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Hygrothermal chamber aging effect on mechanical behavior and morphology of glass fiber-epoxy-nanoclay composites

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Keywords: nanoclay, glass fiber, epoxy, artificial aging, SEM micrographs

Abstract

This work examines the impact of artificial aging on tensile and flexural behavior of epoxy-nanoclay composites (ENCs) and glass fiber-epoxy-nanoclay composites (GFENCs) in the hygrothermal chamber. Epoxy-nanoclay composites made by a general-casting technique, and GFENCs are made by hand layup technique. The specimens are aged in the hygrothermal chamber for 180 days at 40°C with 60% RH. The results revealed that an increase in nanoclay and glass fiber weight percentage enhanced the mechanical behavior of GFENCs. The aging of the sample has a negative influence on the composite materials. But, the increase in nanoclay and glass fiber weight percentage has diminished the impact of aging on the mechanical behavior of composites. SEM micrographs revealed the reason for the failure and influence of aging conditions.

1. Introduction

1.1. Epoxy-nanoclay composites (ENCs)

Epoxy polymer resin has outstanding characteristics, such as extraordinary strength, limited shrinkage, & excellent heat and chemical resistance. Because of its unique properties, epoxy is used in many applications as coats, bonding agents, and matrix constituents for composites [1]. Cured epoxy systems have shown minute endurance to crack instigation and propagation & poor fracture toughness, in its applications [2]. Cured epoxy system exhibits brittle behavior (due to higher crosslink density), while testing. Also, cured epoxy systems show lower fracture toughness, which can be observed from figure 4(a), the pure epoxy displayed a completely smooth surface representing rapid crack propagation and poor fracture toughness. A recent approach is being proposed by adding additives in the polymer resin to improve the mechanical, thermal, and flame-retardant properties [3]. Throughout the last decades, ENCs have been investigated extensively. Due to their favorable properties, ENCs have drawn significant interest in the materials arena. This interest derives from the point that polymers filled with nanoclay can show drastic enhancements in mechanical and thermal properties [4]. There are, however, some disadvantages in the use of these fillers, such as the decline in strain to failure [5]. It is to be remembered that the ability to gain the most of the positive effect in ENCs is from the characteristics of nanoclay due to the proper and homogeneous dispersion in the polymer resin. An effective addition of a small amount of nanoclay, usually up to 5 wt%, could provide an efficient enhancement to the properties of traditional polymer composites [6, 7]. Further increasing the nanoclay content, the properties of composites will decrease owing to the increase in stiffness and agglomeration [8, 9].

1.2. Glass fiber epoxy-nanoclay composites (GFENCs)

There is increasing demand for the use of extraordinary performing and lightweight composites in various applications to replace conventional metals. Glass fiber-epoxy composites are commonly utilized in many applications such as vehicle bodies, wall insulation, bridges, and hulls of the ship among the different composites [10, 11]. Properties of glass fiber-epoxy composites need to be enhanced as it will allow the production of
lightweight structures for civilian applications. As many researchers have proven that adding nanoclay to epoxy resin displays dramatic improvements in the mechanical, thermal, chemical, tribology, fire retardancy, water absorption, toughness and corrosion resistance properties [12]. Also, nanoclay addition leads to an increase in the ‘cross-linking density’ of epoxy, giving rise to increased toughness and fiber-matrix interface toughness [13, 14]. Adding nanoclay guarantees a more significant load transfer to a load-bearing constituent, i.e., glass fiber, by improving the interfacial bonding and preventing the spread out of interfacial crack [7, 15].

1.3. Artificial aging in hygrothermal chamber

Environmental factors viz., temperature, humidity, varying loads, and their combinations can damage composite properties in specific applications [16]. Composite material quality is severely affected by climate exposure and external factors. Composite materials, when exposed to the natural environment, absorb moisture resulting in the reduction in matrix dominated properties viz., maintaining the fibers in the proper orientation, spacing and protecting them from abrasion and the environment and matrix transmits load from the matrix to the fibers through shear at the interface. The engrossed moisture has numerous adverse influences on the properties of the composite. A study on the aging of composites in water is, therefore, useful in improving product durability. Hygrothermal effects can be investigated in numerous methods, including artificial aging and real exposure tests. Real exposure tests take more time to execute. Artificial aging will reduce the time required to saturate the composite material with moisture [17]. Moisture absorption in polymer composites is a function of several variables such as temperature, fiber weight percentage, additives used and time of exposure.

The purpose of the current study is to fabricate and characterize two types of composites materials viz., epoxy-nanoclay composites with varying wt% of nanoclay and GFENCs with varying wt% of glass fiber and nanoclay. This work also represents the effect of artificial aging in the hygrothermal chamber on mechanical behavior viz., tensile and flexural strength, and morphology of fabricated composites.

2. Materials and experimental studies

2.1. Materials

Commercially available epoxy resin (L-12) and hardener (K-6) obtained from ’Atul Polymers.’ The weave E-glass fabric reel (360 GSM) obtained from 'Yuje Enterprises Bengaluru.' Besides, nanoclay (‘Surface modified contains 25–30 wt% trimethyl stearyl ammonium’) obtained from 'Sigma Aldrich.'

2.2. Composite specimen preparation

Two sets of composites are prepared viz., epoxy-nanoclay composites (ENCs) with varying wt% of nanoclay as shown in table 1 and glass fiber epoxy-nanoclay composites (GFENCs) with varying wt% of glass fiber and nanoclay as presented in table 2.

Figure 1 illustrates the detailed procedure of preparation of composite specimen. For ENC specimens, molds are prepared according to required dimensions. GFENCs are prepared by hand lay-up technique, followed with compression molding. Dimensions of the laminates mentioned in figure 1 as $300 \times 300 \times 3 \text{mm}^3$ (L X W X T). The volume is maintained constant with 1% bi-lateral tolerance. The manual compressing machine with stopper is used for compressing the laminates to maintain the uniform thickness of 3 mm. All the laminated composites are marinated with 3 mm thickness. Before compression molding the thickness of casted composites are with non-uniform thickness due to hand lay-up technique. So, it is necessary to use secondary process as compression molding to get uniform thickness. GFENC specimens are cut from the cured laminate according to

<table>
<thead>
<tr>
<th>Constituents (wt%) of ENCs.</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy resin</td>
<td>100</td>
<td>98</td>
<td>96</td>
</tr>
<tr>
<td>Nanoclay</td>
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<td>2</td>
<td>4</td>
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</table>

<table>
<thead>
<tr>
<th>Constituents (wt%) of GFENCs.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
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<td>50</td>
<td>50</td>
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<td>60</td>
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<tr>
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<td>56</td>
<td>50</td>
<td>48</td>
<td>46</td>
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<td>2</td>
<td>4</td>
<td>0</td>
<td>2</td>
<td>4</td>
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</tbody>
</table>
the required dimensions. Table 3 represents the weight and weight percentage details of constituents of 50 wt% GFENCs i.e., type 4, 5 and 6 as presented in table 2 [18].

The GFENC’s average actual densities of three specimens are measured using a simple method of water immersion and theoretical densities are calculated according to the equation (1).

\[
\text{Theoretical density} = \frac{W_f + W_n + W_m}{\left(\frac{W_f}{\rho_f} + \frac{W_n}{\rho_n} + \frac{W_m}{\rho_m}\right)}
\]  

Where, \(W_f\), \(W_n\) and \(W_m\) are weight fraction of glass fiber, nanoclay and matrix respectively. \(\rho_f\), \(\rho_n\) and \(\rho_m\) are density of glass fiber, nanoclay and matrix respectively.

ASTM D2734-94 standard is used to evaluate voids fraction of GFENC [19, 20]. From the theoretical and actual densities of the GFENC, the void fraction is calculated by equation (2).

\[
\text{Void fraction} = \frac{\text{Theoretical density} - \text{Actual density}}{\text{Theoretical density}}
\]  

2.3. Artificial aging in hygrothermal chamber

Fabricated composites are kept in the hygrothermal chamber (Model—SSLI-CC, supplied by Southern Scientific Lab Instruments, Chennai India) for 180 days [21, 22] at 40 °C (+/− 1 °C) with 60% (+/− 3%) relative humidity (RH).
2.4. Testing
Tensile and flexural experiments of ENC specimens are executed in compliance with ASTM D638 & D790 guidelines [23], respectively, because these two standards most often used for ‘plastics’ testing and GFENC specimens ASTM D3039 and D7264, respectively [14], because these to standards are used for polymer matrix composite materials reinforced by high-modulus fibers.

2.5. Scanning electron microscopy analysis
A ZEISS EVO18 scanning electron microscope is utilized to examine the morphology of ruptured surfaces of composite materials. SEM images are used to analyze the reasons for specimen failure.

3. Results and discussion

3.1. Epoxy-nanoclay composites (ENCs)

3.1.1. Tensile strength
Figure 2 shows the tensile properties of unaged and aged composite specimens. The presence of nanoclay has improved the tensile strength of the virgin epoxy by 5% at 2 wt% and 6% at 4 wt% of nanoclay (NC). Surface modified nanoclay addition leads to an increase in cross-linking density of epoxy, giving rise to increased toughness and tensile properties of the epoxy as a result of the load transfer interaction from the matrix to reinforcement [23]. The higher aspect ratio of nanoclay might have enhanced the tensile strength of the polymer matrix by enhancing the nanoclay interaction surface vicinity [24, 25]. Nanoclay serves as an active stress transfer constituent in the ENCs, leading to plastic deformation in the epoxy and eventually increasing the tensile strength [4]. High cross-link density will decrease the fracture toughness of epoxies due to internal stresses induced during curing of the epoxy. Within a high cross-link density epoxy, resistance to crack initiation is very low and the void growth due to plastic deformation is constrained. The presence of nanoclay prohibited crack propagation by generating a large amount of plastic deformation [26, 27].

Under the artificial aging in the hygrothermal chamber, the tensile strength of virgin epoxy specimens has diminished by 21% as compared to unaged specimens. The loss in the tensile strength is because of the plasticization and degradation of the epoxy due to penetration of water molecules in the epoxy [28]. Aged ENCs with nanoclay have reduced the tensile strength by 12% at 2 wt% and 10% at 4 wt% of nanoclay in comparison with unaged ENCs. It can be observed from the figure that adding nanoclay has decreased the percentage of reduction in tensile strength of ENCs compared to pure epoxy. Nanoclay has excellent barrier properties, which leads to establishing the ‘tortuous pathway’ for molecules of water to infiltrate into the ENCs [29].

3.1.2. Flexural strength
The flexural properties of unaged and aged specimens are represented in figure 3. The presence of nanoclay has increased the flexural strength by 9% at 2 wt% and 4% at 4 wt% of nanoclay. The morphology of the nanoclay layers or stacks is exceptionally important for increasing the tensile strength while decreasing the flexural strength at 4 wt% of NC. The flexural properties are largely subjected to the matrix, in parallel with the tensile properties. As a result, for flexural properties, agglomeration and poor dispersion defects are significantly evident. If the nanoclay platelet is located with the axis of the dumbbell, sliding platelets on each other could improve the tensile strength. Though, the flexural strength decreased, due to the increased number of platelets of nanoclay (4 wt%), further parallel and perpendicular to the force exerted [30].

Just like tensile strength under artificial aging, the flexural strength of virgin epoxy specimens is diminished by 22% as compared to unaged specimens. Similarly, aged ENCs with nanoclay have reduced the flexural strength by 13% at 2 wt% and 12% at 4 wt% of nanoclay in comparison with unaged ENCs. Nanoclay has a higher aspect-ratio that exploits as ‘sheet-shaped barriers’ and obstructs the pathway of molecules of water across the epoxy.

3.1.3. SEM analysis
A significant difference is found between the fracture surface of pure epoxy and ENCs, according to microscopic studies. As shown in figure 4(a), the pure epoxy displayed a completely smooth surface representing rapid crack propagation and poor fracture toughness [31, 32]. As compared to pure epoxy, the SEM micrograph (figure 4(b)) of ENC demonstrated a substantially rougher surface. Nanoclay in ENCs physically blocks and delays the propagation of the crack, and the resulting fracture surface of ENC shows river-surface-pattern. The river-surface-pattern provides a clear indication of increased fracture toughness [26].

The fracture surface of aged pure epoxy specimen has a network of micro-cracks throughout the fracture surface, as shown in figure 4(c). Moisture absorption under artificial aging, develops the craze initiation and propagation in epoxy, resulting in the formation of micro-cracks [31]. The fracture surface of aged ENC showed
lesser river-surface-pattern (i.e., lower critical stress intensity factor), compared to unaged ENC. The existence of shear leaps is shown in aged SEM micrograph (figure 4(d)), because shear yielding takes less energy to form a new layer, and the fracture toughness of the moisture absorbed samples is lower than the unaged samples. While shear yielding is found to be the main mechanism of failure in these samples, in these fracture surfaces there is also some sort of crack bifurcation and crack pinning.

3.2. Glass fiber epoxy-nanoclay composites (GFENCs)

3.2.1. Tensile strength

From figures 5(a), (c) and (e) and table 5, it can be witnessed that, the increased glass fiber wt% has enhanced the tensile properties of GFENCs. For unaged GFENCs at 0 wt% of nanoclay, the values of tensile strength and modulus are 249 MPa and 4.90 GPa, respectively. An increase in glass fiber weight percentage enhanced the tensile strength and modulus to 265 MPa & 5.35 GPa and 286 MPa & 5.37 GPa at 50 & 60 wt% glass fiber, respectively. Increased glass fiber weight percentage increases the deformation resistance, which can be seen from stress-strain curves that the reduction in strain to failure [33]. A higher glass fiber weight percentage enhances the fiber-matrix interface, and the interface guarantees an excellent load transfer [34].

Figures 5(a), (c) and (e) and table 5 show that adding nanoclay has increased GFENC’s tensile strength and modulus. Nanoclay addition of 2 and 4 wt% has enhanced the tensile strength to 270 & 281 MPa at 40 wt%,
283 & 298 MPa at 50 wt% and 297 & 319 MPa at 60 wt% of glass fiber. Similarly, tensile modulus has enhanced to 5.37 & 5.62 GPa at 40 wt%, 5.72 & 5.93 GPa at 50 wt% and 6.12 & 6.31 GPa at 60 wt% of glass fiber. Nanoclay guarantees the intensive transfer of load to glass fiber by increasing interfacial bonding and resistance to crack propagation [32].

From figures 5 (b), (d) and (f) and table 5, it can be observed that artificial aging has declined the tensile strength and modulus of GFENCs compared to unaged specimens. Absorbed moisture in the matrix induces swelling and de-bonding of the fiber-matrix [35]. Under artificial aging, molecules of water infiltrate into the micro-voids in GFENCs, leads to crack propagation. Table 4 represents the average actual density, theoretical density and void fraction of 50 wt% glass fiber GFENCs, i.e., type 4, 5 and 6 presented in table 2.

The aging of the GFENCs has reduced the tensile strength and modulus by 5%–18% and 8%–19%, respectively. Increasing glass fiber weight percentage with 0 wt% of nanoclay declined the reduction of tensile strength to 14 and 12% at 50 wt% and 60 wt%, as compared to 18% at 40 wt% of glass fiber. Similarly, the reduction in tensile modulus declined to 16 and 11% at 50 and 60 wt% of glass fiber, as compared to 19% at 40 wt% of glass fiber. Also, nanoclay addition of 2 and 4 wt% has declined the reduction in tensile strength to 16 and 14% at 40 wt%, 13 and 10% at 50 wt%, and 10 and 6% at 60 wt% of glass fiber. Similarly, the reduction

![Figure 3. Flexural properties of ENCs.](image-url)
in tensile modulus decreased to 17 and 15% at 40 wt%, 15 and 11% at 50 wt%, and 10 and 8% at 60 wt% of glass fiber. Epoxy's resistance to moisture absorption is increased by incorporating both glass fiber and nanoclay, which can be attributed to an improvement in the tortuosity path for water molecules to penetrate [36, 37].

3.2.2. Flexural strength

Table 6 shows that the weight percentage increase in glass fiber has improved the flexural properties of GFENCs. For unaged GFENCs at 0 wt% of nanoclay, the values of flexural strength and modulus are 341 MPa and 10 GPa, respectively. Increase in wt% glass fiber improved the flexural strength and modulus to 360 MPa & 11.46 GPa at 50 wt% and 375 MPa & 13 GPa at 60 wt% glass fiber. At increased glass fiber weight percentage, instigation of plastic deformation arises at a greater stress level [38]. The increased weight percentage of glass fiber in GFENCs, therefore, shows significant changes in flexural strength and modulus.

Once again, it can be noticed from table 6, that adding nanoclay has improved GFENC’s flexural strength and modulus. Nanoclay addition of 2 and 4 wt% has enhanced the flexural strength to 391 & 362.5 MPa at 40 wt%, 402 & 375 MPa at 50 wt%, and 417.5 & 392 MPa at 60 wt% of glass fiber. Similarly, flexural modulus has enhanced to 12.67 & 11.21 GPa at 40 wt%, 13.8 & 12.4 GPa at 50 wt% and 15.7 & 14.3 GPa at 60 wt% of glass fiber. At 4 wt% of nanoclay, there is a similar trend like ENCs. It is known that the existence of nanoclay

<table>
<thead>
<tr>
<th>Nanoclay (wt%)</th>
<th>Average actual density (g cm$^{-3}$)</th>
<th>Theoretical density (g cm$^{-3}$)</th>
<th>Void fraction (%)</th>
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<tr>
<td>0</td>
<td>1.744</td>
<td>1.8</td>
<td>3.11</td>
</tr>
<tr>
<td>2</td>
<td>1.77</td>
<td>1.825</td>
<td>2.99</td>
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<tr>
<td>4</td>
<td>1.793</td>
<td>1.843</td>
<td>2.67</td>
</tr>
</tbody>
</table>
enhances the bonding property of the epoxy, which allows a better interfacial bonding in GFENCs [39]. Excellent interfacial bonding delivers superior stress transfer among the constituents and enhances flexural properties [40].

From table 6, it can be observed that artificial aging has diminished the flexural strength and modulus of GFENCs compared to unaged specimens. Under artificial aging, the degradation of flexural properties of GFENCs attributed to matrix micro-cracks. Absorbed moisture might affect the fiber-matrix interface and lead to de-bonding and delamination [41]. The aging of the GFENCs has reduced the flexural strength and modulus by 8%–13% and 7%–12%, respectively. Increasing glass fiber weight percentage with 0 wt% of nanoclay

Figure 5. Stress versus Strain curves of GFENCs under tensile load. (a) Unaged specimens 40 wt.% of glass fiber (b) Aged specimens 40 wt.% of glass fiber (c) Unaged specimens 50 wt.% of glass fiber (d) Aged specimens 50 wt.% of glass fiber (e) Unaged specimens 60 wt.% of glass fiber (f) Aged specimens 60 wt.% of glass fiber.
declined the reduction of flexural strength to 12 and 11% at 50 and 60 wt%, as compared to 13% at 40 wt% of glass fiber. Similarly, the reduction in flexural modulus declined to 10 and 9% at 50 and 60 wt% of glass fiber, as compared to 12% at 40 wt% of glass fiber. Also, nanoclay addition of 2 and 4 wt% has decreased the reduction in flexural strength to 12 and 11% at 40 wt%, 10 and 9% at 50 wt%, and 10 and 9% at 60 wt% of glass fiber. Similarly, the reduction in flexural modulus decreased to 11 and 10% at 40 wt%, 9 and 8% at 50 wt%, and 8 and 7% at 60 wt% of glass fiber. This is due to the pooling of molecules of water in the area of the nanoclay platelet and glass fiber stacks and the modification of the molecular path of the water, resulting in less permeation of the material through the polymer [42].

### 3.2.3. SEM analysis
Fracture surface under tensile load of unaged (figures 6(a) and (b)) and aged (figures (c)–(f)) are shown in SEM micrographs. The presence of large resin clusters is evident in SEM micrographs of unaged samples showing good matrix adhesion to fibers; even after the fracture, the minimum amount of relative de-bonding between the fibers and the matrix is observed [43]. This indicates the presence of a strong bond in the unaged samples at the fiber and matrix interface. From figure 6(c), it is evident that the failure of the specimen is due to the poor fiber–matrix interface because of matrix degradation under the aging condition. Figures 6(c)–(f) exhibit that the failure of the aged specimen is due to crazing, micro-cracking, cracking, and flaking of the matrix material [44, 45].

### 4. Conclusions
Two types of composite materials are fabricated and characterized viz., epoxy-nanoclay composites (ENCs) with varying wt% of nanoclay and GFENCs with varying wt% of glass fiber and nanoclay. Fabricated composites are artificially aged in the hygrothermal chamber. The following conclusions are obtained from this study.

<table>
<thead>
<tr>
<th>Glass fiber wt%</th>
<th>Nanoclay wt%</th>
<th>Unaged</th>
<th>Aged</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Tensile strength (MPa)</td>
<td>Flexural modulus (GPa)</td>
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<th>Glass fiber wt%</th>
<th>Nanoclay wt%</th>
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<th>Aged</th>
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<tr>
<td></td>
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<td>Tensile strength (MPa)</td>
<td>Tensile modulus (GPa)</td>
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<td></td>
<td>4</td>
<td>319</td>
<td>6.31</td>
</tr>
</tbody>
</table>
1. For ENCs, nanoclay has improved the tensile strength and flexural strength of the virgin epoxy.

2. Aging in the hygrothermal chamber, the tensile and flexural strength of virgin epoxy specimens diminished, as compared to unaged specimens.

3. Addition of nanoclay has decreased the percentages of reduction in tensile and flexural strength for aged ENCs.

4. For GFENCs, an increase in wt% of nanoclay and glass fiber improved the tensile and flexural properties.

5. Aging of the GFENCs has reduced tensile and flexural properties.

6. An increase in wt% of nanoclay and glass fiber has diminished the reduction percentage of tensile and flexural properties. Epoxy’s resistance to moisture absorption has increased by incorporating both glass...
fiber and nanoclay, which can be due to an improvement in the tortuosity path for water molecules to penetrate.

7. The reasons for sample failure are identified by SEM micrographs under tensile load for unaged and aged conditions.

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