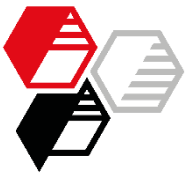




Calcined Neuburg Siliceous Earth in capacitor gaskets

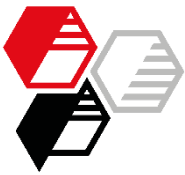
Author: Nicole Holzmayr



Contents

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- Introduction
- Experimental
- Results
- Summary
- Appendix



Status Quo

INTRODUCTION

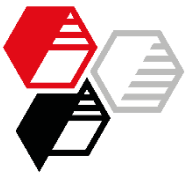
EXPERIMENTAL

RESULTS

SUMMARY

Since 2010, calcined versions of **Neuburg Siliceous Earth** are available – without a surface treatment as well as functional grades.

The calcination of **Neuburg Siliceous Earth** results in improved properties of rubber compounds when compared to the standard **Neuburg Siliceous Earth** grades.



Objective

INTRODUCTION

EXPERIMENTAL

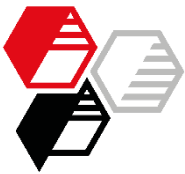
RESULTS

SUMMARY

Comparison of calcined clays Polyfil 80 and Burgess KE and established **Aktisil grades EM and VM 56** in a resin-cured butyl rubber formulation to the **Calcined Neuburg Siliceous Earth** grades **Silfit Z 91** and **Aktifit VM**.

Key properties:

- Processing
- Hardness
- Moduli
- Compression set
- Heat aging



Fillers, Characteristics

INTRODUCTION

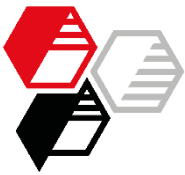
EXPERIMENTAL

RESULTS

SUMMARY

(*) in isopropyl alcohol	Particle Size		Oil absorption [g/100g]	Functiona- lization	Calcination
	d ₅₀ [μm]	d ₉₇ [μm]			
Silfit Z 91	1.8	8.0	56	x	✓
Polyfil 70	2.4	16	59	x	✓
Aktisil VM 56	1.5	5.9	47	Vinyl	x
Aktifit VM	1.8(*)	7.5(*)	51	Vinyl	✓
Burgess KE (*)	4.1(*)	22(*)	70	vinyl	✓

Polyfil 80 was not available in Germany, so we used Polyfil 70 which is identical to grade 80 except for color.



Preparation and Curing of the Compound

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INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

- **Mixing**

Open mill Ø 150 x 300 mm

Batch volume: approx. 1000 g

Temperature: 60 °C mixing

100 °C to avoid bubbles before removing the sheet
off the mill

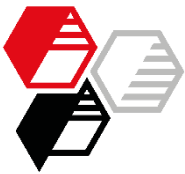
Mixing time: approx. 30 min.

- **Curing**

Press, 6 min. / 200 °C

- **Post-cure**

2 h / 190 °C



High CB Loading Using N 990

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INTRODUCTION

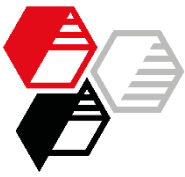
EXPERIMENTAL

High CB, N 990

RESULTS

SUMMARY

Formulation in phr	non-surface treated		surface treated		
	Polyfil 70	Silfit Z 91	Burgess KE	Aktifit VM	Aktisil VM 56
Butyl 268	100	100	100	100	100
N 990	100	100	100	100	100
Polyfil 70	100	-	-	-	-
Silfit Z 91	-	100	-	-	-
Burgess KE	-	-	100	-	-
Aktifit VM	-	-	-	100	-
Aktisil VM 56	-	-	-	-	100
Zinkoxyd aktiv	5	5	5	5	5
Stearic acid	2	2	2	2	2
Dispergator FL	2	2	2	2	2
Tackirol 201	18	18	18	18	18



Medium CB Loading Using N 774 + Additive

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INTRODUCTION

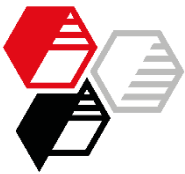
EXPERIMENTAL

Medium CB, N 774,
with additive

RESULTS

SUMMARY

Formulation in phr	non-surface treated		surface treated			
	Polyfil 70	Silfit Z 91	Burgess KE	Aktifit VM	Aktifit VM	Aktisil VM 56
Butyl 268	100	100	100	100	100	100
N 774	50	50	50	50	50	50
Polyfil 70	150	-	-	-	-	-
Silfit Z 91	-	150	-	-	-	-
Burgess KE	-	-	150	-	-	-
Aktifit VM	-	-	-	150	175	-
Aktisil VM 56	-	-	-	-	-	150
<i>Phenodur EP-560</i>	2	2	2	2	2	2
Zinkoxyd aktiv	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
Dispergator FL	2	2	2	2	2	2
Tackirol 201	18	18	18	18	18	18



Medium CB Loading Using N 774, no Additive

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INTRODUCTION

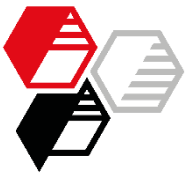
EXPERIMENTAL

Medium CB, N 774,
no additive

RESULTS

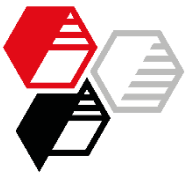
SUMMARY

Formulation in phr	non-surface treated		surface treated			
	Polyfil 70	Silfit Z 91	Burgess KE	Aktifit VM	Aktifit VM	Aktisil VM 56
Butyl 268	100	100	100	100	100	100
N 774	50	50	50	50	50	50
Polyfil 70	150	-	-	-	-	-
Silfit Z 91	-	150	-	-	-	-
Burgess KE	-	-	150	-	-	-
Aktifit VM	-	-	-	150	175	-
Aktisil VM 56	-	-	-	-	-	150
<i>Phenodur EP-560</i>	-	-	-	-	-	-
Zinkoxyd aktiv	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
Dispergator FL	2	2	2	2	2	2
Tackirol 201	18	18	18	18	18	18



Overview

<u>Part 1:</u>	High carbon black loading using N 990
<u>Part 2:</u>	Medium carbon black loading using N 774 and additive
<u>Part 3:</u>	Medium carbon black loading using N 774 without additive
<u>Summary</u>	
<u>Appendix:</u>	Results in detail



High CB Loading Using N 990

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INTRODUCTION

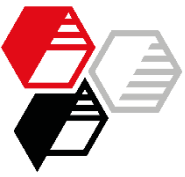
EXPERIMENTAL

High CB, N 990

RESULTS

SUMMARY

Formulation in phr	non-surface treated		surface treated		
	Polyfil 70	Silfit Z 91	Burgess KE	Aktifit VM	Aktisil VM 56
Butyl 268	100	100	100	100	100
N 990	100	100	100	100	100
Polyfil 70	100	-	-	-	-
Silfit Z 91	-	100	-	-	-
Burgess KE	-	-	100	-	-
Aktifit VM	-	-	-	100	-
Aktisil VM 56	-	-	-	-	100
Zinkoxyd aktiv	5	5	5	5	5
Stearic acid	2	2	2	2	2
Dispergator FL	2	2	2	2	2
Tackirol 201	18	18	18	18	18



Processing in General

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INTRODUCTION

EXPERIMENTAL

RESULTS

High CB, N 990

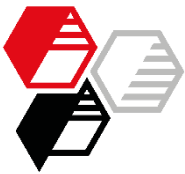
SUMMARY

Problems regarding processing on the lab mill occurred with both **Polyfil 70** and **Burgess KE**:

✗ distinct stickiness on the mill

By using any **Neuburg Siliceous Earth** grade, stickiness was avoided:

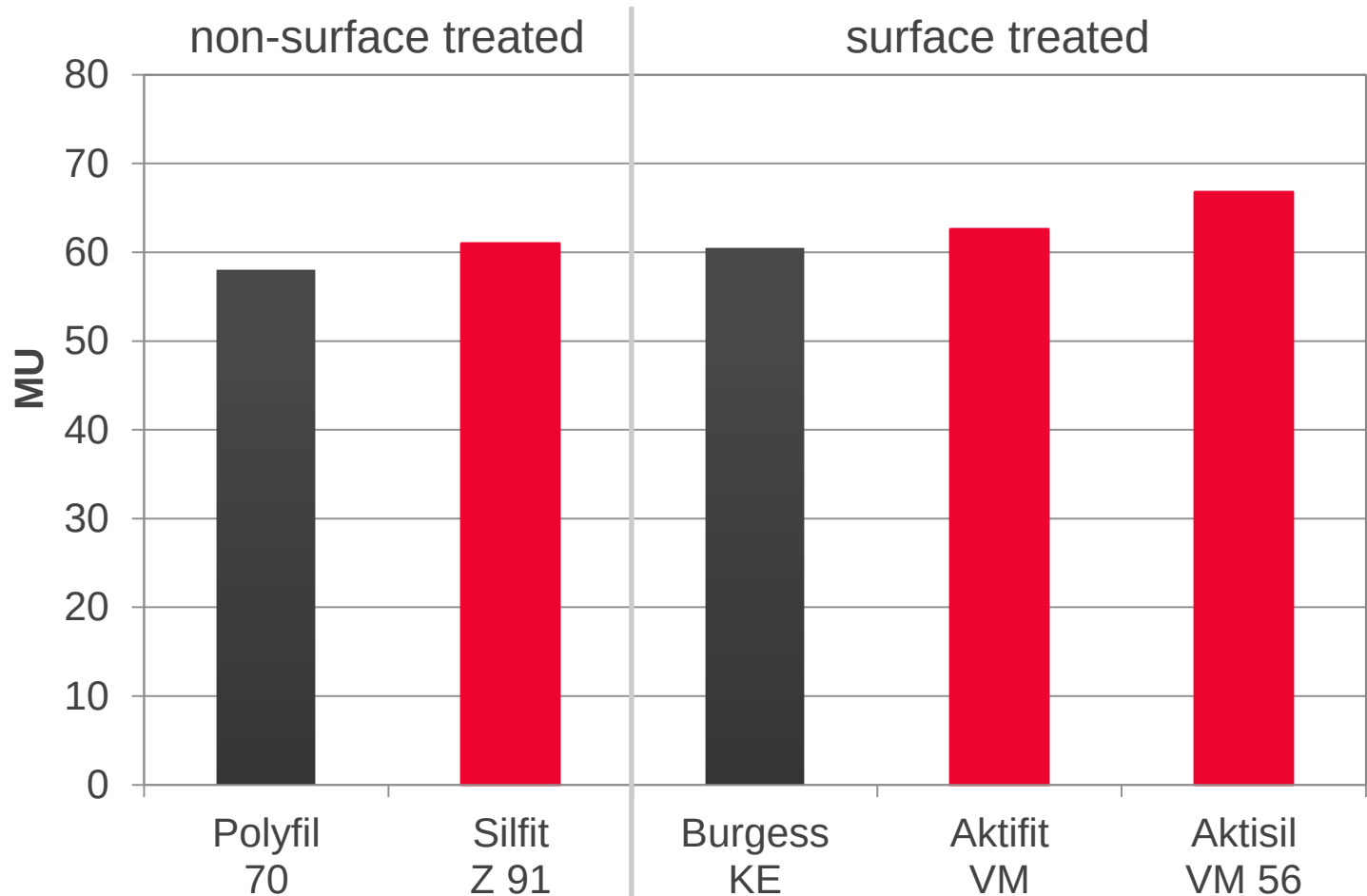
✓ no stickiness with **Neuburg Siliceous Earth**

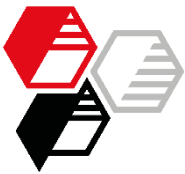


Mooney Viscosity

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DIN 53 523 Part 3, ML 1+4, 120 °C

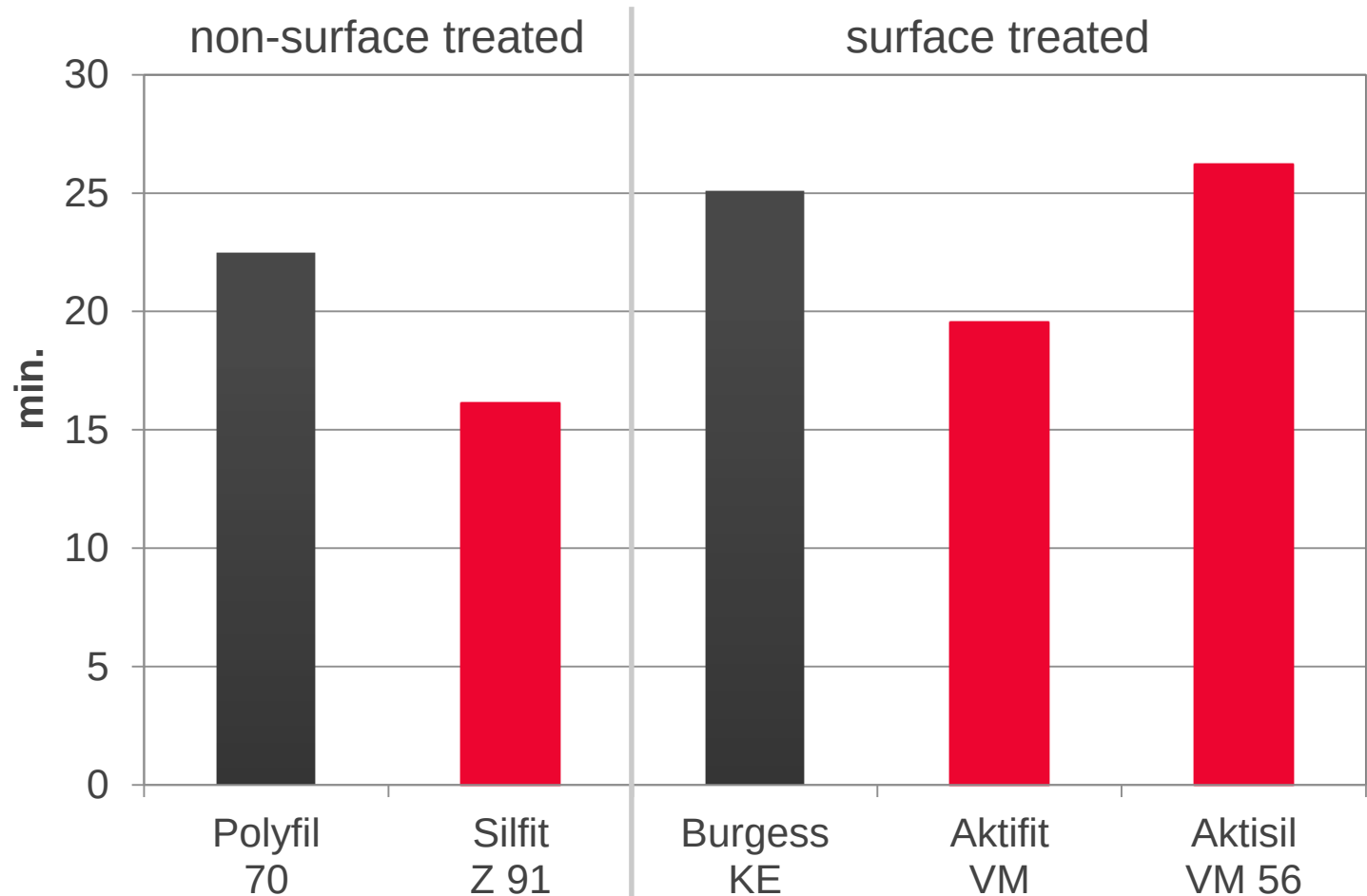


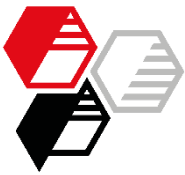


Mooney Scorch Time

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DIN 53 523 Part 4, ML +5, 120 °C

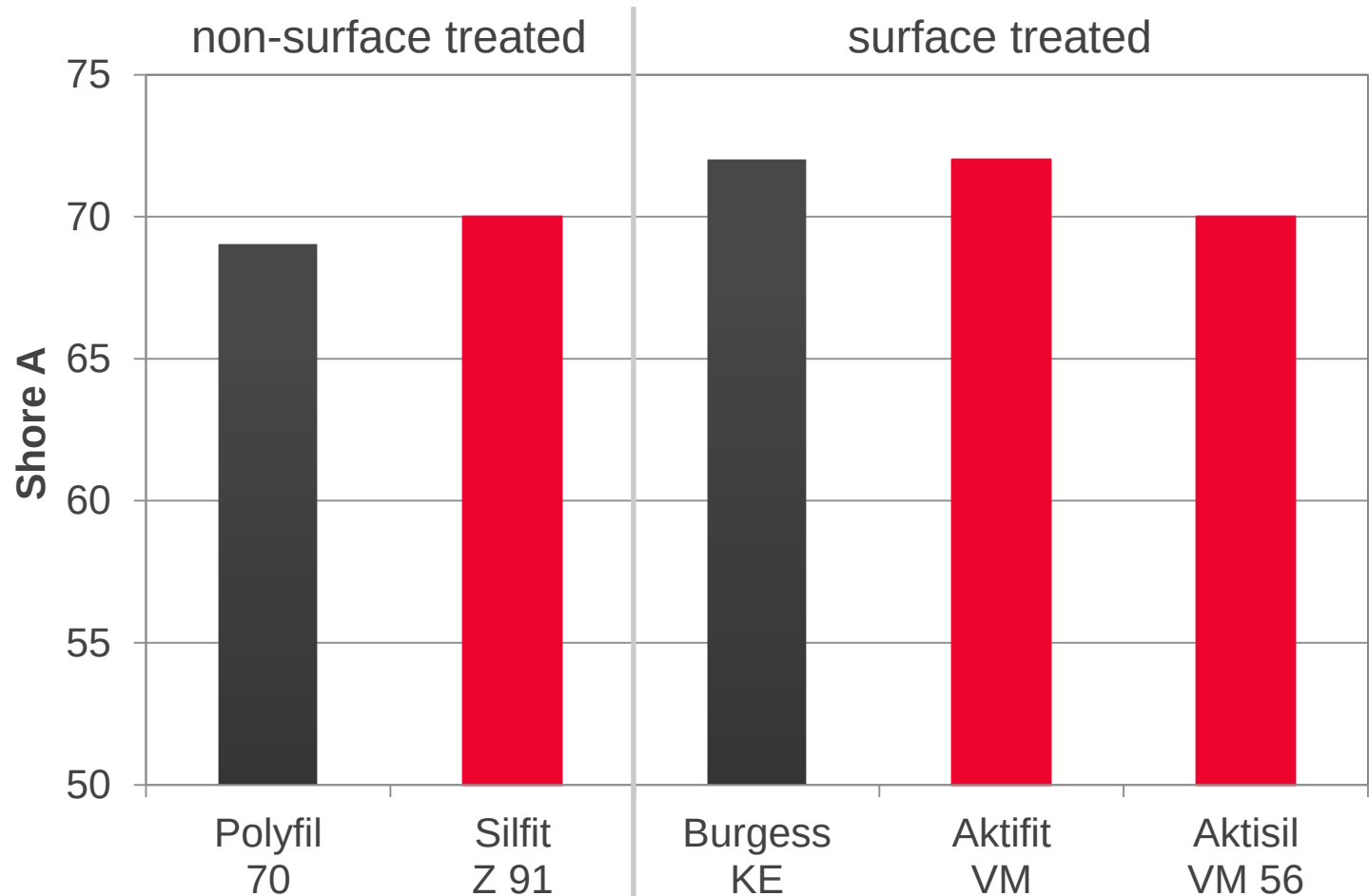


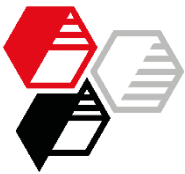


Hardness

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DIN 53 505-A, piled-up S2 dumbbells; post-cured

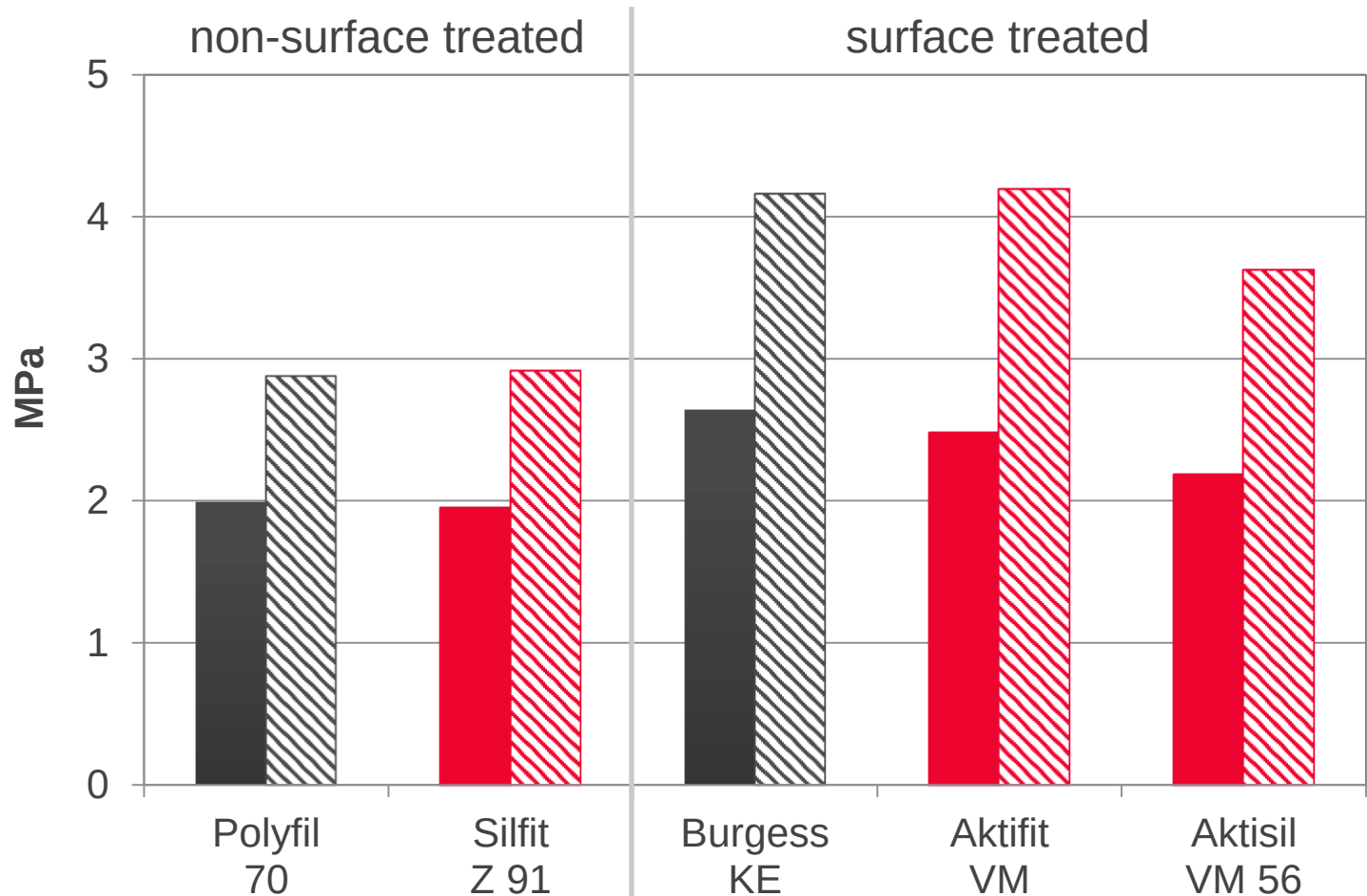


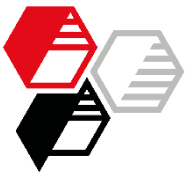


Moduli

DIN 53 504, S2; post-cured

■ Mod. 50 % ▨ Mod. 100 %



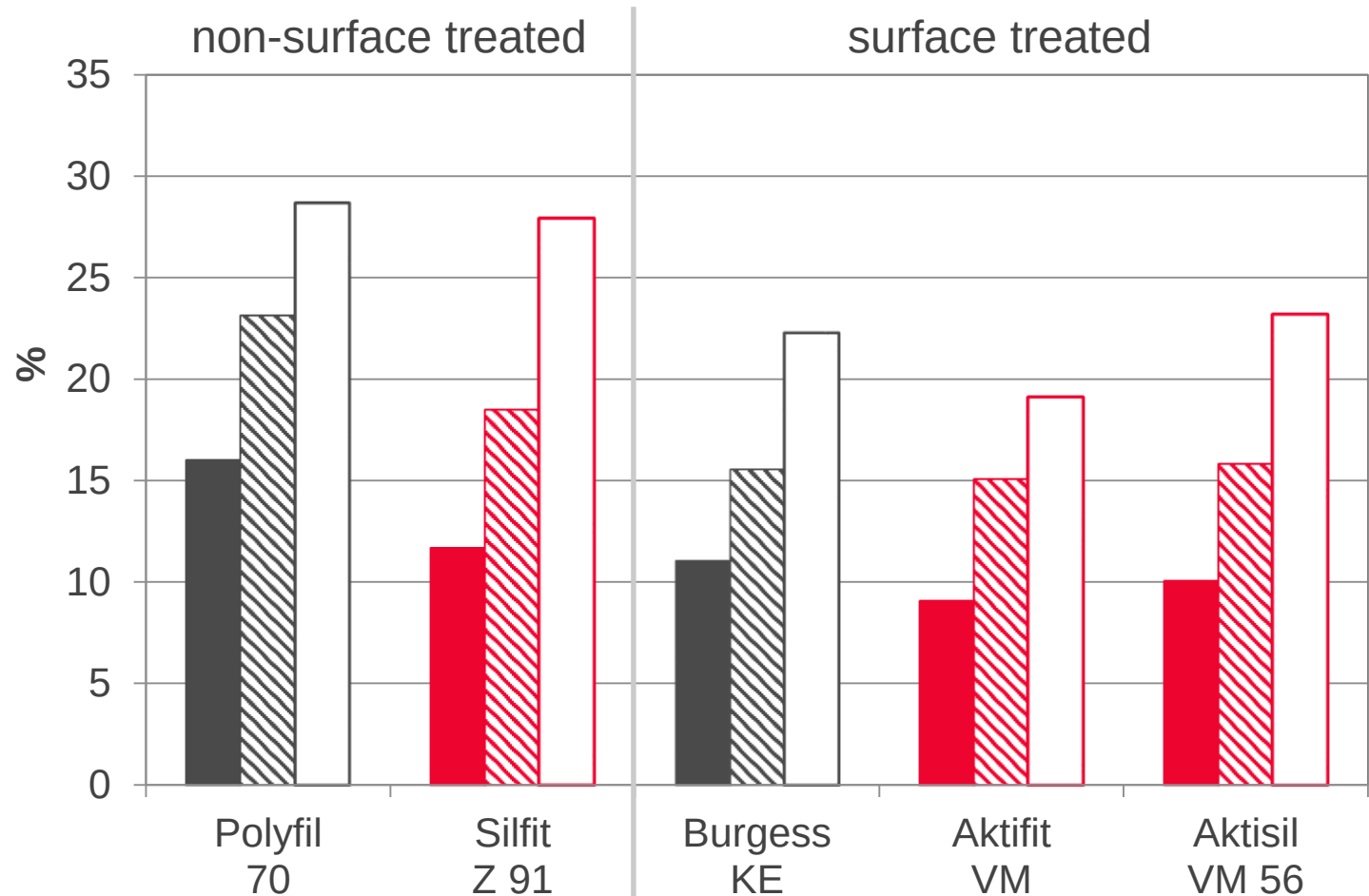


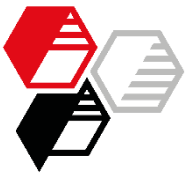
Compression Set, 72 h

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DIN ISO 815-1 B, cooling method A, 25 % defl.; post-cured

■ 105 °C ■ 125 °C □ 135 °C





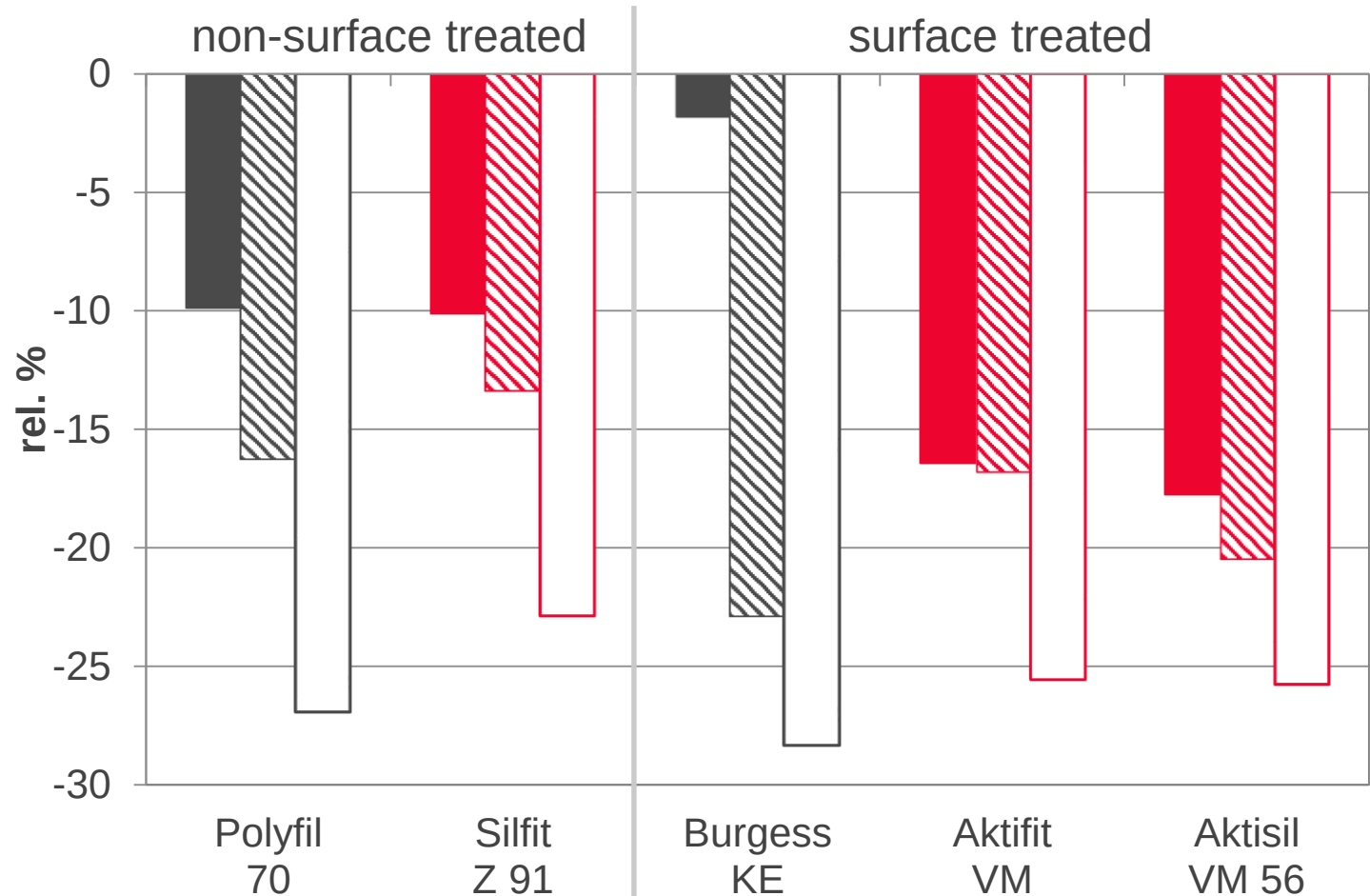
Hot Air Aging, 72 h

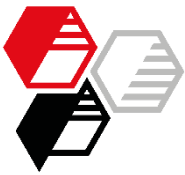
Change of Elongation at Break

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DIN 53 508; post-cured

■ 105 °C ■ 125 °C □ 135 °C





Summary Part 1

High CB using N 990

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Non-surface treated fillers

Silfit Z 91 in comparison to Polyfil 70

Processing	+
Hardness	=
Moduli	=
Compression set	+
Aging	=/+

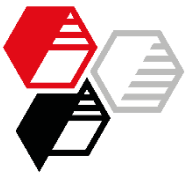
INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

High CB, N 990



Summary Part 1

High CB using N 990

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Surface treated fillers

Neuburg Siliceous Earth grades in comparison to Burgess KE

	Aktifit VM	Aktisil VM 56
Processing	+	+
Hardness	=	-
Moduli	=	-
Compression set	=/+	=
Aging	=	=



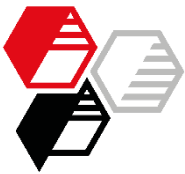
INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

High CB, N 990



Medium CB Loading Using N 774 + Additive

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INTRODUCTION

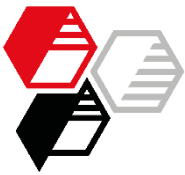
EXPERIMENTAL

Medium CB, N 774,
with additive

RESULTS

SUMMARY

Formulation in phr	non-surface treated		surface treated			
	Polyfil 70	Silfit Z 91	Burgess KE	Aktifit VM	Aktifit VM	Aktisil VM 56
Butyl 268	100	100	100	100	100	100
N 774	50	50	50	50	50	50
Polyfil 70	150	-	-	-	-	-
Silfit Z 91	-	150	-	-	-	-
Burgess KE	-	-	150	-	-	-
Aktifit VM	-	-	-	150	175	-
Aktisil VM 56	-	-	-	-	-	150
<i>Phenodur EP-560</i>	2	2	2	2	2	2
Zinkoxyd aktiv	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
Dispergator FL	2	2	2	2	2	2
Tackirol 201	18	18	18	18	18	18



Processing in General

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INTRODUCTION

EXPERIMENTAL

Medium CB, N 774,
with additive

RESULTS

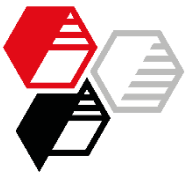
SUMMARY

Problems regarding processing on the lab mill occurred with both **Polyfil 70** and **Burgess KE**:

- ✗ distinct stickiness on the mill

By using **Neuburg Siliceous Earth**, stickiness was reduced:

- ✓ less stickiness with **Neuburg Siliceous Earth**



Mooney Viscosity

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DIN 53 523 Part 3, ML 1+4, 120 °C

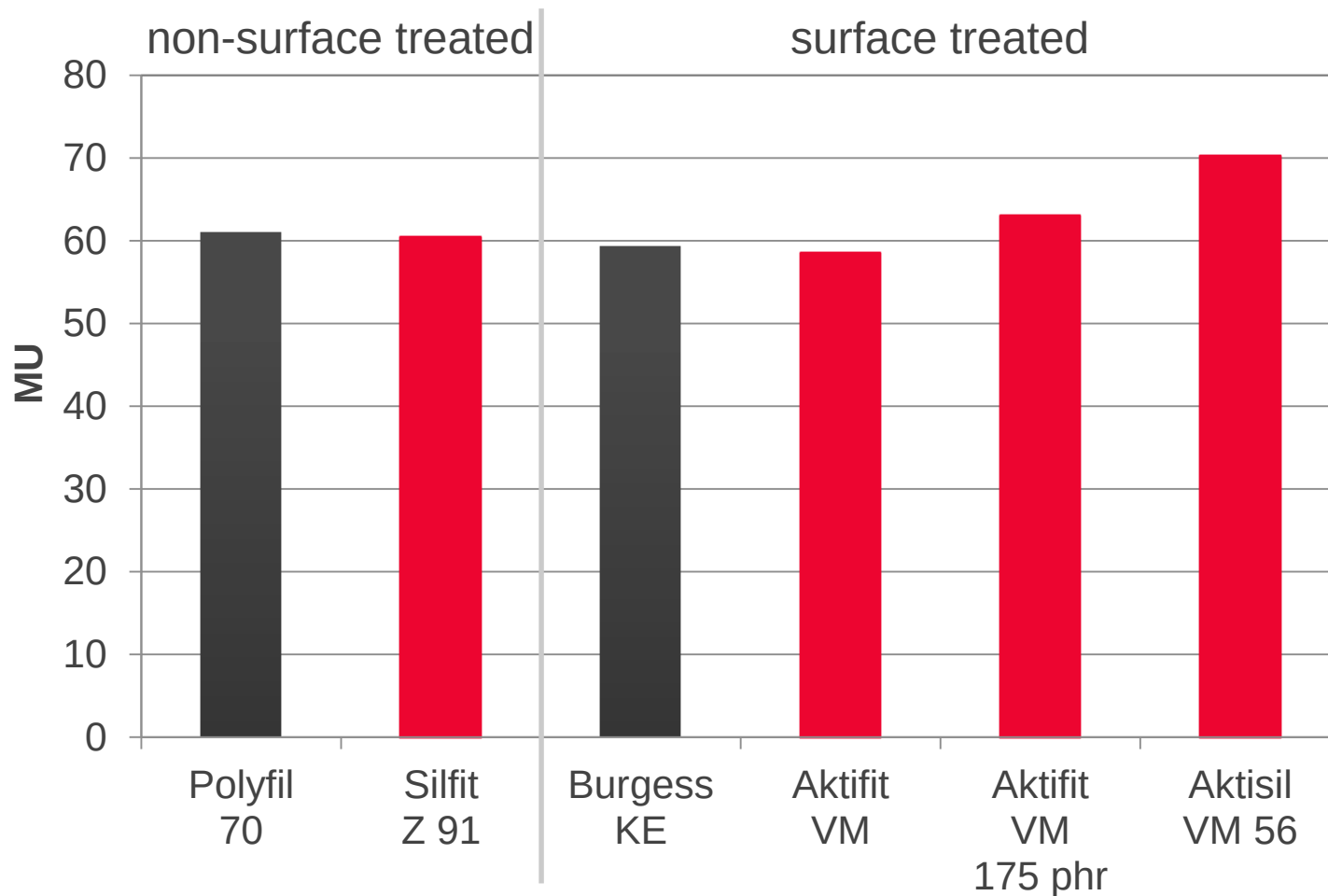
INTRODUCTION

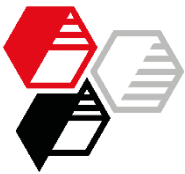
EXPERIMENTAL

RESULTS

Medium CB, N 774,
with additive

SUMMARY

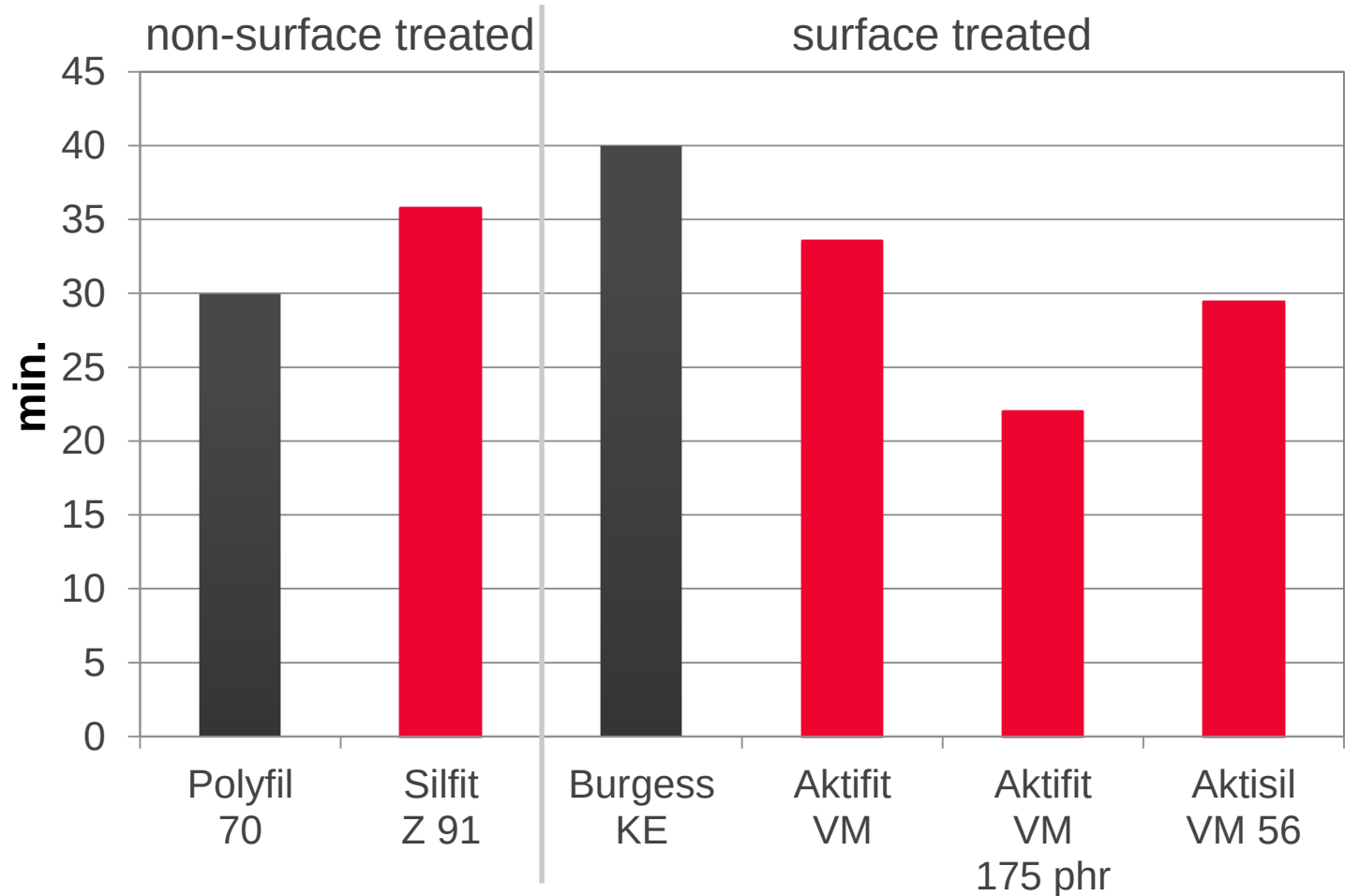


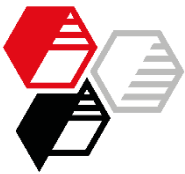


Mooney Scorch Time

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DIN 53 523 Part 4, ML +5, 120 °C

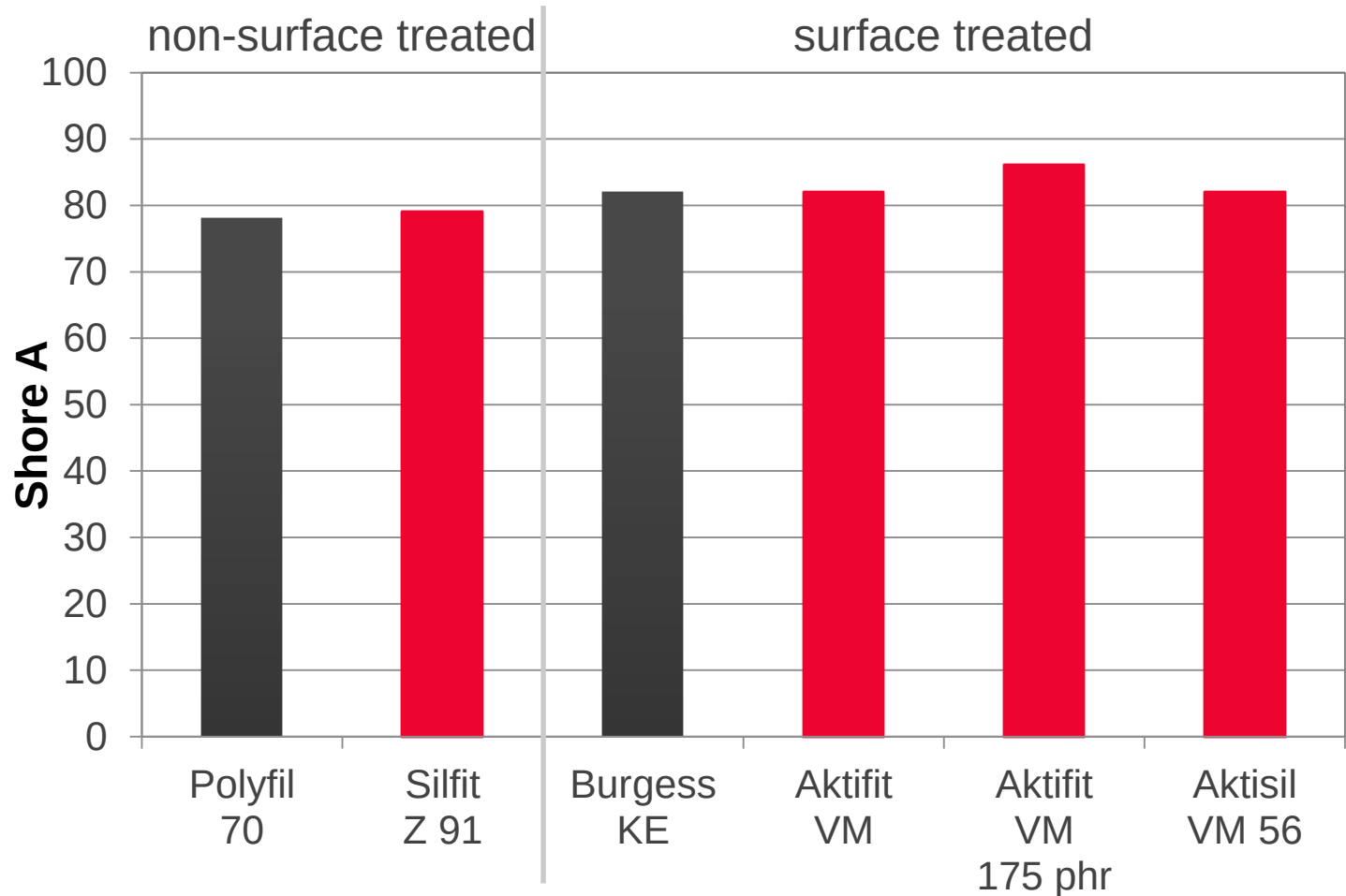




Hardness

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DIN 53 505-A, piled-up S2 dumbbells; post-cured



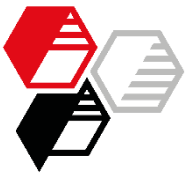
INTRODUCTION

EXPERIMENTAL

RESULTS

Medium CB, N 774,
with additive

SUMMARY

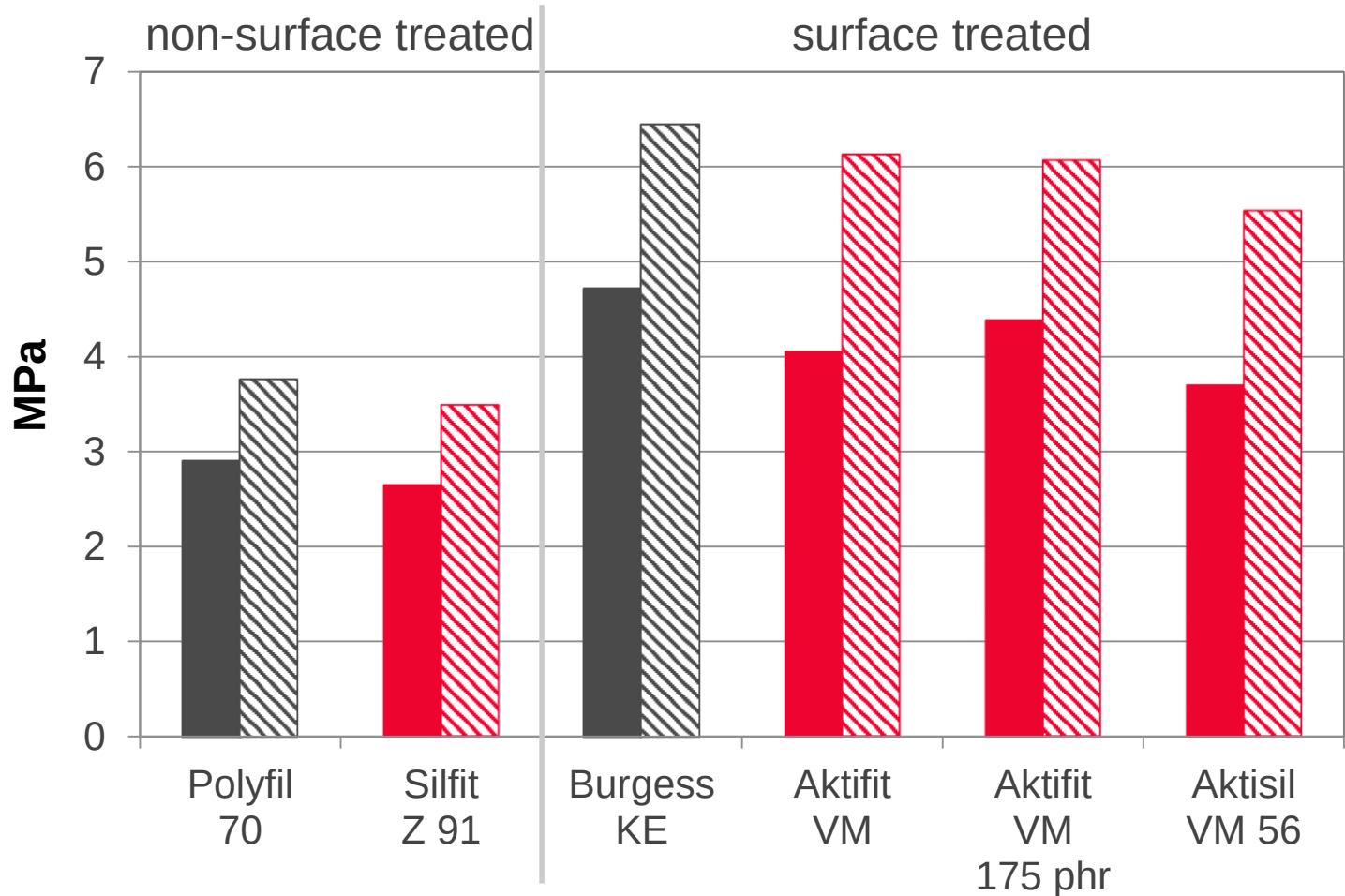


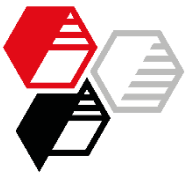
Moduli

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DIN 53 504, S2; post-cured

■ Mod. 50 % ▨ Mod. 100 %

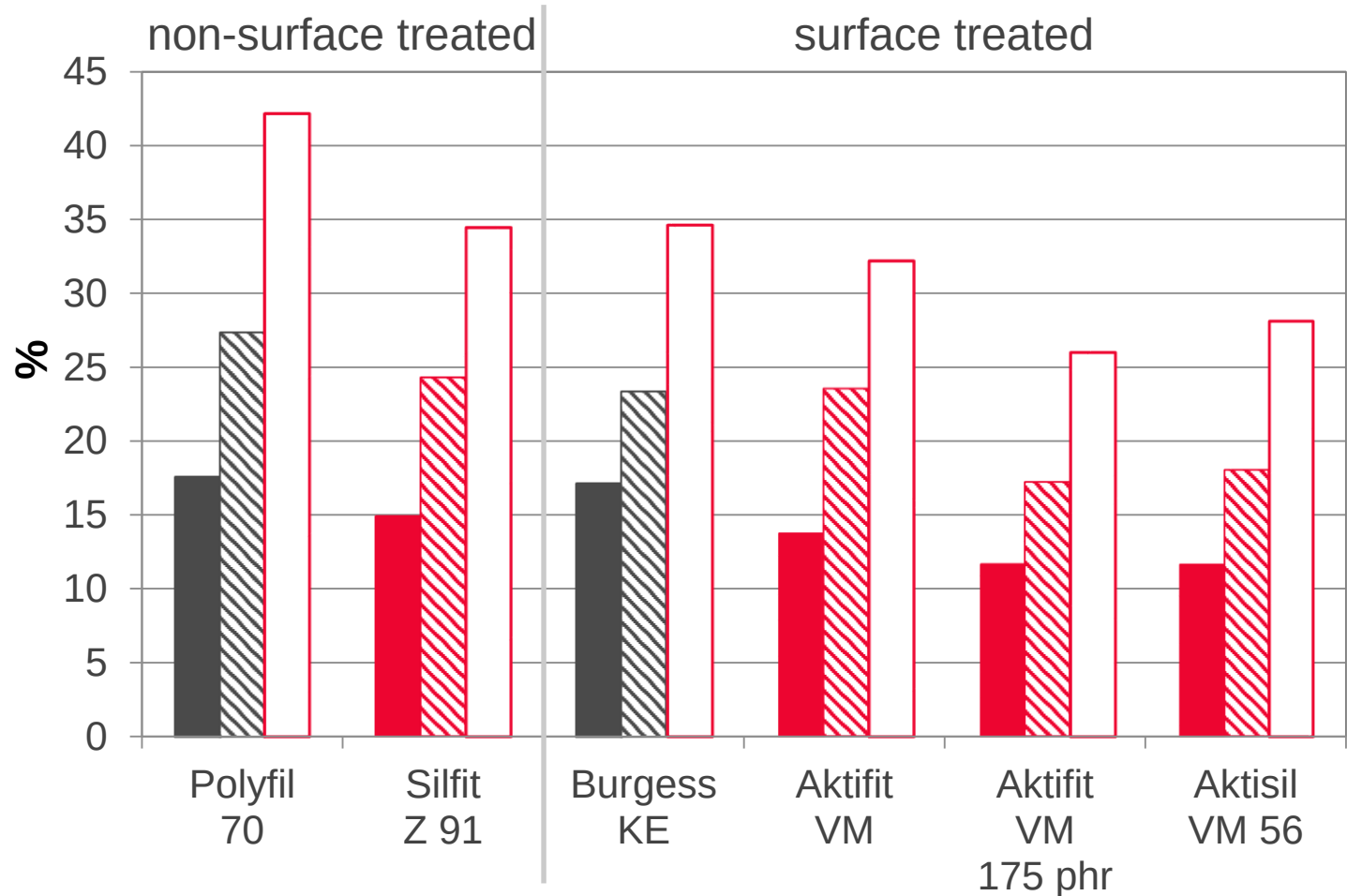


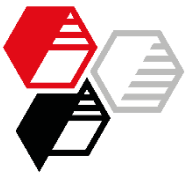


Compression Set, 72 h

DIN ISO 815-1 B, cooling method A, 25 % defl.; post-cured

■ 105 °C ■ 125 °C □ 135 °C





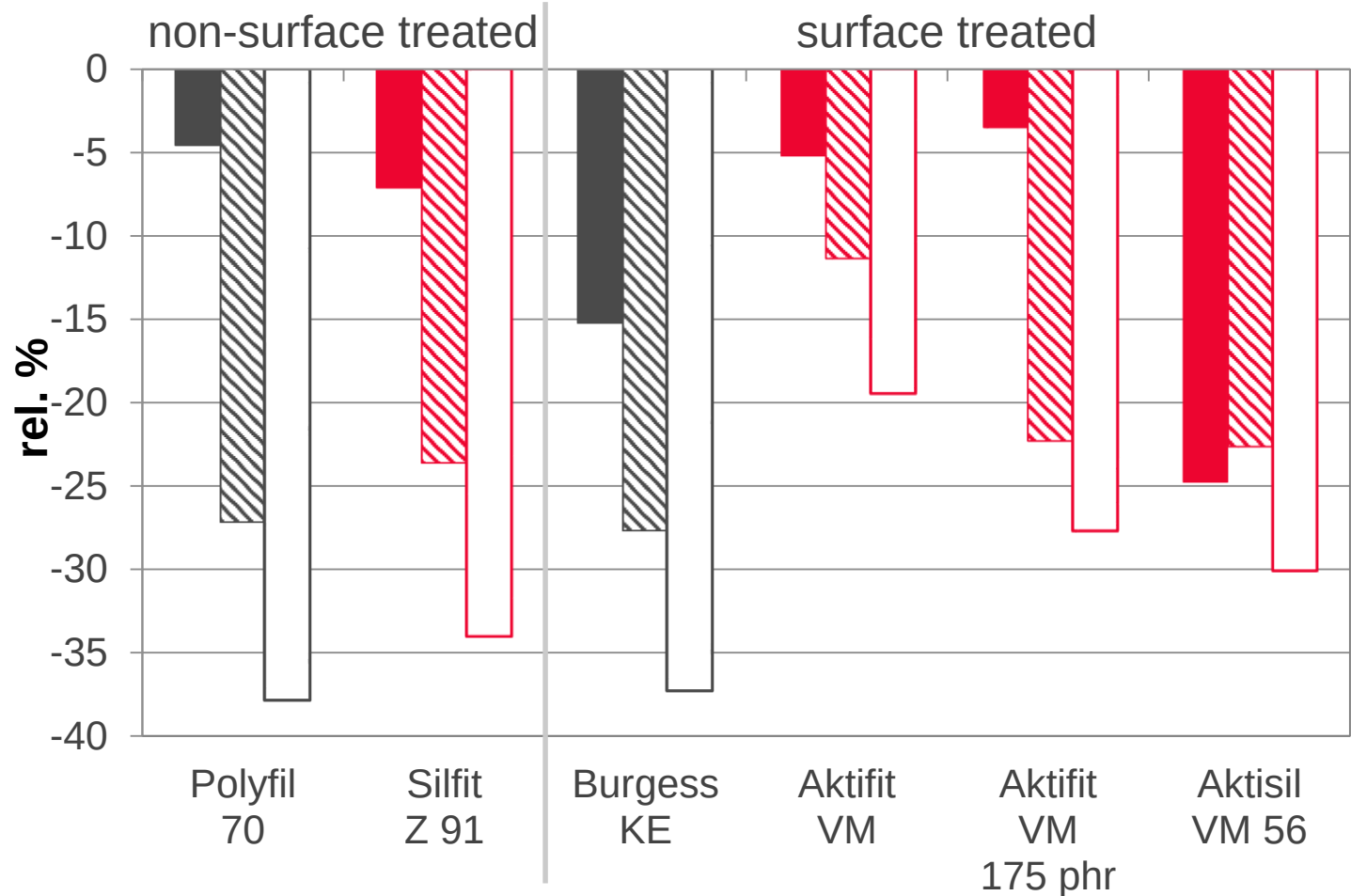
Hot Air Aging, 72 h

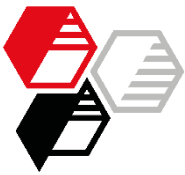
Change of Elongation at B.

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DIN 53 508; post-cured

■ 105 °C ■ 125 °C □ 135 °C





Summary Part 2 – Medium CB using N 774 + Additive

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Non-surface treated fillers

Silfit Z 91 in comparison to Polyfil 70

Processing	+
Hardness	=
Moduli	=/-
Compression set	+
Aging	=/+

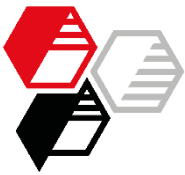
INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

Medium CB, N 774,
with additive



Summary Part 2 – Medium CB using N 774 + Additive

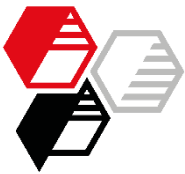
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Surface treated fillers

Neuburg Siliceous Earth grades in comparison to Burgess KE

	Aktifit VM 150 phr	Aktifit VM 175 phr	Aktisil VM 56 150 phr
Processing	+	+	+
Hardness	=	+	=
Moduli	=/-	=	-
Compression set	+	+	+
Aging	+	=/+	=/+





Medium CB Loading Using N 774, no Additive

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INTRODUCTION

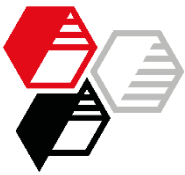
EXPERIMENTAL

Medium CB, N 774,
no additive

RESULTS

SUMMARY

Formulation in phr	non-surface treated		surface treated			
	Polyfil7 0	Silfit Z 91	Burgess KE	Aktifit VM	Aktifit VM	Aktisil VM 56
Butyl 268	100	100	100	100	100	100
N 774	50	50	50	50	50	50
Polyfil 70	150	-	-	-	-	-
Silfit Z 91	-	150	-	-	-	-
Burgess KE	-	-	150	-	-	-
Aktifit VM	-	-	-	150	175	-
Aktisil VM 56	-	-	-	-	-	150
<i>Phenodur EP-560</i>	-	-	-	-	-	-
Zinkoxyd aktiv	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
Dispergator FL	2	2	2	2	2	2
Tackirol 201	18	18	18	18	18	18



Processing in General

INTRODUCTION

EXPERIMENTAL

RESULTS

Medium CB, N 774,
no additive

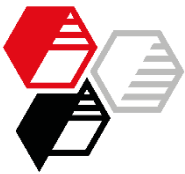
SUMMARY

Problems regarding processing on the lab mill occurred with both **Polyfil 70** and **Burgess KE**:

- ✗ distinct stickiness on the mill

By using **Neuburg Siliceous Earth**, stickiness was reduced:

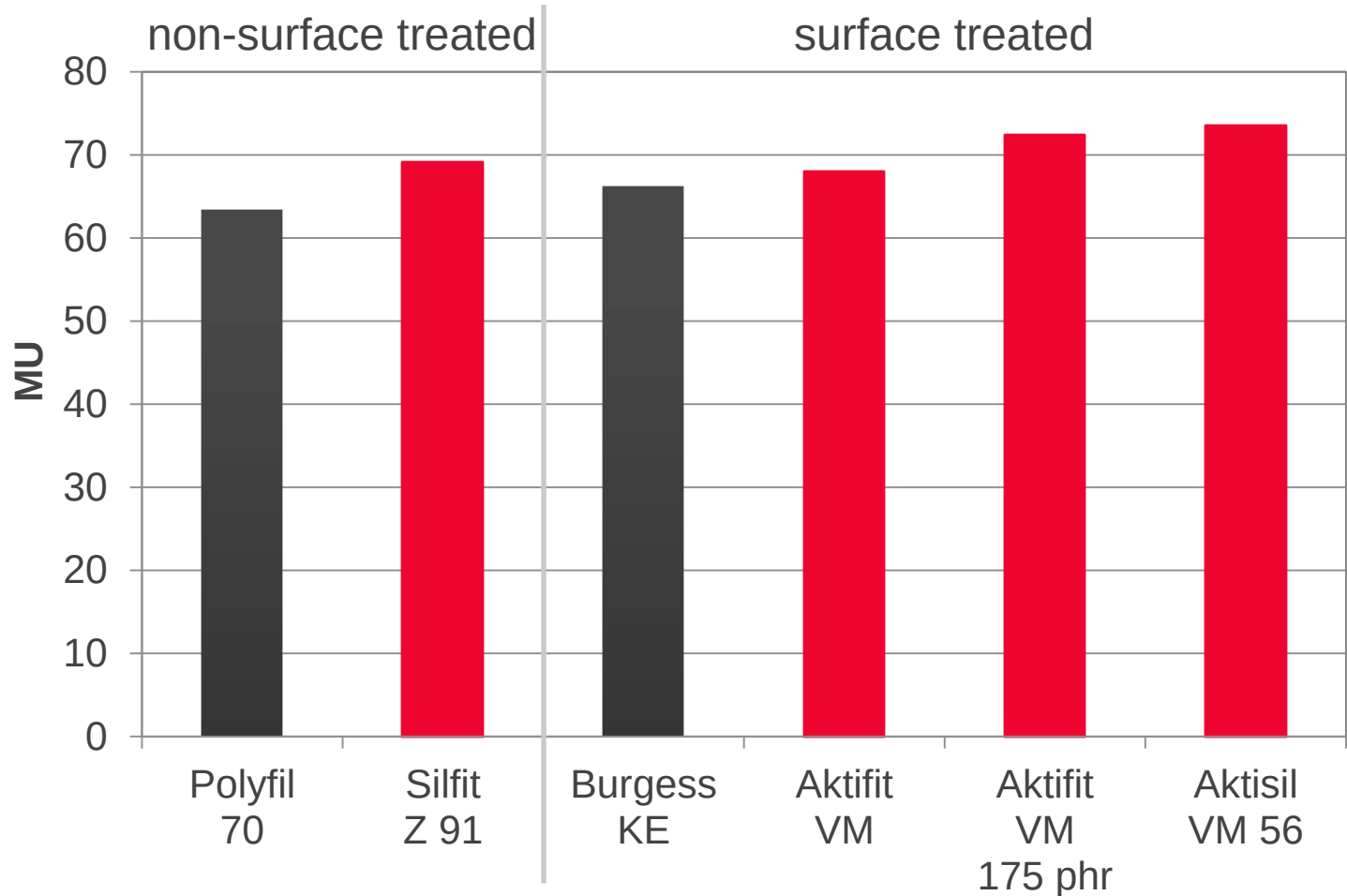
- ✓ less stickiness with **Neuburg Siliceous Earth**



Mooney Viscosity

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DIN 53 523 Part 3, ML 1+4, 120 °C



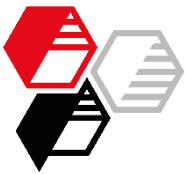
INTRODUCTION

EXPERIMENTAL

RESULTS

Medium CB, N 774,
no additive

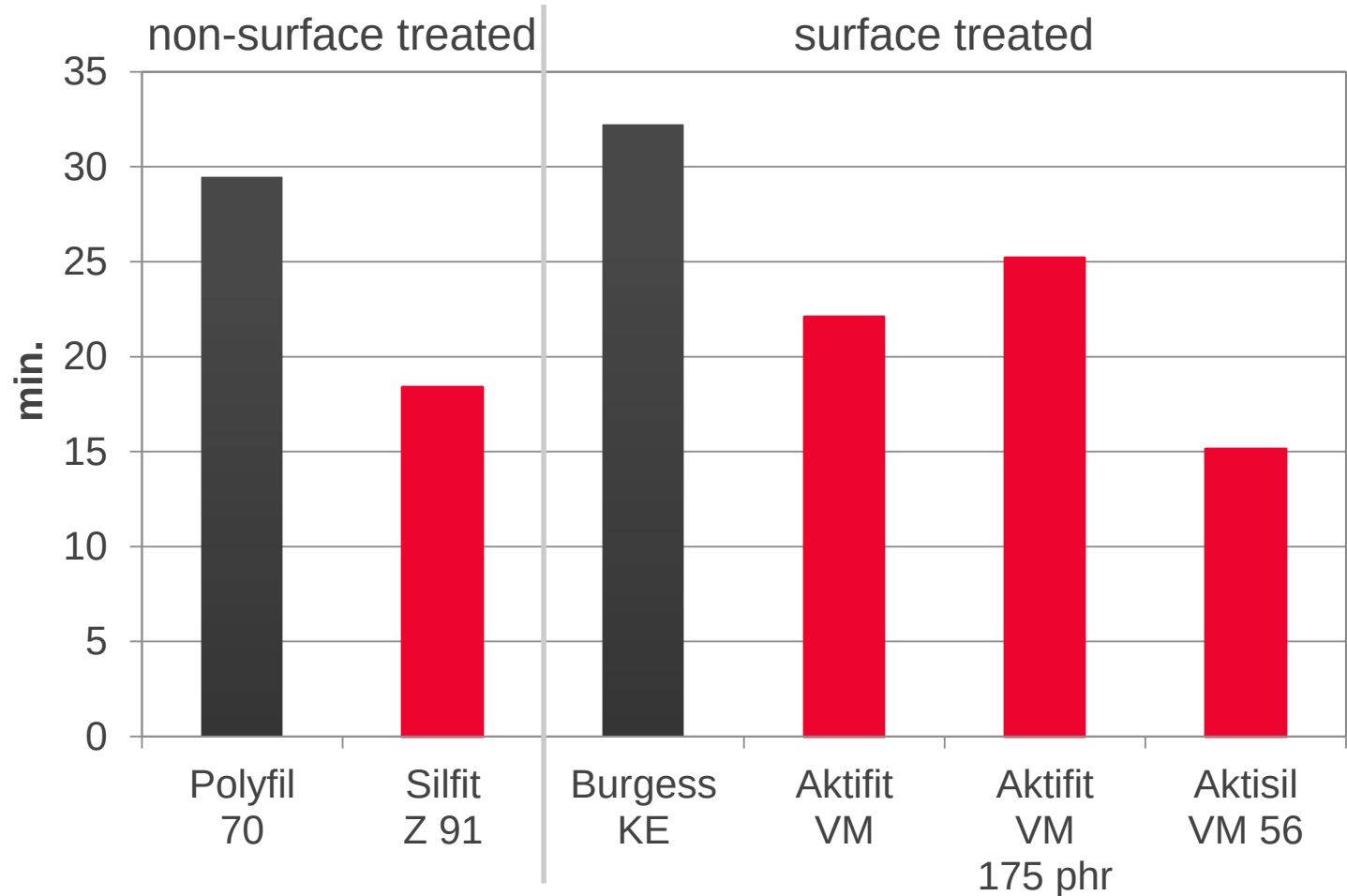
SUMMARY

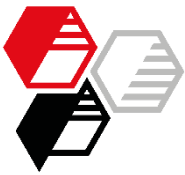


Mooney Scorch Time

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DIN 53 523 Part 4, ML 1+4, 120 °C

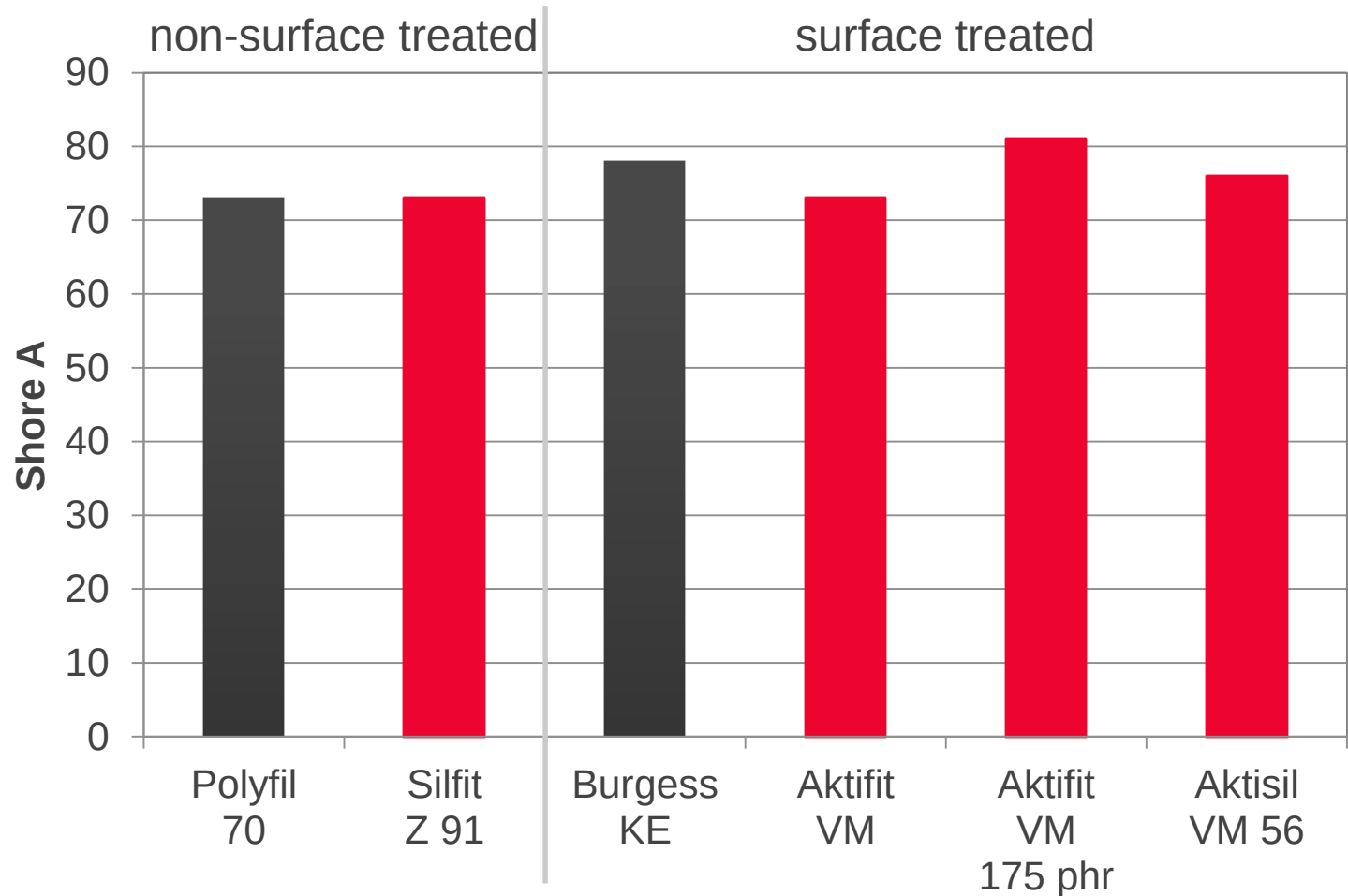




Hardness

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DIN 53 505-A, piled-up S2 dumbbells; post-cured



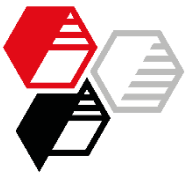
INTRODUCTION

EXPERIMENTAL

RESULTS

Medium CB, N 774,
no additive

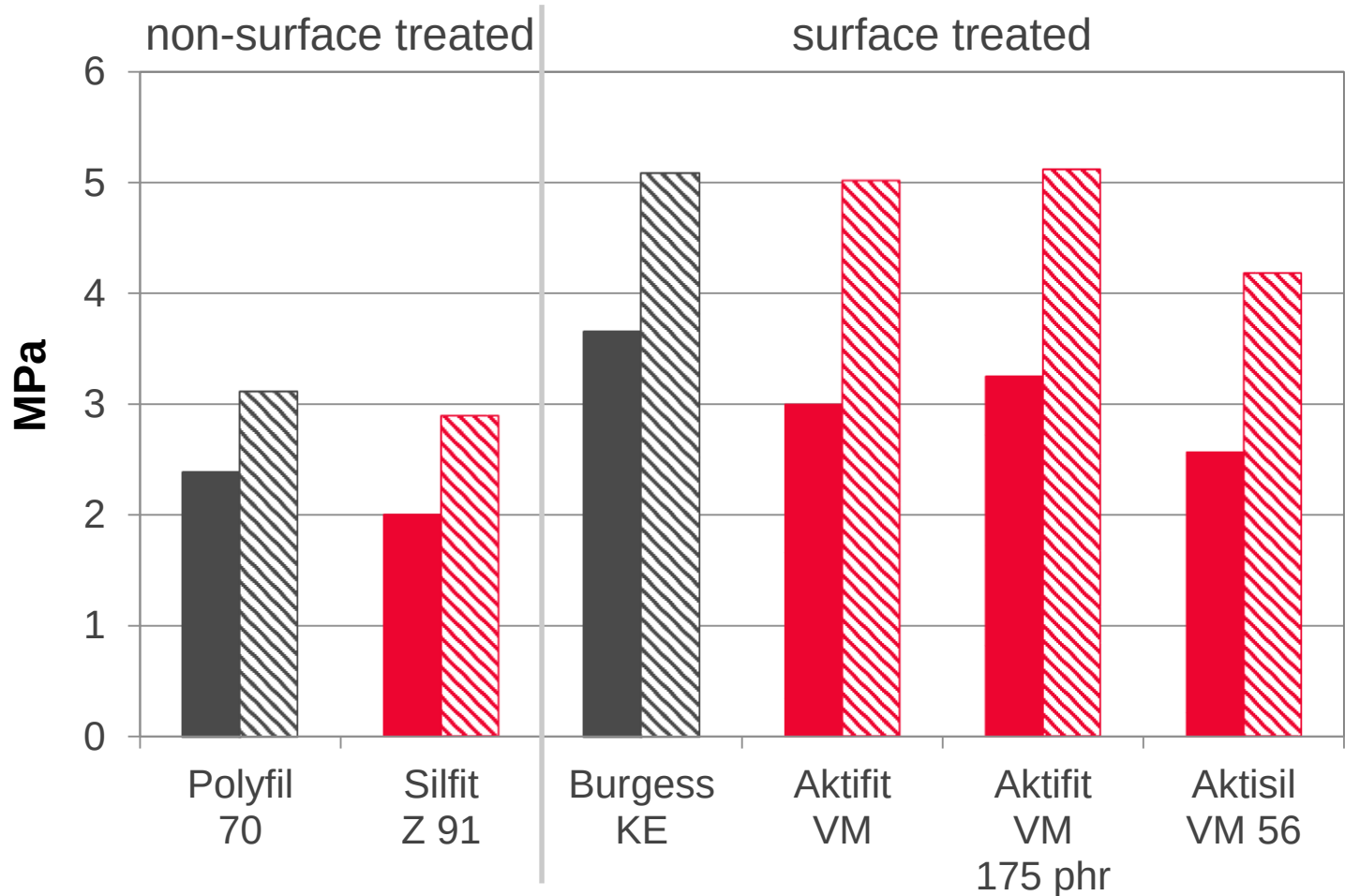
SUMMARY

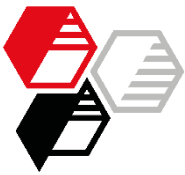


Moduli

DIN 53 504, S2; post-cured

■ Mod. 50 % ▨ Mod. 100 %



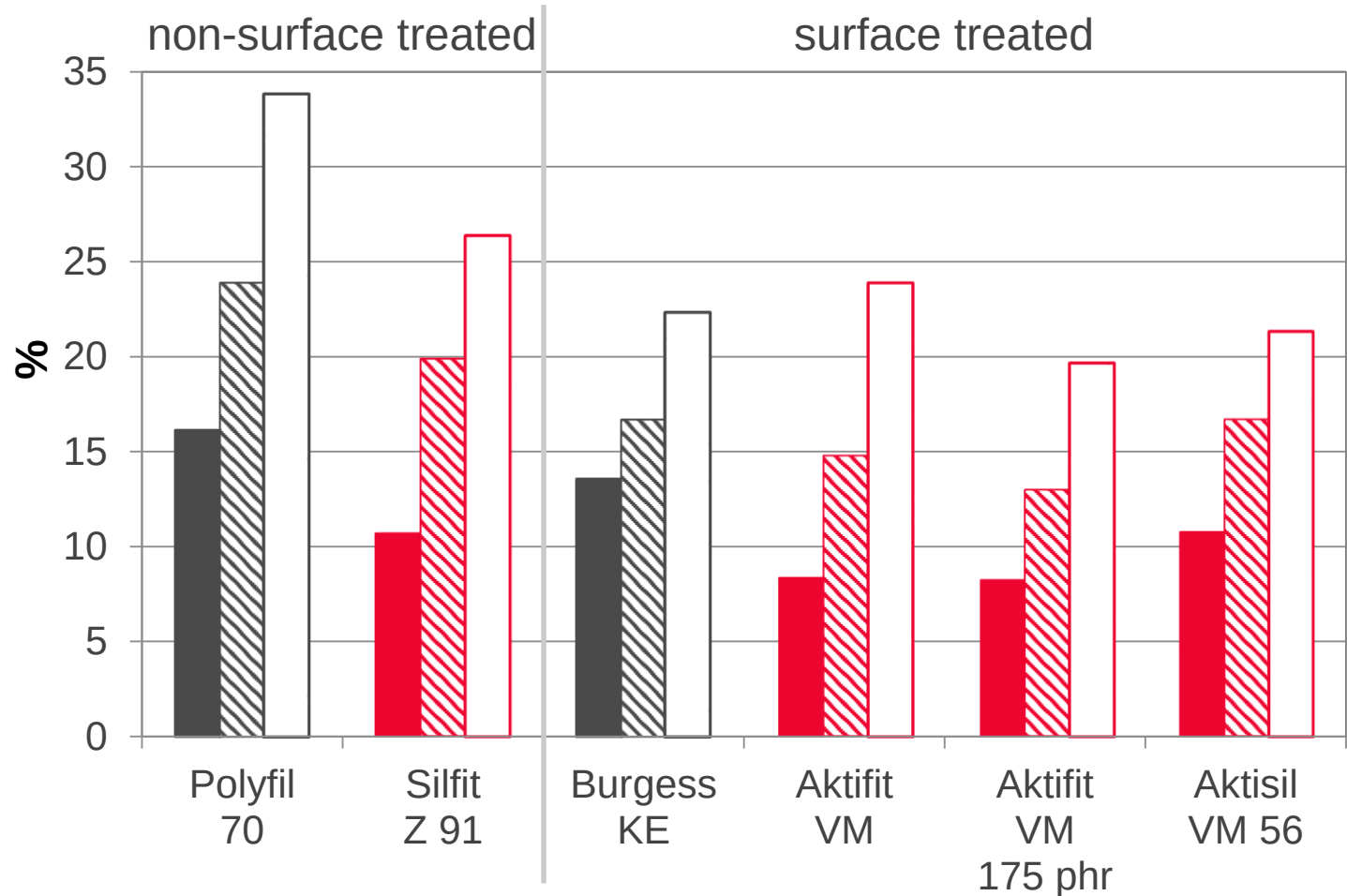


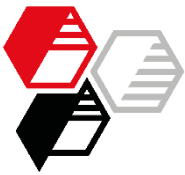
Compression Set 72 h

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DIN ISO 815-1 B, cooling method A, 25 % defl.; post-cured

■ 105 °C ■ 125 °C □ 135 °C





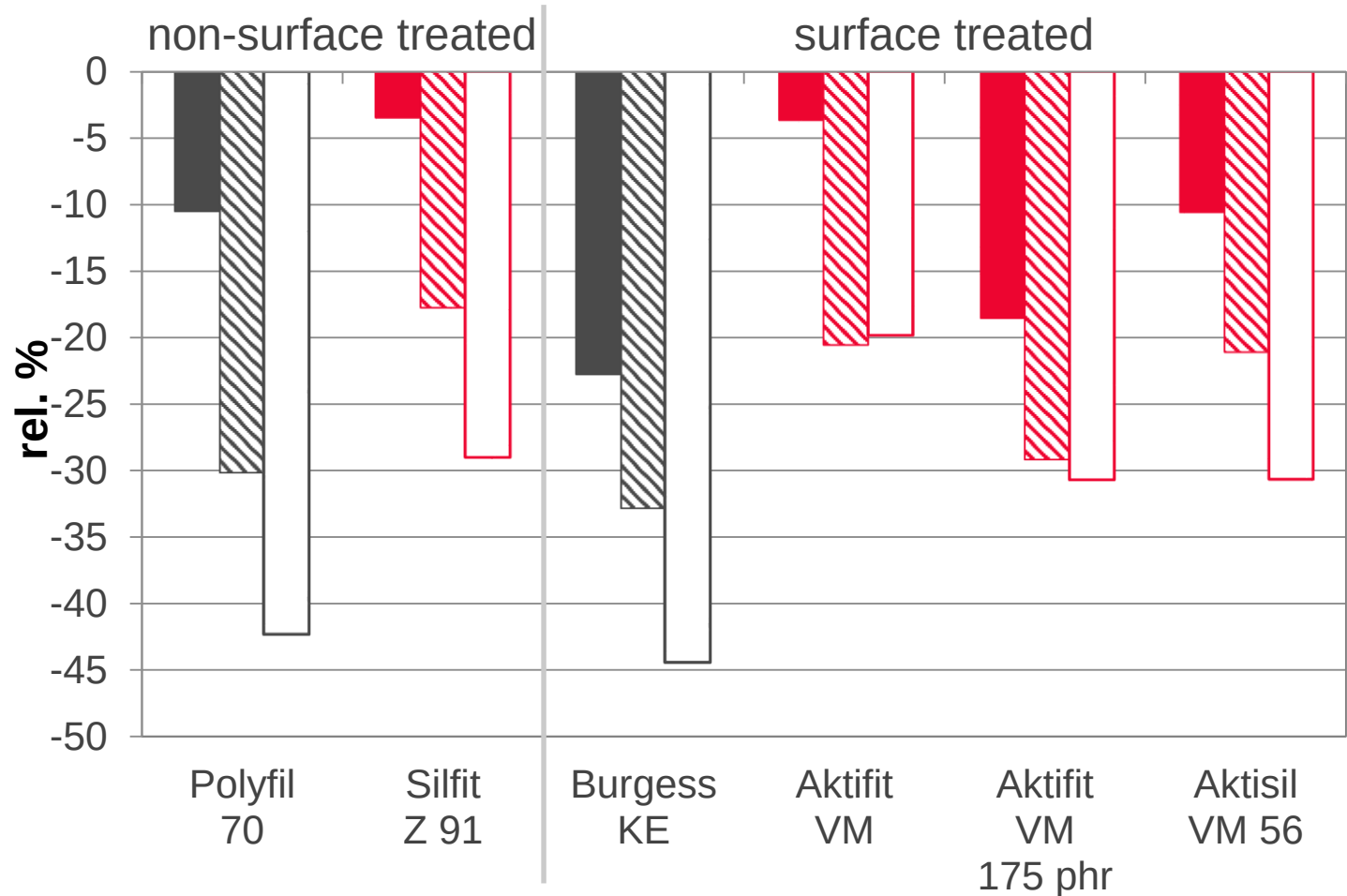
Hot Air Aging, 72 h

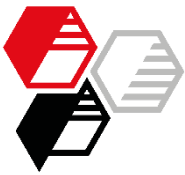
Change of Elongation at B.

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DIN 53 508; post-cured

■ 105 °C ■ 125 °C □ 135 °C





Summary Part 3 – Medium CB using N 774, no Additive

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Non-surface treated fillers

Silfit Z 91 in comparison to Polyfil 70

Processing	+
Hardness	=
Moduli	-
Compression set	+
Aging	+

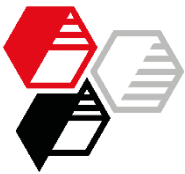
INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

Medium CB, N 774,
no additive



Summary Part 3 – Medium CB using N 774, no Additive

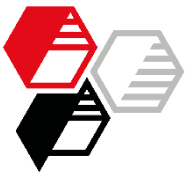
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Surface treated fillers

Neuburg Siliceous Earth grades in comparison to Burgess KE

	Aktifit VM 150 phr	Aktifit VM 175 phr	Aktisil VM 56 150 phr
Processing	+	+	+
Hardness	=	+	=
Moduli	=/-	=	-
Compression set	+	+	=
Aging	+	+	+





Conclusion

High CB Loading, N 990

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Replacing calcined clay by **any evaluated Neuburg Siliceous Earth**
✓ improved processing behavior

non-surface treated

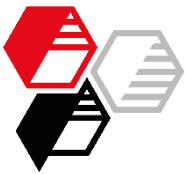
Polyfil 70 → **Silfit Z 91**

- comparable property profile
- + slightly better heat resistance
- + improved compression set

surface treated

Burgess KE → **Aktisil VM 56 or Aktifit VM**

- comparable property profile
 - Aktisil VM 56: lower moduli
 - + Aktifit VM: comparable moduli
- improved compression set



Conclusion

Medium CB Loading, N 774

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Replacing calcined clay by **any evaluated Neuburg Siliceous Earth**
✓ improved processing behavior

non-surface treated

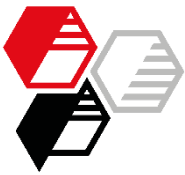
Polyfil 70 → **Silfit Z 91**

- comparable property profile
- + slightly better heat resistance
- + improved compression set

surface treated

Burgess KE → **Aktisil VM 56 or Aktifit VM**

- comparable property profile
- **Aktisil VM 56**
 - + slightly better heat resistance
 - + slightly improved compression set
 - lower moduli
- **Aktifit VM**
 - + better heat resistance
 - + improved compression set
 - slightly lower moduli
 - ↳ improved by increased loading
 - + higher hardness



Conclusion

INTRODUCTION

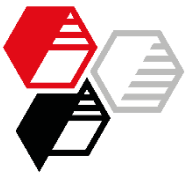
EXPERIMENTAL

RESULTS

SUMMARY

- ✓ **Silfit Z 91** outperforms Polyfil 80 concerning compression set along with comparable mechanical properties.
- ✓ **Aktifit VM** outperforms Burgess KE in the formulation with medium carbon black loading using N 774 in compression set and aging; modulus equals at higher loading along with higher hardness and even further improved compression set.

Calcined Neuburg Siliceous Earth:
Material for good ideas



We supply material for good ideas!

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