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Research Article

Effect of Graphene Fillers on the Water Absorption and Mechanical Properties of NaOH-Treated Kenaf Fiber-Reinforced Epoxy Composites

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This paper is focused on developing composites using kenaf fibers, epoxy polymer, and incorporation of graphene fillers. The kenaf fibers are treated with 5% NaOH to remove the hydrophilic nature and reinforced it with the hydrophobic matrix. The composites are fabricated using the compression moulding technique by keeping 60 wt.% epoxy as constant, and the graphene and kenaf fiber weights are changed accordingly. The samples for tensile, flexural, impact, hardness, and water absorption tests are prepared as per the ASTM D3039, D790, D256, D2240, and D572 standards, respectively. The effect of graphene fillers in the 5% NaOH-treated kenaf fibers reinforced with the epoxy matrix is tested. Among the various samples, sample 4 which has 6% graphene addition in the epoxy matrix reinforced with 5% treated kenaf fiber displayed the highest tensile strength of 63 MPa, flexural strength of 97 MPa, impact strength of 9.56 kJ/m^2, hardness value of 97, and lower water absorption of 5.13%. This is due to the proper dispersion of graphene fillers in the matrix which caused better interfacial adhesion between the fiber and matrix. The water absorption test showed the lowest value in sample S5 as the graphene fillers obstruct water penetration in the fibers. SEM analysis is done on the prepared samples to study the surface flaws and structural changes.

1. Introduction

The usage of synthetic fibers has created many threads to the environment which prompted the researchers to search for an alternative material. Natural fibers diminish ecological risks, and they can be used as a composite by reinforcing it with the polymer matrix [1]. Researchers have worked on natural fibers to identify their expanded applications and benefits. Natural fibers can be obtained from plants, animals, and a mineral base. Plant fibers as reinforcement material play a prominent role in developing natural fiber composites [2–4]. Among the plant fibers, bast and leaf fibers provide good mechanical properties because of their stiffness and hard structure. Generally, plant fibers can be divided into the primary cell wall and secondary cell wall. The primary cell wall comprises disordered cellulose, hemicellulose, lignin, pectin, etc. The secondary cell wall has crystalline cellulose in which S2 cell is responsible for mechanical properties. Hemicellulose acts as a matrix material that surrounds the cellulose structure, and lignin provides extra strength by protecting the fiber from external damage. This makes the cellulose have strength and stiffness. As the cell divides, the

cellulose and lignin amount rise, but the polyose content remains the same throughout [5–7]. The properties of natural fibers when reinforced with polymer matrix includes fiber content, orientation, microfibrillar angle, the interfacial bond between the fiber and matrix, and high water content [8]. However, when plant fibers get reinforced with the hydrophobic matrix, it results in poor bonding leading to less mechanical properties. This is because hemicellulose absorbs more water content from the atmosphere and forms new hydrogen bonds on the surface which makes the fiber to be hydrophilic. This hydrogen bond formation can be reduced by treating the fibers chemically with different processes. In addition to this, the components such as lignin, pectin, wax, and oily substances will be removed to a better extent, and it will result in enhanced stability of the fiber [9–11]. The properties that impact the influence between the fiber and matrix are expressed as follows: (i) proper mating of two unique materials, (ii) dispersion of fiber content in the matrix, (iii) orientation of fiber, and (iv) better surface interaction. Upon treating the fibers with different treatment processes, the mechanical properties of composites get enhanced by reduced water intake and better interfacial adhesion [12–14]. Sreenivasan et al. [15] reported that potassium permanganate-treated short Sansevieria cylindrica-reinforced polyester composites showed minimum water intake when compared to other treated fibers. Mylsamy and Rajendran [16] studied the effect of alkali treatment and optimum treatment percentage on agave Americana fibers. The optimum treatment is found to be 5%, and the treated fiber reduced the noncellulosic components and thereby enhanced the tensile property of the fiber. This is in line with the author. The mechanical and tribological properties can further be enhanced by incorporating fillers with the polymer matrix [17]. Fillers are additive materials that are used to enhance wear resistance, thermal stability, and flame resistance. The property of filler depends on its size, aspect ratio, and chemical composition. Ganesan et al. [18] assessed the effect of nanoclay filler and eggshell powder on the mechanical properties of jute fiber/polyester composite. The author concluded that the incorporation of filler enhanced the mechanical properties of the composite even if it is not chemically treated. The NaOH-treated jute fiber reinforced with eggshell powder (1.5%)+nanoclay (1.5%)/polyester matrix showed the highest flexural strength of 39.52 MPa than the untreated fiber. The increment is due to filler addition which hindered the formation of crack. Venkateshwar et al. [19] investigated the effect of different fillers (Al_2O_3, $CaCO_3$, and TiO_2) on the influence of mechanical properties of Prosopis juliflora epoxy-reinforced composites. The author reported that Al_2O_3 filler mixed uniformly in the matrix which resulted in enhanced bond strength. Benin et al. [20] concluded that Prosopis juliflora/epoxy composites with 12% barium sulfate filler provided superior mechanical properties. This is because the fillers and matrix remain bonded properly. Banyan fiber reinforced with 4% graphene incorporation in the epoxy matrix showed a higher tensile strength of 40.6 MPa and flexural strength of 163.23 MPa. This increment is due to the proper dispersion of graphene fillers in the matrix [21]. Nanofillers for instance graphene and carbon nanotubes have been proven to enhance the mechanical properties and reduced the water absorption content in the composites [22, 23]. For this experimental work, kenaf fiber (*Hibiscus cannabinus*) has been preferred because of its ability to produce in various environmental circumstances [24]. A study demonstrated that kenaf fiber can be used to create panels for furniture, seats, and armrests. It was also suggested that kenaf-reinforced composite materials might be used as noise obstacles and sound dampers [25]. This experiment work highlights the significance of incorporating graphene as a filler material in 5% alkali-treated kenaf fiber-reinforced epoxy composites. The above combination has not been reported in any literature data. The major purposes of the paper are expressed in the following ways: (1) effect of 5% NaOH treatment on kenaf fibers, (2) addition of graphene fillers with various proportions (0%, 2%, 4%, 6%, and 8%) in the matrix, (3) examining the mechanical and water absorption properties in the prepared composites, and (4) analyze the surface morphology of the prepared samples using SEM.

2. Materials

Kenaf fibers were purchased from KCT, Tifac core, Coimbatore, India. Epoxy resin (LY 556) and hardener (HY 951) are chosen as matrix material, and it has been collected from Covai Seenu & Company Coimbatore, Tamil Nadu. As per the manufacturer's suggestions, it is mixed in the ratio of 10 : 1. The resin and hardener are combined to generate a chemical effect that turns the liquid stage into a solid [26, 27]. Graphene as a filler material is added to the matrix material with various proportions (0%, 2%, 4%, 6%, and 8%).

2.1. Treatment of Kenaf Fibers. Kenaf fibers are immersed in 12 litres of distilled water for 1day to eliminate the unwanted particles residing on them. Later, the fibers are left to dry in the air for 24 hours. Dried kenaf fibers are soaked in 5% NaOH to remove the hydrophilic nature to a better extent. This 5% NaOH composition is 60 ml of NaOH in 12 litres of water. Treated kenaf fibers are washed thoroughly in distilled water to remove the additional NaOH present in them. Then, the fibers are left to dry in the air for 1day.

2.2. Fabrication of Graphene-Filled Composites. The treated kenaf fibers were cut into 30 cm length with the help of a cutter based on the moulding constraints. Graphene filler and epoxy resin cannot be mixed thoroughly as the resin chosen is highly viscous. To avoid this constraint, the graphene powder was scattered into the epoxy resin and then whisked for a prolonged period of 40 min at 80°C. Then, the hardener was added to the graphene/epoxy mixture in the ratio of 10 : 1. Aluminium plates of dimension 300 ∗ 300 ∗ 5 mm are chosen for the compression moulding process (Supplier: Modern Plastics Pvt Ltd., Coimbatore, India). To obstruct heating of the plates, white grease is applied to them. For sample 1, the epoxy resin along with the hardener was poured on the surface of the aluminium plate, and then,

the 5% NaOH-treated kenaf fibers are placed above the resin mixture in a unidirectional way. Then, the epoxy resin along with the hardener was poured above the fiber. For other samples, it was prepared by the addition of graphene fillers (2%, 4%, 6%, and 8%) into the epoxy resin, and the same process is repeated as mentioned above. The prepared laminates are processed in the compression moulding machine, and they are kept at a temperature of 130°C, with a pressure of 35 bar for 45 minutes. Later, the laminates are left for the curing process for 50 minutes. With the help of a diamond cutter, the final laminate of dimensions (300 ∗ 300 ∗ 5 mm) is removed from the mould. The prepared laminated can be tested mechanically as per the ASTM standard. Table 1 shows the composition of the prepared sample.

2.3. Physical and Mechanical Tests

2.3.1. Tensile Test. It is one of the mechanical tests to determine the material behaviour under applied load. The samples are prepared as per the ASTM D3039 requirement with a dimension of 250 ∗ 25 ∗ 5 mm and a crosshead speed of 2 mm/min [28]. It was tested in a computerized universal testing machine (Supplier: Aimil Ltd., India). Before the testing process, the samples are mounted onto the machine, and utilizing a hydraulic system, they are gripped to avoid dislocation. The value of each sample tested is noted. A total of three samples were tested in each composition, and the average value is taken for the analysis.

2.3.2. Flexural Test. This test determines how much a material will bend under the applied load. The samples for this test are prepared as per the ASTM D790 requirement with a dimension of 125 ∗ 12.7 ∗ 5 mm and a crosshead speed of 2 mm/min [10]. It was tested in a computerized universal testing machine, and the values for each sample are recorded. A total of three samples were tested in each composition, and the average value is taken for the analysis.

2.3.3. Impact Test. This test determines how much a material can absorb toughness during the applied load. The samples for this test are prepared as per the ASTM D256 requirement with dimensions of 65 ∗ 12.7 ∗ 5 mm [10]. It was tested using digitalized Izod impact test, and the values for each sample are taken. A total of three samples were tested in each composition, and the average value is taken for the analysis.

2.3.4. Hardness Test. This test determines how much a material will experience localized deformation under mechanical indentation or scratching. The samples for this test are prepared as per the ASTM D2240 requirements with dimensions of 20 ∗ 20 ∗ 5 mm [10]. Shore D durometer is used to test the samples. At 6 different locations, the indentations were made, and the mean values are noted.

2.3.5. Water Absorption Test. The resistance to absorbing water is tested using this test. The samples are prepared as per the ASTM D572 requirements with dimensions 64 ∗ 12.7 ∗ 5 mm [29]. The samples are immersed in distilled water for 5 days at room temperature, and the changes are noted accordingly. After a certain interval of time, the samples are taken out and wiped with a cloth, and then, the weight of the sample is measured. The amount of water absorbed in a sample is determined using the below-mentioned formula, where W_b represents the final weight after immersion, W_a indicates the initial weight of a sample, and W shows the percentage of water absorbed.

$$W = \frac{W_b - W_a}{W_a} * 100. \quad (1)$$

Table 1: Composition of the prepared samples.

Sample number	Kenaf fiber (wt.%)	Epoxy resin (wt.%)	Graphene (wt.%)
S1	40	60	0
S2	38	60	2
S3	36	60	4
S4	34	60	6
S5	32	60	8

2.4. SEM Analysis.

A scanning electron microscope was used to analyze the surface flaws and structural changes of the prepared samples using SEM JEOL JSM-6510LA. For this technique, the operating voltage is 25 kV.

3. Result and Discussion

3.1. Tensile Strength. Tensile values of each composite under various loads are presented in Figure 1. 6 wt.% Gr fillers reinforced in treated kenaf/epoxy composite showed a maximum value of 63 MPa, and 0 wt.% Gr fillers showed the minimum value of 36 MPa. With the addition of Gr fillers, the tensile strength increased, and beyond a certain limit, it begins to decline. Sample S4 which have 6 wt.% Gr fillers proved to have maximum tensile strength and optimum content. This value is obtained because of the nature of graphene incorporated as a filler material. Graphene fillers obstruct the water molecules to penetrate the fiber, thereby creating proper bonding between the fiber and matrix. Graphene filler has a strong C-C bond which makes it difficult to rearrange its positions resulting in brittle nature [30]. Furthermore, the 6 wt.% Gr reinforced in treated kenaf/epoxy composite bonded properly, and graphene particles are dispersed uniformly in the matrix, thereby enhancing tensile strength than other Gr variations. 6 wt.% Gr fillers provided better interlocking with the fiber-matrix adhesion, and when stresses are applied, it is distributed evenly in the composite [31]. In sample S5, 8 wt.% Gr fillers reinforced in treated kenaf/epoxy composite showed a tensile strength of 58 MPa which is greater than 0 wt.% Gr, 2 wt.% Gr, and 4 wt.% Gr fillers samples and less than 6 wt.% Gr filler sample. With an increase in Gr fillers, the agglomeration of particles tends to take place, and crack propagation will not occur which indicated that the addition of graphene fillers makes the composite resulting in enhanced brittleness instead of higher tensile strength [21]. 8 wt.% Gr fillers will undergo a necking process within a short duration, and the

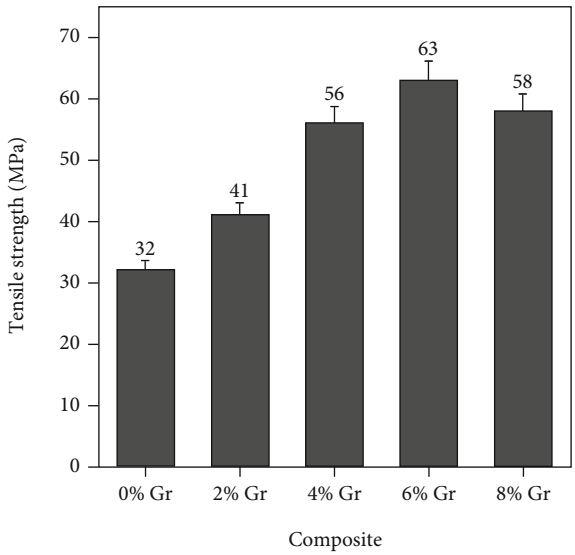

Figure 1: Tensile strength of graphene-filled kenaf/epoxy composites.

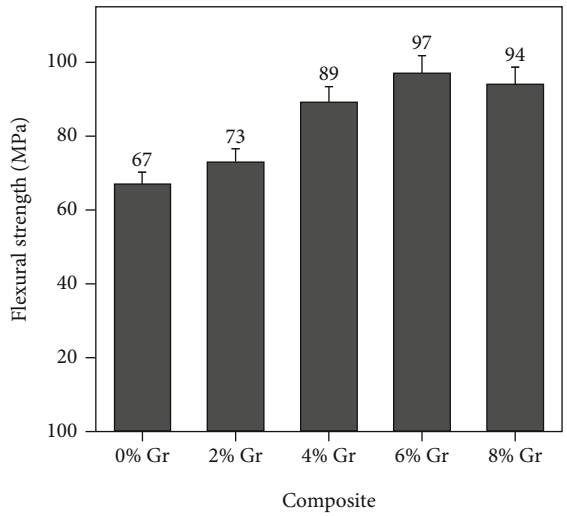

Figure 2: Flexural strength of graphene-filled kenaf/epoxy composites.

material breaks rapidly because of the brittle nature of graphene. 4 wt.% Gr fillers showed a value of 56 MPa which is greater than 0 wt.% Gr and 2 wt.% Gr and lower than 6 wt.% Gr and 8 wt.% samples. This decrement is because this concentration is not sufficient to hinder void formation in the matrix when compared to the 6 wt.% Gr filler sample [32]. 2 wt.% Gr filler sample showed a tensile strength of 41 MPa which is greater than 0 wt.% Gr and lesser than other graphene samples. This concentration reduced void content comparatively less than other Gr samples. In sample S1, the kenaf fibers are treated with 5% NaOH, and it is reinforced with epoxy resin to remove the noncellulosic contents to a good extent. The tensile strength of 32 MPa is attained due to the removal of impurities. 5% NaOH effectively removes this, but some constituents tend to degrade and created a void in it resulting in ease of water penetration also decrease in tensile strength.

3.2. Flexural Strength. The values of each sample for the flexural test are presented in Figure 2. Sample S4 displays the highest flexural strength of 97 MPa followed by sample S5 (94 MPa), sample S3 (89 MPa), sample S2 (73 MPa), and sample S1 (67 MPa). Sample S4 showed the highest flexural strength because the 6 wt.% Gr fillers with the matrix enhanced the bonding in the interface, thereby promoting better load sharing capability. Also, 6 wt.% graphene addition proved to be an optimum concentration because the fillers are dispersed uniformly in the matrix, thereby reducing holes and enhancing the flexural properties. 8 wt.% Gr fillers in sample S5 showed flexural strength of 94 MPa which is lower than sample S4 and higher than all other samples. This is due to the reason that 8 wt.% Gr fillers caused agglomeration in the matrix; also when reinforced with treated kenaf fiber, it resulted in improper interfacial bonding between the fiber/matrix. With 8 wt.% Gr addition, the flexural property of the composite starts to decline because of delamination between the layers. Also, 8 wt.% Gr fillers did not disperse throughout which caused some debris in that region, and when the load is applied, the sample showed lower flexural properties and enhanced brittleness of the composite [21]. 4 wt.% Gr and 2 wt.% Gr fillers in samples 3 and 2 are insufficient to hinder void formation causing fiber pull-out in the surface and resulting in lower flexural strength of value 89 MPa and 73 MPa when compared to 6 wt.% Gr and 8 wt.% Gr filler samples, respectively. In sample S1, treated kenaf fibers removed the hydrophilic nature in the fiber and have been well bonded with the matrix. But the load distribution did not occur evenly in the composite because of the microgaps present in it leading to a decrease in flexural strength of a value 67 MPa when compared to other varying Gr filler samples.

3.3. Impact Strength. It determines how much a material can absorb energy under applied load. The values of each sample for this test are displayed in Figure 3. Sample S4 showed maximum impact strength of 9.56 kJ/m^2 and minimum impact strength of 4.85 kJ/m^2. 6 wt.% Gr fillers in sample S4 showed the highest impact strength (9.56 kJ/m^2) as 6 wt.% graphene addition absorbed more energy before the inception of brittle behaviour. This is because the optimum concentration of graphene into the matrix resulted in better interlocking between the fiber and the matrix. The increase in impact strength depends on factors like the toughness of the composite, compatibility between the fiber and matrix, and proper dispersion of filler material into the matrix [21]. 4 wt.% Gr fillers in sample S3 showed an impact strength of 7.32 kJ/m^2 which is higher than 8 wt.% Gr filler samples. 4 wt.% Gr fillers absorbed more energy than 8 wt.% Gr fillers, and this is due to the fact that 4 wt.% Gr fillers did not make the composite crack easily, whereas the addition of Gr fillers beyond 6 wt.% made the composite embrittle. 8 wt.% Gr filler in sample S5 showed an impact strength of 7.18 kJ/m^2 which is greater than 0 wt.% Gr and 2 wt.% Gr and less than 6 wt.% Gr and 4 wt.% Gr filler samples. This is due to the agglomeration of graphene particles in the matrix region which caused the composite to lose its

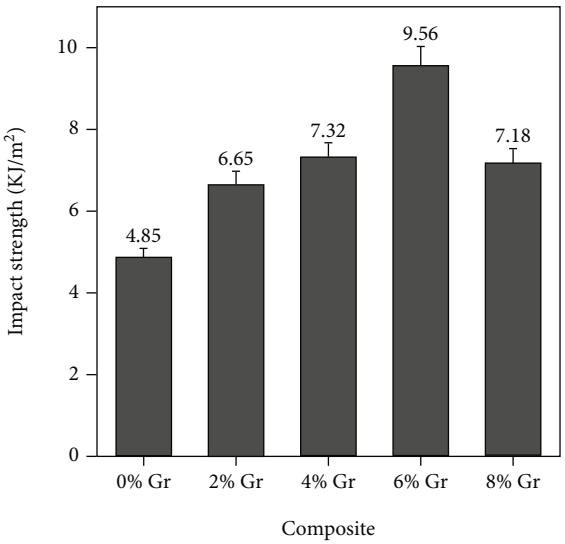

Figure 3: Impact strength of graphene-filled kenaf/epoxy composites.

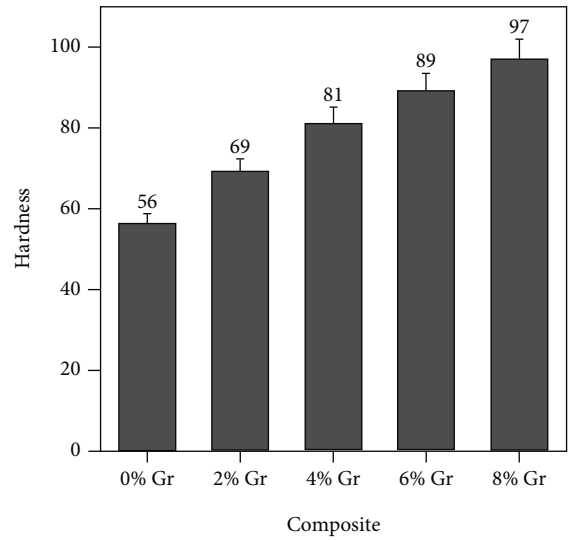

Figure 4: Hardness values of graphene-filled kenaf/epoxy composites.

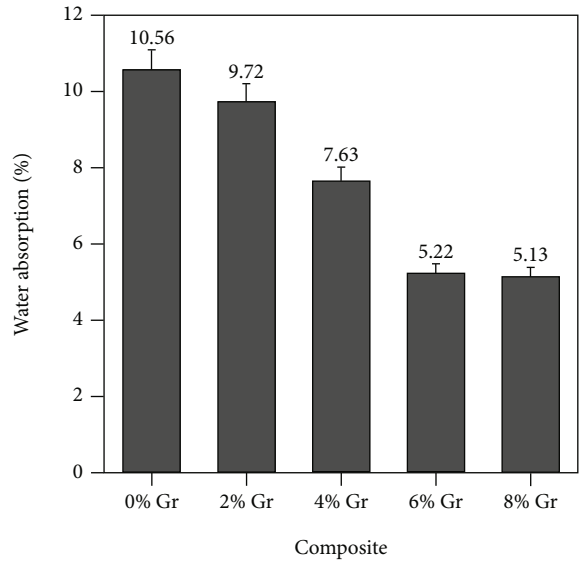

Figure 5: Water absorption of graphene-filled kenaf/epoxy composites.

ductile nature and hence resulted in a reduction in impact strength. 2 wt.% Gr filler in sample S2 showed an impact strength of 6.65 kJ/m^2 which is lower than 6 wt.% Gr, 4 wt.% Gr, and 8 wt.% Gr fillers and greater than 0 wt.% Gr filler sample. This is due to the reason that 2 wt.% Gr did not hinder the void formation as compared to 6 wt.% Gr, 4 wt.% Gr, and 8 wt.% Gr filler samples. Sample S1 which has 0 wt.% Gr filler showed the lowest impact strength of 4.85 kJ/m^2 when compared to all other Gr fillers with varying samples. This is because when it is treated, the fibers are split into smaller ones making it withstand load, but the microgaps which are present in the composite led to a reduction in lower impact strength.

3.4. Hardness. It will determine how much a material can withstand penetration depth when the load is applied to it. If the material is brittle, then the hardness will be more [21]. The hardness value of each sample is displayed in Figure 4. 8 wt.% Gr filler in sample S5 showed a maximum hardness value of 97, and 0 wt.% Gr in sample S1 showed a lower hardness value of 56. 8 wt.% Gr filler in sample S5 showed a higher hardness value because the brittle nature of graphene particles also resisted penetration when the load is applied on it when compared to all other samples. 6 wt.% Gr filler in sample S4 showed a hardness value of 89, and this is greater than 0 wt.% Gr, 2 wt.% Gr, and 4 wt.% Gr fillers and also lesser than 8 wt.% Gr filler samples. 6 wt.% Gr filler showed uniformed dispersion of graphene in the matrix and enhanced the interfacial bonding between the fiber/matrix, but the addition of more graphene fillers showed resistance to penetration. 4 wt.% Gr filler in sample 3 showed a hardness value of 81 which is greater than 0 wt.% Gr and 2 wt.% Gr and lower than 8 wt.% Gr and 6 wt.% Gr filler samples. 4 wt.% Gr filler did not lock the void formation resulting in fiber pull-out from the surface which resulted in lowering of hardness value. In the sample S2, 2 wt.% Gr filler in the matrix is not sufficient to increase the hardness value, so this composite displayed a hardness value of 69 which is higher than 0 wt.% Gr filler but higher than other Gr filler samples. The treated kenaf fibers reinforced with epoxy matrix in sample S1 showed a hardness value of 56 which is lower than all other Gr filler samples. 5% NaOH-treated kenaf fibers provided a rough surface, so the sample resisted penetration initially, but after a certain period, it failed because of some hydrophilic nature in the fiber.

3.5. Water Absorption Tests. This test is used to determine the hydrophilic nature of the fibers. The values of water absorption tests for all samples are displayed in Figure 5. Sample S5 which have 8 wt.% Gr filler reinforced in treated kenaf/epoxy composite showed more resistance to water absorption than other samples. Graphene plays an obstacle

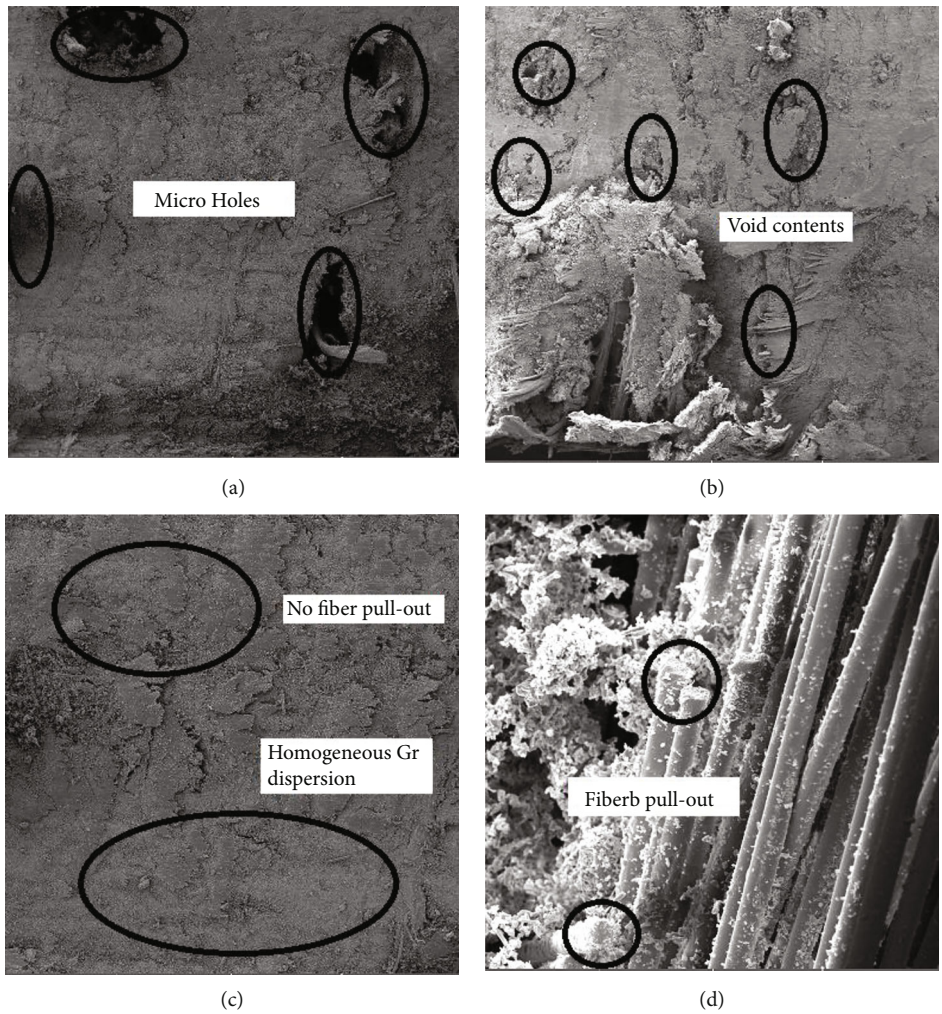

Figure 6: (a–d) represents SEM images for various concentration of graphene filler reinforced in treated kenaf/epoxy composite.

in transporting water to the composites [22]. With the addition of graphene fillers, the void content is minimized, thereby locking it with the matrix and leading to better interfacial adhesion between the fiber and matrix. Due to this reason, the water absorption test displayed lower valves. Sample S1 showed high water absorption of 10.56% when compared to all other Gr filler samples. The kenaf fibers are treated with 5% NaOH to remove the impurities and noncellulosic content like hemicellulose and pectin, and then, it is reinforced with epoxy resin. Even after treating the kenaf fiber, it possesses some hydrophilic nature and void content. With 2 wt.% Gr addition in sample S2, the gaps present in the matrix are removed partially, and some holes allowed the fibers to interact with water molecules, thereby absorbing water content. Sample S2 showed a water absorption value of 9.72 than sample S1 but higher than all other Gr filler samples. The addition of graphene fillers resisted water penetration by locking the void formation. In sample S4, 6 wt.% Gr addition resisted water absorption as the concentration of filler addition locks the void from interacting with the water molecules. 6 wt.% Gr filler showed a value of 5.22 which is lower than sample S5.

3.6. SEM Analysis. Figure 6(a) represents the SEM image of sample S1 which consists of 5% NaOH-treated 40 wt.% kenaf fiber reinforced with epoxy resin. This sample showed a rough surface due to the removal of noncellulosic components in it, but there are a few microholes that are present in the matrix after the treatment. These microholes may create stress concentration leading to the deterioration of mechanical properties of the composites [33]. In sample S2, the 2 wt.% Gr addition disperses well with the matrix by hindering void formation to some extent, so the water absorption is reduced, and the interfacial bond will be better than 0 wt.% Gr filler sample. This change can be seen in Figure 6(b). In sample S3, 4 wt.% Gr filler reinforced in treated kenaf/epoxy composites showed proper bonding and minimal void content in the matrix. Figure 6(c) represents the SEM image of sample S3 which showed well dispersion of graphene with the matrix, no fiber pull-out, and proper interfacial adhesion between the fiber and matrix. Above 6 wt.% Gr incorporation with the matrix leads to fiber pull-out. According to Zhang et al. [33], after 5% NaOH treatment, the interfacial adhesion between the kenaf fibers and the matrix was greatly improved with 6 wt.% Gr. Treated kenaf fibers with 6 wt.% Gr had extremely greater tensile strength than other

composition, and the fracture of treated kenaf fibers could greatly enhance the mechanical strength of composites. In Figure 6(d), the SEM image of sample S5 has 8 wt.% Gr incorporation with the matrix which displayed agglomeration in the matrix region causing weak interfacial adhesion between the fiber and matrix. The poor fiber/matrix interface could not provide sufficient stress transport; thus, the mechanical properties of the composites decreased significantly. Severe fiber/matrix debonding with nanofillers could be witnessed due to the poor interfacial adhesion between fibers and the matrix, as shown in Figure 6(d). The unnecessary voids and microholes in the composites (S1 and S2) were witnessed at lower/higher weight fraction of graphene, which may reduce the reinforcing effect of the nanofillers resulting in the reduction of mechanical properties [21].

4. Conclusion

The mechanical and water absorption test is carried out for the various concentrations of reinforcement graphene filler with kenaf. Increment in mechanical properties strongly depends on the uniform dispersion of graphene in the matrix. The major findings of incorporating graphene in the treated kenaf/epoxy composites are discussed as follows. Graphene plays a major role in obstructing water penetration in the composite, thereby minimizing void concentration in it. The investigation proved that the addition of graphene improved the performance of the composite thermal stability. With more than 6 wt.% Gr filler reinforced in treated kenaf/epoxy composites, the properties like tensile, flexural, and impact strength start to decline, but maximum hardness value of 97 is achieved at 8 wt.% Gr filler sample. Beyond 6 wt.% graphene addition, the composite becomes embrittle causing agglomeration in the matrix and reduction in mechanical properties. SEM analysis is done to study the morphological surface of various graphene concentrations reinforced in treated kenaf/epoxy composites. 6 wt.% Gr filler sample is found to optimum, and this displayed no fiber pull-out, better bonding in the SEM analysis.

Data Availability

The data used to support the findings of this study are included in the article.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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