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Investigation of epoxy composites for outdoor insulation under accelerated ultraviolet exposure

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Abstract

Epoxy and its composites have shown remarkable properties to be used for outdoor high voltage insulation applications. However, outdoor environment that contains UV-A radiations deteriorates the organic structure of epoxy-based insulators. To analyze the surface degradation and chemical changes in structure due to UV radiations, neat epoxy (NEPOX), 15 wt.% epoxy microcomposite (EMC 15) and 5 wt.% epoxy nanocomposite (ENC 5) were prepared and subjected to a specially fabricated UV chamber under accelerated conditions. The samples were analyzed with visual inspection, STRI hydrophobicity classification, and Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR) spectroscopy. The analyses confirmed the pattern of gradual degradation of surface structure due to the appearance of ester and carbonyl groups due to oxidation of hydrocarbon chains. FTIR peaks of C=O stretching at ∼1725.52 increased by 157.44%, 75.86% and 33.33% for NEPOX, EMC 15 and ENC, respectively. This shows that ENC-5 expressed better resistance against surface oxidation as compared to EMC 15 and NEPOX. Addition of fillers enhanced the chain intactness and nano filler performed even better due to greater surface area and surface energy.

1. Introduction

Polymeric insulators have been studied extensively in the recent past due to their promising properties over the conventional ceramic insulators [1, 2]. Despite all the advantages over ceramics, polymeric insulators suffer from degradation due to their organic nature [3–6]. Ultraviolet light can cause gradual decomposition of chemical structure of the organic Polymeric insulators. Such degradations can result in discoloration, loss of hydrophobicity, surface chalking and cracking, chemical changes, loss in breakdown strength and mechanical weaknesses [7, 8]. According to the parameters defined in CIGRE WG D1.14, UV weathering is one of the twelve physical parameters in the evaluation of polymeric materials for usage in outdoor insulation [9].

Various polymers are being employed for high voltage insulation applications. Most significant studies include the effect of UV weathering on the polymeric insulators. Lin et al studied UV-A irradiated liquid silicone rubber for 1000 h and deduced surface oxidation, chain scission and loss in hydrophobicity [10]. In another study, Lee et al concluded that ultraviolet radiations caused abrupt increase in esters and carbonyl groups on the surface of epoxy/glass fiber composites. This surface degradation was attributed to continuous thermodynamic reactions which caused reorientation of oxygen groups [11]. Amin and co-authors found degradation in absorption peaks and hydrophobicity of High Temperature Vulcanized (HTV) SiR when subjected to ultraviolet...
radiations [12]. Wang and team concluded that ultraviolet treatment made the hydrophobicity of ATH (Al₂O₃) filled silicone rubber worse and also increased the rigidity of the samples [13]. Rao et al made an investigation on the leakage current and pollution flashover voltage of silicone rubber under UV light. An increase in leakage current and decrease in flashover voltage was seen with increase in the aging period [14]. Hedir et al studied physicochemical properties of UV treated Cross Linked Polyethylene (XLPE) concluding increase in crosslinking of chains and photo oxidation which was confirmed with the presence of carbonyl and DBI groups [15]. Israr et al investigated silicone rubber and epoxy composites under ultraviolet radiations elucidating reduction in absorption peaks of hydrocarbon groups and loss of hydrophobicity. Asymmetric C–H stretching of ~2966.18 in neat epoxy was 22% of virgin sample. The reduction in peaks was lowest in nanocomposites. Similarly, in silicone rubber Si–O–Si chain expressed 75%, 58% and 60% reduction in peak for neat, microcomposite and nanocomposite, respectively. These reductions in absorption peaks were referred to depolymerization phenomenon [16]. SiR/ATH micro composites were studied under UV radiations and 70.2% decrease in volume resistivity and 367.7% increase in dielectric loss factor was measured. Hydrophobicity class of the samples also moved from HC-1 to HC-2 [8]. Long term UV and moister aging of silicone rubber membranes resulted in loss of hydrophobicity, surface oxidation and increase in dielectric loss [17]. Another study evaluated the degree of aging of UV treated epoxy through polarization/depolarization current method and elucidated an increase in currents with increase in aging time. This increase indicated the degree of aging of the samples [18]. Hedir et al reported accelerated UV aging of XLPE for high and medium voltage insulation. UV aging degraded surface and dielectric properties of XLPE due to severe oxidation and chain scission [19]. Ning and authors studied space charge characteristics of ultraviolet radiations treated MgO filled epoxy nanocomposites concluding that 3 wt% ~ 5 wt% exhibited best resistance against aging. The volume resistivity and dielectric loss factor were also less affected for this concentration [20]. Zhai and team prepared 2wt.%, 6wt. % and 10wt.% epoxy/aluminum composites and studied their ultraviolet aging. They concluded that 6wt.% composite expressed best performance with 57.5% and 65% reduction in bending strength and yellowing value than pristine samples, respectively [21]. Gorur et al studied tracking, erosion and flashover performance of epoxy/silica insulators for usage on 25 kV outdoor system. It was seen that insulators had sufficient resistance against tracking and erosion and did not flashover even at 20% higher voltage than the nominal value [22]. In another study, epoxy/silica nano-dielectrics were evaluated for high voltage outdoor insulation and it was concluded that nanosilica filled dielectrics showed improved breakdown strength and dielectric properties [23]. It is clear from the literature that UV light is the primary reason for degradation of polymeric insulators. Hence epoxy based insulators must be tested against ultraviolet radiations before usage in outdoor environment. To date, work on ultraviolet aging of epoxy/silica composites under both dry and wet conditions is not reported.

This work is conducted to investigate the impact of accelerated UV-A radiations on structure and physical properties of epoxy/silica composites under dry conditions. The analysis techniques of visual inspection, hydrophobicity classification, and SEM and FTIR spectroscopy were used for this purpose.

2. Material procurement and composite preparation

2.1. Procurement of materials

The materials used to prepare the composites were epoxy resin, nanosilica (12 nm) and microsilica (5 μm). Epoxy resin was procured from a German company General Electric while nanosilica was bought from Degussa Chemicals, China and microsilica was procured from Wuhan Newreach Chemicals, China.

2.2. Sample preparation

Different epoxy composite with varying filler concentration neat epoxy (NEPOX) without filler, epoxy with 5% Nanosilica (ENC 5) and epoxy with 15% Microsilica (EMC 15) were prepared. First of all, dispersion of silane (3-Glycidoxypropyltrimethoxysilane) pre-treated fillers were dispersed into the ethanol. Functionalized fillers and Bisphenol-A were mixed at 3600 rpm in a shear mixer for 15 min. Sonication was performed on the prepared solution for 30 min in an ultrasonic bath to ensure fine mixing. After that the solution mixture was placed in a
Memmert oven at 100 °C for 48 h and consequently all the ethanol evaporated. Then the solution was mixed with hardener at 7200 rpm for 20 min and was placed in vacuum at 27 mmHg for degassing and debubbling. After pouring into molds, the prepared slurry was placed at room temperature for 1 day. The samples were placed in oven at 90 °C for 3 h in the post curing process. The diameter and thickness of each sample was 80 mm and 3 mm, respectively. Neat epoxy was used as a reference to compare the performance of the micro and nanocomposites. The names and sample code of all samples are given in table 1.

2.3. Experimental aging set up
An ultraviolet aging chamber having dimensions of 0.5 m × 1 m × 1 m was fabricated of acrylic sheet. The schematic diagram of fabricated chamber in the figure 1 shows how eight UV-A lamps (354 nm) were mounted on interior of the four vertical edges. Two aluminum plates were placed to hang the samples in the chamber. The intensity of UV lights was fixed at 5 mW cm⁻² in the chamber measured with ML-8511 sensor. The experimental setup was built mainly according to ISO-4892 standard. Other standards such as ASTM D2865M-06 and IEC 62217ed2.0 were also used as guides. The samples were aged for 1000 h in four cycles with each cycle consisting of 250 h.

2.4. Analysis techniques
The following techniques were utilized to inspect the behavior of insulators before and after UV-A aging.

2.4.1. Visual inspection
Visual inspection was done to analyze the surface deformation and color changings of the samples during the aging process. Samples were visualized in the day light and their photographs were taken with High Definition (HD) camera.

2.4.2. Hydrophobicity classification
Swedish Transmission Research Institute (STRI) classification was utilized to study the hydrophobic behavior of the samples. Each sample was sprayed with distilled water for 20 s continuously after each cycle and their hydrophobicity was evaluated within 10 s by comparing their images with STRI standard image guide (HC 1 to HC 6). The images were taken in an inclined position at an approximate angle of 45°. The hydrophobicity was evaluated between HC 1 and HC 6, where HC 1 class exhibits the most hydrophobic and HC 6 the least hydrophobic character.

2.4.3. Fourier transform infrared (FTIR) spectroscopy
FTIR analysis was carried out by using Agilent Technologies’ Cary 630 FTIR spectrometer to detect the oxidation of hydrocarbon chain and structural changes in the samples due to ultraviolet aging. After every cycle, spectrographs in absorption form were obtained from 500 cm⁻¹ to 4250 cm⁻¹ range.

2.4.4. Scanning electron microscopy (SEM)
Scanning Electron Microscopy (SEM) analysis was performed to analyze the surface degradation due to UV radiations of the samples. SEM micrographs were taken after every cycle using SEM VEGA III, TESCAN.
3. Results and discussion

3.1. Visual inspection

Visual inspection of the samples was done after every cycle during the aging period. Neat epoxy and its composites did not express any cracks or major degradation during the aging time, however, changes in color and surface roughness were observed. The transparent NEPOX became opaque and more yellowish. These color changes are evident in the visual inspection images provided:

- **Figure 2.** Visual inspection images of NEPOX (a) unaged (b) 1000 h aged.
- **Figure 3.** Visual inspection images of EMC 15 (a) unaged (b) 1000 h aged.
- **Figure 4.** Visual inspection images of ENC 5 (a) unaged (b) 1000 h aged.
changes are attributed to the formation of ether as well as carbonyl group (C=O) due to UV-A radiations. It is well documented that ultraviolet radiations oxidize the surface of polymeric insulators [11, 24, 25]. Kumar et al concluded that epoxy absorbed UV radiations due to which aromatic groups in bisphenol-A glycidyl ether were oxidized [26]. Similarly, EMC 15 also appeared yellowish, but the intensity of yellow color was less as compared to the neat epoxy. This may be due to the incorporation of silica filler that enhanced the intactness of chain and induced resistance against oxidation. ENC 5 showed least degradation of hydrocarbon chain as it showed a little change in color as compared to NEPOX and EMC 15 due to addition of nanosilica that provides greater surface intactness and high surface energy. These increased surface characteristics hinder surficial changes due to which
greater stability against environmental stresses can be observed. Comparison of virgin and 1000 h aged NEPOX, EMC 15 and ENC 5 is given in figures 2–4, respectively.

3.2. Hydrophobicity classification

The hydrophobic behavior of the samples was studied after every cycle and it was observed that surface hydrophobicity was greatly affected by the ultraviolet radiations. Hydrophobicity of the samples was evaluated by standard STRI classification guide in which HC 1 is the highest hydrophobicity class and HC 6 is the lowest hydrophobicity class, as illustrated in figure 5. All the samples exhibited their highest hydrophobicity class prior to aging and loss in hydrophobicity was observed after the application of UV radiations. However, recovery was also seen in case of ENC 5 and EMC 15. Figures 6–10 represent the hydrophobicity images of unaged, 250 h aged, 500 h aged, 750 h aged and 1000 h aged samples, respectively. UV treatment induced non-uniform surface
energy on the surface of the samples which distorted the circular shape of the droplets. NEPOX showed HC-2 class before aging which turned to HC-3 after 500 h of aging and remained the same till 1000 h. This loss in hydrophobicity is due to surface oxidation of epoxy as described in visual inspection as well. It is also attributed to scission of chains and formation of polar groups on the surface due to ultraviolet radiations \[11, 27\].

\[\text{Figure 11. Variation trends in hydrophobicity with aging period.}\]

\[\text{Figure 12. SEM images of NEPOX (a) unaged (b) 1000 h aged.}\]

\[\text{Figure 13. SEM images of EMC 15 (a) unaged (b) 1000 h aged.}\]
silica filled epoxy (EMC 15) showed better resistance to change in hydrophobicity compared to neat epoxy. EMC 15 initially showed HC-2 class of hydrophobicity and moved to HC-1 after 250 h. It retained its initial class till 750 h and ended at HC-3 after 1000 h. The better hydrophobicity trend of EMC 15 confirms the role of silica filler in improving the surface behavior of epoxy [28]. ENC 5 exhibited better hydrophobic character than both NEPOX and EMC 15. ENC 5 started at HC-1 and touched HC-2 after 500 h. However, it recovered to HC-1 in the next cycle and ended at HC-1 after 1000 h. The addition of fillers improved the stability in terms of hydrophobicity of composites against UV radiations, due to the better intactness of silanol groups of silica with hydrophobic methyl group of epoxy [29]. ENC 5 showed even better results as the surface intactness and surface energy of EMC 15 may be less as compared to ENC 5 due to relatively large size of micro filler than nano filler. Overall, all the samples exhibited acceptable hydrophobic behavior with ENC 5 expressing the highest hydrophobicity class of HC-1. Hydrophobicity variation trend of all the samples can be seen in figure 11.

3.3. Scanning electron microscopy (SEM)
Scanning Electron Microscopy (SEM) was used to study the surficial changes in NEPOX, EMC 15 and ENC 5 due to aging. Images of unaged and 1000 h aged samples were taken at a distance of 5 μm. Figures 12–14 show images of unaged and 1000 h aged NEPOX, EMC 15 and ENC 5, respectively. It was seen from the images that all the samples exhibited surface degradation due to UV treatment. Greater degradation of surface, holes and loss of material was observed in case of NEPOX due to excessive oxidation caused by UV radiations. This loss of material from the surface of NEPOX may have caused roughness and irregular morphology which may have contributed to loss of hydrophobicity. EMC 15 expressed better surface intactness than neat epoxy due to the presence of silanol group (Si–OH) that provides better surface intactness. ENC 5 in addition to the abundance of silanol group has greater surface area and chain intactness provided by nanosilica [30–32]. This chain intactness and high surface area resist in distortion of surface morphology making ENC 5 less susceptible to surface degradation. SEM findings regarding all prepared materials also validate its hydrophobicity and visual inspection results.

Figure 14 SEM images of ENC 5 (a) unaged (b) 1000 h aged.

| Group name                           | Wave number (cm\(^{-1}\)) |
|--------------------------------------|---------------------------|
| Asymmetrical Carbon-Hydrogen Stretching of \(-\text{CH}_3\) | ~2967.18                  |
| Asymmetrical Carbon-Hydrogen Stretch of \(-\text{CH}_2\) group | ~2927.61                  |
| Symmetrical Carbon-Hydrogen Stretch of \(-\text{CH}_2\) group | ~2871.68                  |
| Stretching vibration in C==O         | ~1725.52                  |
| Carbon-Carbon Stretching vibration in aromatic | ~1606.52                  |
| Carbon-Carbon Stretching aromatic ring | ~1508.16                  |
| Asymmetrical deformation of Ethyl     | ~1296.01                  |
| Symmetrical Aromatic C–O–C Stretch   | ~1033.72–1100.55          |
| –Carbon Hydrogen aromatic deformation | ~829.29                   |
| –\text{CH}_2 Rocking                 | ~772.15                   |
3.4. Fourier transform infrared (FTIR) spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was used to study the changes in chemical structure of NEPOX, EMC 15 and ENC 5. The analysis was made after each cycle in absorption form. All the samples expressed variation in absorption peaks due to ultraviolet radiations. Prominent groups in epoxy and their respective wavenumbers are given in table 2. Important hydrocarbon groups, such as asymmetric C–H stretching of methyl group at $\sim 2966.18 \text{ cm}^{-1}$, asymmetric stretching of ethyl group at $\sim 2927.61 \text{ cm}^{-1}$ and symmetric stretching of ethyl group at $\sim 2871.68 \text{ cm}^{-1}$, expressed degradation in all the samples. Samples were subjected to severe oxidation due to UV radiation which is evident from increase in absorption peaks of Carbon Oxygen stretching at $\sim 1725.52 \text{ cm}^{-1}$ and Carbon Carbon stretching at $\sim 1606.52 \text{ cm}^{-1}$. In NEPOX, asymmetric...
stretching of \(-\text{CH}_3\) group, asymmetric stretching of \(-\text{CH}_2\) group, and symmetric stretching of \(-\text{CH}_2\) group expressed 41.30%, 27.77% and 32.25% decrement in absorption peaks after 1000 h of UV treatment. However, absorption values of carbonyl group and carbon double bond were increased by 157.44% and 37.25%, respectively. Degradation of hydrocarbons and increase of C=O and C=C is due to oxidation which caused scission of hydrocarbon groups resulting in formation of carbonyl groups [33, 34]. Spectrographs of NEPOX, EMC 15 and ENC 5 after each cycle are given in figures 15–17, respectively. Like NEPOX, EMC 15 and ENC 5 also exhibited degradation in absorption peaks after aging but the degree of their decrement was less as compared to neat epoxy. EMC 15 expressed 30.76%, 28.94% and 31.42% degradation in asymmetric \(-\text{CH}_3\) stretch, asymmetric \text{CH}_2\ stretch and symmetric \text{CH}_2\ stretch after 1000 h, respectively. The absorption of C=O and C=C increased by 75.86% and 56% in case of EMC 15. ENC 5 showed 26.67%, 25% and 29.16% decrease in
the absorption peaks of asymmetric $-\text{CH}_3$ stretching, asymmetric $-\text{CH}_2$ stretching and symmetric $-\text{CH}_2$ stretching, respectively.

ENC 5 was also affected by oxidation but it was less than both NEPOX and EMC 15. The absorption peaks of $\text{C}=\text{O}$ stretching and $\text{C}=\text{C}$ stretching at $\sim1725.52$ cm$^{-1}$ and $\sim1606.52$ cm$^{-1}$ were increased by 33.33% and 5.12%, respectively. The better performance of ENC 5 than both NEPOX and EMC 15 is discernable which is attributed to the incorporation of nano silica which provided greater chain intactness and high surface energy to nanocomposites [35]. EMC 15 expressed better performance than neat epoxy but lower than ENC 5 which is due to relatively greater size of micro silica which provided smaller surface area as compare to the nano silica. It can be deduced from the above discussion that inclusion of nano silica increased the stability of the functional

Figure 17. FTIR imaged of ENC 5 (a) virgin (b) 250 h aged (c) 500 h aged (d) 750 h Aged (e) 1000 h aged.
groups due to greater intactness of the chain. The absorbance values of NEPOX, EMC 15 and ENC 5 after each cycle are given in tables 3–5, respectively.

4. Conclusion

The performance evaluation of epoxy/silica composites for outdoor insulation under intense UV-A radiation was made with visual inspection, hydrophobicity classification, SEM and FTIR. It was observed that all the samples underwent severe oxidation under accelerated conditions. Due to the oxidation, color of the samples changed to yellow. This surface change also reduced the hydrophobicity of the samples. ENC 5 exhibited better resistance to hydrophobicity loss followed by EMC 15 and NEPOX. Holes and loss of material were observed along with filler exposure in SEM findings. Greater surface degradation was seen in NEPOX as compared to EMC 15 and ENC 5. FTIR results indicated reduction in peaks of hydrophobic methyl group with increase in peaks of C=O and C=C. The increase in carbonyl group compliments findings of hydrophobicity and SEM.

### Table 3. Peak’s intensities in NEPOX.

| Group name                                                | Wave number (cm⁻¹) | Absorbance         |
|------------------------------------------------------------|--------------------|--------------------|
| Asymmetrical Carbon-Hydrogen Stretching of –CH₃           | ~2966.18           | Virgin             |
| Asymmetrical Carbon-Hydrogen Stretch of –CH₂ group        | ~2927.61           | 0.046 0.036 0.037 0.032 0.027 |
| Symmetrical Carbon-Hydrogen Stretch of –CH₂ group         | ~2871.68           | 0.031 0.032 0.036 0.033 0.026 |
| Stretching vibration in C=O                               | ~1725.52           | 0.047 0.063 0.067 0.112 0.121 |
| Carbon-Carbon Stretching vibration in aromatic            | ~1606.52           | 0.051 0.053 0.037 0.060 0.070 |
| Carbon-Carbon Stretching aromatic ring                    | ~1508.16           | 0.084 0.075 0.079 0.076 0.065 |
| Asymmetrical deformation of Ethyl                         | ~1296.01           | 0.046 0.049 0.046 0.061 0.051 |
| Symmetrical Aromatic C–O–C Stretch                        | ~1033.72–1100.55   | 0.201 0.119 0.157 0.203 0.184 |
| –Carbon Hydrogen aromatic deformation                      | ~829.29            | 0.140 0.117 0.131 0.218 0.190 |
| –CH₂ Rocking                                               | ~772.15            | 0.183 0.112 0.140 0.145 0.135 |

### Table 4. Peak’s intensities in EMC 15.

| Group name                                                | Wave number (cm⁻¹) | Absorbance         |
|------------------------------------------------------------|--------------------|--------------------|
| Asymmetrical Carbon-Hydrogen Stretching of –CH₃           | ~2966.18           | Virgin             |
| Asymmetrical Carbon-Hydrogen Stretch of –CH₂ group        | ~2927.61           | 0.039 0.028 0.029 0.026 0.027 |
| Symmetrical Carbon-Hydrogen Stretch of –CH₂ group         | ~2871.68           | 0.035 0.026 0.023 0.017 0.024 |
| Stretching vibration in C=O                               | ~1725.52           | 0.029 0.049 0.052 0.056 0.051 |
| Carbon-Carbon Stretching vibration in aromatic            | ~1606.52           | 0.025 0.037 0.044 0.033 0.039 |
| Carbon-Carbon Stretching aromatic ring                    | ~1508.16           | 0.108 0.059 0.062 0.061 0.060 |
| Asymmetrical deformation of Ethyl                         | ~1296.01           | 0.053 0.038 0.035 0.030 0.050 |
| Symmetrical Aromatic C–O–C Stretch                        | ~1033.72–1100.55   | 0.146 0.093 0.122 0.106 0.092 |
| –Carbon Hydrogen aromatic deformation                      | ~829.29            | 0.130 0.092 0.101 0.093 0.090 |
| –CH₂ Rocking                                               | ~772.15            | 0.108 0.088 0.108 0.081 0.79 |

### Table 5. Peak’s intensities in ENC 5.

| Group name                                                | Wave number (cm⁻¹) | Absorbance         |
|------------------------------------------------------------|--------------------|--------------------|
| Asymmetrical Carbon-Hydrogen Stretching of –CH₃           | ~2966.18           | Virgin             |
| Asymmetrical Carbon-Hydrogen Stretch of –CH₂ group        | ~2927.61           | 0.036 0.026 0.027 0.032 0.028 |
| Symmetrical Carbon-Hydrogen Stretch of –CH₂ group         | ~2871.68           | 0.024 0.024 0.021 0.019 0.017 |
| Stretching vibration in C=O                               | ~1725.52           | 0.036 0.043 0.048 0.040 0.048 |
| Carbon-Carbon Stretching vibration in aromatic            | ~1606.52           | 0.039 0.034 0.040 0.031 0.041 |
| Carbon-Carbon Stretching aromatic ring                    | ~1508.16           | 0.064 0.054 0.057 0.049 0.038 |
| Asymmetrical deformation of Ethyl                         | ~1296.01           | 0.035 0.035 0.032 0.035 0.024 |
| Symmetrical Aromatic C–O–C Stretch                        | ~1033.72–1100.55   | 0.154 0.083 0.111 0.125 0.084 |
| –Carbon Hydrogen aromatic deformation                      | ~829.29            | 0.108 0.084 0.092 0.084 0.062 |
| –CH₂ Rocking                                               | ~772.15            | 0.141 0.080 0.099 0.128 0.085 |
From the findings of all the techniques, it was concluded that ENC 5 expressed best resistance among other samples due to large surface area provided by nanosilica and polymer chain intactness which reduced the scission of hydrocarbon groups. This builds a strong UV shielding layer in nanocomposites. So, it can be deduced that nanocomposite can perform better in outdoor insulation.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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