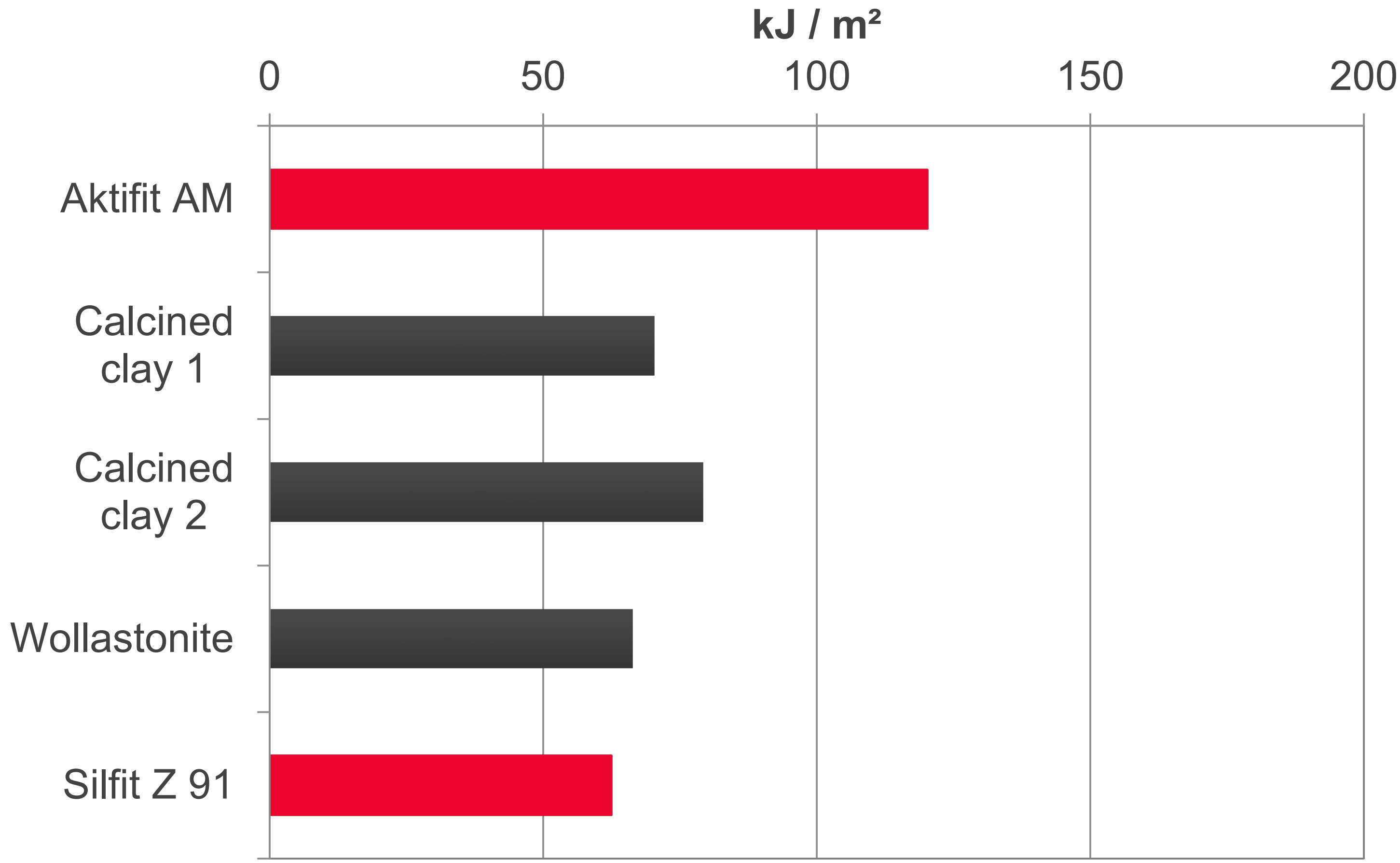
Impact Strength
Charpy 23 °C

DIN EN ISO 179-1 / 1eU



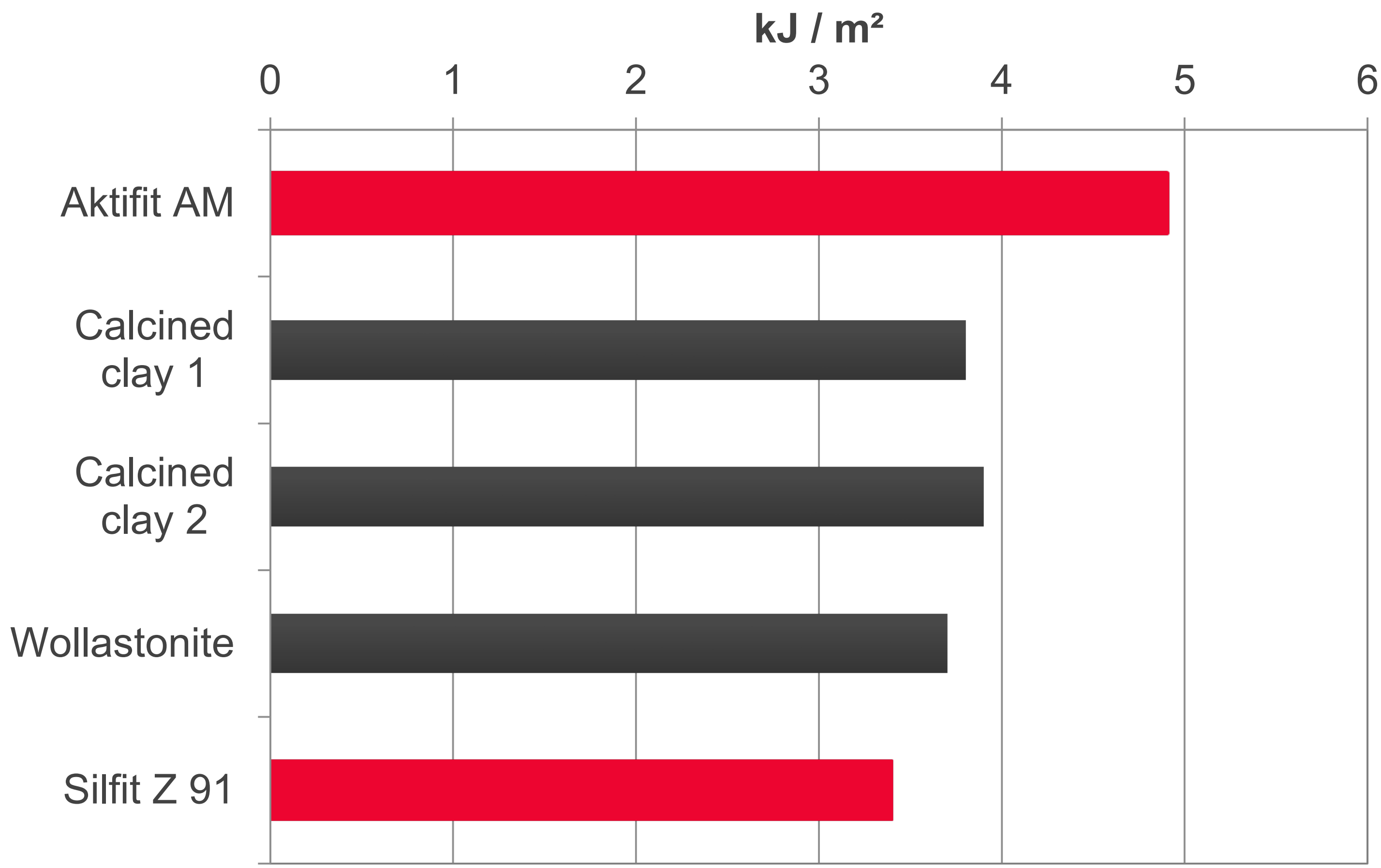
Impact Strength
Izod 23 °C

DIN EN ISO 180/U



Notched Impact Strength
Charpy 23 °C

DIN EN ISO 179-1 / 1eA



Black Coloring without Graying

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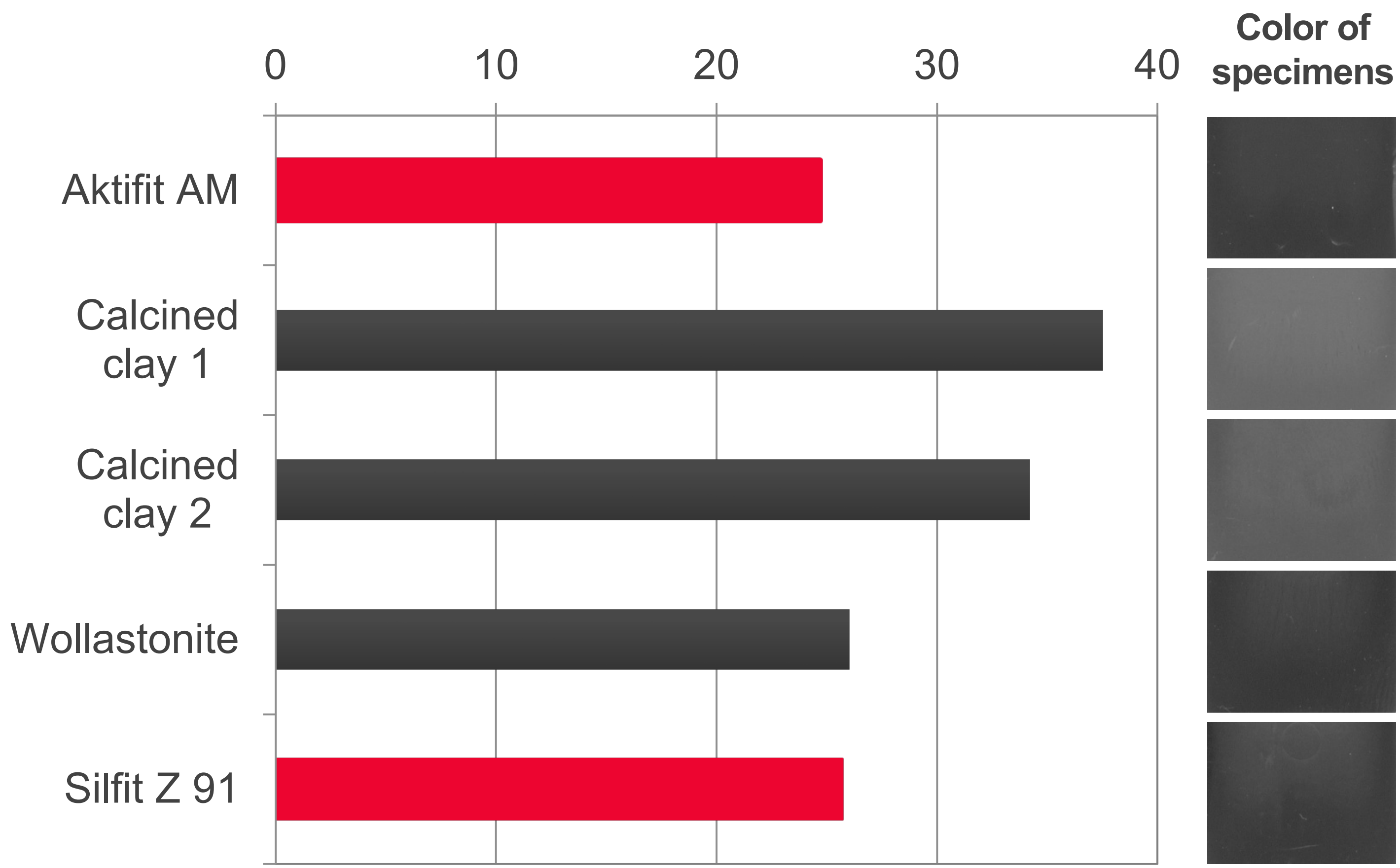
ISO 7724, CIE-Lab Lightness L*



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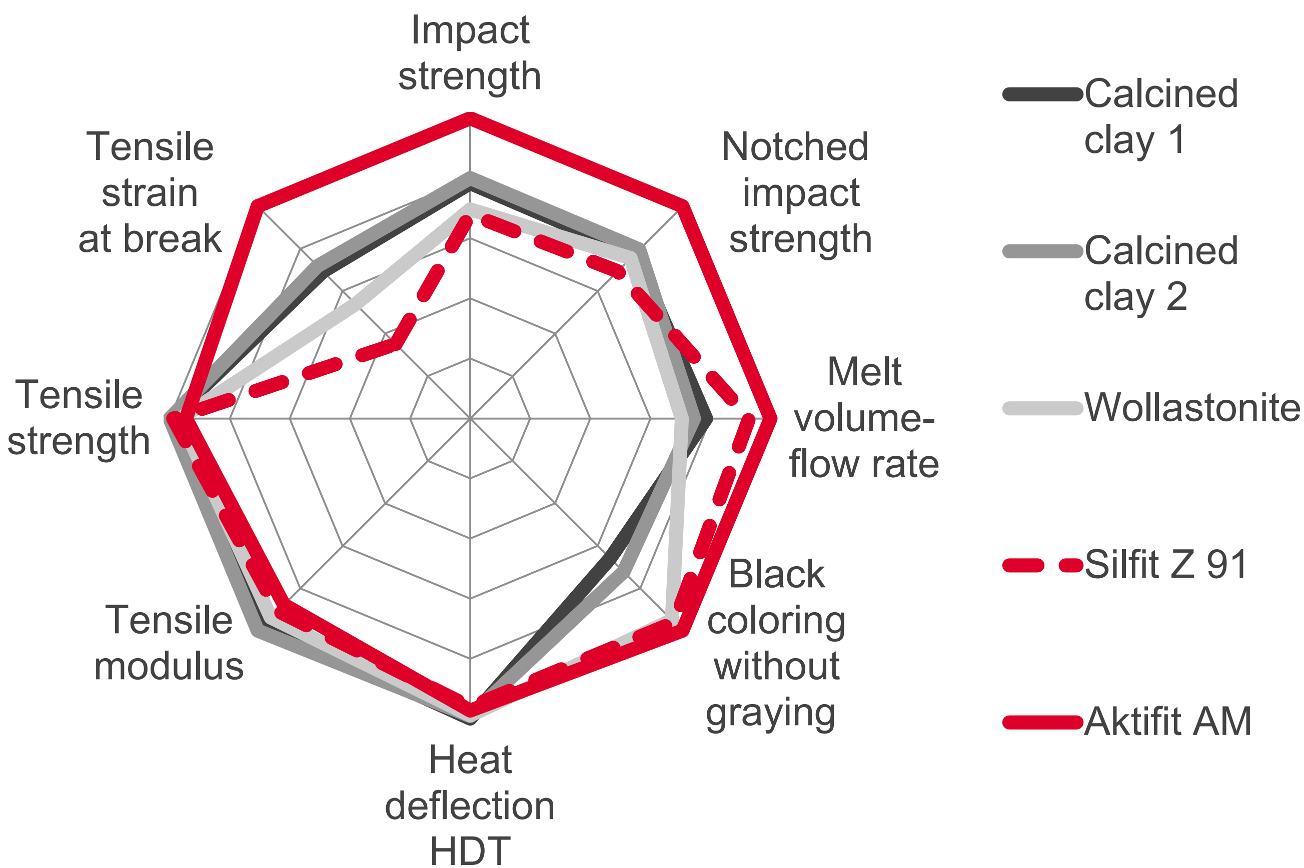

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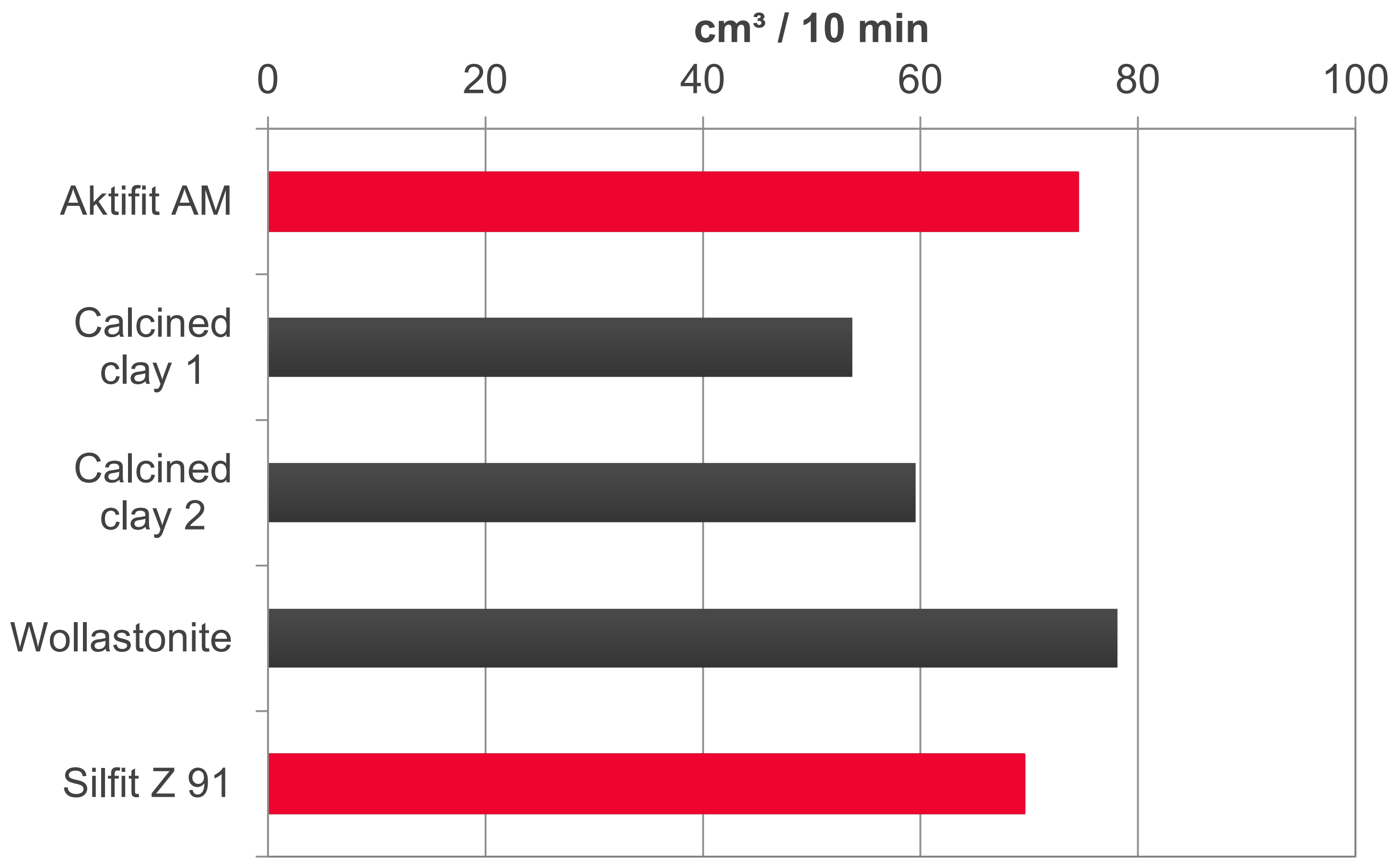
Melt Volume-flow Rate

DIN ISO 1133, 275 °C, 5 kg



Heat Deflection
Vicat Softening Point

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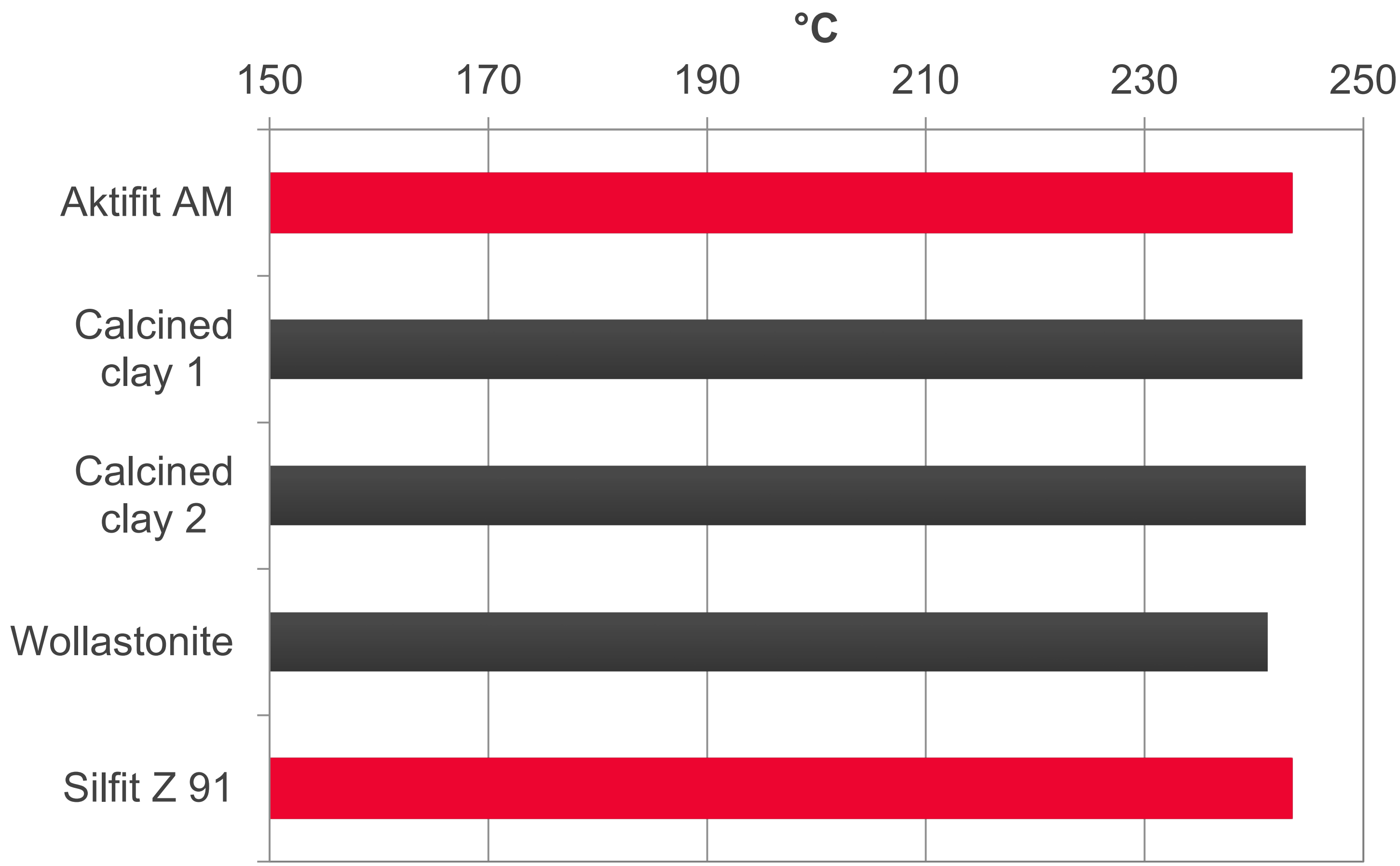
DIN ISO 306; B50



Heat Deflection
Heat Distortion Temperature

DIN EN ISO 75-1, -2; Method B (0.45 MPa)

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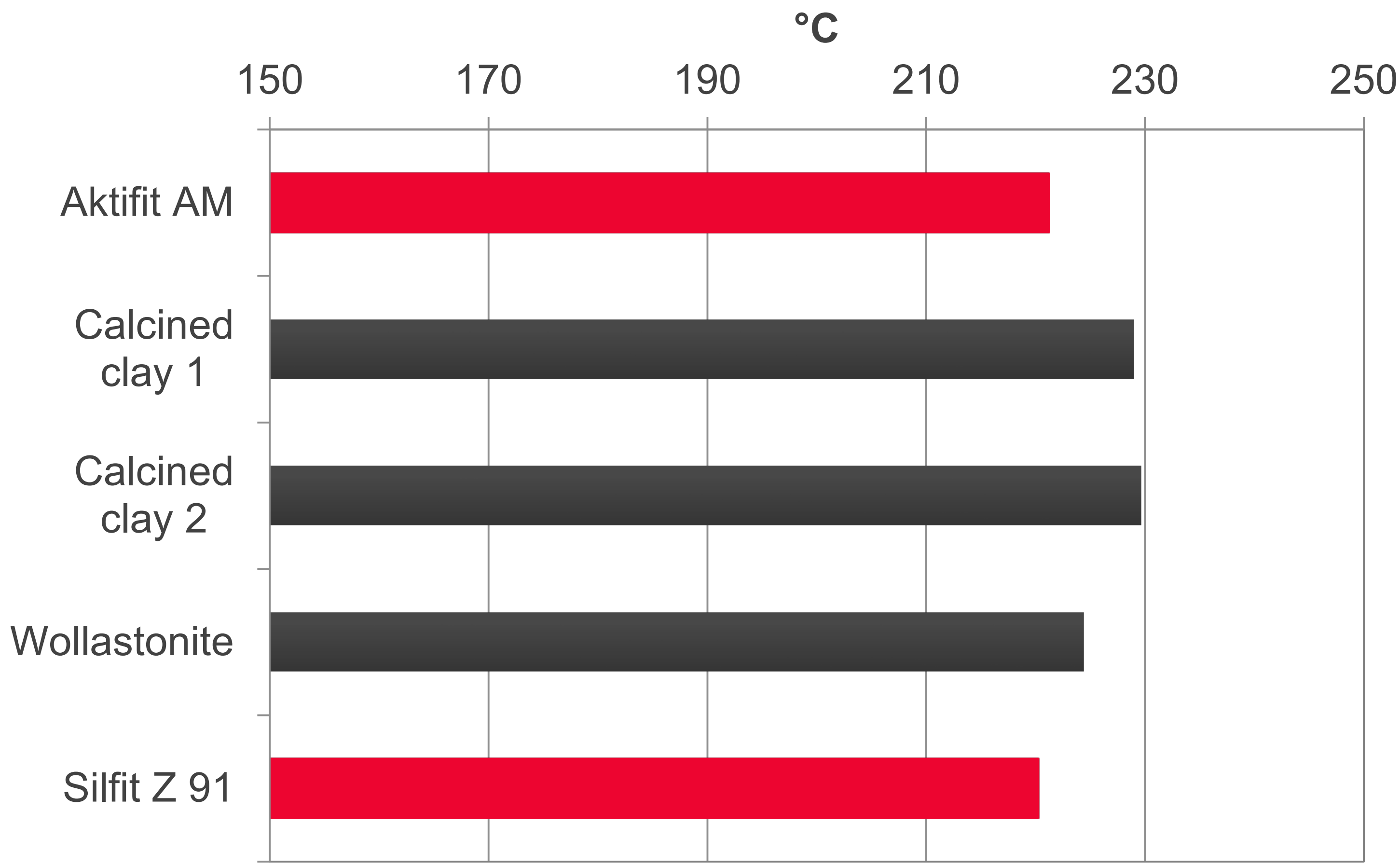
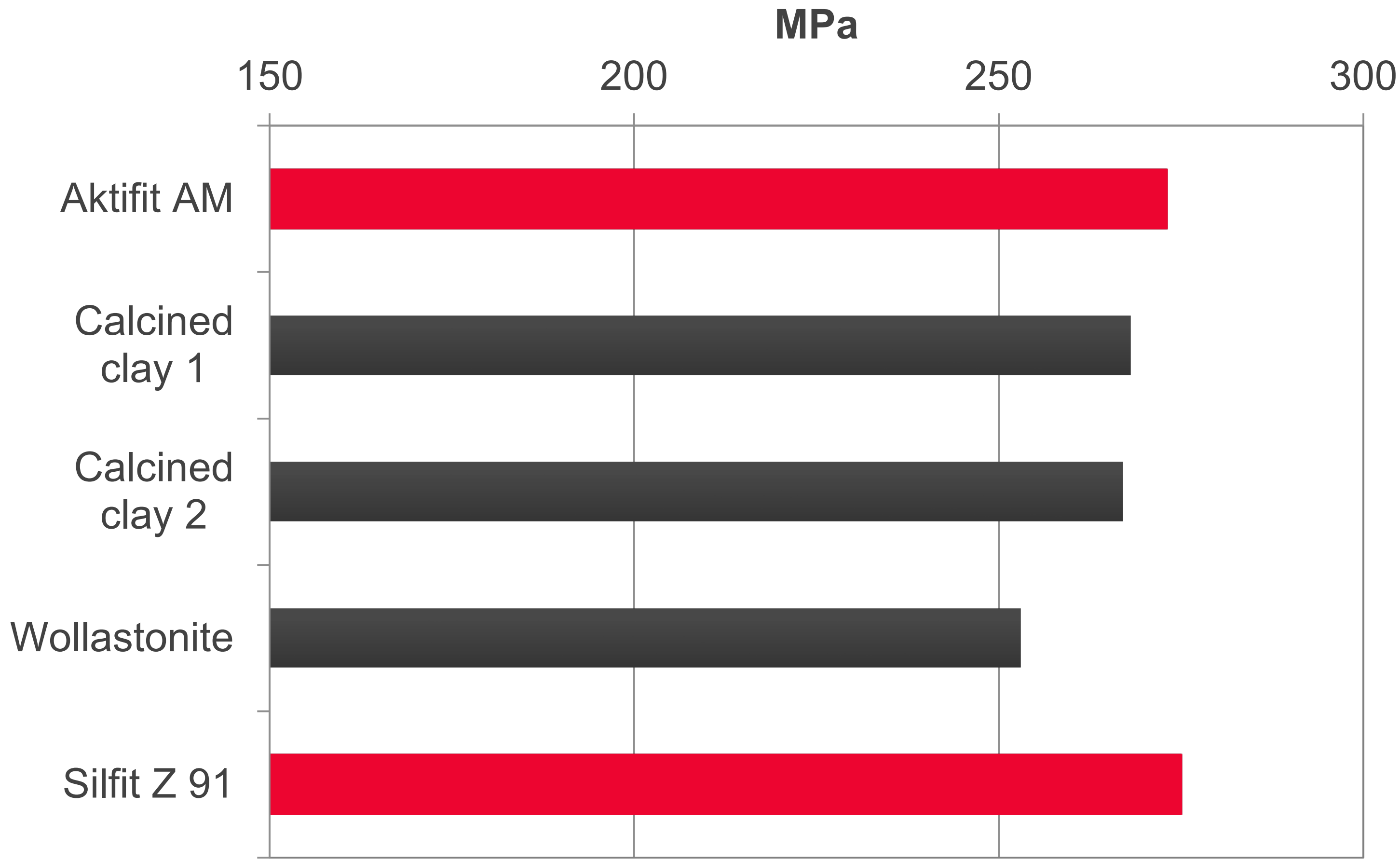


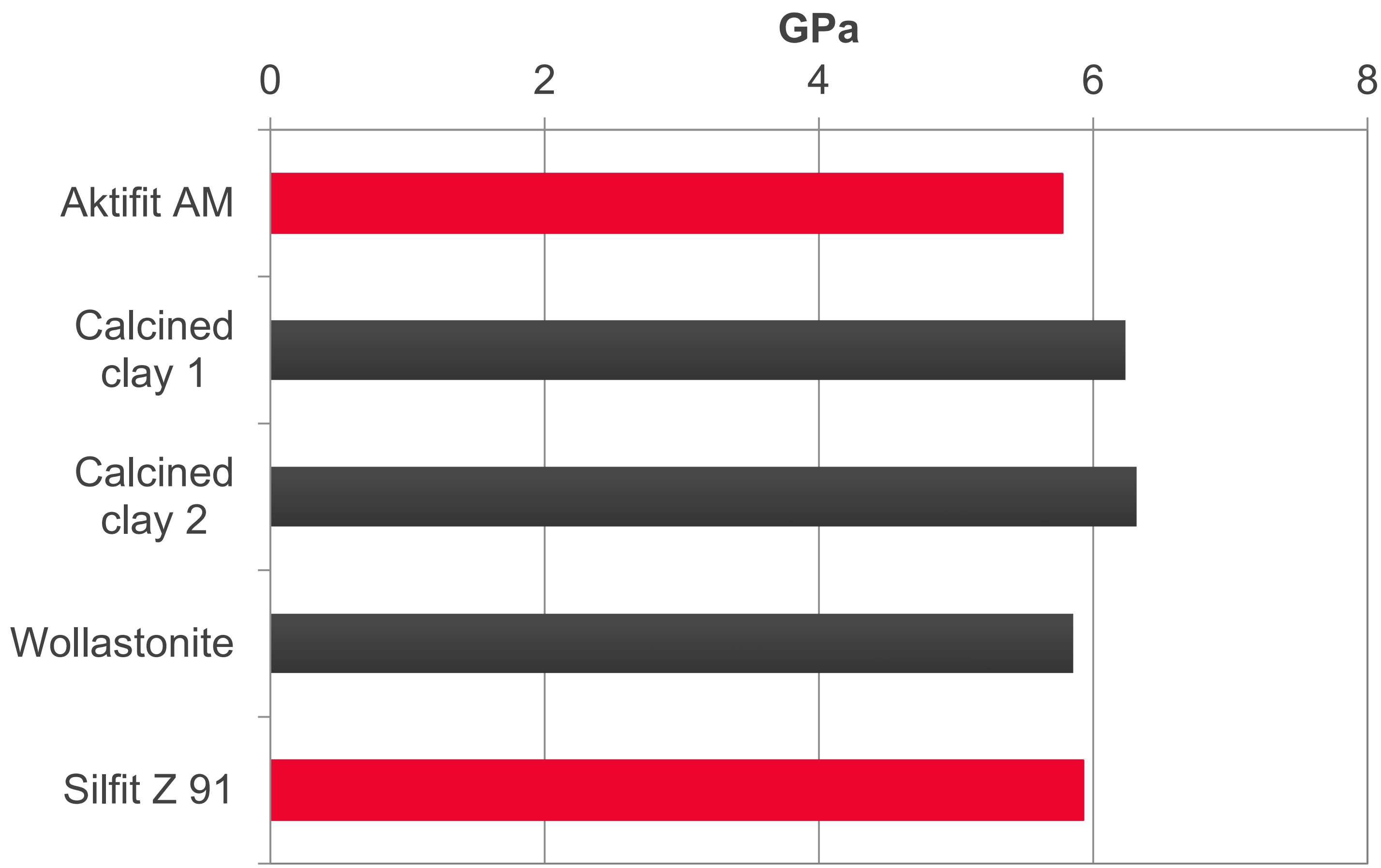
Ball Indentation Hardness

DIN EN ISO 2039-1; H961N/30s



Tensile Modulus

DIN EN ISO 527-1,-2; 0.5 mm/min



Tensile Strength

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DIN EN ISO 527-1,-2; 5 mm/min

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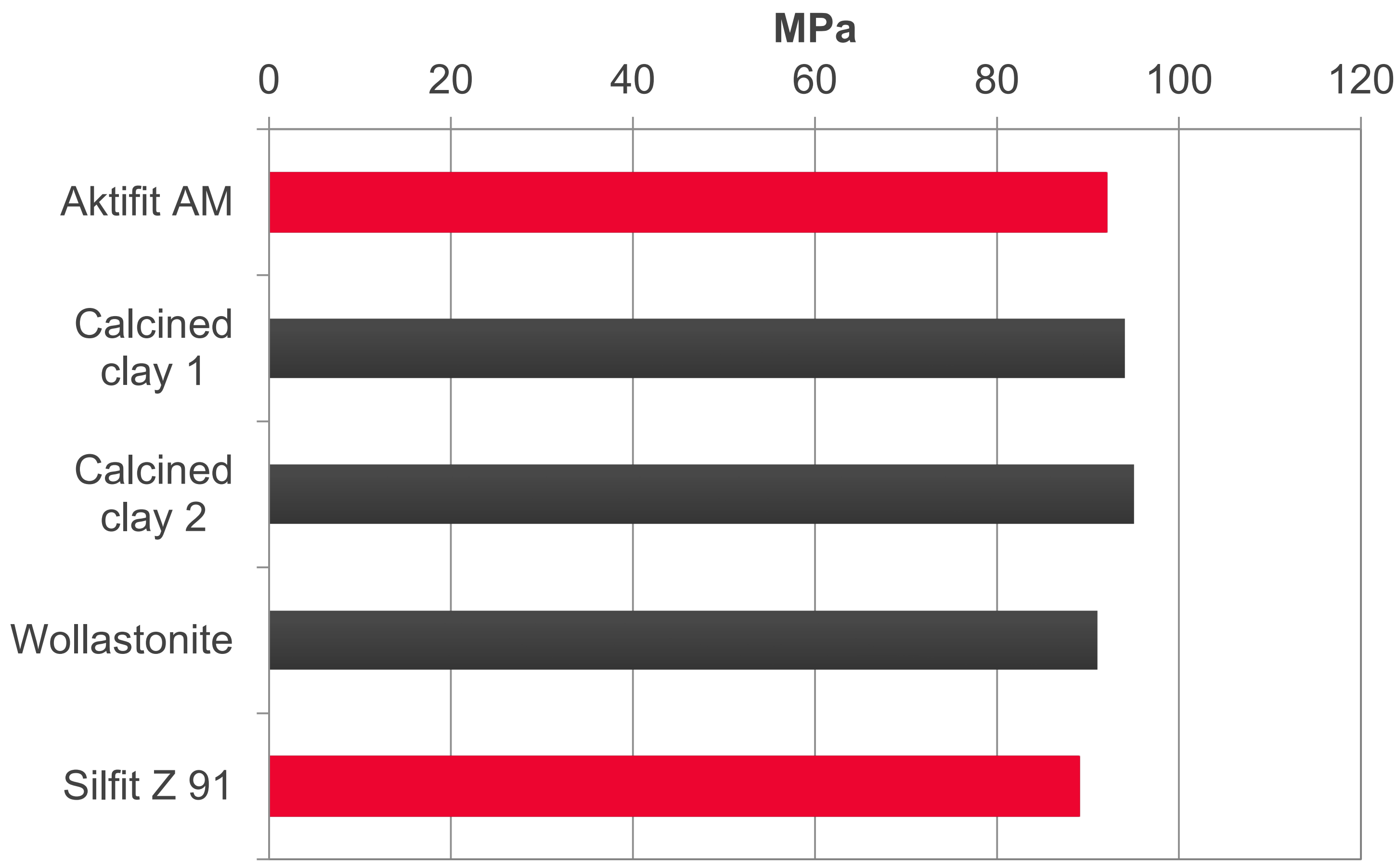

Tensile Strain at Break

DIN EN ISO 527-1,-2; 5 mm/min

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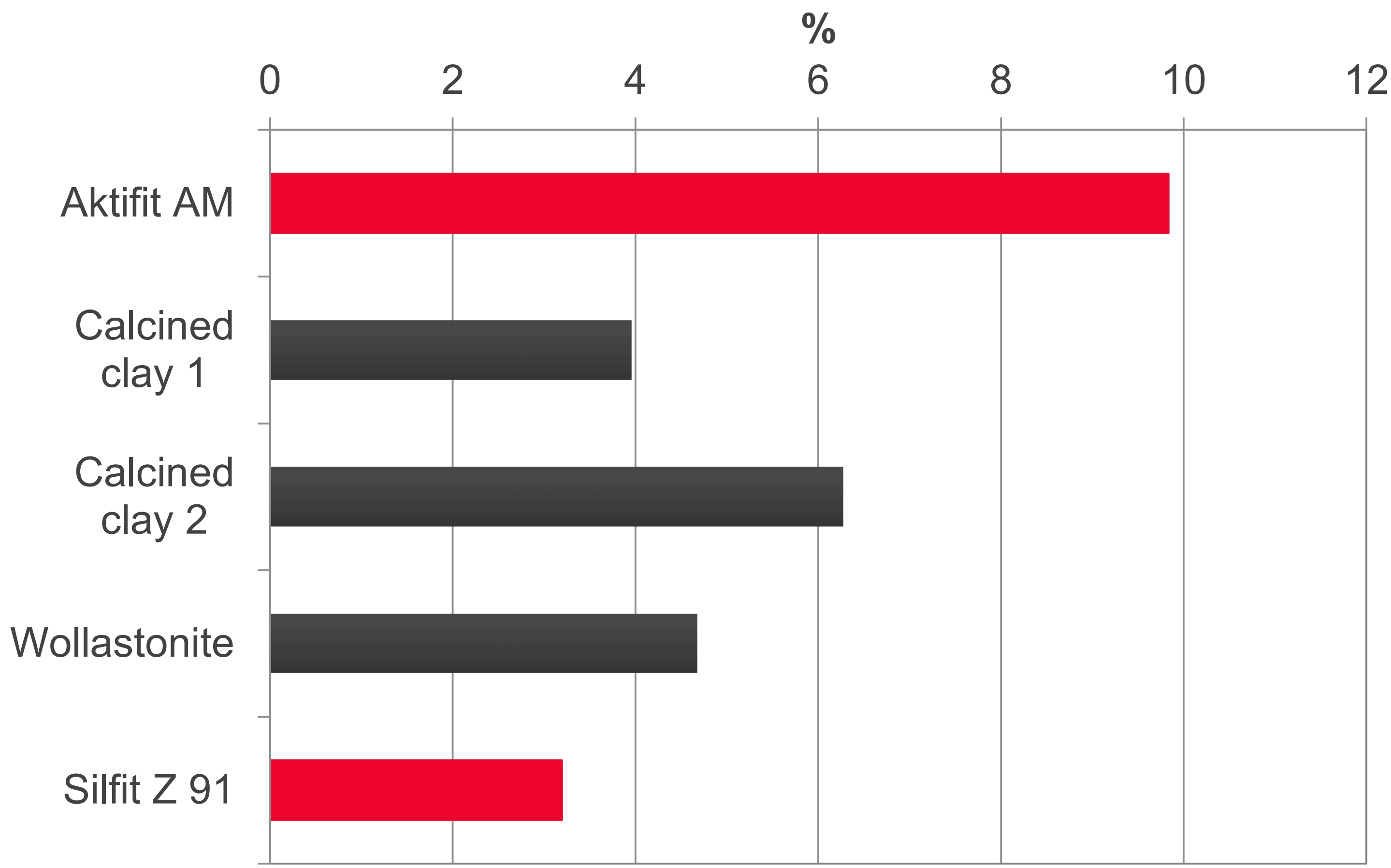
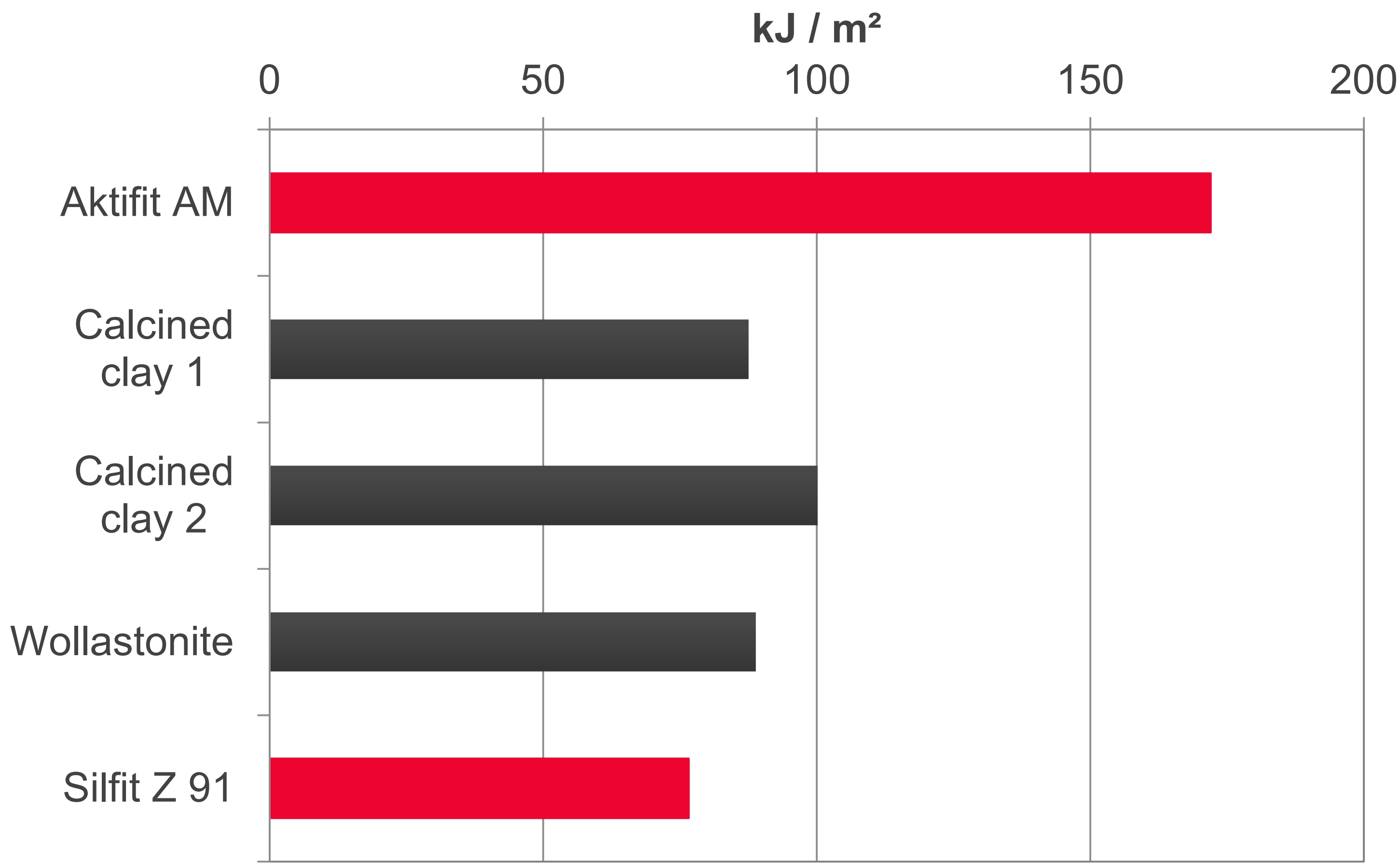


Impact Strength
Charpy 23 °C

DIN EN ISO 179-1 / 1eU



Impact Strength
Charpy -30 °C

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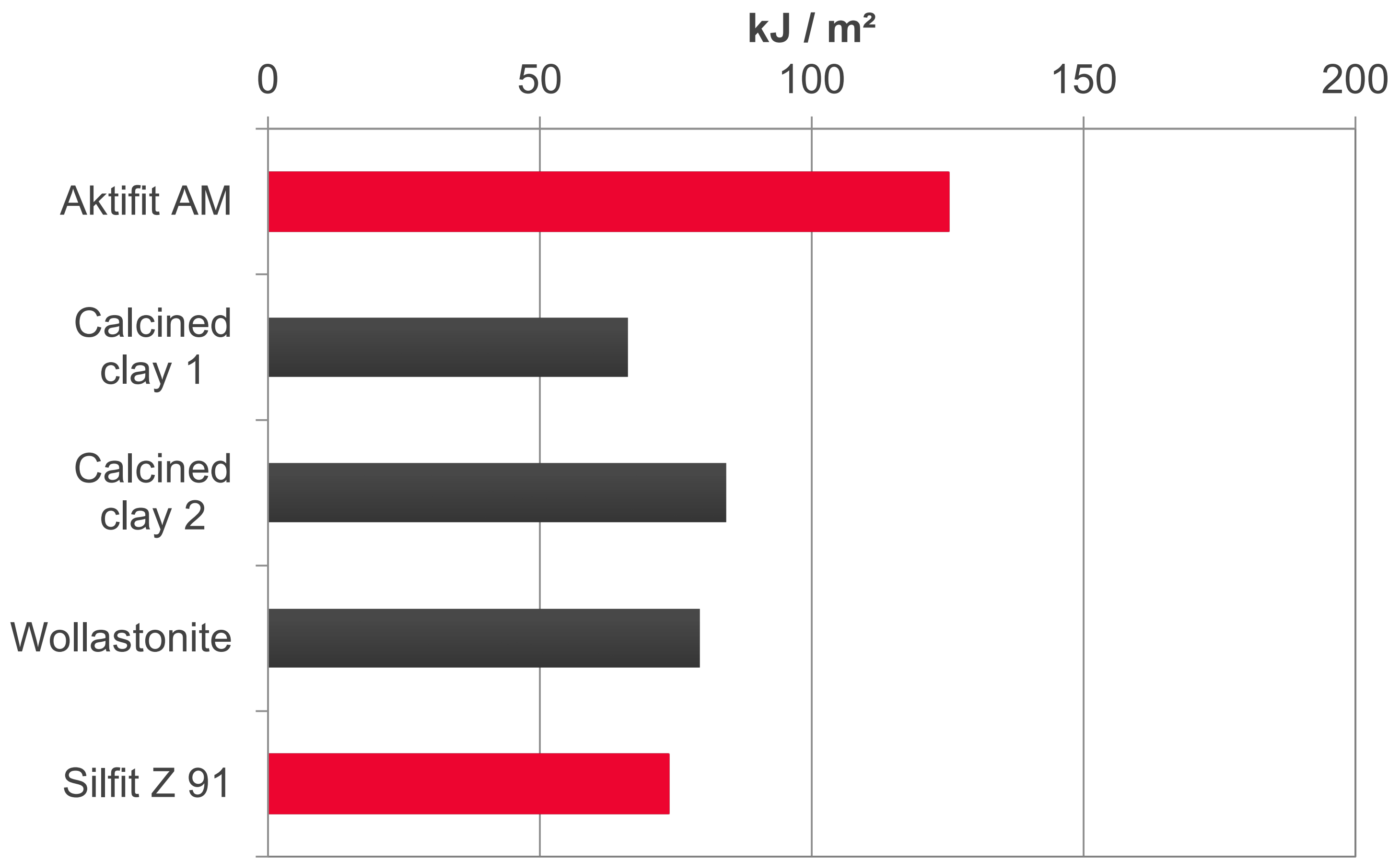
DIN EN ISO 179-1 / 1eU



Impact Strength
Izod 23 °C

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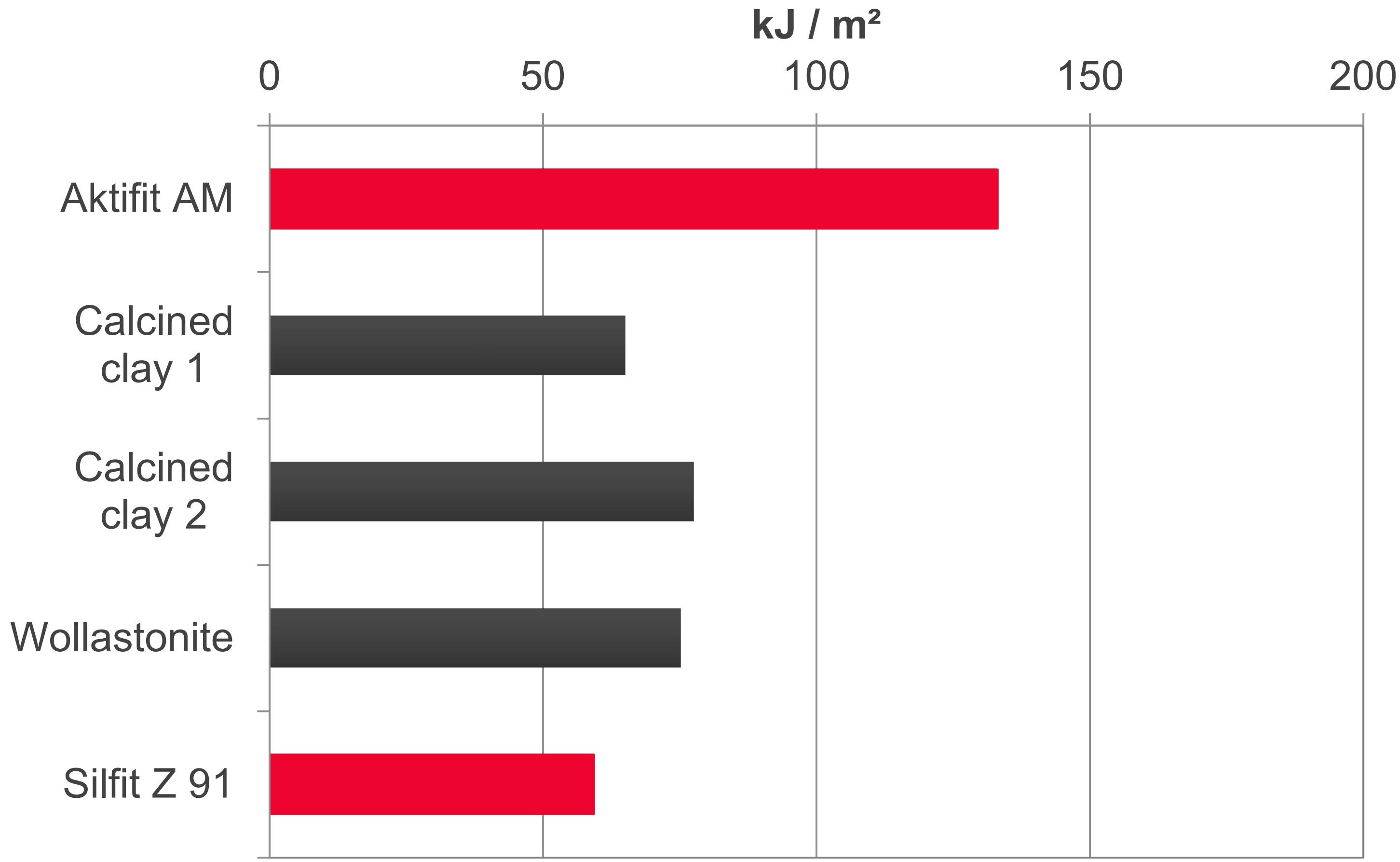
DIN EN ISO 180/U



Notched Impact Strength
Charpy 23 °C

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DIN EN ISO 179-1 / 1eA

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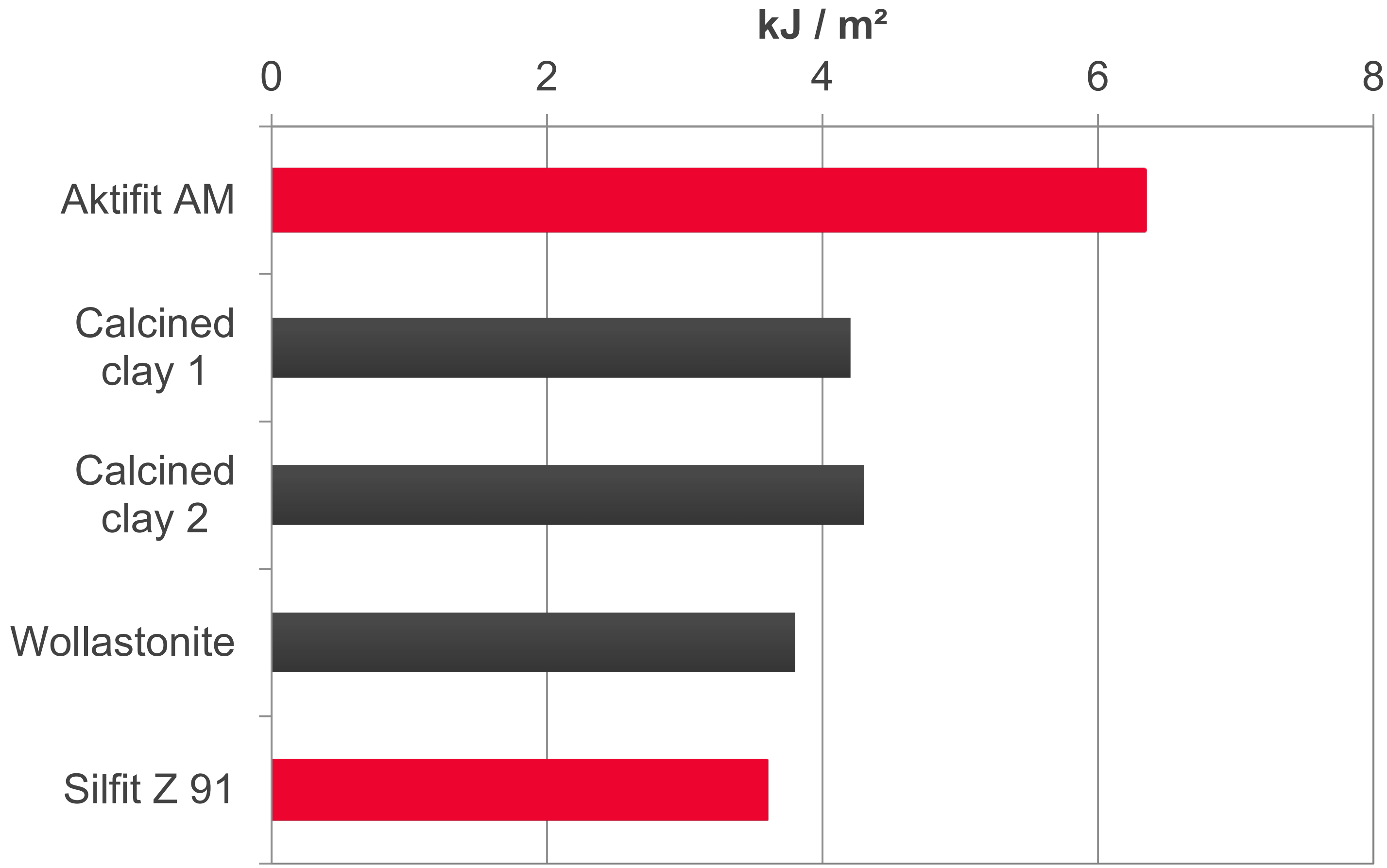

Black Coloring without Graying

Results obtained with polyamide 6 show that the tested clays led to a clearly noticeable lighter color or graying of black-colored compounds, respectively.

With Aktifit AM, this was **not** the case.

This has also been observed in earlier studies on black-colored polyamide 66 compounds

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Black Coloring in PA 6

The tests were carried out with a second grade of polyamide 66:

Schulamid® 66 MV 2

The ranking of the fillers was the same as with Ultramid® A3K.

Summary tables can be found in the appendix.

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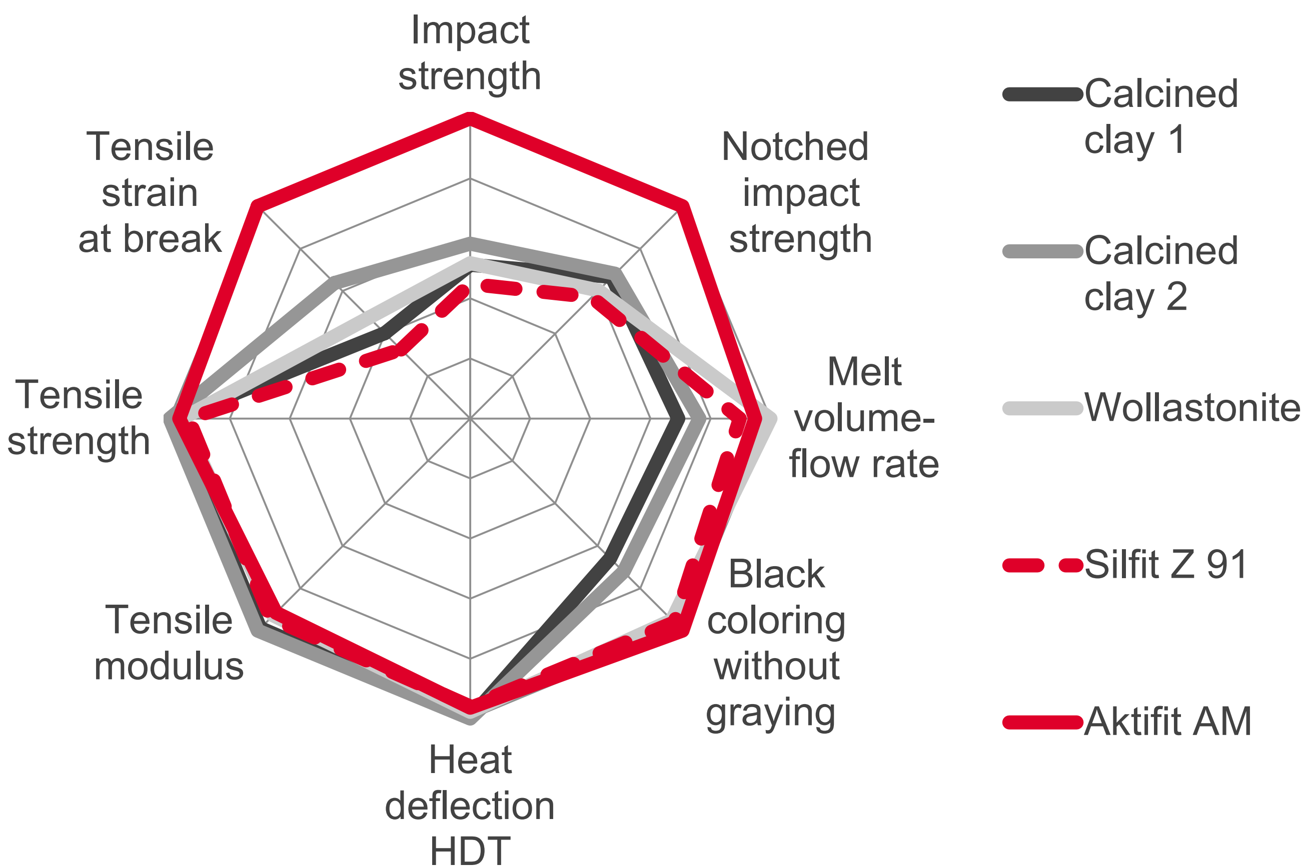

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Summary

Compared to other fillers suitable for low-warpage parts, Aktifit AM offers the following benefits:

- High melt flow rate
- Comparable heat deflection
- Similar tensile strength
- No graying of black-colored compounds
- Very high tensile strain at break
- Excellent impact strength, even at low-temperature
- Low-temperature impact strength very often higher than with competitive fillers at room temperature (PA 66)

We supply material for good ideas!

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Summary Table (1)
Durethan B 31 SK dry-as-molded

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		Calcined clay		Wollastonite	Calcined Neuburg Siliceous Earth	
		Grade 1	Grade 2	block-like particle structure	Silfit Z 91	Aktifit AM
Melt Volume-flow rate	cm³/10 min	77	73	69	91	98
Heat distortion temperature HDT/B	°C	189	188	187	182	184
Ball Indentation Hardness	MPa	218	217	204	230	221
Tensile Modulus	GPa	6.0	6.2	5.8	5.7	5.4
Tensile Strength	MPa	87	87	84	86	83
Tensile Strain at Break	%	7.2	7.5	5.6	3.6	10.4
Impact Strength Izod	kJ/m²	70 C	79 C	66 C	62 C	120 C
Impact Strength Charpy 23 °C -30 °C	kJ/m²	98 C 66 C	100 C 72 C	87 C 74 C	85 C 70 C	125 C 74 C
Notched Impact Strength Charpy 23 °C -30 °C	kJ/m²	3.8 C 3.3 C	3.9 C 3.2 C	3.7 C 3.1 C	3.4 C 2.6 C	4.9 C 3.6 C

Summary Table (2)
Durethan B 31 SK conditioned

		Calcined clay		Wollastonite	Calcined Neuburg Siliceous Earth	
		Grade 1	Grade 2	block-like particle structure	Silfit Z 91	Aktifit AM
Tensile Modulus	GPa	2.0	2.1	1.8	1.9	1.8
Tensile Strength	MPa	52	48	44	31	52
Tensile Strain at Break	%	18	10 [1)]	25	19	30
Impact Strength Charpy 23 °C	kJ/m²	314 C	17 C [1)]	236 C	105 C	N
Notched Impact Strength Charpy 23 °C	kJ/m²	6.0 C	3.2 C [1)]	4.1 C	3.2 C	6.7 C

[1)] After conditioning, the results of clay 2 were inexplicably low.

Summary Table (3)
Ultramid A3K dry-as-molded

		Calcined clay		Wollastonite	Calcined Neuburg Siliceous Earth	
		Grade 1	Grade 2	block-like particle structure	Silfit Z 91	Aktifit AM
Melt Volume-flow rate	cm³/10 min	54	60	78	70	74
Vicat Softening Point	°C	244	245	241	243	243
Heat distortion temperature HDT/B	°C	229	230	224	220	221
Ball Indentation Hardness	MPa	268	267	253	275	273
Tensile Modulus	GPa	6.2	6.3	5.9	5.9	5.8
Tensile Strength	MPa	94	95	91	89	92
Tensile Strain at Break	%	4.0	6.3	4.7	3.2	9.8
Impact Strength Izod	kJ/m²	65 C	78 C	75 C	59 C	133 C
Impact Strength Charpy 23 °C / -30 °C	kJ/m²	88 C 66 C	100 C 84 C	89 C 79 C	77 C 74 C	172 C 125 C
Notched Impact Strength Charpy 23 °C / -30 °C	kJ/m²	4.2 C 3.7 C	4.3 C 3.8 C	3.8 C 3.5 C	3.6 C 3.1 C	6.3 C 3.9 C

Summary Table (4)
Ultramid A3K conditioned

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		Calcined clay		Wollastonite	Calcined Neuburg Siliceous Earth	
		Grade 1	Grade 2	block-like particle structure	Silfit Z 91	Aktifit AM
Ball Indentation Hardness	MPa	125	119	117	119	114
Tensile Modulus	GPa	3.2	3.2	2.9	2.8	2.7
Tensile Strength	MPa	65	66	56	41	64
Tensile Strain at Break	%	13	18	15	11	25
Impact Strength Charpy 23 °C	kJ/m²	191 C	239 C	161 C	84 C	460 C
Notched Impact Strength Charpy 23 °C	kJ/m²	5.9 C	5.8 C	4.4 C	3.5 C	7.7 C

Summary Table (5)
Schulamid 66 MV 2 dry-as-molded

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		Calcined clay		Wollastonite	Calcined Neuburg Siliceous Earth	
		Grade 1	Grade 2	block-like particle structure	Silfit Z 91	Aktifit AM
Melt Volume-flow rate	cm³/10 min	47	45	57	54	59
Heat distortion temperature HDT/B	°C	221	223	215	218	214
Ball Indentation Hardness	MPa	232	218	209	237	234
Tensile Modulus	GPa	6.2	6.2	5.8	5.7	5.6
Tensile Strength	MPa	90	92	88	86	86
Tensile Strain at Break	%	4.7	7.4	4.5	3.1	8.5
Impact Strength Izod	kJ/m²	71 C	79 C	73 C	66 C	134 C
Impact Strength Charpy 23 °C -30 °C	kJ/m²	97 C 69 C	103 C 84 C	84 C 74 C	88 C 81 C	159 C 108 C
Notched Impact Strength Charpy 23 °C -30 °C	kJ/m²	4.1 C 3.7 C	4.1 C 3.8 C	3.9 C 3.7 C	3.7 C 3.4 C	6.0 C 4.2 C

Summary Table (6)
Schulamid 66 MV 2 conditioned

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			Calcined clay		Wollastonite	Calcined Neuburg Siliceous Earth	
			Grade 1	Grade 2	block-like particle structure	Silfit Z 91	Aktifit AM
Tensile Modulus		GPa	2.6	2.7	2.3	2.4	2.3
Tensile Strength		MPa	60	62	53	38	60
Tensile Strain at Break		%	18	21	16	17	28
Impact Strength Charpy	23 °C	kJ/m²	242 C	288 C	186 C	108 C	N (455 C)
Notched Impact Strength Charpy	23 °C	kJ/m²	6.0 C	6.2 C	5.4 C	3.6 C	9.1 C

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