Investigation of dielectric properties and methylene intactness under multiple environmental stresses for high voltage epoxy composites

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Abstract

Epoxy decays its dielectric characteristics and exhibits degradation of main hydrocarbon on exposure to multiple environmental stresses. Inorganic oxides-based epoxy composites have been performing well in many applications and short-term testing; therefore, evaluation of their dielectric and structural characteristics under extreme weathering conditions may also unleash enhancement in these characteristics. To explore dielectric properties and degradation of main hydrocarbon group, neat epoxy and silica-based epoxy microcomposite (15% micro-silica loading) and nanocomposites (5% nano-silica loading) have been prepared and subjected to acid rain, heat, ultra-violet radiation, salt fog, and humidity in a chamber that was specially fabricated in view of service conditions. Interesting results were obtained before and after aging. Enhanced intactness of methylene group was observed in nanocomposite followed by micro composite. Similarly, for epoxy nanocomposites not only higher dielectric constant, lower energy dissipation and conductivity was recorded before application of stresses, but also nanocomposite showed superior sustainability in these properties after aging. In all analyses, microcomposites performed better than neat epoxy but in conductivity the results of both samples were found comparable.

1. Introduction

Tremendous advancements in the field of composites are the reasons for consideration of replacement of conventional polymers with polymer composites in various fields such as energy storage, dielectric, electrical insulation and high voltage apparatus [1–7]. Prominent polymers for dielectric, high voltage outdoor and electrical cable insulation applications are Silicone rubber, EPDM, PVC, EPR, XLPE and epoxide etc [8–10]. Among various mentioned polymers, epoxy is highly preferred over other polymers where hard, fire resistive polymer in a one-unit-construction is required as good alternative to ceramics [11]. However, epoxy not only lags behind ceramics in few characteristics but its characteristics e.g. structural properties, dielectric and mechanical characteristics etc also degrade during service [9, 12, 13]. On the other hand, composites’ formation has been proved to improve several characteristics of epoxy and can lead to ultimate replacement of its competitive options i.e. rest of the polymers and ceramics [14–16]. All polymers in general but especially epoxy in particular needs detailed investigation of characteristics before it can be confidently recommended for high performance applications. Most important problems in epoxy are its short life span and loss of dielectric properties and degradation of main hydrocarbon due to multiple environmental stresses such as (ultra-violet-A) UV-A radiation, water, heat etc [17–19].
Keeping in view the insulation and dielectric applications, the micro and nano fillers of insulating nature should be selected. For this purpose, there are several classes of fillers but inorganic oxides of silica, titania, and zinc oxide are favorable for obtaining required enhancements in nano/micro polymer dielectrics [9, 20]. In literature, many encouraging results are presented which strengthen the effect of enhancing overall characteristics of epoxy micro and nanocomposites through incorporation of micro and nano inorganic oxides. For example, Zakya Rubab et al discovered that preparing epoxy composites through addition of submicron titania particles enhances glass transition temperature, young modulus, tensile strength and thermal oxidative temperature [21]. Qian Xie et al studied electrical characteristics of silica and titania wire based epoxy composites in comparison to neat epoxy. DC volume resistivity, Thermally Stimulated Discharge Current (TSDC) and permittivity were found few folds higher in case of epoxy-titania composites [22]. Epoxy acrylic-alumina composites were studied by Jiepeng Bian et al for dielectric properties and it was found that irregular alumina-based composites exhibited improved dielectric strength as compared to spherical shaped alumina and unfilled samples [23]. Similarly, Kishan Ahn et al used titanium oxide (TiO$_2$) for coating of copper nano-wires that were subsequently used for formation of epoxy composites. At the same weight friction with uncoated wires, the coated wires showed significantly higher values of thermal conductivity [24]. P Pareetha et al explored alumina based epoxy composites for AC breakdown analysis and discovered that at 1% loading of alumina showed highest dielectric strength [25]. However, long-term aging performance under multiple stresses was suggested. After performing the long-term electro-thermal aging of the samples by the authors in [26], the samples at 5% loading of alumina exhibited 9 times higher shelf life in comparison to unfilled epoxy. This showed the importance of long-term service analyses rather than relying on initial characterization. Among wide range of fillers, silica is most cost effective, easily available and performed best in basic characteristics of composites such as thermal [27], mechanical [28], electrical [29] etc properties [30]. Besides this encouraging literature for long

Table 1. Prepared sample concentrations.

| Sample                     | Code  |
|----------------------------|-------|
| Neat epoxy                 | NE    |
| Epoxy with 5% nano-silica | ENC 5 |
| Epoxy with 15% micro-silica| EMC 15|

Table 2. Weathering conditions for aging.

| Applied Stress          | Summer | Winter |
|-------------------------|--------|--------|
| Test Voltage (kV)       | 2.5    | 2.5    |
| Length of cycle (days)  | 11     | 17     |
| Temperature (°C)        | 47.2   | 35.3   |
| UV-A (hours)            | 10     | 8      |
| Acid rain (4.5 pH)      | 6 times| 2 times|
| Salt fog (5000 μS cm$^{-1}$) | 0 times| 4 times|

Figure 1. Prepared samples (a) neat epoxy (b) epoxy nanocomposite (c) epoxy microcomposite.
term dielectric and structural performance’s investigation of epoxy silica, we also found both micro and nano-silica as great contributors in life enhancement, weathering performance of silicone rubber [4, 31, 32], EPDM [33, 34] and HNBR [35]. The available literature and our previous findings have been helpful in studying the effect of environmental stresses on dielectric properties as well as main hydrocarbon intactness.

Figure 2. (a) Schematic diagram of (b) Photographic image of the aging chamber.
Keeping in view the above-mentioned motivations, this work is dedicated for investigation of dielectric properties of epoxy micro and nanocomposites before and after degradation. Furthermore, main hydrocarbon group of epoxy is analyzed continuously to better understand the degradation mechanism at structure level.

Figure 3. Absorbance of symmetric CH$_3$ stretch for (a) Neat epoxy (b) Epoxy Nanocomposite (c) Epoxy microcomposite at different aging intervals.
Figure 4. Percentage of symmetric CH$_3$ stretch for (a) Neat epoxy (b) 5% epoxy (c) 15% epoxy composites at different aging intervals.
2. Experimental

2.1. Materials
Resin Diglycidyl ether of bisphenol-A (DGEBA) (Eposchon®), equivalent to 189 ± 5 g/eq. of epoxy, and the hardener cycloaliphatic amine (EPH 555®) equivalent to 86 g/eq of amine hydrogen were used. Both Resin and hardener were obtained from Justus Kimia Raya, Indonesia. Nano-silica (AEROSIL® 200) with average diameter of 12 nm and specific surface area of 200 m² g⁻¹ was obtained from Degussa, USA. Micro-silica was used with average particle size of 5 μm, obtained from Wuhan NewReach chemicals China. Silane (DOW CORNING® Z-6040) was used for surface functionalization of fillers, while ethanol used as solvent was procured from Sigma-Aldrich® USA.

2.2. Fabrication of samples
Nano-and micro-silica were first functionalized and dispersed into ethanol. For surface functionalization of the fillers, silane(3-glycidoxypropyltrimethoxysilane) was employed as surfactant. Solution was kept in ultrasonic bath for 90 min. After that, epoxy resin was added into the solution and mixed in a high shear mixer at 3600 rpm for 15 min before putting the mixture in ultrasonic bath for few hours again. Then, after a thorough mixing, the solution was kept in vacuum oven to evaporate ethanol. Thereafter, hardener was added and kept in a shear mixer at 7200 rpm for 120 min. The solution was put in an ultrasonic bath to ensure degassing and then transferred into molds for keeping at room temperature for 24 h. Concentrations of prepared samples are given in table 1, whereas prepared samples are shown in figure 1.

2.3. Aging setup
Electrical Power Research Institute (EPRI) [36] and IEC-61109 standards [37] were used to construct the aging chamber with dimensions 1 m × 2 m × 1 m. Acrylic sheets were used to fabricate the chamber walls, while floor of the chamber was made of stainless steel. Experiment was carried out under different environmental stresses representative of summer and winter weather. Multiple environmental stresses such as salt fog, heat, humidity,

| Cycle | NE Status | EMC Status | ENC Status |
|-------|------------|------------|------------|
| 0     | Virgin     | Virgin     | Virgin     |
| 1     | Degradation| Stability  | Stability  |
| 2     | Stability  | Stability  | Stability  |
| 3     | Stability  | Stability  | Degradation|
| 4     | Recovery   | Stability  | Degradation|
| 5     | Degradation| Degradation| Degradation|
| 6     | Degradation| Degradation| Degradation|
| 7     | Recovery   | Stability  | Degradation|
| 8     | Degradation| Degradation| Recovery   |
| 9     | Recovery   | Recovery   | Recovery   |
| 10    | Stability  | Recovery   | Degradation|
| 11    | Recovery   | Degradation| Degradation|
| 12    | Degradation| Recovery   | Degradation|
| 13    | Degradation| Recovery   | Degradation|
| 14    | Degradation| Degradation| Stability  |
| 15    | Recovery   | Degradation| Recovery   |
| 16    | Degradation| Recovery   | Recovery   |
| 17    | Recovery   | Degradation| Recovery   |
| 18    | Recovery   | Recovery   | Degradation|
| 19    | Degradation| Recovery   | Degradation|
| 20    | Recovery   | Degradation| Stability  |
| 21    | Recovery   | Recovery   | Degradation|
| 22    | Recovery   | Stability  | Degradation|
| 23    | Degradation| Degradation| Degradation|
| 24    | Degradation| Degradation| Degradation|
| 25    | Degradation| Degradation| Degradation|
| 26    | Degradation| Degradation| Degradation|
| 27    | Degradation| Degradation| Degradation|
| 28    | Degradation| Degradation| Degradation|
acid rain, were established inside the chamber. For energizing the samples, 2.5 kV (RMS) voltage supply was provided through 220 V/2500 V step up transformer. Temperature was maintained at 47.2 °C for summer and 35.3 °C for winter cycles, whereas acid rain with pH 4.5 was applied 6 times during summer and 2 times during winter. Moreover, the samples were exposed to UV-A radiations for 10 h during summer and 8 h during winter. Salt fog of 5000 μS cm⁻¹ was applied 4 times during winters only. The length of the cycle was maintained as 11 days for summers and 17 days for winters. A total of weather cycles were applied. The experimental conditions are summarized in table 2 [38]. Schematic diagram and photos of aging setup are given in figure 2. The specified values of different stresses applied during summer and winter weathers are given in table 2.

2.4. Instruments and conditions

2.4.1. Backbone degradation studies

Fourier Transform Infrared Spectroscopy (FTIR) was performed by using the Bruker platinum ATR Model Alpha, Germany with a spectral range of 4000–500 cm⁻¹. The samples were directly placed on a diamond crystal. Absorption peak of interest was recorded after each weather cycle i.e. Intensity of asymmetric stretch of CH₃ group at ~2966 cm⁻¹ after each weather cycle.
2.4.2. Dielectric properties

For measurement of dielectric properties, samples were cut into the 2 mm diameter and pressed between electrodes of LCR meter of Wayne Kerr model 6500B with frequency range of 100 Hz to 5 MHz at room temperature.

3. Results and discussion

3.1. Backbone and degradation analysis

Main hydrocarbons support retention of properties for most of the polymers. Consequently, degradation of themain hydrocarbon due to oxidation and scission affects the properties of polymers [39]. In the case of the epoxy dielectrics, asymmetric stretch of CH$_3$ group at $\sim$2966 cm$^{-1}$ is of utmost importance. Therefore, absorbance analysis of epoxy dielectrics in NE, EMC and ENC was conducted after each consecutive summer and winter cycle. It was observed that unfilled epoxy showed highest degradation in comparison to both composites. Figure 3 shows trajectory of absorbance intensities for NE, EMC and ENC as aging cycles pass. In case of NE, 23% degradation was observed after 1st cycle. However, the degradation level was subsequently

![Figure 6. (a) Virgin epoxy microcomposite (b) aged epoxy microcomposite.](image-url)
retained till 7th cycle followed by temporary recovery at two points. By comparison, no loss was observed till 4th cycle but 20% degradation occurred after 4th and 5th cycle in both ENC and EMC. However, difference in retention of CH$_3$ was clearly recorded after 6th and 7th cycle. A 50% decrease in the retention was seen at the end of 7th cycle in the case of EMC. However, for ENC the decrease was 25% and 35% after 6th and 7th cycles, respectively. Thereafter, for NE (EMC) a sudden decrease of 62% (70%) was observed in 8th cycle but in case of ENC recovery was observed. After 9th cycle, recovery was observed for NE and EMC and degradation was 12% and 70% respectively. On the other hand for ENC 20% degradation was seen after 9th cycle. Degradation level was retained till 10th cycle for NE, while recovery was exhibited by EMC followed by 25% degradation. Similarly, ENC exhibited overall 50% degradation. At 11th cycle, temporary recovery was seen for NE but then losses of 28%, 34% and 56% were recorded after 12th, 13th and 14th cycles, respectively. For NE, recovery was observed after 15th cycle. For EMC, between 11th and 15th cycles a total 60% degradation was seen but recovery was observed a few times during these cycles. Similarly, for ENC degradation was 60% after 11th and 12th cycle that enhanced to 65% during 13th and 14th cycles. ENC showed recovery till 17th cycle and then continued to decrease till 23rd cycle. Degradation in CH$_3$ for ENC was 55%, 60% and 70% for 21st, 22nd and 23rd cycles, respectively. For NE and EMC random increasing and decreasing trends of degradation were recorded. At the end of aging experiment (28th cycle), overall, 67%, 60% and 40% degradations were recorded for NE, EMC and

Figure 7. (a) Virgin epoxy nanocomposite (b) aged epoxy nanocomposite.
ENC respectively percentage trajectory can be seen from figure 4. More random trends were present in case of NE and EMC. The reason for this super intactness of main hydrocarbon in epoxy nanocomposites, explored in next sections, is extraordinary affinity of nano-silica with main network that may have been caused by abundant silanol groups present on the surface of nano-silica. Micro-silica also possesses silanol groups but the results were relatively more enhanced in the nanocomposite due to the fact that nano-silica has almost 9.5 times more specific area than micro-silica. This increased several folds the interaction of nano-silica with main polymer network [33, 38, 40]. Figure 3(a) illustrates trend of absorbance intensities for NE, EMC and ENC over the complete aging period of 28 cycles. Percentage degradation after each cycle is given in table 3. From the table it is observed that degradation of polymer methylene group occurs after many cycles and also recovery was observed at many steps after degradation but at the end most stability was seen in case of epoxy nanocomposite. Absorbance decreased continuously and during last cycles a continuous decreasing slope is clearly visible in neat epoxy. FTIR spectra of virgin and aged samples are given in figures 5–7.

3.2. Dielectric properties of unaged and aged samples
For comparative analysis of NE, ENC and EMC, dielectric constant ($\varepsilon'$) and dielectric loss were calculated both before and after aging to evaluate the impact of silica as well as effect of multiple stresses. Dielectrics and insulators should not only possess these properties but also retain them after exposure to environmental stresses. Dielectric constant ($\varepsilon'$) is a measure of charge stored in a material when a range of frequencies is applied to the material. These properties are frequency dependant and should be evaluated for a range of frequencies. Generally, at lower frequencies, higher polarization of material takes place due to which high dielectric constant value is observed. However, with increasing frequency, already polarized material fails to arrange itself in

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**Figure 8.** (a) Dielectric constant of unaged composites (b) Dielectric constant of aged composites.
response to the fluctuating current and heat losses. On the other hand, dielectric loss is energy that is consumed for hopping of electrons with fluctuating current. At a low frequency, more energy is consumed to cause the hopping of electrons due to which high dielectric loss is observed. By comparison, at a high frequency, less energy gets consumed for hopping of electrons and consequently dielectric loss decreases. At high frequencies the displacement current is so high that its ratio of the real power loss to the reactive component is minimal and therefore the dissipation factor is also minimal.
3.2.1. Dielectric constant

Dielectric constant was analyzed before and after aging. Figure 8(a) shows dielectric constant graph of unaged samples. A pronounced effect of micro and nano-silica filler on dielectric constant of neat epoxy is clearly visible. The dielectric constant of neat epoxy was below 1.4 (approximately 1.2 on average) at almost all frequencies. Conversely, EMC showed an average of 1.3 in the frequency range with a more decreasing trend at low frequency but less decreasing trend at higher frequency. However, the dielectric constant of ENC was not only higher than that of EMC but also maintained it above 2.0 for all frequencies.

Figure 8(b) shows dielectric constant trends of aged samples under multiple environmental stresses. After applying aging conditions, decrease in dielectric constant of all the samples was observed due to degradation of grain boundaries that allowed separation of charges. The dielectric constant of NE decreased from average 1.2 to average 1.0. A slight degradation from 1.3 to 1.2 was observed in case of EMC. Similarly after degradation, ENC 5 showed degradation in dielectric constant but highest average dielectric constant of approximately 1.3. The reason for this enhanced characteristics is the higher dielectric constant of silica in comparison to neat epoxy as well as less filler-filler interaction in the case of ENC [41, 42]. Figure 9 shows one to one comparison of dielectric constant values of aged v/s unaged samples.

3.2.2. Dissipation factor

In general, as evident from literature, the dissipation factor should increase with the dielectric constant and the same behaviour was recorded for all samples. As shown in figure 10(a), the dielectric loss was higher at low frequencies than that at high frequencies, in accordance with the above established facts. It is evident from literature that material with high dielectric constant also exhibits high dissipation. It was observed that
dissipation also decreases, along with dielectric constant, after degradation. All the samples showed loss in range from 0.025 to 0.6. It is pertinent to note that despite highest dielectric constant ENC showed loss of 0.6 at low frequencies. The loss was further decreased to 0.2 at higher frequencies and after degradation value of

Figure 11. Comparison of dielectric loss of aged V/S un-aged (a) neat epoxy (b) epoxy nanocomposite (c) epoxy micro composite.
dissipation became 0.1. For EMC, in both cases before and after degradation, least dielectric loss was recorded at low frequencies but a moderate loss value of 0.1 for unaged and 0.05 for aged sample was seen at higher frequencies. The NE showed moderate behavior with the same decreasing trend and exhibited average dielectric loss of approximately 0.2 (0.1) at low frequencies and approximately 0.05 (0.025) at high frequencies for unaged (aged) samples. Overall, very low amount of dissipation was observed along with high dielectric constant values.

Figure 10(b) illustrates relationship between dielectric loss and frequency after aging, whereas comparison of aged v/s unaged composites can be seen in figure 11. As expected, a decreasing trend was also observed after applying aging conditions. For NE at start of low frequencies, an unexpected behavior was also observed. This has occurred due to machine error caused by the high impedance at low frequencies. When impedance of material is high, its phase angle is not measured accurately and results in this abnormal behavior. The slope of decrease was much less in EMC and ENC. This is due to the extraordinary dielectric and insulating nature of silica. In case of ENC more pronounced results may be due to the greater intactness more hydrogen bonding due to silanol groups and increased filler-polymer interaction resulting in enhanced stability [8, 43, 44].

3.2.3. Conductivity

Generally, due to the increase in frequency, placement of neighboring sites of cationsis disturbed resulting in increased conductivity ($\sigma_{AC}$). Although this general trend was observed for all samples, the response of each epoxy formulation was different. Low values of current were observed at low frequencies but at high frequencies due to disturbance in polarization high values of current were recorded. Conductivity is directly related to dielectric constant and heat loss, and its value also increases with increase in dielectric constant. The same trend of superior insulating and dielectric nature was recorded in all samples with negligible conductivity at low frequencies and maximum conductivity of only 2.5 $\mu$S cm$^{-1}$ for aged ENC. In case of unaged samples, NE, ENC and EMC showed almost comparable values at low frequencies but conductivity was highest for EMC. This

![Figure 12](image-url)
unusual trend may be due to greater disturbance of cations in EMC which was reduced in case of ENC due to small size of particles. Conductivity of all the samples linearly increased to approximately 2, 1.5 and 2 $\mu$S cm$^{-1}$ for unaged EMC, ENC and NE, respectively.

Figure 12 AC conductivities of aged v/s un-aged (a) Neat epoxy (b) epoxy nanocomposite (c) epoxy micro composite.

Figure 13. Comparison of AC Conductivity of aged v/s un-aged (a) Neat epoxy (b) epoxy nanocomposite (c) epoxy micro composite.
conductivity reached 2.5 $\mu$S cm$^{-1}$. Nevertheless, this was very satisfactory for a material with high dielectric constant. Conductivity of EMC was higher than NE. At high frequencies, aged EMC and NE showed conductivities of 1.5 $\mu$S cm$^{-1}$ and 1.0 $\mu$S cm$^{-1}$, respectively. This result is also in-line with main hydrocarbon intactness and dielectric properties discussed in sections 3.1 and 3.2 respectively.

One to one comparison of aged v/s unaged samples is illustrated in figure 13. It is pertinent to note that values of conductivity decreased after aging because movement and speed of electrons decreases due to structural degradation.

4. Conclusion

In order to investigate the dielectric characteristics and stability of main asymmetric CH$_3$ group of epoxy/silica nano and micromposites under multiple weathering stresses, neat epoxy and its silica based, nanocomposite (ENC) and microcomposite (EMC) were prepared. The prepared samples were subjected to accelerated multiple environmental stresses. At the end of experiment, neat epoxy and EMC showed 67% and 60% losses, respectively. While, ENC showed overall least degradation of 40% in asymmetric CH$_3$ stretch. Similarly, average dielectric constants of neat epoxy, EMC and ENC were approximately 1.29, 1.32 and 2.1; which reduced to 1.13, 1.23 and 1.27, respectively, after exposure to multiple environmental stresses. The dielectric loss values for EMC and ENC were 0.1 and 0.6 that decreased to 0.075 and 0.1, respectively, after aging. ENC not only showed highest dielectric constant at all frequencies but also exhibited least degradation with aging and increase in frequency. The same trend of superior dielectric nature was recorded in the case of epoxy nanocomposite after aging with negligible conductivity and maximum conductivity of only 2.0 $\mu$S cm$^{-1}$. After aging, these values increased and were recorded as 2.5 $\mu$S cm$^{-1}$ at higher frequency. In all respect, the epoxy nanocomposite showed highest stability, high dielectric constant with low heat losses and conductivity even after applying multiple environmental stresses. By overall performance the samples can be arranged in descending order i.e. ENC is better than EMC which in turn is better than NE.

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