

Enhancement of Acid Stability of Silicone Elastomers by Using Inert Fillers

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ABSTRACT

This paper shows a novel approach to reduce the aging of silicone elastomers for outdoor insulators by using different filler surface treatments and inert fillers such as barium sulphate (BaSO_4). Silicone rubber composite insulators are used in overhead transmission lines (OHTL) since many years, however under specific environmental conditions, they can show a reduction of mechanical properties, embrittlement and the formation of superficial cracks. Besides other factors, these aging effects can be attributed to strong acids, which can be generated by high electrical field stresses and acidic pollution on the insulator surface. In this study, silicone elastomers used for outdoor insulation, were equipped with aluminum hydroxide with various surface treatments and inert fillers, which provokes a significant improvement of aging performance. Silicone elastomer compounds equipped with barium sulphate show improved aging resistance under acidic environment, like a reduced mass loss when immersed in concentrated nitric acid, decreased crack formation and enhanced stability of mechanical properties with comparable erosion resistance.

Index Terms — silicones, barium sulphate, filler, acid resistance, silanization, insulation

1 INTRODUCTION

THE purpose of high voltage insulators is to isolate components at high voltage potential from grounded structure as well as to transfer the weight of conductors or other components at high voltage potential to the load bearing and grounded structure like lattice towers or foundations.

Insulators can be made from different materials, the most common ones are glass, ceramic or different kinds of polymers. In Figure 1, a typical design of a composite long rod insulator can be seen. The main component is the epoxy resin reinforced glass-fibre core which acts as electrical insulator and carries the mechanical load of components at high voltage potential. In order to improve the weathering resistance, the core is covered by a high temperature vulcanizing silicone rubber (HTV-SIR) which must withstand all environmental

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and artificial stresses. By adding sheds, the creepage distance is increased and leakage currents can be reduced which would lead to a flashover of the insulator by formation of a conductive layer of pollution on the surface of the insulator. Both silicone rubber components, shed and sheath are filled with approximately 50 wt% [1] aluminium hydroxide. Due to this, flame retardancy and resistance against electrical erosion can be ensured.

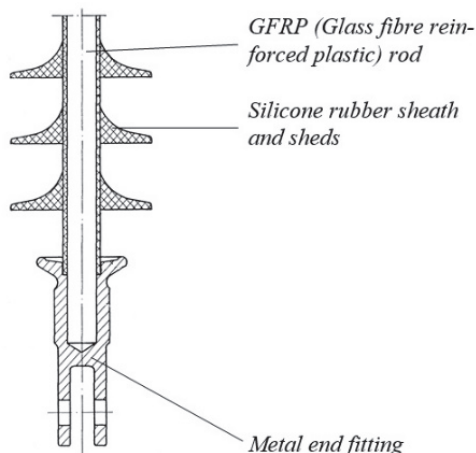


Figure 1. Schematic structure of a composite insulator.

The silicone rubber compound consists of the matrix system polydimethylsiloxane (PDMS) filled with the primary filler silica. PDMS including silica can be considered as silicone base. By changing the molar-mass of the used PDMS, the chemical structure (cyclic, linear, side groups) or modifying the amount of silica, the mechanical properties of the silicone rubber can be adjusted [2]. Producers add crosslinkers such as peroxides, pigments and process-additives to customize the compound for the final application and processing. Due to its ability to create a cooling effect at temperatures between 180 and 560°C, aluminium hydroxide (ATH) is the most common filler for high voltage insulation [3]. The tracking and erosion resistance of ATH filled insulating materials are tested in various standardized test setups, like the inclined plane test acc. to DIN IEC 60587 [4] or the salt fog aging test according to IEC 62217 [5, 6]. In this publication, peroxide cured HTV silicone rubber elastomers are discussed exclusively, but the advantages of inert fillers can be transferred to any other material used for outdoor insulation such as EPDM, room temperature vulcanized silicones (RTV) or liquid silicone rubbers (LSR).

The incorporation of inert fillers, like silica in order to improve mechanical properties [2] and the erosion resistance of silicone elastomers [7] was shown by many authors, however the use of barium sulphate or silica to improve the degradation and acid resistance has not been described so far. Barium sulphate itself originates from barites (natural barium minerals) as well as from synthetic precipitation reaction of barium carbonate and sulfuric acid [8]. Beyond the excellent chemical resistance, the incorporation of nano-sized barium sulphate in polyurethane elastomers leads to an antimicrobial nature of the resulting polymers [9]. Some publications show

that silicone insulators suffer also from biofilms and fungi [10]. However, the main reason for choosing barium sulphate is the chemical inertness.

A second inert filler, which is used for many years is silica. All compounds which are used in this publication are already formulated with silica as the primary filler. Incorporation of silica improves the mechanical properties, which are normally insufficient with unfilled systems. In order to compare the properties of barium sulphate with another inert filler, silica has been chosen.

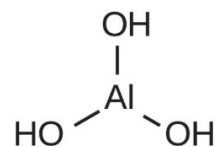


Figure 2. The chemical structure of aluminum hydroxide.

According to relevant literature sources, the stability of aluminium hydroxide against strong acids is relatively low [2, 11]. Surface treatment of this filler has a big influence on the acid stability of the final elastomer [12]. Due to the polar character of the hydroxyl group (-OH), aluminium hydroxide is hydrophilic. The PDMS polymer-network with its methyl groups is hydrophobic, which reduces the ability to bond the filler to the matrix. In order to improve the filler-matrix bonding, the filler can be pre-treated [13]. Besides industrial processes to add silanes to fillers, like “grafting to” or “grafting from” processes, silanes can also be added by spray coating of the filler. Another technology is to incorporate pure silanes in a liquid state or on highly loaded substrates during the compounding process. Also if higher amounts of silane are consumed, the insertion of silanes during compounding is one of the most common technologies due to its simplicity and cost effectiveness. Because of the big impact of the filler treatment to the acid resistance of the product, also this aspect will be discussed in this paper.

Since 2015, Cigré working group D1.62 is treating the topic of “Surface Degradation of Polymeric Insulating Materials for Outdoor Applications”. The group of experts analysed more than seventy cases of deterioration at composite insulators and came to the conclusion, that erosion, cracks and hardness change are the mostly discussed and published topics of aging of polymer insulators for overhead transmission lines and substations [14]. The most important influencing parameters are according to the survey electrical stress, UV-radiation and salt pollution [15], followed by an acidic environment. The main causes of cracks in silicone elastomers seem to be UV-radiation, corona discharges and an acidic pollution. The aim of this research was to increase the acid resistance of silicone rubber elastomers because of its importance for the reliability of high voltage composite insulators.

2 METHODOLOGICAL APPROACH

2.1 MATERIAL COMPOSITION

The materials used in this research are standard products, which are available on the market. The silicone compounds

consisted of a base polymer, aluminium hydroxide, pigment, crosslinker and the optional ingredients are silane, silica and barium sulphate. The base silicone is a translucent PDMS with a density of 1.18 g/cm³ and a tear strength of about 11 N/mm after curing. It is filled with reinforcing silica.

ATH is a white powder with a density of 2.4 g/cm³ and a medium particle size of $d_{50}\% = 1.3 \mu\text{m}$. As cross linker, 2,5-dimethyl-2,5-di(tert-butylperoxy)hexane was used. Barium sulphate is a white powder with a particle size of 1.3 μm in this study. As second inert filler, silica powder with 16 μm and a density of 2.65 g/cm³ was used. The chemical structure of the vinyltrimethoxysilane is shown in Figure 3. It is a liquid of light-yellow colour with a purity of 98%. At 25°C, it has a relative density of 0.968 g/cm³. Within this publication it is referred to as vinyl silane.

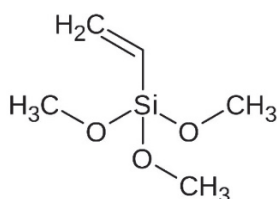


Figure 3. Chemical structure of vinyltrimethoxysilane.

In all formulations, similar amounts of base polymer, pigment, cross linker and filler were used. The total amount of ATH is 52% by weight, for compounds with inert fillers, a total filler amount of 54% by weight was chosen and the remaining filler amount to achieve 54 wt%, was replaced by unsilanized ATH. For the in-situ silanized compound, 2.2 parts per hundred rubber (phr) by weight of silane per was added. Table 1 provides an overview of the used compounds.

Table 1. Silicone rubber (SIR) compound overview.

SIR Compounds	Silane (phr)	ATH (phr/wt.%)	Inert (phr/wt.%)
52 wt% untreated ATH	-	110 / 52	-
52 wt% precoated ATH	-	110 / 52	-
52 wt% insitu coated ATH vinyl silane	2.2	110 / 52	-
ATH +25 wt% SiO ₂	-	55 / 25	55 / 25
ATH +9 wt% BaSO ₄	-	100 / 9	20 / 9
ATH +13.5 wt% BaSO ₄	-	90 / 13.5	30 / 13.5
ATH +18 wt% BaSO ₄	-	80 / 18	40 / 18
ATH +36 wt% BaSO ₄	-	40 / 36	80 / 36
54 wt% BaSO ₄	-	0 / 0	120 / 54

At all compounds, 100 phr silicone base, 0.9 phr peroxide crosslinker and 1.3 phr colour masterbatch was used.

“phr” stands for parts per hundred rubber.

The compounds have been produced in a z-blade mixer type UMI-X 1.0 T of HF mixing group. At the beginning of the mixing process, silicone base compound, 1.3 parts per hundred rubber (phr) of pigment-masterbatch and 0.9 phr of a cross linker paste containing 45% of 2,5-dimethyl-2,5-di(tert-butylperoxy)hexane (Figure 4) were weighed with a mettler toledo ME204T/00 ($\pm 0.001\text{g}$), added to the mixer and homogenised for 120 s at 25 rpm.

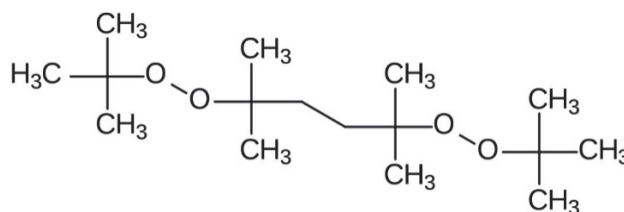


Figure 4. Chemical structure of the used cross linker 2,5-dimethyl-2,5-di(tert-butylperoxy) hexane.

After a homogeneous mass was achieved, 50 wt% of the filler and the silane was added. After 150 seconds at the same speed and 360 seconds at 70 rpm, the remaining filler content was added. After 150 s at 25 rpm and 360s at 70 rpm, final homogenization was made for 600 s at 25 rpm and vacuum. During the whole mixing process, blades and chamber are cooled to a max. temperature of 30°C.

2.2 PREPARATION AND CONDITIONING OF SPECIMEN

The specimen (150 x 150 x 2 mm) were produced on a 1000 kN hydraulic press. Thereafter, the uncured compound was added to the press for 2 minutes at 180°C. After curing for another 10 minutes at the same temperature and a 7 days desiccator preconditioning at $23 \pm 2^\circ\text{C}$ with relative humidity of $50 \pm 5\%$, the desired specimens have been punched for the test. The relative humidity in the desiccator was adjusted by a saturated solution of magnesium nitrate hexahydrate (Merck KGaA).

2.3 ACID IMMERSION

In order to test the resistance of different silicone rubber compounds against acid, specimens of each compound have been immersed in 1M nitric acid for 168 h. Similar tests have been used by several authors [16–18]. To avoid the risk of neutralisation, the specimens were immersed in 100 ml acid. During immersion for 168h, the temperature was kept constant at 30 °C and the pH value was measured every day.

2.4 DETERMINATION OF MASS LOSS

Mass loss was measured on small cylinders with $\varnothing 26 \text{ mm}$ and a height of 10 mm. The difference in mass was determined before and after storage by means of a scale (Mettler Toledo ME204T/00 $\pm 0.001\text{g}$). For this purpose, the test specimens (minimum 3 pieces) were conditioned in a desiccator under the conditions mentioned in Section 2.2. After storage, the specimens were conditioned under same conditions for at least 24h.

2.5 MECHANICAL TESTS

Tensile strength, tear resistance, elongation at break and Shore A were measured to analyse the influence of inert fillers and filler treatment to the mechanical properties. Therefore, those tests were conducted with untreated (original) and acid aged specimens. For all tests, the samples were conditioned like mentioned above. Tensile strength and elongation at break (E@B) were determined according to DIN 53504 specimen type S2. All mechanical tests, except the Shore A hardness

have been performed on a Wolpert universal mechanical test device at a feed rate of 200 mm/min. The original length was 20 mm, calculation of tensile strength and elongation at break can be calculated with the following equations:

$$\text{Elongation at break } \varepsilon_{break} = \frac{l_{break} - l_0}{l_0} * 100 \quad (1)$$

$$\text{Tensile strength } \sigma_{max} = \frac{F_{max}}{\text{Original cross-section}} \quad (2)$$

Tear resistance was measured according to ASTM D624 at a velocity of 500 mm/min. For this test, the specimens were pre-cut at the middle of the sample and the force is measured until rupture occurred.

$$\text{Tear resistance } T_s = \frac{F_{max}}{\text{Material thickness}} \quad (3)$$

The surficial hardness Shore A was performed according to ISO 868. At this test, the cylindric specimens of absorption tests were used. By pushing the test device Durometer Zwick Roell 3114 on the sample surface for three seconds, the value can be read.

2.6 ELECTRICAL EROSION TEST

Outdoor insulators are exposed to humidity and other environmental impacts. In combination with high electrical field stress, discharges can occur. According to [22, 23], local temperatures up to 1200°C occur due to those discharges. For this specific type of electrical deterioration, a test setup was developed. The inclined plane test according to DIN EN 60587 is combining electrical stress with the flow of a contaminant along an inclined sample surface. Because of electrical field applied to the specimen, discharges occur on the edges of the contaminant droplets. A schematic test-setup is displayed in Figure 5. A local concentration of electrical discharges can lead to digging erosion. The effect of building surficial erosion paths is called tracking. According to DIN EN 60587 [9], the test is failed if a current of 60 mA is achieved or the specimens start to burn or 50% of the specimen length is bridged by tracking or digging erosion created a hole in the specimen during the test durance of 6h. In the test setup for this research, a static AC-voltage of 4.5 kV and a contaminant flow of 0.6 ml/min ±10% was used. The procedure was performed on five specimens [24].

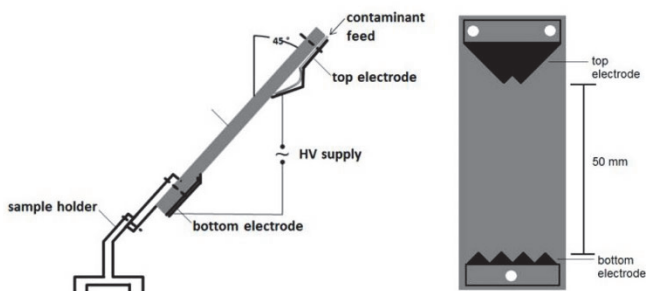


Figure 5. Inclined plane test according to DIN 60587.

3 RESULTS AND DISCUSSION

The aim of this research was to increase the acid stability of standard elastomers to produce high voltage composite insulators or silicone coatings. Therefore, a focus was set on the surficial treatment of the filler aluminium hydroxide (ATH) and the application of barium sulphate and silica as inert fillers. The error bars represent the maximum and minimum values, if not otherwise specified.

3.1 MASS LOSS DUE TO ACID IMMERSION

The test results, shown in Figure 6 reveal another aspect of the application of barium sulphate as a possible new filler. Due to the high density of BaSO₄, 4.4 g/cm³, the weight of the silicone rubber samples is significant higher as compared to ATH filled specimens (the density of ATH is 2.4 g/cm³). One of the big advantages of composite insulators is the light weight compared to other existing materials like glass and ceramic. Because of the mechanical design of the tower structure and handling efforts during shipment and installation, low weight insulators are preferred by the market. Considering this aspect, only a low amount of BaSO₄, or another inert filler should be applied. However, the influence of inert fillers to the mass reduction caused by acid is significant. It seems, that uncoated ATH particles are being dissolved in acid. The in-situ silanized ATH doesn't show a better performance compared to the untreated ATH. Over all ATH filled compounds, a relative mass loss of 3.5 % can be observed. As also described by [19], it is possible, that ATH is being dissolved in the strong acid as shown in Equation (4):

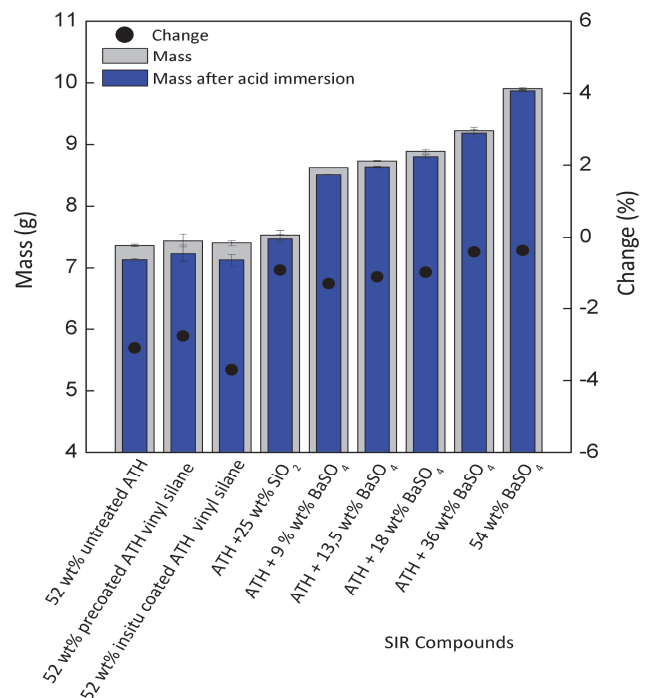
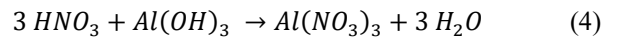


Figure 6. Arithmetic average values of the mass of specimens before and after immersion for 168h in 1M-HNO₃ including the percentual change.

Contrary to the findings of other authors, the ATH filler treatment or the applied silanization, the process doesn't seem to have a big influence on the solubility of the filler particles during acid immersion.

Another noticeable trend is the decreasing mass loss with increasing BaSO₄ content. A significant difference between SiO₂ and BaSO₄ cannot be observed. The positive effect of adding 25.5 wt% of SiO₂ to the compound fits into the trend of decreasing mass loss compared to the increased inert filler content. The lower density of silica leads to a lower weight of the material compared to the BaSO₄ filled specimens.

3.2 REDUCTION OF TENSILE STRENGTH

The silicone rubber elastomer doesn't have to transfer loads during installation. Due to handling (e.g. de-installation of insulator sets) the mechanical properties of the housing material must be good enough to withstand deformations or mechanical stresses of handling. It is a common practice to lift composite insulators by holding it on the sheds or also stand on insulators during maintenance work. Brittle housing materials will be damaged or break under such conditions which can also affect the reliability and safety of the insulator. That's why mechanical properties are an important factor of the performed investigation. As shown in Figure 7 the effect of nitric acid immersion to the tensile strength of silicone rubber is very strong. The filler treatment seems to have a positive effect on the tensile strength. It cannot be concluded, that the performance of inert fillers is overall better compared to ATH filled compounds. The best results were achieved by compounds with inert fillers in a medium amount.

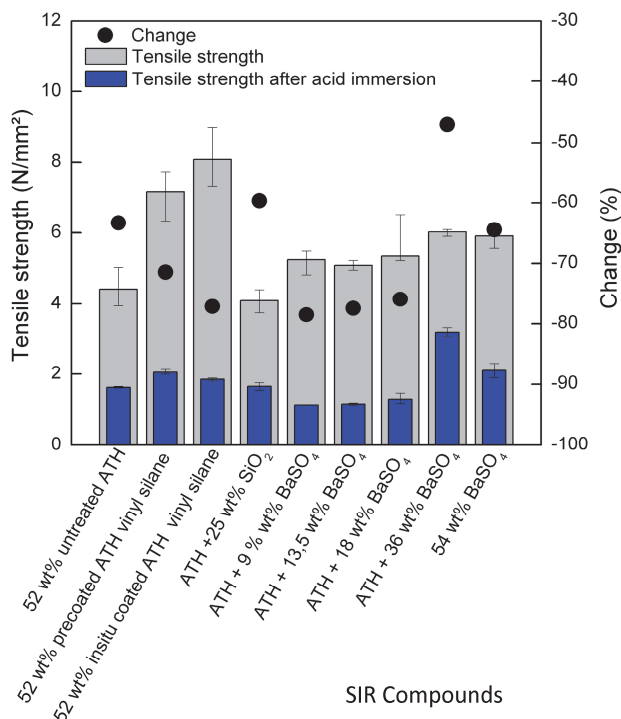


Figure 7. Change of tensile strength due to immersion of specimens (tensile test according to DIN 53504) for 168h in 1M-HNO₃

3.3 ELONGATION AT BREAK

By looking at the elongation of the specimen during tensile test, another effect of the inert filler can be observed. With gradual increase of BaSO₄, the elongation can be enhanced. Silicon oxide shows similar results. Further to that, it can be concluded that compounds with high amounts of ATH are losing their elasticity tremendously up to an ATH content of 13.5–36 wt%. Incorporating ATH with silanes to the compound reduces the initial elongation dramatically. Additionally, an improvement of the acid stability by adding silanes cannot be determined by these results. Also the procedure, how the silanes are incorporated, doesn't seem to have a big influence.

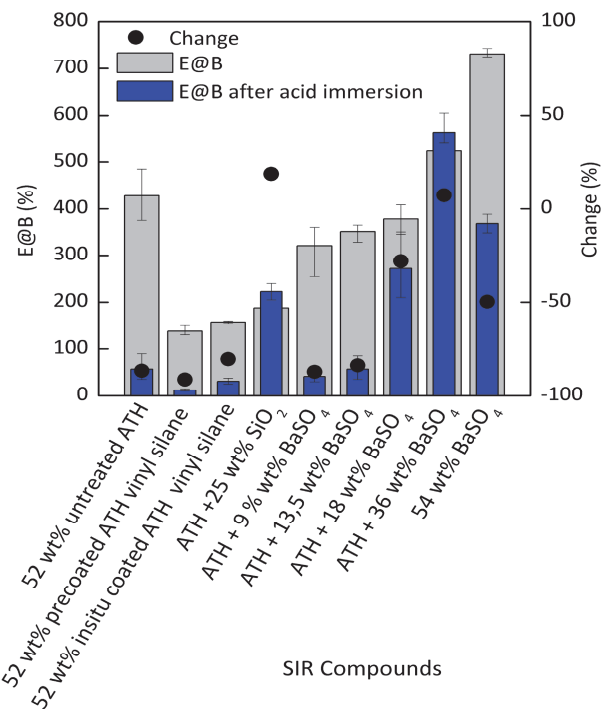


Figure 8. Measurement of elongation at break during tensile test (E@B) before and after acid immersion

3.4 TEAR RESISTANCE

By comparing the tear strength of the investigated silicone compounds, a slightly higher tear strength can be noticed for all inert filled specimens. Silica is already added as the primary filler in order to enhance the mechanical properties of the PDMS compound. This effect is also clear for the SiO₂ filled compound. This specimen shows the best tear strength and the lowest reduction of tear strength due to acid immersion. The silanization process does not seem to have a significant influence on the tear strength. Besides this, the in-situ silanized ATH particles show the lowest reduction of tear strength.

3.5 CHANGE IN HARDNESS

On the BaSO₄ samples a reduced hardness can be observed compared to the conventional compounds. SiO₂ does not seem to have this effect. By acid attack, the hardness decreases in

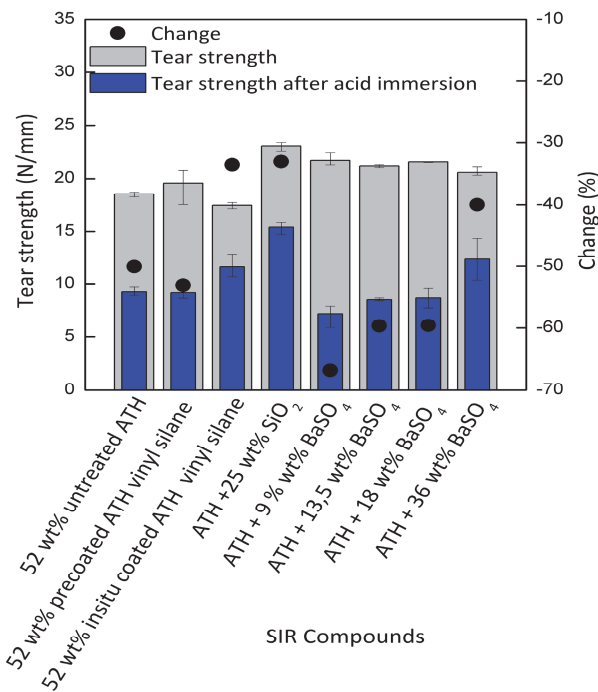


Figure 9. Determination of the tear resistance. Besides the arithmetic average values before and after acid immersion, the maximum and minimum values are shown, as well as the percentual change.

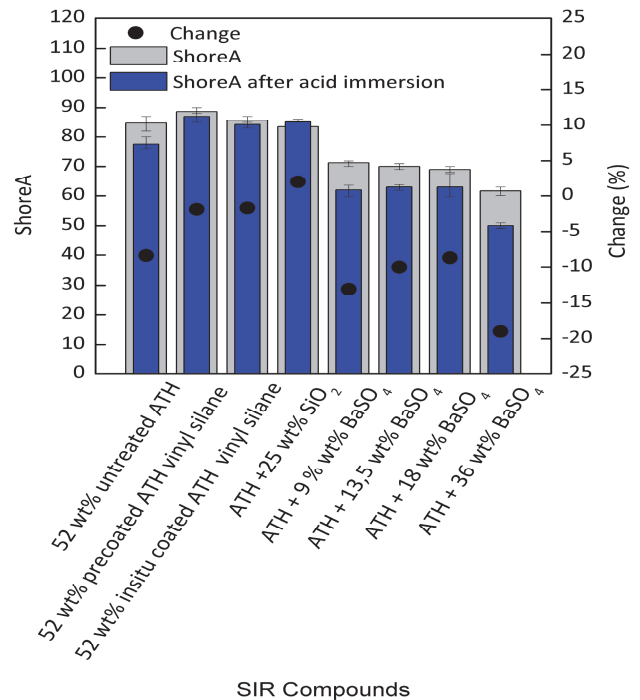


Figure 10. Change in Shore A hardness by acid attack.

most of the cases which can be explained by a possible swelling of silicone samples. The lowest rate of change can be observed at vinyl-silanized ATH filled compounds and the SiO₂ filled one.

The highest change was achieved at high BaSO₄ filled specimens. Adding silanes to ATH filled compounds increases the stability of the hardness and reduces the swelling effect. The method, how the silane is applied, doesn't have a significant influence. It cannot be concluded that SiO₂ shows a better performance compared to BaSO₄ filled compounds due to the difference in mode of changing. However, the difference between aged and unaged specimens is lower compared to the barium sulphate compounds.

3.6 RESISTANCE AGAINST ELECTRICAL EROSION

At electrical stresses of 5 to 7 kV/cm, corona discharges start on water droplets [3]. If the electrical field is limited by metal hardware, high local field stresses will occur due to pollution and humidity. A very important aspect is that ATH or another flame-retardant filler is still necessary for outdoor insulation. The used inert fillers aren't flame retardants, however tracking length and eroded mass wasn't affected until reaching a threshold value, which is in the range between 18 wt% w/w and 36 wt% by weight. The best tracking and erosion resistance was achieved by the compound with in-situ coated ATH. Compounds with 36 wt% BaSO₄ by weight and higher fail in the test. This result can be considered as a not acceptable for the application as electrical insulators. Filler treatment seems to have a slight influence on that property.

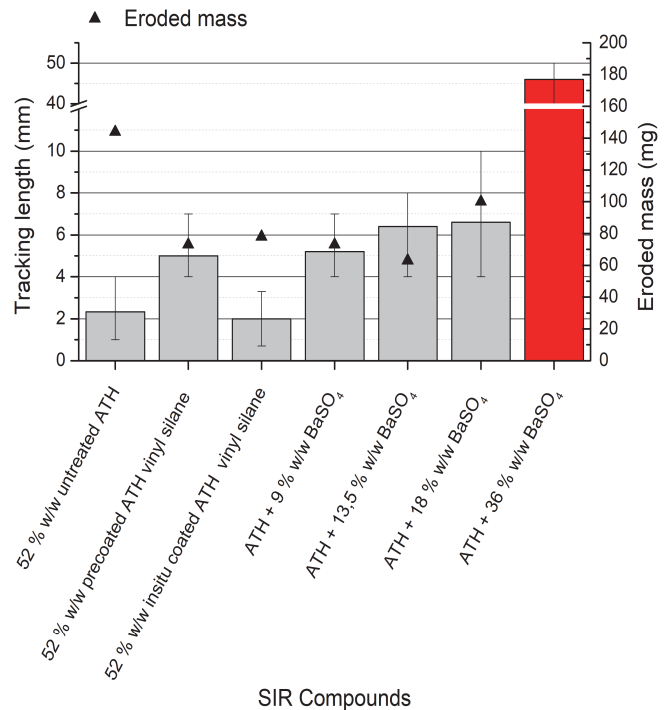


Figure 11. Results of inclined plane test.

4 CONCLUSIONS

The purpose of this investigation is to improve the acid resistance of VMQ silicone rubber by adding inert fillers. Since acids can be created by electrical stress and the nitrogen in the air, acid resistance is one of the most important properties of silicone composite insulators [3]. Besides the

general applicability of silicone rubber elastomers equipped with inert fillers for outdoor insulation, the effect of different kinds of inert fillers to the aging properties was evaluated. In addition to the type and quantity of inert filler, the ATH surface treatment and process was considered. By measuring the mechanical and electrical properties prior and after acid storage, the relative change was analysed.

The main findings of this publication are:

- Acid aging of ATH filled silicone elastomers leads to mass loss and a significant reduction of mechanical properties, such as Tensile strength, tear resistance and elongation at break.
- Due to the substitution of untreated ATH by inert fillers, the mass loss during acid immersion and the reduction of mechanical material properties can be reduced significantly.
- Filler-Functionalization improves the filler matrix adhesion which can be seen at the tensile strength, but doesn't lead to an improved acid stability.
- In order to achieve sufficient tracking and erosion resistance, the maximum content of inert fillers should be in the range of 18–36 wt%. Additionally, Meyer et al. showed that also the tracking resistance of silica filled silicone rubber compounds are comparable to ATH filled compositions, due to the high thermal conductivity [7].
- Due to the improved adhesion to the silicone matrix, the lower density, higher tear resistance and elongation at break and comparable acid resistance with barium sulphate, silica would be the preferred inert filler for silicone elastomers.

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