Impact of shear mixing time of epoxy-silica nanocomposites on its electrical and mechanical properties

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Abstract

The shear mixing time was varied to study its influence on electrical and mechanical behaviour of epoxy-silica nanocomposites. Epoxy resin along with 1 wt.% of silica nanofillers were subjected to shear mixing at different time spans. Water droplet-initiated corona inception voltage (CIV) was observed to increase with the shear mixing time, under AC and DC voltages. A marginal increase in contact angle as a function of shear mixing time was noticed. The CIV due to water droplet and the contact angle has shown a direct correlation. The epoxy-silica nanocomposite sample fabricated with 7 h of shear mixing time has led to 46.68% increment in CIV under AC voltage and 24.81% increment in contact angle compared to base epoxy resin. The ultimate tensile strength (UTS) and the Young's modulus of nanocomposites have increased with shear mixing time and at 7 h of shear mixing time has shown increase in ultimate tensile strength and Young's modulus of 45.78% and 43.77%, respectively, as compared to base epoxy resin. The strain versus time plot obtained from the digital image correlation (DIC) studies has shown that maximum strain occurs at a time period before breakdown has increased with shear mixing time up to 7 h. The time at which the maximum strain occurs has increased to 91.49% before the breakdown of epoxy-silica nanocomposite sample which was prepared with 7 h of shear mixing time as compared to base epoxy resin.

Abbreviations

CIV Corona Inception Voltage
DIC Digital Image Correlation
SEM Scanning Electron Microscope
UHF Ultra-High Frequency
UTS Ultimate Tensile strength

1. Introduction

Epoxy resin is an indispensable material, which can be used as spacers in GIS and also an insulating material in power system equipment such as dry type transformers [1, 2]. Base epoxy resin is reinforced with different size, shapes and weight percentages of nanoparticles to enhance electrical, thermal and mechanical properties [3–5]. The high aspect ratio and specific surface area of filler particles helps in achieving desired properties such as high breakdown strength, low surface charge accumulation, low dielectric loss and high mechanical strength [6–9]. Imai et al have stated that the inclusion of SiO₂ particles reduces thermal expansion coefficient of epoxy nanocomposites [10]. Also, inclusion of the silica fillers improves the mechanical strength of the composites [11]. Selection of appropriate filler-concentration plays a crucial role in the preparation of composite material, since the low filler-concentration in the base matrix results in uneven dispersion and high filler-concentration.
leads to agglomerations [12]. Also, the efficient filler dispersion in the polymer matrix relies on the shear mixing rate and time during the sample preparation, for improving the properties of the material [13]. In literature, it is indicated that the shear mixing is an effective and scalable method of dispersing nanoparticles in epoxy, which can break the agglomerates of the nanoparticles apart. Pullicino et al have studied the effect of variation in shear mixing rate and time in epoxy nanocomposites and have indicated that the optimum combination of shear mixing time and rate results in better dispersion of nanoparticles [14]. They have varied different shear mixing rates but they have limited to 1 h and 2 h of shear mixing times. Hence, in the present study, the shear mixing time during the preparation of the nanocomposites is varied by keeping shear mixing rate as constant, in order to identify the suitable shear mixing time for enhancing the overall properties of epoxy nanocomposites.

Water droplet presence on the insulating material because of fog, rain and humid environmental conditions can initiate micro discharges that occur at the triplet junction of a water droplet, air and the insulator surface. This results in conductive paths on the surface of the insulating material leading to surface flashover. Sarathi et al have performed corona inception studies in water droplet presence on the insulating material, and have stated that UHF signals are radiated due to water droplet-initiated discharges [15]. Hence, it is necessary to identify the voltage at the initiation of corona phenomenon due to these accumulated water droplets on the surface of the insulators.

Recent studies have indicated that the superior in tensile properties in case of polymeric nanocomposites can be obtained by good matrix-filler interfacial adhesion and proper filler dispersion [16–19]. Also, the bonding at the matrix-filler interface plays an important role in effective load transfer and also on the strain distribution of the composite material [20, 21]. Digital image correlation (DIC) is an image-based technique which can be applied for strain field displacement calculation [22]. Hence, this technique is applied to tensile test, to study the influence of shear mixing time variation on mechanical behaviour of epoxy-silica nanocomposites.

Hence, to study the impact of shear mixing time on epoxy-silica nanocomposites, the following methodical studies were performed: (i) water droplet-initiated corona inception studies, (ii) Contact angle and (iii) Mechanical characterisation by Digital image correlation studies.

2. Experimental studies

2.1. Preparation of samples

The materials used in the preparation of the epoxy nanocomposite specimens are the Bisphenol-A epoxy resin (CY205) as base matrix, silica nanoparticles of 15 nm APS (supplied by Nanostructured & Amorphous Nano Express 2 (2021) 010031 A Sharma et al Table 1. Sample details.

| Sample code | Sample details |
|-------------|----------------|
| Pure        | Base epoxy resin |
| 2 h         | 1 wt.% epoxy-silica nanocomposite prepared with 2 h of shear mixing time. |
| 5 h         | 1 wt.% epoxy-silica nanocomposite prepared with 5 h of shear mixing time. |
| 7 h         | 1 wt.% epoxy-silica nanocomposite prepared with 7 h of shear mixing time. |
| 10 h        | 1 wt.% epoxy-silica nanocomposite prepared with 10 h of shear mixing time. |

Figure 1. Schematic diagram of sample preparation.
Materials, Inc. Houston, Texas, USA) and Tri-ethylene tetra-amine (TETA) hardener as curing agent. The nanocomposites were prepared by mixing various weight percentages (0, 1, 2, 3, 5 and 10 wt.%) of silica to find the optimum weight percentage. Then, epoxy nanocomposites with 1 wt.% of silica fillers with base epoxy at different shear mixing timings, varying at regular intervals of 2, 5, 7 and 10 h at constant speed of 2000 rpm and the sample codes and corresponding details were represented in table 1. Hence, one pure epoxy sample and 4 nanocomposite samples were selected for present study. Following procedure as shown in figure 1 has been adopted for sample preparation: the nano silica powder was heated at a temperature of 110 °C for 24 h to remove the traces of moisture in it. Thereafter that nano filler was dispersed in ethanol and was kept in sonic water bath for 45 min. Then, the above mixture was mixed with epoxy resin and sonicated at high frequency sonication process (20 kHz) for one hour. The mixture was then subjected to shear mixing of 2, 5, 7 and 10 h respectively for preparation of different samples. Thereafter, again the mixture was subjected to high frequency sonication process (20 kHz) for one hour. Then, this mixture was transferred to an oven to evaporate the excess ethanol. At the last stage, the hardener was mixed to the above mixture and mixed thoroughly. After degassing, the final mixture was poured in various moulds for different characterisations. The Scanning Electron Microscopic (SEM) images of the epoxy nanocomposite samples prepared with 2, 5, 7 and 10 h of shear mixing time were presented in figure 2. It is noticed that the agglomeration count is reducing with increment in shear mixing time up to 7 h and the agglomeration count is noticed to increase in 10 h sample compared to 7 h sample.

2.2. Contact angle measurement and water droplet-initiated discharges

The layout sketch to measure corona inception voltage (CIV) because of water droplet presence on the epoxy nanocomposite sample under AC and DC voltages is depicted in figure 3. The experimental setup consists of a...
function generator, high voltage Trek amplifier, the test electrode, UHF sensor and digital storage oscilloscope. The test electrodes consisted of 2 stainless steel electrodes, whose tips were cut at 45° (IEC 60112), and kept on the sample, separated by 10 mm distance. One electrode was connected to the high voltage source through a resistance of 10 MΩ, and the other one was connected to the ground. Deionized water of 20 μl volume was used as a droplet. A UHF sensor was placed near the test setup and connected to the high bandwidth digital storage oscilloscope (LeCroy Wavepro 7300 A, 3.5 GHz bandwidth, operated at 20GSa/s). Figure 4 shows the frequency response of the UHF sensor. In the present work, the contact angle was measured adopting sessile droplet method with volume of water droplet of 10 ul. The contact angle was determined using the equation (1) [23].

\[
\theta = 2 \tan^{-1} \left( \frac{2h}{d} \right)
\]

where, \(h\) is the height and \(d\) is the base length of water droplet.

2.3. Digital image correlation (DIC) technique
DIC technique is an effective tool in calculating strain field displacement. Its principle is to identify the geometrical changes on the surface patterns (speckled patterns as represented in figure 5) after experiencing strain, as compared to the patterns before strain. To investigate the effect of shear mixing time on epoxy-silica nanocomposites, the prepared samples are conventionally tested with universal Testing Machine at 2 mm min⁻¹ displacement rate, at room temperature. ASTM D-638 (Type IV) standard is maintained for samples with the dimensions 115 × 6 × 3 mm³ and 25 mm gauge length. For reliability and repeatability of results, test was conducted on 3 samples of same shear mixing time. The experimental steps were taken in the following manner. Initially, the specimen is coated with thin white paint and speckled with black dots. Under the application of tensile load, images at every instant of time were recorded continuously by a camera placed in front of the universal testing machine. The displacement obtained in the speckle pattern is calculated using, the VIC-2D software.
3. Results and discussion

3.1. Water droplet-initiated corona inception voltage

Figure 6 depicts the CIV values of epoxy-silica nanocomposite specimens with variation in nanofillers weight percentages, under AC, +DC and -DC voltages. Katada et al have investigated water droplet-initiated discharges on polymer insulating surface under different voltage profiles and have indicated that the tips of the water droplet under applied voltage have induced charges, which are subjected to tensile forces that are proportional to magnitude of applied voltage [24]. CIV due to water droplet sitting on the sample is low for the pure epoxy and significantly increases with inclusion of 1 wt.% nanofillers in to base matrix. Sarathi et al have performed the water droplet-initiated corona inception studies on epoxy-clay nanocomposites under positive and negative DC voltages and have noticed that there is an enhancement in the CIV value of nanocomposites when compared to base epoxy resin, up to certain filler wt% and further increment in filler wt.% had showed no further enhancement of CIV [25]. Similar trend is observed in the present study, with increment in filler wt.%, only a

![Figure 6. Water droplet initiated CIV under AC and DC voltages with nanofillers wt.% variation.](image1)

![Figure 7. Water droplet initiated CIV under AC and DC voltages with variation in shear mixing time.](image2)
marginal variation in CIV is noticed in case of both AC and DC voltages. Hence, depending on the CIV values under AC, +DC and -DC voltages, epoxy nanocomposite with 1 wt.% of silica fillers has been selected for further analysis.

Figure 7 depicts the water droplet-initiated CIV values of 1 wt.% epoxy-silica nanocomposites with variation in shear mixing times, under different voltage profiles. The CIV values tend to increase as the shear mixing time increases and almost becomes constant. The interfacial bonding between nanoparticles and polymer is a key factor in enhancing the properties of polymer nanocomposites, which can be achieved by uniform dispersion of nanoparticles in polymer matrix [13]. Hence, better dispersion of nanofillers in the base epoxy matrix results in the improvement of the CIV values compared to base epoxy resin. Increment in the shear mixing time up to a certain time, has ensured the better dispersion of nanofillers in the epoxy matrix. Hence, with increment in the shear mixing time, better nanofiller dispersion is achieved and it led to the increment in the water droplet initiated CIV values. But, after certain extent of shear mixing time (above 7 h in the present study), no significant improvement in the CIV values is noticed. Lack of adhesion between the nanoparticles and polymer can possibly form strongly bonded nanoparticle-aggregates during the nanocomposite preparation, which results in an early failure at the interface of the nanocomposite or can lower the properties of polymer nanocomposite compared to that of base polymer [13]. Thus, in the present study, the CIV results have reflected that the 7 h of shear mixing time is optimum. This trend is same for both the AC and DC voltages. However, the CIV is higher under -DC than +DC and AC voltages. The CIV under AC voltage is lower as the rate of voltage change is high compared to DC [26]. The high energy electrons accessible for discharge phenomenon near the electrode, in high field region are more in number under +DC voltage than -DC [27]. Hence, the CIV values under +DC voltage are lesser than that of CIV under -DC voltage. The increase in CIV can also be attributed to the increase in hydrophobicity of the nanocomposite as observed in the contact angle analysis. As the contact angle is more, the water droplet spread out will be lower for the given volume and hence the CIV because of water droplet will be lower.

3.2. Contact angle
The variation of contact angle of the test specimens with respect to the shear mixing time is represented in figure 8. The contact angle with 1% nano filler at 2 h shear mixing time jumps 17.3% from the pure sample and thereafter 6.8% at 5 h shear mixing time. It can be inferred that the contact angle remains almost constant after five hours of shear mixing time and reduced slightly in case of 10 h mixing time. The wetting behaviour of the surface has been related to the surface roughness and the surface energy of the material [28]. Since the pure epoxy resin is hydrophilic in nature, the contact angle is notice to be less. When the silica nanoparticles are reinforced into epoxy matrix, significant improvement in contact angle is noticed. With increment in shear mixing time, the contact angle has improved. It is because of the better dispersion of the nanofillers in to the base matrix. Proper dispersion of the nanofillers have aided for the improvement of the hydrophobicity of the composite.

3.3. Mechanical characterisation by digital image correlation studies
The tensile tests were performed three times per each sample and the three stress-strain characteristics of Pure epoxy and 7 h samples are represented in figure 9 as Set-1, Set-2 and Set-3. The average and standard deviation of the tensile parameters calculated from three set of readings of each sample are tabulated in table 2. Out of three set of values per each sample, the stress-strain characteristics with extreme set of values (Set-1 and Set-3) are
Figure 9. Tensile stress-strain curves of (a) Pure epoxy and (b) 7 h samples.

Figure 10. Tensile stress-strain curve for epoxy-silica nanocomposites at different shear mixing time.

Table 2. Average value and standard deviation of tensile properties of epoxy-silica nanocomposites.

| Shear Mixing Time | Load at maximum tensile stress (N) | Young's Modulus, E (MPa) | Ultimate tensile Strength (MPa) | Static toughness (kJ m⁻³) |
|-------------------|------------------------------------|--------------------------|--------------------------------|--------------------------|
| Pure epoxy        | 753.93 ± 78.52                     | 1602.52 ± 119.29         | 41.31 ± 4.31                   | 663.47 ± 83.25           |
| 2 h               | 1208.69 ± 50.46                    | 1737.48 ± 74.43          | 67.28 ± 2.79                   | 1521.87 ± 105.28         |
| 5 h               | 1150.76 ± 58.17                    | 1814.66 ± 76.99          | 63.93 ± 3.23                   | 1265.46 ± 70.15          |
| 7 h               | 1044.51 ± 103.63                   | 2500.95 ± 231.86         | 57.60 ± 3.71                   | 769.13 ± 98.75           |
| 10 h              | 649.94 ± 62.77                     | 2176.17 ± 180.28        | 38.06 ± 3.68                   | 355.35 ± 98.42           |

Table 3. Tensile properties of epoxy-silica nanocomposites.

| Shear Mixing Time | Load at maximum tensile stress (N) | Young's Modulus, E (MPa) | Ultimate tensile Strength (MPa) | Static toughness (kJ m⁻³) |
|-------------------|------------------------------------|--------------------------|--------------------------------|--------------------------|
| Pure epoxy        | 747.46                             | 1653.86                  | 40.96                           | 649.42                   |
| 2 h               | 1217.38                            | 1758.86                  | 67.63                           | 1572.39                  |
| 5 h               | 1164.83                            | 1825.46                  | 64.71                           | 1295.49                  |
| 7 h               | 1074.73                            | 2377.71                  | 59.71                           | 825.78                   |
| 10 h              | 634.36                             | 2159.51                  | 37.15                           | 308.34                   |
Enhancement in tensile properties such as Young's modulus at the maximum load point which reflects the intrinsic brittle nature of the nanocomposites. In the present study, the reduction in tensile parameters of nanocomposites with above 7 h of shear mixing time is noticed. This could be due to efficient load transfer between matrix and filler in 7 h sample. However, at 10 h of shear mixing time has resulted in lower static toughness compared to that of base epoxy. The result obtained can be attributed to the fact that the filler dispersion and interfacial adhesion plays a vital role in the mechanical characterization of a material [29].

From table 3, Young’s modulus was found to increase with the time of shear mixing. Also, the ultimate tensile strength and the load at for breakpoint of the nanocomposites is higher than the pure epoxy sample. However, the ultimate tensile strength and the corresponding load decreases slightly below the pure epoxy at 10 h of shear mixing time. The static toughness values of the test specimens were represented in table 3.

Toughness indicates the ability of the test specimen to absorb energy by undergoing plastic deformation without fracturing, under applied tensile stress. The toughness value is dependent on the tensile strength and strain of the specimens. Addition of nanofillers to base epoxy matrix has successfully improved the static toughness of the nanocomposites, up to 7 h sample compared to base epoxy sample. This could be due to efficient load transfer between matrix and filler up to 7 h sample. However, at 10 h of shear mixing time has resulted in lower static toughness compared to that of base epoxy resin. The result obtained can be attributed to the fact that the filler dispersion and interfacial adhesion plays a vital role in the mechanical characterization of a material [29].

After the addition of the filler to the pure epoxy and shear mixed for 2 h, the nano particles start dispersing and tend to make stronger bonds with the base epoxy matrix. As the time of the shear mixing is increased it is observed that the Young’s modulus of nanocomposites improved up to 7 h of shear mixing time. This could be due to efficient load transfer between matrix and filler in 7 h sample. The interfacial bonding between nanofillers and polymer is a key factor in enhancing the properties of polymer nanocomposites, which can be achieved by uniform dispersion of nanoparticles in polymer matrix [13]. Hence, better dispersion of nanofillers in the base epoxy matrix results in the improvement of the material properties compared to base epoxy resin. Increment in the shear mixing time up to a certain time, has ensured the better dispersion of nanofillers in the epoxy matrix and it led to the increment in the tensile properties. But, after certain extent of shear mixing time (above 7 h in the present study), no significant improvement in the material properties have been noticed. Lack of adhesion between the nanoparticles and polymer can possibly form strongly bonded nanoparticle-aggregates during the nanocomposite preparation, which results in an early failure at the interface of the nanocomposite or can lower the properties of polymer nanocomposite compared to that of base polymer [13]. It is noticed from figure 2 that the agglomeration count is reducing with increment in shear mixing time up to 7 h and the agglomeration count is noticed to increase in 10 h sample compared to 7 h sample. Pullicino et al have studied the effect of variation in shear mixing rate and time in epoxy nanocomposites and have indicated that the reduced tensile strength in epoxy nanocomposites is due to the larger sizes of agglomerates which act as stress raisers and result in premature failure [14]. Kim et al have indicated that the tensile strength and the ductility show inverse relationship [30]. The highest shear mixing time in the present study i.e., 10 h of shear mixing time have reflected least tensile strength compared to other nanocomposites, providing the highest ductility. Hence, in the present study, the reduction in tensile parameters of nanocomposites with above 7 h of shear mixing time could be due to the presence of higher agglomerates, which might possibly lead to poor interfacial bonding between epoxy matrix and nanoparticles.

Einstein, Kerner and Nielsen models are the well-known mathematical models for characterizing mechanical behaviour of nanocomposites [31]. These theories require the calculation of the volume fractions of the particles and the nanofillers volume fraction was determined using rule of mixtures as shown in equation (2).

\[
V_p = \frac{\phi_m}{\phi_m + (1 - \phi_m) \frac{\rho_p}{\rho_m}}
\]  

(2)

Where \(V_p\) represent volume fraction and \(\phi_m\) is the mass fraction, \(\rho_p\) and \(\rho_m\) are the nanofillers density and matrix density respectively.

As per Einstein theory, the Young’s modulus of composite materials having a dispersed filler in a base matrix at low concentration of spherical particles, having a good matrix-filler adhesion can be defined by the equation (3) [32, 33].

\[
E_c = E_m[1 + 2.5V_p]
\]  

(3)

where \(E_c\) and \(E_m\) are Young’s moduli of the composite and the matrix material.

In the Kerner’s model, the particles are considered to be properly bonded to suspending medium, and are considered as spheres in the mean. Hence, Young’s modulus of the composite material that consists of spherical particles is given by the equation (4) [34, 35].
where \( \nu_m \) represents matrix Poisson ratio.

The Nielsen model is mainly based on the Einstein’s coefficient and a function containing the maximum volumetric packing fraction of the filler phase \([36]\). The expression for Young’s modulus of composite materials is given by the equation (5)

\[
E_c^{\text{Nielsen}} = E_m \left[ \frac{1 + \frac{V_p}{1 - V_p} \left( 1 - V_{\text{p max}} \right)}{1 + \Psi V_{\text{p max}}} \right] 
\]

where \( \Psi \) is a factor which is based on the maximum packing fraction \( V_{\text{p max}} \) of the fillers and is given by equation (6).

\[
\Psi = 1 + V_p \left( 1 - \frac{V_{\text{p max}}}{V_{\text{p max}}} \right)
\]

The coefficients \( A_1 \) and \( B_1 \) are defined for spherical particle by:

\[
A_1 = \frac{7 - 5V_m}{8 - 10V_m} \quad \text{and} \quad B_1 = \frac{E_p}{E_m} \left( \frac{1}{E_p + A_1} \right)
\]

The ultimate tensile stress \( (\sigma_c) \) and volume fraction \( (V_f) \) of nanocomposites can be related by considering the poor adhesion of nanoparticles with base matrix can be calculated as shown in equation (8) \([37]\).

\[
\sigma_c = \sigma_m (1 - aV_f^b)
\]

where \( \sigma_c \) and \( \sigma_m \) are the ultimate tensile stress of the nanocomposites and matrix respectively. \( a \& b \) are constants based on the shape and arrangement of the nanofillers. The values of \( a \) and \( b \) are equal to 1.21 and 0.66 when there is no adhesion between the nanofillers and the matrix \([38]\).

Table 4 represents the theoretical values of Young’s modulus calculated based on experimental results and theoretical models. The Young’s modulus calculated from theoretical models, tends to increase with the increment in shear mixing time and is within acceptable range till the 5 h of shear mixing time after which it is giving a high deviation. Since, the deviation between experimental values and theoretical values of Young’s modulus is very less up to 5 h of shear mixing time, it is observed that the theoretical models have successfully estimated the Young’s modulus of the epoxy nanocomposites. Again, with shear mixing time greater than 7 h, the deviation is noticed to be high. From the SEM images of the epoxy nanocomposite samples, it is noticed that the agglomeration count is reducing with increment in shear mixing time up to 7 h and the agglomeration count is noticed to increase in 10 h sample compared to 7 h sample. Hence, in the present study, the reduction in tensile parameters of nanocomposites prepared with above 7 h of shear mixing time, could be due to the higher agglomerates, which might possibly lead to poor interfacial bonding between epoxy matrix and nanoparticles.

**Table 4. Young’s Modulus as per the various models and experimental values.**

| Model            | Young’s Modulus for 1 wt% of SiO₂ (GPa) | Sample | Young’s Modulus Calculated Experimentally (GPa) |
|------------------|----------------------------------------|--------|-----------------------------------------------|
| Pure             | 1.65                                   |        |                                               |
| Einstein Model   | 1.67                                   | 2 h    | 1.76                                          |
| Kerner’s Model   | 1.73                                   | 5 h    | 1.82                                          |
| Nielsen Model    | 1.67                                   | 7 h    | 2.37                                          |
|                  |                                        | 10 h   | 2.16                                          |

**Table 5. Ultimate Tensile stress as per theoretical model and experimental values.**

| Ultimate tensile Strength (MPa) calculated as per the theoretical model | Sample | Ultimate tensile Strength (MPa) of nanocomposites | Percentage deviation |
|------------------------------------------------------------------------|--------|---------------------------------------------------|-----------------------|
| 39.68                                                                  | 2 h    | 67.63                                             | 70.43%                |
|                                                                        | 5 h    | 64.71                                             | 63.08%                |
|                                                                        | 7 h    | 59.71                                             | 50.48%                |
|                                                                        | 10 h   | 37.15                                             | 6.37%                 |

\[
E_c^{\text{Kerner}} = E_m \left[ 1 + \frac{V_p}{1 - V_p} \left( 1 - V_{\text{p max}} \right) \right] 
\]

**Table 4.** Young’s Modulus as per the various models and experimental values.

**Table 5.** Ultimate Tensile stress as per theoretical model and experimental values.

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Table 6. Strain distribution along the gauge length of the epoxy-silica nanocomposite samples obtained from DIC method at different instants of time during elongation.

| C   | At 0 Sec | At 10 Sec | At Break Point |
|-----|----------|-----------|----------------|
| Pure|          |           |                |
| 2 h |          |           |                |
| 5 h |          |           |                |

Nano Express 2(2021)010031 A Sharma et al
| C  | At 0 Sec | At 10 Sec | At Break Point |
|----|----------|-----------|----------------|
| 7 h| ![Image](image1.png) | ![Image](image2.png) | ![Image](image3.png) |
| 10 h| ![Image](image4.png) | ![Image](image5.png) | ![Image](image6.png) |
Also, the experimental values could have been deviated from the expected theoretical values due to this poor interfacial bonding between epoxy matrix and nanoparticles. Hence, according to Young's modulus calculation from theoretical models, it is considered that the nano particles would have dispersed perfectly in the range of 5 to 7 h of shear mixing.

Table 5 shows the calculated value of the ultimate tensile stress of the nanocomposite ($\sigma_c$) when calculated by keeping the values of $a$ and $b$ as 1.21 and 0.66 and when there is no matrix-filler adhesion. With 2, 5 and 7 h of shear mixing, the percentage deviation between theoretical and experimental value of ultimate tensile stress where there is no matrix-filler adhesion, is very high. Whereas, the theoretical value of ultimate tensile strength when there is minimum to no matrix-filler adhesion, is approximately equal to the experimental value of ultimate tensile strength of the nano composite prepared with 10 h of shear mixing time, justifying that the poor matrix-filler interfacial bond due to excessive shear mixing.

Table 6 depicts the strain distribution of the sample during the entire process of elongation at the starting point, at 10 s and at the breaking point of the test samples. The strain field distribution can be used in identifying the highly-stressed areas, just before the occurrence of fracture. Thus, the DIC method is advantageous than the normal tensile test, by representing the strain field corresponding to deformations [39, 40]. Also, the strain magnitude at any point on the surface of the specimen can be deciphered by the colour scheme for each sample and each instant of time. It is observed that the average strain on the sample increases with time and suddenly drops around the location of breakage. Also, the variation of $e_{yy}$ along with the time is depicted in the figure 11. It is observed that the elongation progresses linearly with time for all the samples. It is noticed that the nanocomposite specimen with 2 h of shear mixing time has higher strain magnitude and it is noticed that the strain magnitude at any time instant before breaking, reduces with increment in shear mixing time up to 7 h of shear mixing time. Even though the 5 h sample has reached breakpoint in less time, it is noticed from figure 10 that the 5 h sample has recorded higher tensile strength and lesser strain magnitude compared to 2 h sample. The 7 h sample has reflected lesser strain magnitude and is also able to withstand stress for more time. But, the 10 h of shear mixed sample recorded higher strain magnitude than 7 h samples and withstood only lesser time, indicating poor load transfer between matrix and filler interfaces.

4. Conclusion

The important conclusions accrued in the current study are as follows:

- Inclusion of 1 wt.% of silica in to epoxy matrix have shown a significant improvement in the water droplet-initiated CIV and further increment in weight percentage depicted only a marginal increment.
- The contact angle and CIV under both AC and DC voltages, tends to increase with increase in shear mixing time up to 7 h of mixing time.
• The 7 h sample has recorded 46.68% increment in CIV under AC voltage and 24.81% increment in contact angle compared to base epoxy resin.

• The Young’s modulus tends to increase with addition of silica fillers and tends to increase with increase in shear mixing time up to 7 h of mixing time and reduce after 10 h of mixing time. It is because of the effective filler dispersion in case of nanocomposite specimen with 7 h shear mixing time.

• The 7 h sample has shown 45.78% increment in ultimate tensile strength and 43.77% increment in Young’s modulus compared to base epoxy resin.

• The theoretical value of ultimate tensile strength when there is minimum to no matrix-filler adhesion is approximately equal to the experimental value of ultimate tensile strength of the nanocomposite prepared with 10 h of shear mixing time, justifying that the poor matrix-filler interfacial bond due to excessive shear mixing.

• The strain versus time plot obtained from the digital image correlation studies have reflected 91.49% increment in the time at which the maximum strain occurs before breakdown of 7 h sample compared to base epoxy resin.

Overall, it can be concluded that the optimum shear mixing time to disperse 1 wt.% nano-silica in epoxy matrix uniformly is found to be 7 h, which significantly improved electrical and mechanical behaviour of epoxy-silica nanocomposites.

Data availability statement

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

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