Potable water seal EPDM E-DIN EN 681-1 (07/16)
Partial replacement of carbon black
by Neuburg Siliceous Earth

Author: Karin Müller

HOFFMANN MINERAL®
We supply material for good ideas

Contents

Status Quo

- Range of application: Elastomers in contact with drinking water Hardness range 70 IRHD (recommended by Arlanxeo)
- Raw materials must comply with strict requirements according to the German Federal Environment Agency (Umweltbundesamt, UBA) Positive List part 1, part 2 (till12/21) and part 3 for the drinking water application. PAH[1] content in the mixture has to be below 10 % of the required limit value of the German Drinking Water Ordinance 2011
- Purex HS 45: reduced PAH content, high price fluctuations with long-term rising cost level, temporary supply bottlenecks
- Neuburg Siliceous Earth: Price increases only slightly in the long term, high calculability and availability, reliable delivery times. Purity requirements BfR[2] Part 1 A LII fulfilled, reduces PAH content in the compound

[1]Polycyclic aromatic hydrocarbons
[2]German Federal Institute for Risk Assessment

Objective

Partial replacement of the carbon black Purex HS 45 by

Neuburg Siliceous Earth

in compliance with the standard E-DIN EN 681-1 (07/16)

and additional positive effect on costs.

Requirements profile

E-DIN EN 681-1 (draft July 2016)

Elastomeric seals - Material requirements for pipe joint seals used in water and drainage applications - Part 1: Vulcanized rubber

- WB Cold drinking water supply (T ≤ 50 °C) and continuously hot drinking water supply (T ≤ 110 °C)
- WD Cold non drinking water supply (T ≤50 °C) and continuously hot non drinking water supply (T ≤110 °C)
- Hardness 70 IRHD (+/-5 IRHD)

Base Formulation

		phr
Keltan®2650	EPDM amorphous	100.0
Zinkoxyd aktiv	Zinc oxide	3.0
Stearic acid	Processing aid	0.3
Purex HS 45	**FEF-Carbon black, reduced PAH content**	80.0
Caldic PIB 190	Polyisobutylene, Plasticizer	15.0
Safic Chem OMB	BPH, 2,2'-methylene-bis-(4-methyl-6-tert. butylphenol), Antioxidant	0.75
Luperox 101-XL 45	DHBP, 2,5-Dimethyl-2,5-Di(tert-butyl peroxide)hexane, Peroxide / Crosslinker	5.33
TAC 70	Triallyl cyanurate, Activator	0.7
Total		**205.08**

All selected raw materials meet the criteria of the UBA Positive List for the production of elastomers in contact with drinking water Part 1 and Part 3 (as of July 2021).

Formulation Variations

Dosages in phr for equal hardness

Purex HS 45	Carbon black replacement level	Sillitin Z 86	Aktisil VM 56
80			
60	25 %	40	40
50	37 %	60	60
40	50 %	80	80

Fillers and Characteristics

HOFFMANN MINERAL

		Purex HS 45	Sillitin Z 86	Aktisil VM 56
Density	[g/cm³]	1.8	2.6	2.6
Particle size d_{50}	[µm]		1.9	2.2
Particle size d_{97}	[µm]		9.0	10
Sieve residue > 40 µm	[mg/kg]		20	20
Sieve residue 45 µm / 325 mesh	ppm	≥ 50		
Oil absorption	[g/100g]		55	45
Jod adsorption	[mg/g]	43 +/- 5		
Specific surface area BET	[m²/g]		12	9
STSA surface area	[m²/g]	39 +/- 5		
Functionalization		none	none	Vinyl

Preparation and Curing of the Compounds

INTRODUCTION
EXPERIMENTAL
RESULTS
SUMMARY
APPENDIX

- **Mixing**

  Open mill Ø 150 x 300 mm

  Batch volume: approx. 800 cm³

  Temperature: 50 °C

  Mixing time: approx. 20 min.

- **Curing**

  Press: 180 °C

  Curing time: t_{90} + 10 % resp. 12 min.

- **Post cure**

  Unless otherwise stated, 2 h / 125 °C

Test Standards

INTRODUCTION

EXPERIMENTAL

RESULTS

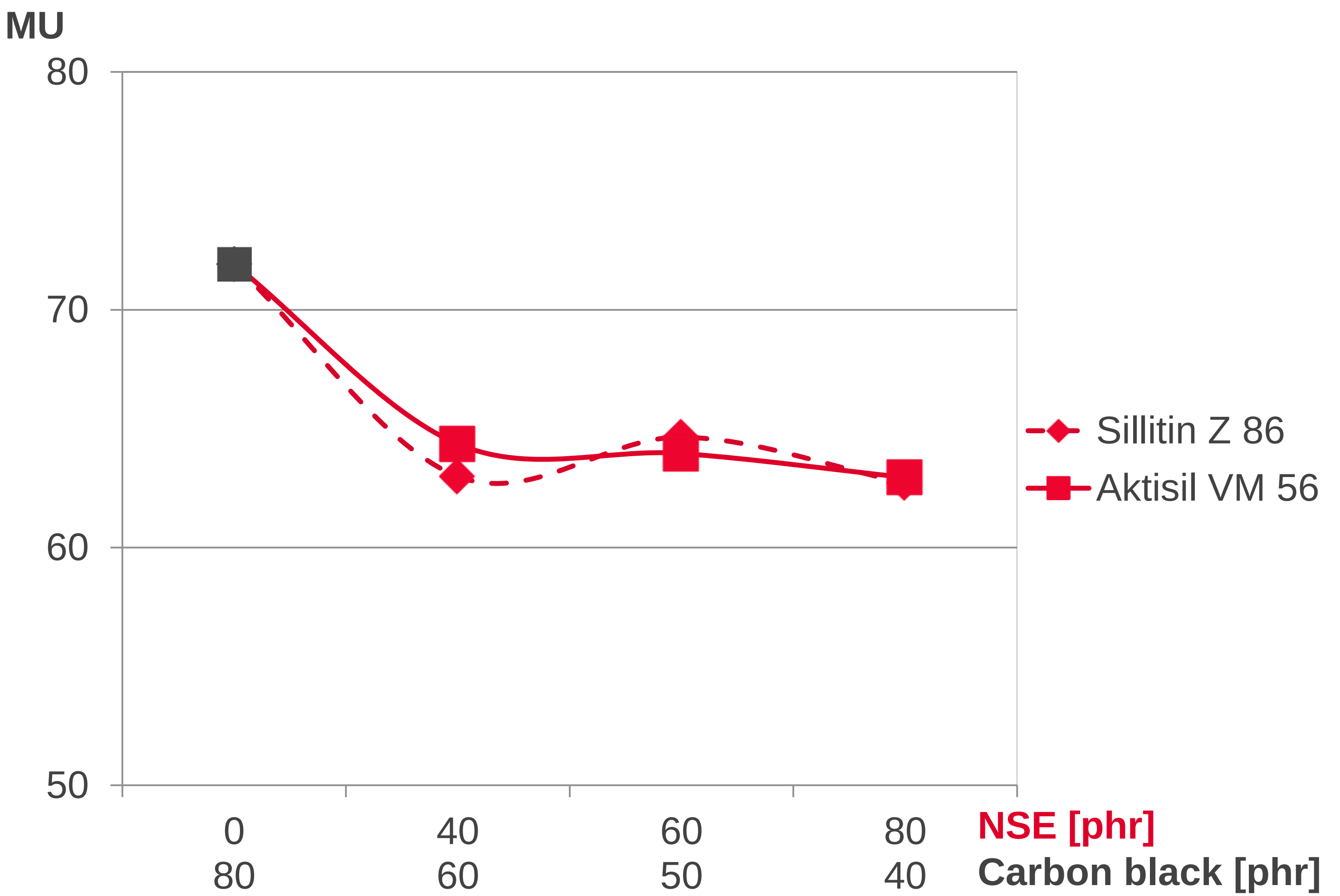
SUMMARY

APPENDIX

Test	Standard
Mooney Viscosity, ML 1+4	DIN ISO 289-1
Mooney Scorch, ML +5	DIN ISO 289-2
Rotorless curemeter	DIN 53 529 Part 3
Hardness	DIN ISO 7619-1
Tensile strength	DIN 53 504, S2
Modulus 100 %	DIN 53 504, S2
Elongation at break	DIN 53 504, S2
Rebound	DIN 53 512
Tear resistance, trouser specimen	DIN ISO 34-1, A
Tear resistance, delft	DIN ISO 34-2, A
Compression set	DIN ISO 815-1, B
Compression set	DIN ISO 815-2, B
Hot air aging	DIN 53 508
Immersion distilled water	ISO 1817

Mooney Viscosity

DIN ISO 289-1, ML 1+4 100 °C



Conversion Time t_{90}

INTRODUCTION

EXPERIMENTAL

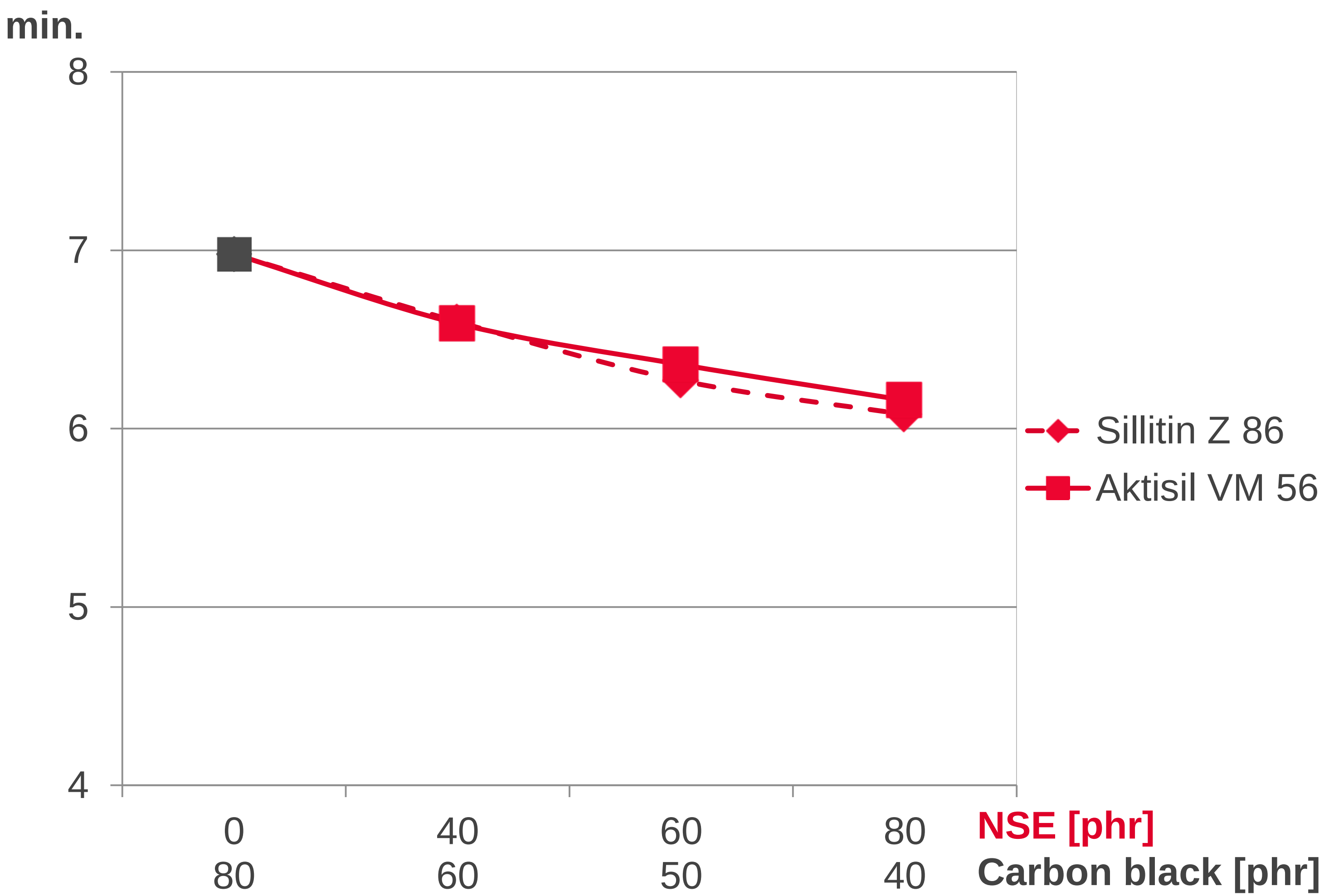
RESULTS

• Rheology

SUMMARY

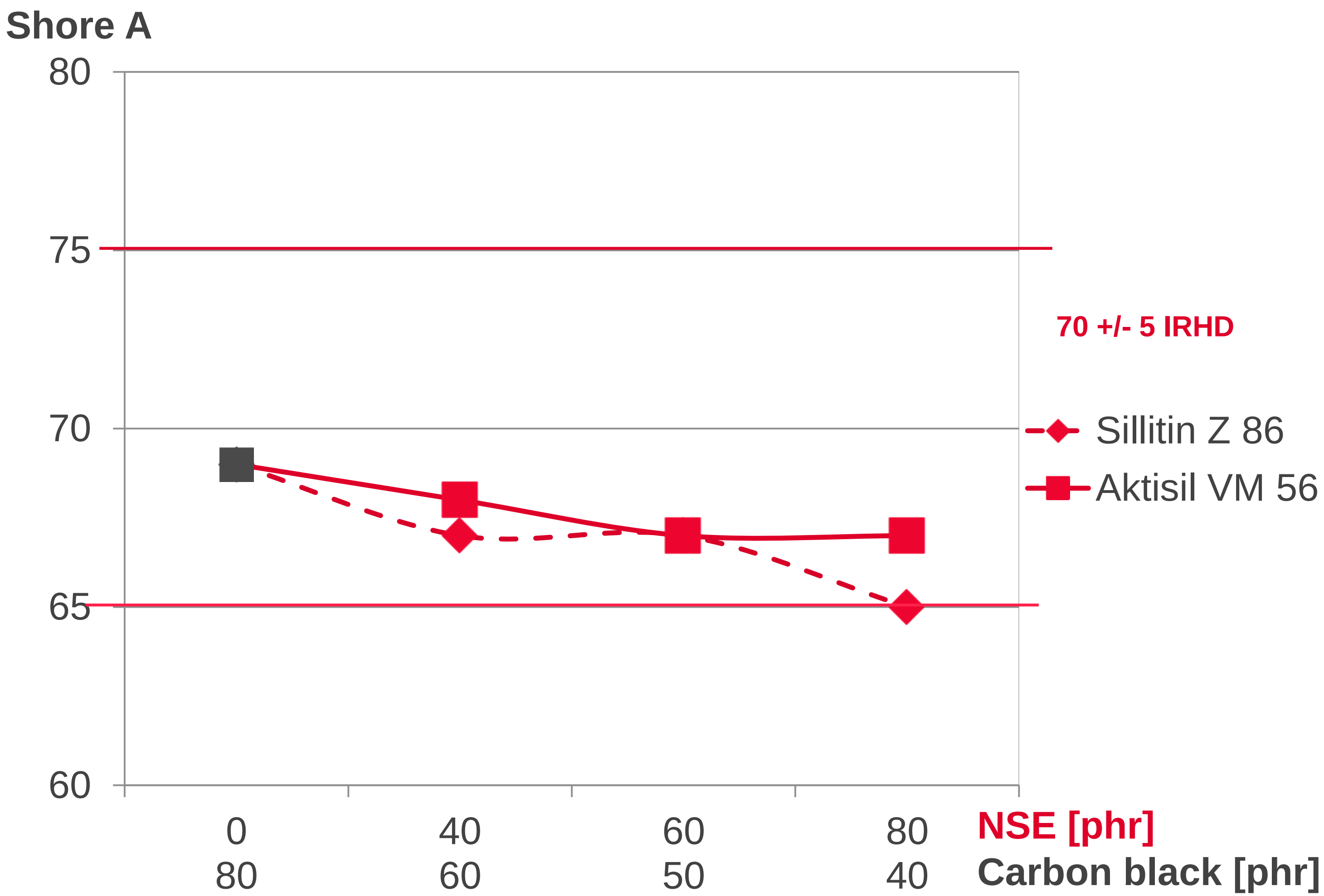
APPENDIX

DIN 53 529-A3, 180 °C, 0,2° deflection - Göttfert Elastograph



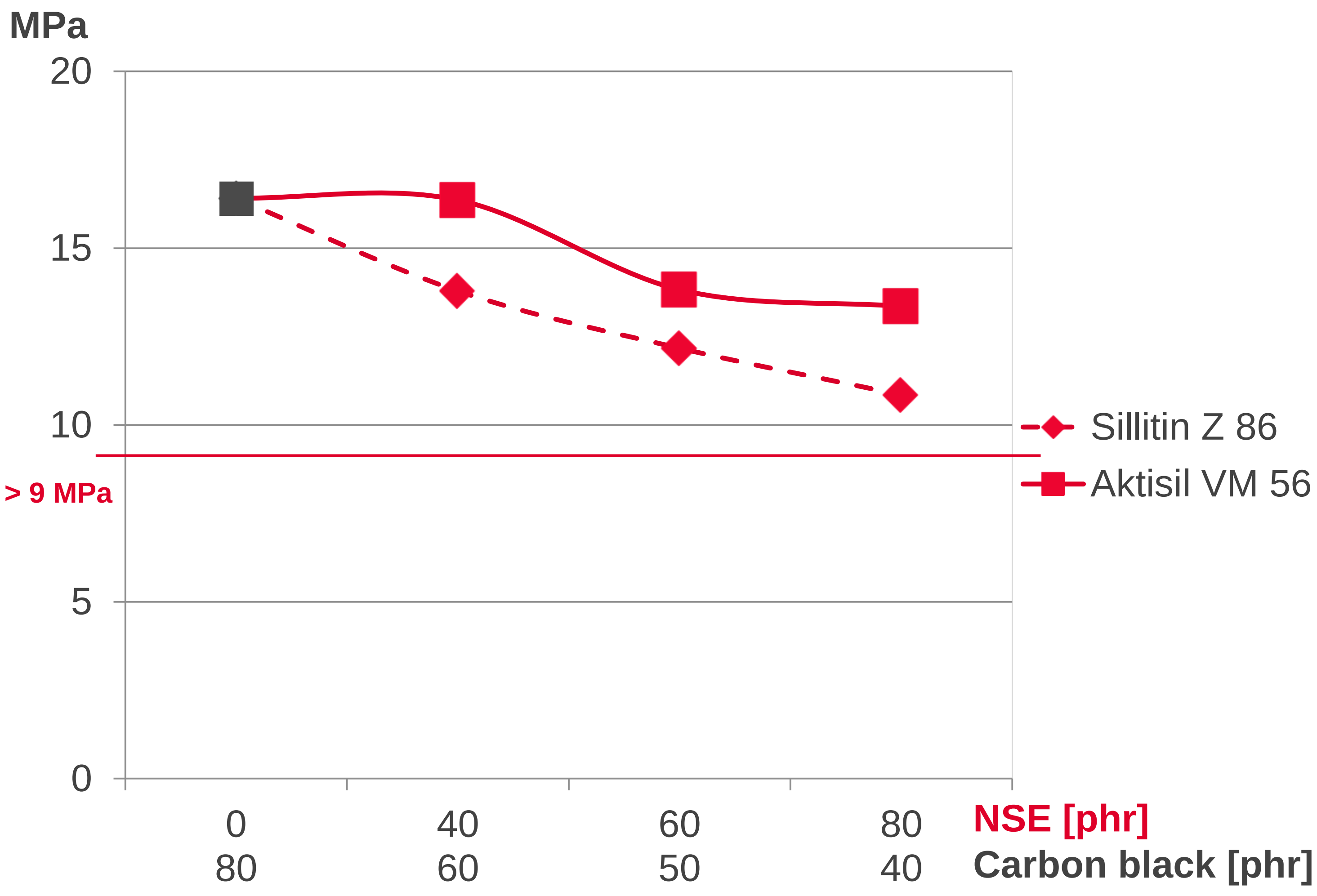
Hardness

DIN ISO 7619-1, piled-up S2 dumbbells



Tensile Strength

DIN 53 504, S2



Elongation at Break

INTRODUCTION

EXPERIMENTAL

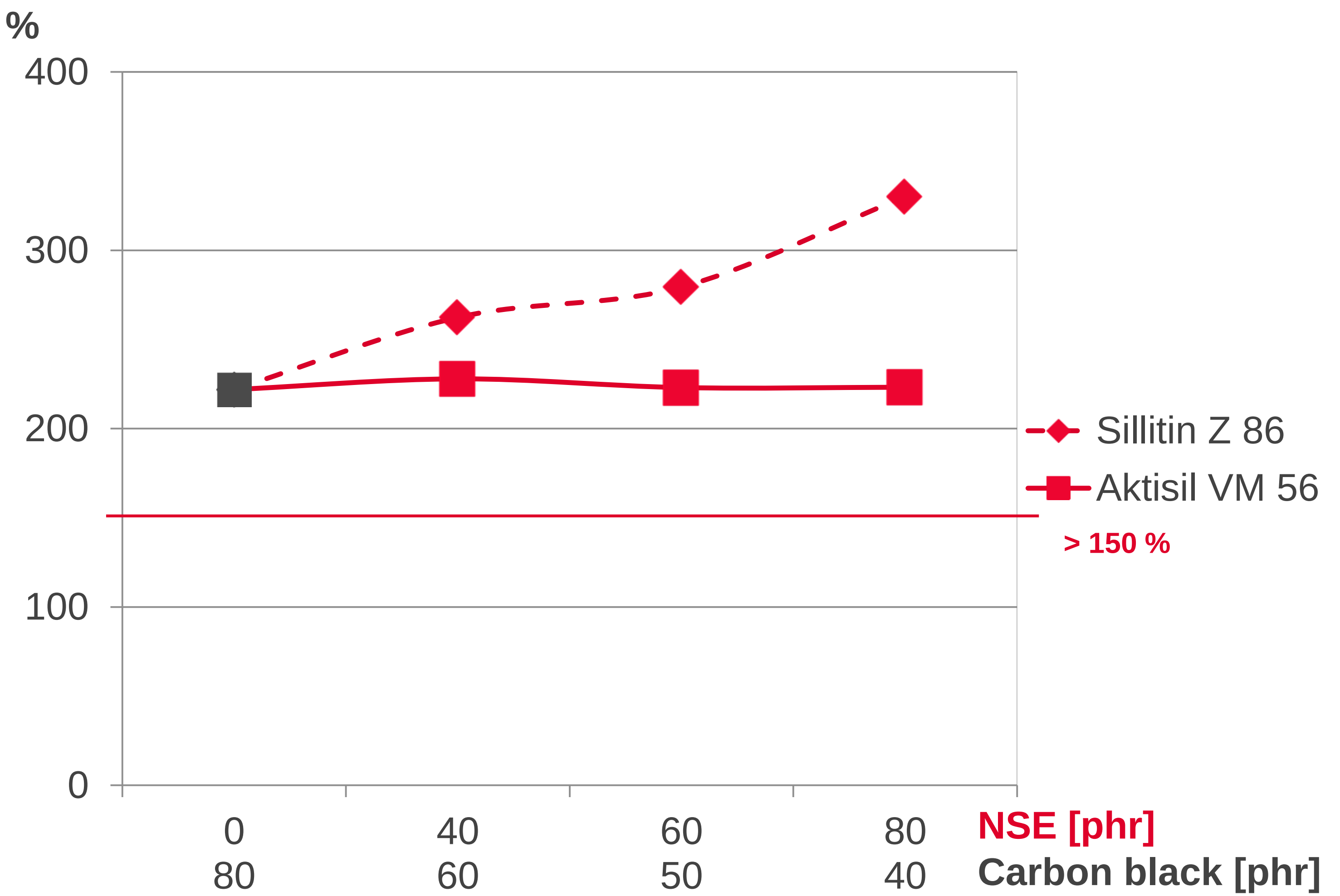
RESULTS

• Mechanical properties

SUMMARY

APPENDIX

DIN 53 504, S2



Tear Resistance

INTRODUCTION

EXPERIMENTAL

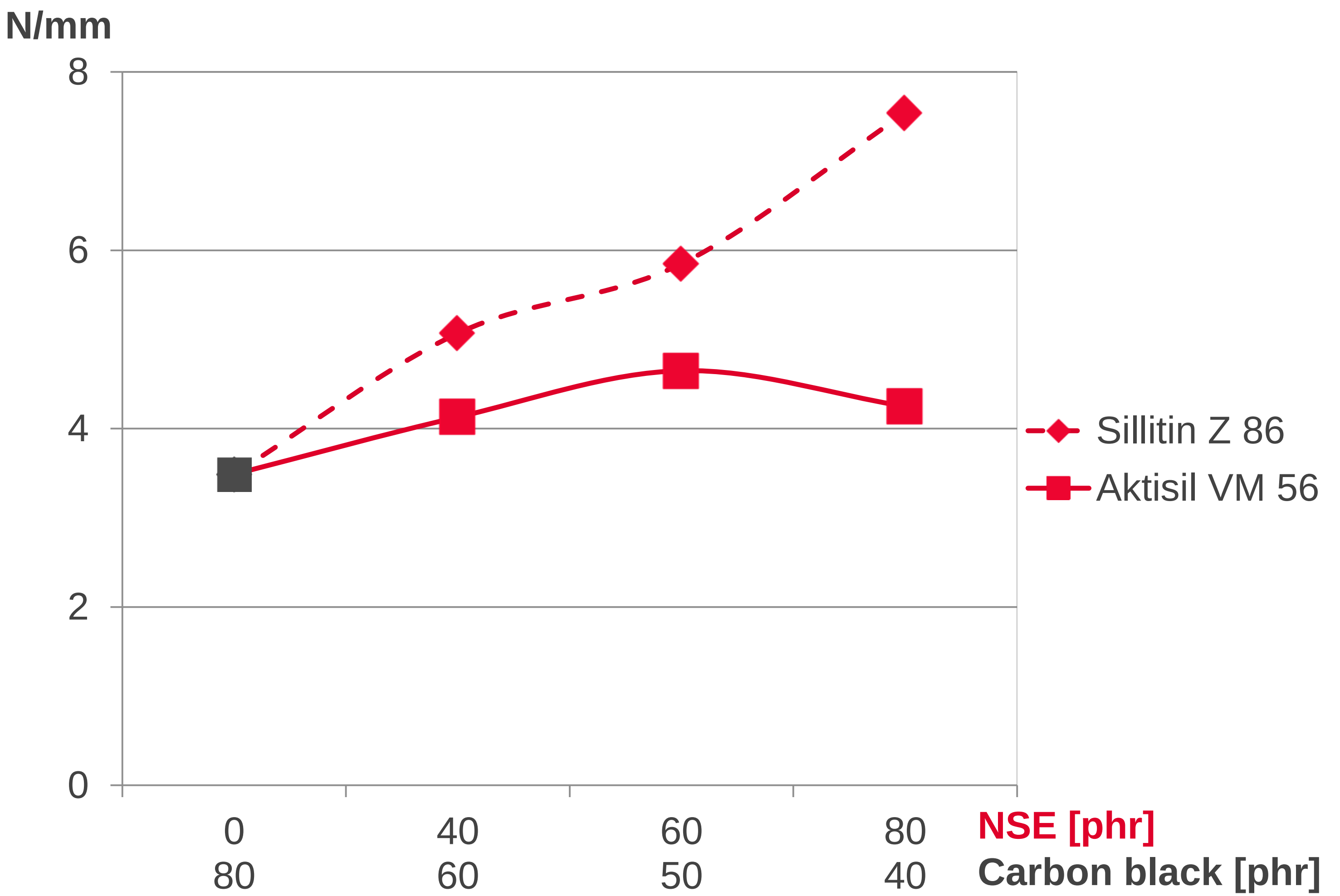
RESULTS

• Mechanical properties

SUMMARY

APPENDIX

DIN ISO 34-1, A (trouser)



Tear Resistance

DIN ISO 34-2, A (delft)

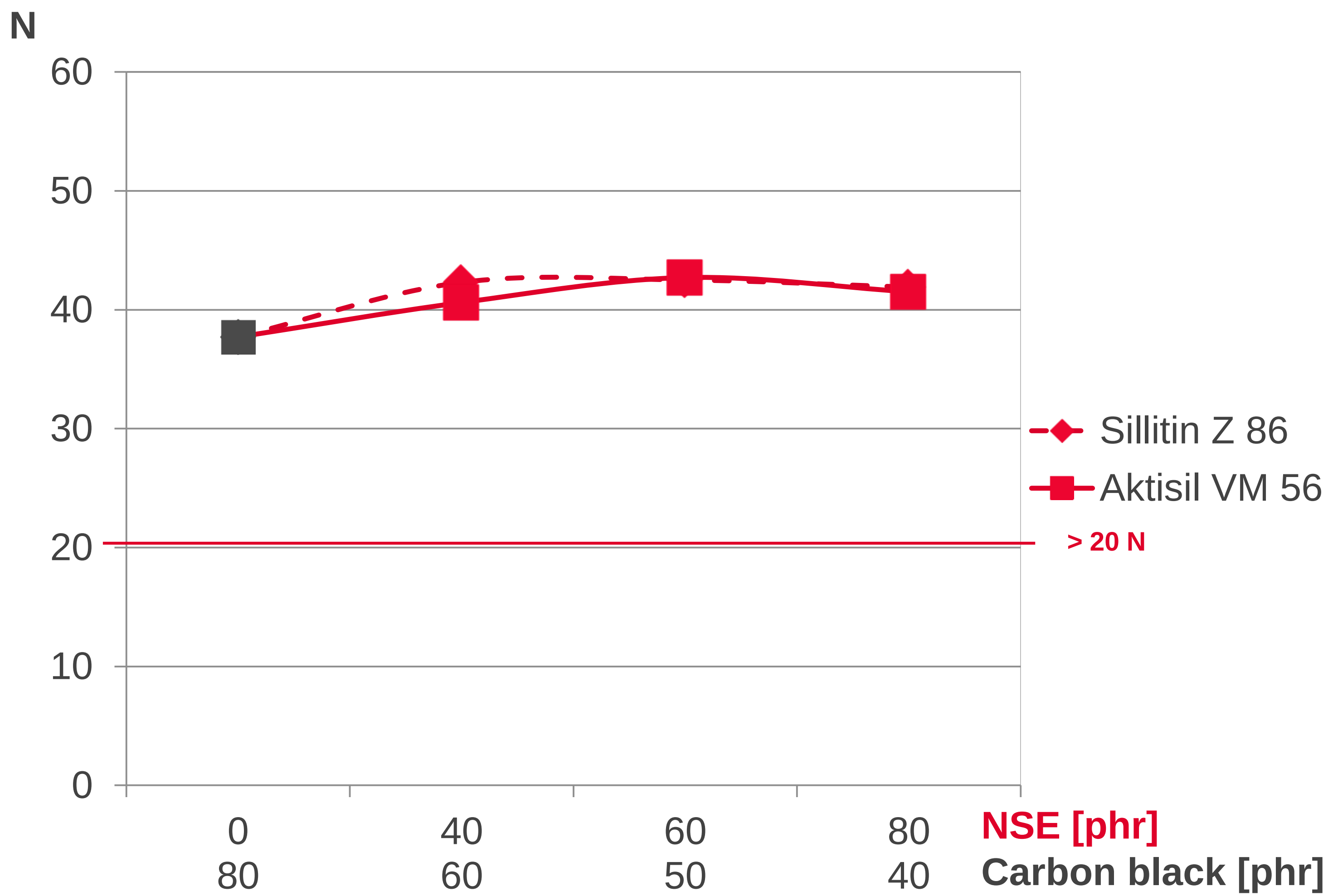
INTRODUCTION

EXPERIMENTAL

RESULTS
- Mechanical properties

SUMMARY

APPENDIX



Compression Set 72 h / 23°C
Press Cure t_{90} + 10 %

INTRODUCTION

EXPERIMENTAL

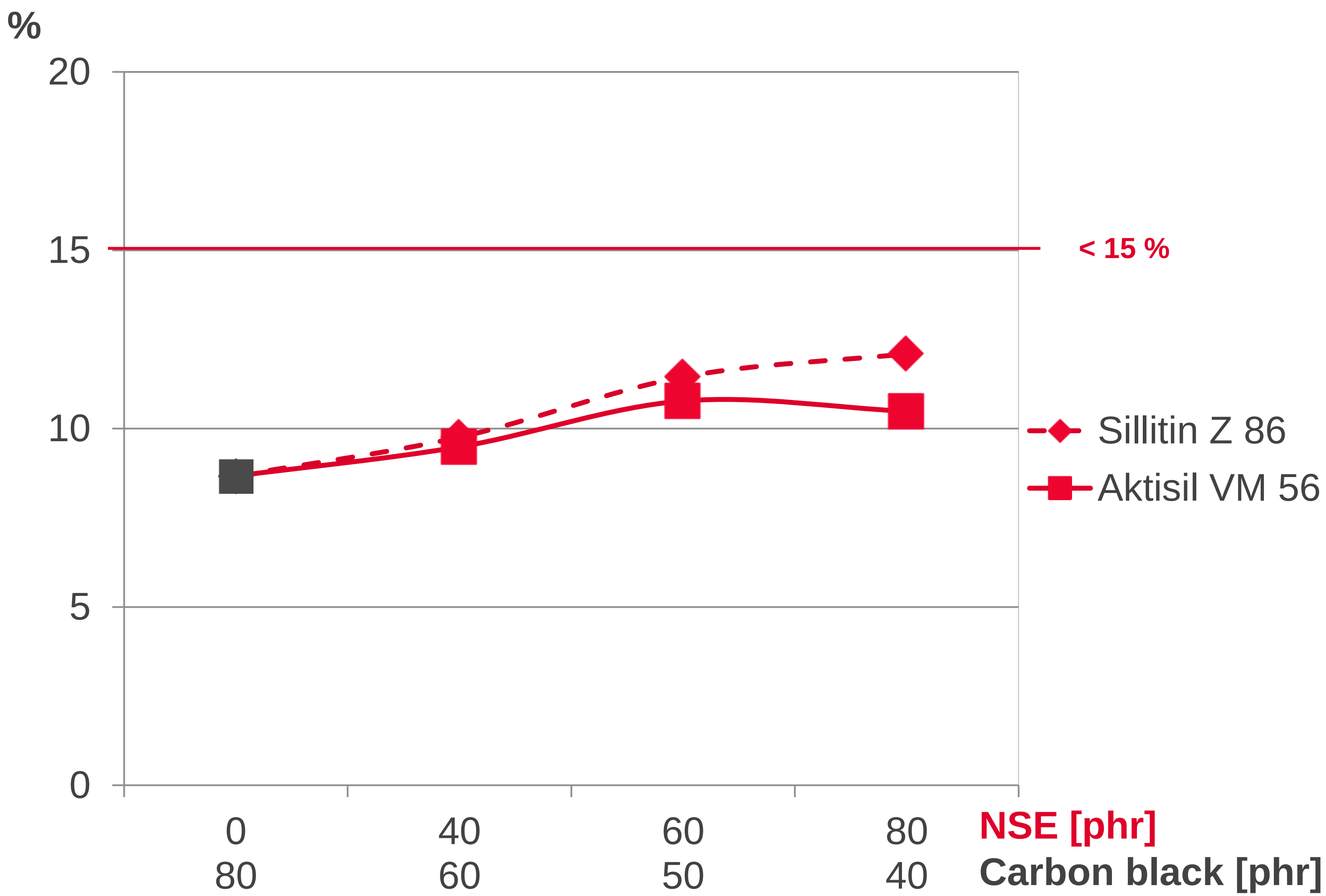
RESULTS

• Mechanical properties

SUMMARY

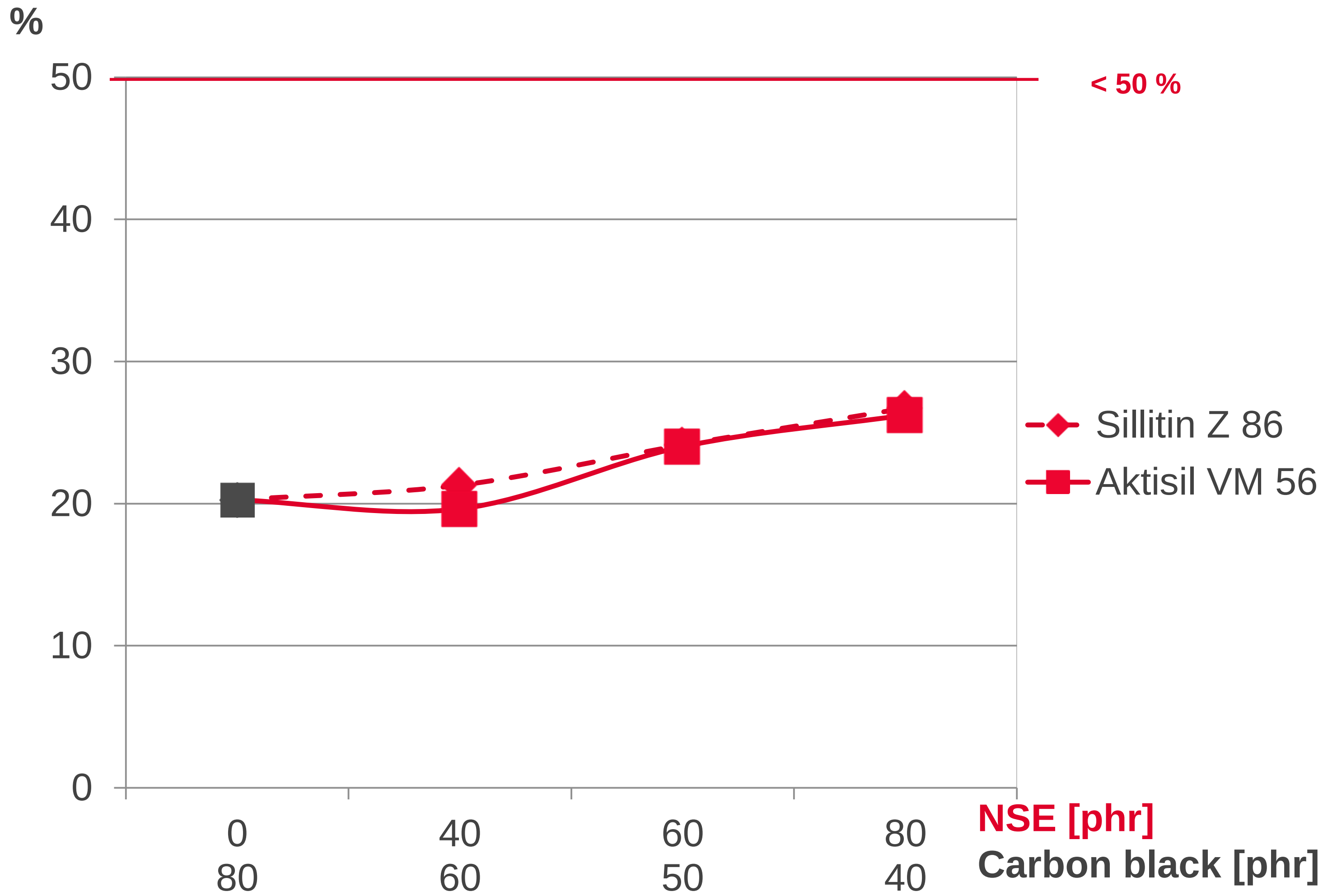
APPENDIX

DIN ISO 815-1 B, 25 % deflection



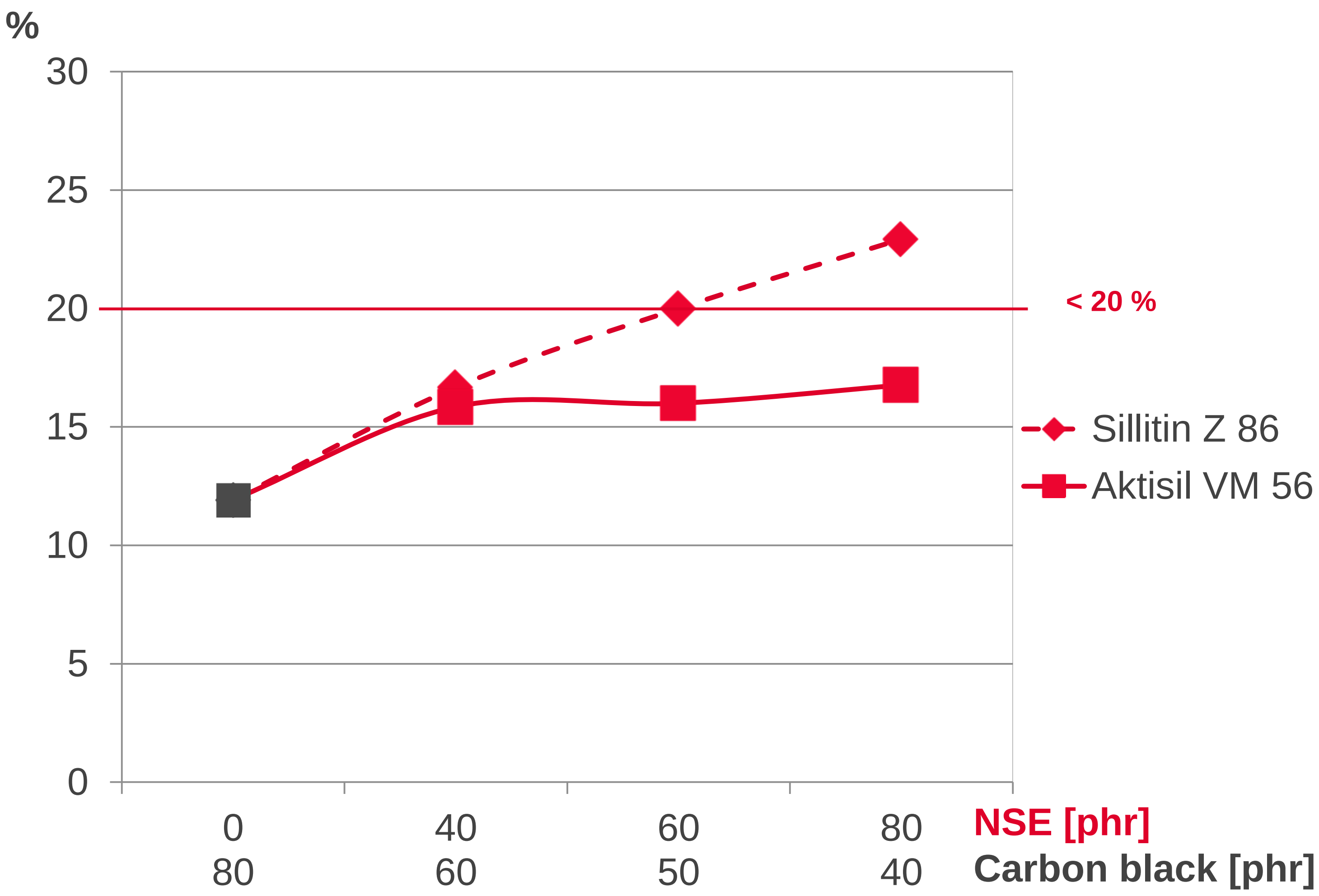
Compression Set 72 h / -10 °C
Press Cure t_{90} + 10 %

DIN ISO 815-2 B, 25 % deflection



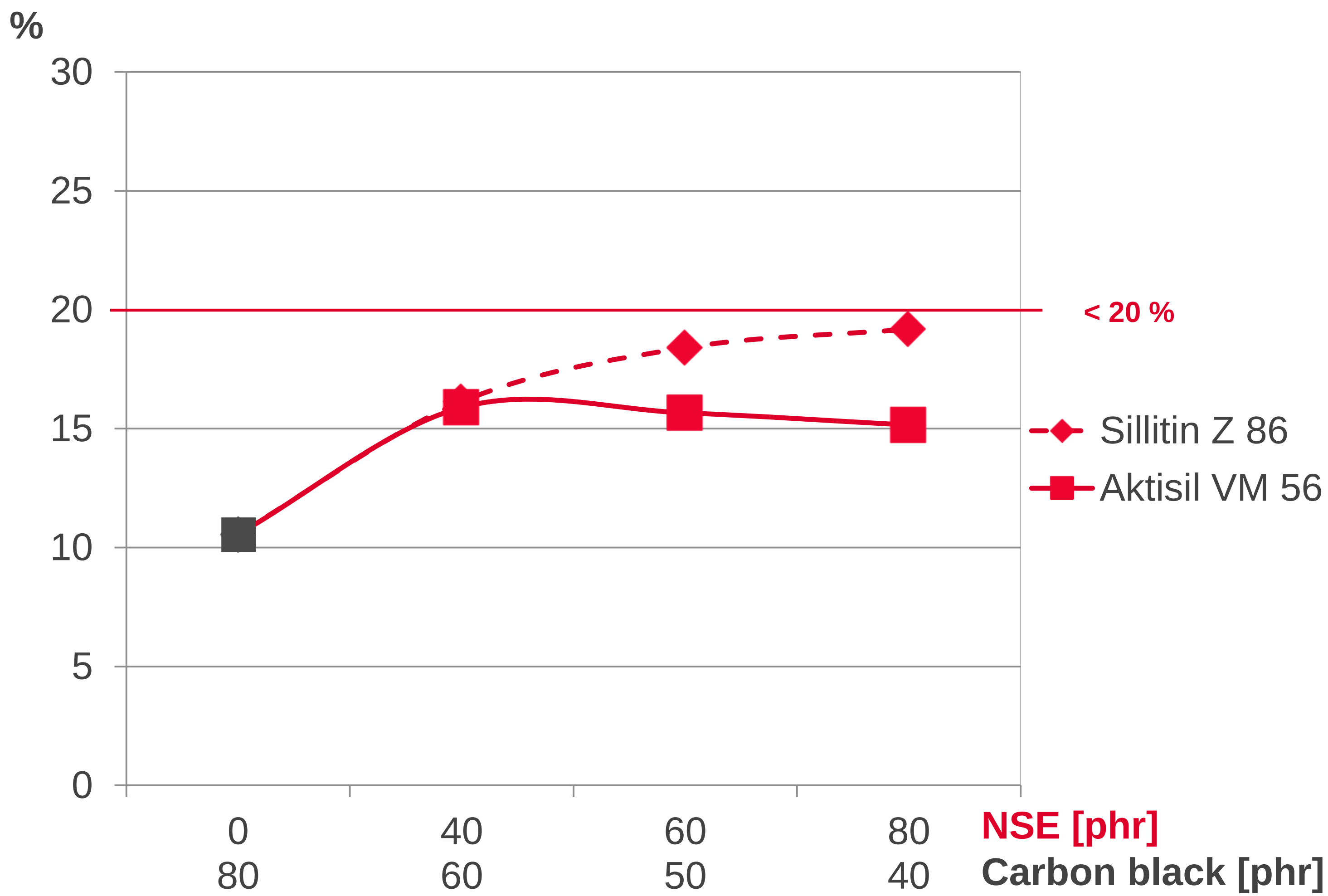
Compression Set 24 h / 125 °C
Press Cure t_{90} + 10 %

DIN ISO 815-1 B, 25 % deflection



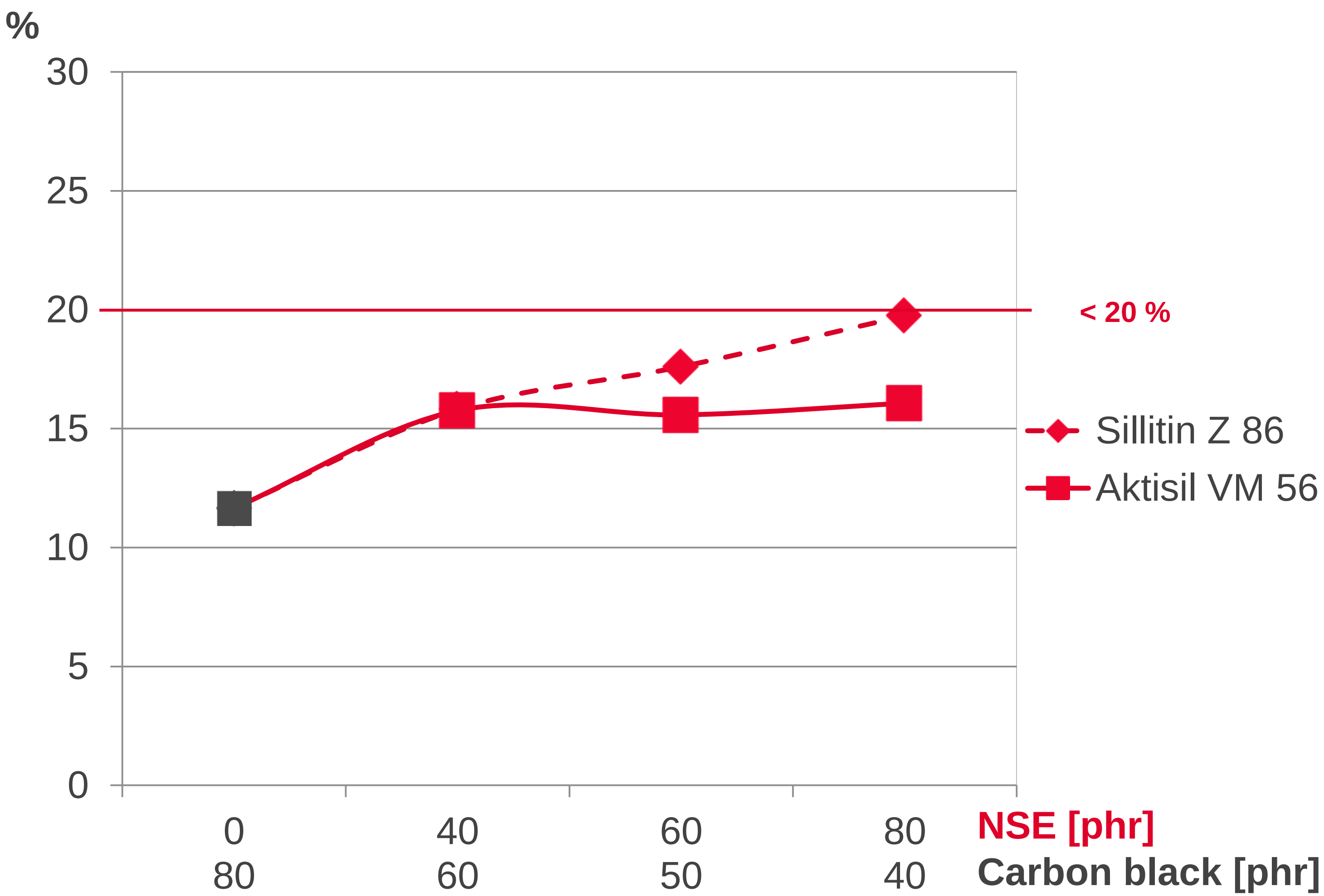
Compression Set 24 h / 125 °
Press Cure 12 min.

DIN ISO 815-1 B, 25 % deflection



Compression Set 24 h / 125 °C
Post Cure 2 h / 125 °C

DIN ISO 815-1 B, 25 % deflection

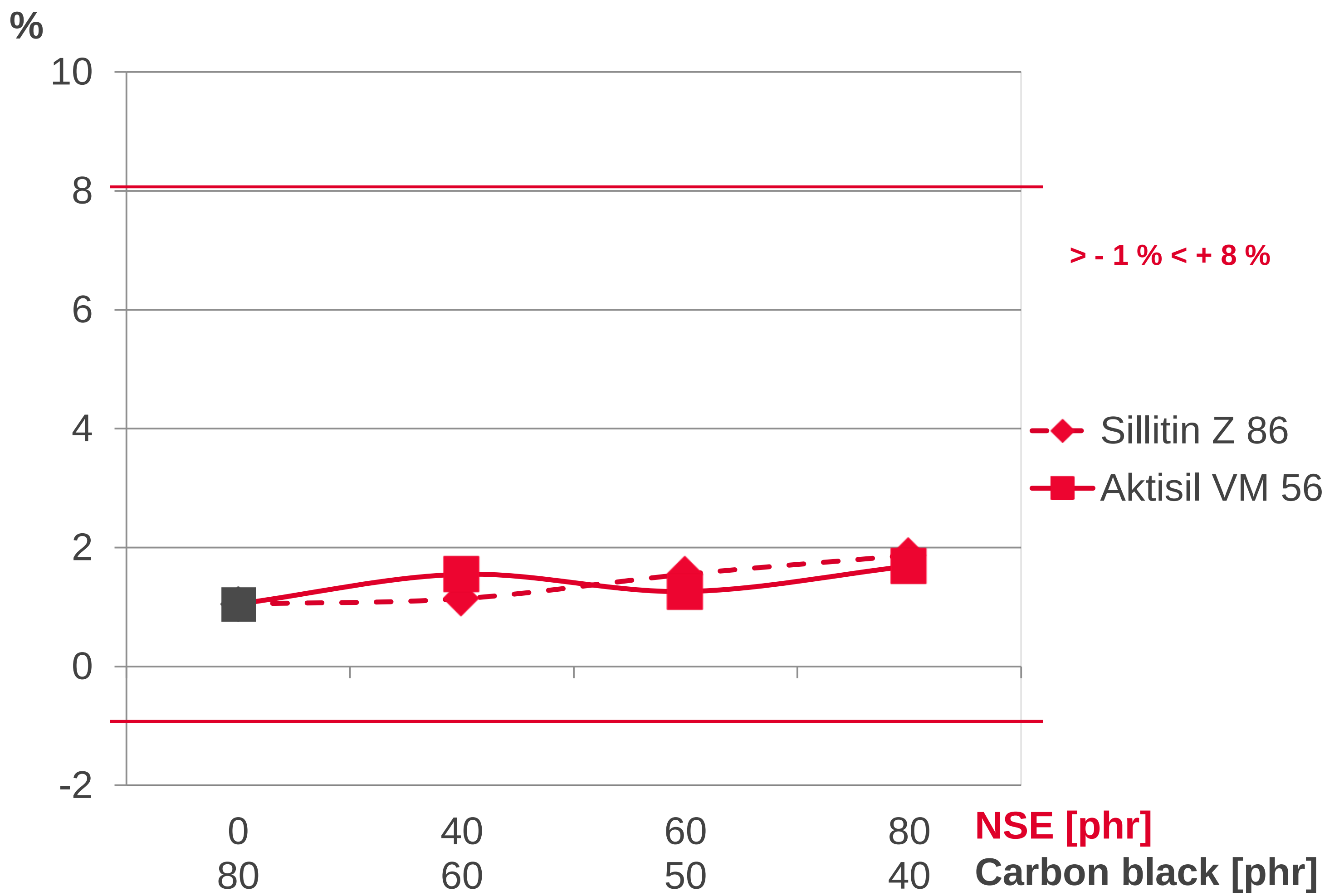


Immersion in Distilled Water
Change of Volume

HOFFMANN MINERAL

- INTRODUCTION
- EXPERIMENTAL
- RESULTS
  - • Immersion in distilled water
- SUMMARY
- APPENDIX

168 h / 95 °C



Hot Air Aging
Change of Hardness

INTRODUCTION

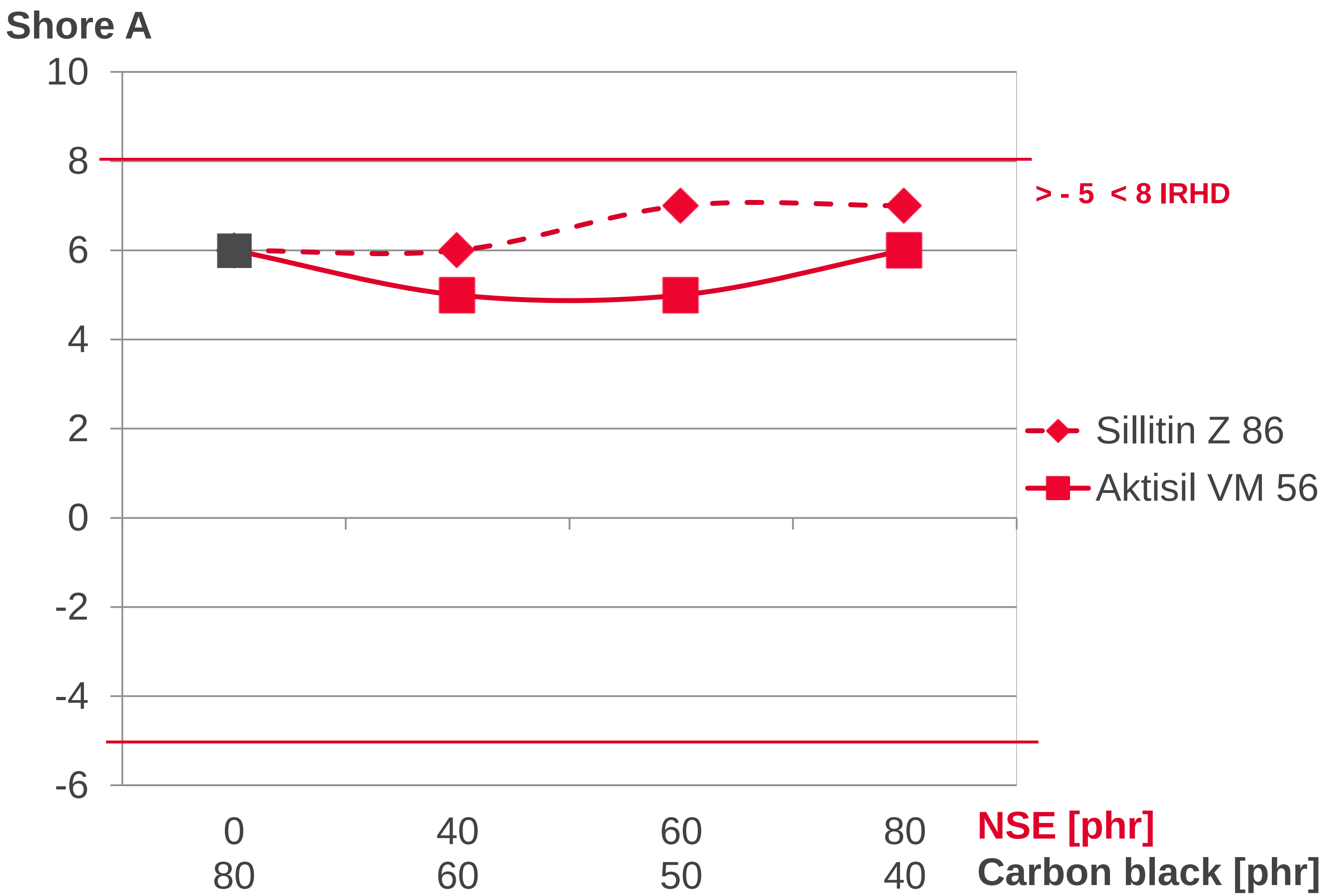
EXPERIMENTAL

RESULTS
• Hot air aging

SUMMARY

APPENDIX

168 h / 125 °C



Hot Air Aging
Change of Tensile Strength

INTRODUCTION

EXPERIMENTAL

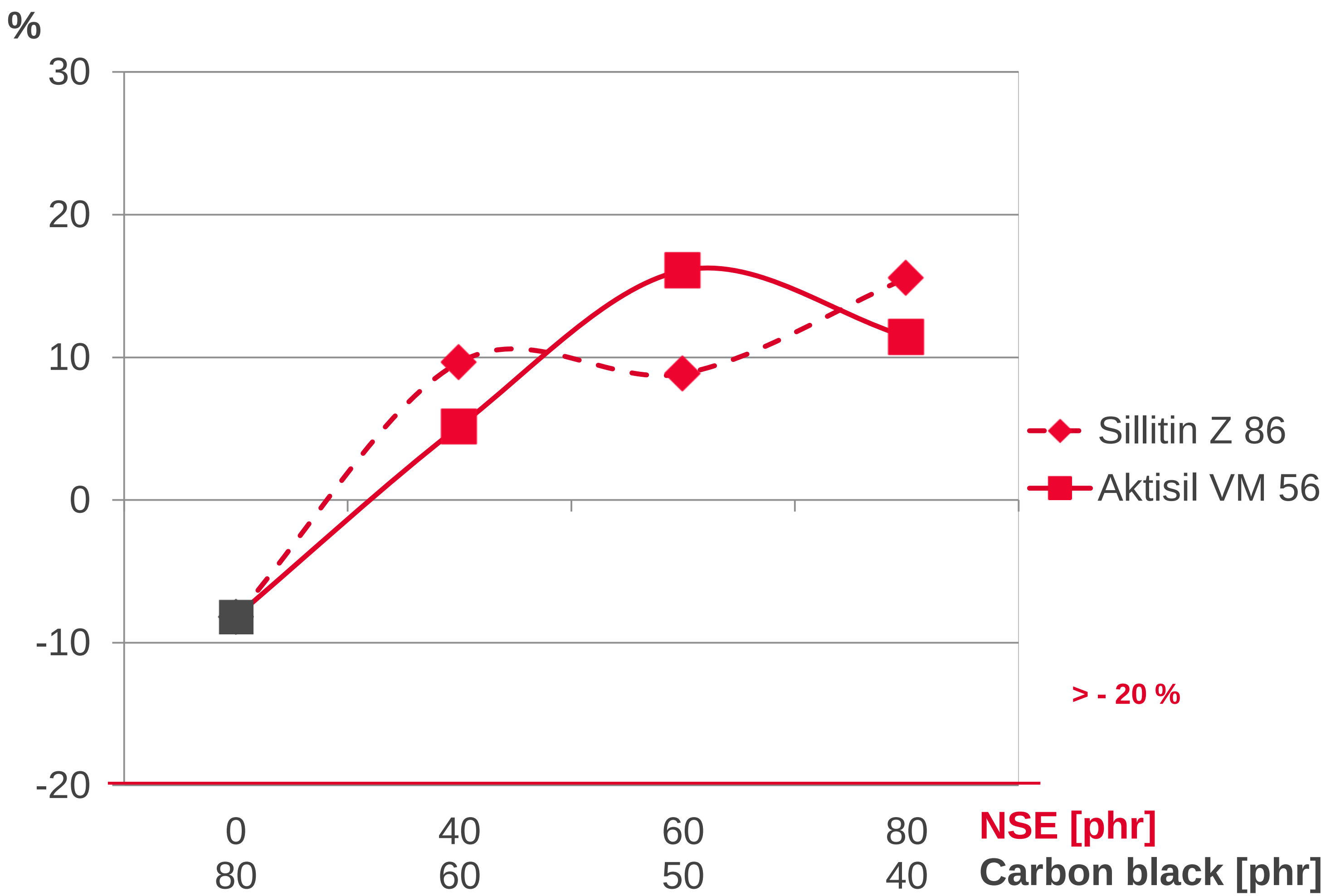
RESULTS

• Hot air aging

SUMMARY

APPENDIX

168 h / 125 °C



Hot Air Aging
Change of Elongation at Break

INTRODUCTION

EXPERIMENTAL

RESULTS
• Hot air aging

SUMMARY

APPENDIX

168 h / 125 °C

Cost Aspects

HOFFMANN MINERAL

INTRODUCTION

EXPERIMENTAL

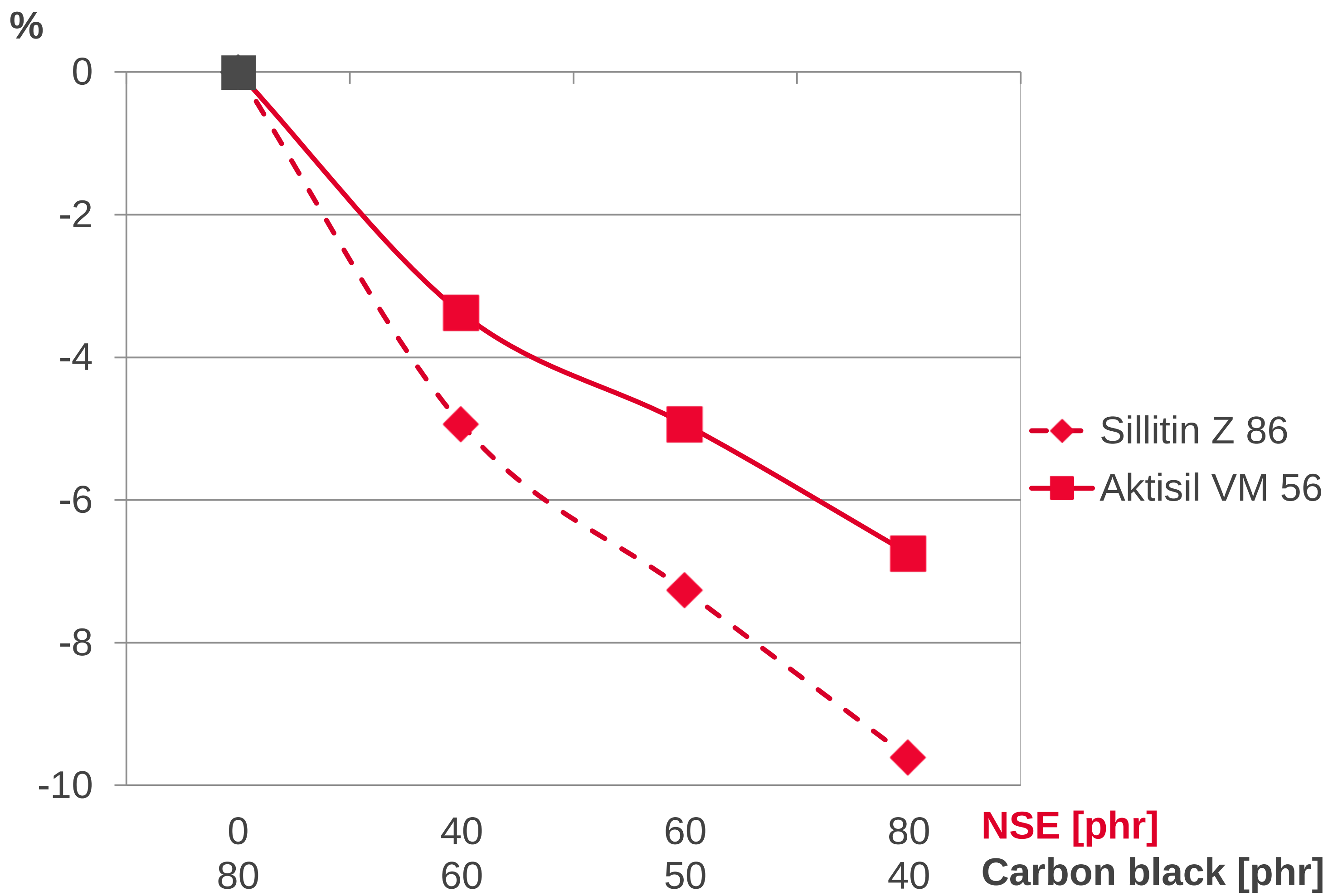
RESULTS

• Cost aspects

SUMMARY

APPENDIX

based on price per litre of mixture, Germany September 2021



Cost Aspects

INTRODUCTION

EXPERIMENTAL

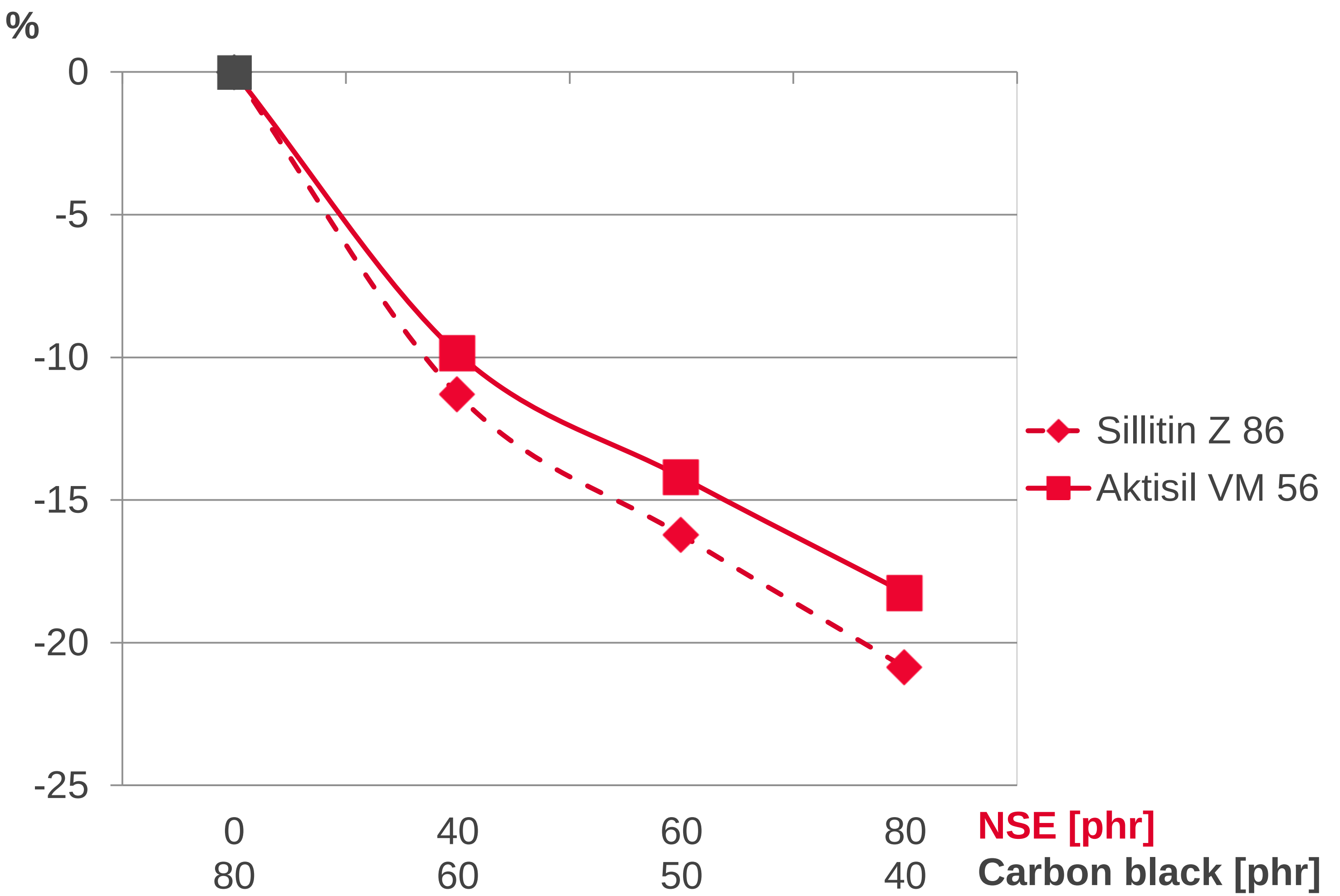
RESULTS

• Cost aspects

SUMMARY

APPENDIX

based on kg - price of the mixture, Germany September 2021



Summary 1

Aktisil VM 56

- Significant cost reduction
- Fulfils the requirement profile of the standard even up to 50 % carbon black replacement at a high level
- Lower Mooney viscosity and shorter conversion time t_{90}

Summary 2

Sillitin Z 86

- Even more significant cost reduction potential than with Aktisil VM 56
- Positive effect on tear resistance DIN ISO 34-1 trouser specimen
- Lower Mooney viscosity and shorter conversion time t_{90}
- 25 % carbon black replacement fulfils requirement profile
- 37 % carbon black replacement requires longer conversion time or post cure due to compression set requirement, change of elongation at break after hot air aging is borderline
- 50 % carbon black replacement, as the most cost-effective variant, however requires formulation optimization: For example, by replacing the antioxidant BPH with polymeric TMQ, both compression set and properties after hot air aging are significantly improved. Polymeric TMQ currently listed in Positive List part 2 of the German Federal Environment Agency (as of September 2021).

We supply material for good ideas!

HOFFMANN MINERAL GmbH
Muenchener Straße 75
DE-86633 Neuburg (Donau)

Phone: +49 8431 53-0
Internet: www.hoffmann-mineral.de
E-mail: info@hoffmann-mineral.com

Our applications engineering advice and the information contained in this memorandum are based on experience and are made to the best of our knowledge and belief, they must be regarded however as non-binding advice without guarantee. Working and employment conditions over which we have no control exclude any damage claim arising from the use of our data and recommendations. Furthermore we cannot assume any responsibility for patent infringements, which might result from the use of our information.

INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

APPENDIX

Table of Results

		Purex HS 45	Sillitin Z 86 + Purex HS 45			Aktisil VM 56 + Purex HS 45		
		80 phr	40 phr 60 phr	60 phr 50 phr	80 phr 40 phr	40 phr 60phr	60 phr 50 phr	80 phr 40 phr
Rheology								
Mooney viscosity, ML 1+4, 100 °C	MU	72	63	65	63	64	64	63
Mooney viscosity, ML 1+4, 120 °C	MU	53	47	50	47	47	48	48
Temperature curemeter	°C	180						
Rotorless curemeter M_{min}	Nm	0.10	0.08	0.09	0.08	0.08	0.08	0.08
Rotorless curemeter M_{max}	Nm	0.80	0.76	0.73	0.70	0.79	0.70	0.71
Rotorless curemeter M_{max}-M_{min}	Nm	0.71	0.68	0.65	0.62	0.71	0.62	0.63
Rotorless curemeter V_{max}	Nm/min.	0.22	0.24	0.24	0.24	0.24	0.23	0.25
Rotorless curemeter t_5	min.	0.47	0.48	0.47	0.46	0.48	0.47	0.45
Rotorless curemeter t_{90}	min.	7.0	6.6	6.3	6.1	6.6	6.4	6.2

Table of Results

		Purex HS 45	Sillitin Z 86 + Purex HS 45			Aktisil VM 56 + Purex HS 45		
		80 phr	40 phr 60 phr	60 phr 50 phr	80 phr 40 phr	40 phr 60phr	60 phr 50 phr	80 phr 40 phr
Mechanical properties after press cure								
Hardness	Sh. A	69	67	67	65	68	67	67
Tensile strength	MPa	16	14	12	11	16	14	13
Modulus 100 %	MPa	5.0	4.7	4.6	3.8	5.5	5.2	5.3
Elongation at break	%	222	262	279	330	228	223	223
Tear resistance Trouser tear	N/mm	3.5	5.1	5.9	7.5	4.1	4.7	4.3
Tear resistance Delft	N	38	42	43	42	41	43	42
Compression set, 72 h / 23 °C, 25 % defl.	%	8.7	9.8	11	12	9.5	11	10
Compression set, 72 h / -10 °C, 25 % defl.	%	20	21	24	27	20	24	26
Compression set, 24 h / 125 °C, 25 % defl. Press cure t_{90} + 10 %	%	12	17	20	23	16	16	17
Compression set, 24 h / 125 °C, 25 % defl. Press cure 12 min.	%	11	16	18	19	16	16	15
Compression set, 24 h / 125 °C, 25 % defl. Post cure 2 h / 125 °C	%	12	16	18	20	16	16	16

Table of Results

		Purex HS 45	Sillitin Z 86 + Purex HS 45			Aktisil VM 56 + Purex HS 45		
		80 phr	40 phr 60 phr	60 phr 50 phr	80 phr 40 phr	40 phr 60phr	60 phr 50 phr	80 phr 40 phr
Mechanical properties after immersion in distilled water 168 h / 95 °C								
Hardness	Sh. A	70	67	66	64	69	68	67
Tensile strength	MPa	17	15	13	11	16	14	14
Elongation at break	%	208	289	325	391	239	247	250
Δ Hardness	Sh. A	+1	0	-1	-1	+1	+1	0
Δ Tensile strength	%	+3.8	+6.5	+9.5	+4.6	-2.3	+2.4	+2.0
Δ Elongation at break	%	-6.4	+10	+16	+18	+5.0	+11	+12
Change of volume	%	+1.1	+1.1	+1.6	+1.9	+1.6	+1.3	+1.7
Mechanical properties after hot air aging 168 h / 125 °C								
Hardness	Sh. A	75	73	74	72	73	72	73
Tensile strength	MPa	17	15	14	13	15	16	15
Elongation at break	%	169	194	212	215	170	188	176
Δ Hardness	Sh. A	+6	+6	+7	+7	+5	+5	+6
Δ Tensile strength	%	-8.2	+9.7	+8.9	+16	-5.2	-16	-11
Δ Elongation at break	%	-20	-27	-29	-39	-24	-18	-25