HOFFMANN MINERAL
Unternehmensgruppe Hoffmann

Calcined Neuburg Siliceous Earth in medium and high voltage cable insulations

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1 Introduction

Neuburg Siliceous Earth grades are already being used in cable sheath and cable insulation compounds.

They impart medium to high tensile strength and low elongation at elevated temperatures, as well as good extrusion properties.

Furthermore, Neuburg Siliceous Earth makes it possible to obtain high volume resistivity levels.

In the area of high voltage cable insulations, calcined clays have until today represented the state of art, as they offer good dielectrical properties also at high temperatures and high voltages, even under exposure to high air humidity and moisture.

In order to further improve the dielectrical properties, for applications in the medium voltage range vinyl silane is added in situ. For high voltages, over and above the in situ addition of vinyl silane, surface treated calcined clays are used.

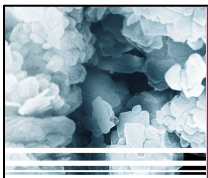
As Hoffmann Mineral constantly endeavors to enlarge ist product portfolio in order to offer customers an increased variety of applications, a calcined version of Neuburg Siliceous Earth was developed which is today available either without surface treatment as Silfit Z 91, or after treatment with a special vinyl functional group under the name of Aktifit VM.

The present study is to show the effects of calcined Neuburg Siliceous Earth - both with and without surface treatment - with respect to the basic properties as well as the electrical properties.

For comparison, calcined clays are evaluated which already find application in medium and high voltage cable insulation compounds.

In addition, the question will be taken up if an in situ addition of vinyl silane aside from the use of surface treated fillers is really necessary.

2 Experimental
2.1 Base formulation



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Base Formulation

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	with additional vinyl silane	without additional vinyl silane
EPDM*	100.00	100.00
Masterbatch (ZnO, Pb_3O_4, PE)*	14.61	14.61
Vulkanox HS/LG	1.28	1.28
Perkadox BC-FF	1.83	1.83
Paraffinic wax*	5.01	5.01
Silquest A-172 NT	0.75	- - - -
Mineral filler	60.00	60.00
Total	**183.48**	**182.73**

Formulation and raw materials (*) have been kindly provided by Brugg Kabel AG in Brugg, Switzerland.

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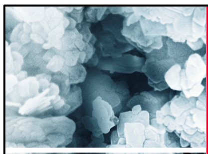
EPDM	kindly provided by Brugg Kabel AG
Masterbatch	kindly provided by Brugg Kabel AG
Vulkanox HS/LG	TMQ, antioxidant
Perkadox BC-FF	dicumyl peroxide
Paraffin wax	kindly provided by Brugg Kabel AG
Silquest A-172 NT	vinyl silane
Mineral filler	see 2.2 "Mineral fillers and compound preparation"

Compound variations with and without added vinyl silane were evaluated with regard to their basic and electrical properties.

2.2 Mineral fillers and compound preparation



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Fillers and Characteristics

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	Particle size d_{50} [µm]	Particle size d_{97} [µm]	Oil absorption [g/100g]	Specific surface area BET [m²/g]	Surface treatment with vinyl silane
Silfit Z 91	2.0	10	59	7.6	none
Aktifit VM	2.2	9.4	57	7.0	yes
Satintone W	3.3	20	71	7.5	none
Polestar 200 R	3.6	19	60	6.5	none
Polarite 103 A	4.4	16	71	6.1	yes
Polarite 503 S	2.9	13	60	10	yes
Translink 37	3.0	18	61	7.4	yes

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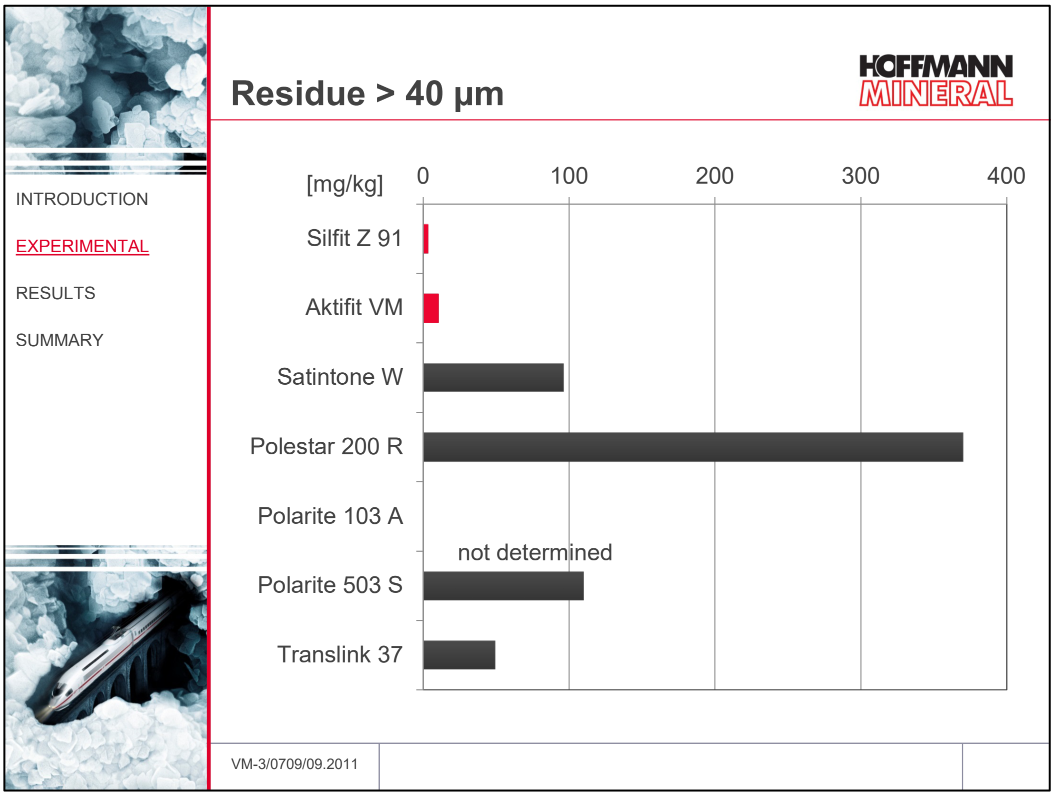
Silfit Z 91 and Aktifit VM were evaluated in comparison with surface treated and untreated calcined clays.

With Satintone W and Polestar 200 R, two coarser untreated calcined clays were included which have roughly a similar surface area as Silfit Z 91. With respect to the oil absorption, Polestar 200 R is comparable with Silfit Z 91, the oil absorption of Satintone W is somewhat higher.

The particle size of Polarite 503 S, Polarite 103 A and Translink 37 increases slightly in this order, which means these surface treated calcined clays are coarser than Aktifit VM. Polarite 103 A offers a somewhat higher oil absorption compared with the other surface treated calcined fillers which within each other give oil absorptions at a similar level. the specific surface area of Polarite 503 S is higher than for the other fillers which come out more or less at a similar level.



Calcined Neuburg Siliceous Earth shows markedly lower sieve residues than the tested competitive fillers. Thus, prolonged intervals of filter changes may be expected by the use of Calcined Neuburg Siliceous Earth.

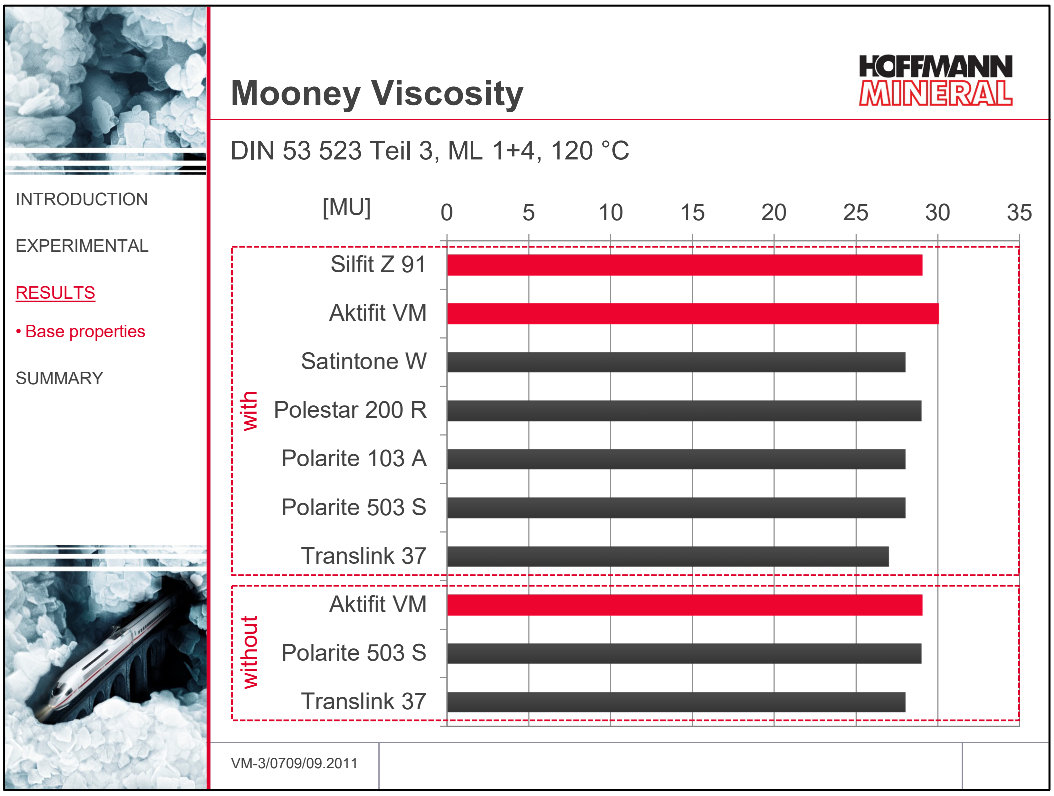
Compounding took place on an open laboratory mill (Schwabenthan Polymix 150 L). The rubber was masticated at 80 °C, followed by all other producers in the order of the formulation as quoted. In order to melt the masterbatch which was added, the roller temperatures were increased to 110 °C. After reaching 110 °C, this temperature was kept constant for five minutes. The rollers were then cooled down again to 80 °C and the peroxide was mixed in. Typical mixing time was 20 minutes.

The compounds were press-cured for 6 minutes at 180 °C.

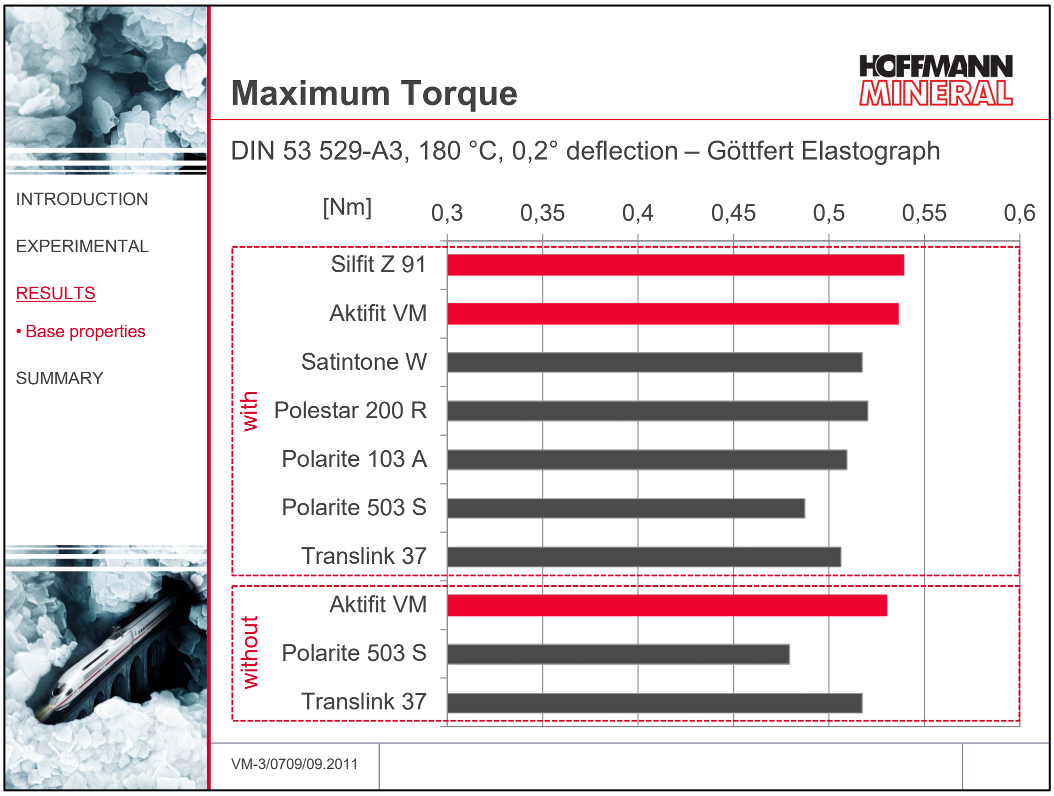
The 1 mm sample sheets for the electrical tests were cured between plastic films in order to avoid any dirtying of the surface which might affect the results.

3 Results

3.1 Viscosity and cure characteristics

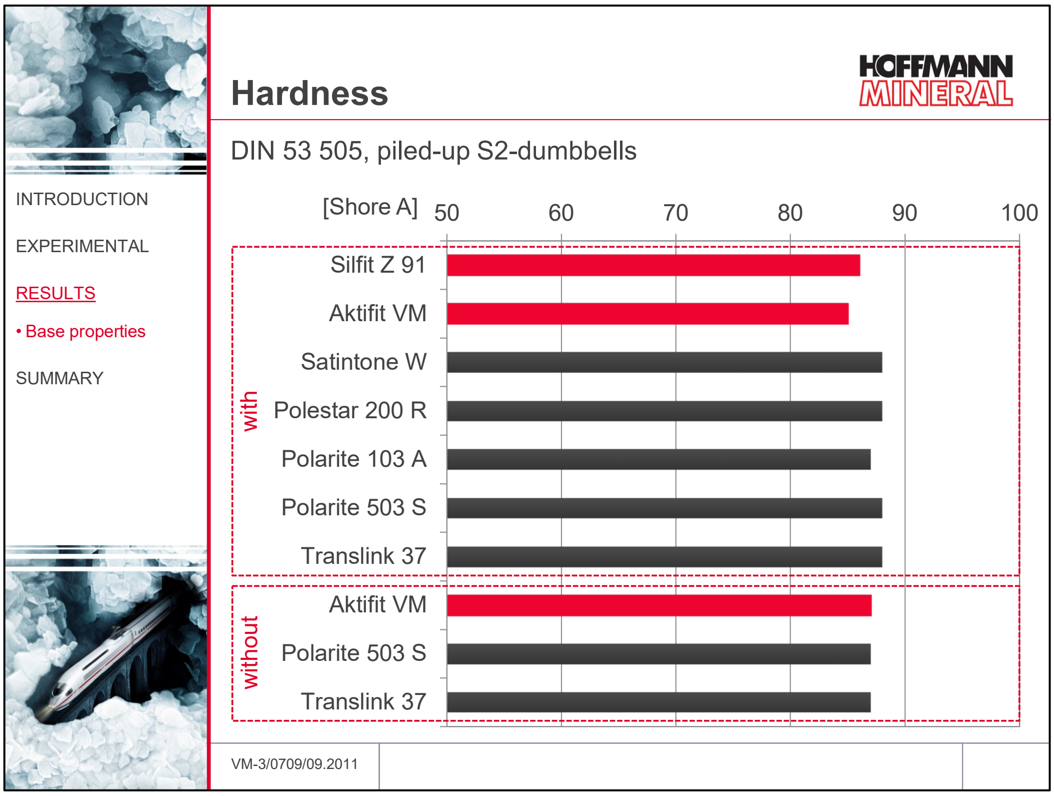


Quite obviously, there are no significant differences regarding viscosity between the individual fillers tested.
Even taking out the in situ added vinyl silane does hardly change the results.

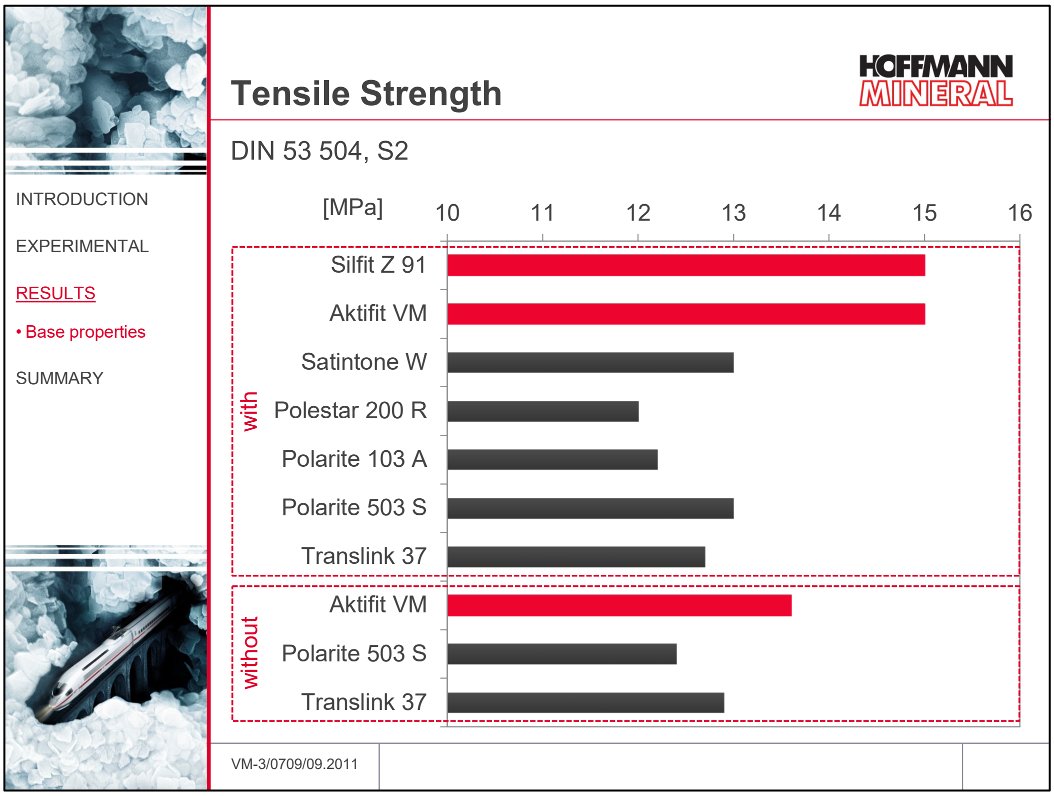


The calcined Neuburg Siliceous Earth grades attain higher Vulcameter torque maxima compared to the other fillers tested.

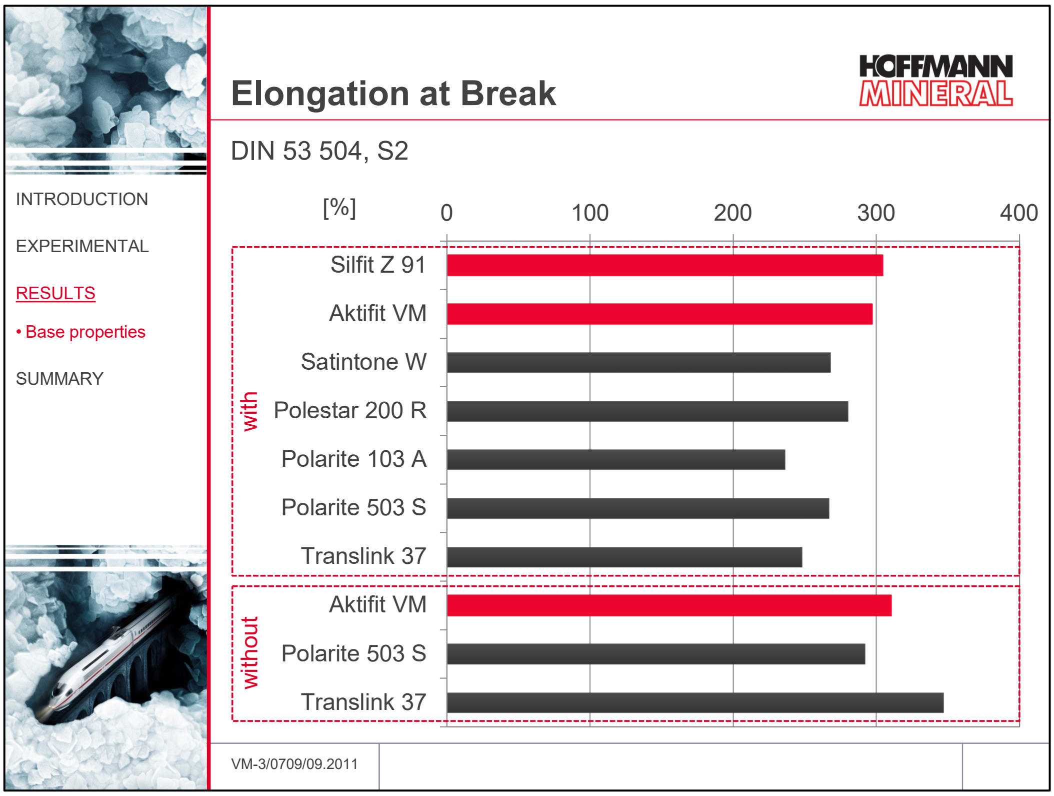
3.2 Mechanical properties



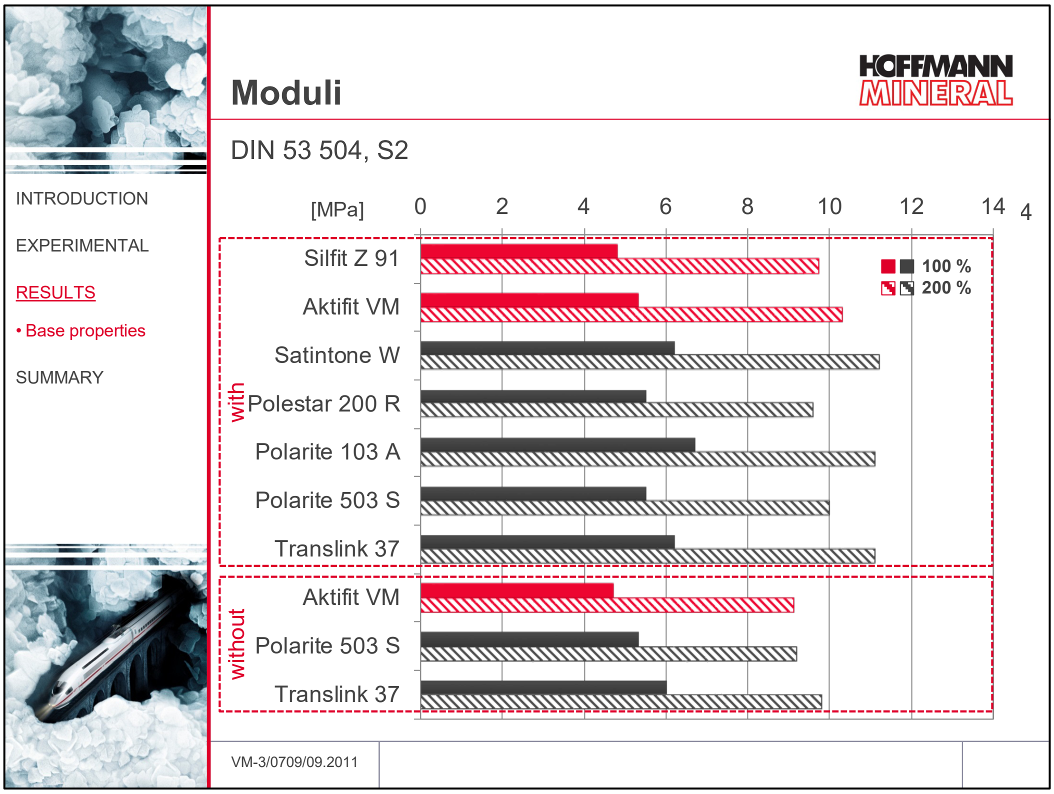
Hardness was determined on three piled-up S2 dubbell specimens.
No significant hardness differences are seen between the individual filler. Even taking out the in situ added vinyl silane does not affect the results.



The calcined Neuburg Siliceous Earth fillers attain higher tensile strength levels with but also without in situ added vinyl silane. As evident from the graph, already the untreated Silfit Z 91 gives a higher tensile strength than all other competitive fillers tested. The same is true for Aktifit VM.
Without in situ added vinyl silane, the tensile strength with Aktifit VM comes out somewhat reduced, but still about 1 MPa higher compared with Polarite 503 S or Translink 37.

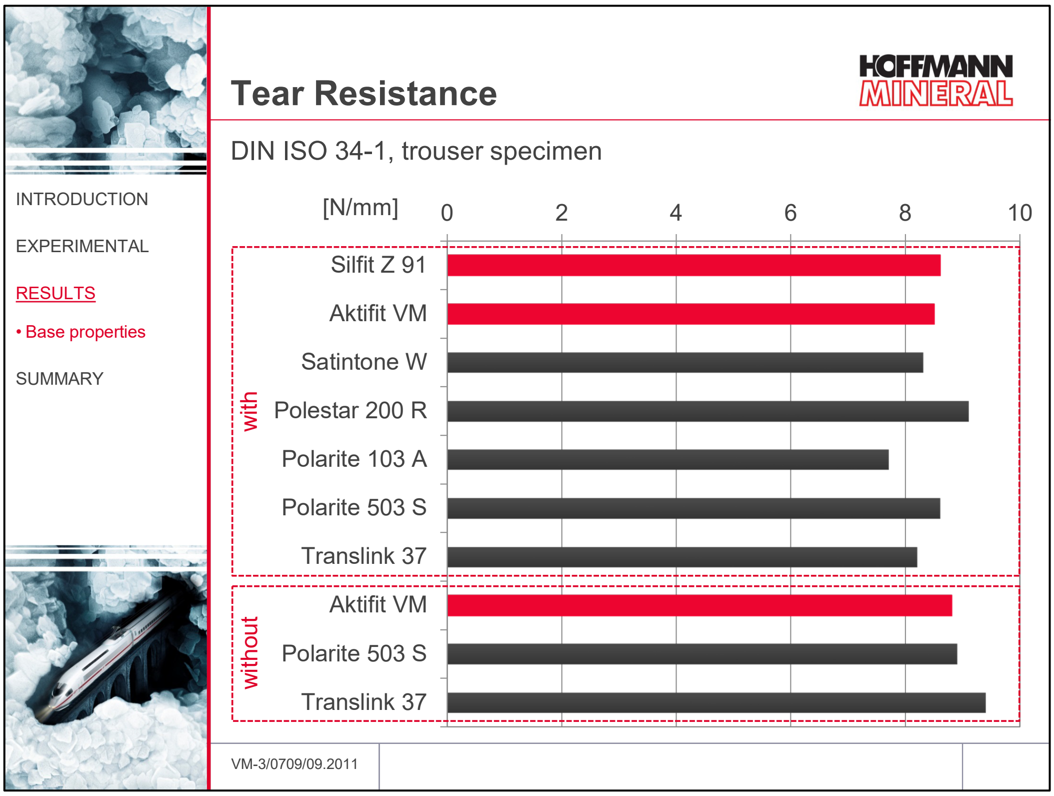


The elongation at break levels obtained with calcined Neuburg Siliceous Earth are somewhat higher than those realized especially with the surface treated calcined clays. Without the in situ added vinyl silane, the results move closer to each other.

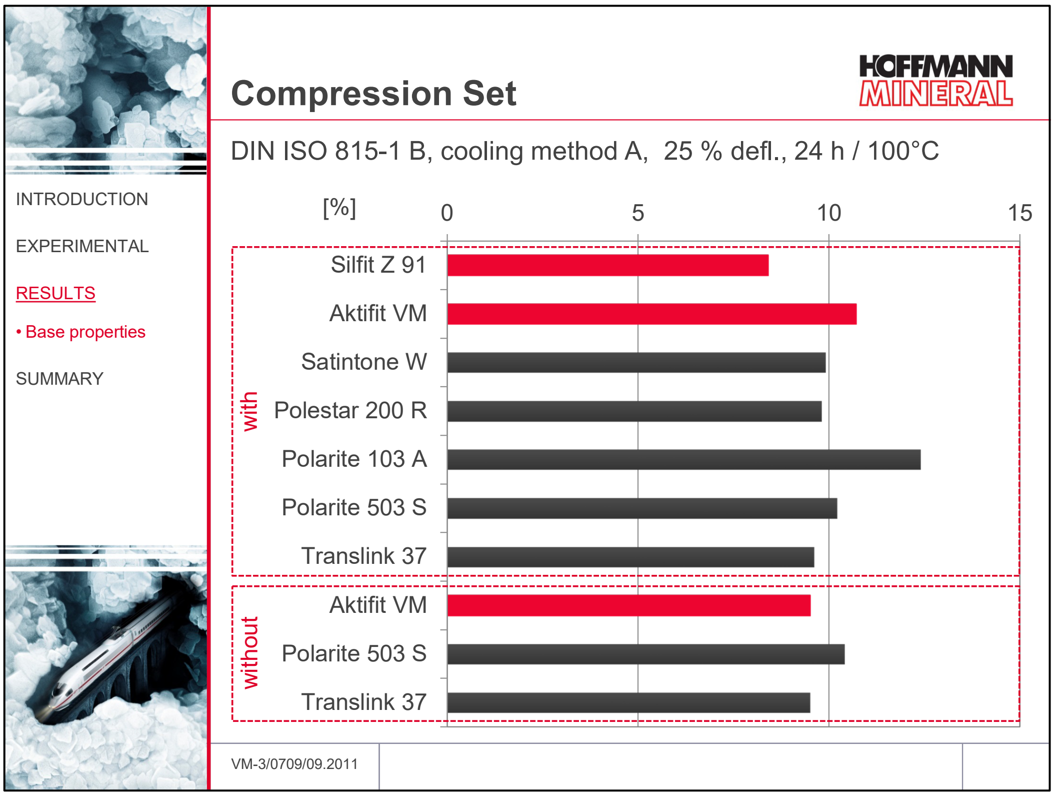


Regarding the moduli, with added vinyl silane there appear no differences between the individual fillers.

Without in situ added vinyl silane, the moduli come out somewhat lower, but this effect is true for all tested fillers to a similar degree.



Likewise, for tear resistance no differences can be observed - neither between the individual fillers nor for the presence respectively absence of added vinyl silane.



The compression set does not represent an immediate characteristic of cable insulation compounds but it gives indications about the capacity for elastic recovery after deformation. The selection of fillers shows no remarkable effect on the compression set. With all fillers tested very similar results are obtained.

Leaving out the addition of vinyl silane does not affect the results either.

3.3 Hot air aging

Hot air aging (according to DIN 53 508) was run for 168, 500 and 1000 hours at 135 °C on S2 dumbbell specimens.

Each time tensile strength, moduli, elongation at break and hardness were determined and the changes from the original levels after cure calculated.

It became evident that there were no significant differences between the individual fillers as well as for the compounds with or without in situ added vinyl silane.

Furthermore, neither the temperature nor the time period of aging affected the results. The changes, for instance, in tensile strength or elongation at break came out all at about ($\pm$) 5 %, the hardness changed ($\pm$) 3 Shore A.

As all compounds tested showed a similar behaviour this will not be further taken up here.

3.4 Electrical properties

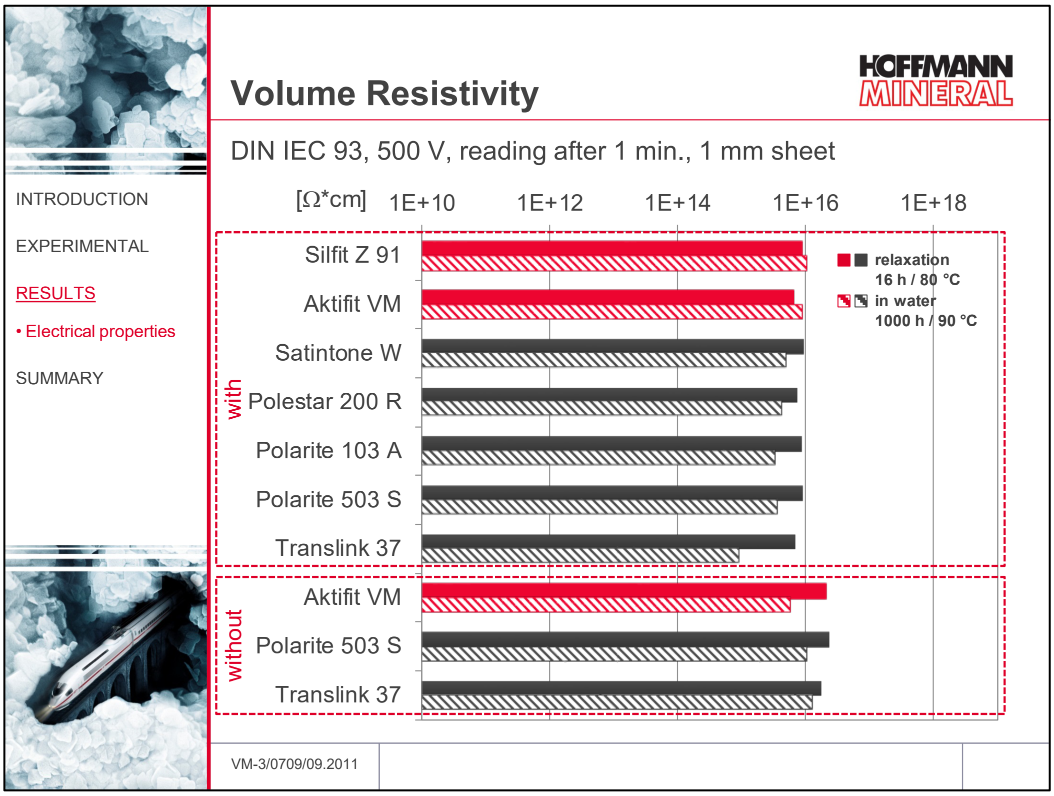
The electrical properties were tested on sample sheets of 1 mm thickness which were cured - as already mentioned - between plastic films.

The relaxation (16 h / 80 °C) and the water immersion tests were run at Hoffmann Mineral. The volume resistivity and the loss factor tan δ were determined after relaxation and after 1000 hours immersion in deionized water of 90 °C.

The determinations of the volume resistivity took place with Hoffmann Mineral using always the same sample sheets. This means, the sample sheets were taken out of the water after the predetermined immersion time, cooled for two hours and then measured. Subsequently, the sheets were re-immersed in the water.

Prior to the measurements, the thickness of the individual sheets was determined in order to detect a possible swelling of the samples. No changes in thickness were observed over the test period, which is why this point will not be further discussed here.

For the determination of the loss factor tan δ, three circular samples with a diameter of about 50 mm were punched out for each immersion time and compound from the 1 mm sheets. At the end of the immersion period, the circular samples were taken out of the water and, the same compounds together, packed in tight-closing PE bags. In order to avoid a total drying-out of the samples, a little amount of deionized water was added into the bags. The samples were then sent to Kabel Brugg AG in Switzerland where the loss factors tan δ were determined.



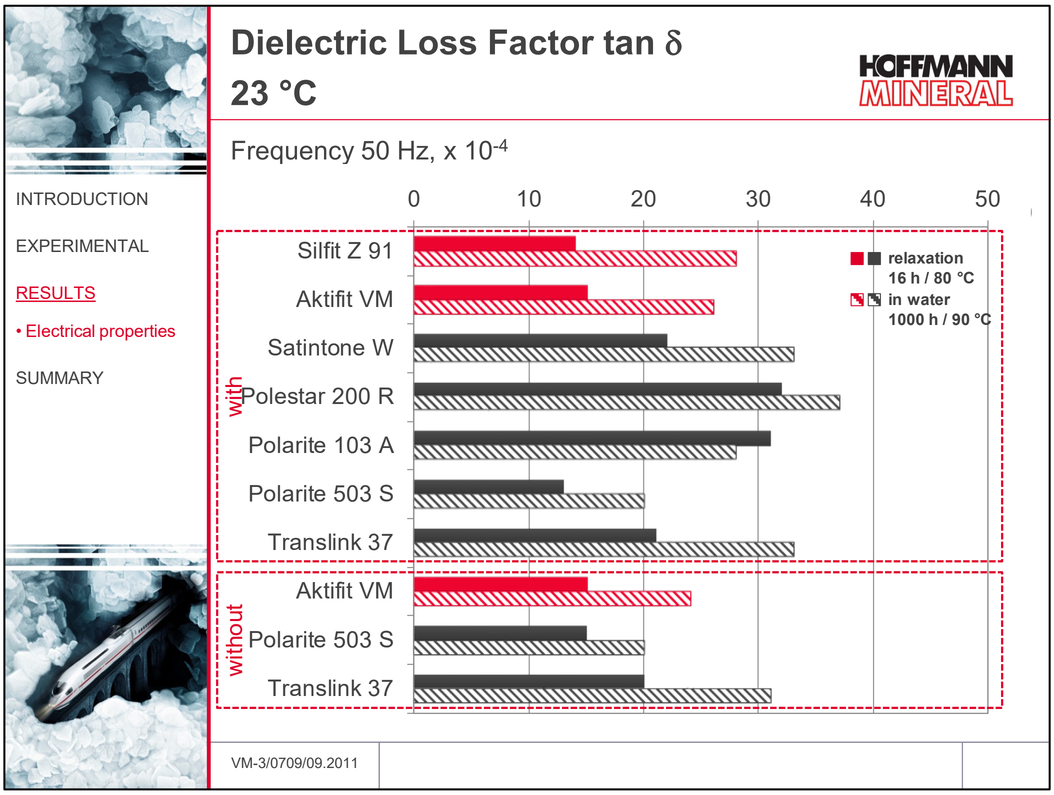
As the graph for the dry results (after relaxation) shows, the calcined Neuburg Siliceous Earth grades impart high volume resistivites which are on one level with the calcined clay as usually applied in this application.

It can also be noted that when leaving out additional vinyl silane, no negative effect on the volume resistivity is observed.

The wet results after 1000 hours water immersion are outstandingly high with Silfit Z 91 and Aktifit VM, i.e. they show no change from the dry levels. By contrast, the competitive fillers in part lead to lower results.
Without added vinyl silane the results remain almost the same.

As the determination of the volume resistivity is carried out with direct current, these results only give an indication of the insulating properties of the compounds but do not characterize the dielectric losses.

These were examined separately, as shown in the following graphs.

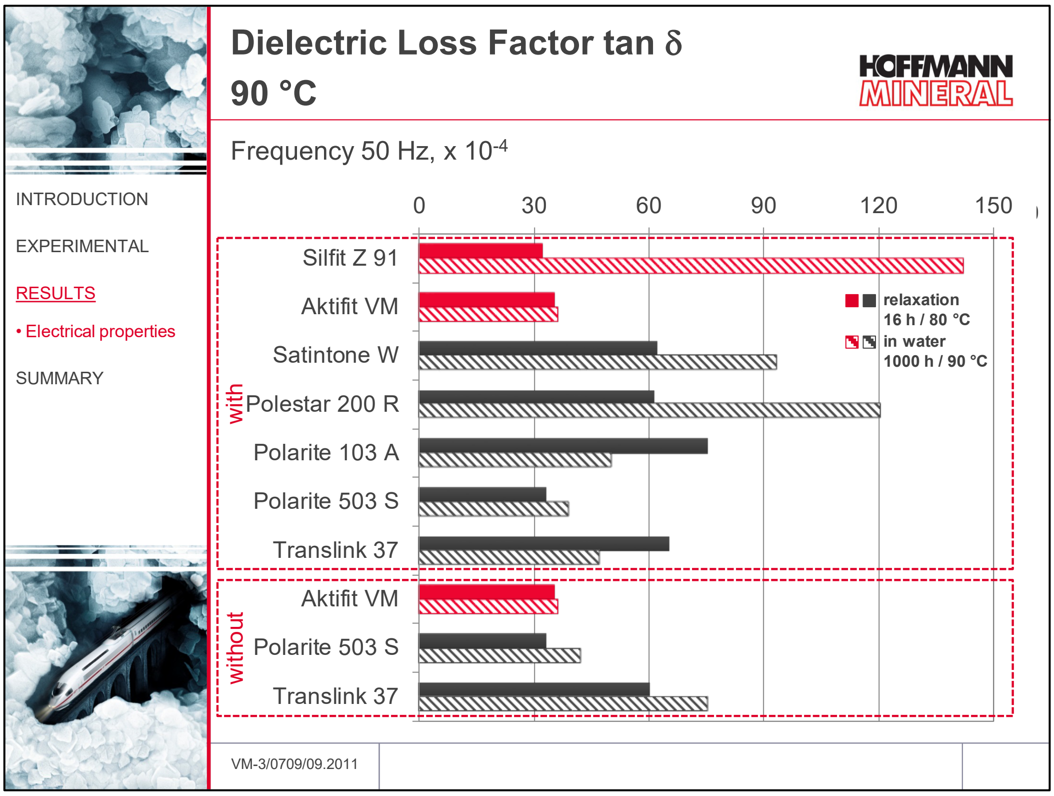


With Silfit Z 91 and Aktifit VM very good results can be obtained.

For instance, the dielectric loss factor with Silfit Z 91 after relaxation comes out similar with Aktifit VM which is on one level with Polarite 503 S and thus clearly below the result with Translink 37.

After 1000 hours water immersion, the dielectric loss factor with Silfit Z 91is lower than for the two untreated calcined clays and as well for Translink 37 although this filler has been treated with vinyl silane.

In comparison with Translink 37, Aktifit VM even without added vinyl silane offers a better result, which is only slightly higher than the result with Polarite 503 S.



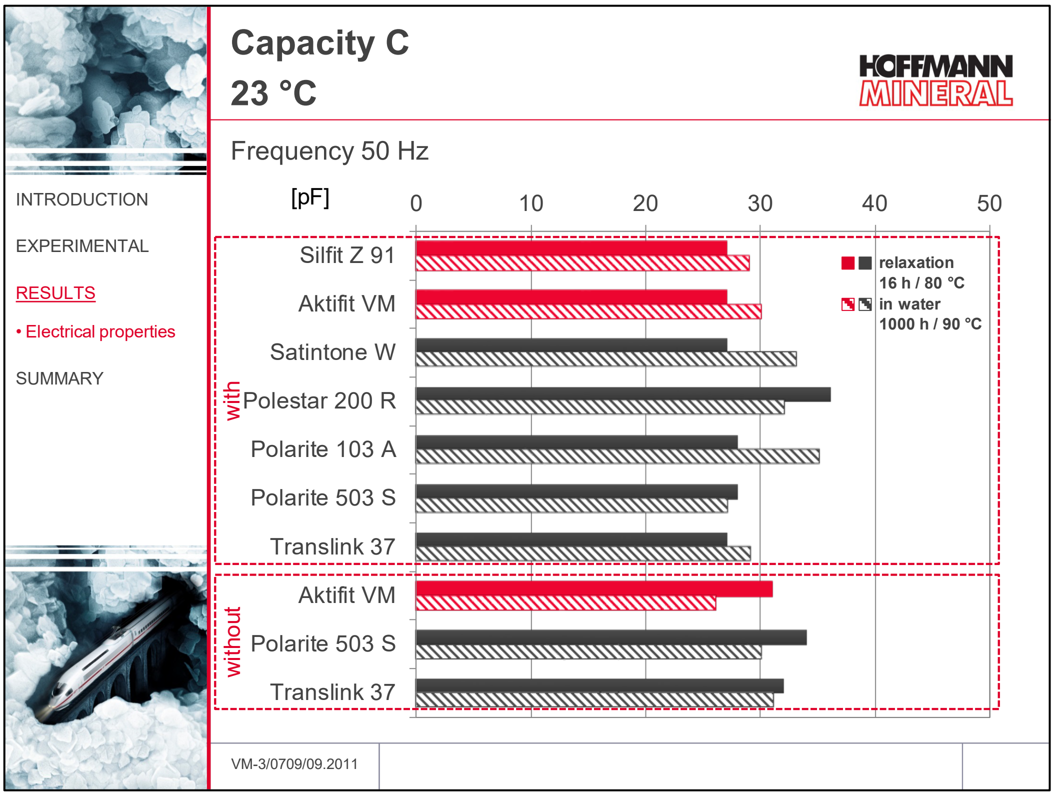
As the usual service temperature of medium and high voltage cables is around 90 °C, the dielectric loss factor was also determined on samples at 90 °C. The industry standard of the dielectric loss factor is about 30 x 10^{-4}.

With added vinyl silane, at the sample temperature of 90 °C after relaxation (dry), the situation is similar to 23 °C, but at a higher numerical level. This way, Silfit Z 91 and Aktifit VM have caught up with Polarite 503 S, and therefore offer an improvement over Translink 37. This improved performance of Aktifit VM vs. Translink 37 remains valid even without vinyl silane addition.

Despite added vinyl silane, the loss factors tan δ after 1000 hours water immersion increase for the untreated fillers Silfit Z 91, Satintone W and Polestar 200 R, while Aktifit VM marks the lowest, i.e. the best result together with Polarite 503 S. While Polarite 503 S shows a slight increase of the loss factor due to the water immersion, Aktifit VM does not give evidence of any negative effect of the water exposure. By consequence, at 90 °C it is also possible to realize an equal low loss factor tan δ with Aktifit VM as with the competitive fillers.

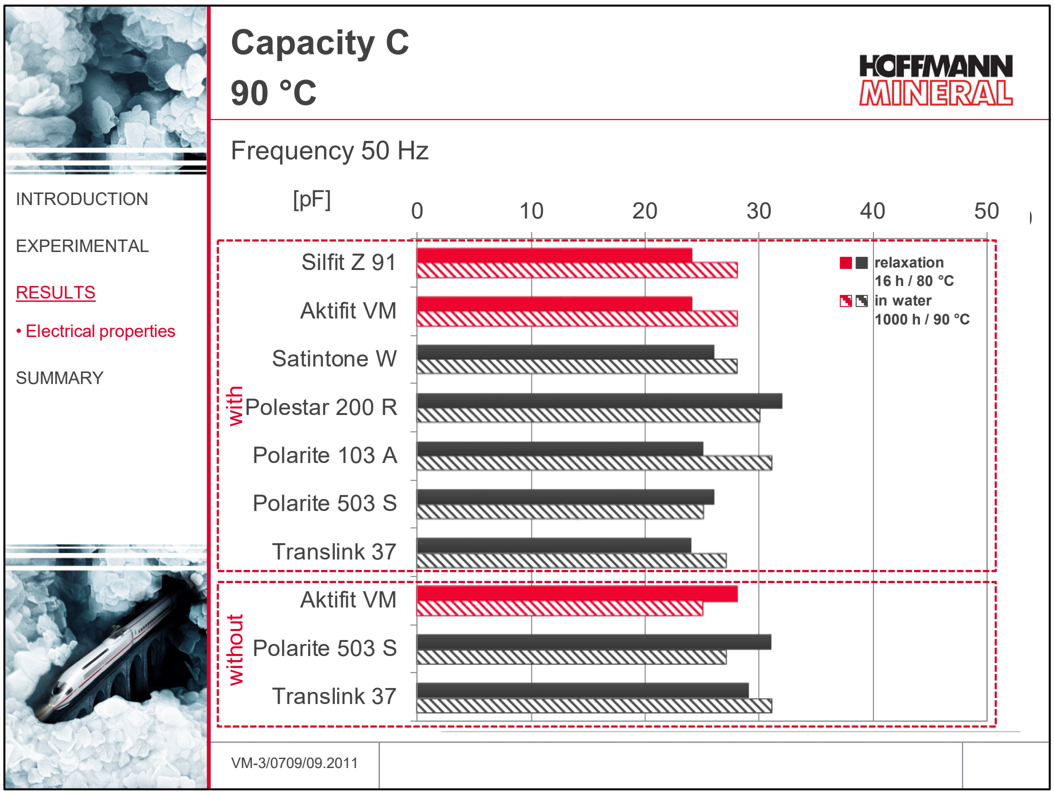
This is also true for the formulations without added vinyl silane. Here it is evident that the loss factor tan δ for Translink 37 - and less distinct for Polarite 503 S - increases to a high value, while the level of Aktifit VM hardly changes.

The required industry standard is met by Aktifit VM with outstanding reliability and this even without further addition of vinyl silane.



The capacity is related to geometry of the specimens and must not be understood as a material constant. Since the dielectric constant εR is a factor for the calculation of the capacity and the specimen dimensions were widely constant, these two electrotechnical parameters correlate well with each other.

At a temperature of 23 °C no significant effects of the fillers or the compound variations can be observed. After water immersion the results are inconsistent too.



At a temperature of 90 °C - which is the usual service temperature - no marked differences can be found between the variations. Nevertheless it should be noticed here that the results for Silfit Z 91 and Aktifit VM are mostly at the lower end and thus better.

4 Summary

Calcined Neuburg Siliceous Earth allows to obtain basic properties comparable with calcined clays along with higher tensile strength levels.

At the same time, benefits concering extended intervals of filter changes may be expected with Calcined Neuburg Siliceous Earth.

The dielectrical properties obtained with Silfit Z 91 are on a similar or slightly superior level compared with the untreated calcined clays.

Aktifit VM offers the best level of dielectric properties - at least comparable with Polarite 503 S - and therefore ranks markedly superior to Translink 37.

The use of suitable surface treated fillers apparently requires no further addition of vinyl silane - at least by using Aktifit VM in the present formulation.

It can safely be stated that Calcined Neuburg Siliceous Earth grades are excellent fillers for the use in medium and high voltage cable insulation compounds.