

Neuburg Siliceous Earth
in adhesives based on
silane-terminated polyether
e. g. parquet adhesives

Author: Petra Zehnder
Hubert Oggermüller

Contents

- 1 Introduction

- 2 Experimental
 - 2.1 Base formulation
 - 2.2 Fillers and their characteristics
 - 2.3 Test design
 - 2.4 Preparation of batches

- 3 Results
 - 3.1 Value-driven approach
 - 3.1.1 Viscosity
 - 3.1.2 Skin formation time and cure rate
 - 3.1.3 Mechanical properties
 - 3.1.4 Storage stability
 - 3.1.5 Conclusion: value-driven approach
 - 3.2 Cost-driven approach
 - 3.2.1 Viscosity
 - 3.2.2 Skin formation time and cure rate
 - 3.2.3 Mechanical properties
 - 3.2.4 Storage stability
 - 3.2.5 Raw material costs
 - 3.2.6 Conclusion: cost-driven approach
 - 3.3 Maximum performance
 - 3.3.1 Viscosity
 - 3.3.2 Skin formation time and cure rate
 - 3.3.3 Mechanical properties
 - 3.3.4 Storage stability
 - 3.3.5 Conclusion: maximum performance approach

- 4 Suggestions: fillers and start formulations

- 5 Summary

1 Introduction

Aside from the widely introduced silicone and polyurethane systems, also innovative hybrid polymers based on silane-terminated polyethers are able to meet the requirement profile for modern adhesive and sealant applications.

Here it is possible to formulate adhesives and sealants which are free from solvents, isocyanates and tin, therefore healthwise and ecologically safe, and which in addition are distinguished by excellent adhesion and outstanding mechanical properties.

The present study will present Neuburg Siliceous Earth and Calcined Neuburg Siliceous Earth grades as functional fillers for (parquet) adhesives based on a silane terminated polyether.

The objective was to improve the strength of the adhesive and take advantage of this effect for upgrading traditional compounds formulated with the established filler calcium carbonate.

2 Experimental

2.1 Base Formulation

The starting point of the study was a guide formulation from Wacker Chemie. The main ingredients are a silane terminated polyether as the polymer, and polypropylene glycol as plasticizer. The standard filler was natural calcium carbonate. Vinyl silane is used as a chemical drying agent, and amino silane as adhesion promoter. The rheology control was effected with a hydrophobic fumed silica.

| | | Base Formulation | | HOFFMANN MINERAL |
|---|--------------------|--|--|---------------------------------|
| INTRODUCTION <u>EXPERIMENTAL</u> RESULTS SUMMARY APPENDIX | | | | parts or % by weight |
| | GENIOSIL® STP-E 10 | Polymer Silane-terminated polyether | | 25.5 |
| | Caradol ED 56-200 | Plasticizer Polypropylene glycol | | 15.0 |
| | GENIOSIL® XL 10 | Drying agent Vinyl silane | | 2.0 |
| | HDK H 18 | Rheological additive Fumed silica | | 2.5 |
| | GCC | Filler Ground calcium carbonate | | 54.0 |
| | GENIOSIL® GF 96 | Adhesion promoter Amino silane | | 1.0 |
| | Total | | | 100.0 |
| | | VM-05/0610/02.2019 | | |

2.2 Fillers and their Characteristics

The table summarizes the most important typical properties of the fillers.

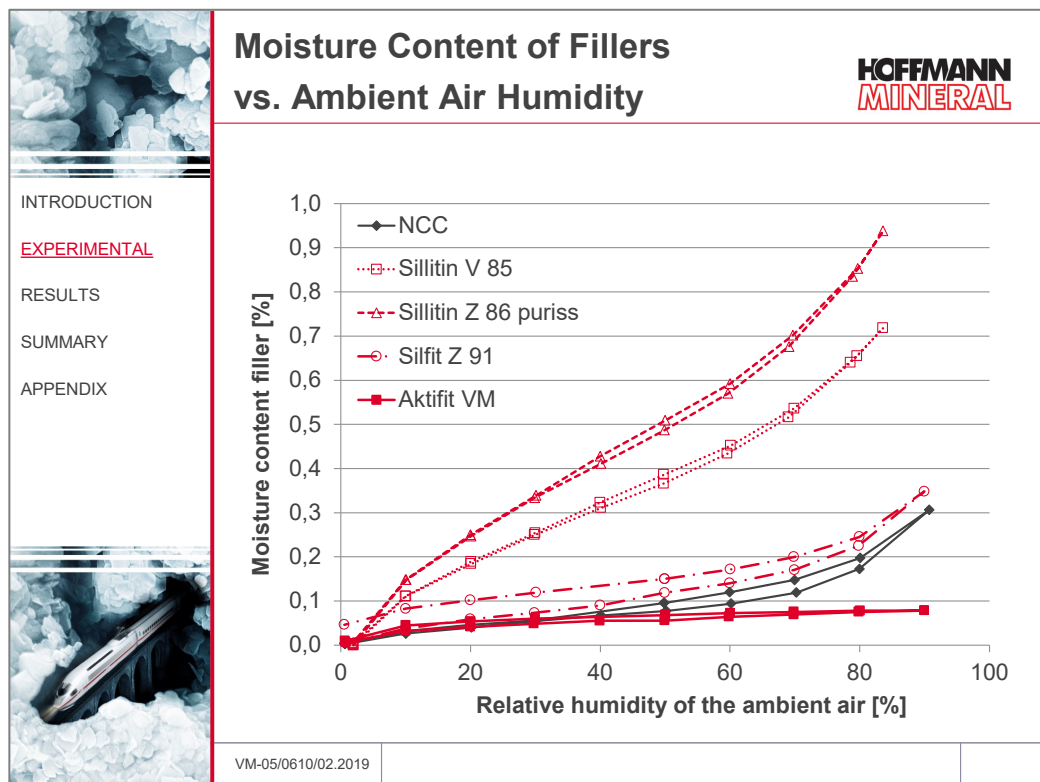
| | | Fillers Characteristics | | | | | HOFFMANN MINERAL |
|---|-------------------------------|------------------------------------|-------------|------------------------------------|-------------------------------------|---|-----------------------------|
| INTRODUCTION <u>EXPERIMENTAL</u> RESULTS SUMMARY APPENDIX | | | GCC | Neuburg Siliceous Earth | | Calcined Neuburg Siliceous Earth | |
| | | | | Sillitin V 85 | Sillitin Z 86 puriss | Silfit Z 91 | Aktifit VM |
| | Volatile matter at 105 °C | [%] | 0.1 | 0.4 | 0.6 | 0.2 | 0.1 |
| | Particle size d ₅₀ | [µm] | 5 | 4 | 1.8 | 2 | 2 |
| | Particle size d ₉₇ | [µm] | 21 | 18 | 8 | 10 | 9 |
| | Oil absorption | [g/100g] | 20 | 40 | 50 | 57 | 52 |
| | Specific Surface Area BET | [m ² /g] | 2.4 | 7 | 10 | 7.9 | 7.9 |
| | Surface treatment | | --- | --- | --- | --- | Vinyl silane |
| | | | hydrophilic | | | | hydro- phobic |
| | | VM-05/0610/02.2019 | | | | | |

The natural calcium carbonate included in the base formulation is a marble flour of average fineness (d_{50} : 5 μm).

In line with the grain size of the competitive product, Sillitin V 85 was chosen to work with as the coarsest grade of Neuburg Siliceous Earth, although here already a higher specific surface area and oil number are present. All other Neuburg Siliceous Earth grades have a higher fineness and a similar or even higher oil number and a higher specific surface area.

Sillitin V 85 and Sillitin Z 86 puriss count among the standard siliceous earth grades. Silfit Z 91 and Aktifit VM are calcined versions, and therefore offer more pronounced brightness and color neutrality. Further differences exist with respect to surface characteristics and surface treatment.

The standard siliceous earth grades and Silfit Z 91, as untreated products, have to be regarded as hydrophilic, while the vinyl silane treated Aktifit VM is hydrophobic. It is characterized by a very low volatile content and shows, in addition, an extremely low moisture uptake under humid climatic conditions. The following graph illustrates how the (equilibrium) moisture content of the fillers changes with the humidity of the ambient air.



The curves refer to the moisture uptake with increasing humidity of the surrounding air, as well as the moisture loss with decreasing humidity of the ambient air.

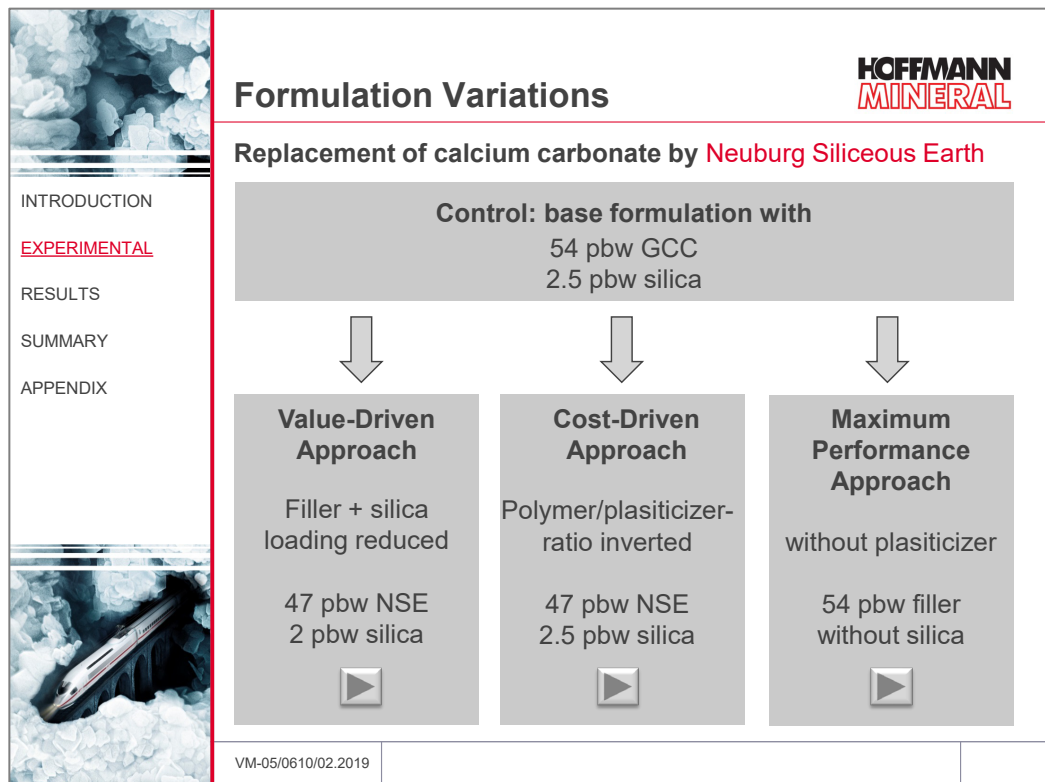
The standard siliceous earth grades give evidence of a distinct dependence of the filler moisture content on the ambient climate, they take up – corresponding to their BET surface area – relatively much moisture, but give it off again under drying conditions to the same extent. The calcined Silfit Z 91 under dry ambient conditions shows a markedly lower moisture content which only increases with higher ambient air humidity.

Particular attention, however, deserves the calcined and vinyl silane treated Aktifit VM: independent on the climatic conditions its moisture content remains at an almost constant level below 0.1 %, even with extremely high air humidity. This means a pre-drying of the filler prior to its use can be left out.

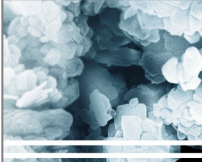
2.3 Test Design


Starting from the base formulation, three different approaches were studied.

- The first part of the tests is oriented towards value determination. In order to keep the viscosity at higher shear with Neuburg Siliceous Earth (NSE) low, the addition of filler and silica will be somewhat reduced (47 resp. 2 parts by weight).
- The second approach goes in the direction of cost orientation. In order to save costs, the polymer portion will be reduced along with an increase of the plasticizer addition, with the result of an inverted ratio between polymer and plasticizer.
- The third variant was supposed to point to maximum performance, i.e. to show which tensile strength and lap shear strength can be obtained by working with Neuburg Siliceous Earth as a reinforcing filler. For this, formulations without plasticizer nor silica are studied.



2.4 Preparation of Batches





Preparation of Batches

- Planetary mixer, two bar blades with scraper
- Cold process (room temperature)
- typical preparation time approx. 10-15 min

INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

APPENDIX

- Feed polymer, plasticizer and drying agent
- Add rheological additive while stirring
- Add filler (not pre-dried) while stirring

Disperse: 2 min at 600 rpm

- Add adhesion promoter


Disperse: 1 min at 600 rpm under vacuum

- Remove compound from the mixing tools

Disperse: 1 min at 600 rpm under vacuum

Deaerate: 1 min at 200 rpm under vacuum

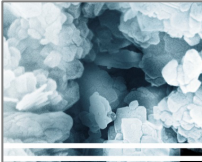
- Fill into cartridge




VM-05/0610/02.2019

3 Results


3.1 Value-Driven Approach





Formulation Variations Value-Driven Approach

| | GCC | | Neuburg Siliceous Earth | |
|----------------------|-----------------|-------------|-------------------------|----------|
| | pbw or weight-% | pbw | pbw | weight-% |
| Polymer | 25.5 | 25.5 | 27.6 | |
| Plasticizer | 15.0 | 15.0 | 16.2 | |
| Drying agent | 2.0 | 2.0 | 2.2 | |
| Rheological additive | 2.5 | 2.0 | 2.2 | |
| Filler | 54.0 | 47.0 | 50.7 | |
| Adhesion promoter | 1.0 | 1.0 | 1.1 | |
| Total | 100.0 | 92.5 | 100.0 | |

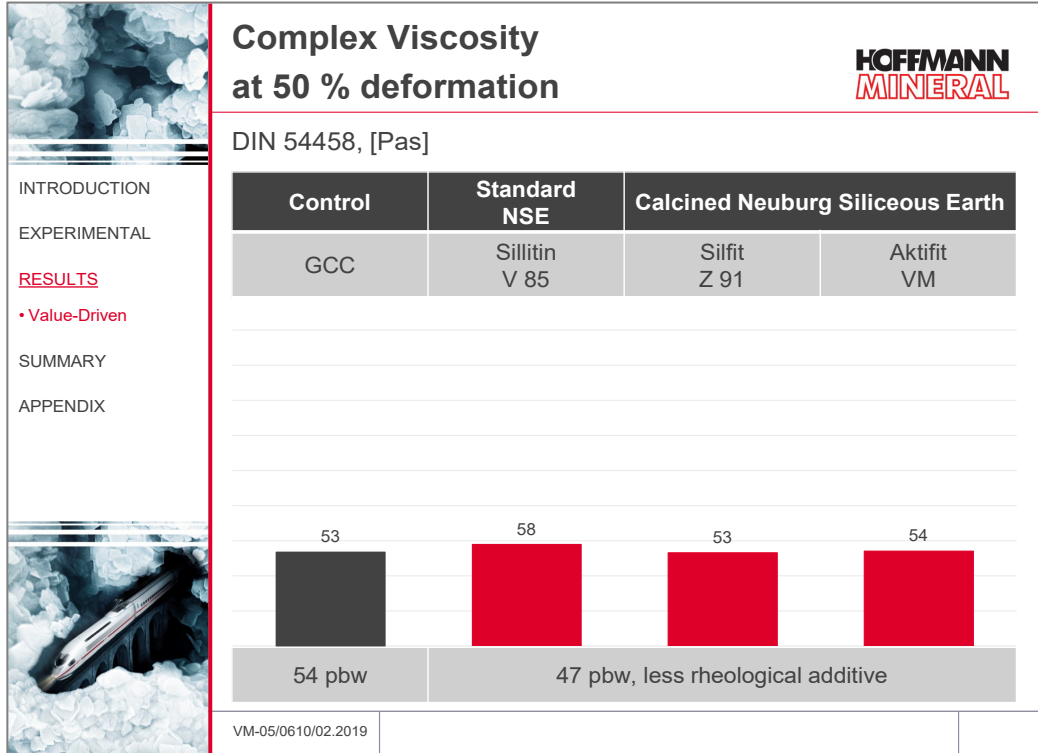


VM-05/0610/02.2019

The table shows the base formulation with ground calcium carbonate and the formulation version adjusted for Neuburg Siliceous Earth with reduced filler and silica content. Among the Neuburg Siliceous Earth grades, Sillitin V 85, Silfit Z 91 and Aktisil VM are included.

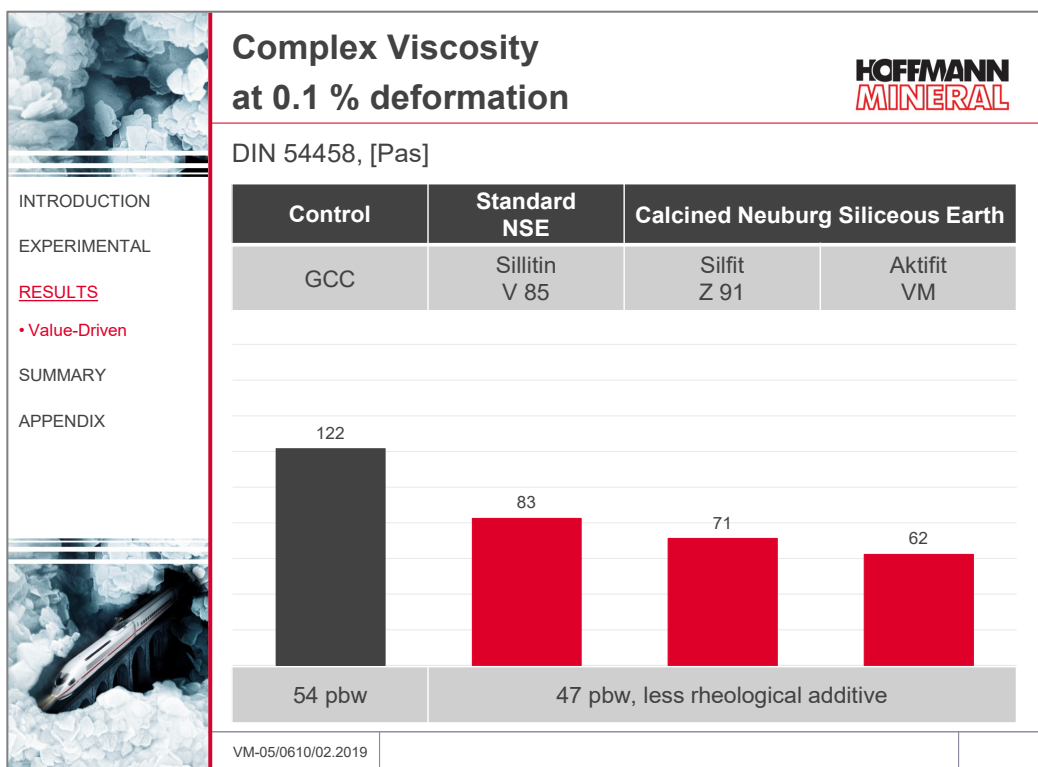
3.1.1 Viscosity

The complex viscosity was measured with a plate-plate rheometer in a deformation controlled oscillation mode at a constant frequency of 10 Hz. The measuring plate had a diameter of 25 mm, the gap distance was 0.5 mm. During the first days after the preparation of the batches, post-wetting effects still give rise to erratic results. For the overall assessment, therefore, the results after 4 weeks following the preparation were taken into account.



All formulations come up with about the same low complex viscosity at larger deformations. For application purposes, this means that all batches are extruded with similar ease as the formulation with GCC.

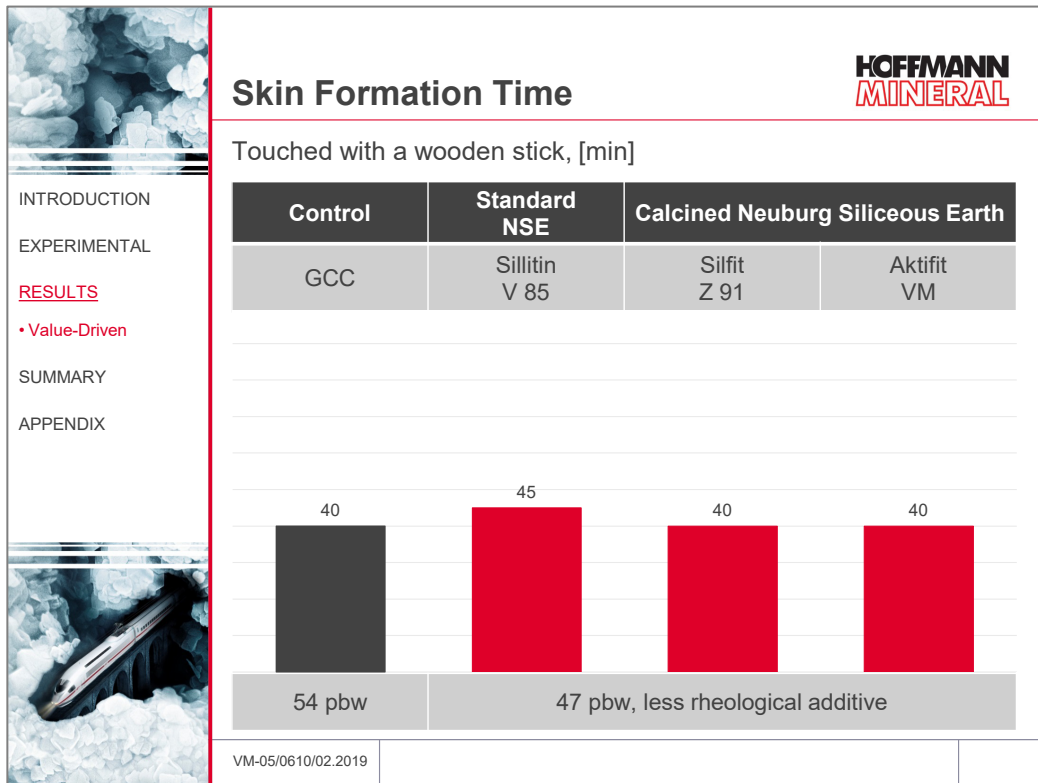
At low deformation, a quasi-static condition is simulated. Here, among others, non-sag properties and the behavior after application can be evaluated.



The Neuburg Siliceous Earth grades at low deformation give a lower complex viscosity than the calcium carbonate. This indicates a stronger flow tendency, which is, however, easily compensated by increasing the silica addition to 2.5 to 3 parts by weight.

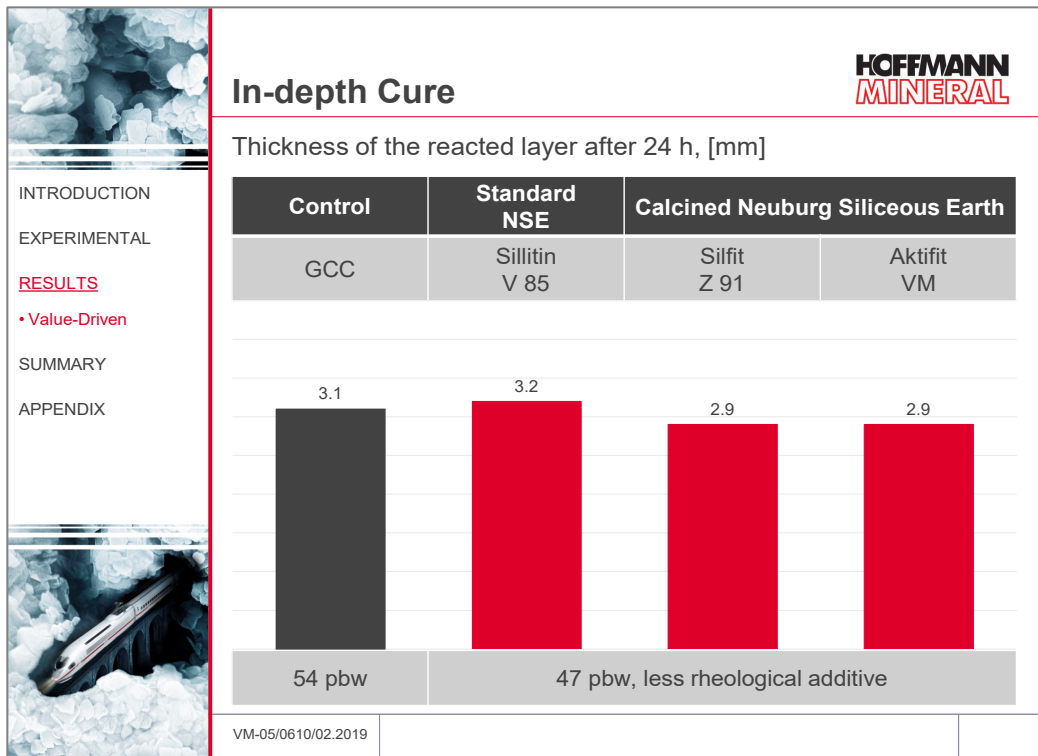
3.1.2 Skin Formation Time and Cure Rate

For the evaluation of skin formation time, the compounds were squeezed out from a cartridge as strands of approx. 10 mm in diameter. Stored at standard conditions 23 °C/50 % r.h, the strands were touched with a wooden stick after definite periods of time. The time after which no material residues remained on the stick, was counted as the skin formation time.



The skin formation time came out very similar for all fillers tested.

In order to measure the cure rate, approx. 4 ml of the batches were filled into small PE containers (Ø 1.8 cm, height 1.5 cm), and the surface was evenly wiped off. After 24 hours of storage at standard conditions 23/50, the cured layer was taken away, still liquid remainders were removed, and the thickness of the cured layer was determined.



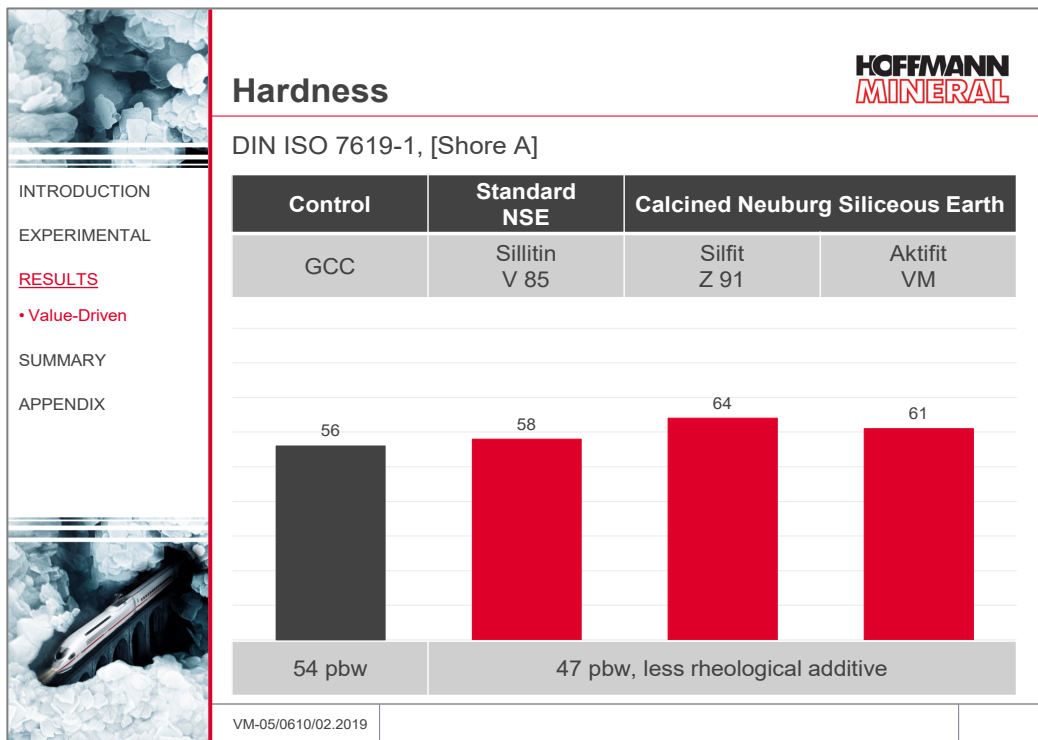
While Sillitin V 85 imparts a similar cure rate as GCC, the thickness of the reacted layer with the Calcined Siliceous Earth grades comes out somewhat lower. The results, however, are always close to each other, so the minimum differences should not be overestimated.

3.1.3 Mechanical Properties

For determining hardness and tensile properties, from a sample sheet of about 2 mm thickness which was cured for four weeks at standard climate 23/50, S2 dumbbells were punched out.

a) Hardness

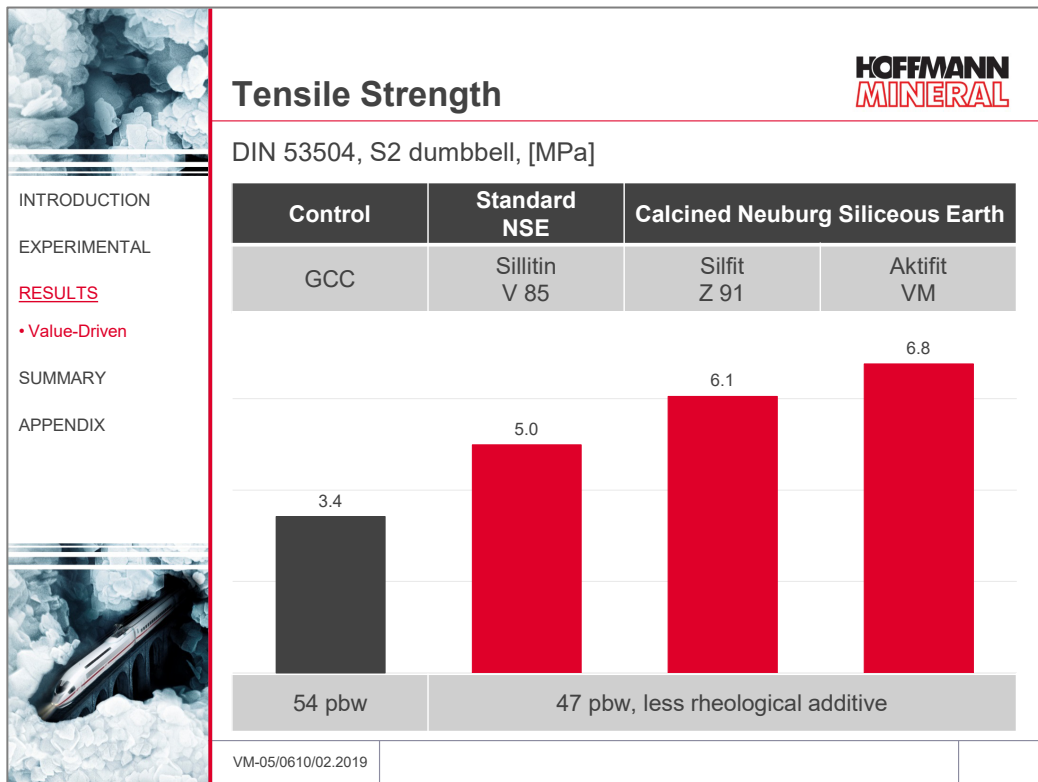
Hardness was measured on piled-up S2 dumbbells (total height approx. 6 mm).



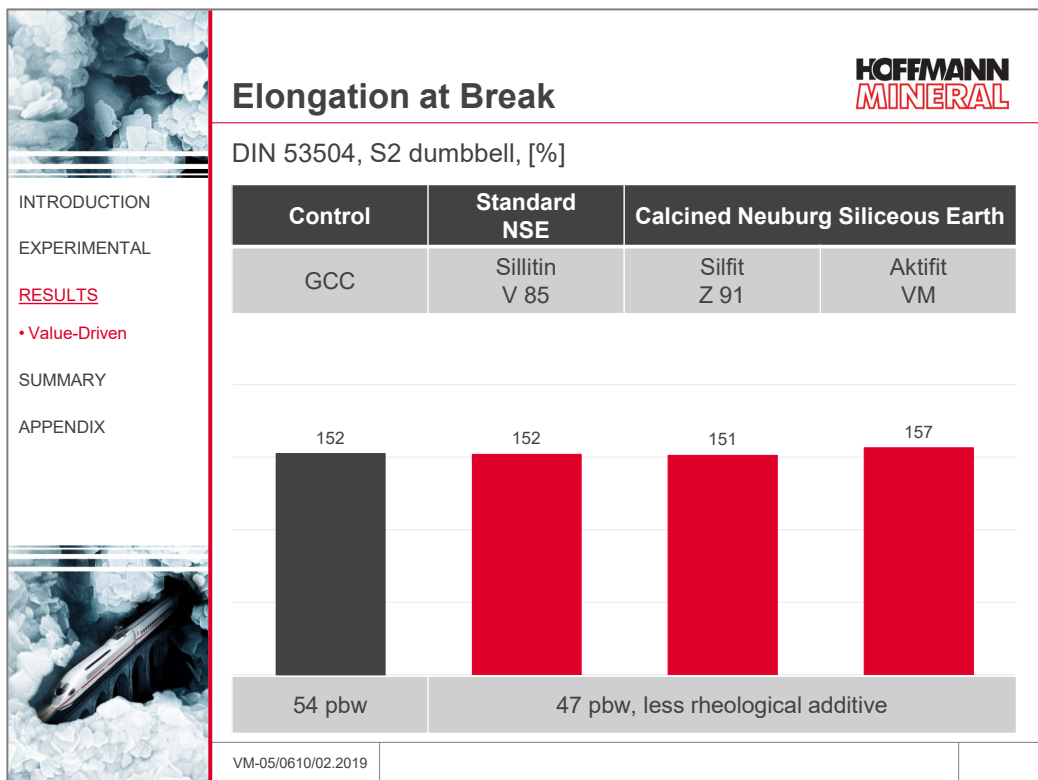
Calcined Neuburg Siliceous Earth – despite reduced filler content – produces a hardness increase.

b) Tensile Properties

The cross-head speed for the tests was 200 mm/min.



Despite the somewhat lower filler loading, Neuburg Siliceous Earth is able to impart a significantly increased tensile strength. Already Sillitin V 85, the coarsest and highly cost effective grade, brings about a marked improvement. With the calcined grades, the tensile strength in comparison to calcium carbonate can partly even be increased by a factor of two.



While the tensile strength is markedly increased with Neuburg Siliceous Earth, the elongation at break at the same time remains at the level of the base formulation with GCC. The reinforcing potential of Neuburg Siliceous Earth comes out in a markedly higher energy absorption capacity up to break, which can be determined as the integral of the stress over the strain (elongation).

c) Lap Shear Strength on Wood

Two oak mosaic parquet slats measuring 160 x 23 x 8 mm were bonded overlapping with a bond surface area of 23 x 26 mm (approx. 600 mm²).

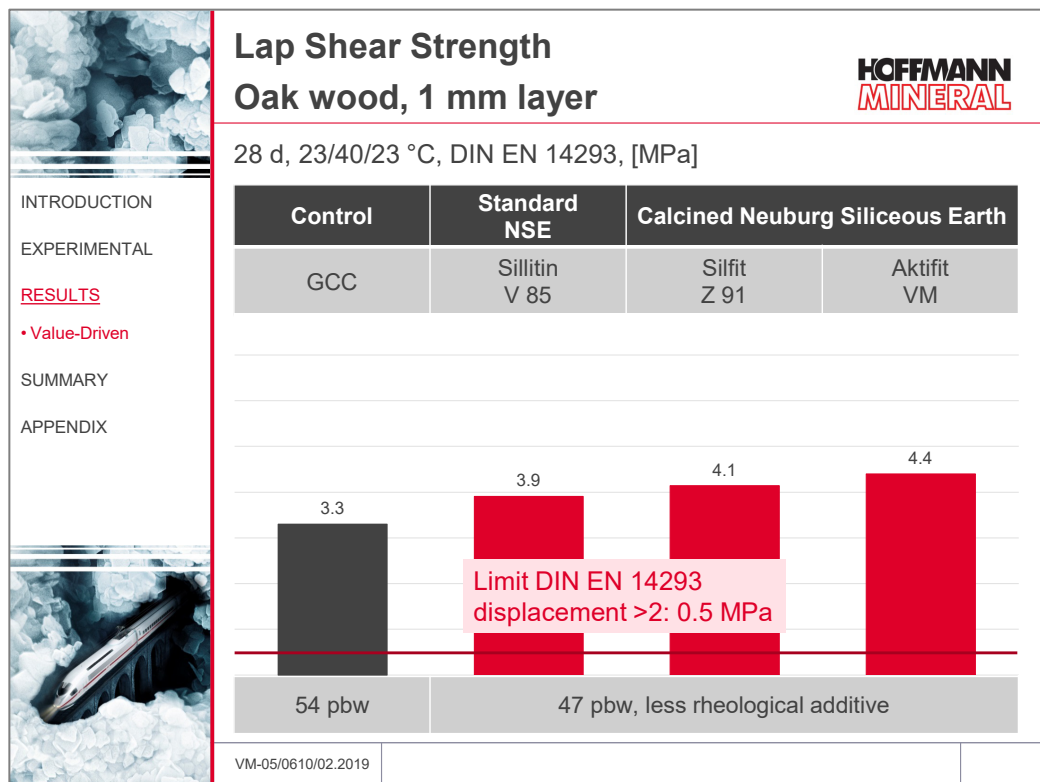
For the evaluation as “hard” adhesive, the sample was applied with a notched trowel (toothing B3 according to TKB6) at a right angle to the longitudinal direction of the slats, and a second slat laid on top. By loading the bond area with 2 kg for 60 s, an adhesive layer thickness of about 0.1 mm was obtained.

For the evaluation as “soft” adhesive, the sample was applied onto the full surface area with a spatula. The adhesive layer thickness of 1 mm was adjusted with the help of suitable spacers when pressing the parquet slats together.

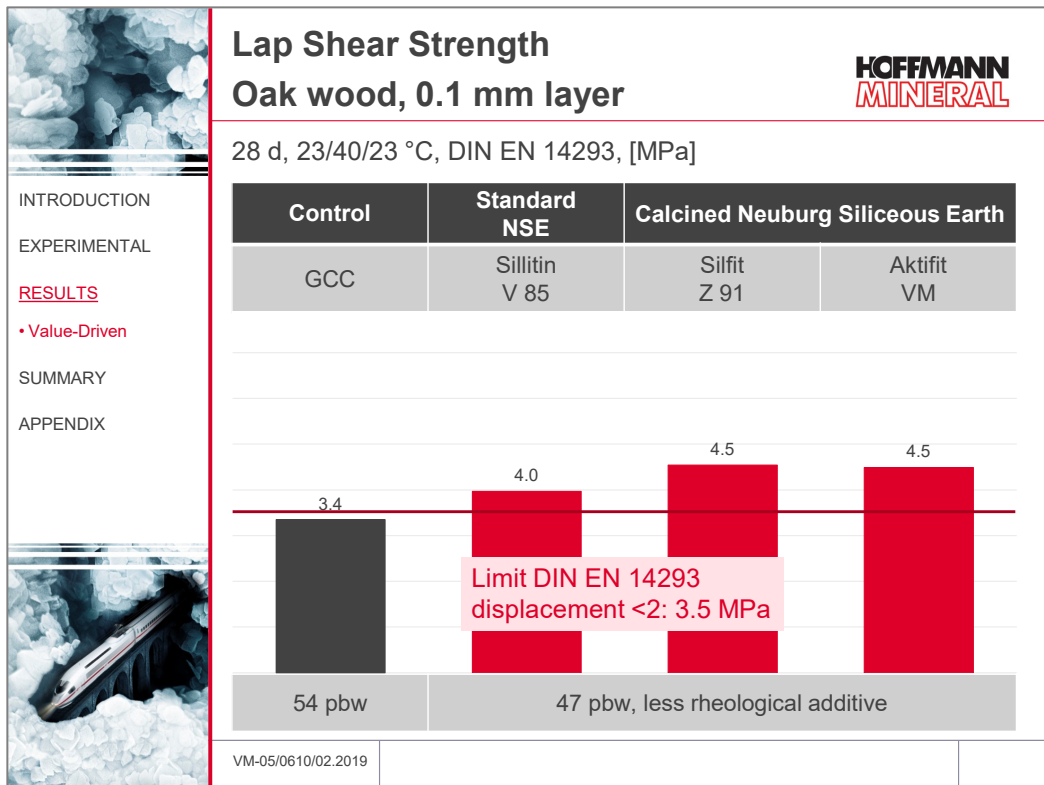
In both tests, excess adhesive residues were removed after the bonding.

The samples with an adhesive layer thickness of 1 mm and half of the samples with an adhesive layer thickness of 0.1 mm were stored for 28 days (7 d at standard climate 23/50 + 20 d at 40 °C + 1 d at standard climate 23/50). For determining the strength properties at an early stage, the other half of the samples with an adhesive layer thickness of 0.1 mm was evaluated already after 3 days at standard climate 23/50.

The test was run according to the standard DIN EN 14293 with a crosshead speed of 20 mm/min.

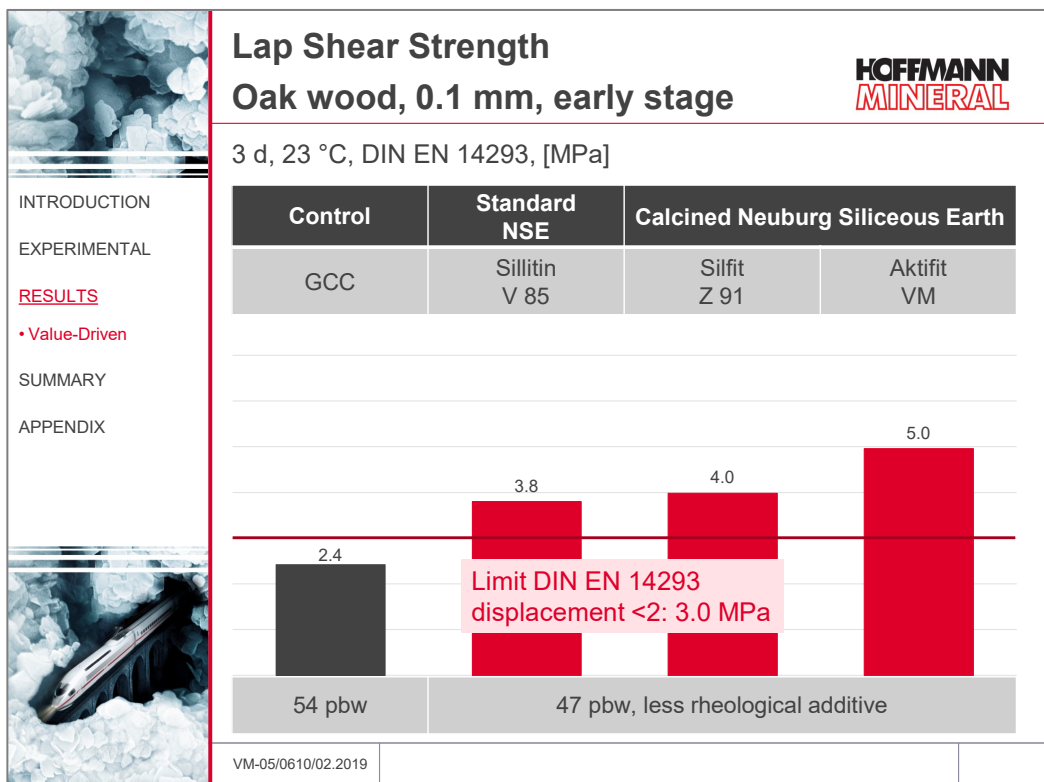


The standard DIN EN 14293 for “soft” parquet adhesives – as defined by a displacement above 2 at an adhesive layer thickness of 1 mm – calls for a lap shear strength of at least 0.5 MPa. This minimum requirement is easily exceeded without problems by all tested formulation variants. All batches gave evidence of good adhesion on the oak wood substrate. The product variants with Neuburg Siliceous Earth distinctly enhance the tensile level with an improvement of up to 33 %.



For “hard” adhesives – defined by a displacement below 2 – the standard specifies a lap shear strength of at least 3.5 MPa after 28 days of curing. Contrary to the control formulation with GCC, which just misses the limit, the batches with Neuburg Siliceous Earth have no problem in meeting this requirement. In so doing, the calcined siliceous earth grades give the best results.

In addition, the DIN EN 14293 standard requires for “hard” adhesives a lap shear strength of 3.0 MPa after curing for 3 days at standard climate conditions.

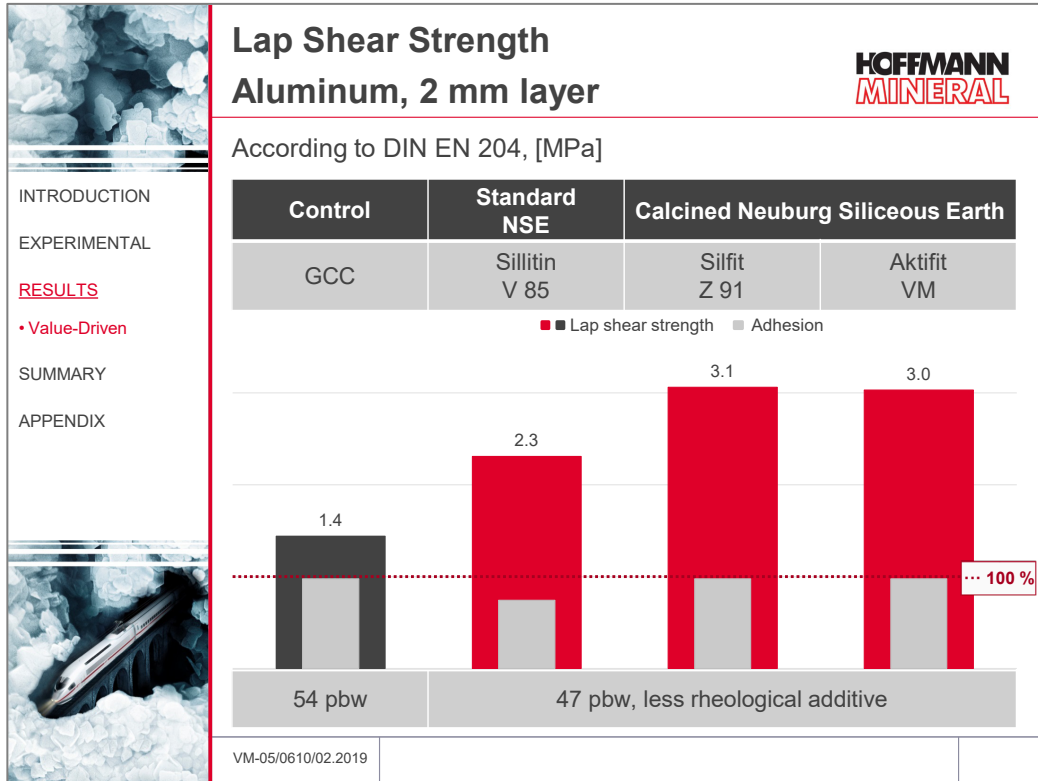


Here again the batches with Neuburg Siliceous Earth come out markedly higher than the minimum requirements, whereas with GCC also this limit cannot be overcome.

d) Lap Shear Strength on Aluminum / Hot Water Resistance

In order to assess the performance as multifunctional adhesives, pure aluminum was chosen as an additional substrate.

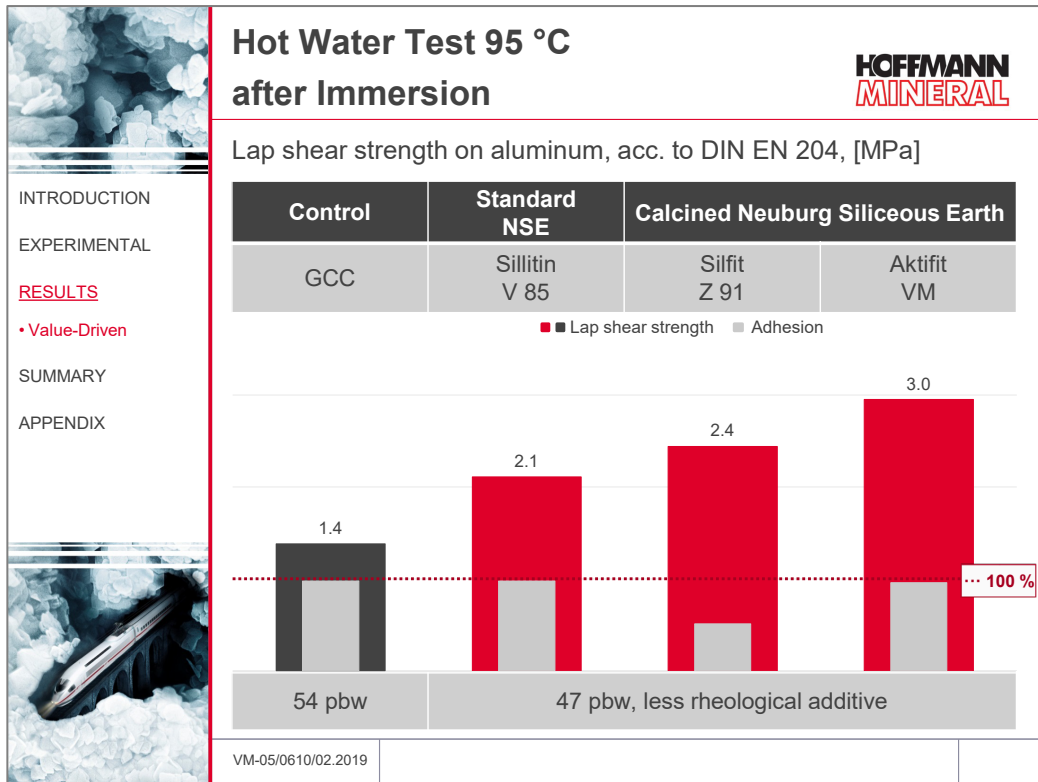
Two aluminum sample sheets measuring 100 x 25 x 1.5 mm were lap-bonded at an adhesive area of 25 x 12.5 mm (312.5 mm²) with an adhesive layer thickness of 2 mm. The tests were carried out after a curing time of 14 d at standard climate 23/50.



The Neuburg Siliceous Earth grades here again come off significantly better than the calcium carbonate. In particular, Silfit Z 91 and Aktifit VM give rise to an improvement of the lap shear strength of 120 %.

The light gray bars refer to the percentage area of the bonded surface which contains adhesive residues after the breakage of the specimen. The formulations (with the exception of Sillitin V 85) give evidence of a very good adhesion with 100 % cohesive failure.

The aluminum samples were subjected to a hot water test on the basis of DIN EN 204. They were immersed for 6 h in deionized water at 95 °C, and subsequently 2 h in cold water of 20 °C. Then the tests were run on the still wet samples.



For the hot water resistance, the positive results with the Neuburg Siliceous Earth grades have been confirmed.

Even the cost effective system with Sillitin V 85 surpasses the calcium carbonate formulation considerably.

Despite partial loss of adhesion with Silfit Z 91, the good strength is largely maintained. Aktifit VM shows the same outstanding lap shear strength and 100 % cohesive failure prior to and after the hot water immersion, and therefore this grade proves particularly advantageous with respect to hot water resistance.

3.1.4 Storage Stability

After 6 months of storage of the adhesives in a customary standard PE cartridge at room temperature, no premature crosslinking could be observed. The compounds showed no gelling, and could be squeezed out without problems.

3.1.5 Conclusion: Value-Driven Approach

Compared to the standard filler GCC, Neuburg Siliceous Earth offers the following benefits:

- Control of rheology via filler and fumed silica loading
- Higher tensile strength without losses in elongation at break
- Markedly higher lap shear strength
- High lap shear strength even after immersion in hot water
- Compliance with the requirements of DIN EN 14293 for “soft” as well as “hard” adhesives

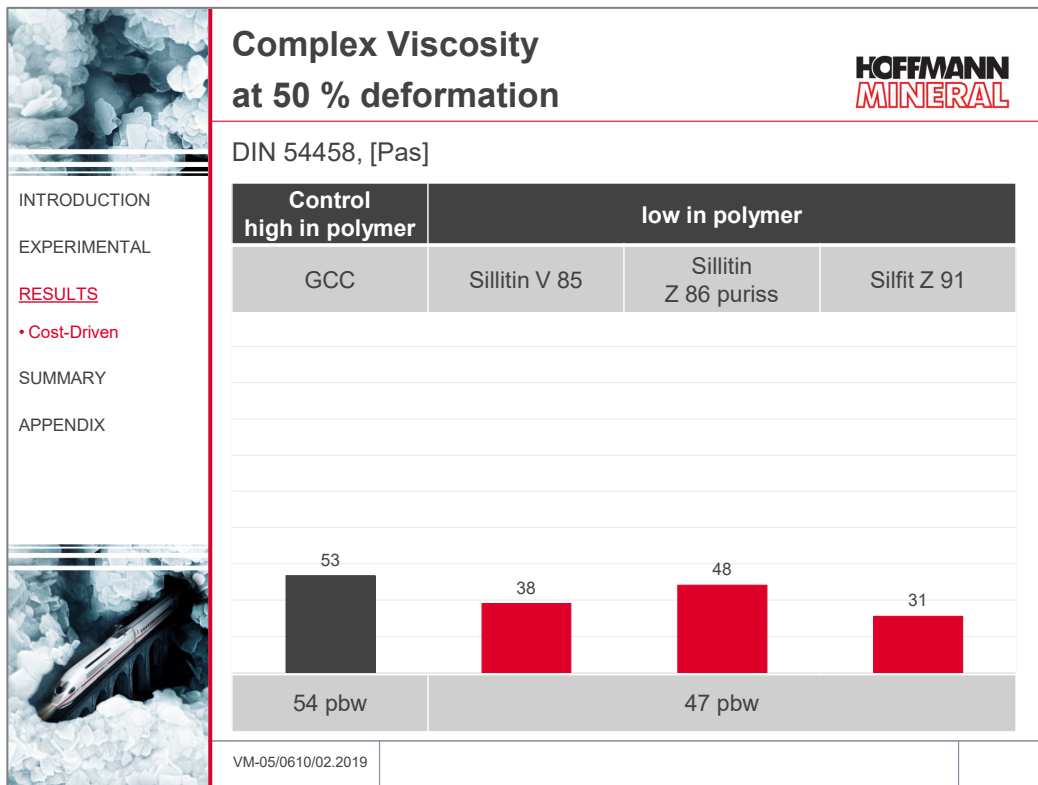
3.2 Cost-Driven Approach

| | | Control with GCC high in polymer | | Neuburg Siliceous Earth low in polymer | |
|--|----------------------|----------------------------------|-------------|--|----------|
| | | pbw or weight-% | pbw | pbw | weight-% |
| INTRODUCTION EXPERIMENTAL RESULTS • Cost-Driven SUMMARY APPENDIX | Polymer | 25.5 | 15.5 | 16.7 | |
| | Plasticizer | 15.0 | 25.0 | 26.9 | |
| | Drying agent | 2.0 | 2.0 | 2.2 | |
| | Rheological additive | 2.5 | 2.5 | 2.7 | |
| | Filler | 54.0 | 47.0 | 50.4 | |
| | Adhesion promoter | 1.0 | 1.0 | 1.1 | |
| | Total | 100.0 | 93.0 | 100.0 | |
| | VM-05/0610/02.2019 | | | | |

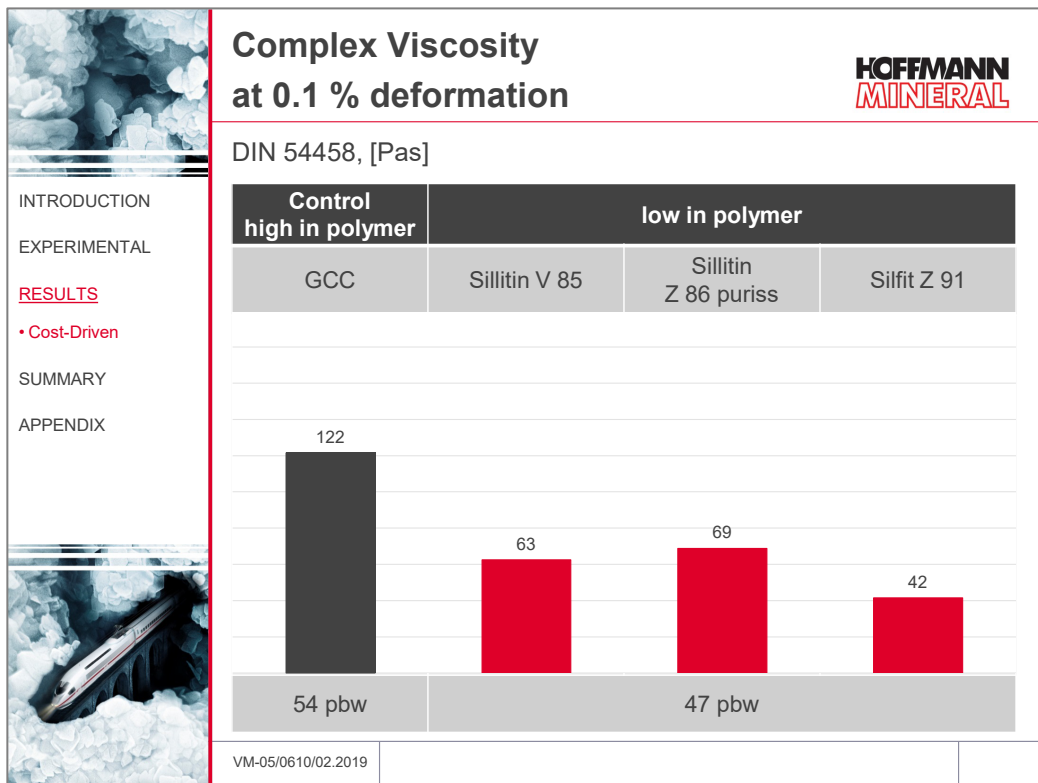
The cost-oriented part of the study is based on the high strength as obtained with Neuburg Siliceous Earth. The starting point again is the polymer-rich base formulation with calcium carbonate used as a reference. In order to save costs, the polymer portion in the formulation will be reduced, and at the same time the addition of plasticizer (polypropylene glycol) increased. The filler loading of Neuburg Siliceous Earth, as in the value-oriented study, is reduced to 47 pbw. The rheological additive silica, however, in favor of good non-sag properties, is again added at the original level of 2.5 pbw.

As cost-favorable fillers here Sillitin V 85 and Sillitin Z 86 puriss, two standard grades of Neuburg Siliceous Earth, were selected, and as a color neutral version the calcined, untreated Silfit Z 91.

3.2.1 Viscosity

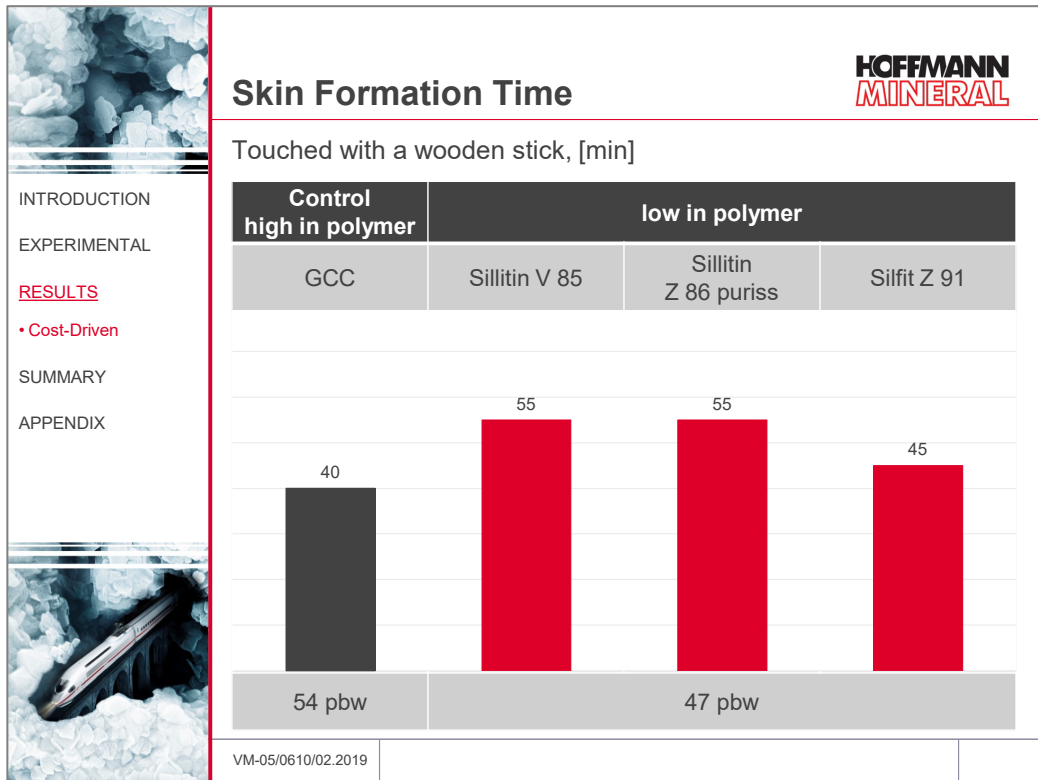


Caused by the reduced filler content and the inverted polymer/filler ratio, the formulations with Neuburg Siliceous Earth at larger deformation show a lower, at maximum comparable viscosity level compared to the base formulation with GCC.

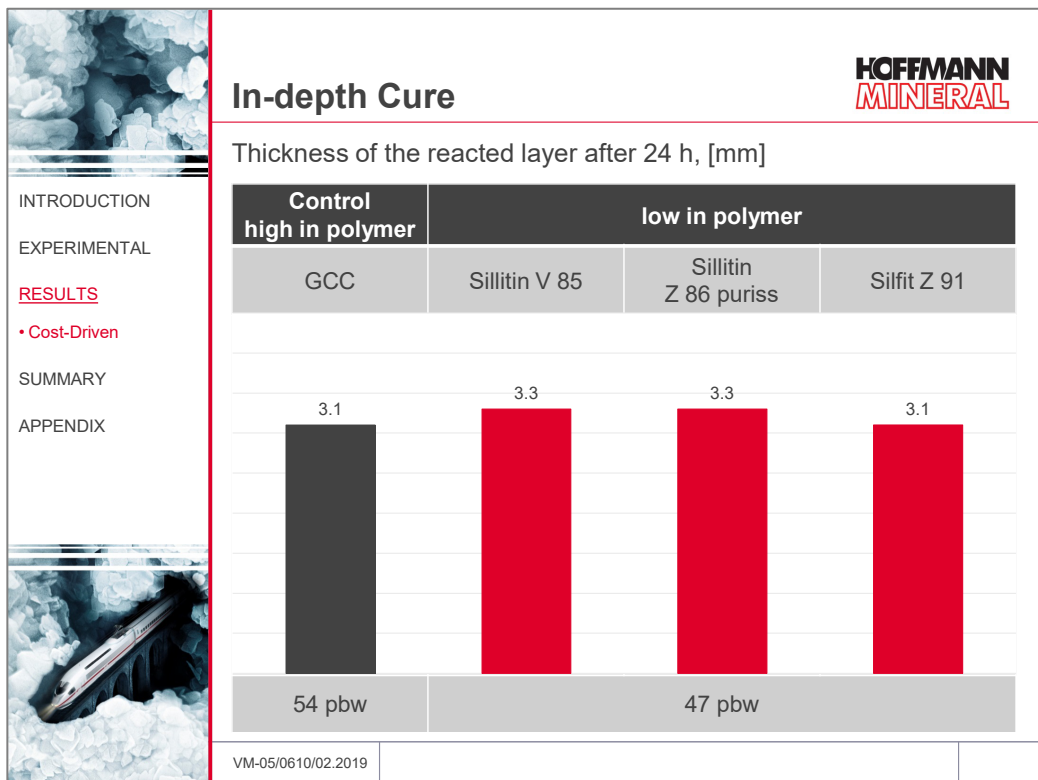


At low deformation, the viscosity with Neuburg Siliceous Earth comes out distinctly lower, and the flow tendency stronger. This is particularly true for the calcined Silfit Z 91. These trends could well be counteracted by proper adjustment of the filler and rheological additive loading, if higher non-sag is desired.

3.2.2 Skin Formation Time and Cure Rate



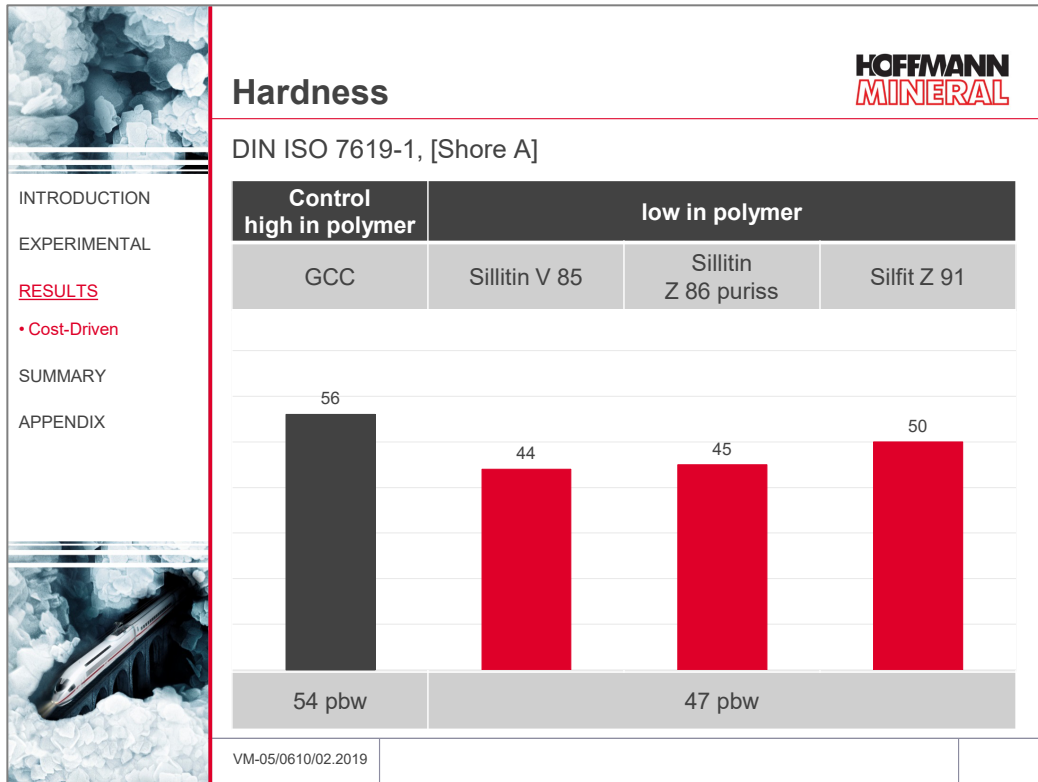
Compared with the polymer-rich formulation with GCC, the skin formation time is generally prolonged as a result of the increase in plasticizer content. This means a longer open time is available for placing the parquet. In particular the standard Siliceous Earth grades Sillitin V 85 and Sillitin Z 86 puriss offer themselves for use of this effect. When working with Silfit Z 91, the skin formation takes place at a similar rate as with the base formulation.



The cure rate, by contrast, remains little affected. With a high plasticizer content, the standard grades rather tend towards a more rapid hardening.

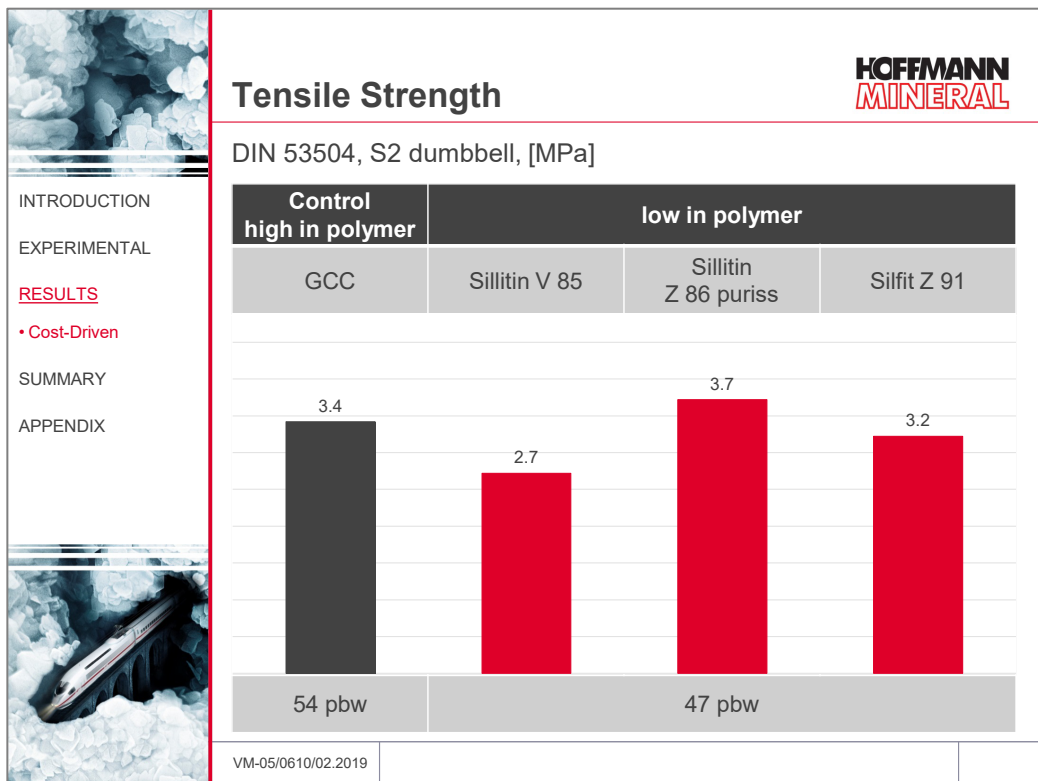
3.2.3 Mechanical Properties

a) Hardness

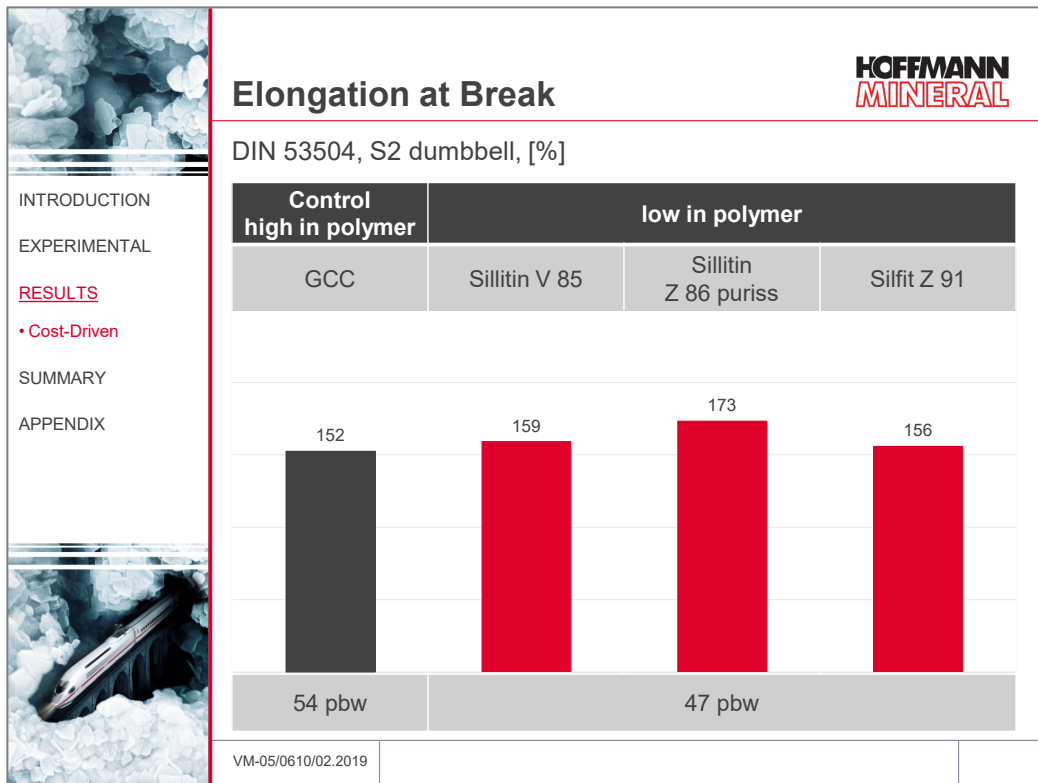


With the modified polymer/plasticizer ratio and reduced filler content, the hardness drops below the level of the polymer-rich base formulation with GCC. The use of the calcined grade Silfit Z 91 makes it possible to compensate this effect to some extent.

b) Tensile Properties

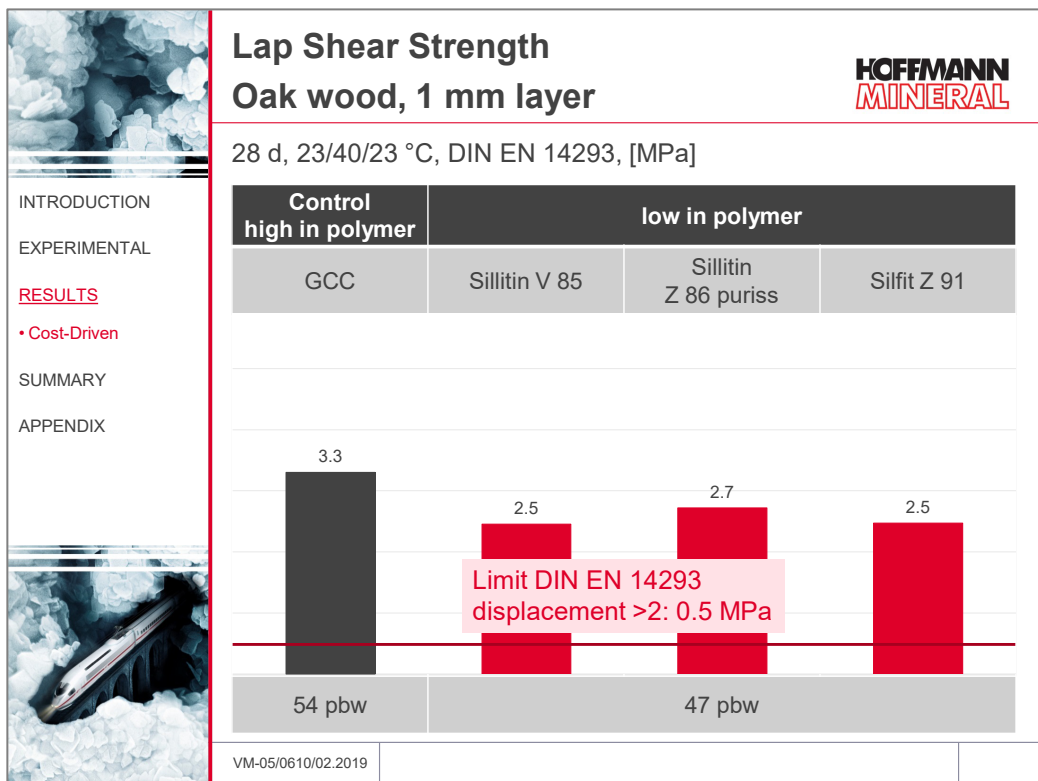


With the finer particle size of the Siliceous Earth grades Sillitin Z 86 puriss and Silfit Z 91, the tensile strength rather remains at the level of the polymer-rich reference with GCC. The coarser grade Sillitin V 85 leads to a slight loss.



The elongation at break too remains on the level of the reference formulation with GCC. Sillitin Z 86 puriss shows a tendency towards higher elongation.

c) Lap Shear Strength on Wood



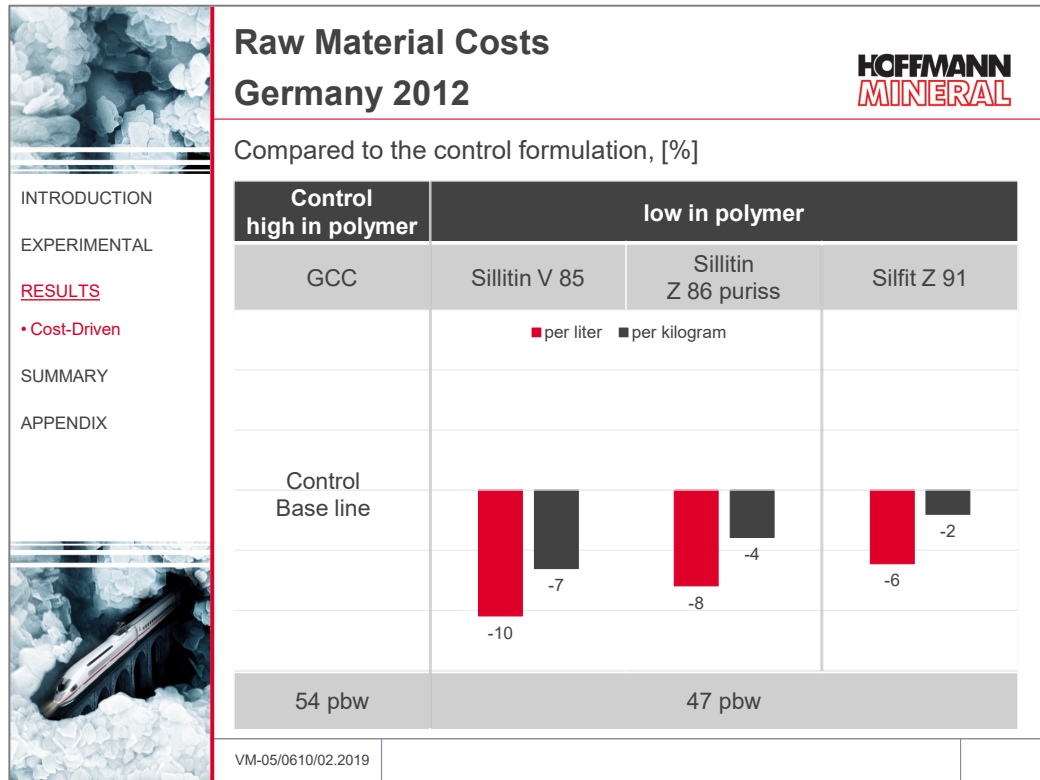
The lap shear strength admittedly is somewhat lower compared with the polymer-rich GCC formulation, but still goes much beyond the requirement of DIN EN 14293 for “soft” adhesives. All formulations give evidence of good adhesion to the oak wood substrate.

3.2.4 Storage Stability

After storing the formulation batches for 6 months in commercial standard PE cartridges at room temperature, no premature crosslinking could be observed. None of the batches showed signs of gelling, and all were ready for squeezing out without problems. Sillitin Z 86 puriss gave evidence of a slight increase in viscosity.

3.2.5 Raw Material Costs

The bar diagram illustrates the raw material costs per liter and per kilogram of the formulations in comparison with the polymer-rich reference formulation with calcium carbonate. The calculations were based on raw material prices in Germany in 2012.



With respect to cost savings, the area of “soft parquet adhesives”, as a result of the strength obtained with Neuburg Siliceous Earth grades, offers several chances. With a savings potential of 10 % (based on volume) resp. 7 % (based on weight), the formulation with Sillitin V 85 comes out as the most cost favorable choice.

Sillitin Z 86 puriss offers higher strength along with still 8 resp. 4 % savings.

Even with the distinctly color neutral Silfit Z 91, there remains a savings potential of 6 resp. 2 % along with good strength.

3.2.6 Conclusion: Cost-Driven Approach

Compared with the control formulation (high in polymer content), Neuburg Siliceous Earth grades offer:

- Similar tensile strength and elongation at break
- Somewhat lower lap shear strength
- Compliance with the requirements of DIN EN 14293 for “soft” parquet adhesives
- Cost saving potential by reduced polymer / increased plasticizer content

3.3 Maximum Performance Approach

In the last part of this study, the maximum performance capability of Neuburg Siliceous Earth grades, in comparison with GCC, will be evaluated in formulations without plasticizer. The objective is adhesion with the “highest load transmission possible”.

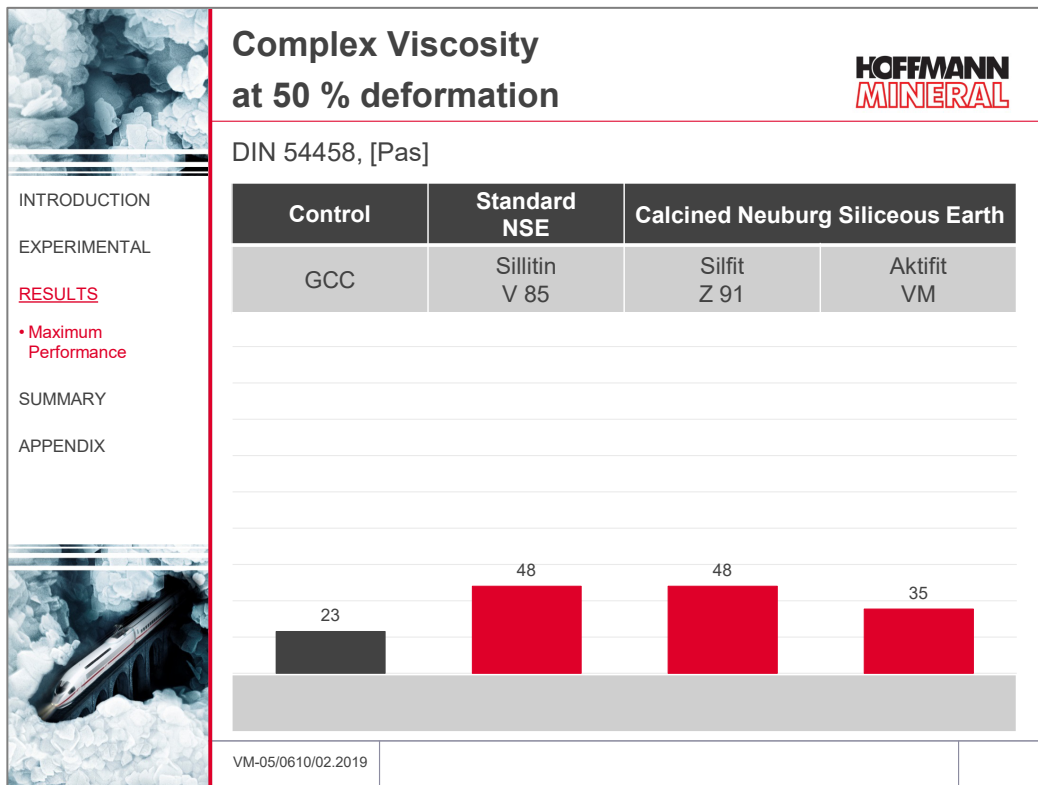
| Formulation Variations | | Maximum Performance | |
|--|----------------------|-----------------------------|--|
| | | HOFFMANN MINERAL | |
| INTRODUCTION EXPERIMENTAL RESULTS • Maximum Performance SUMMARY APPENDIX | | without plasticizer | |
| | | pbw or weight-% | |
| | Polymer | 42.14 | |
| | Plasticizer | --- | |
| | Drying agent | 2.31 | |
| | Rheological additive | --- | |
| | Filler | 54.38 | |
| | Adhesion promoter | 1.17 | |
| | Total | 100.00 | |
| VM-05/0610/02.2019 | | | |

In order to just evaluate the filler effects, the formulation was only composed of polymer, filler, drying agent and adhesion promoter. The rheological additive was left out, due to it also contributes to the strength, and additionally tends to increase the viscosity.

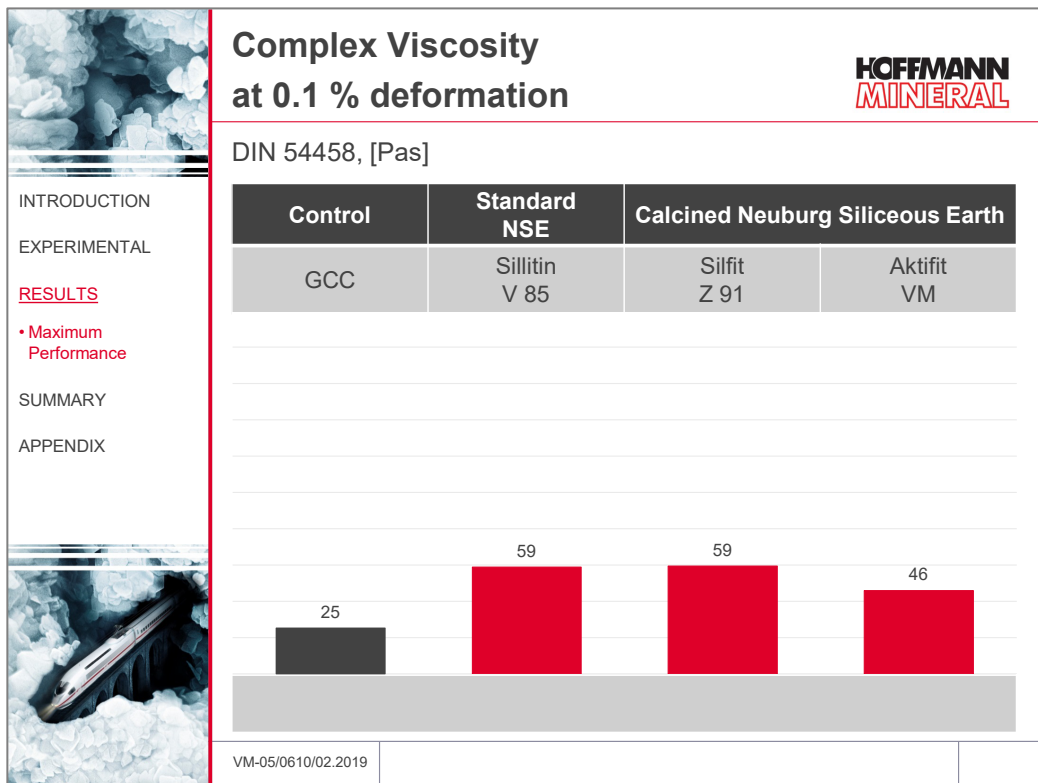
The batch recipes were identical for all formulations, with a filler content of around 54 %.

Among the Neuburg Siliceous Earth grades, again Sillitin V 85, Silfit Z 91 and Aktifit VM were included.

3.3.1 Viscosity

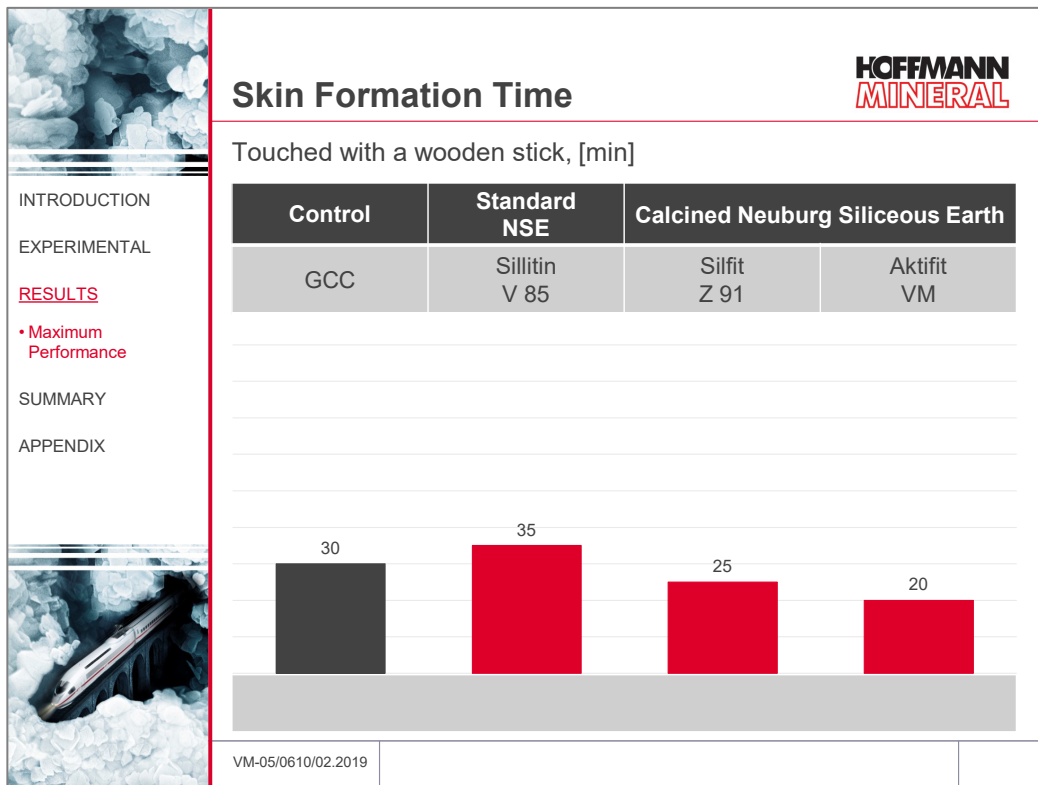


Under higher deformation the complex viscosity with Neuburg Siliceous Earth in direct comparison is higher than with GCC. Within the Neuburg Siliceous Earth grades, this increase turns out lower with the surface treated calcined grades.

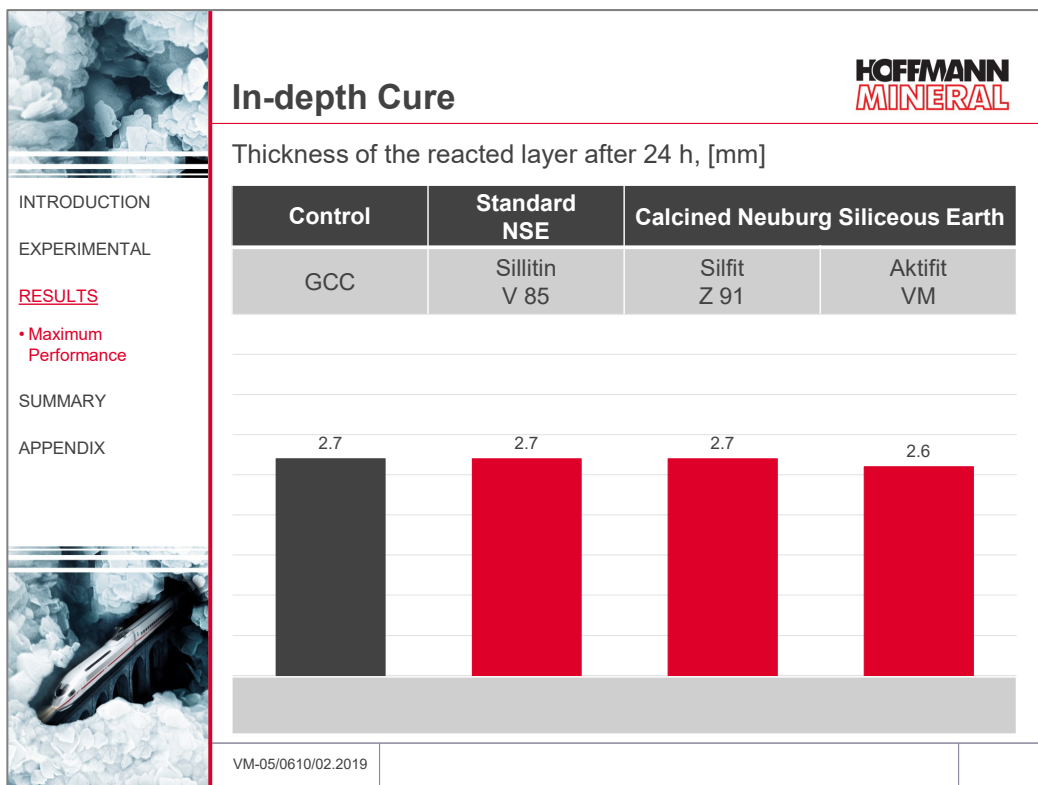


At low deformation, the Neuburg Siliceous Earth grades result in only marginally higher measuring values. Also under quasi-static conditions the batches give evidence of a pronounced flow tendency.

3.3.2 Skin Formation Time and Cure Rate



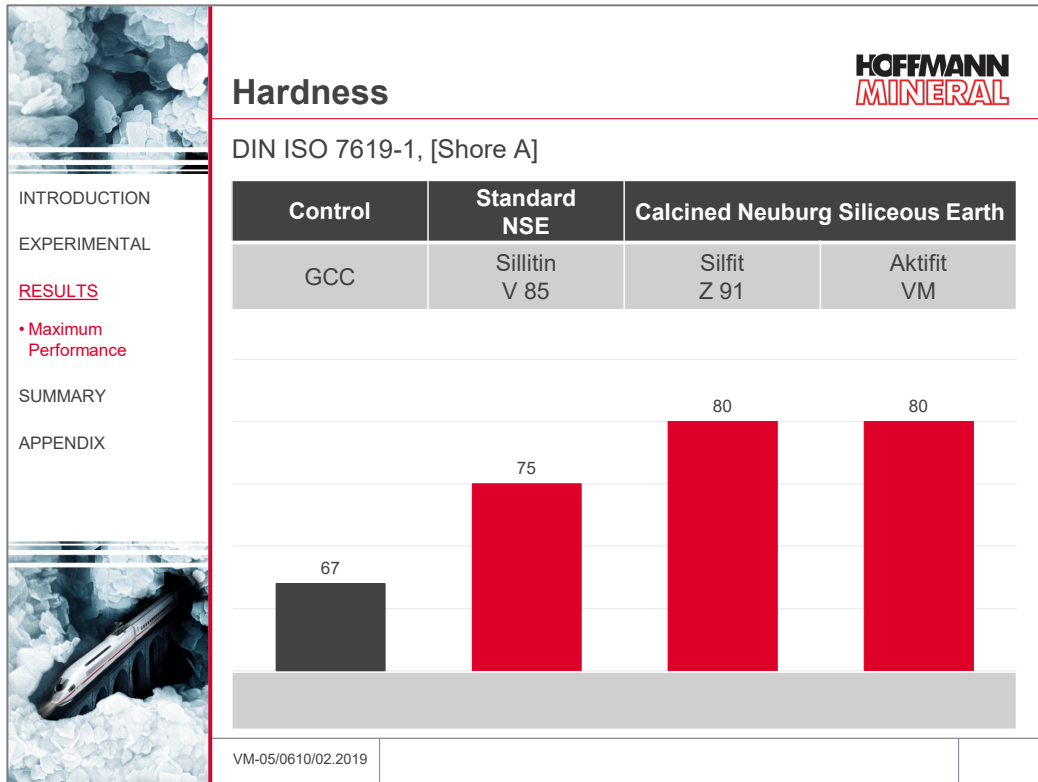
The formulation with the standard grade Sillitin V 85 requires a marginally longer time for skin formation. With the Calcined Siliceous Earth grades – especially with the surface treated product – the skin formation time comes out somewhat shorter.



Concerning the cure rate, no significant differences can be observed after 24 hours.

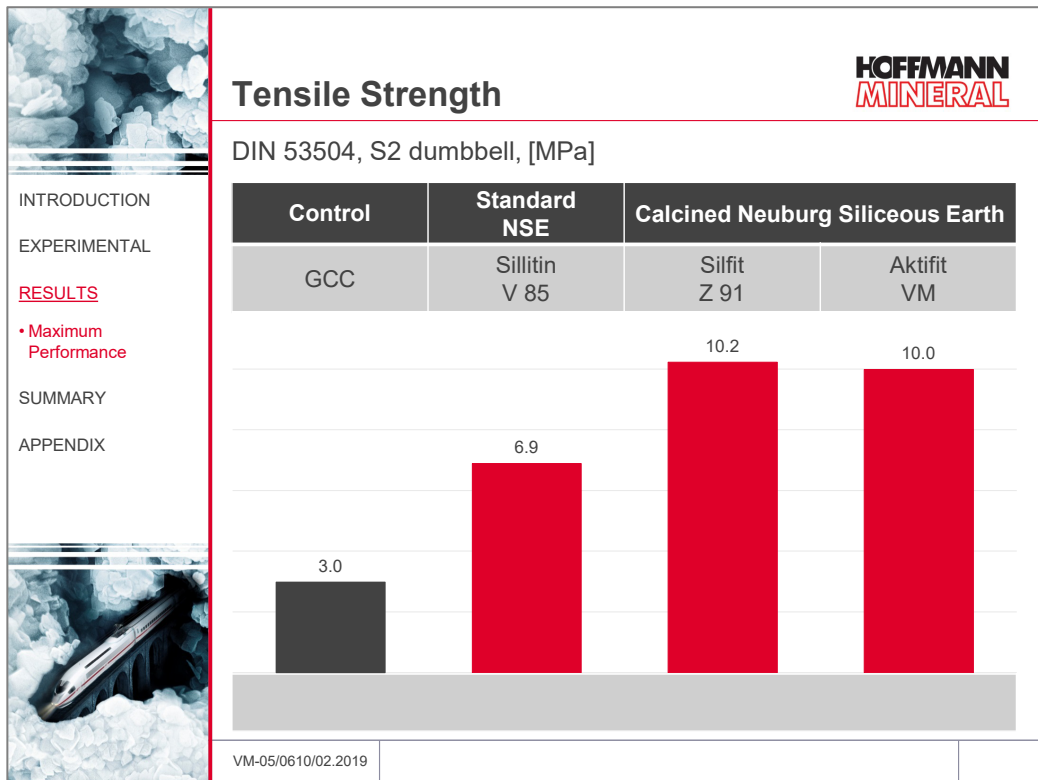
3.3.3 Mechanical Properties

a) Hardness

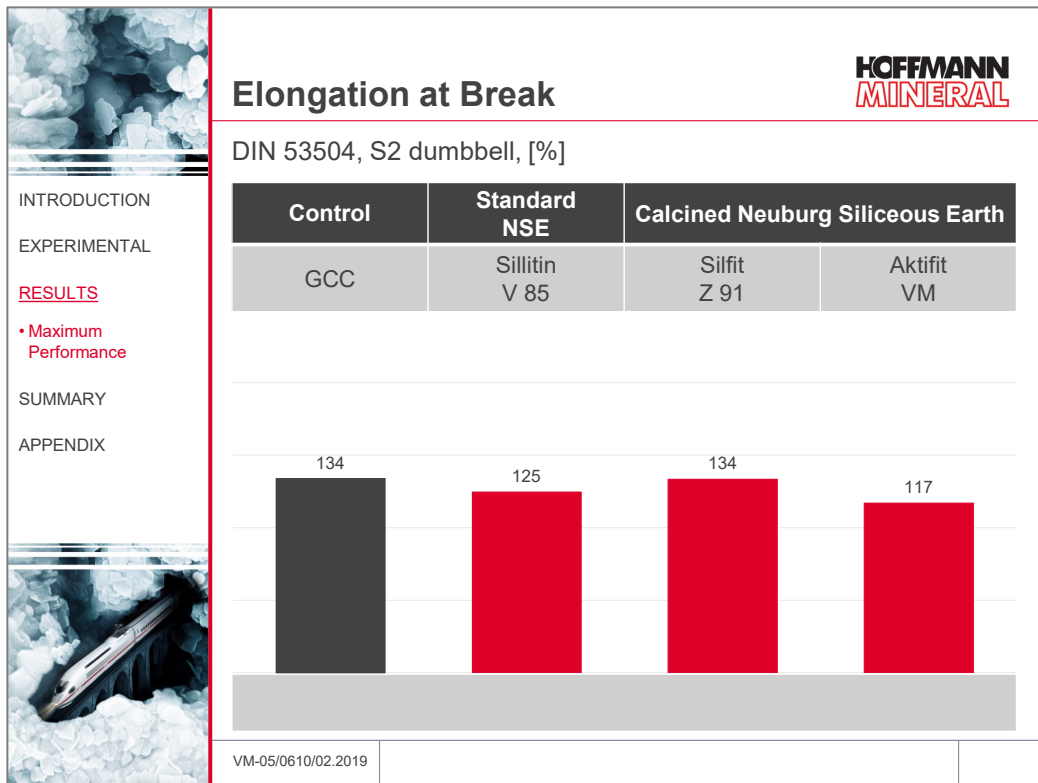


In the formulations without plasticizer, Neuburg Siliceous Earth grades result in a marked increase of the Shore hardness, which is most pronounced with the calcined products.

b) Tensile Properties

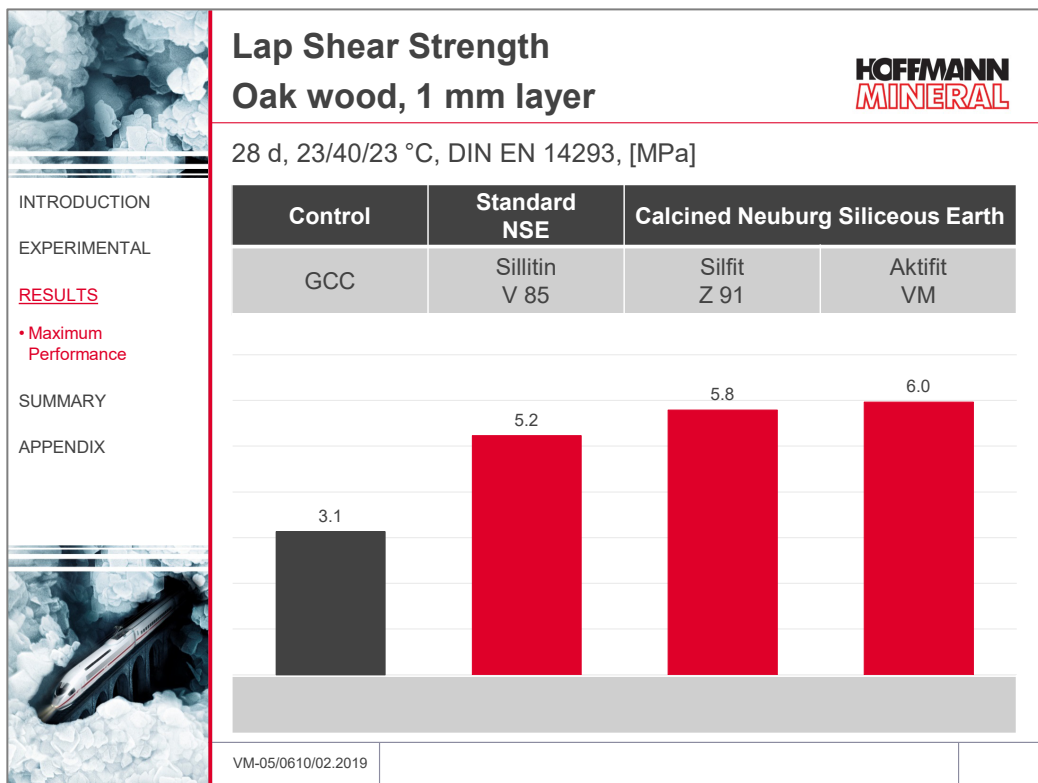


Neuburg Siliceous Earth gives rise to an extraordinarily high tensile strength. With Sillitin V 85, the tensile strength can already be more than doubled vs. GCC. Silfit Z 91 and Aktifit VM with approx. 10 MPa give the very best results, which is more than three times the level of the GCC formulation.

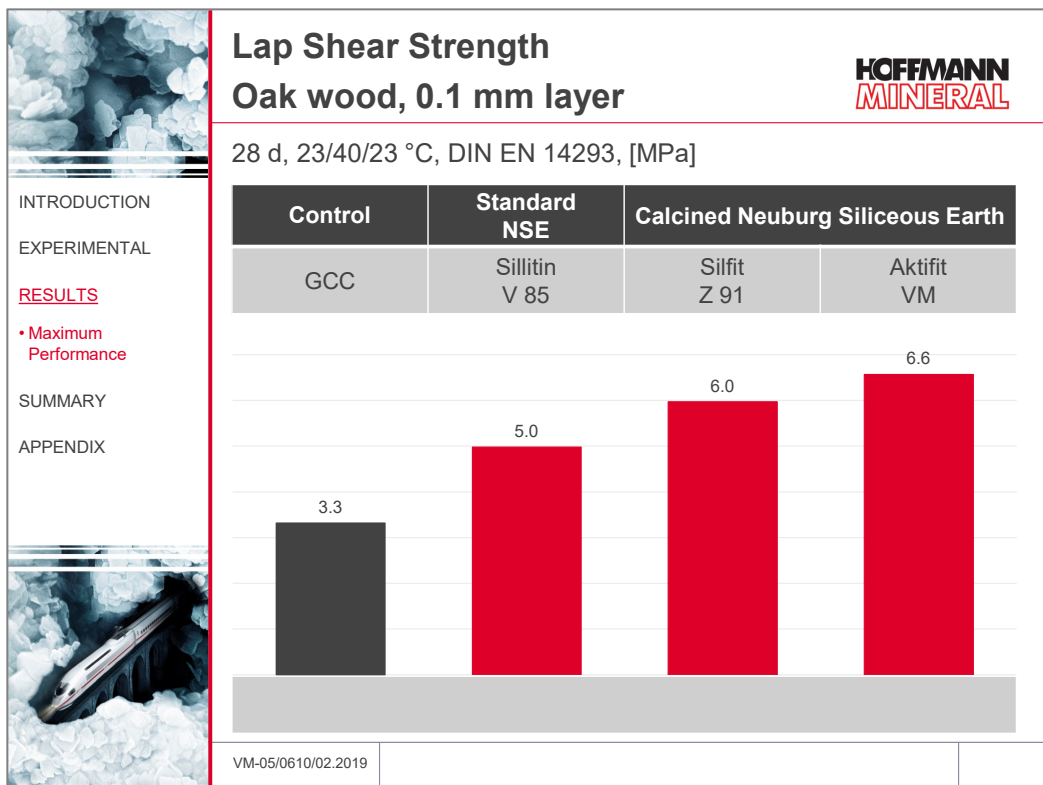


This is all the more remarkable as the elongation at break does not show significant differences. Only the hydrophobic Aktifit VM remains somewhat behind the other fillers.

c) Lap Shear Strength on Wood



Despite very good adhesion, the extremely high tensile strength cannot fully be transferred to the substrate, yet compared with calcium carbonate the lap shear strength is still increased by a factor of 2 – in particular with the calcined grades. With this, the inherent strength of the oak wood is attained, so that partly wood fiber break-outs can be observed.



The same favorable results are also obtained at the lower layer thickness.

3.3.4 Storage stability

After 6 months of storage in commercial PE cartridges at room temperature, also the formulations without plasticizer did not show any sign of premature crosslinking. There was no evidence of gelling, and the materials could be extruded without problems.

3.3.5 Conclusion: Maximum Performance Approach

In comparison with GCC, Neuburg Siliceous Earth grades offer:

- High hardness
- Extraordinarily high tensile strength up to 10 MPa
- Almost unchanged elongation at break
- Markedly increased lap shear strength, more than 5 MPa appear possible

4 Suggestions: Fillers and Start Formulations

Among the product portfolio of Neuburg Siliceous Earth, the following fillers are particularly suited to formulate (parquet) adhesives based on silane terminated polyethers:

- Sillitin V 85**
 - Very cost effective
 - High strength
- Sillitin Z 86 puriss**
 - Cost effective
 - Higher strength
- Silfit Z 91**
 - Low moisture content
 - White and color-neutral
 - Cost effective
 - Very high strength
- Aktifit VM**
 - Very low moisture content and extremely low moisture absorption even under humid conditions
 - White and color-neutral
 - Very high strength
 - Excellent hot water resistance and adhesion on aluminum

| INTRODUCTION EXPERIMENTAL RESULTS SUMMARY APPENDIX | Start Formulations | | | HOFFMANN MINERAL |
|--|-----------------------------|--|-------------------------------------|--------------------------------|
| | parts by weight | | | |
| | Requirement | Parquet adhesive with increased strength "soft" or "hard" | Economic parquet adhesive "soft" | Adhesive with maximum strength |
| | GENIOSIL® STP-E 10 | 25.5 | 15.5 | 42.1 |
| | Caradol ED 56-200 | 15.0 | 25.0 | --- |
| | GENIOSIL® XL 10 | 2.0 | 2.0 | 2.3 |
| | HDK H 18 | 2.5 | 3.0 – 2.0 | 0 – 2.0 |
| | Sillitin / Silfit | --- | 47.0 – 54.0 | --- |
| | Sillitin / Silfit / Aktifit | 47.0 | --- | 54.4 |
| | GENIOSIL® GF 96 | 1.0 | 1.0 | 1.2 |

VM-05/0610/02.2019

Suppliers:

| | |
|--------------------|-----------------|
| GENIOSIL® STP-E 10 | Wacker Chemie |
| Caradol ED 56-200 | Shell Chemicals |
| GENIOSIL® XL 10 | Wacker Chemie |
| HDK H 18 | Wacker Chemie |
| GENIOSIL® GF 96 | Wacker Chemie |

5 Summary

The use of Neuburg Siliceous Earth in the polymer-rich base formulation allows to meet both the requirement of DIN EN 14293 for “soft” parquet adhesives with a displacement of more than 2 and the specification for “hard” parquet adhesives with a lap shear strength of at least 3.5 MPa (resp. 3.0 MPa after 3 days). The rheological properties can be optimized by reducing the filler and silica content. Of particular interest vs. calcium carbonate are the markedly increased tensile strength and lap shear strength imparted by Neuburg Siliceous Earth.

Selected surface modification of Neuburg Siliceous Earth offers a potential for improving further properties, such as the hot water resistance of the bond.

The high strength imparted by Neuburg Siliceous Earth can be used in a formulation version with reduced polymer content, which still comes up to the requirements of DIN EN 14293 for “soft” parquet adhesives.

Formulations without plasticizer addition allow to arrive at strength results of an outstandingly high level.

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.