Degradation analysis of RTV-SiR based composites under both polarities DC voltage for insulators coating

Israr Ullah a, Mohammad Akbar a, Haris Ahmad Khan b,∗

a Faculty of Electrical Engineering, Ghulam Ishaq Khan Institute of Engineering Sciences and Technology, Topi, Swabi, KPK 23460, Pakistan
b Farm Technology Group, Wageningen University & Research, Wageningen, The Netherlands

1. Introduction

The insulation system of high voltage transmission lines is of utmost importance for reliable and uninterrupted power supply of electricity to consumers. The ceramic insulators were introduced in the power transmission systems back in 1880 and since then, are being used extensively around the globe for many decades because of their good dielectric properties and mechanical strength [1,2]. These insulators are heavy and quite labor-intensive during erection as well as maintenance in case a fault occurs. When installed in the polluted region, these insulators are prone to flashovers even if generous insulation is provided. Polymeric insulators offer a good alternative to a ceramic insulator for use in pollution environment along with several other benefits [3–5]. In Pakistan, where 100% high voltage AC transmission lines are constructed with ceramics insulators, it is impossible economically and technically to replace them with polymeric insulators. The operating electric utility companies in Pakistan remained constantly engaged to counter the flashover problems. Several methods such as hand cleaning, extending creepage distance, etc. were adopted as practiced internationally [6,7]. Besides other techniques against flashover, insulator coating with polymeric materials has been proposed and adopted [8].

When a polymeric insulator is employed in service, it is energized at a certain voltage level and remains exposed to weather pollution and other environmental stresses, changes can occur in the characteristics of the insulator. These stresses may cause changes in characteristics of the insulator which could be physical (erosion & tracking), chemical (depolymerization), or physic-chemical (hydrophobicity). Such changes may adversely affect the insulator performance which is collectively termed as insulator aging. An insulator that is energized in actual service conditions is said to be exposed to natural aging. On the other hand, when a laboratory test is performed to reproduce service stresses at an accelerated pace to shorten the test duration, it is termed as accelerated multi-stress aging. In the present research work, multiple environmental and electrical stresses were reproduced and implemented in an accelerated way in a controlled weathering chamber to simulate the actual

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ABSTRACT

In a high voltage transmission system, insulator pollution highly affects the reliability of operation. Air pollutants from surrounding environment accumulated on insulator surface after some time during field operation. Which ultimately polluted the insulators and severely affects their performance in high voltage lines. To combat the pollution effects, high voltage ceramic insulators are coated with room temperature vulcanized silicone rubber (RTV-SiR) as a one-time interminable solution these days. In this study, an attempt has been made to investigate the aging behavior of several composite coatings. Different test samples for coating with various loading concentrations of alumina tri-hydrate (ATH) and silica (SiO2) were fabricated and subjected to multi-stress aging under both polarities of DC voltage. A weather cycle representing a heavily polluted region of Pakistan was simulated in a chamber for 10,000 h. Various diagnostic techniques of aging like hydrophobicity assessment, leakage current monitoring, Fourier transform infrared spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) were utilized. The aging effect on investigated composites was also evaluated through measurements of mechanical properties. Considering the cumulative effect on aging detected through the measurement of several relevant parameters, it has been established that hybrid composite is most resistant to aging. Further, the polarity effect on aging was manifested showing positive polarity as having a more adverse effect on aging as compared to negative polarity.
field conditions representing a polluted environment. Selected coating composites were then energized and investigated under the synergetic impact of multiple stresses involving thermal, electrical, UV, humidity, salt fog and acid rain.

The first step of pollution flashover begins with the accumulation of pollutants on the surface of insulators [9]. During wetting of these insulators due to high humidity, fog, and rain, a conductive path create which allows leakage current to flow on the surface and dry bands are formed. As a result, the electric field distributes on the surface non-uniformly and dry band arcing takes place. Under the severe level of pollution, these dry band arcing leads to complete flashover along the string of insulators. The polymeric coatings offer the advantage of not forming a continuous layer of wetting as water beads away from the surface due to its hydrophobic nature. Over time, the coating may lose its hydrophobic property and lead to flashover [10,11]. Amongst the family of polymeric insulating materials, RTV-SiR has shown superior hydrophobicity, leakage current suppression, temperature resistance, UV resistance, and high flame retardancy [12,13]. The RTV-SiR due to its molecular structure has the hybrid nature of rubber as well as inorganic nature [14].

Many researchers used RTV-SiR coatings to enhance the anti-pollution performance of ceramic insulators in heavily polluted areas. The performance analysis was performed with different characterization methods and techniques like hydrophobicity classification, scanning electron microscopy (SEM), Fourier transforms infrared spectroscopy (FTIR), and analysis of leakage current [15-18].

Silicone rubber (SiR) is available in various types offering a variety of properties for different applications and environments. Some of the most commonly used SiR in electrical and electronics applications are: 1) High temperature vulcanized silicone rubber (HTV-SiR) and, 2) Room temperature vulcanized silicone (RTV-SiR). Besides other applications in day-to-day use, silicone rubber provides advantageous properties in electrical and electronics applications i.e. in insulation, sensors and actuators. RTV and HTV silicone offer better characteristics among other silicone rubbers (i.e. high tensile strength, better curing, easy processing, actuation, compressive mechanical behavior and better hydrophobicity) [19]. Several studies show further improvements in the properties for various applications by incorporating carbon nanotube (CNT), graphic fillers (GR) as a filler [20,21]. As an outcome, RTV-SiR has shown excellent weather-resistant ability and good insulating properties over other polymeric insulators. However, when exposed to aggressive environmental stresses such as UV radiations, fog, humidity, temperature, pollution, and acid rain RTV-SiR materials may degrade [22,23]. Therefore, to improve the service life of the RTV-SiR coatings, the inclusion of inorganic fillers has been suggested wherein its type, shape, and size are considered as important. A filler of different types and sizes enhances the different properties of the composites. The work in [24] compared composites of different sizes of micro and nano-based fillers. The authors found that the nano-based filler reduces leakage current and is more resistant to erosion. There are many micro and nano-based inorganic fillers that can be used to prepare polymer composites [25]. Silica (SiO₂) is one of the most promising inorganics fillers as it offers anti-tracking capability, good mechanical strength, more erosion resistance, and good thermal ability [26]. Micro and nano-based silica has been used as filler with varying percentages in different studies [27]. The performance of nano-based silica RTV-SiR composites showed superior performance compared with micro-based silica composites. KhatKid et al. exposed composites in the multi-stress environment and different characterizations were performed using SEM, FTIR, leakage current analysis, classification of hydrophobicity, and hardness test [28]. Moreover, nano-silica fillers were also used in other polymeric materials like EPDM and epoxy. The effect of nano-silica in EPDM and epoxy also showed good results after being analyzed in the multi-stress lab aging [29]. The other filler which is prominent for polymeric composites is alumina tri-hydrate (ATH). In a research study, it was proclaimed that the composites of ATH-filled RTV-SiR showed better performance in comparison to silica-based composites. Moreover, it was shown that silica-ATH hybrid composite has higher hydrophobicity than other composites [30]. Khan et al. found that the ATH-filled SiR composites have better tracking performance and mechanical strength than the silica-based composites [31]. In more recent work, it was reported that ATH-filled SiR composites have good leakage current suppression performance and also possess good thermal stability [32]. It is reported in the literature that polluted insulators performed poorly under DC stress as compared to an equivalent AC stress [33,34]. Kannan et al. [35] noticed that DC insulators collect more pollution in a given environment compared with AC insulators. Similarly, in two different studies, the authors found that leakage current and associated surface degradation of insulators HVDC system are more than the HVAC system [36,37].

Despite a great deal of literature, researchers around the globe are still consistently making efforts to better understand polymeric materials and their composites which could offer long life in all sorts of environmental conditions prevailing globally. This is particularly true for HVDC lines operating in multi-stress conditions and under severe pollution. It is in this context that we decided to study and understand the service performance of RTV-SiR coating of ceramic insulators of various formulations to determine the most suitable material suiting our environment. A variety of filler reinforcement was implemented in silicone rubber to see its effect on the aging behavior (life expectancy) of these materials in an accelerated environment.

Firstly, various specimens of RTV-SiR coatings with reinforced micro and nano-silica/ATH fillers were fabricated. These composite samples were placed in a weather chamber with simulated the environmental conditions of an industrial city of Hattar, Pakistan. The chosen area was preferred for long-term aging study because of its high pollution index due to a mix of various pollution-causing industries. The weather cycle of the specific area was designed based on 30 years of meteorological data acquired from the Meteorological Department, Pakistan. The calculated weather cycle consists of two parts i-e the summer cycle and the winter cycle. Six different carefully chosen samples were exposed to multi-stress environmental aging under DC voltage for a longer duration of 10,000 h. The aging performance of samples was analyzed using different measured data and through the deployment of various characterization methods, like leakage current, ultimate strength, hardness, weight loss analysis, FTIR, and hydrophobicity classifications. Detailed weathering evaluation of RTV-SiR composite coatings was carried out in terms of electrical, chemical, and mechanical properties and was compared with the corresponding data of unaged counterparts. Polarity-specific effect on aging was also investigated and analyzed.

2. Experimental preparations

2.1. Fabrication of composites

The base RTV-SiR (RTV 615) was acquired from WilKON distributors, China while a silica nano-filler of 10 nm was procured from Wuhan, Newreach Chemicals, China. ATH fillers of 15 µm and 20 µm were procured from Huber Engineering Materials. The auxiliary additives like stearic acid (SA), Zinc Oxide (ZnO), tetra-methyl-thiram-disulfide (TMTD), diethyl phthalate (DOP), and mercapto-benzo-thiazole (MBT) for the preparation of the composites were procured from local industries. Primarily silicone rubber (RTV 615) was selected as the base polymer having low viscosity vinyl polydimethylsiloxane silicone rubber (rang 3000-7000 cps). This type of SiR is a two-part silicone rubber compound; a liquid silicone rubber, and a curing agent with a ratio 10:1. Preparation of the specimens was performed in the following three steps. In the first step, dry filler was gently added to RTV 615-A (liquid state), while keeping the mixing speed low to properly mix the fillers. For blending of base RTV-SiR with micro ATH and micro-nano ATH/SiO₂ fillers ASTM D1418-10a standards were followed [38]. The micro fillers
of ATH/ SiO\textsubscript{2} were stirred for 15 min in a 100 ml solution of ethanol followed by ultra-sonication for 30 min. The ATH/silica filler was first kept in an oven for 24 h and maintained at a temperature of 150 °C to remove any moisture. After solution mixing, when the fillers got properly wet, the mixing speed was gradually increased to 50 rpm and was kept unchanged until the mixture did not show any visible signs of lumps. In the second step, after 2–3 min of cooling, the curator RTV-615B was stirred with the prepared mixture with the help of a sonicator. To eliminate bubbles in the mixture, degasification was performed with the help of a vacuum desiccator. Thereafter, a hardener was added and mixed with the prepared solution. Finally, in the third step, the mixture was poured into steel molds of the required shape and size. Steel molds were kept at room temperature for 24 h to set the compositions. RTV 615-B was added to the mixture after 2–3 min of cooling and mixed at low speed for two minutes with a sonicator to ensure uniform filler dispersion. The mixture was then degassed in a vacuum at 27 mm-Hg until the bubbles were entirely eliminated. Vulcanization was done using condensation-curing technique at room temperature in an environment of controlled laboratory engaging a fully automated material processing and metering systems to prepare the samples. As per standard DL/T 627-2012 [39], humidity and temperature of silicone rubber were selected as 56–58% and 25 °C ± 2 °C respectively, using a programmable constant temperature and humidity chamber. After the initial mixing steps and addition of cross linkers, fillers and other auxiliary materials, in the third step, the silicone rubber mixture was poured into moulds and left to cure for 24 h at room temperature. The samples were then post-cured in a 90 °C oven for 4 h. Table 1 shows the filler wt% ratio in the composition of each sample. Samples with codes: PS1, PS2, and PS3 were selected to be exposed to positive DC stress, while the remaining three samples (NS1, NS2, and NS3) were energized with negative DC stress. The DC stress magnitude was kept the same for all polarities.

2.2. Test chamber

A chamber measuring 152.4 cm × 91.44 cm × 121.92 cm was fabricated using acrylic sheets and adopting guidelines of Electric Power Research Institute (EPRI), USA [40]. The specimens were installed in the chamber as recommended in IEC/TR 62730 [41] and were energized with a DC source of ±2.4 kV. Three conducting bars were installed in the chamber to connect positive, negative, and ground terminals to suitably stress the insulating samples. Samples were placed in soft edge copper electrodes on both sides and mounted with the main rods in the chamber as shown in Fig. 1.

To maintain the temperature within the chamber, three electric heaters of 1000 W each were installed along with a temperature sensor. The winter and summer cycles of Hattar, an industrial city of Pakistan were designed using weather data of the Pakistan Meteorological Department. Multi-stress accelerated aging technique is applied in polymeric insulators as a lab aging.

Investigation to reproduce the long-term impact of a real field environment in a short time span. In current research 10,000 h, aging was performed according to IEC1109/IEC61109 modified standard [42, 43], which represents the actual field aging of approximately 15 years for the specified area. Lab aging time for the specific area is calculated based on acceleration factor whereby 28 days of lab aging is considered equivalent to 365 days of the actual field with an acceleration factor of 365/28 or roughly 13. Hence, accelerated aging of 10,000 h is approximately equivalent to 15 years of exposure in an outdoor environment. Lab aging time duration of 28 days of lab for 365 days in the field is based on meteorological data of about 40 years of the selected region. Furthermore, various IEC standards are available in actual and modified form, utilized worldwide by researchers for the aging investigation of composites insulators in a shorter time.

The summarized duration of cycles designed and associated environmental parameters are shown in Table 2. Weather cycles were then simulated in the controlled aging chamber in an accelerated mode according to the above-mentioned conditions. For UVA radiations, six UVA-340 fluorescent lamps having a rating of 20 W each were installed to maintain the UV weathering requirement of 1.1 mW/cm\textsuperscript{2}. The intensity of UV radiation was measured with the help of the Lutron light meter, model UV-340A. A humidifier, having a sensor and controller was used to adjust and measure humidity inside the chamber. A nozzle was placed in an appropriate position in the chamber to apply salt fog and acid rain. For acid rain, a dilute solution of HCl having a pH of 4.5 was used. To energize the samples in the chamber, the creepage distance was maintained as 25 mm/kV following IEC61109 and IEC60815 standards [44,45]. A small piece of each sample measuring 2 × 2 mm was taken off from each sample after every cycle for further tests and characterization.

3. Parametric measurements and characterizations

The aged samples were critically analyzed through various relevant measurements and by employing various characterization techniques. A summary is given below.

3.1. Hydrophobicity assessment

The hydrophobicity of a polymeric insulator plays an important role to analyze its surface condition. Multiple-stress aging may cause the scission of important hydrophobic groups in the molecular structure of the polymer. In this study, measurement and analysis of surface hydrophobicity of each sample were carried out using a method developed by Swedish Transmission Research Institute (STRI) and was designated as STRI hydrophobicity classification guide [46]. This method assigns a class to surface hydrophobicity varying between HC1 to HC6, representing the least loss of hydrophobicity to maximum loss of hydrophobicity, respectively. The STRI procedure was adopted after every cycle to measure any loss of hydrophobicity. The procedure requires spraying tap water for 20 s and taking images with the help of a high-resolution camera within 10 s after the spray. The images are then compared with various classes of hydrophobicity and are accordingly assigned a class designated in the STRI guide from HC1 to HC6.

3.2. Leakage current investigation

Leakage current measurement through the test samples throughout the aging period offers useful data to know the stressed-caused degradation. Towards this end, the leakage current was measured after each aging cycle using UT-70B digital multimeter.

3.3. Scanning electron microscopy (SEM)

Surface morphological changes due to aging in all samples are investigated at the micro-level using Scanning Electron Microscopy (SEM). Microscopy of each sample is performed after the completion of the entire aging test. Each specimen is coated with gold sputtering of 2.5 nm before the microscopy which is necessary for insulating materials investigations.

### Table 1

| S. No | Composition       | Sample code | Quantity |
|-------|-------------------|-------------|----------|
| 1     | Unfilled RTV-SiR  | PS1, NS1    | 2 samples (one for each polarity) |
| 2     | 10% micro-ATH     | PS2, NS2    | 2 samples (one for each polarity) |
| 3     | 10% micro-ATH +5% nano-silica | PS3, NS3 | 2 samples (one for each polarity) |
3.4. FTIR spectroscopy

FTIR spectroscopy of each test sample was carried out after each aging cycle to measure the molecular changes in the functional groups. Absorption peaks of each sample were obtained with the help of the FTIR spectrometer (Perkin-Elmer spectrum 2000). Potassium bromide powder was used as a transparent material for all the tests.

3.5. Hardness and weight measurement of aged samples

To measure the impact of multiple stresses on the mechanical properties, the hardness and weight of each sample were measured after each cycle. Hardness was measured with the help of the Shore-A Durometer (0–100HA) following ASTM D2240 standard, whereas the weight was measured with the help of the GT-500 electronic compact scale. These two parameters also show the water absorbance behavior of every sample.

3.6. Stress-strain curve evaluation

For reliable operation of the power transmission system, the mechanical integrity of outdoor composite insulators is of high importance. As per the requirement of the test, all the specimens were cut in dumbbell-shape, having a 24 mm gauge length and 5 mm gauge width. A universal testing machine (Instron 5567, Massachusetts, USA) was used which had a strain rate of 10 mm/min and a cell load of 30 kN. The stress-strain curve was obtained for each sample using the UTM machine where the ultimate tensile strength of each test specimen was assessed as per ASTM D1708–06a. Measurements were conducted both on un-aged as well as on 10,000 h-aged SiR composite samples.

4. Experimental results

4.1. Hydrophobicity assessment

To better understand the aging performance of composite coating material in an outdoor environment, the measurement of surface hydrophobicity is of great significance. Surface hydrophobic properties of composites may change due to the formation of the hydroxyl groups and the occurrence of crosslinking in polymer chains under multi-stresses with a constant DC electric field. To compare surface hydrophobicity, variations in water repellency of all specimens were investigated after every 1344 h (4 aging cycles) using the STRI hydrophobicity classification guide [46]. Based on the procedure outlined in the standard, 5–7 photographs of each sample were taken with a digital camera and compared with the standard image of STRI guide to better determine the hydrophobicity class of the composites. STRI guide allocates hydrophobicity class between HC1 and HC7 representing the most hydrophobic to the least hydrophobic surface, respectively. Initially, all RTV coating blends exhibited good hydrophobicity falling in class HC-1 according to STRI classification as shown in Fig. 2. In the case of sample PS1, NS1 and PS3, hydrophobicity level dropped to HC-3 while remaining composites maintained their hydrophobicity level as HC-2 up to the first four aging cycles representing an aging time of 1344 h. Significant recovery of hydrophobicity to level HC-1 can be seen in filled composites at the end of 2688 hours of aging.

A prominent decline of hydrophobicity class was noticed in each sample beyond 4032 h of aging. Unfilled silicone specimens PS1 and NS1 for instance, reached the maximum loss of hydrophobicity to level HC-6 after 5376 h with only the least recovery to HC-5. Pure silicone (PS1) experienced relatively more degradation and hence its hydrophobicity class fell to HC5-HC6 at the end of aging showing thereby its least hydrophobic character among all other studied samples. The poor hydrophobicity of the composites may change due to the formation of the hydroxyl groups and severe photo-oxidation under UV radiations which results in the formation of –CH–OH from hydrophobic –CH2– to hydrophilic –CH2OH, thus creating (Si–CH2OH) hydroxyl group with peroxide due to the reaction of oxygen contents with PDMS [47]. Moreover, loss of low molecular weight silicone (LMWs) polymer (known as “cyclic”) may also be a possible reason for the slow recovery of unfilled samples [48].

In the case of samples PS2 and NS2 filled with micro ATH, the highest drop of hydrophobicity to class HC-4 is noticed after 5376 h of aging. However, significant reclamation was noticed in the remaining phases and finally, its hydrophobicity recovered to class HC-3 and HC-2, respectively. Similarly, PS3 and NS3 suffered the maximum hydrophobicity loss to class HC-5 after 6720 h of total aging but finally recovered.
to class HC-2 showing a recovery of better water repellency characteristics. Reasons for hydrophobic recovery could be the reclamation of the polymer chain, resistance to chain scission, high mobility of SiR backbone chain, and ease in the diffusion of cyclics from the bulk to the surface of these composites [49].

4.2. Leakage current investigation

Surface leakage current of test samples was measured to investigate their aging as a function of time under the influence of positive/negative DC along with other environmental stresses. The current was measured after each weathering cycle which was 264 h and 408 h for summer and winter, respectively. Leakage current was measured using 1 MΩ resistor in series with ground terminal. The voltage drop across the resistor was measured using a UT70B digital meter. Leakage current value can then be calculated from voltage drop. On each sample, the measurement was repeated 4–5 times and an average value of current was recorded after each cycle of aging. Data of measured leakage current on all samples under positive and negative DC voltage are shown in Fig. 3a and b, respectively. It can be seen from the results that leakage current did not follow any specific pattern as has a tendency to vary from sample to sample depending on surface degradation with aging particularly in the context of its hydrophobicity performance. The recovery of surface hydrophobicity also takes place in a complex manner depending on the polymeric material itself which affects the flow of leakage current.

Initial values recorded for all specimens were in the range of −3.2 to −3.9 μA and +3.8 to +4.1 μA for negative and positive DC stress, respectively. It can be seen that regardless of filler loading and polarities of DC stress, all samples showed a gradual increase in leakage current. With aging, unfilled SiR samples PS1 and NS1, showed the maximum increment of 10.65 μA and 6.62 μA in leakage current, respectively. Additionally, both specimens showed a hike in leakage current from the

Fig. 2. Variation in hydrophobicity classes with aging cycles.

Fig. 3. Leakage current as a function of aging: (a) samples energized under positive DC, (b) samples energized under negative DC.
13th to 18th cycles of aging and reached the highest final values of $+14.74 \mu A$ for PS1 and $-9.91 \mu A$ for NS1. High loss of LMW silicone due to the synergetic effect of multiple stresses in unfilled samples could be the most probable reason for such an increase [48]. As compared to pure silicon specimens, composites filled with micro ATH observed a minor growth in leakage current. Samples PS2 and NS2 for example exhibited an increase in leakage current from start to end of aging as 5.01 $\mu A$ and 3.73 $\mu A$, respectively. The better leakage current may be due to the remaining alumina which enhances the protective shielding after the evaporation of dehydrated water at high temperatures thus improve the insulating properties [50].

Moreover, hybrid composites among all samples showed a lower value of leakage i.e. better current suppression performance over the complete aging duration. For instance, sample PS3 showed a total increase of 1.97 $\mu A$ from an initial (3.88 $\mu A$) to final (5.85 $\mu A$) over the entire aging time. Similarly, NS3 showed a marginally lower increase of 1.44 $\mu A$ in leakage current. Hydrophobic layer formation over the surface contaminants due to nano-silica particles may be the probable reason for such a lower magnitude of leakage current [51]. Furthermore, high surface resistivity, molecular changes, and diffusion of LMWs polymer from the bulk to the surface in filled samples may also result in lower leakage current. Somewhat similar observations are elucidated in the hydrophobicity-leakage current relation reported earlier [47].

Comparing the overall leakage current data, it is found that samples under positive polarity have a high leakage current as compared to negative polarity indicating a significantly severe pollution effect and high rate of degradation under positive voltage. This may be due to the migration of more ions onto the surface resulting in high surface conductivity under positive polarity of DC akin to the process of electrolysis discussed earlier in [50].

### 4.3. Scanning electron microscopy (SEM)

The surface morphology of each aged coating specimen is investigated through scanning electron microscopy. Surface irregularities, dents, cracks, pores and agglomeration, etc., induced in materials due to stresses impact is evaluated via SEM micrographs. In Fig. 4, various surface roughness and fracture are analyzed and explained accordingly. Perceptible degradation of coating blends can be observed at the micro-level as shown in Fig. 4. Unfilled sample PS1 under positive voltage presented deep dents with irregular surface cracks, agglomerations, and surface pores. Whereas sample NS1 showed comparatively fewer pits with rare cracks instead of profound depths and apertures. A higher degree of polymer chain scission and oxidative degradation due to UV, thermal and electrical stresses may lead to such cracks and dents. Comparable observations are discussed in literature [22,52].

Sample PS2 under positive voltage is affected fairly high as compared to NS2 under negative polarity DC but unlike pure silicone, no major dents are perceived for both direction voltages. For instance, PS2 exhibited slightly high particles agglomeration on the surface however in counterpart under negative DC these changes are at a minor level. Similarly hybrid composites PS3, depict relatively small and even accumulations with minor surface irregularities for positive voltage while NS3 energized under negative polarity exhibited minute cluster with pits however no major rupture and surface tracks are obtained. Among all specimens, PS3 shows better overall performance against the combination of severe stresses followed by NS3 in negative DC voltage. The reason may be the interaction of more conductive ions and their flow in positive DC voltage [50]. Comparative SEM analysis of changes at the micro-level describes the better performance of coatings with hybrid fillers.

### 4.4. FTIR spectroscopy

To evaluate the alterations in chemical structure, Fourier transforms infrared (FTIR) spectroscopy was performed after the combined summer and winter cycles equivalent to 672 h of aging. To simplify the analysis, four spectrographs out of a total of fifteen were selected after aging with equal intervals of combined summer-winter cycles for each polarity and are presented in Figs. 5 and 6. Additionally, the chemical group with each absorbance peak against the corresponding wavenumber is indicated in IR spectra. To assess the extent of variations in chemical structure, the percentage absorption peak height of significant functional groups at the end of the aging test is presented in Table 5.

The percent growth and drop in heights of absorption peaks are tabulated for each aged specimen to its non-aged taking the peak height value of absorbance in the un-aged state as 100% for reference. The O-H hydroxyl peak in the range of 3200–3700 cm$^{-1}$ wavenumber is considered essential for hydrophilicity in PDMS. The appearance of bumps in IR spectra amid wavenumber 3200 cm$^{-1}$ and 3700 cm$^{-1}$ can be seen in unfilled silicone samples for both positive and negative DC. Alike other results discussed earlier in this study, the peaks of curvature are dominant in the FTIR graph for positive DC as shown in Fig. 5. For instance, this value is measured at 385.2% for PS1 and 221.1% for NS1 showing the highest percentage increase in this region, which portrays more degradation and loss of hydrophobicity in pure silicone samples. A

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**Fig. 4.** SEM images of aged specimens.
similar trend is also observed in the previous sections of hydrophobicity classification and leakage current. This may be attributed to the rise of hydrophilic –OH contents due to the materialization of Si (CH$_2$CH$_2$Si) and Si-OH bonds on the surface in addition to the photodecomposition triggered by UV radiation [53].

Sample PS2 shows a 171.3% increase in OH content due to aging, followed by PS3 (139.3%). Similarly, under negative polarity, the increase in OH is 110.5% for NS3 and 119.1% for NS2 thus showing relatively less loss in hydrophobicity compared with positive polarity. In both polarities, hybrid samples show high resistance against surface hydrophilicity due to aging. The introduction of micro and nanofillers in the polymer matrix may be the probable reason which improved the
barrier against surface aging [54,55].

Moreover, the percentage change in peak intensities for Si (CH$_3$) at (840–790 cm$^{-1}$) is found maximum for PS1 which shows 15.5% of the un-aged specimen and thus pointing maximum drop of around 84.5% in PDMS side-chain bonds. In the case of PS3, this change is 31.5% showing a drop of 68.5% of the fresh sample followed by PS2 with 28.4%. Relatively lower loss in the side-chain of PS2 may be due to ATH filler which decomposes into alumina and water at high temperatures. Hydration water content evaporates and removes surface heat while remaining alumina provides better shielding thus improve the properties of the polymer [50].

While considering the change in absorbance peak height at Si-O-Si (1130–1000 cm$^{-1}$), the important siloxane backbone of silicone exhibited by PS1 is 17.5%, while PS3 retained 38.3% with high resistance to Si-O-Si bond breakage followed by PS2 (29.3%). A high degree of depolymerization due to the synergetic impact of aging may be the reason for such loss in pure silicone specimens [56]. Similarly, in C-H symmetric stretch of CH$_3$ at (2963–2960 cm$^{-1}$) samples, PS1 and NS again perceived maximum loss in absorbance peaks as seen in Table 3, which indicate high stretching of C-H thus high variations in the polymer chain. Such a type of stretching results in shrinkage of the polymer chain and harden the sample’s surface. Variations in the hardness of the specimens are discussed in detail in the next section. Similar observations are also reported earlier in [57]. Identical patterns of drops are obtained for Si–CH$_3$ at wavenumbers 1280–1255 cm$^{-1}$ and remaining groups which are presented clearly in Table 3.

Variations in absorbance peak height of these functional groups for negative polarity voltage are found relatively lower compared to positive DC as shown in Fig. 6. The combined impact of UV, humidity, high temperature, and contamination along with high voltage DC is responsible for variation in the chemical structure of PDMS. Conclusion elucidates that hybrid composites performed exclusively better in the aging test which is attributed to the lower concentration of SiO$_2$ nanoparticles thus increased surface area in polymer base matrix due to the strong interaction of polymer base and additive particles [58]. It is obvious from FTIR results that the anti-aging properties of coating materials can be improved up to a great extent by adding micro/nanofillers to matrix RTV silicone.

### Table 3

| Functional group | Wavenumber (cm$^{-1}$) | PS1 | PS2 | PS3 | NS1 | NS2 | NS3 |
|------------------|-----------------------|-----|-----|-----|-----|-----|-----|
| Si-O of Si(CH$_3$)$_2$ | (840–790) | 15.50% | 28.40% | 31.50% | 22.10% | 31.40% | 72.80% |
| Si-O-Si Asymmetric stretch | (1130–1000) | 17.50% | 29.30% | 38.30% | 29.70% | 36.00% | 70.60% |
| Si-CH$_3$ symmetric bending | (1280–1255) | 9.60% | 34.00% | 60.50% | 27.50% | 62.30% | 62.90% |
| CH$_3$ in Si-CH$_3$ | (1440–1410) | 10.90% | 69.00% | 90.20% | 56.60% | 84.10% | 71.20% |
| C-H Symmetric CH$_3$ stretch | (2963–2960) | 20.60% | 56.60% | 68.70% | 47.50% | 75.20% | 61.40% |
| O-H water content | (3700–3200) | 385.20% | 171.30% | 139.30% | 221.10% | 119.10% | 110.50% |

4.5. Hardness and weight analysis

Sunlight initiates degradation in a polymer when exposed to an open environment due to the breakdown of polymeric C-C bond creating small fragments and thus result in brittleness of the polymeric material [59]. The hardness of all blends is measured to investigate the variations in mechanical properties due to the collegial effects of UV, thermal and other stresses under positive and negative DC voltage. Measurements are recorded after every summer/winter cycle from the start of the aging. Average value of hardness shore-A is selected after at least three measurements and standard deviations of the data with error bars are represented. Fig. 7 reveals the alterations in hardness for coating specimens as a function of aging time. It can be observed that initial hardness values of micro and micro-nano-filled hybrid are higher than unfilled SiR which means that the hardness of composites is augmented with the incorporation of micro/nano additives to the base matrix of silicone. The figure also shows a trend of loss and recovery of the hardness of all
samples as aging cycles continue.

It can be seen in the hardness-aging cycles graph (Fig. 7), that all specimens except NS3 show a significant rise of hardness after the 5th cycle of weathering while a gradual rise and drop is observed after the 10th cycle. The maximum percentage increase in hardness is 9.3% obtained for PS1 followed by PS2 and PS3 showing 6.6% and 5.2%, respectively which are stressed under positive DC. Minimum increment in hardness is obtained for NS3 as 2.65 HA with aging and is followed by NS2 with 3.16 HA. To have a comparison of polarity, we see that the hardness values of samples PS1 and NS1 (before-after) aging are (50.33–55) HA, and (50.33–53.87) HA, respectively. However, after calculating the net percentage increase in hardness, it is found that all blends perceived a moderate increment as compared to recovery.

The reason behind this increasing-decreasing trend may be due to striving competition amongst cross-linking and macromolecule decomposition in the elastomer [53]. An increment in hardness may be attributable to increased cross-linking in polymer chain under the impact of UV radiation, thermal stresses, and variation of pH on the surface which results in the initiation of such mechanism [60]. Likewise, the formation of small fragments in polymers due to C-C bond breakdown may also result in the hardening of these materials. In both polarities, the filled RTV-SiR performed better showing relatively only a small change in hardness. This is probably due to the nanofillers in polymer structure which increased the shielding effect against UV and other stresses [47].

To investigate the degree of mechanical degradation due to long-term multi-stress aging, it is also critical to measure the change in the mass (gain or loss) of the polymers. Percent change in weight calculated from the measurements after the conclusion of aging is presented in Fig. 8. Measurements are repeated 4 times for every sample and the average value is selected to record the readings with maximum possible accuracy. Fig. 8 shows that aging has caused loss of weight in all samples though to a varying degree.

The weight/aging time graph explicates that a maximum percent weight loss of 7.7% occurred in unfilled SiR (PS1) stressed under positive DC followed by NS1 (3.78%) with negative DC. Extra polymer chain scission, loss of cycles after diffusion from the bulk, and chemical decomposition may have caused maximum variation in pure RTV-SiR [53]. Unfilled specimen PS1 under positive voltage observed the highest weight loss after aging. The plausible reason may be the weak shielding against UV radiation, high temperature and consistent unidirectional DC stress, which gradually deteriorates its properties due to the resultant chain scission, hydrolysis reaction, loss of LMWs and decomposition of the polymer chain. After the pure SiR blends, the percentage measured loss is 1.44% and 1.2% for PS2 and NS2, respectively. The sample, PS3 shows the maximum sustainability against weight loss and performed extremely well. Similarly, the weight loss recorded is 1.13% only for NS3 which is fairly higher as compared to PS3 but still falls among the lowest amid other composites. The enhanced weather shielding as a result of the addition of nanofillers to the base matrix may have offered such a high resistance against the impact of multi-stresses, especially UV.

The outcome of hardness and weight loss measurements indicate that as compared to negative polarity, positive polarity has a more dominant effect similar to other measurements reported in earlier sections. The possible reason for the polarity effect may be the high surface conductivity resulted from more ions migration from pollutants consequently letting high leakage current flow and cause further heating. This temperature rise may boost the aforesaid chemical reactions thus leading to a high degree of aging under positive polarity [50].

4.6. Stress-strain curve

To examine the detailed response of mechanical properties of composites in long-term multi-stress environments under DC, the deformation behavior of RTV SiR and other composites was also studied. For this purpose, the stress-strain curve and tensile strength of all the specimens were measured both prior-to-aging and after the completion of all aging cycles. Using dumbbell-shaped samples, stress-strain curves were obtained through elongation in a specific environment with applied force to get the ultimate tensile strength for each blend. The stress-strain curve so obtained for each sample in un-aged and aged conditions is presented.

![Fig. 8. Percentage weight loss after complete aging.](image-url)
in Fig. 9.

Here, an apparent decline in the stress-strain curve can be seen after aging for all the samples representing an ultimate loss in elasticity regardless of fillers and their concentration. This may again be attributed to the initiation of crosslinking in polymers and hence their eventual rise in brittleness. Results are almost in line with the hardness analysis performed in the previous section.

Fig. 10 explicates the relative change in ultimate tensile strength for each composite before and after aging. It can be observed that all samples experienced some loss of tensile strength due to aging but to a varying degree. The sample PS3 under positive DC exhibited the lowest loss of 16.08% followed by PS2 with a loss of 20.78%. The sample PS1 as expected experienced the highest loss of 42.18%. The lower drop in ultimate rupture strength of composites samples may be attributed to the appropriate dispersion of additives, which improve interactions of nanoparticles with base matrix and plenty of silanol groups of nano-

Fig. 9. Stress-Strain relation curve for non-aged and aged samples.

Fig. 10. Percentage loss in tensile strength with aging.
silica [61]. Additionally, intact hydrogen bonding between silanol groups present in silica and -OH groups of PDMS matrix may also improve the ultimate strength of the base matrix [62].

In the case of composites stressed under negative DC, similar behavior of all investigated samples was observed except that the percentage drop of mechanical strength was comparatively less. Overall findings revealed that at the end of the complete aging period the hybrid composite blends PS3 and NS3 maintained better sustainability against multi-stress aging followed by micro-filled specimens. Relatively speaking, unfilled samples experienced maximum loss in ultimate strength.

5. Discussion

Changes in surface hydrophobicity are considered an initial insignia of commencement of degradation in aging studies of insulators. From Fig. 2, it can be seen that all specimens show a loss as well as recovery of hydrophobicity through class measurement by STRI criterion. Each test is performed in a repeated manner for the measurement of consistency in recorded data. Results of some essential findings of diagnostic techniques are summarized in Table 4. Impact of various stresses and their corresponding overall effects on other essential parameters like hydrophobicity and leakage current are discussed in detail.

Unfilled samples (PS1 and NS1), after complete aging, depicted hydrophobicity loss following to class HC6 (most hydrophilic) among all investigated composites. While PS2/NS2 (ATH filled) and PS3/NS3 (ATH-Silica filled) samples show improved performance with the good final hydrophobic class as shown in Table 4. Increased concentration of oxygen on silicone composites’ surface due to formation of hydrophilic silica-like layer and silanol group on PDMS may lead to the loss of hydrophobicity. Severe oxidation as a result of the reaction of oxygen content with PDMS may deform hydrophobic CH3 to CH2 thus creating hydroxyl with peroxides [63]. Also, leakage current is highly linked with the hydrophobicity of surface, the more the hydrophobic surface the lower the leakage current to flow. Leakage current data from Fig. 3 and hydrophobicity classification in Fig. 2, shows that filled composites PS2 and PS3 offered high resistance to these changes in comparison to unfilled silicone. This could be due to enhanced filler resistance and shielding effect imparted to the base PDMS matrix which affects LMWs mobility thus slowing down the recovery but reduce the impact of oxidation reaction initiated by synergetic impact of stresses. Sample PS3 and NS3 perform exclusively well with better recovery. This may be due to the ease of LMWs molecule transportation due to nano silica filler which improves the mobility in addition to better shielding against other deterioration factors.

FTIR results further clarify hydrophobicity loss and leakage current rise as shown in Figs. 5 and 6. The increased OH contents (wavenumber following in the range: 3200–3700 cm−1) in samples due to oxidation and other reactions as a result of multi-stresses lead to the hydrophobicity loss which in turn results in more leakage current on the surface. For instance, pure silicone sample (PS1) experienced high hydrophobicity loss, maximum rise in OH hydroxyl content and leakage current as shown in Table 4. On the other hand, the hybrid filled sample (PS3) has comparatively lower values. An almost similar trend is observed for samples energized under negative DC voltage. These results depict that multi-stresses initiate various chemical reactions which degrade the material and cause hydrophobicity loss and increase leakage current. Higher leakage current in turn, results in different electrical phenomena like dry band arcing, corona discharges and rise in surface temperature which accelerates the chemical reactions in combinations with other stresses and ultimately causes the decline of desired properties of insulating material.

Other reasons for hydrophobicity loss and leakage current are surface degradation by various reactions (depolymerization, chain scission, decomposition, hydrolysis, loss of LMWs, etc.) due to synergetic multi-factors stresses. As shown in Fig. 4, SEM images of pure silicon show more surface deterioration as compared with other composites. While minimum topological alterations are observed in PS3 and NS3 which portrays enhanced enactment to these changes. Although rough surface helps in improving the mobility of LMWs to some extent and thereby improves recovery but on the other side, it attracts more pollution/moisture forming a conductive layer after wetting thus enhance the leakage current. Although the incorporation of filler may have some negative effects on the recovery process of hydrophobicity but its effect on improved shielding against other stresses enhances the overall performance of the composite.

Similarly, investigation of mechanical characterization like hardness, weight loss, tensile strength and elongation-at-break showed enhanced anti-aging characteristics and high enactment of ATH/ATH-silica filled composites (PS2 and PS3) against aging under positive voltage. In polymeric coatings investigation, these properties are of key importance. An increase in hardness and brittleness of polymers may result due to breakage of C-C bond due to exposure to UV radiations resulting in small fragments formation. These degradation cause surface roughness which can be verified from SEM micrographs in Fig. 4. Furthermore, thermal and electrical degradation in the presence of other stresses causes weight loss of polymer due to depolymerization, loss of silicone cyclics and other reactions. Results of hardness and weight elucidate the highest loss in weight and increased hardness for pure silicone while (PS3 and NS3) showed improved enactment. Outcomes of tensile strength are almost in conformance with hardness measurements. Variations in other corresponding bonds can be validated from FTIR results at their specific wavenumbers from Table 3. Concluding the above key findings, it can be summarized that hybrid filled composites (PS3 and NS3) evince better overall performance among all investigated specimens. These are followed by PS2 and NS2.

In summary, results of SEM, FTIR, leakage current, hydrophobicity and mechanical tests clearly indicate that incorporation of ATH filler into base PDMS matrix improves the performance of silicone to a greater extent while the incorporation of ATH-silica combined fillers dominantly enhanced the shielding effect and offers a moderate impact on the mobility of silicone LMWs cyclics.

6. Conclusion

The present investigation became a priority research area in Pakistan since most of the 500 kV and 200 kV AC transmission lines use ceramic insulators and anti-pollution design has always remained a challenge.

| Samples | PS1 | PS2 | PS3 | NS1 | NS2 | NS3 |
|---------|-----|-----|-----|-----|-----|-----|
| HC Class | 6   | 3   | 2   | 6   | 2   | 2   |
| Leakage Current (µA) | 14.74 | 8.96 | 5.85 | 9.91 | 6.88 | 4.84 |
| OH Content % rise (3200–3700 cm−1) | 385.2 | 171.3 | 193.3 | 231.1 | 199.1 | 118.5 |
| Hardness (% Increase) | 9.27 | 5.41 | 4.42 | 7.03 | 4.55 | 4.39 |
| % Weight Loss | 7.7 | 1.44 | 0.63 | 3.78 | 1.2 | 1.13 |
| % Loss in Tensile Strength (MPa) | 42.18 | 20.78 | 16.08 | 33.69 | 17.69 | 13.98 |
The use of polymeric coatings for such existing lines appears to be the only workable solution as other means of maintenance are very costly and un-implementable due to other logistic difficulties. The recent introduction of HVDC using polymeric insulators is the first such experiment in Pakistan. With the goal to find the best possible coating material for our existing HVAC lines, the present study was launched. Having better knowledge of the coating performance, the electric utility may opt to extend the use of such coatings on the extended portion of transmission lines located in polluted regions. As DC pollution is relatively more severe, we applied DC stress on top of existing local environmental stresses, to simulate the most adverse conditions for the comparison of various possible coatings and their relative merit.

The present experimental study is devoted to understanding the behavior of RTV-SiR coating as well as several other hybrid samples involving the incorporation of micro and nano-size fillers in the base SiR under the dual stress of positive and negative polarities. SiR blends with micro and micro-nano ATH & silica show amended resistance to the hydrophobicity loss. Measurements of surface current clarified that NS3 offered better leakage current suppression followed by the counterpart PSP3 under positive DC. Stress-strain curves and ultimate strength assessments show high intactness for NS3 and PS3. Furthermore, FTIR results clarified that unfilled SiR experienced loss in main peaks at wavenumber 1008 cm⁻¹, which demonstrates the occurrence of de-polymerization. Similarly, many minimum changes are observed in the surface topography of hybrid samples.

Overall results elucidate that performance of RTV- SiR can be enhanced by reinforcing hybrid fillers (micro ATH & nano-silica) into the base PDMS matrix. Moreover, it is observed that the degradation phenomenon in the aging analysis is comparatively grievous in the case of positive DC.

CRediT authorship contribution statement

Israr Ullah: Conceptualization, Methodology, Formal analysis, Writing – original draft, Software. Mohammad Akbar: Formal analysis, Supervision. Haris Ahmad Khan: Formal analysis, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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