

Comparison of mineral fillers in non-conductive car body seals

Authorship: Karin Müller
Hubert Oggermüller

Content

1	Introduction
2	Laboratory trials
2.1	Objectives and test design
2.2	Preliminary tests of electrical conductivity
2.3	Experimental
2.4	Results
2.4.1	Mooney viscosity
2.4.2	Curing properties
2.4.3	Mechanical properties
2.4.4	Electrical properties
2.4.5	Extrusion properties
2.5	Summary of results
3	Pilot plant trials
3.1	Experimental
3.2	Results
3.2.1	Mooney viscosity
3.2.2	Curing properties
3.2.3	Mechanical properties
3.2.4	Electrical properties
3.2.5	Extrusion properties
3.2.6	Optical properties
3.3	Summary of results
4	Conclusions and outlook
5	Appendix

1 Introduction

Since a few years, the automotive industry increasingly has been working with light metals such as aluminum and magnesium. In contact with steel and conventionally formulated sealing profiles, electrochemical corrosion can occur at the less noble metal. In order to avoid that this happens, electrically insulating sealing sections are required.

In the pertinent compounds, consequently, the loading of the traditional major filler carbon black has to be reduced and must be compensated by non-conductive fillers. Here mineral fillers are the materials of choice, as they have already been used as co-fillers, but primarily with respect to esthetic effects at the extruded rubber surface. In the modified formulations, the mineral filler now has to assume the load-carrying properties of the section.

This leads to the question: Which mineral filler to use?

The present work compares a number of light-colored fillers from numerous classes in such compounds with the objective of developing optimum formulations. With priority, fillers based on Neuburg Siliceous Earth were included, and here also the effects of a surface treatment with functional groups (Aktisil grades) were studied.

2 Laboratory Trials

2.1 Objectives and test design

The starting point of the trials was a guide formulation from DSM Elastomers Europe for window duct and channel seals with a medium filler loading.

With respect to the requirements of the car manufacturers, the key test results compiled in *Figure 1* should be complied with.

The definition of the key laboratory data was done under consideration of the uncertainty factor which exists for the transfer of laboratory to production conditions. This is particularly the case for the volume resistivity which can come out one to two orders of magnitude lower for production compounds because of the poorer filler dispersion in internal mixers.



	Part 1: Laboratory Trials 
INTRODUCTION <u>LAB TESTS</u> • EXPERIMENTAL • RESULTS • SUMMARY PILOT PLANT TRIALS SUMMARY APPENDIX	<p>The starting point was a guide formulation for window duct and channel seals from DSM Elastomers Europe with a medium filler loading (40 vol.-% EPDM).</p> <p>With respect to the requirements of automotive companies, the following laboratory results wer to be obtained:</p> <ul style="list-style-type: none">• Hardness 60 to 70 Shore A• Tensile strength > 8 MPa• Compression set 22 h / 70 °C < 20 %• Volume Resistivity > 10⁹, ideally > 10¹⁰ Ω*cm <div data-bbox="549 1368 1386 1408"><div>VM-4/0409/03.2025</div><div></div><div></div></div>

Fig. 1

The control formulation shown in *Table 1* filled with straight carbon black represents a typical example based on a high molecular EPDM grade with long chain branching and a polymer content of about 40 volume percent. In response to modern requirements, the curing system did not contain any nitrosamine generating, i.e. potentially cancerogenic accelerators.



	Base Formulation 	
INTRODUCTION <u>LAB TESTS</u> • EXPERIMENTAL • RESULTS • SUMMARY PILOT PLANT TRIALS SUMMARY APPENDIX	EPDM – 65 Shore A	
		phr
	Keltan 8340 A	100.00
	Zinc Oxide activ	5.00
	Stearic acid	1.00
	Lipoxol 3000	2.00
	Kezadol GR	5.50
	Carbon black N 550	135.00
	Sunpar 2280	65.00
	Rhenogran DPG-80	0.50
	Rhenogran MBTS-80	1.30
	Rhenogran ZBEC-70	2.00
	Rhenogran S-80	0.75
	Rhenodure S/G	1.00
	Rhenocure TP/G	2.00
	Vulkalant E/C	0.50
	Santocure CBS pdr-d	0.50
	Total	322.05
	VM-4/0409/03.2025	

Table 1

2.2 Preliminary tests of electrical conductivity

In view of the primary importance of the electrical properties, preliminary trials for the determination of the volume resistivity were carried out under variation of the loadings of carbon black N550 and Sillitin Z 86. In so doing, an equal hardness of 65 Shore A was aimed at. As a rough guideline, it was confirmed that for equal hardness 1 part of carbon black N550 can be replaced by 2 parts Sillitin Z 86 (*Table 2*).



	Preliminary Tests 						
INTRODUCTION <u>LAB TESTS</u> • EXPERIMENTAL • RESULTS • SUMMARY PILOT PLANT TRIALS SUMMARY APPENDIX	Test for determining the volume resistivity as a function of the loading of carbon black / mineral filler at nearly identical hardness of 65 Shore A						
		phr					
	Carbon black N 550	135	105	90	75	67,5	60
	Sillitin Z 86	0	65	95	125	140	155
		Vol.-%					
	Carbon black N 550	27.2	20.5	17.4	14.4	12.9	11.4
	Sillitin Z 86	0.0	8.7	12.6	16.4	18.3	20.2
	VM-4/0409/03.2025						

Table 2

As can be seen in *Figure 2*, the volume resistivity decreases significantly above a carbon black content of about 11 volume percent. With about 13 vol.-% carbon black, already a level of $10^{10} \Omega\text{cm}$ is obtained, with still higher black loadings the resistivity goes down to about $10^5 \Omega\text{cm}$.

For the development work in progress, therefore, only the range below 12.9 vol.-% carbon black looked acceptable. Despite the optimum dispersion conditions on the laboratory mill, this loading evidently is already in the percolation region. For the continuing trials, therefore, a carbon black loading of 11.4 vol.-% was chosen, which means in this formulation 60 phr carbon black N550 + 155 phr mineral filler.

The higher loading of the mineral filler, in addition, offers the advantage that the individual effects of the grades come out more clearly.

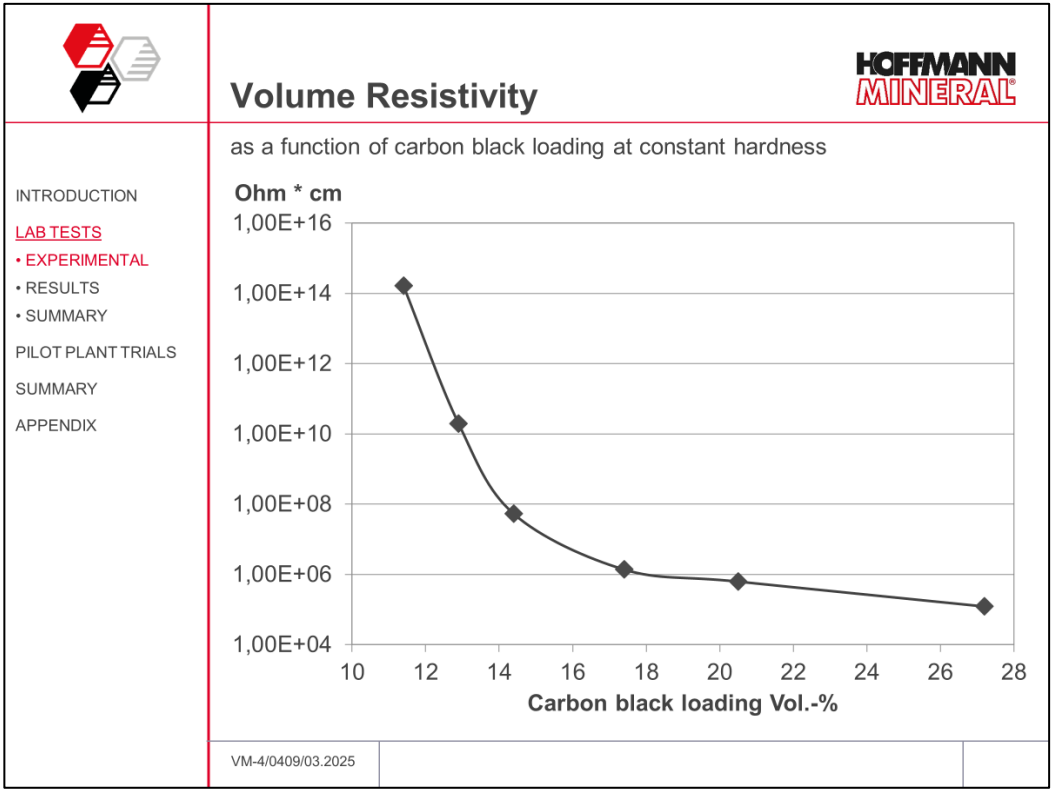



Fig. 2

2.3 Experimental

The mineral fillers included in the test program are listed with some of their characteristical properties in *Table 3*. The selection, in order to work with a broad choice, concerned typical grades of Neuburg Siliceous Earth, calcium carbonate (whiting), talc and clay; because of their high moisture content, precipitated silicates were not included.

The typical properties as indicated were always determined with the aid of the same analytical methods, and therefore should allow a direct comparison for an overall characterisation of the fillers. The median grain size d_{50} has always to be regarded in combination with the specific surface area, as an anisotropy of the grain shape as well as a porosity of the particle aggregates and agglomerates can influence the results.

Among the products based on Neuburg Siliceous Earth, Sillitin P 87 represents a version of Sillitin Z 86 with a finer particle size, while Sillitin N 85 has a higher average particle size. Aktisil MM was treated with a mercapto functional group, Aktisil PF 216 with a tetrasulfane funktional group.



Fillers, Characteristics

HOFFMANN
MINERAL®

INTRODUCTION

LAB TESTS

• EXPERIMENTAL

• RESULTS

• SUMMARY

PILOT PLANT TRIALS

SUMMARY

APPENDIX

Filler Class	Product	Particle size		Oil absorption [g/100g]	Specific surface area BET [m²/g]
		d ₅₀ [µm]	d ₉₇ [µm]		
Neuburg Siliceous Earth	Sillitin N 85	3.0	16	45	10
	Sillitin Z 86	1.9	9	55	11
	Sillitin P 87	1.5	6	55	12
	Aktisil MM	2.2	10	45	7
	Aktisil PF 216	2.2	10	50	8
Calcium Carbonate	Surface treated CaCO ₃	2.7	24	20	2
	CaCO ₃	2.4	13	30	5
Talc	American Talc	4.7	17	50	11
Clay	English calcined Clay	3.5	18	60	8
	English soft Clay	7.7	34	45	7
	English hard Clay	1.8	11	50	30
	English Clay	3.0	12	55	13

VM-4/0409/03.2025

Table 3

The compounding and the curing of the sample sheets were done as described in *Fig. 3*.



		<h1>Preparation and Curing of the Compound</h1>			
<p>INTRODUCTION</p> <p><u>LAB TESTS</u></p> <ul style="list-style-type: none">• EXPERIMENTAL• RESULTS• SUMMARY <p>PILOT PLANT TRIALS</p> <p>SUMMARY</p> <p>APPENDIX</p>		<ul style="list-style-type: none">• Mixing Open mill Ø 150 x 300 mm Batch volume: approx. 1000 g Temperature: 50 °C Mixing time: approx. 20 min.• Curing Press, 170 °C, $t_{90} + 10 \%$			
		VM-4/0409/03.2025			

Fig. 3

The extrusion trials were run in accordance with ASTM D2230 on a laboratory extruder whose parameters are listed in *Figure 4*.




		Extrusion			
<p>INTRODUCTION</p> <p><u>LAB TESTS</u></p> <ul style="list-style-type: none">• EXPERIMENTAL• RESULTS• SUMMARY <p>PILOT PLANT TRIALS</p> <p>SUMMARY</p> <p>APPENDIX</p>		<p>In accordance with ASTM D 2230</p> <ul style="list-style-type: none">• Schwabenthan Extruder Polytest 30R D = 30 mm, L/D ratio = 15• Temperature profile: 70 / 70 / 110 °C• Variations: Constant screw speed 50 rpm• Garvey rating: 1st digit: Die swell 2nd digit: 30° edge 3rd digit: Surface 4th digit: Corners• Assessment 1 = poor to 4 = very good• Maximum rating: 4 x 4 = 16 			
		VM-4/0409/03.2025			

Fig. 4

The parameters of the determination of the volume resistivity are given in *Figure 5*.


	<h2 style="text-align: center;">Volume Resistivity</h2> <div style="text-align: right;">HOFFMANN MINERAL</div>	
<p>INTRODUCTION</p> <p>LAB TESTS</p> <ul style="list-style-type: none"> • EXPERIMENTAL • RESULTS • SUMMARY <p>PILOT PLANT TRIALS</p> <p>SUMMARY</p> <p>APPENDIX</p>	<p>DIN IEC 93</p> <p>Test outline:</p> <ul style="list-style-type: none"> • Dimension of plates: 10 x 10 cm • Thickness of plates: about 2 mm • Electrode set-up: circular plate electrode with protective ring • Test method: Voltage / Amperage method • Test voltage: 100 V • Recording time: 1 min. after application of voltage • Test temperature: 23 °C • Evaluation: $\rho = R_x \cdot A / h$ <p>with</p> <p>ρ volume resistivity in $\Omega \cdot \text{cm}$</p> <p>R_x volume resistance in Ω</p> <p>A effective surface area of the protected electrode (24 cm²)</p> <p>h median thickness of test plate in cm</p>	
	<p>VM-4/0409/03.2025</p>	

Fig. 5

2.4 Results

2.4.1 Mooney viscosity

The Mooney viscosity of all compounds which contain mineral fillers along with carbon black, with the exception of the hard clay, comes out lower than the straight carbon black control. The lowest viscosity is obtained with the surface treated and untreated calcium carbonates, followed by soft clay and talc. The Neuburg Siliceous Earth grades place themselves in the medium region along with the calcined and the medium active clays (*Fig. 6*).

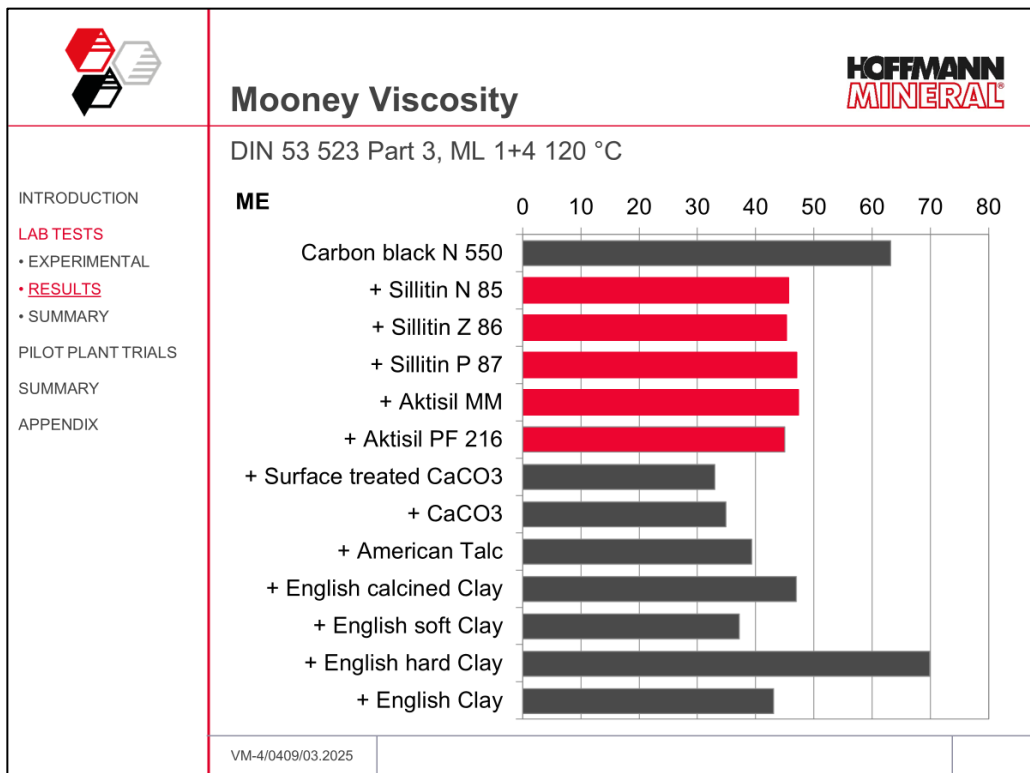


Fig. 6

2.4.2 Curing properties

The time t_{90} as an index for the rate to full cure is made longer and in part significantly, by the mineral fillers, above all by hard clay and talc. Fairly low effects are found with calcined clay, the two calcium carbonates as well as Aktisil MM and Sillitin N 85. The other products came off close to each other in the middle region (*Fig. 7*).

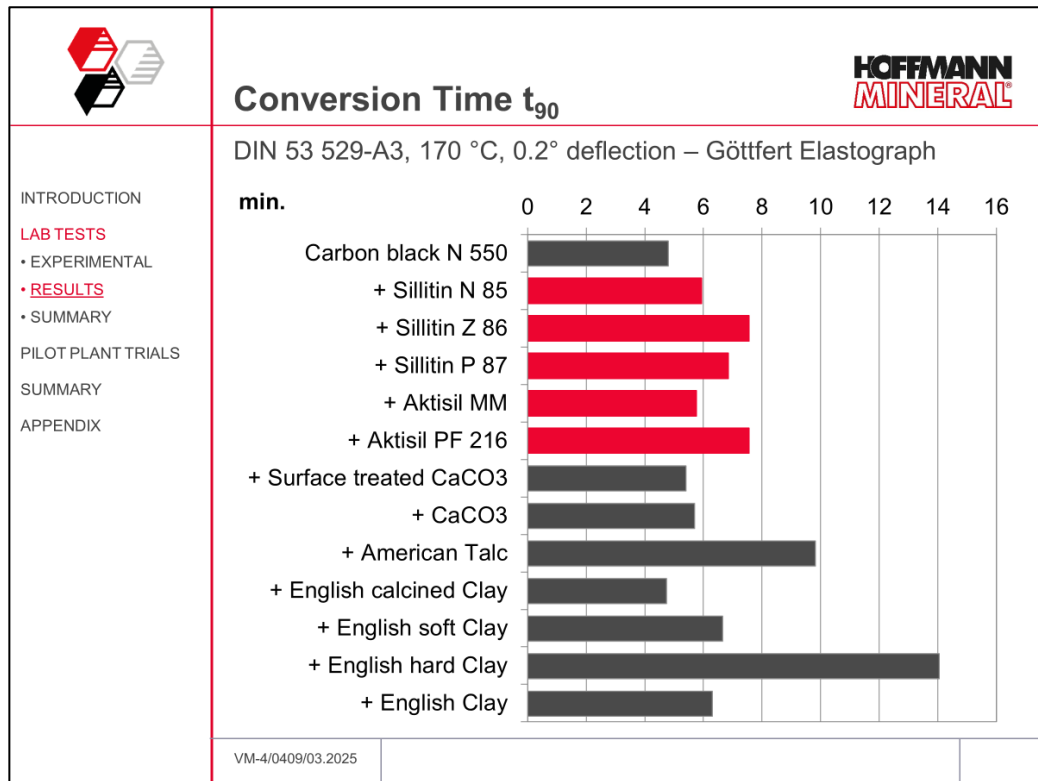


Fig. 7

2.4.3 Mechanical properties

The replacement of part of the carbon black by twice the mass of a mineral filler in most cases arrives at nearly the same Shore hardness, only the two calcium carbonates give markedly lower results. Aktisil PF 216 and hard clay are found on the same level as straight carbon black, medium active clay, talc and Aktisil MM somewhat lower. The surface treatment of the Neuburg Siliceous Earth grades gives rise to somewhat higher hardness compared with the base material Sillitin Z 86 (Fig. 8).

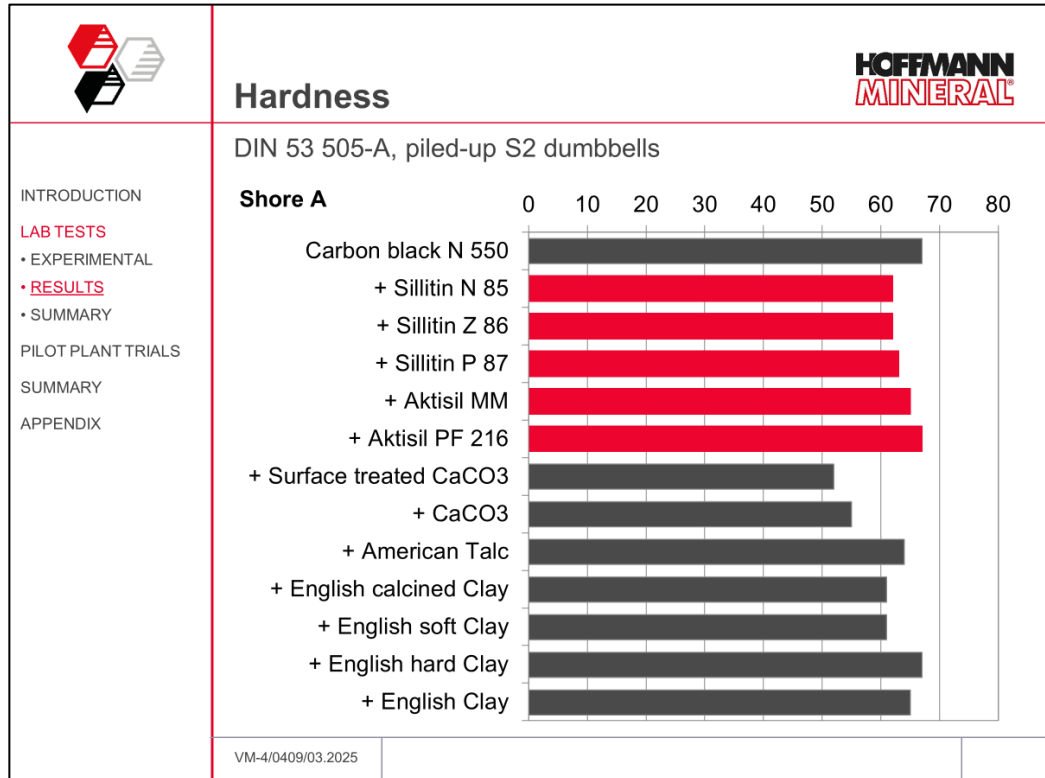


Fig. 8

With respect to tensile strength, the relatively most favorable results are obtained with talc, the untreated clays and the surface treated Aktisil grades. The untreated Sillitin N 85 along with the calcined clay and the calcium carbonates are at a level of 8.5 MPa. If sometimes only just, all compounds meet the specified limit of >8 MPa (Fig.9).

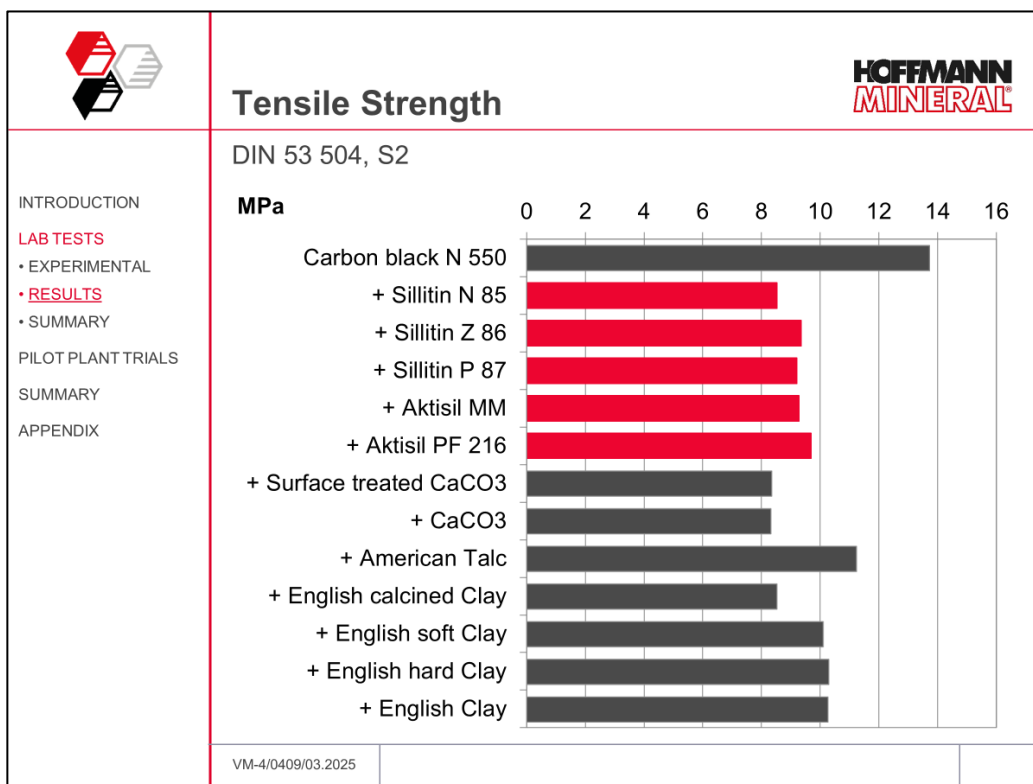


Fig. 9

The elongation at break comes out increased with almost all the mineral fillers. Only Aktisil PF 216 gives nearly the same level as the straight carbon black loading, Aktisil MM a markedly higher result. The other fillers do not show great differences (Fig.10).

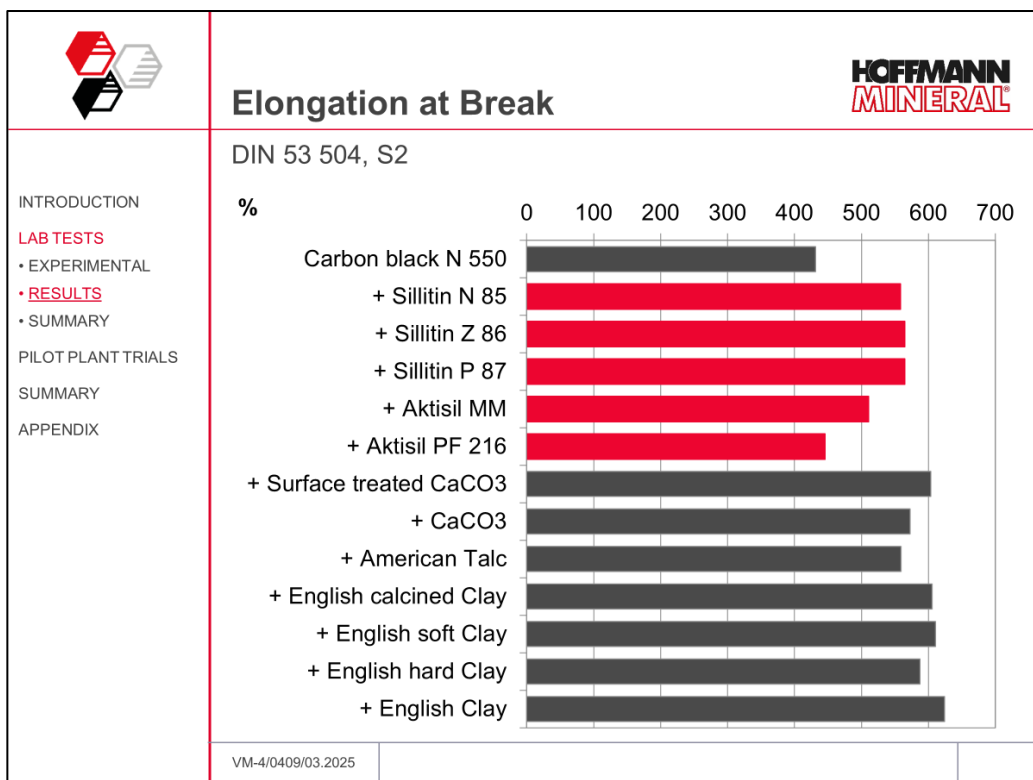


Fig. 10

More differentiated, by contrast, is the picture for the 100 % modulus. The high level of the straight carbon black compound is only and just reached with Aktisil PF 216, followed by Aktisil MM and distinctly lower the talc. Untreated and treated calcium carbonate come out lowest. The other fillers among each other are on a similar level, with the soft clay at the lower end (Fig. 11).

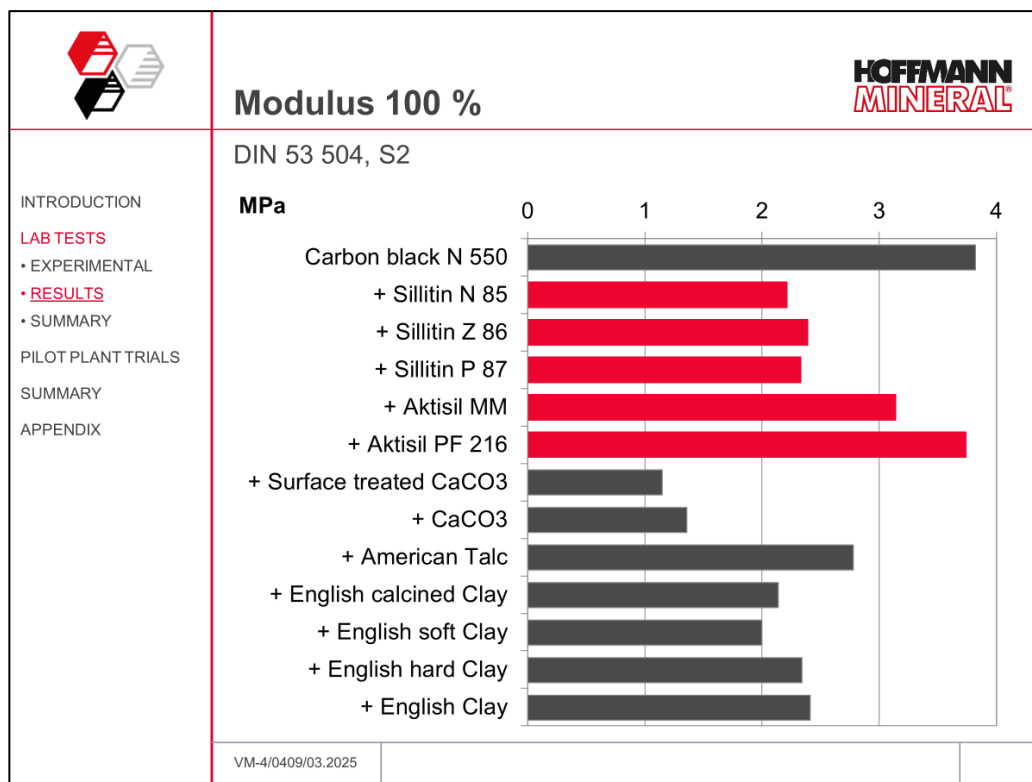


Fig. 11

With regard to tear strength, the straight carbon black loading is surpassed by various blends with mineral fillers. The best result is obtained with the hard clay, followed by the medium-active clay. But also the siliceous earth grades, excepted Aktisil PF 216, arrive at levels of talc, soft and calcined clay just above the carbon black control. Markedly lower finish the two calcium carbonates (Fig. 12).

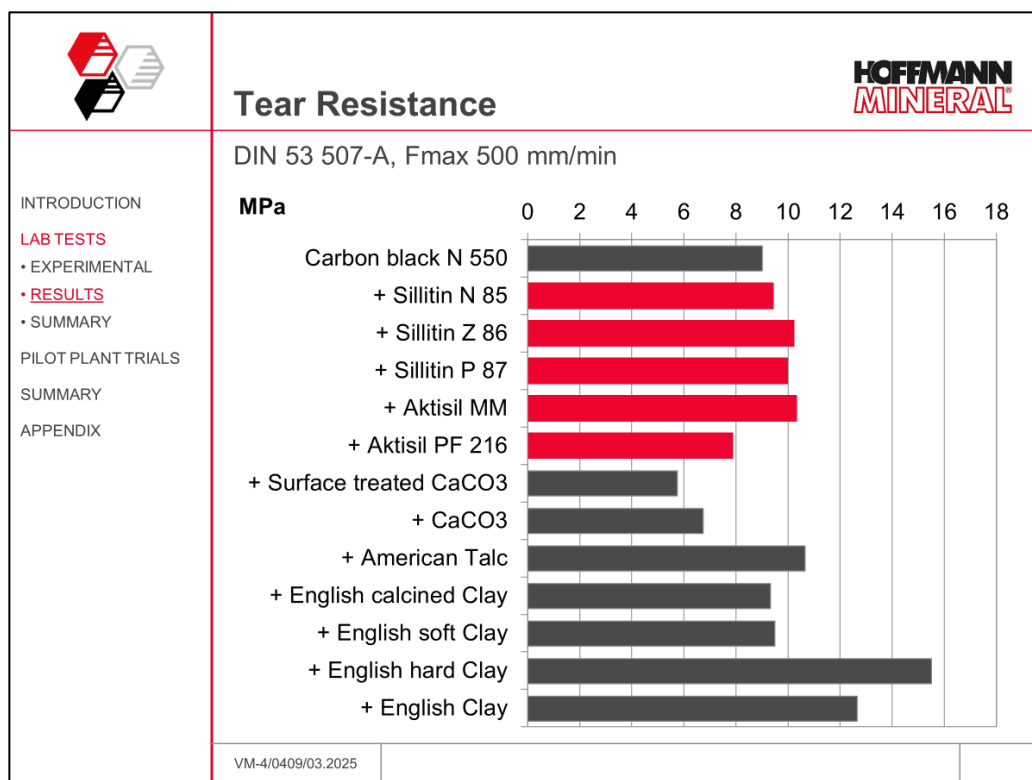


Fig. 12

For the compression set in accordance with ISO 815, the mineral fillers are split into two groups. In particular the Neuburg Siliceous Earth products, and above all the Aktisil grades, similar to the two calcium carbonates arrive at a more favorable, i.e. lower level than the straight carbon black loading. The calcined clay comes out somewhat poorer. The other clays as well as the talc give still poorer results and do no longer meet the specified condition of <20 % (Fig.13).

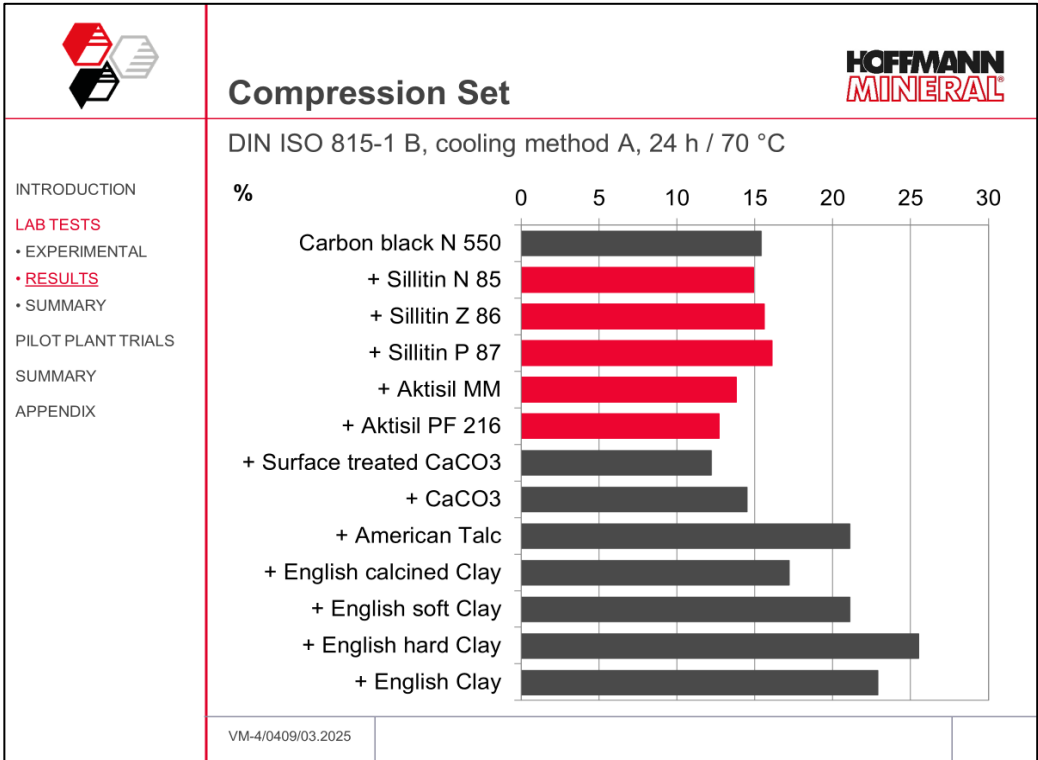


Fig. 13

The more stringent conditions of the compression set test according to the VW standard PV 3307 do hardly change the overall picture, but the blends with untreated and surface treated calcium carbonate here come out with less favorable results than the straight carbon black loading. The results with Sillitin Z 86 and Aktisil MM are slightly below the carbon black control. Aktisil PF 216, by contrast, allows to arrive at markedly better levels (Fig.14).

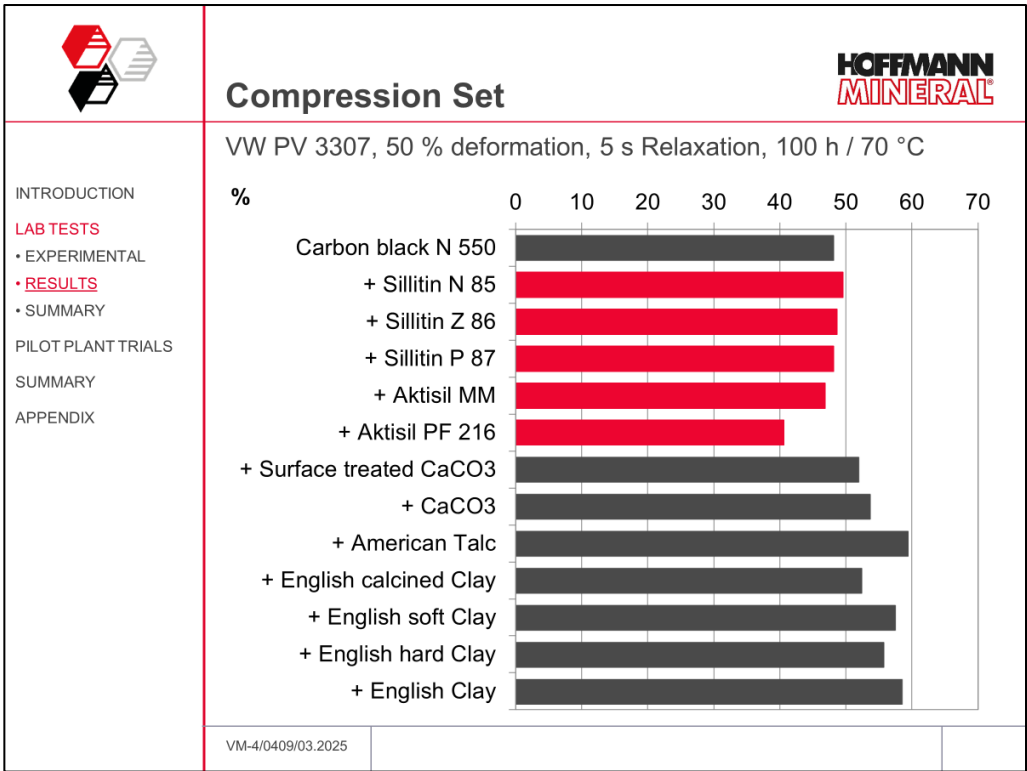


Fig.14

2.4.4 Electrical properties

The whole of the mineral fillers reduce the electrical conductivity (i.e. give higher resistivity), but generally to a varying degree. Above all, the talc and the hard clay impress, compared with the other fillers, with a lower resistivity by one or two orders of magnitude. As all mineral fillers intrinsically should show similar insulation properties, the differences are probably a result of a poorer carbon black dispersion (*Fig. 15*).

Under production conditions in the factory, lower levels by one or two orders of magnitude for the volume resistivity must be expected (*see Fig. 38*).

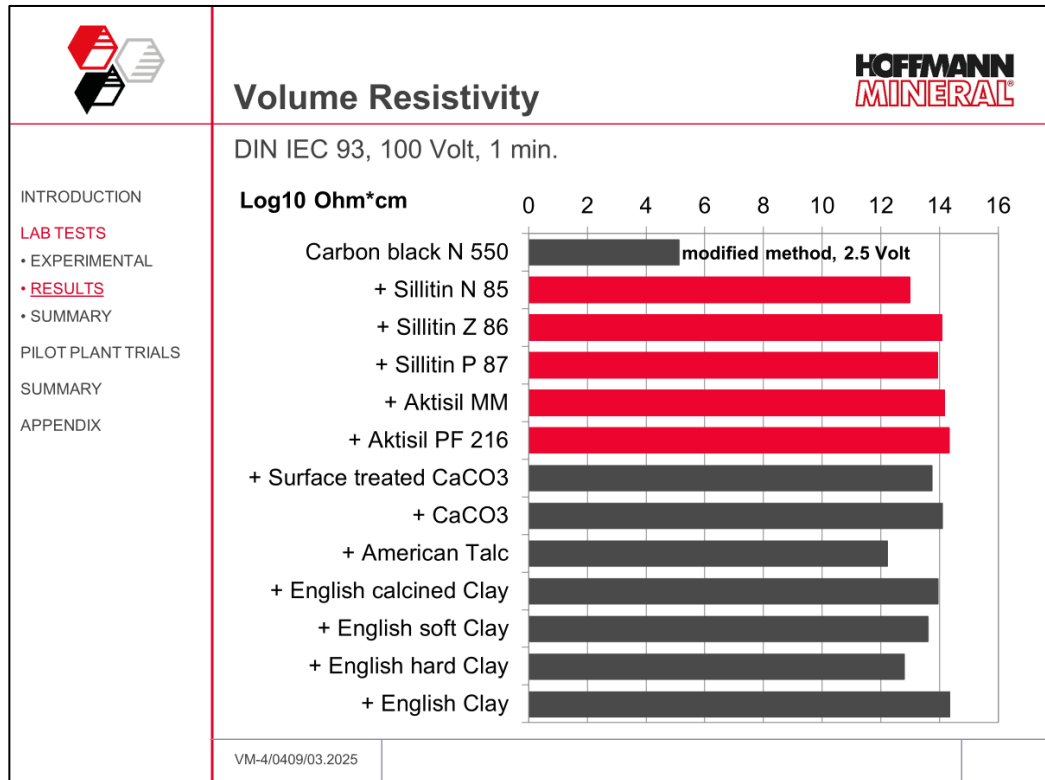


Fig. 15

2.4.5 Extrusion properties

The extrusion properties of the compounds can be characterized by various tests, but only a combination of the results obtained will allow an allround statement.

At a constant screw rate of 50 rpm the output in length and the die swell were determined, and the profile quality assessed according to ASTM.

Concerning the throughput (length output), the Neuburg Siliceous Earth grades along with the hard and medium-active clay come out at least at the level of the straight carbon black loading. Calcined clay, talc and soft clay as well as the two calcium carbonates have negative effects on the output (*Fig.16*).

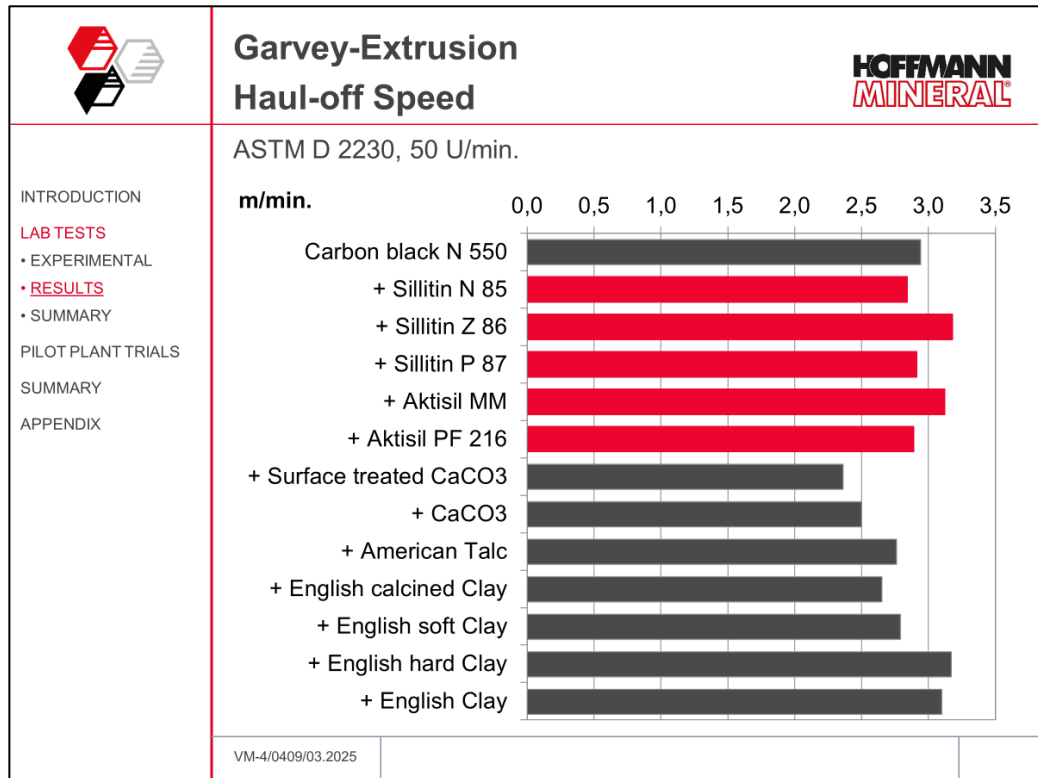


Fig. 16

The extrusion quality with the Garvey die for Sillitin 87 and Aktisil MM comes close to the straight carbon black compound. Almost similarly favorable ranks Aktisil PF 216, followed by Sillitin Z 86, calcined and medium-active clay. Calcium carbonate and Sillitin N 85 position themselves with relatively favorable edge rating in the middle region, while hard clay, talc and the surface treated calcium carbonate reach only moderate results, and the soft clay makes the weakest end (Fig.17).

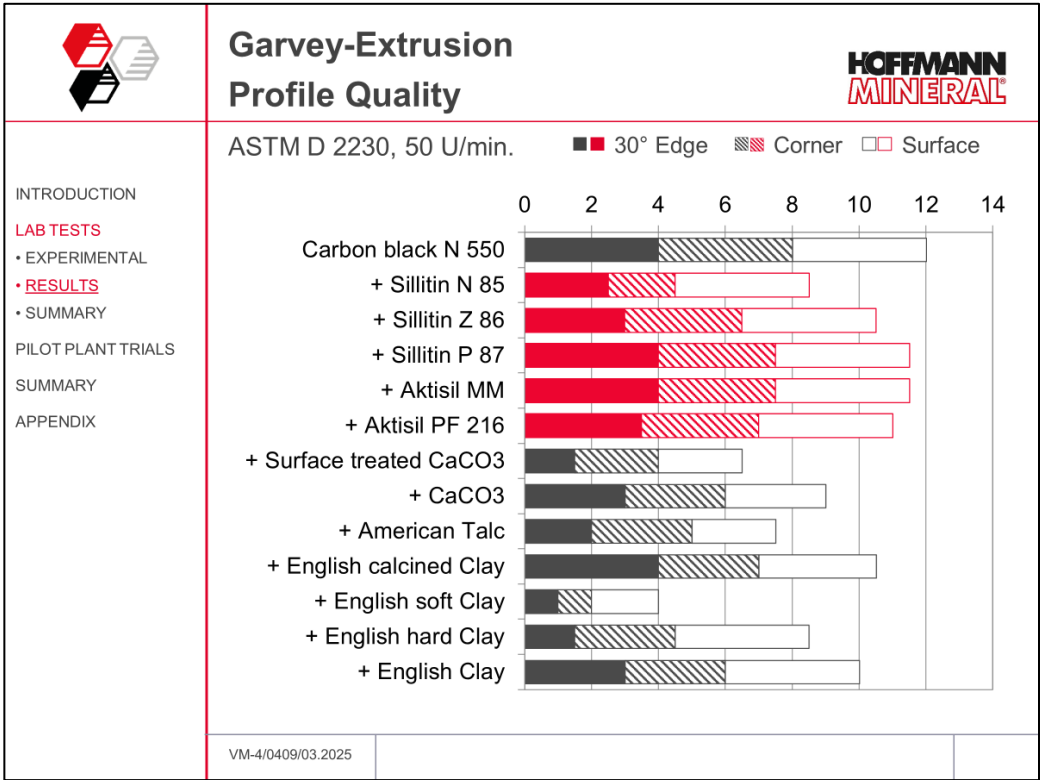


Fig. 17

When assessing die swell, it has to be taken into account that torn profile edges can lead to assessment errors, which is why the results only allow a rough ranking. In particular, the two calcium carbonates impress with markedly higher levels, and talc with the lowest result. Sillitin N 85, Aktisil MM and the soft clay more or less match the level of the straight carbon black control compound. A very low die swell is offered by talc, followed by the medium-active and the hard clay as well as Sillitin P 87. Also the calcined clay and Sillitin Z 86 impart a somewhat lower die swell than carbon black alone (Fig. 18).

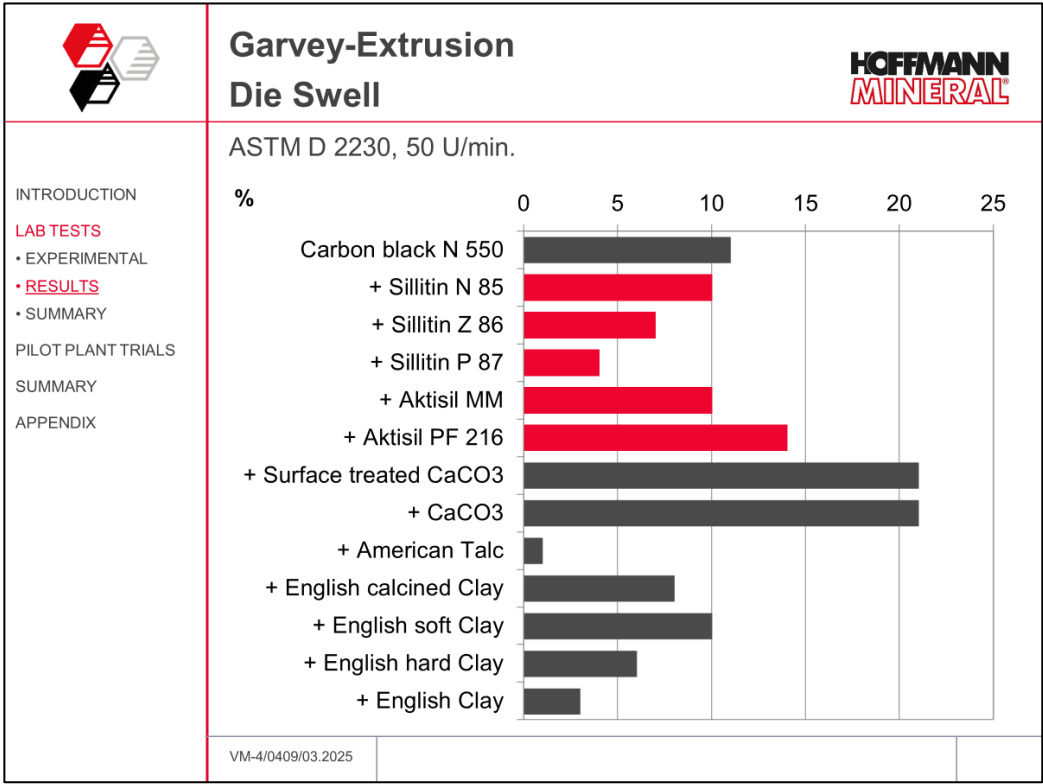


Fig. 18

Figure 19 where the profile quality is plotted versus the output length, offers a meaningful allover summary of the extrusion characteristics of the filler blends evaluated. The products based on Neuburg Siliceous Earth, with the exception of Sillitin N 85, are positioned close to the carbon black at the upper right of the diagram, and appear to represent, therefore, the best suited fillers for such extrusion compounds.

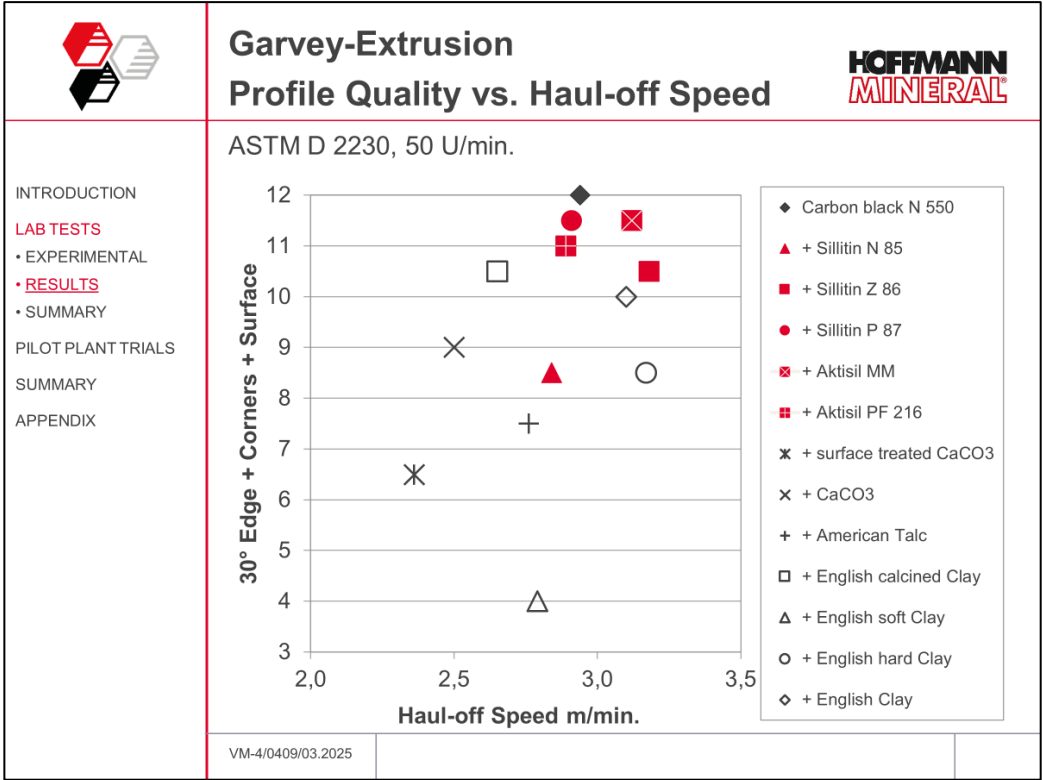


Fig. 19

2.5 Summary of results

Fig. 20 and Fig. 21 summarize the results of the laboratory trials.

<div> <div> </div> <div> <h3>Laboratory Trials</h3> <h4>Summary (1)</h4> </div> <div> </div> </div>	
<div> <div>INTRODUCTION</div> <div>LAB TESTS</div> <div>• EXPERIMENTAL</div> <div>• RESULTS</div> <div>• SUMMARY</div> <div>PILOT PLANT TRIALS</div> <div>SUMMARY</div> <div>APPENDIX</div> </div>	<p>With mineral fillers non-conductive car body seals can be produced which – with the only exception of tensile strength – do not give evidence of diminished performance properties.</p> <ul style="list-style-type: none"> • The calcium carbonates show their main deficiency in the mechanical and extrusion properties, but at short cure times they give good compression set results. • Talc imparts good tensile strength levels, but even at long cure times compression set comes out poor, and extrusion properties are just moderate. • With clays, excepted the calcined version, good mechanical properties are obtained, but they show weak points in extrusion properties and particularly in compression set results. For the calcined clay, more or less the reverse is true.
VM-4/0409/03.2025	

Fig. 20



	<h2>Laboratory Trials Summary (2)</h2> 
<p>INTRODUCTION</p> <p>LAB TESTS</p> <ul style="list-style-type: none"> • EXPERIMENTAL • RESULTS • SUMMARY <p>PILOT PLANT TRIALS</p> <p>SUMMARY</p> <p>APPENDIX</p>	<ul style="list-style-type: none"> • The Neuburg Siliceous Earth grades are distinguished above all by their outstanding extrusion properties and low compression set. • The most suitable grades are Aktisil MM and Aktisil PF 216. They impart very good extrusion properties along with high tensile moduli and excellent compression set.
	<div>VM-4/0409/03.2025</div>

Fig. 21

3 Pilot plant trials

3.1 Experimental

The compounding, extrusion and continuous cure were carried out at the pilot plant of DSM Elastomers Europe in Geleen (The Netherlands).

For the pilot plant trials (*Table 4*) nearly the same compound formulation was used as for the laboratory trials (*Table 1*). Only the modifications as given in *Figure 22* were applied.




	<h2>Part 2: Pilot Plant Trials</h2> 
<p>INTRODUCTION</p> <p>LAB TESTS</p> <p>PILOT PLANT TRIALS</p> <ul style="list-style-type: none"> • EXPERIMENTAL • RESULTS • SUMMARY <p>SUMMARY</p> <p>APPENDIX</p>	<p>The starting point was, with minor adjustments, the same formulation as was used for the laboratory trials. The following changes were applied:</p> <ul style="list-style-type: none"> • Calcium oxide was increased to 10 phr, in order to ensure blister-free extrusions. • Because of availability, Lipoxol 3000 was replaced by Breax 3400. • Use of Rhenogran CBS-80 instead of Santocure CBS, however same dosage.
	<div>VM-4/0409/03.2025</div>

Fig. 22



HOFFMANN
MINERAL®

Base Formulation

EPDM – 65 Shore A

	phr	phr
Keltan 8340 A	100.00	100.00
Zinc Oxyde active	5.00	5.00
Stearic acid	1.00	1.00
Breax 3400	2.00	2.00
Kezadol GR	10.00	10.00
Carbon black N 550	135.00	60.00
Mineral Filler	-	155.00
Sunpar 2280	65.00	65.00
Rhenogran DPG-80	0.50	0.50
Rhenogran MBTS-80	1.30	1.30
Rhenogran ZBEC-70	2.00	2.00
Rhenogran S-80	0.75	0.75
Rhenodure S/G	1.00	1.00
Rhenocure TP/G	2.00	2.00
Vulkalant E/C	0.50	0.50
Rhenogran CBS-80 %	0.50	0.50
Total	326.55	406.55

VM-4/0409/03.2025

INTRODUCTION

LAB TESTS

PILOT PLANT TRIALS

• EXPERIMENTAL

• RESULTS

• SUMMARY

SUMMARY

APPENDIX

Table 4

The preparation of the compounds in the pilot plant was done under the conditions listed in *Figure 23*. There was a noticeable difference in the compound temperature at the end of the mixing process: the straight carbon black compound arrived at 135 °C, the compounds with the filler blends only at 100-120 °C.


	<div> <div>HOFFMANN MINERAL</div> <div>Preparation of the Compound</div> </div>
<div> <div>INTRODUCTION</div> <div>LAB TESTS</div> <div>PILOT PLANT TRIALS</div> <div>• EXPERIMENTAL</div> <div>• RESULTS</div> <div>• SUMMARY</div> <div>SUMMARY</div> <div>APPENDIX</div> </div>	<div> <div> <div>Mixing parameters</div> <div>Internal mixer Farrel 3D, tangential, volume 70 liters</div> <div>Rotational speed: 35 rpm</div> <div>Fill factor: 72 % by volume</div> <div>Starting temperature (at wall): 50 °C</div> <div>Mixing time: 6 min</div> <div>Final mix temperature: Carbon black 135 °C Carbon black + Mineral Filler 100 – 120 °C</div> </div> <div> <div>Mixing cycle</div> <div>0 - 1 min Polymer</div> <div>1 - 2 min All others except accelerator and sulfur</div> <div>2 - 4 min Mixing</div> <div>4 - 4.5 min Sweep down</div> <div>4.5 - 6.0 min Mixing</div> <div>Dump on open mill (30 °C), cool, and add accelerator and sufur</div> </div> <div> <div>VM-4/0409/03.2025</div> </div> </div>

Fig. 23

The extrusion trials at the pilot plant were run under the conditions given in *Figure 24*. With the temperature conditions of the filler blend compounds a satisfactory extrusion of the straight carbon black compound was not possible, so the temperature here had to be suitable increased.


	<div>HOFFMANN MINERAL®</div>		
Extrusion			
<div>INTRODUCTION</div> <div>LAB TESTS</div> <div>PILOT PLANT TRIALS</div> <div>• EXPERIMENTAL</div> <div>• RESULTS</div> <div>• SUMMARY</div> <div>SUMMARY</div> <div>APPENDIX</div>	Vacuum Extruder , Diameter _D : 90 mm, Length: 16 D		
	Temperatures °C	Straight carbon black	Carbon black + Mineral Filler
	Zone 1 (screw)	75	50
	Zone 2	75	40
	Zone 3	80	50
	Zone 4	90	60
	Zone 5 (head)	110	70
	Rational speed: 15 to 18 rpm		
	Output rate: 7.5 m/min constant		
	The extrusion of the straight carbon black compound was not possible with the temperature profile of the carbon black + mineral filler compounds!		
	VM-4/0409/03.2025		

Fig. 24

In summary it can be stated that the filler blends at mixing as well as at extruding generate less heat, or require a lower temperature. This difference may purposefully be used with advantage for process, energy and cost optimization efforts (*Fig. 23 and 24*).

The cure of the extruded sections took place under the conditions given in *Figure 25*.


	<div> <div>Curing</div> <div>HOFFMANN MINERAL</div> </div>	
<div> <div>INTRODUCTION</div> <div>LAB TESTS</div> <div>PILOT PLANT TRIALS</div> <div>• EXPERIMENTAL</div> <div>• RESULTS</div> <div>• SUMMARY</div> <div>SUMMARY</div> <div>APPENDIX</div> </div>	UHF channel: 2 x 6 kW, length 6 m, air temperature 200 °C, typical discharge temperature 185 °C	
	Hot air tunnel: length 3 x 9 m, air temperature 270 °C	
	Cooling basin: length 2 x 9 m, water temperature 10 °C	
	VM-4/0409/03.2025	

Fig. 25

The test specimens were prepared as described in *Figure 26*. Color and gloss determinations were run at the underside of the profile bottom, for which the profile sections concerned were rotated by 180° prior to cure.


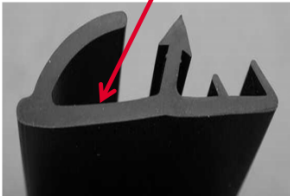
	<div style="text-align: right;">HOFFMANN MINERAL</div> <h2 style="text-align: center;">Preparation of Test Specimens</h2>	
<p>INTRODUCTION</p> <p>LAB TESTS</p> <p><u>PILOT PLANT TRIALS</u></p> <p>• EXPERIMENTAL</p> <p>• RESULTS</p> <p>• SUMMARY</p> <p>SUMMARY</p> <p>APPENDIX</p>	<ul style="list-style-type: none"> The optical and mechanical properties were evaluated on samples taken from the bottom of the extruded profiles. <div style="text-align: center;">  </div> <ul style="list-style-type: none"> The sheets for the determination of volume resistance were cured from the compound mixed in the internal mixer. 	
	<small>VM-4/0409/03.2025</small>	

Fig. 26

With all the difficulties of quantifying color impressions, an easily interpreted assessment can be obtained with the CIELAB color system via the L*a*b* levels (*Fig. 27 and Fig. 28*).


	<div style="text-align: right;">HOFFMANN MINERAL</div> <h2 style="text-align: center;">Measurement of Color</h2>	
<p>INTRODUCTION</p> <p>LAB TESTS</p> <p><u>PILOT PLANT TRIALS</u></p> <p>• EXPERIMENTAL</p> <p>• RESULTS</p> <p>• SUMMARY</p> <p>SUMMARY</p> <p>APPENDIX</p>	<p>ISO 7724</p> <ul style="list-style-type: none"> Parameter <p>Spectral photometer (Luci 100, Dr. Lange)</p> <p>Light D 65</p> <p>Geometry d/8°, without gloss trap</p> <p>Observation angle 10°</p> Definition <p>L*: brightness (0: ideal black; 100: ideal white)</p> <p>a*: red / green (positive values: red tints; negative values: green tints)</p> <p>b*: yellow / blue (positive values: yellow tints; negative values: blue tints)</p> 	
	<small>VM-4/0409/03.2025</small>	

Fig. 27

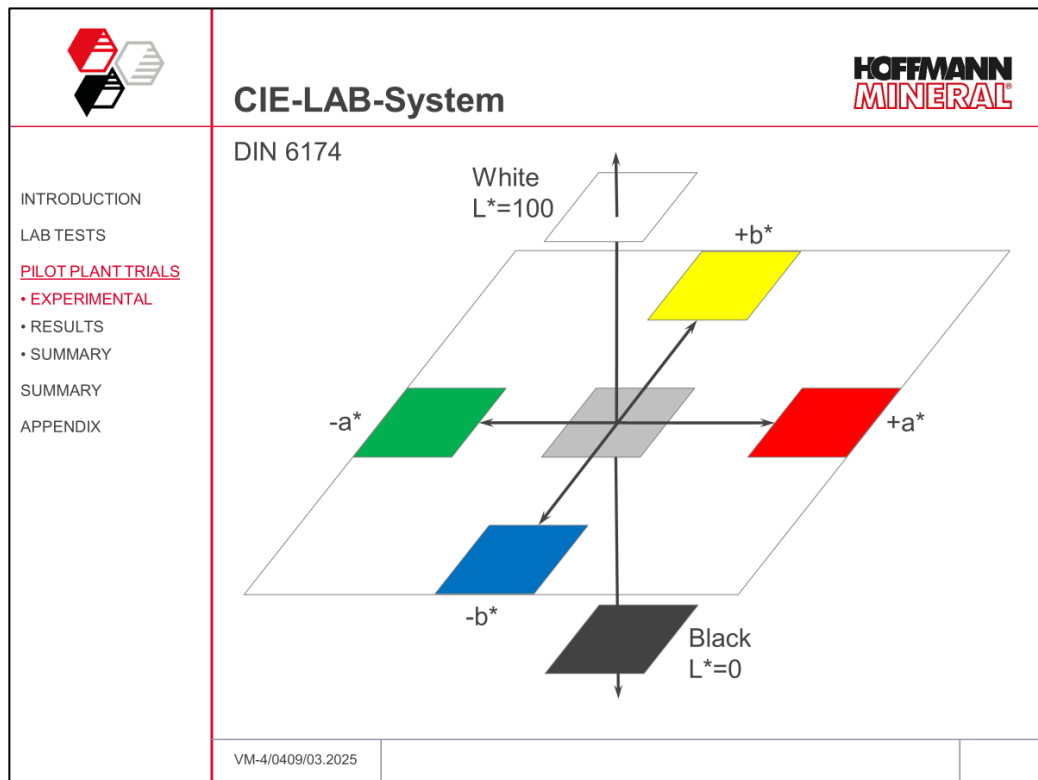


Fig. 28

3.2 Results

3.2.1 Mooney viscosity

The Mooney viscosity of the compounds from the internal mixer basically showed the same ranking as the laboratory mixes, but the overall level was shifted upwards by 5 to 10 points. Again the compounds with mineral fillers, excepted the hard clay, came off lower throughout than the straight carbon black compound. The last rank is occupied by the surface treated and the untreated calcium carbonate. Along with the calcined and the medium-active clay, the Neuburg Siliceous Earth grades are found in the center span (Fig. 29).

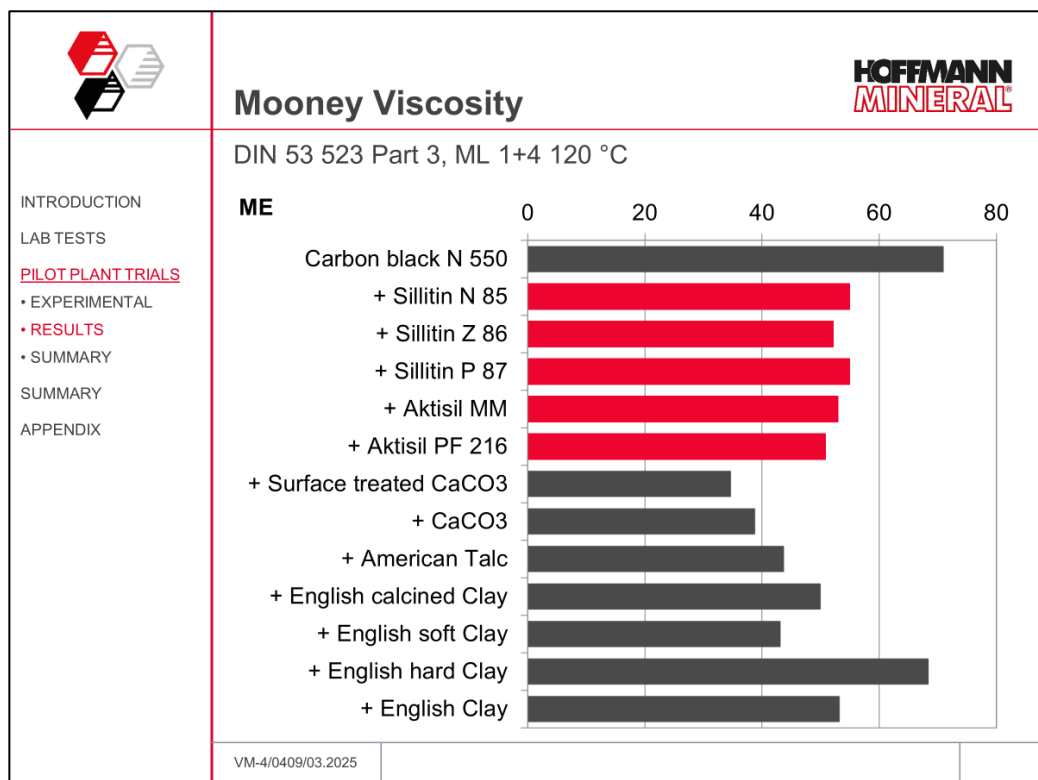


Fig. 29

3.2.2 Cure properties

The conversion time t_{90} as an index for the time to full cure comes out about 1 to 2 minutes longer for the banbury compounds. Talc and the medium-active clay show a marked re-tarding effect, Sillitin N 85 looked indifferent as did also the two calcium carbonates and the calcined clay (Fig. 30).

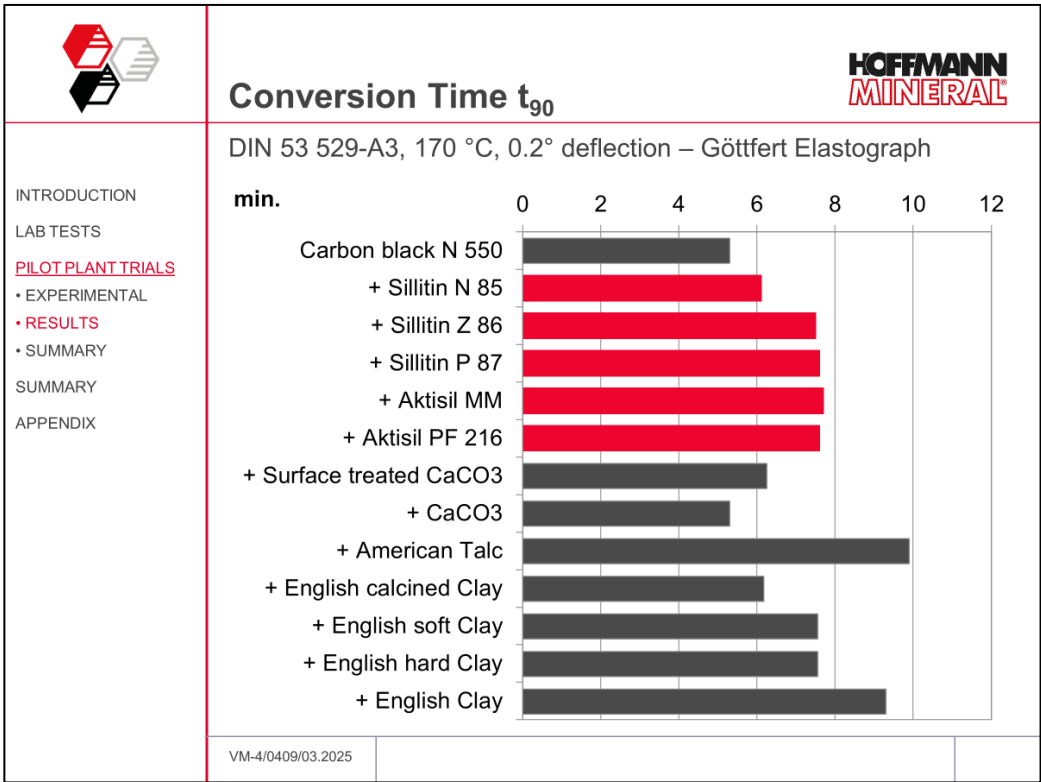


Fig. 30

3.2.3 Mechanical properties

With respect to hardness all fillers, excepted the two calcium carbonates, reach levels around 60 Shore A. Hard clay and Aktisil PF 216 come off at the slightly higher level of the carbon black control compound. Compared with the corresponding laboratory compounds, the hardness generally is lower by about 5 points. The especially low results of the two calcium carbonates, in particular for the surface treated material may well be due to a high porosity of the extruded sections (Fig. 31).

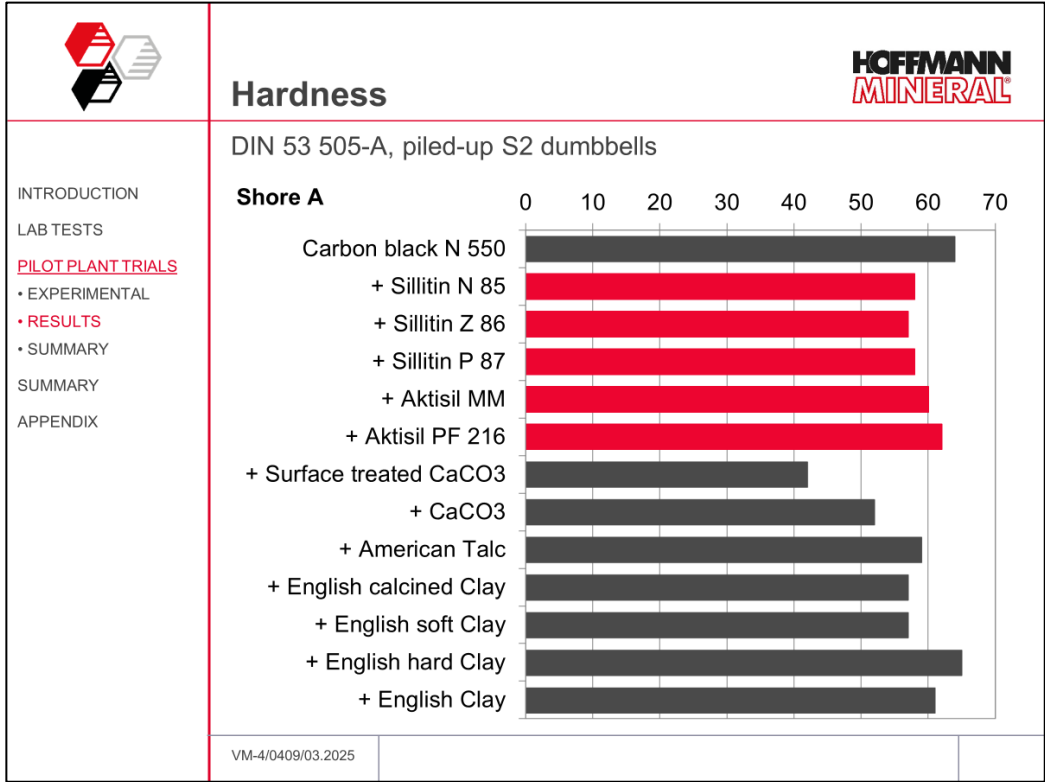


Fig. 31

As already found for the laboratory mixes, all blends with mineral fillers result in a lower tensile strength than the straight carbon black loading. Rather favorable are still talc, hard and medium-active clay as well as Aktisil PF 216 and MM. Likewise, Sillitin Z 86 and Sillitin P 87 position themselves just above the aspired limit of 8 MPa. Considering a safety margin, the calcined and the soft clay with 7.5 MPa are found in the limiting region of the customer specification, while the untreated and in particular the surface treated calcium carbonate by far are not able to meet those requirements (*Fig. 32*).

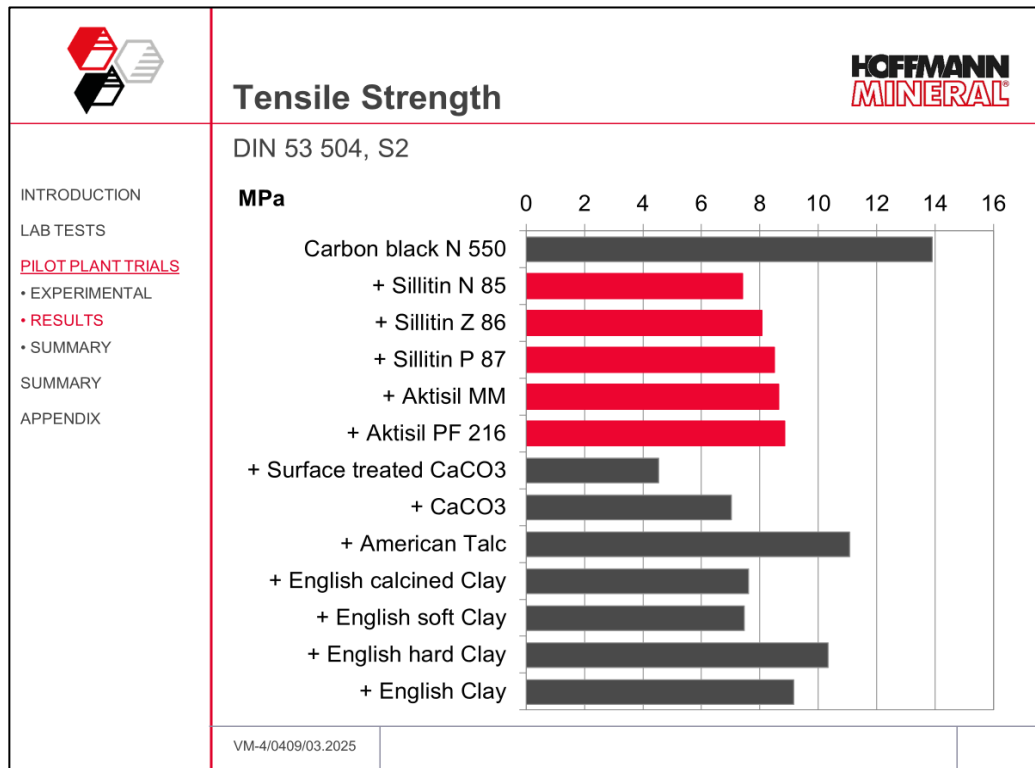


Fig. 32

The elongation at break, by contrast, comes out higher with all filler blends. Aktisil PF 216 finds itself closest to the straight carbon black loading, followed by the surface treated calcium carbonate. Talc is positioned at the upper end, the other products are also lifted to about the same level and do not show big differences (*Fig. 33*).

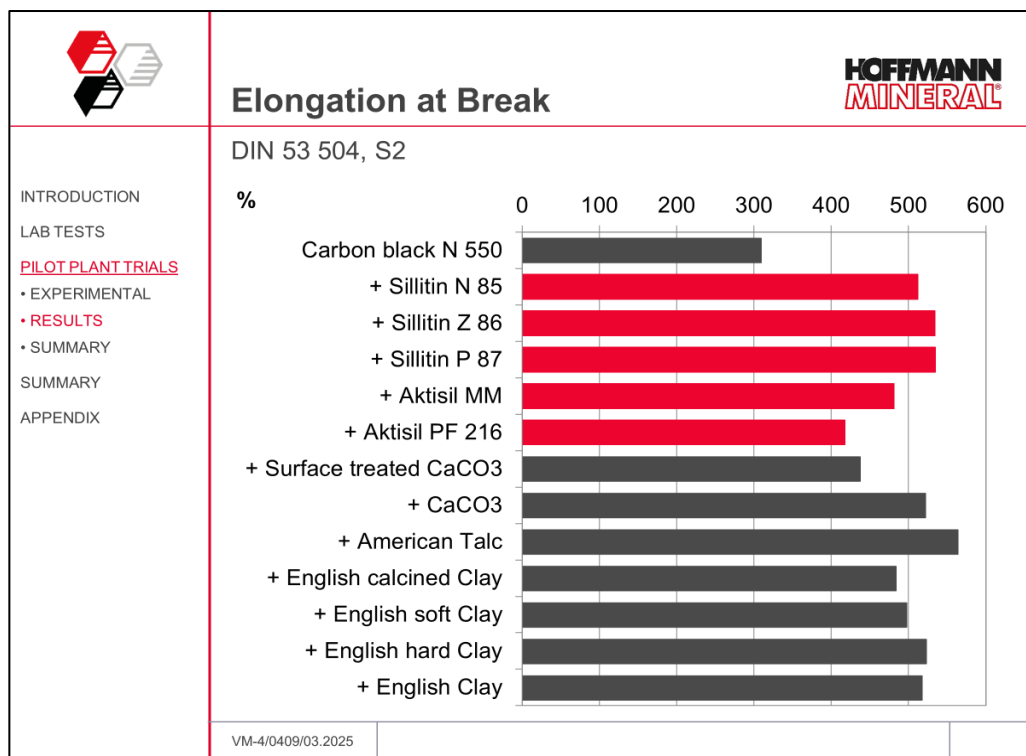


Fig. 33

The 100 % modulus of most of the filler blends is only half as high as in the straight carbon black compound. Its high level is only approached by Aktisil PF 216, followed by Aktisil MM and markedly lower the hard clay. Talc and the medium-active clay form the center span, and way down are found the two calcium carbonates (Fig. 34).

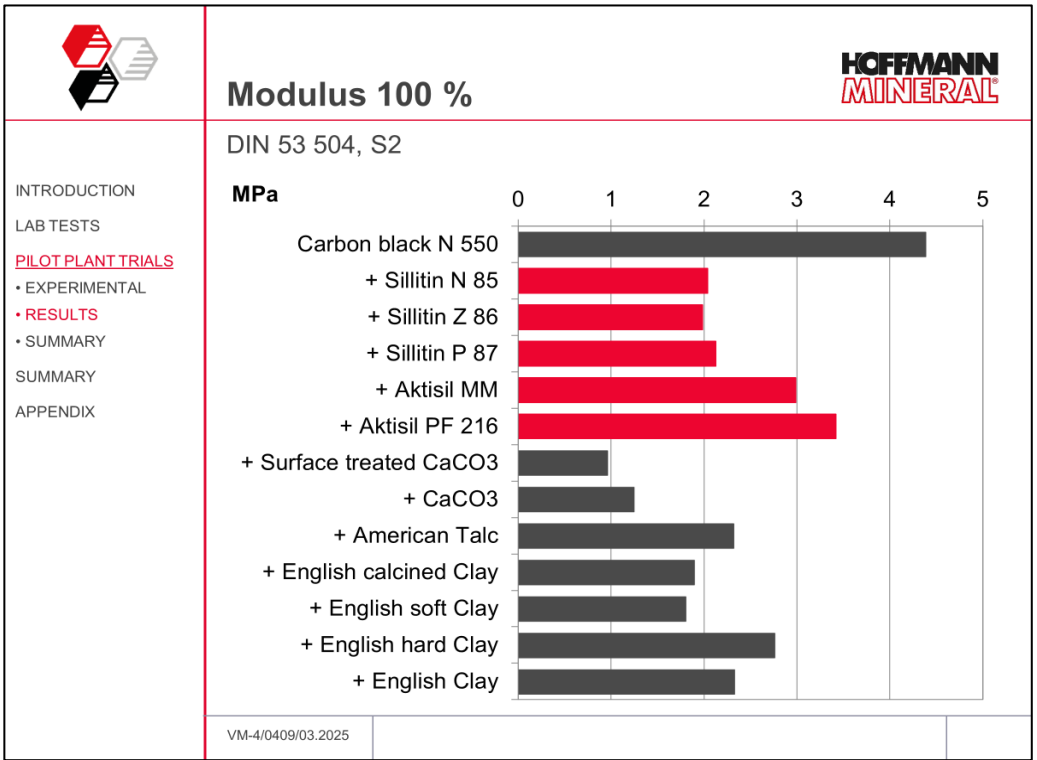


Fig. 34

As is evident from Figure 35, also the tear resistance shows significant differences, and here numerous blends with mineral fillers are able to surpass the straight carbon black loading. Aktisil MM followed by the hard clay largely leads the field. Talc, the medium-active clay and the other siliceous earth products come off somewhat lower, but still above the straight carbon black control. The calcined clay and the soft clay do not fully attain the carbon black level, and the two calcium carbonates again are found at the low end of the scale.

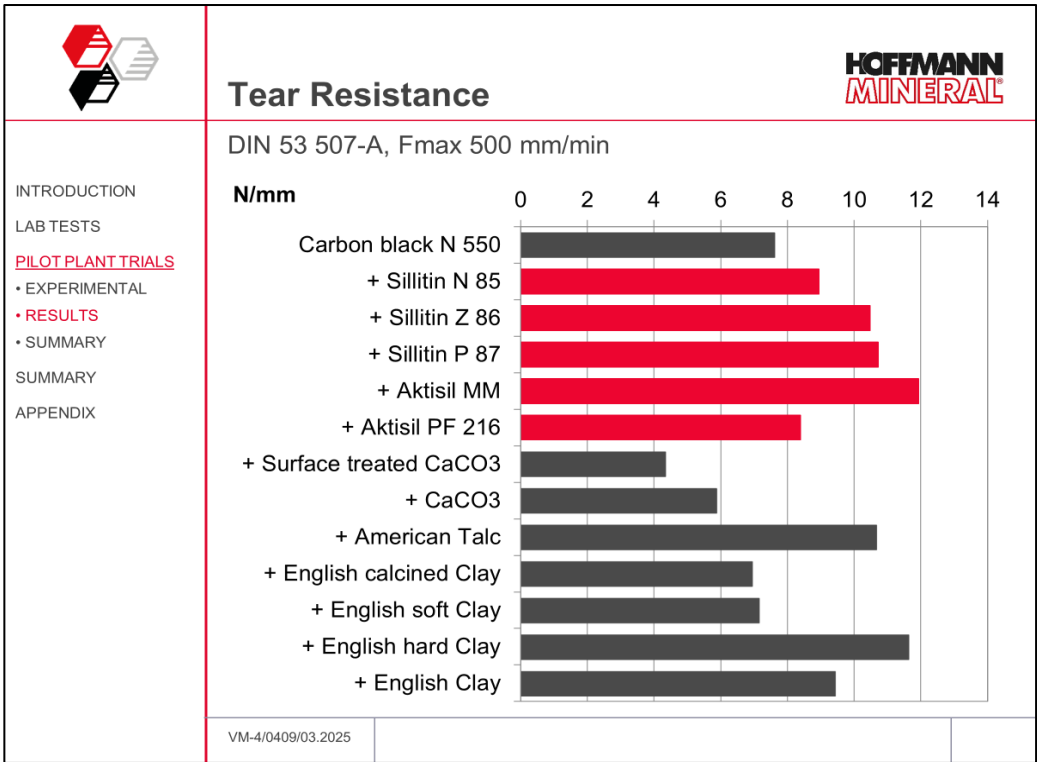


Fig. 35

The compression set according to the VW standard PV 3307 was determined both after 5 seconds and after 60 minutes relaxation time. When tested at room temperature, Aktisil PF 216 clearly arrived at the best result, after the short relaxation time distinctly better than straight carbon black. By contrast, Aktisil MM and untreated calcium carbonate were just at level with the carbon black, followed by Sillitin N 85, Z 86 and the calcined clay. Somewhat weaker are Sillitin P 87 and the surface treated calcium carbonate, while talc and the other clays form a less satisfactory group (Fig. 36).

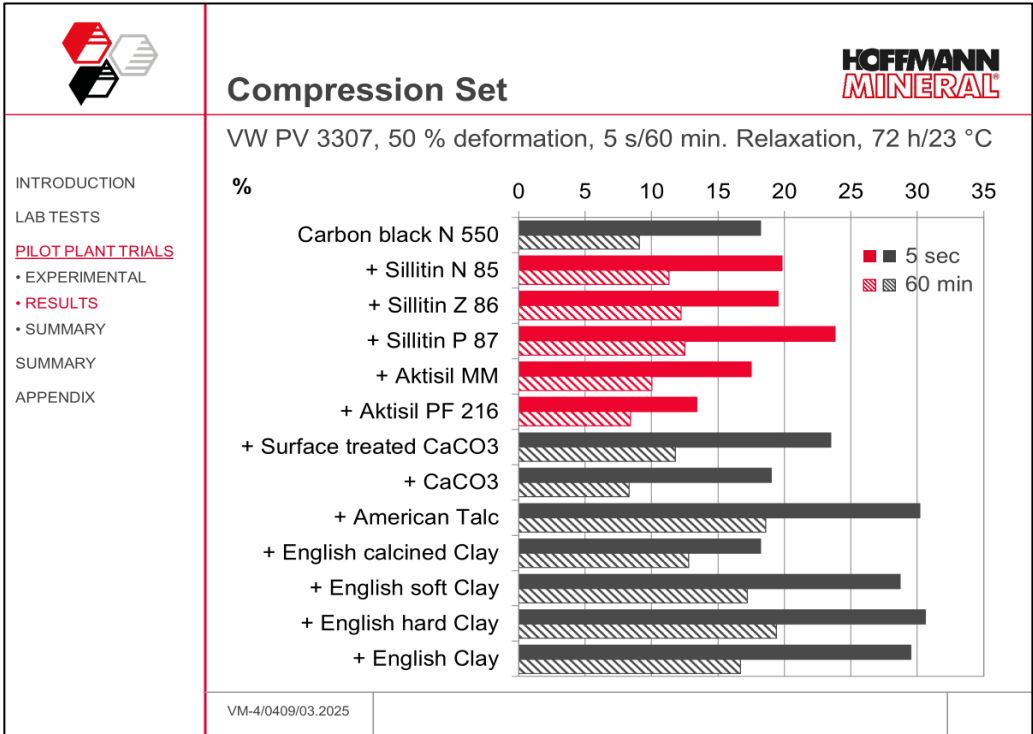


Fig. 36

The test at higher temperature basically leads to comparable results, and here just Aktisil PF 216, Aktisil MM and calcium carbonate offer a similarly low level as the straight carbon black loading. The calcined clay, Sillitin Z 86, Sillitin N 85, Sillitin P 87 and the surface treated calcium carbonate are situated in the center span, the other clays and talc in a less favorable region. The meager result of the uncalcined clays and of talc may go back to their platelet structure (Fig. 37).

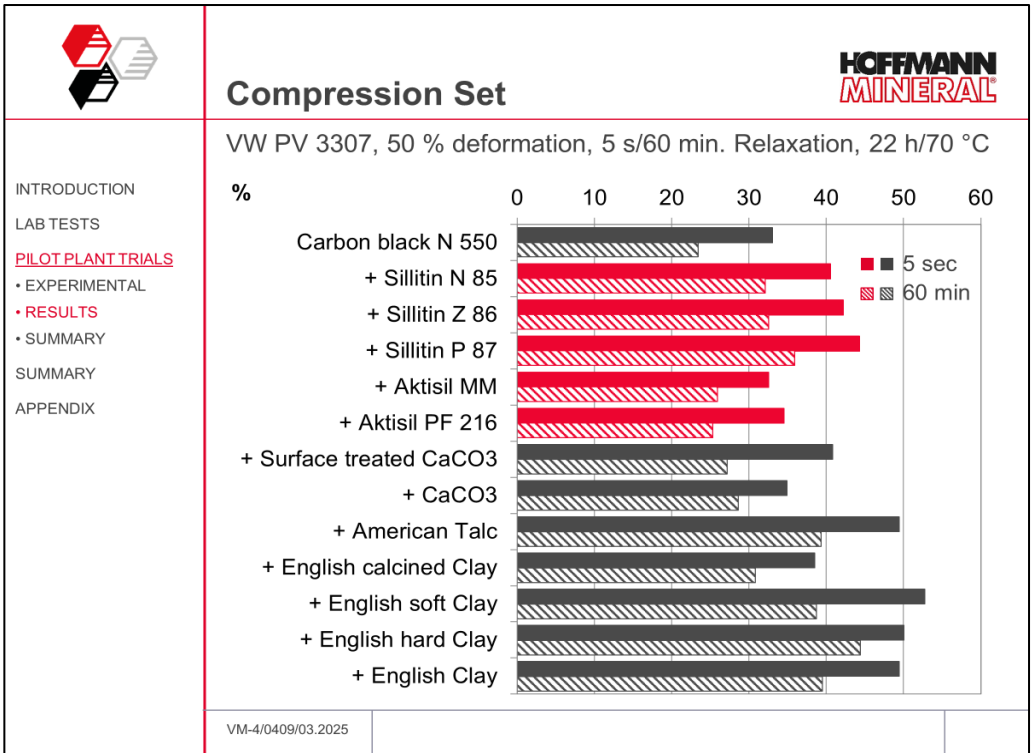


Fig. 37

3.2.4 Electrical properties

For volume resistivity, the two calcium carbonates, with results around $10^{10} \Omega\text{cm}$, take a fairly unsatisfactory position, barely meeting the specification requirements. The soft and the hard clay, talc and Sillitin N 85 follow at a slightly higher level, but are surpassed by Aktisil MM, the medium-active clay, Sillitin Z 86 and Sillitin P 87 with results above $10^{12} \Omega\text{cm}$. The lowest conductivity, with resistivities above $10^{13} \Omega\text{cm}$, is obtained with the calcined clay and with Aktisil PF 216 (Fig. 38).

Compared with the laboratory compounds, the resistivity results are lower by about 1.5 orders of magnitude, which fully confirms expectations (see item 2.4.4 and Figure 15). The differences in the electrical conductivity, similar to the laboratory mixes, have to be attributed to effects of the dispersion of the carbon black and the mineral fillers, partly affected by the grain shape. Only the straight carbon black compound shows almost no response to the mixing procedure. The banbury compound even comes off slightly higher compared with the laboratory mix.

So far, the volume resistivity after water immersion has not been discussed. In order to run tests close to application conditions, this important property should be included in future studies, as the practical use of the sections normally takes place in the «wet area» of the cars.

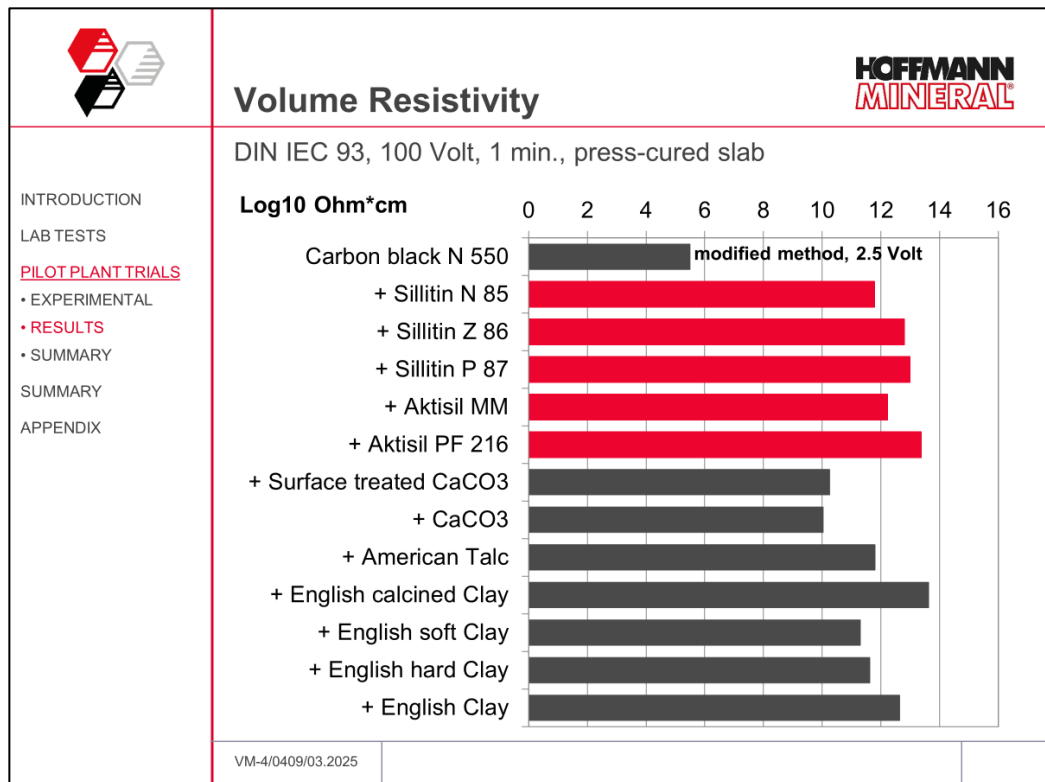


Fig. 38

3.2.5 Extrusion properties

For the extrusion tests, a profile shape was chosen which differentiates above all the edge formation. In particular, the center part which resembles a fir tree represents a critical region. The dimensional stability or collapse resistance of the extruded sections could be determined through the distance between the leg of the profile (left in the picture) to the profile bottom (Fig. 39).

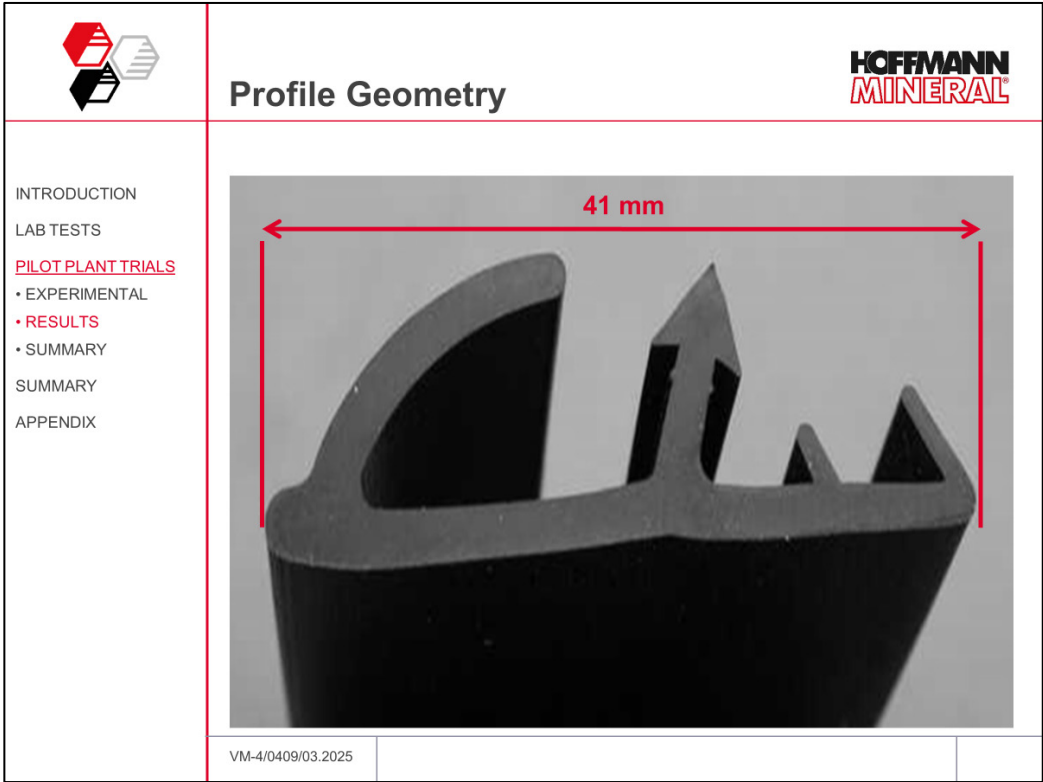


Fig. 39

Magnifications of all profile images are found in the appendix.

The profile pictures in *Figure 40* show a very good edge formation for the straight carbon black compound, along with a glossy surface and high collapse resistance. Such compounds, if and when a glossy surface is desired, under such aspects have to be called outstanding. There is, however, a certain limitation by a bluish gleam on the surface which appears after a certain period of time.

The whole of the other compounds with blends of carbon black and mineral fillers, by contrast, exhibit a dull surface without a bluish gleam, see *item 3.2.6*.

In detail, Sillitin N 85 gives rise to good surfaces, but shows some weakness in the formation of the fir tree edge. Sillitin Z 86 with a smooth upper edge und less torn-up lateral edges comes out markedly more favorable. A very good result is obtained with Sillitin P 87 where all edges come out smooth, and the profile leg shows a large distance towards the bottom.

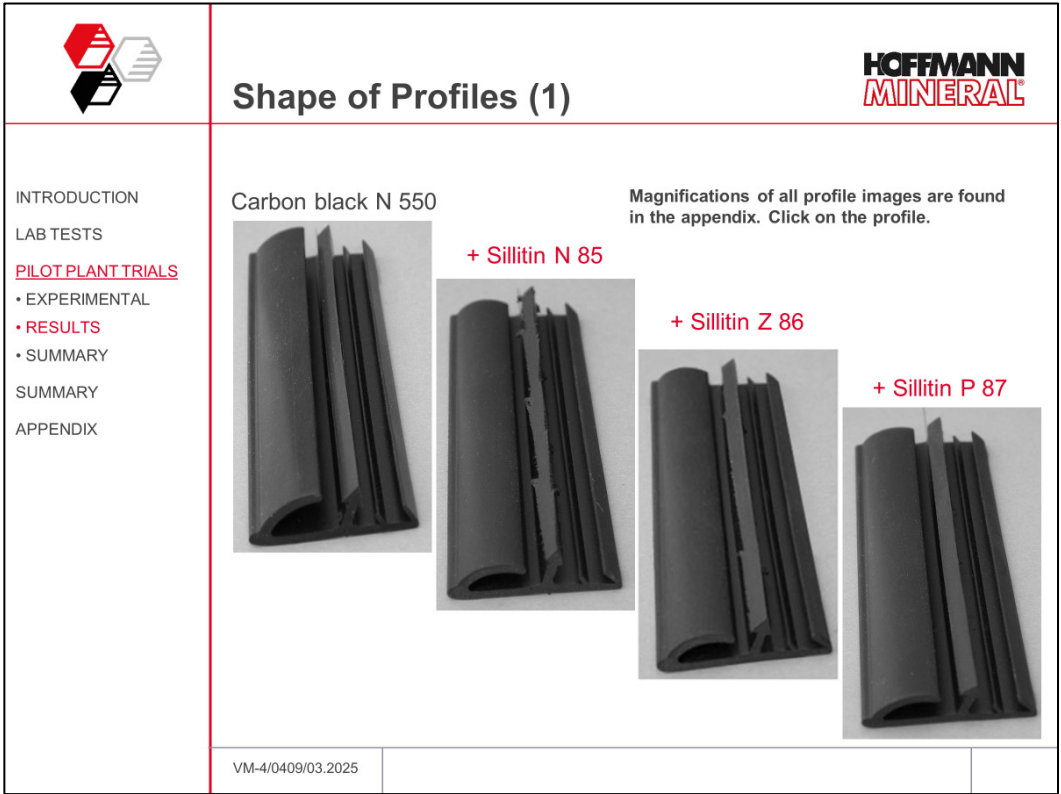
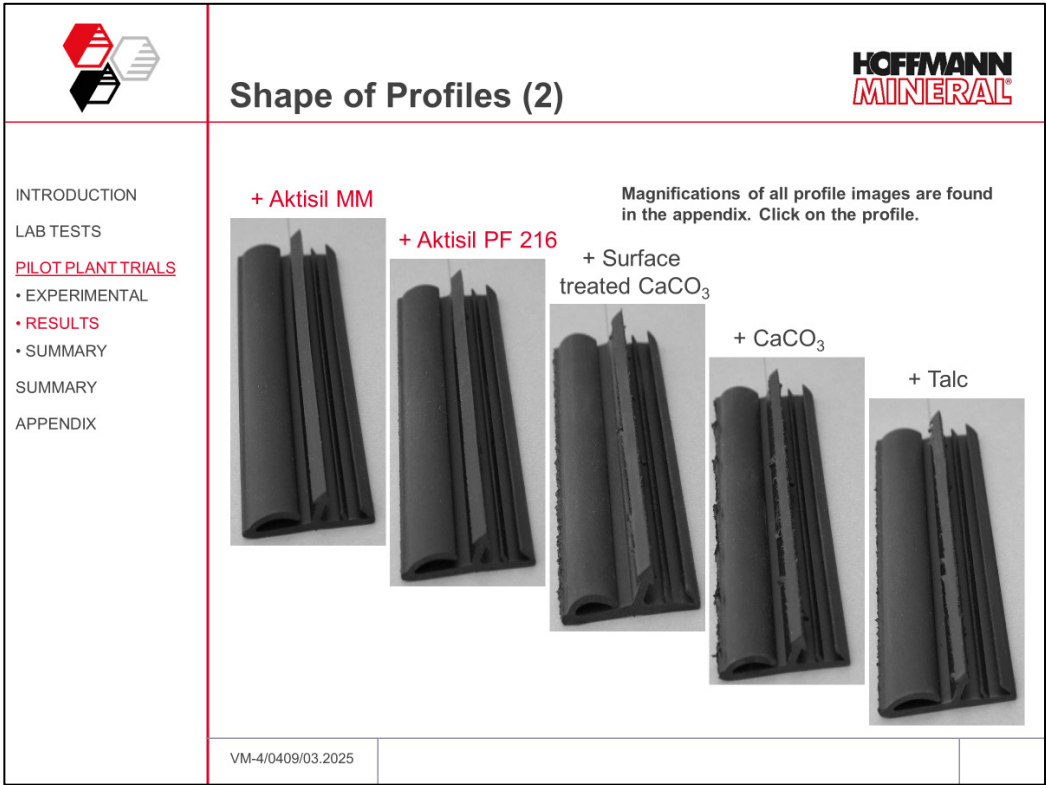


Fig. 40

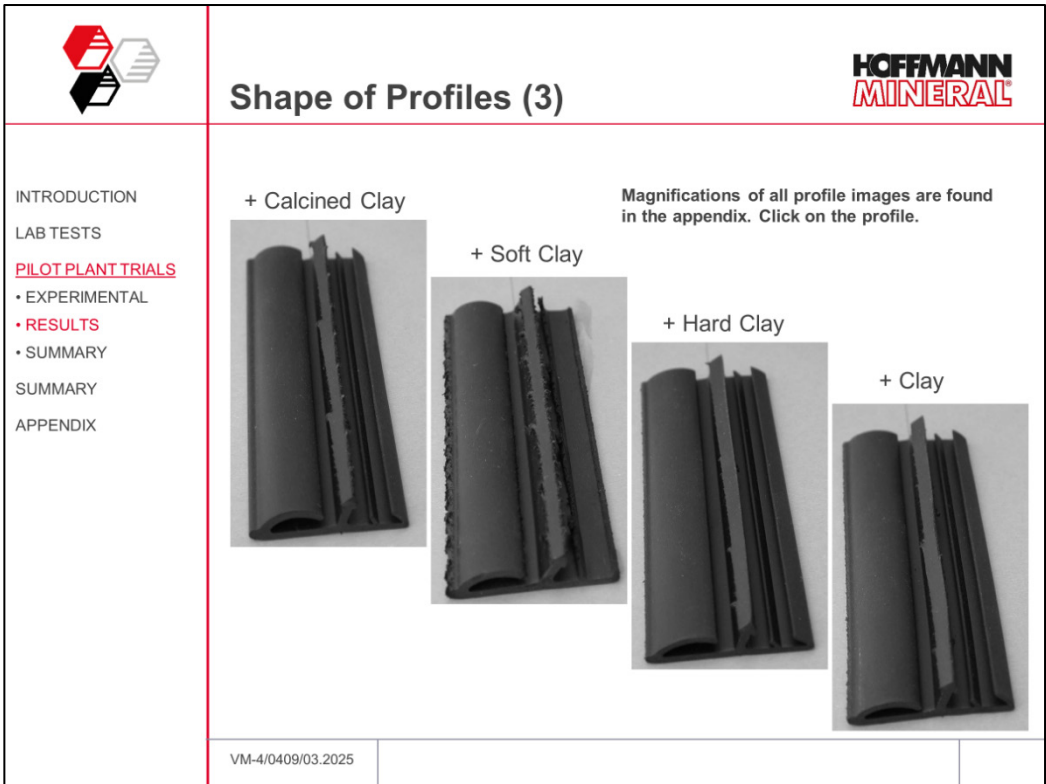
Aktisil MM too on all sides gives smooth edges, but a somewhat smaller distance to the profile leg. Aktisil PF 216 shows slightly rougher edges at the fir tree and at the left outside edge. The surface treated calcium carbonate leads to heavily torn-open profile edges on all sides and a rough surface, above all evident on the profile leg. The untreated calcium carbonate lies somewhat more favorable, similar to the talc whose outside edge is a little less frayed (Fig. 41).



VM-4/0409/03.2025

Fig. 41

As Figure 42 reveals, the calcined clay imparts smooth outer edges, but markedly frayed fir tree edges. With the soft clay, the right-hand lip is no longer developed at all, the whole of the edges are heavily frayed, and the surface is rough throughout. Hardly more favorable rank the hard and the medium-active clay.



VM-4/0409/03.2025

Fig. 42

The field of profile quality as a combination of edge formation and surface quality for the mineral fillers is led by Sillitin P 87 and Aktisil MM, but Sillitin Z 86 and Aktisil PF 216 follow at a short distance. Already Sillitin N 85 positions itself less favorably, and via the calcined and the other clays, talc and calcium carbonate the assessment goes down with soft clay and surface treated calcium carbonate bringing up the rear (Fig. 43).

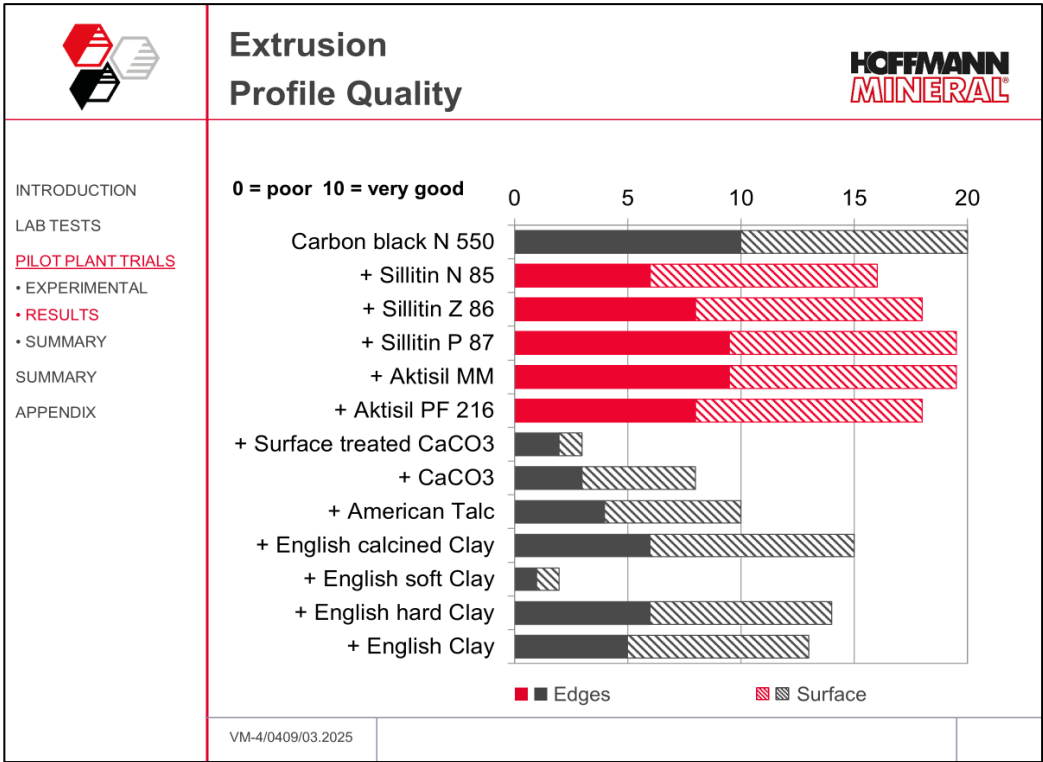


Fig. 43

A porosity of the extruded sections was determined via the density difference between the profiles and press-cured sheets from the same compound. In addition, profile cuts were assessed visually (the ranking is indicated in the bars of the figure). Only the surface treated calcium carbonate gave rise to very pronounced formation of blisters. The soft clay and the untreated calcium carbonate generated fine bubbles. The other fillers practically did not cause any porosity (Fig. 44).

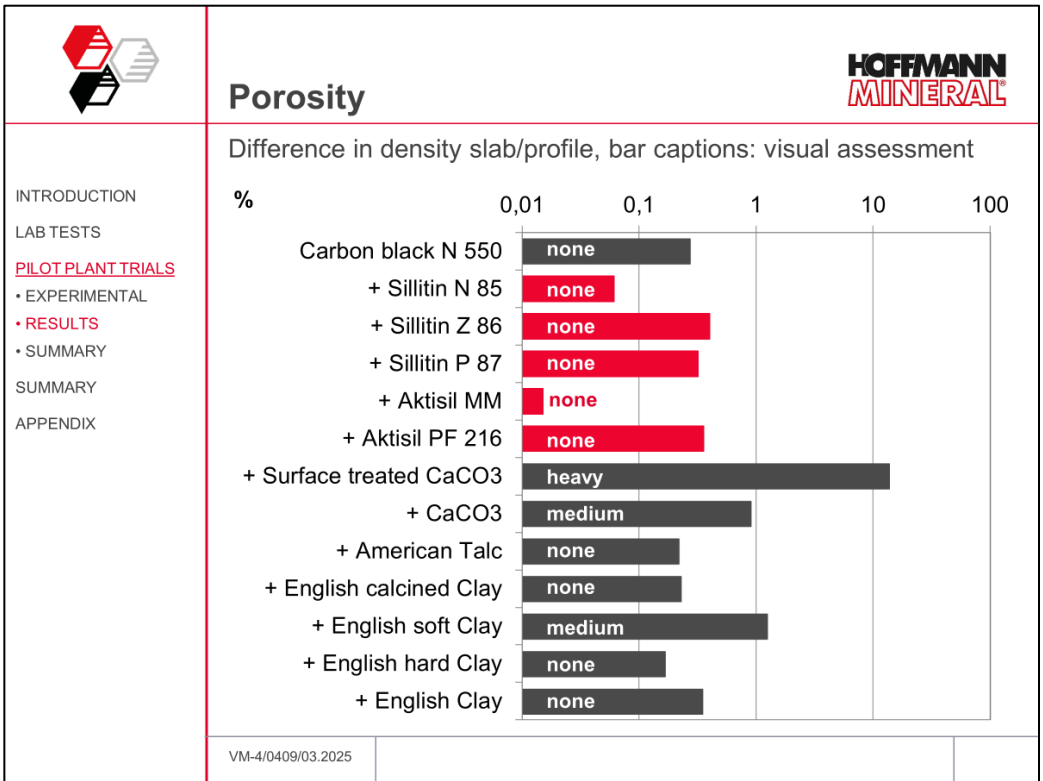


Fig. 44

The dimensional stability or collapse resistance of the sections, measured via the distance between the profile leg and bottom, is brought down with all mineral fillers. Relatively favorable results are obtained with Sillitin P 87, most of the other products are situated in the center span. With the two calcium carbonates and the soft clay, the profile leg even falls down to the bottom (Fig. 45).

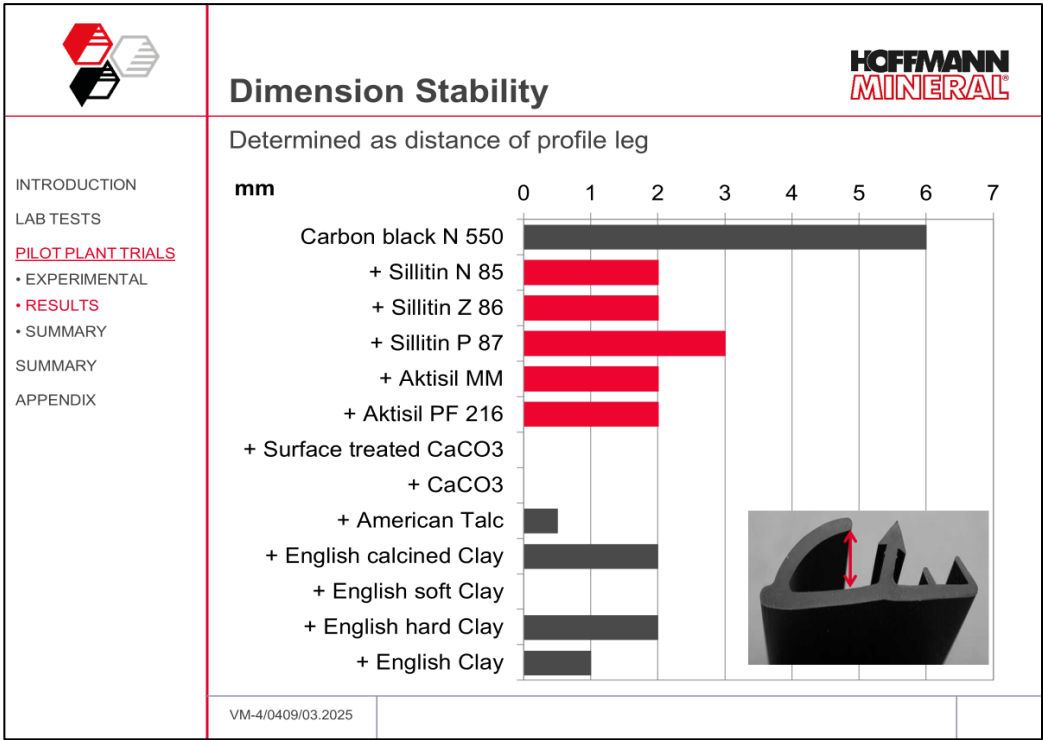


Fig. 45

3.2.6 Optical properties

As expected, the straight carbon black compound shows the highest gloss, while all mineral fillers give rise to a matting effect, which not necessarily means a drawback, as in Europe gloss is not so much desired. Between the mineral fillers in the blends, hardly any differences can be observed. If the corresponding surface quality is also taken into consideration, the calcined clay and in particular the products based on Neuburg Siliceous Earth are to be recommended (Fig. 46).

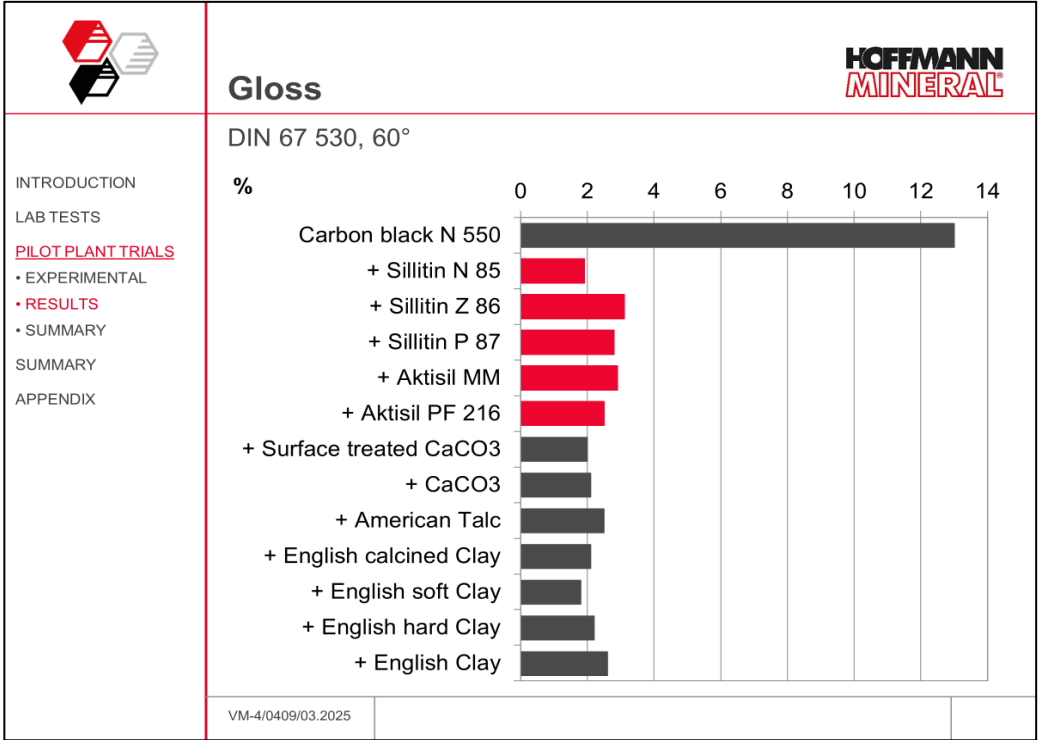


Fig. 46

According to *Figure 47*, all additions of mineral fillers lead to higher L* values compared with the straight carbon black loading, which means they give rise to a somewhat lighter black color. Except for talc with a slightly higher result, all other products are found nearly at the same level. The test system chosen to some extent also makes a correction for the existing gloss, but small effects onto the measurement results cannot be excluded.

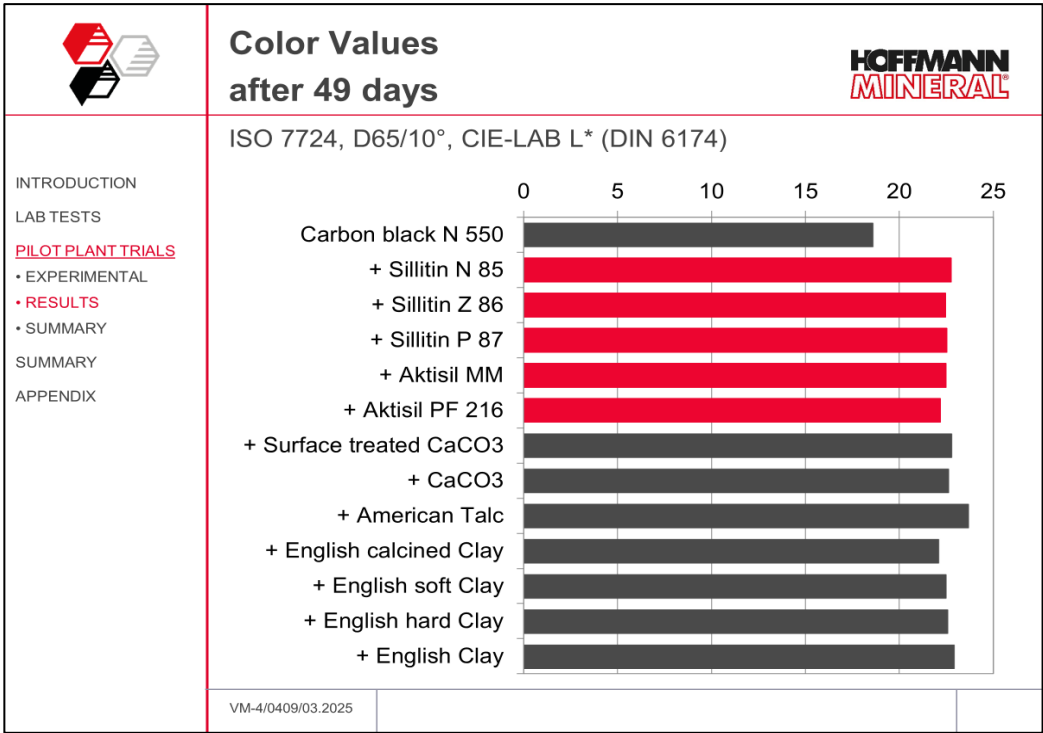


Fig. 47

The a* and b* values indicate the varied colors, starting from 0 for completely uncolored, i.e. according to the L* value white, grey or black. a* stands for the green/red, b* for the blue/yellow axis. The more a* and b* come out on the negative side, the more the color moves towards green/blue. A greatly negative figure for b*, therefore, is an indication of a marked bluish gleam on the extrusions. All mineral fillers tend to shift the b* results in the direction of the zero axis, which means they decrease the bluish gleam of the straight carbon black compound. The differences between the individual products most likely can be explained by purely statistical variation (*Fig. 48*).

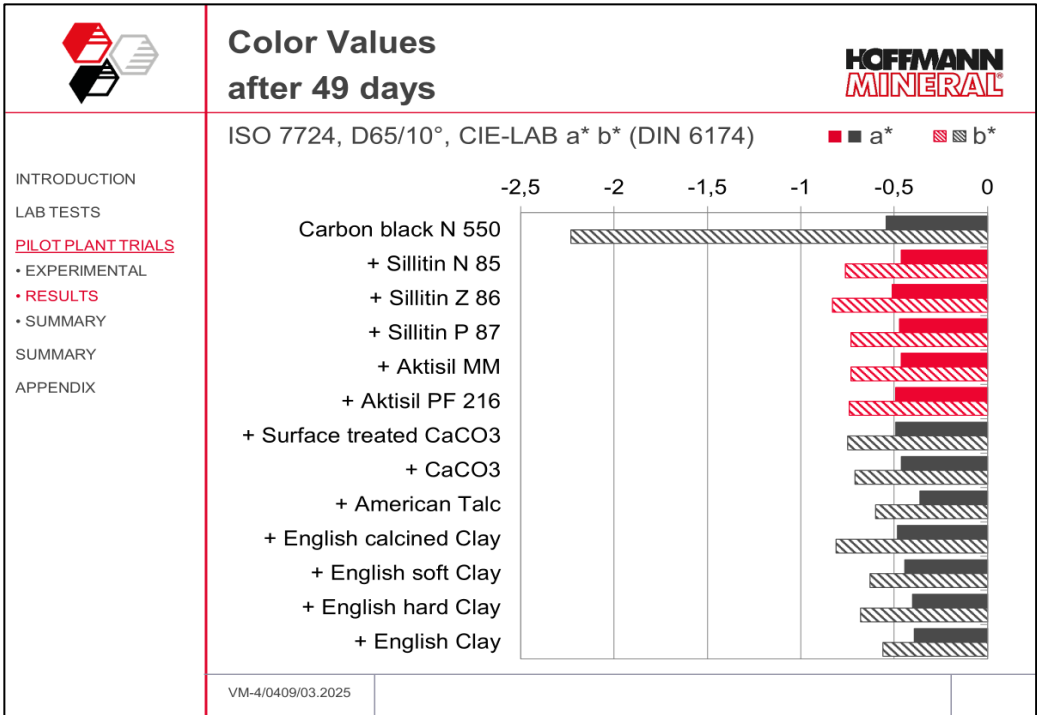


Fig. 48

3.3 Summary of results

Fig.49 and Fig. 50 summarize the results of the pilot plant trials.



	Pilot Plant Trials Summary (1) 
<p>INTRODUCTION</p> <p>LAB TESTS</p> <p><u>PILOT PLANT TRIALS</u></p> <ul style="list-style-type: none"> • EXPERIMENTAL • RESULTS • SUMMARY <p>SUMMARY</p> <p>APPENDIX</p>	<ul style="list-style-type: none"> • The calcium carbonates exhibit their main weak points in the mechanical properties as well as during extrusion and in electrical resistivity, however they impart good compression set resistance. • Talc gives good mechanical properties, but unsatisfactory compression set and moderate extrusion properties. • Calcined clay is characterized by moderate mechanical properties, medium extrusion properties and good compression set along with high electrical resistivity. • Soft clay comes off with very poor extrusion properties and moderate mechanical properties und also poor compression set resistance.
	<div>VM-4/0409/03.2025</div>

Fig. 49



	Pilot Plant Trials Summary (2) 
<p>INTRODUCTION</p> <p>LAB TESTS</p> <p><u>PILOT PLANT TRIALS</u></p> <ul style="list-style-type: none"> • EXPERIMENTAL • RESULTS • SUMMARY <p>SUMMARY</p> <p>APPENDIX</p>	<ul style="list-style-type: none"> • Hard clay imparts good mechanical properties und moderate extrusion properties, but there are definite weak points relative to compression set. • Medium activity clay comes close to the hard clay, but only reaches moderate mechanical properties along with higher electrical resistivity. • The Neuburg Siliceous Earth grades distinguish themselves above all by their excellent extrusion properties, high electrical resistivity and good compression set along with high tear resistance. • Particularly recommended are Aktisil MM and Aktisil PF 216. These grades impart very good extrusion properties in combination with high tensile moduli and excellent compression set results.
	<div>VM-4/0409/03.2025</div>

Fig. 50

4 Conclusion and outlook


	<div> <div>Conclusion</div> <div>HOFFMANN MINERAL®</div> </div>	
<div> <div>INTRODUCTION</div> <div>LAB TESTS</div> <div>PILOT PLANT TRIALS</div> <div>SUMMARY</div> <div>APPENDIX</div> </div>	<p>The basic statement from the laboratory tests largely also apply to the pilot plant trials which were close to production conditions.</p> <ul style="list-style-type: none"> • With mineral fillers electrically insulating car body seals can be produced, which with the exception of lower tensile strength and partly less optimum extrusion properties do not give evidence of poorer performance levels. Some of the compounds do already meet the requirements of pertinent specifications. • A further optimization can be visualized via the loading ratio of carbon black to mineral filler, as in view of the partly very high resistivity there is still room for a higher carbon black loading. In this context, also the volume resistivity after water immersion should be taken into account. 	
	VM-4/0409/03.2025	

Fig. 51

Acknowledgement

The pilot plant mixing and the profile extrusion reported above were carried out, as mentioned, at the facilities of DSM Elastomers Europe in Geleen. We owe DSM sincere thanks and appreciation for an excellent cooperation.

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.

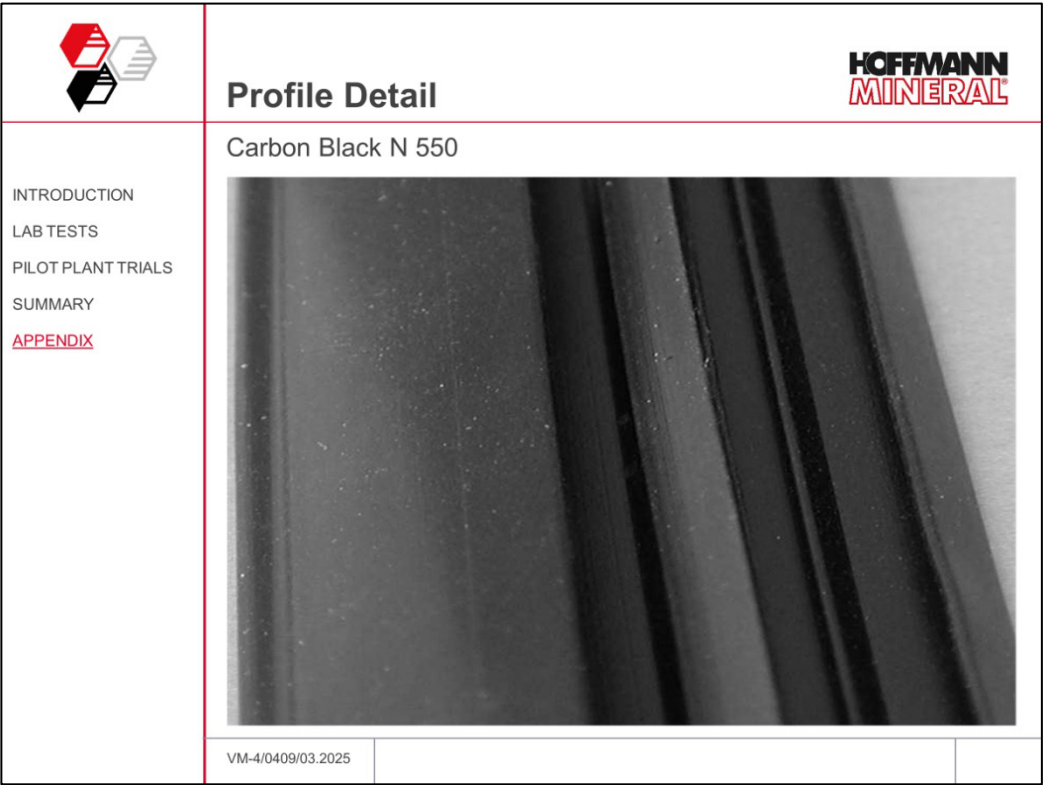


Fig. 52

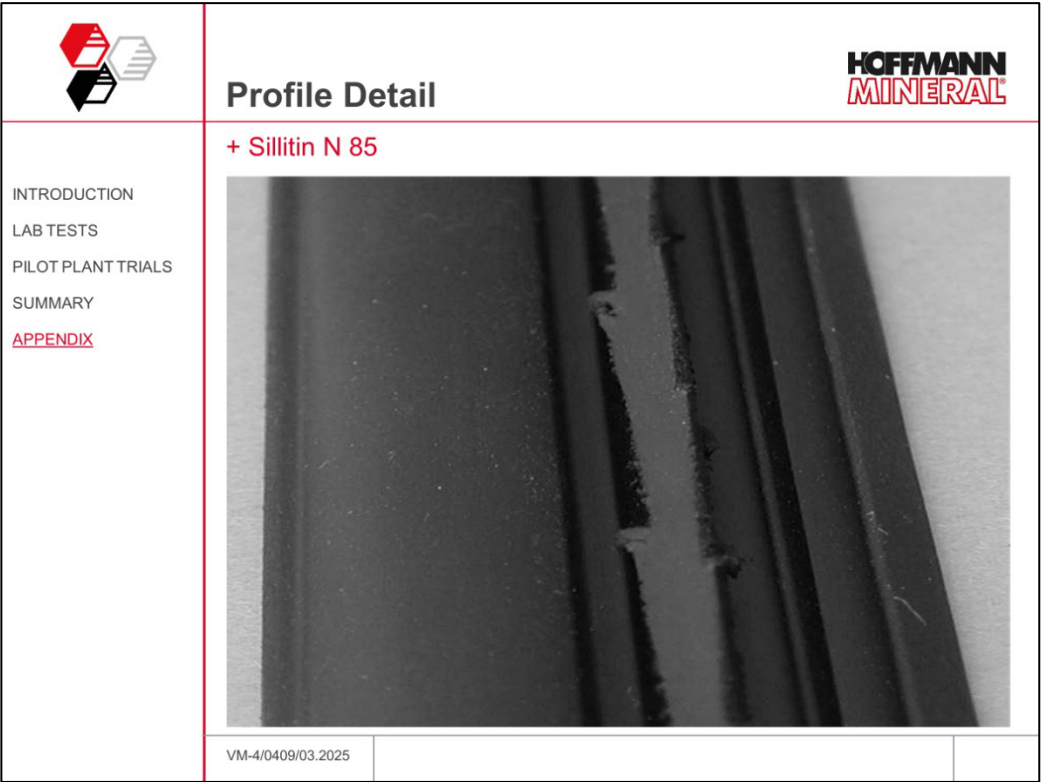


Fig. 53

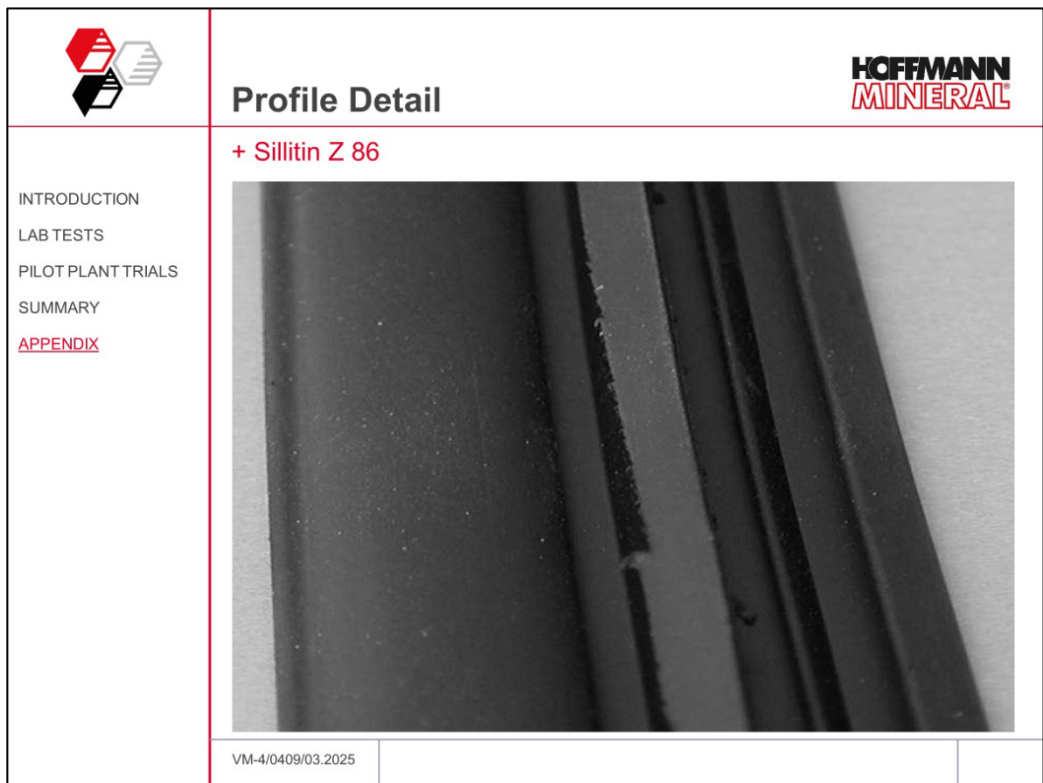


Fig. 54

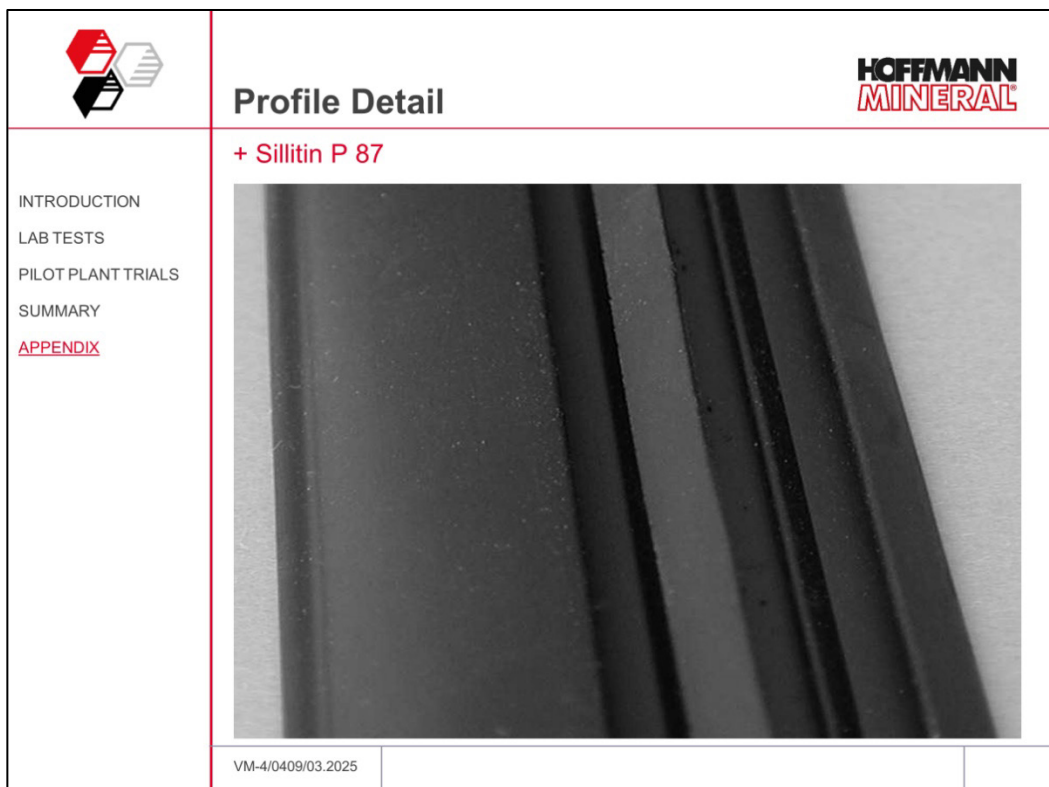


Fig. 55

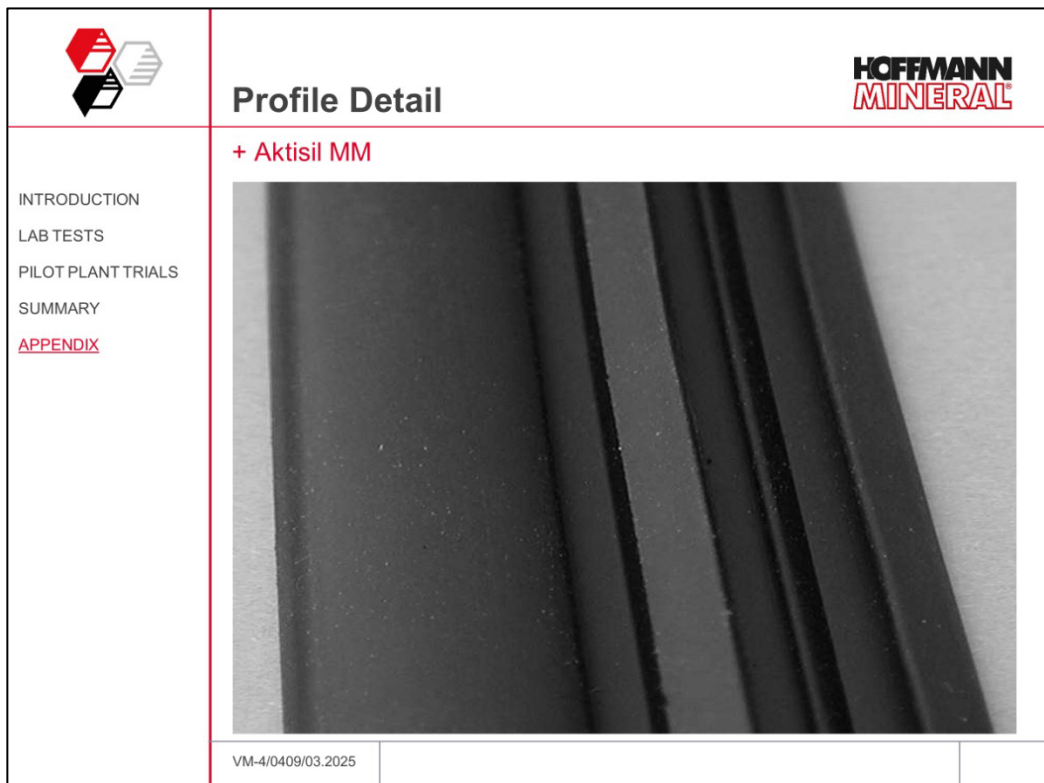


Fig. 56

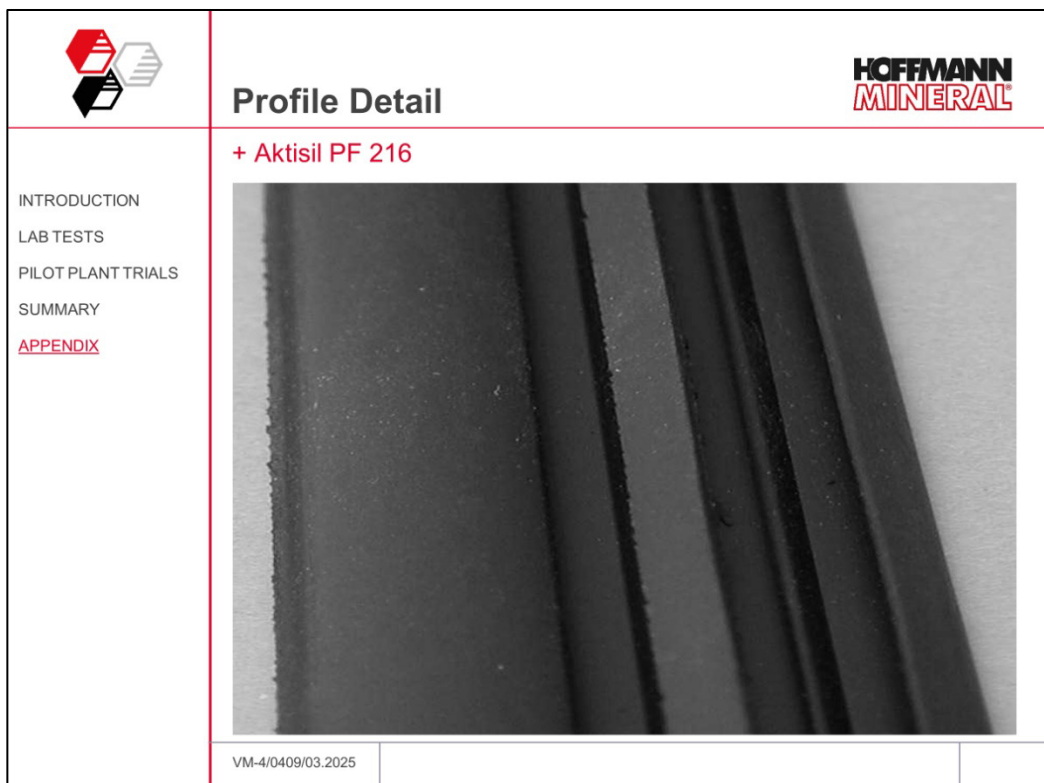


Fig. 57

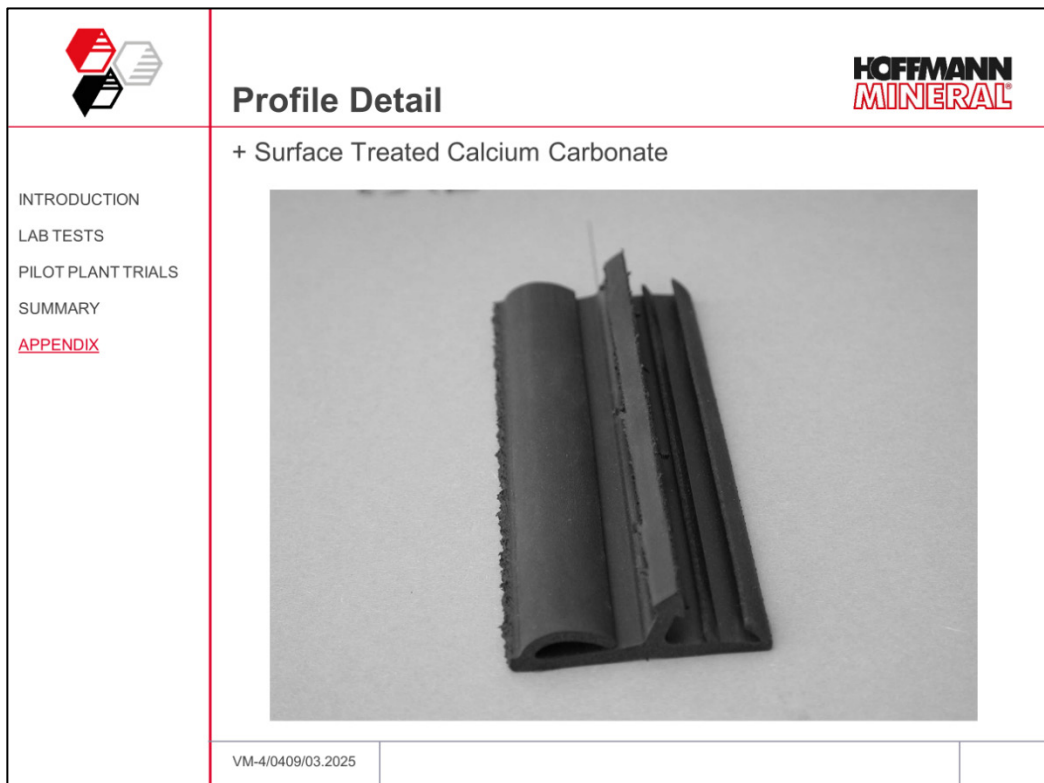


Fig. 58

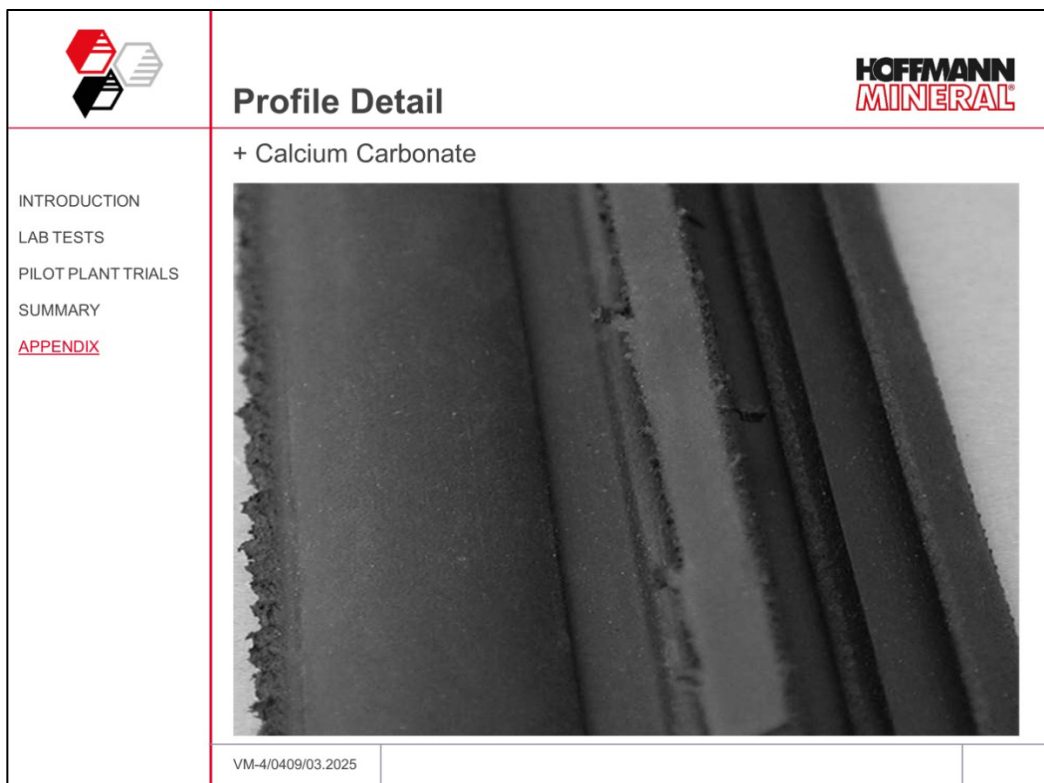


Fig. 59

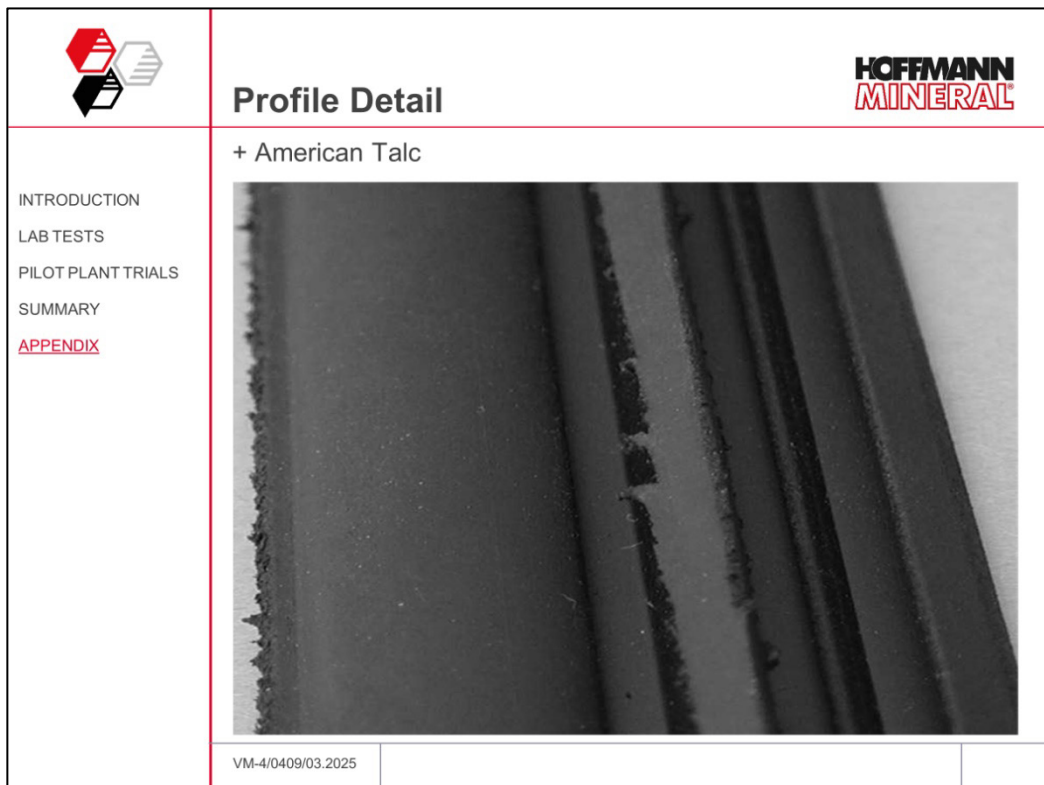


Fig. 60

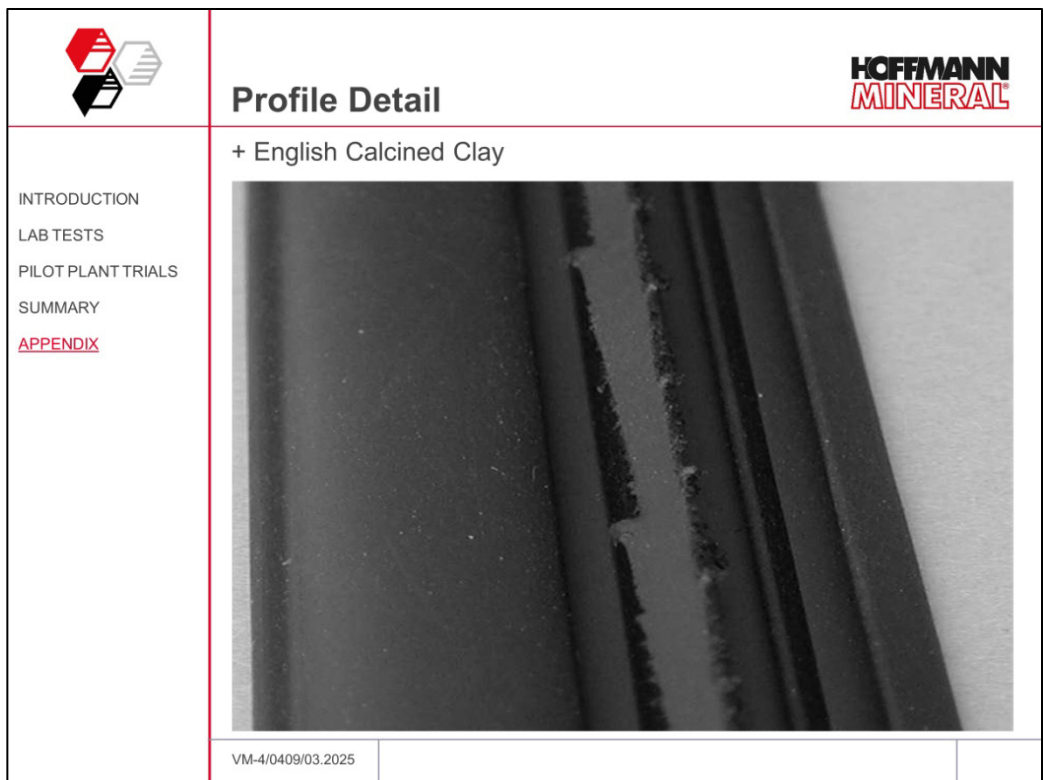


Fig. 61

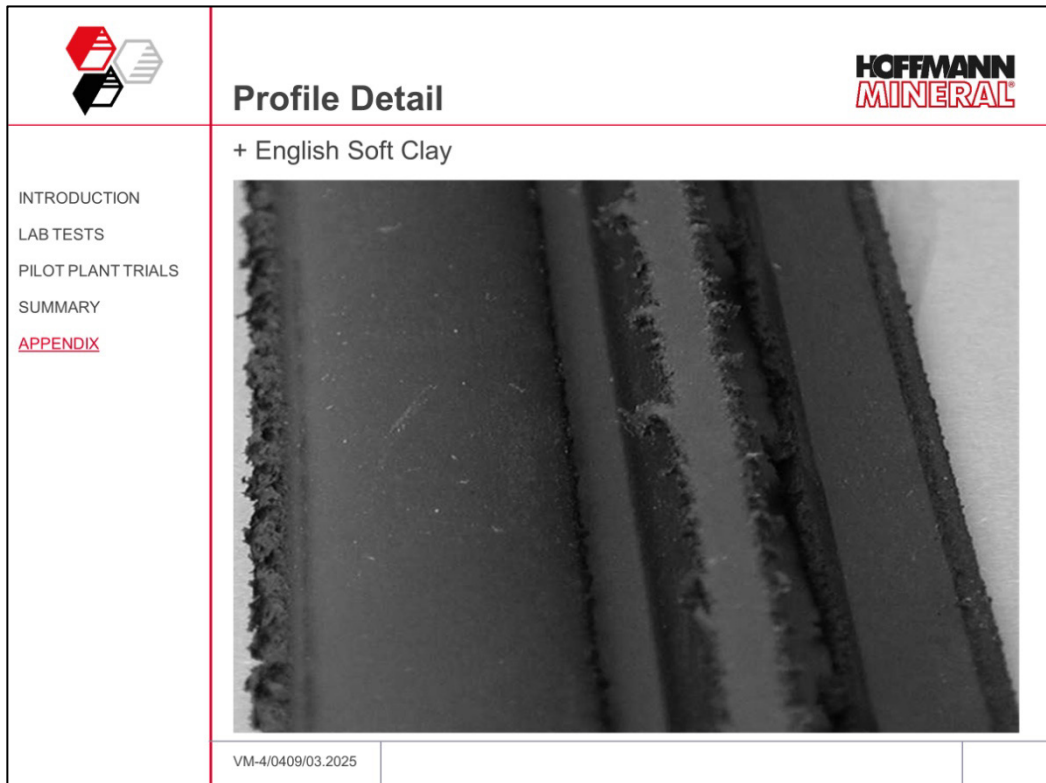


Fig. 62

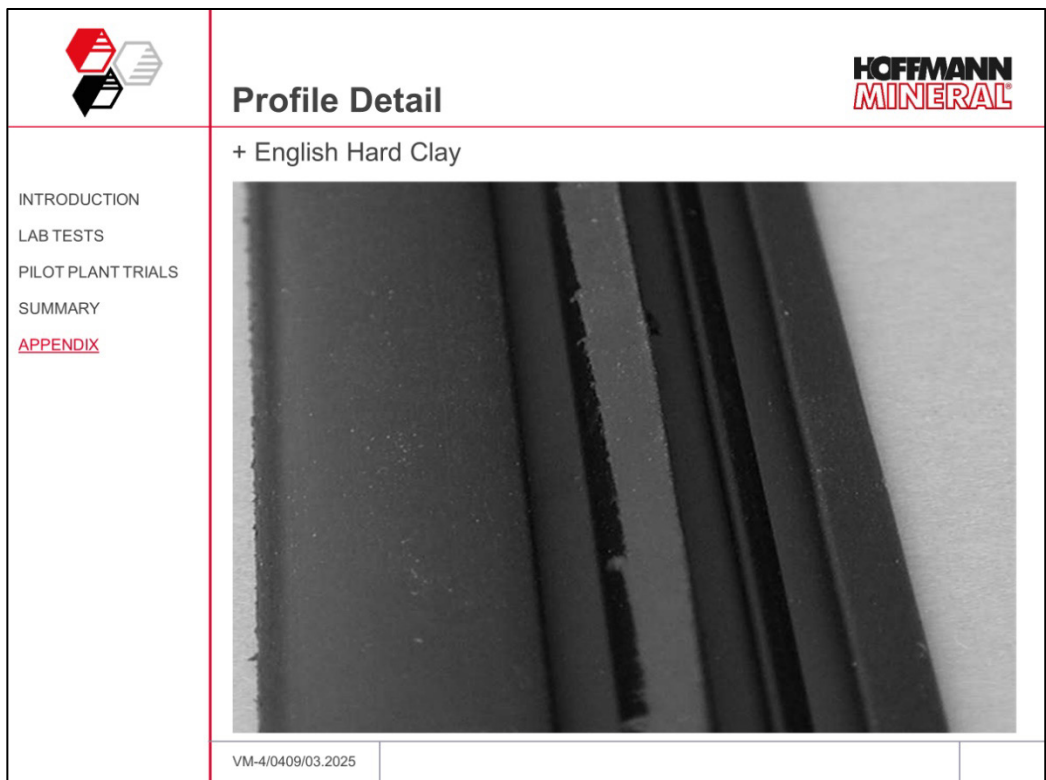


Fig. 63


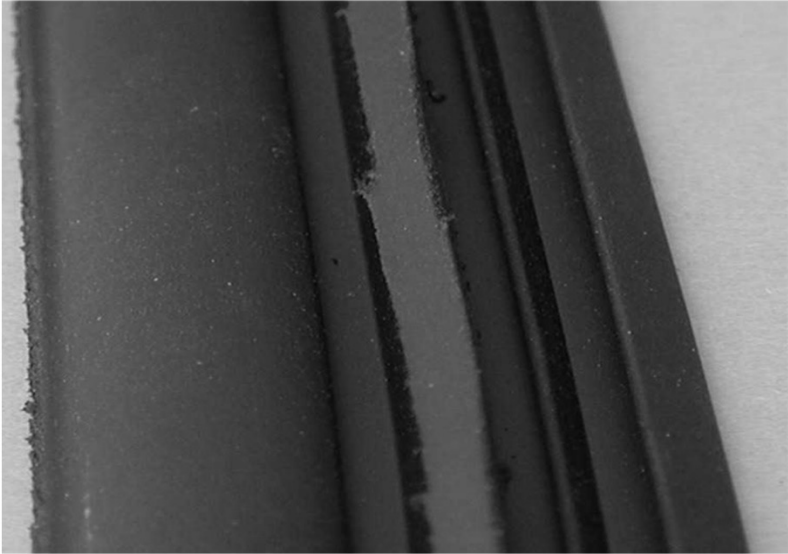
	<div> <div> Profile Detail </div> <div> HOFFMANN MINERAL </div> </div>
<div> <div>INTRODUCTION</div> <div>LAB TESTS</div> <div>PILOT PLANT TRIALS</div> <div>SUMMARY</div> <div>APPENDIX</div> </div>	<div> <div>+ English Clay</div> <div>  </div> <div> <div>VM-4/0409/03.2025</div> <div></div> <div></div> </div> </div>

Fig. 64