Air intake hose EPDM -

Replacement of carbon black N990

with Neuburg Siliceous Earth

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1 Introduction

For the present study on the example of a highly loaded molding formulation, an automotive air intake hose was chosen (Table 1).

In order to arrive at a loading as high as possible along with acceptable properties, traditionally the compounds are partly filled with N990 (MT) carbon black.

Following the prices of fossil raw materials, carbon black prices also suffer from large fluctuations. The long time trend, however, clearly goes upwards.
The price of mineral fillers is only marginally affected by fossil raw materials; there rather is a very slow increase over the long term without any fluctuations.

The rather frequent supply problems with carbon black N990 can be avoided by working with siliceous earth fillers.

Hoffmann Mineral assures reliable short delivery times and a high degree of availability of the Neuburg Siliceous Earth product portfolio.

2 Objectives

Carbon black N990 should be replaced with Neuburg Siliceous Earth fillers in order to reduce costs while maintaining the technical property profile.

3 Experimental

3.1 Compound Formulations

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Base Formulation

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		phr
Keltan 5469	EPDM crystalline + 100 phr mineral oil	200.0
Zinkoxyd aktiv	zinc oxide acitive	5.0
Stearic Acid	processing aid	1.0
Corax N550	FEF carbon black	115.0
Carbon black N990	**MT carbon black**	**150.0**
Omya BSH	whiting surface treated	50.0
Triethanolamine 98 %	filler deactivator	0.9
Sunthene 4240	naphthenic oil, plasticizer	25.0
Rhenogran DPG-80	accelerator	0.5
Rhenogran TP-50	accelerator	2.0
Rhenogran ZBEC-70	accelerator	2.0
Rhenogran MBTS-80	accelerator	1.3
Rhenogran CLD-80	accelerator	1.0
Vulkalent E/C	cure retarder	0.5
Rhenogran TBBS-80	accelerator	0.63
Rhenogran S-80	sulfur, curing agent	0.75
Summary		**555.58**

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Table 1

Keltan 5469	EPDM, crystalline + 100 phr mineral oil
Zinkoxyd aktiv	zinc oxide active
Stearic acid	processing aid
Corax N550	FEF carbon black
Carbon black N990	MT carbon black
Omya BSH	whiting, surface treated
Triethanol amine 98 %	filler deactivator
Sunthene 4240	naphthenic oil, plasticizer
Rhenogran DPG-80	diphenyl-guanidine, 80 % polymer bound
Rhenogran TP-50	zinc-dialkyl-dithiophosphate, 50 % polymer bound
Rhenogran ZBEC-70	zinc-dibenzyl-dithiocarbamate, 70 % polymer bound
Rhenogran MBTS-80	dibenzo-thiazyl-disulfide, 80 % polymer bound
Rhenogran CLD-80	dithio-caprolactam, 80 % polymer bound
Vulkalent E/C	n-(trichloromethylthio)-benzenesulfonanilide
Rhenogran TBBS-80	n-tert-butyl-2-benzothiazylsulfenamide, 80 % polymer-bound
Rhenogran S-80	sulfur, 80 % polymerbound

The compound can be highly loaded with carbon black N990 while maintaining a good property profile, which behavior resembles the siliceous earth products. The total carbon black loading arrives at 265 phr and is split between 115 parts of Corax N550 and 115 phr Carbon black N990. In addition, the compound contains 50 phr of whiting. The total plasticizer content comes up to 125 phr (100 phr extender oil in the rubber, and 25 phr added in liquid form). In total, this leads to a hardness of 60 Shore A.

Design of Experiments

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Dosage in phr for equivalent hardness

Control	Calcined Neuburg Siliceous Earth		Neuburg Siliceous Earth	
Carbon black N990	Aktifit AM	Silfit Z 91	Sillitin Z 86	Sillitin N 75*
150	180			

*The tests were carried out with Sillitin N 82. This product is no longer available.
Recommended: Sillitin N 75.

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Table 2

The carbon black N990 was replaced by Aktifit AM, Silfit Z 91, Sillitin Z 86 resp. Sillitin N 75 at a ratio of 1:1,2. The siliceous earth fillers had to be added at a slightly higher amount in order to keep the hardness at the same level *(Tab. 2).*

Fillers, Characteristics

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		Carbon black N990	Aktifit AM	Silfit Z 91	Sillitin Z 86	Sillitin N 75
Density	[g/cm³]	1,8	2,6	2,6	2,6	2,6
Particle size d_{50}	[µm]		2,0	2,0	1,9	3,0
Particle size d_{97}	[µm]		10,0	10,0	9,0	16,0
Sieve residue > 40 µm	[mg/kg]		10	10	20	25
Sieve residue 45 µm (Sieve Nr. 325)	ppm	18				
Oil absorption	[g/100g]		55	55	55	45
DBP absorption	[ml/100 g]	38				
Specific surface area BET	[m²/g]		7	8	12	12
CTAB Surface area	[m²/g]	7				
Funktionalization		none	Amino	none	none	none

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Table 3

The typical properties of carbon black N990 do not show big differences vs. the siliceous earth; one of the biggest differences is the density of 1,8 vs. 2,6 g/cm³ *(Tab. 3).*

3.2 Mixing of Compounds and Vulcanization

Preparation and Curing of the Compound

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- **Mixing**

  Open mill Ø 150 x 300 mm

  Batch volume: approx. 600 cm³

  Temperature: 50 °C

  Mixing time: approx. 20 min.

- **Curing**

  Press: 180 °C

  2 mm sheet: 5 min.

  Compression set and rebound specimen: 10 min.

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Fig. 1

All compounds were mixed on a laboratory mill (Schwabenthan Polymix 150 L) at 20 rpm and a batch size of about 600 cm^3. The mixing times were adjusted according to the fillers, and registered.

The compounds were cured in an electrically heated press at 180 °C. The 2 mm sheets were cured for 5 minutes, and the rebound and compression set samples for 10 minutes *(Fig. 1)*.

4 Results

4.1 Mooney Viscosity und Mooney Scorch Time

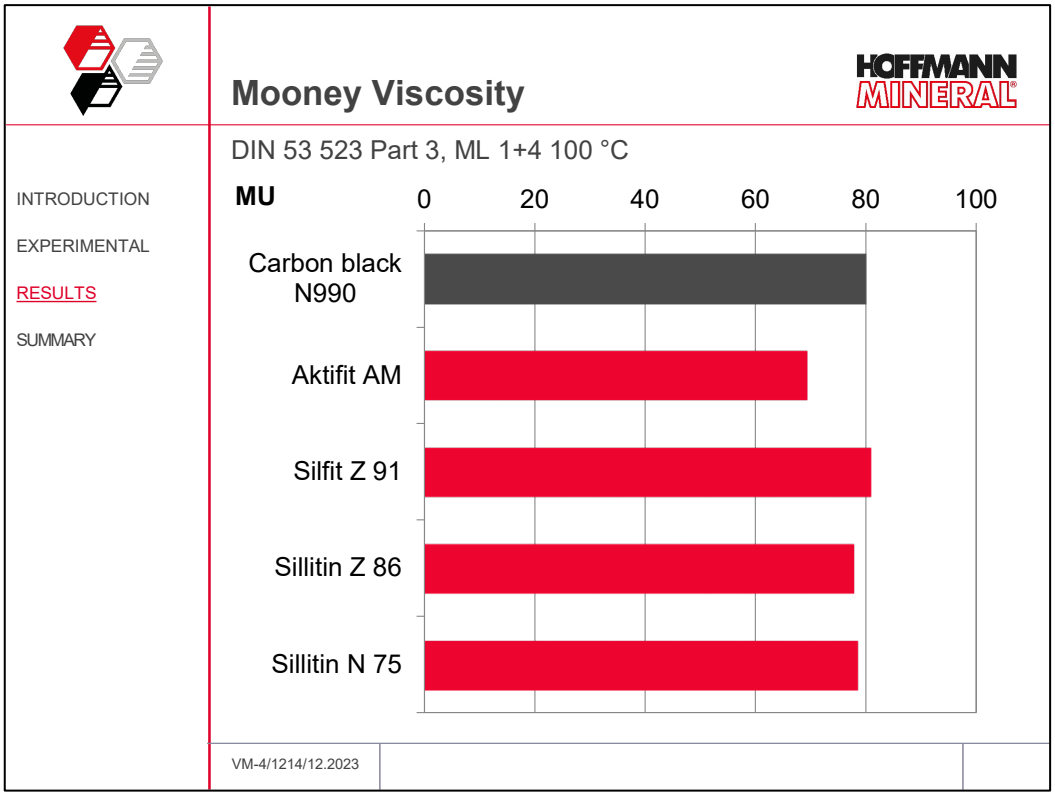

Fig.2

Replacing carbon black N990 with the siliceous earth grades mentioned, except for Aktifit AM the Mooney viscosity hardly changes at all. With Aktifit AM, a somewhat lower Mooney viscosity was measured *(Fig. 2).*

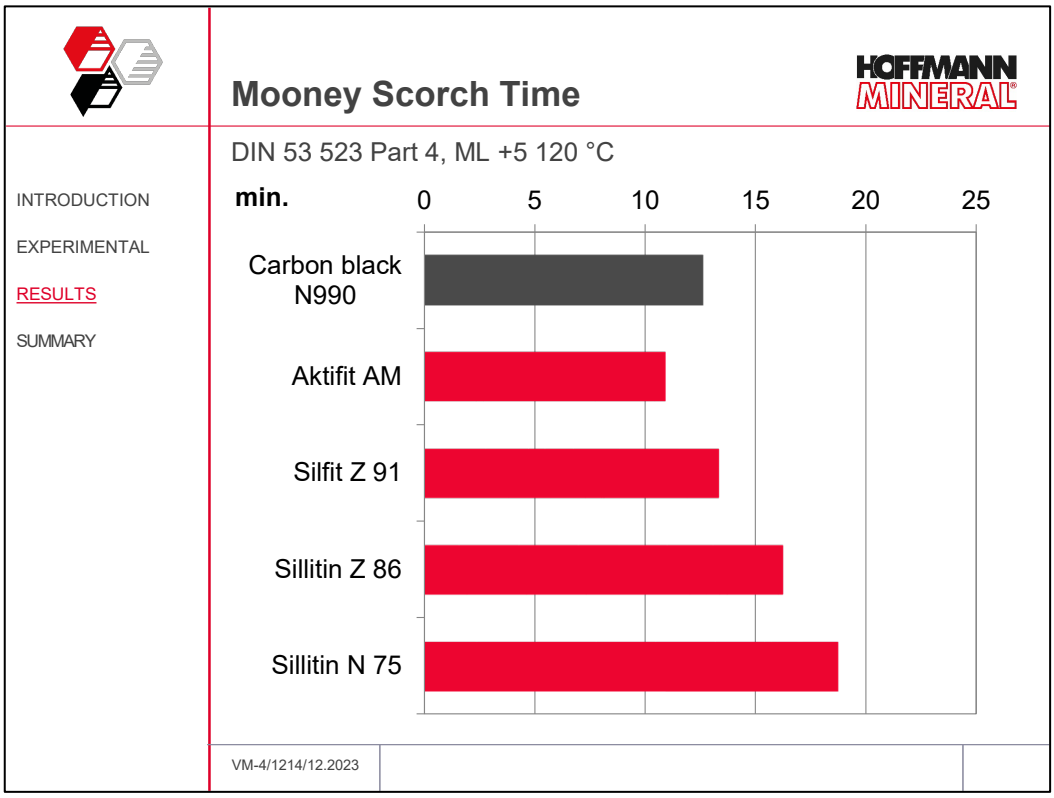

Fig. 3

Mooney scorch time, as an index of the initial cure properties during processing, was found somewhat longer with Sillitin Z 86 and Sillitin N 75. In other words, with these fillers the risk of an onset of cure during processing is lower. With Silfit Z 91, there is no change, while the scorch time with Aktifit AM tends to be slightly shorter *(Fig. 3)*.

4.2 Cure Properties

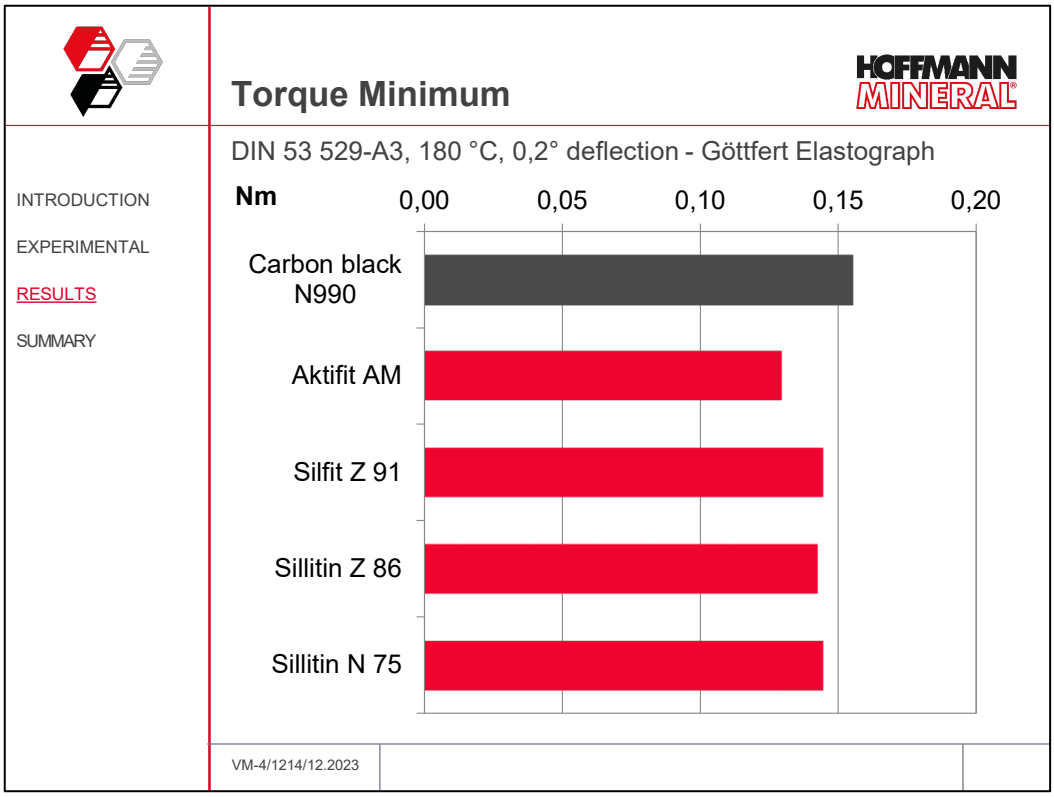

Fig. 4

If carbon black N990 is replaced by Neuburg Siliceous Earth, the torque minimum in the Vulkameter test will be slightly lower *(Fig. 4)*.

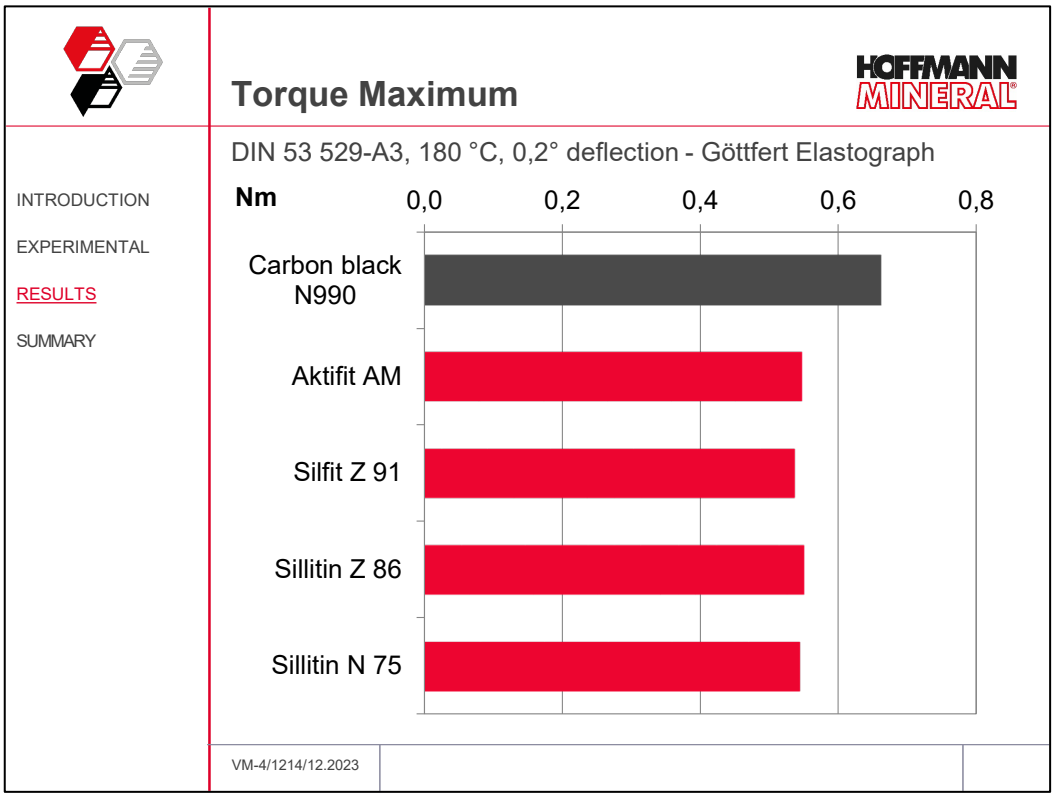

Fig. 5

Likewise, the torque maximum comes out somewhat lower by this replacement *(Fig. 5).*

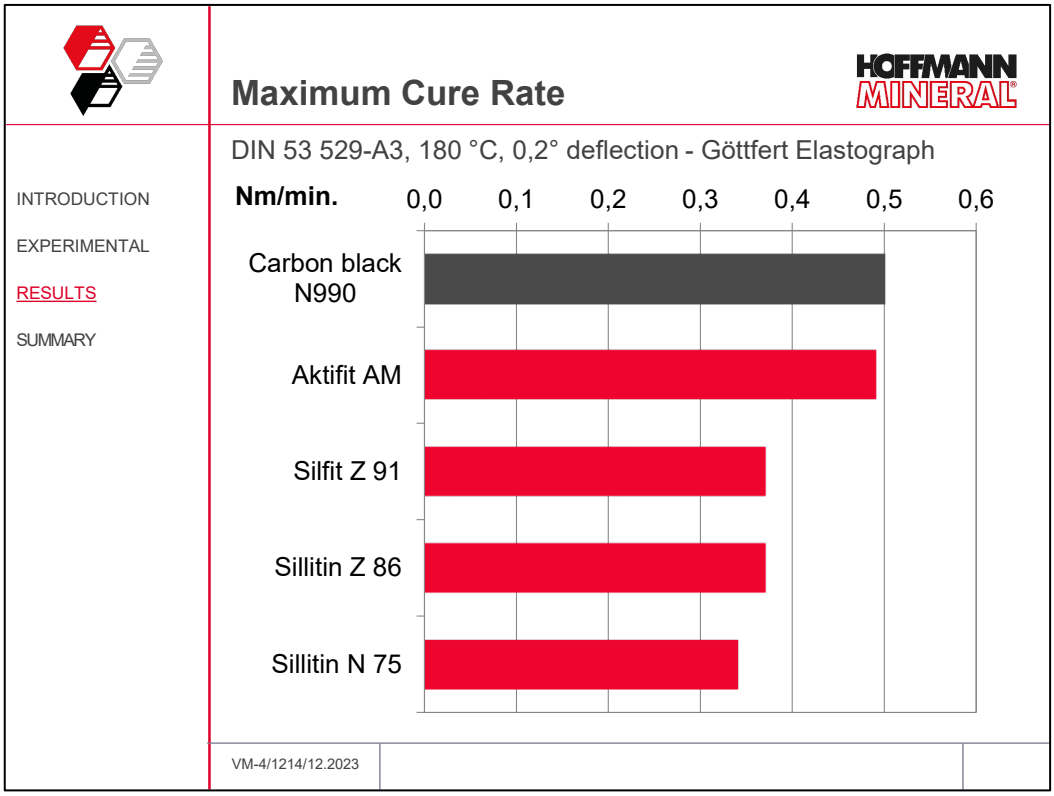

Fig. 6

The compound with Aktifit AM shows the same cure rate as the straight carbon black formulation. With Silfit and Sillitin grades, the peak of the cure rate is reduced *(Fig. 6).*

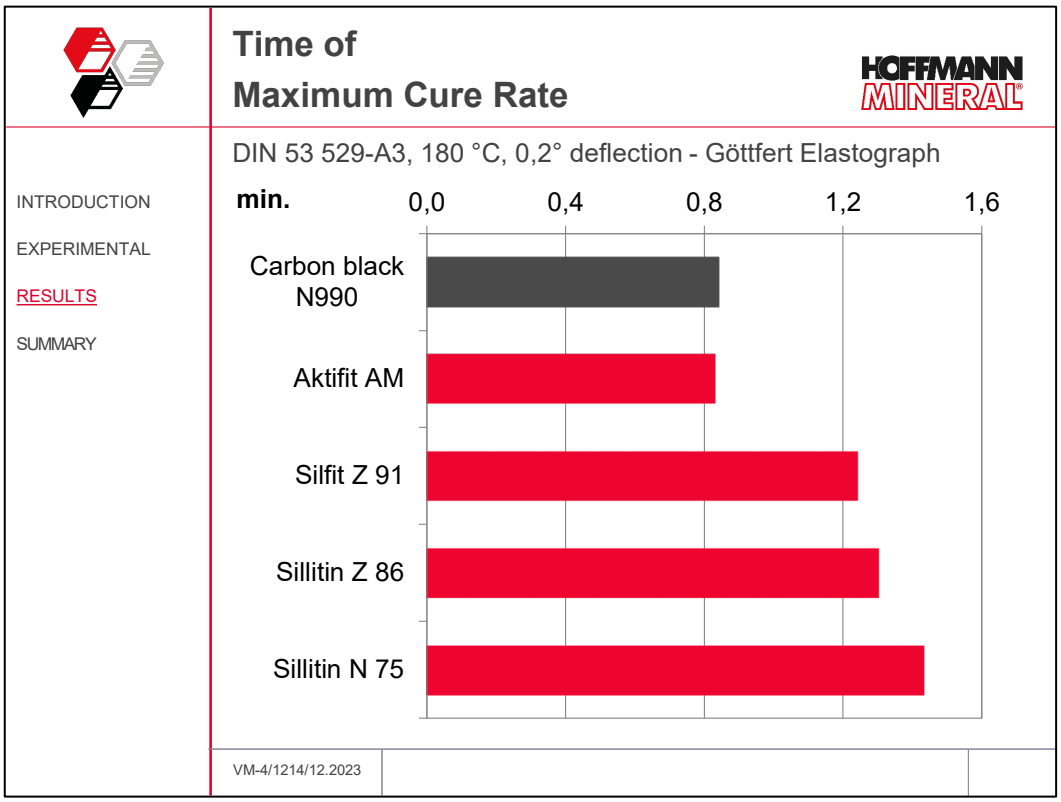

Fig. 7

Likewise, the time of the maximum cure rate with Aktifit AM is the same as in the straight carbon black compound, while Silfit and Sillitin grades take markedly longer *(Fig. 7).*

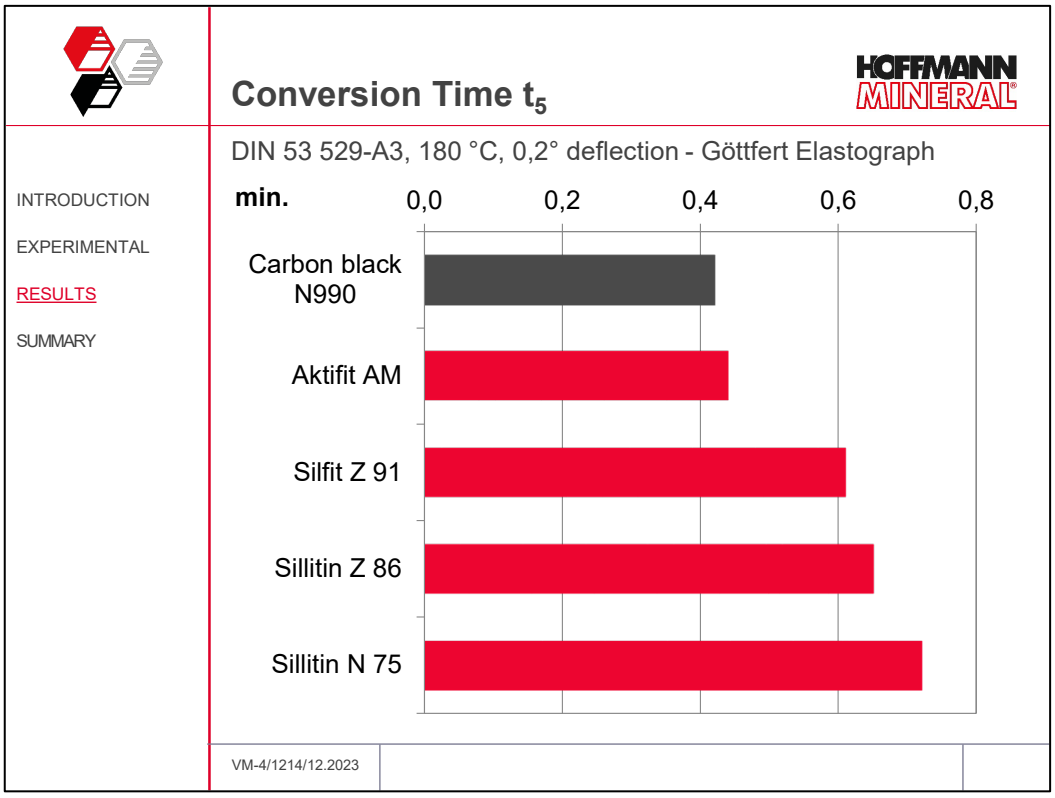

Fig. 8

With Aktifit AM, also the conversion time t_5 is at level with the carbon black N990 containing compound. The other siliceous earth grades show somewhat longer t_5 times *(Fig. 8).*

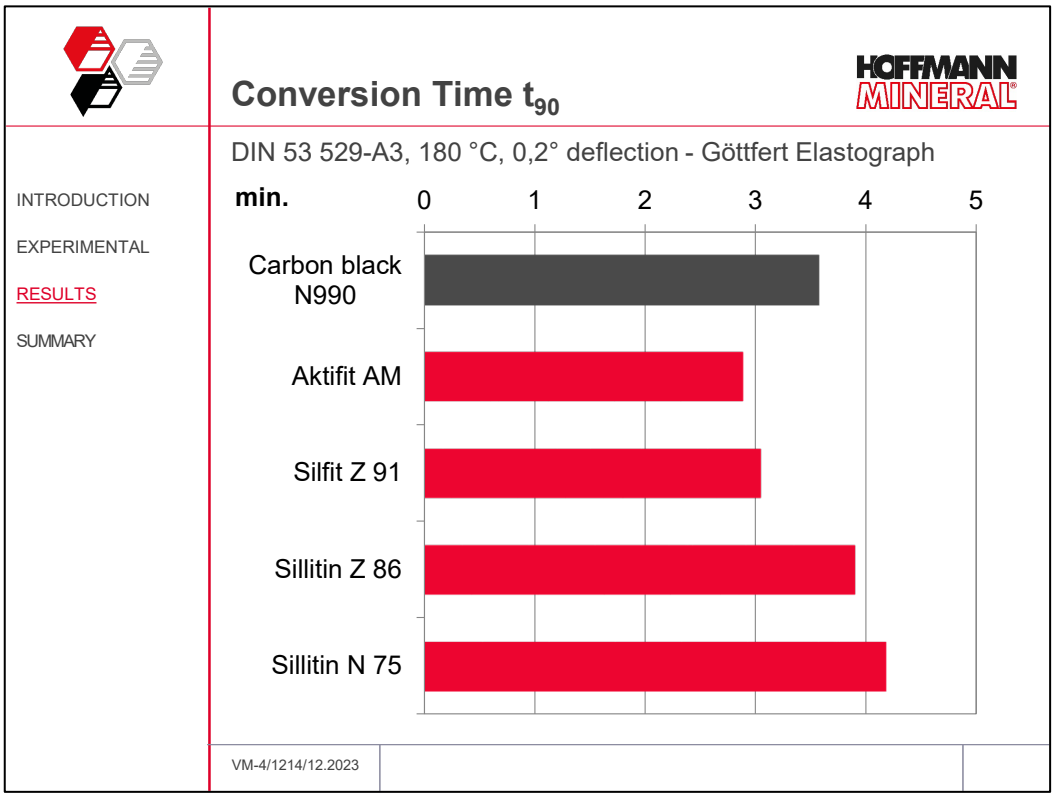

Fig. 9

The conversion time t_{90}, an index for the required time to full cure, comes out slightly shorter with the calcined siliceous earth grades, which should allow a shortening of the cycle times. By contrast, the standard grades Sillitin Z 86 and Sillitin N 75 take marginally more time compared with the straight carbon black compound *(Fig. 9).*

4.3 Mechanical Properties

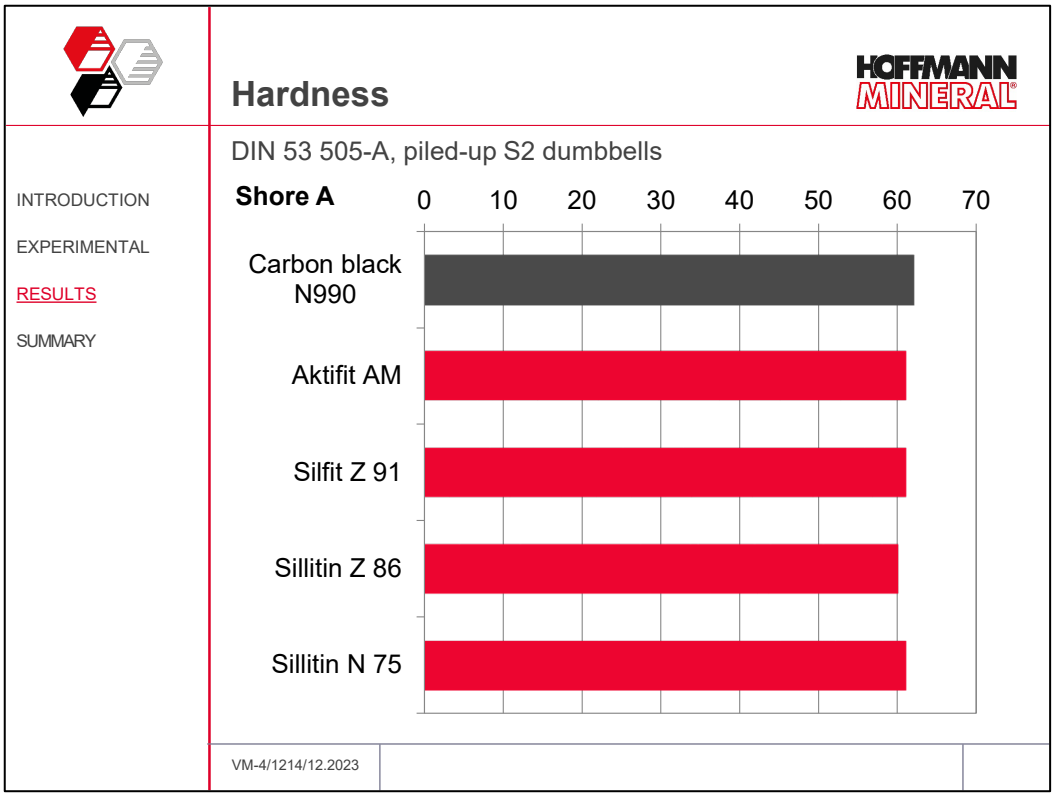

Fig. 10

The different formulations were all adjusted to a hardness of close to 60 Shore A (Fig. *10).*

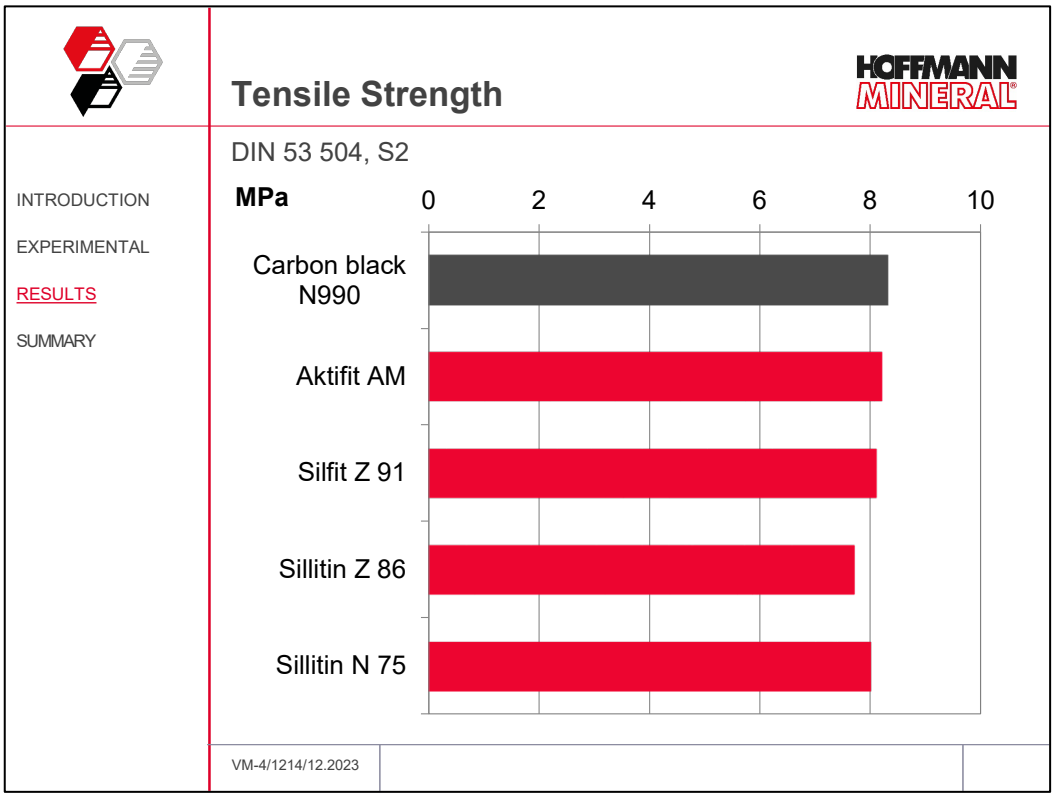

Fig. 11

Tensile strength does practically not change by replacing carbon black N990 with the siliceous earth grades *(Fig. 11).*

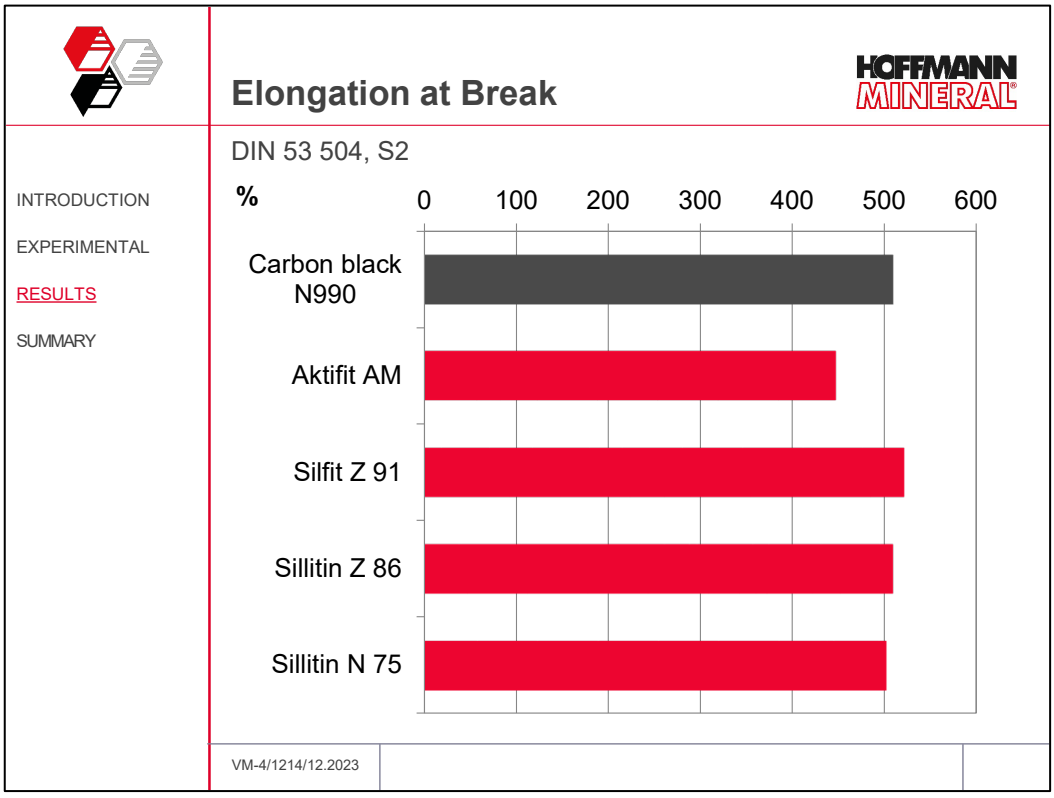

Fig. 12

The same is true for the elongation at break, with the exception of Aktifit AM which, as a result of the surface treatment, gives rise to a somewhat lower result, while the other grades do not show any significant differences versus the control compound with only carbon black *(Fig. 12).*

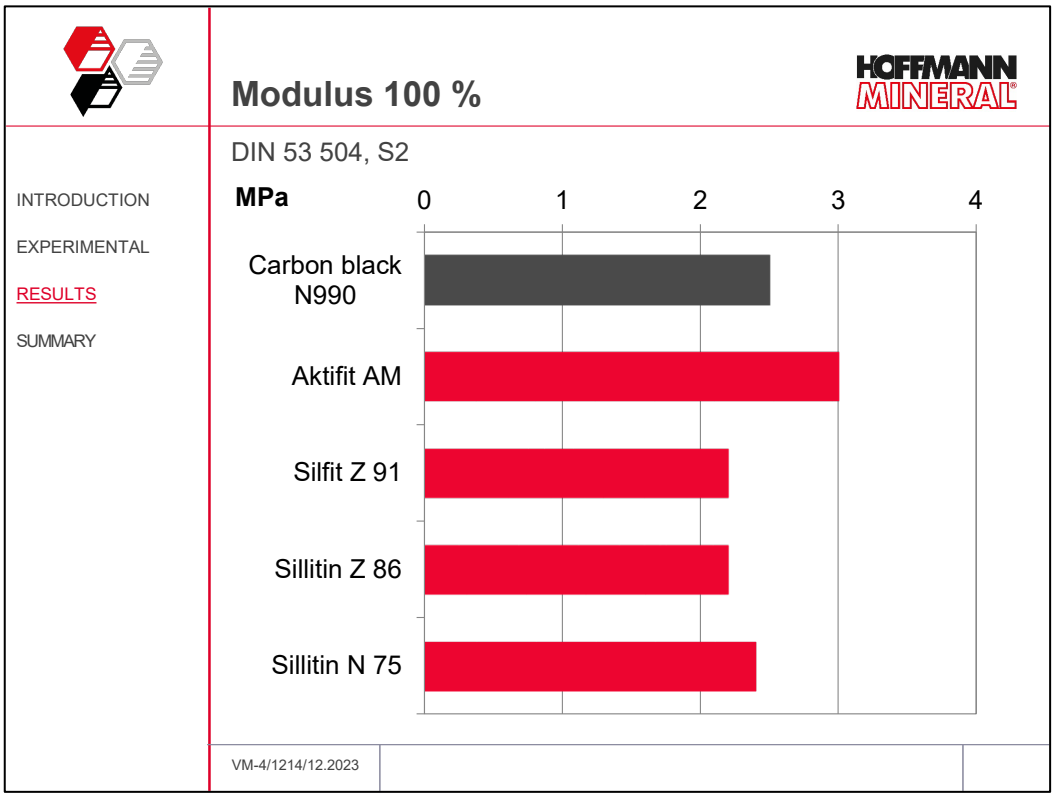

Fig. 13

Similarly, the tensile modulus with Aktifit AM comes out somewhat higher than with carbon black N990. The untreated siliceous earth grades result in slightly lower modulus levels *(Fig. 13).*

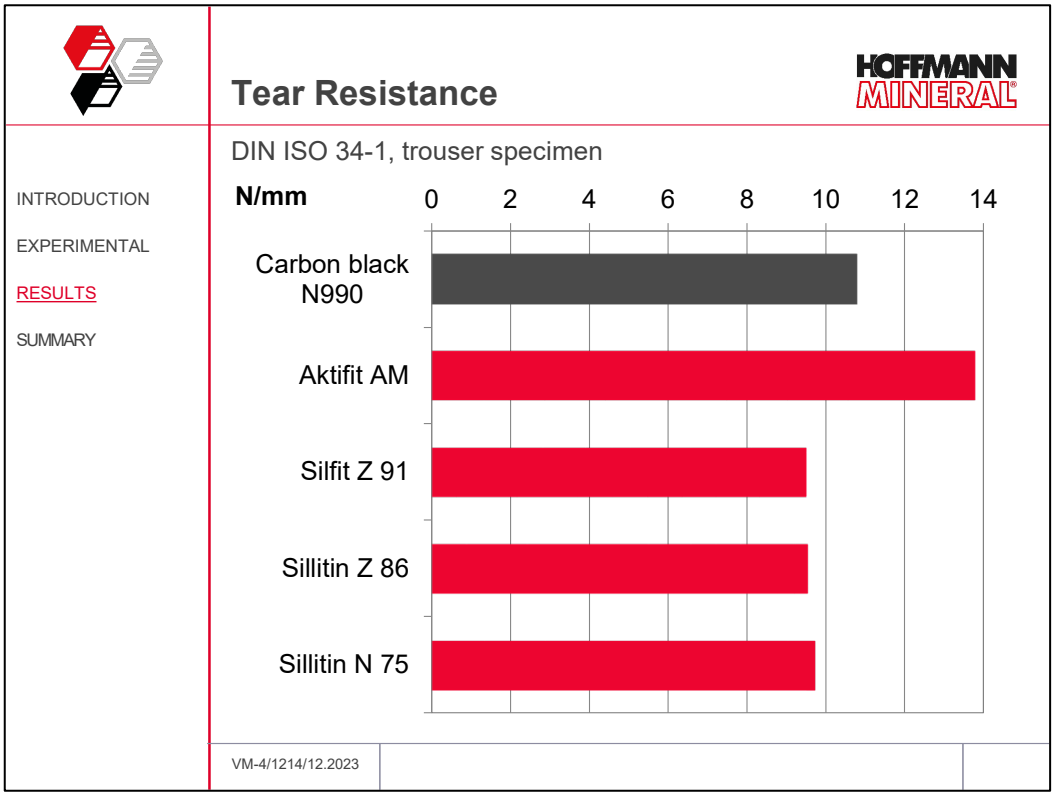

Fig. 14

The tear resistance of the trouser sample is higher with Aktifit AM than with carbon black N990 too. The untreated siliceous earth grades results in slightly lower tear resistance *(Fig. 14).*

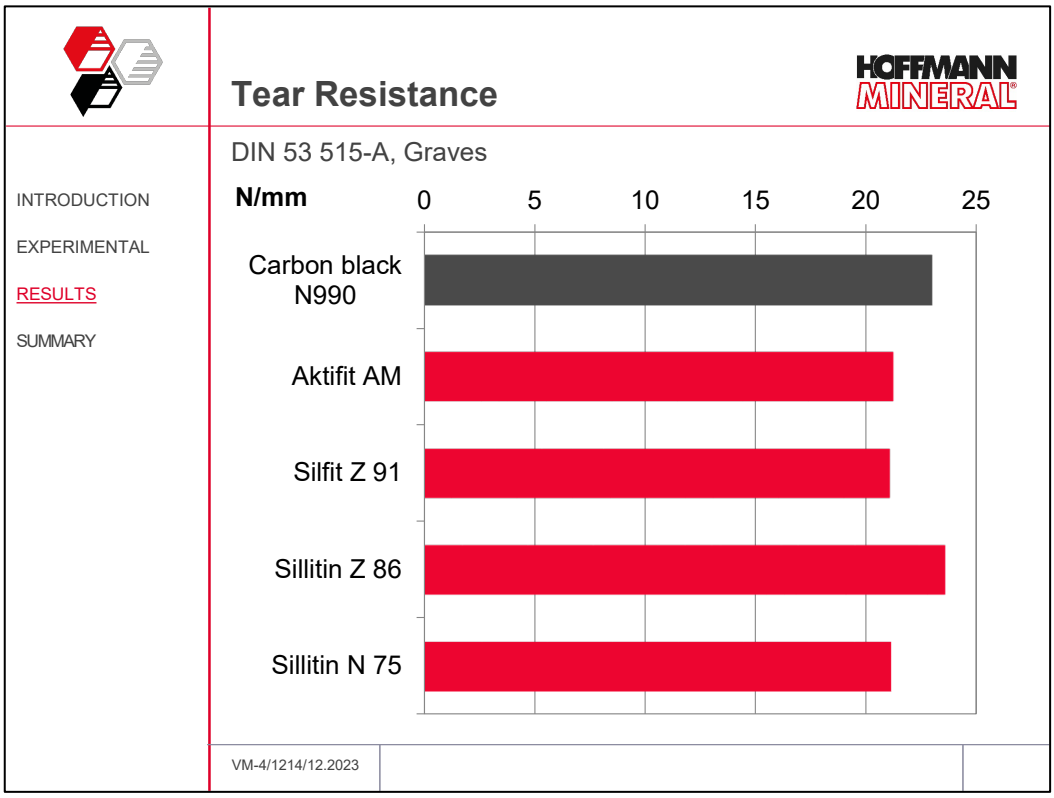

Fig. 15

With Sillitin Z 86 the result is slightly better. The other siliceous earth grades tend to have a slightly lower tear resistance using the Graves method *(Fig. 15).*

Depending on the method used the results are slightly different, however the level can be rated as similar.

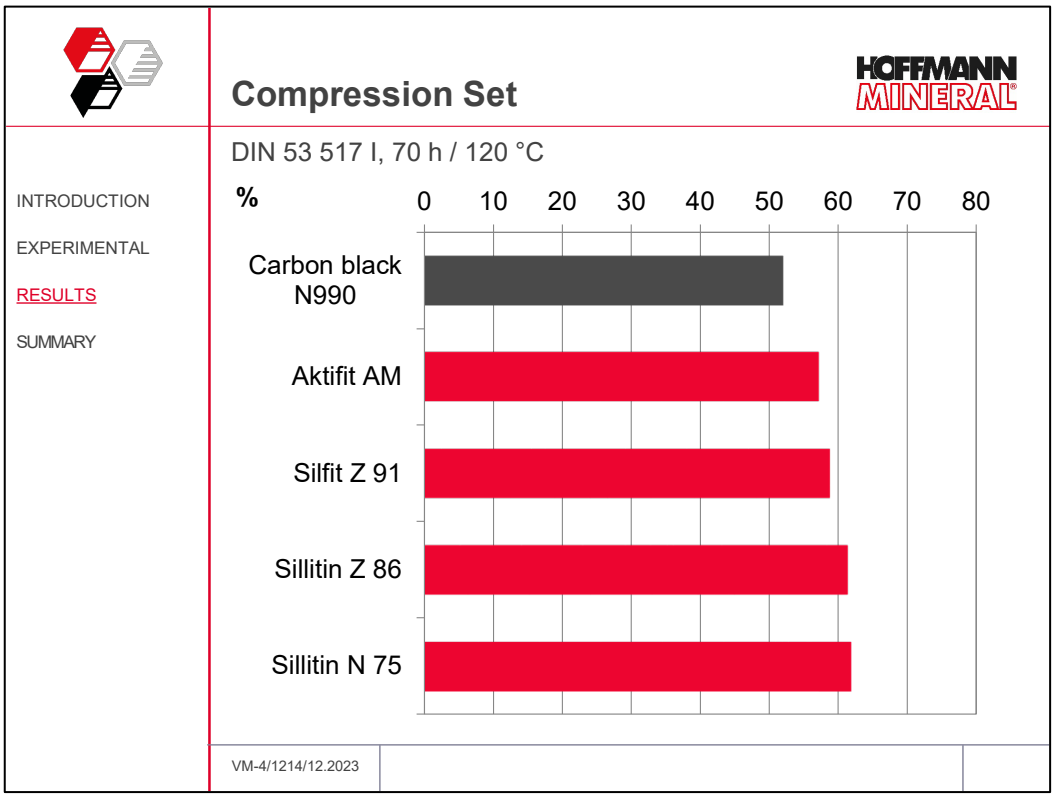

Fig. 16

Compression set only plays a minor role in the specifications for air intake hoses. As a result, the slight increase in tests at 120 °C looks acceptable, with Aktifit AM again coming up with the optimum result *(Fig. 16).*

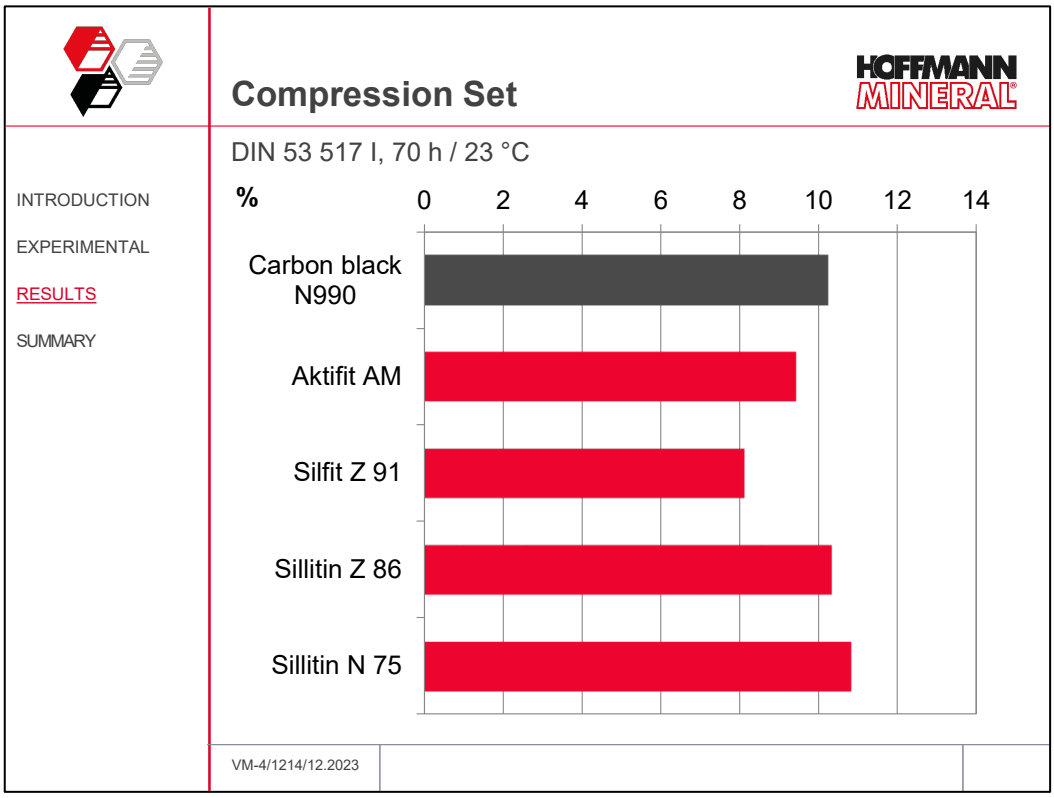

Fig. 17
By comparison, the compression set at 23 °C with Aktifit AM and Silfit Z 91 shows up even better than with carbon black N990. With Sillitin grades, the results hardly differ from the straight carbon black compound *(Fig. 17)*.

4.4 Immersion in Oil IRM 903

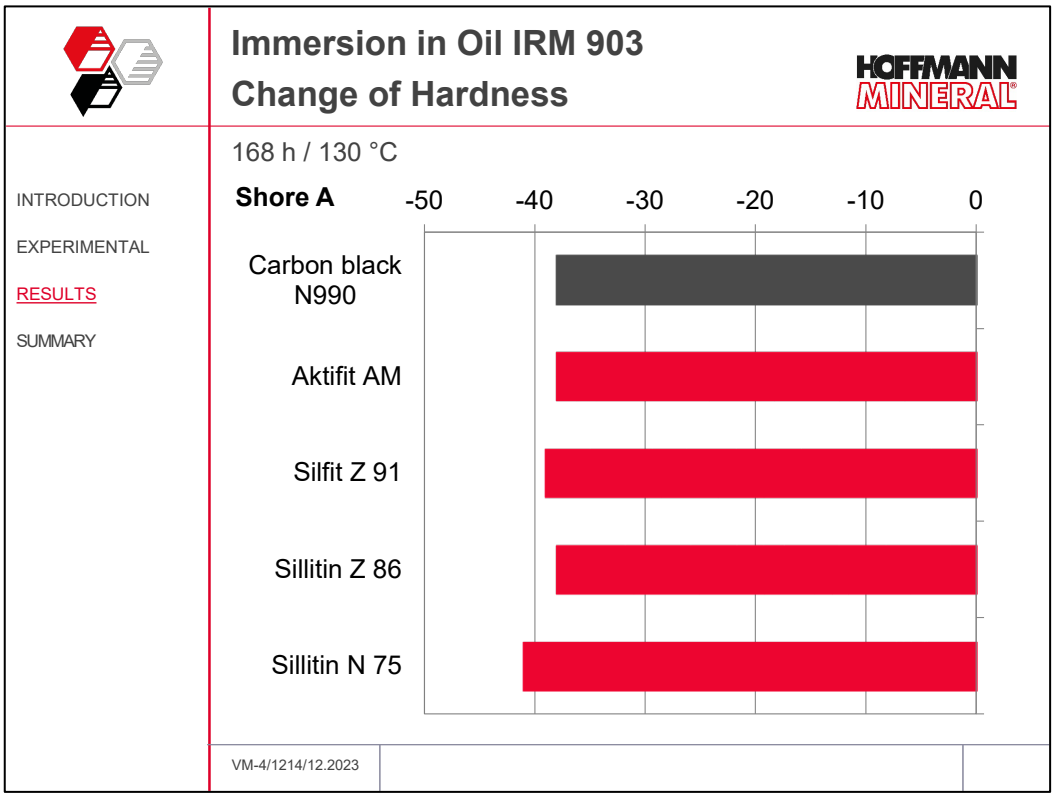

Fig. 18
Further tests evaluated the resistance against the aggressive standard oil IRM 903, i.e. under demanding conditions for an EPDM compound. Hardness decreased for all compounds to an equal extent, only Sillitin N 75 showes a somewhat bigger change *(Fig. 18)*.

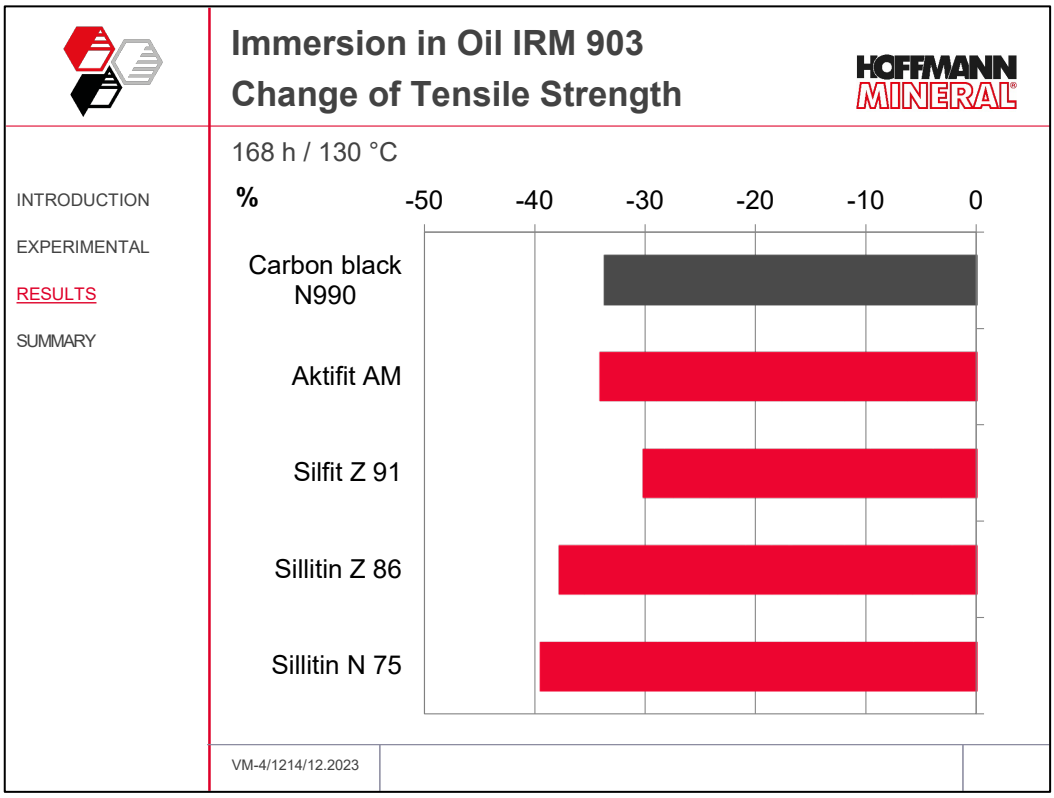

Fig. 19

The change of tensile strength is lowest with Silfit Z 91, even lower compared with straight carbon black. By contrast, the changes with Sillitin come out slightly higher *(Fig. 19).*

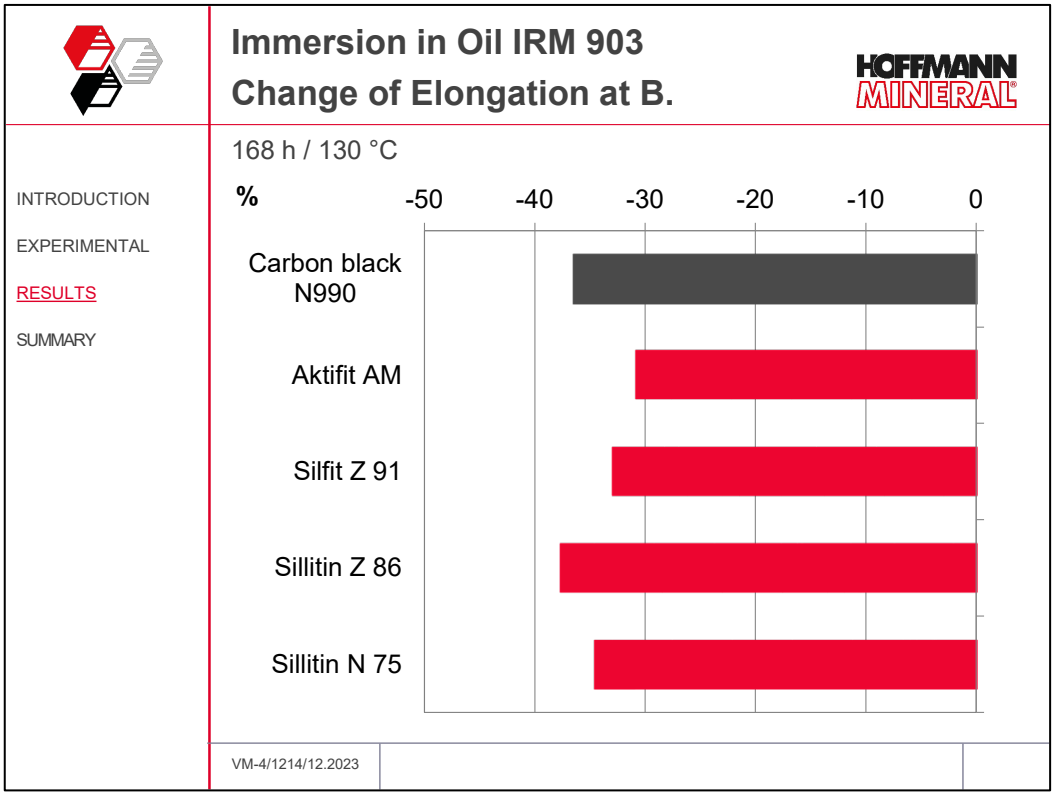

Fig. 20

Elongation at break changes with Aktifit AM and Silfit Z 91 slightly less than with the MT black, while the Sillitins reach the level of the control compound *(Fig. 20).*

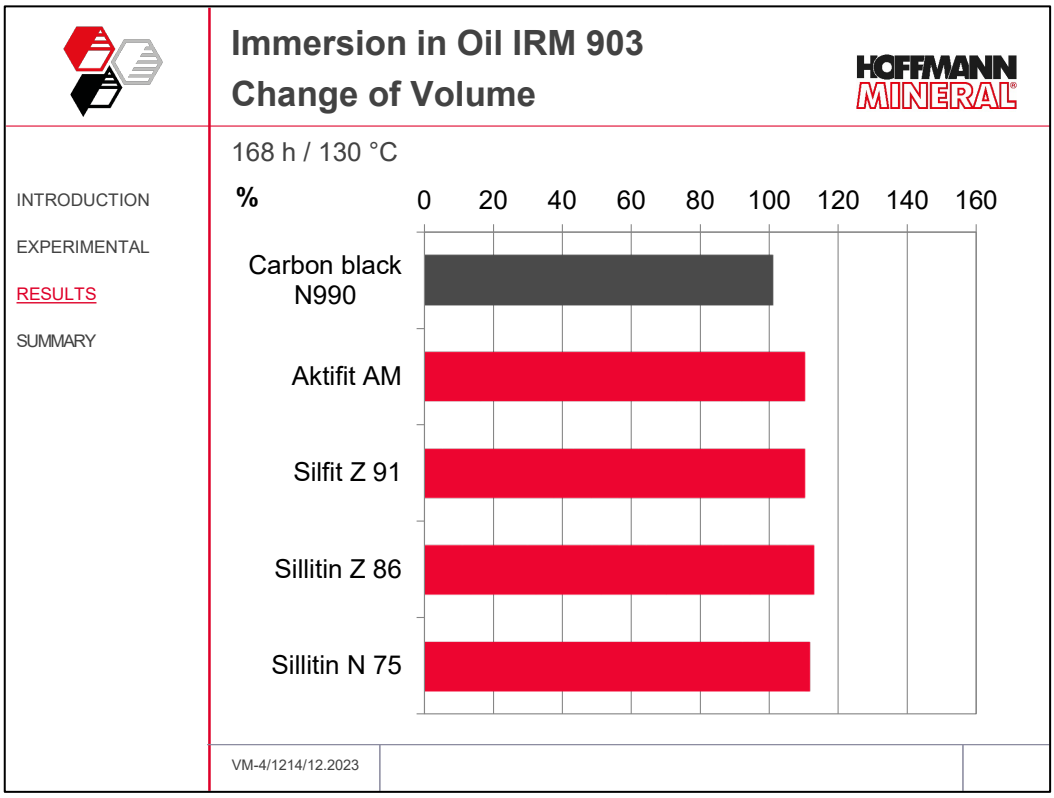

Fig. 21

Volume changes (swelling) with the siliceous earth grades are only marginally higher than with carbon black N990, without showing significant differences between the individual grades *(Fig. 21).*

4.5 Hot Air Aging

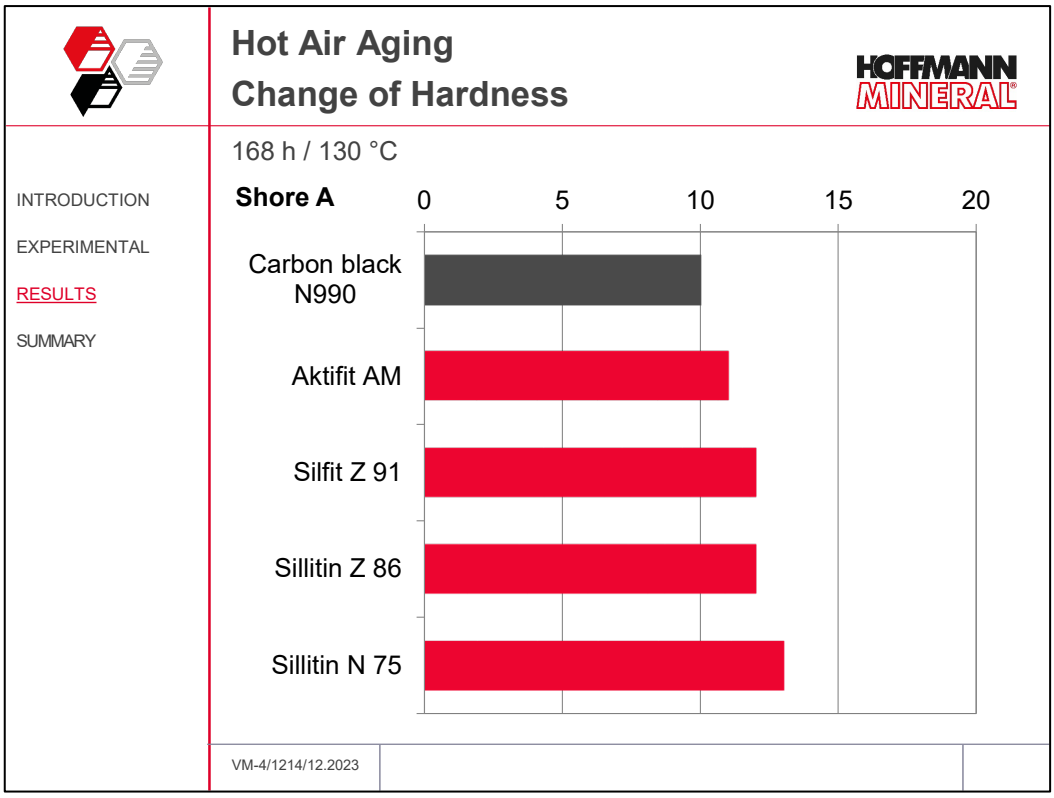

Fig. 22

The hardness change during hot air aging is slightly more pronounced with the siliceous earth grades compared with the straight carbon black compound *(Fig. 22).*

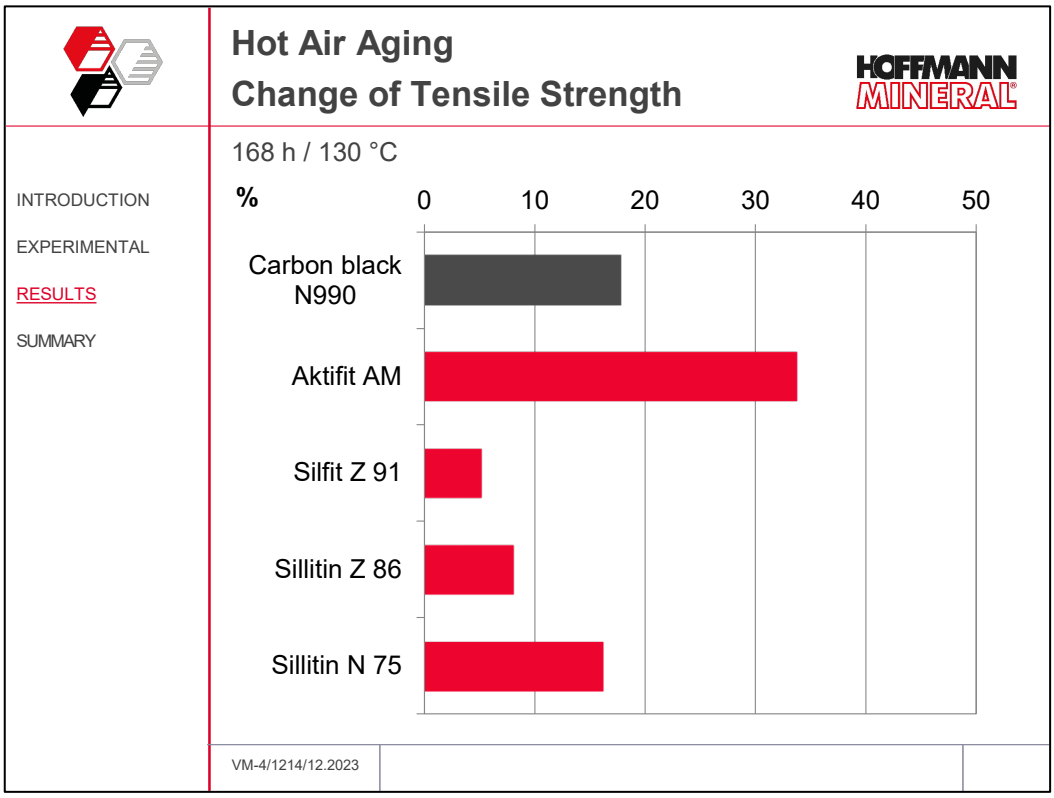

Fig. 23

The compound with Aktifit AM shows a higher change of the tensile strength compared with the straight carbon black control compound, while the results with Silfit Z 91 and Sillitin Z 86 come out somewhat lower, and Sillitin N 75 remains at the level of the control compound *(Fig. 23).*

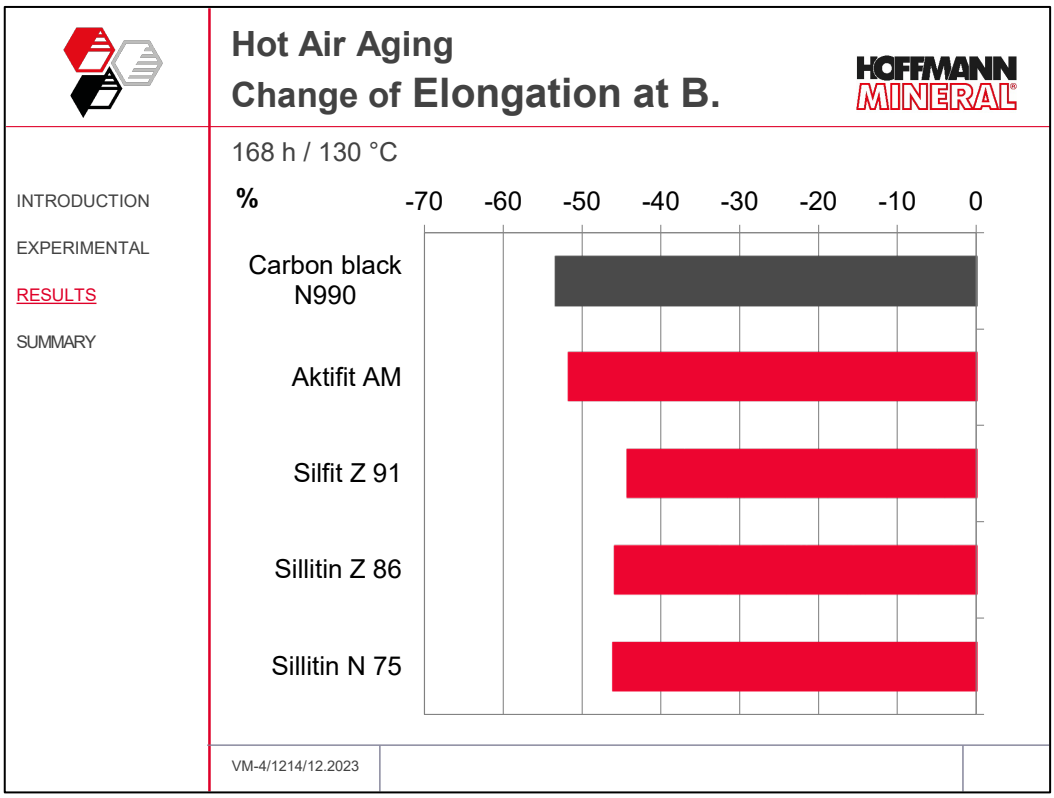

Fig. 24

Aktifit AM and carbon black N990 give rise to an equal change of elongation at break, but Silfit Z 91, Sillitin Z 86 and Sillitin N 75 come out somewhat lower *(Fig. 24).*

4.6 Cost Aspects

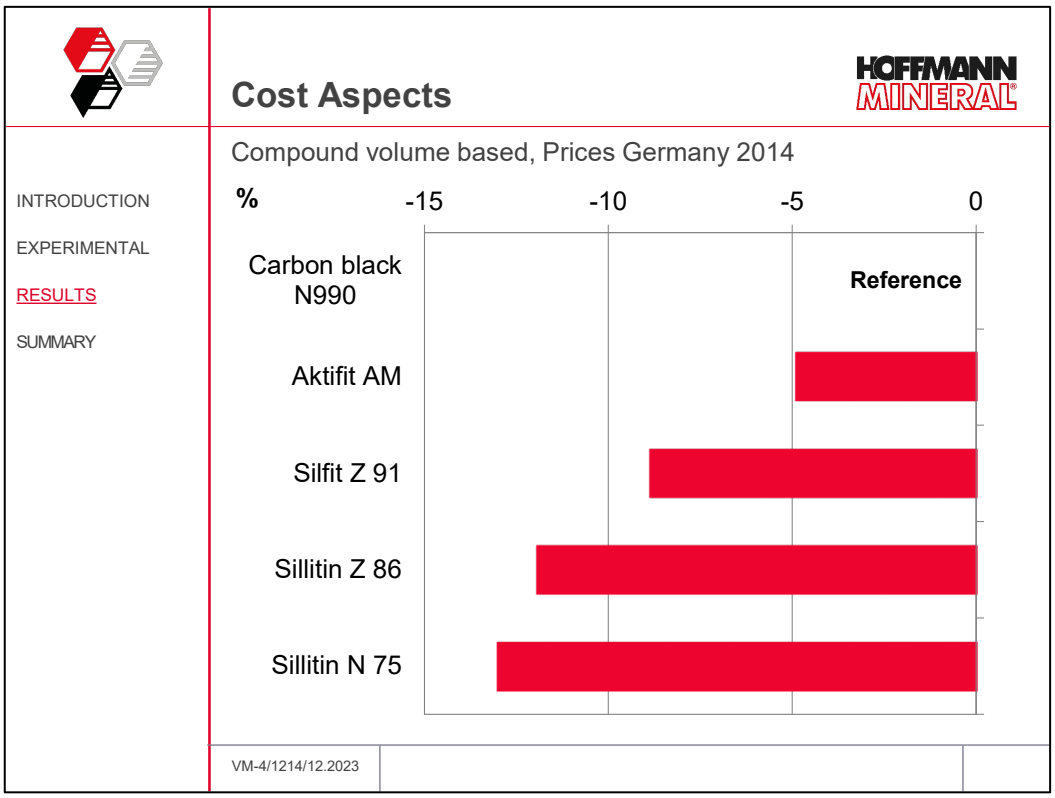

Fig. 25

With the use of siliceous earth products, a marked reduction of the compound raw material costs can be realized. This is all the more of interest, as the compound cost as indicated already has taken into account the different densities compared to working with carbon black N990.

Therefore, it is possible, despite the relative higher price due to the surface treatment in comparison with the untreated grades, to arrive with Aktifit AM at a cost reduction of 5 %.

Silfit Z 91, and even more pronounced the two Sillitin grades, even allow a cost reduction of 9 % and more *(Fig. 25).*

5 Summary

- The replacement of Carbon Black N990 in the EPDM air intake compound by Neuburg Siliceous Earth fillers leads to a very similar property profile.
- Aktifit AM allows to exactly reproduce the scorch behavior of Carbon Black N990, and in addition to obtain a shorter time to full cure along with a cost reduction of 5 %.
- Silfit Z 91 as well as Sillitin Z 86 and Sillitin N 75 lead to a cost reduction of 9 % and more.
- Aktifit AM and Silfit Z 91 impress by good processing properties and the absence of mold fouling.
- The Neuburg Siliceous Earth fillers offer the advantage of unlimited availability and reliable delivery.

Conclusion: Carbon Black N990 can be replaced in the air intake hose compound by Neuburg Siliceous Earth with cost ad vantages and without technical drawbacks.

Our technical service suggestions and the information contained in this report are based on experience and are made to the best of our knowledge and belief, but must nevertheless be regarded as non-binding advice subject to no guarantee. Working and employment conditions over which we have no control exclude any damage claims arising from the use of our data and recommendations. Furthermore, we cannot assume any responsibility for any patent infringements which might result from the use of our information.