Effect of grain size on the structure and properties of coir epoxy composites

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Abstract

The reinforcement of different polymers by natural fibers has recently been the center of attention among many research groups around the globe. The major factors that affect the mechanical performance for the composites are the surface treatment for the reinforcing material as well as the variation in the fiber diameters. In this study, coir fibers with different grain sizes varied between 250 and 950 μm were chemically treated by alkali solution and employed as a reinforcing agent for epoxy. The morphology and the mechanical performance represented by impact and flexural strength for the neat polymer and the resulted composites were investigated. The weight fraction was 10.0 wt %, and the approach employed for preparing the samples was hand layup technique. The obtained results confirmed a mechanical improvement for the composites compared to the neat polymer. Flexural strength was considerably improved ($\sim 94\%$) for the composite with higher grain size of 950 μm . On the other hand, the impact strength was also significant as it was improved by 92% for the composite of lower grain size of 250 μm . The scanning electron microscopy (SEM) results were correlated with the mechanical performance of the polymer and its composites.

Keywords Epoxy · Coir fibers · Impact strength · Flexural strength · SEM

1 Introduction

Recently, the issues of global warming and environmental threats have been attracting a significant concentration [1]. These issues have encouraged researchers to seek for possible solutions and looking for alternative materials and green technology in many industrial sectors. The latter are relying on the production cost of composites and their functional applications such as transportation (automobiles, railway coaches, aerospace), building and construction industries (ceiling paneling, partition boards) and furniture packaging [2–4].

Natural fiber composites (NFCs) are becoming increasingly attractive for their diverse use in many applications. These materials possess unique properties such as improved specific strength, high modulus,

biodegradability, eco-friendliness, eco-efficiency nature, low rate of carbon emission and ease of fabrication [5–7]. Natural fillers that used to reinforce polymer matrices offer a new group of materials that provide notable mechanical characteristics with various applications [8–10]. Coir fibers (CFs) are versatile lignocellulosic fibers and are comprehensively utilized in the scope of various industrial applications. *Cocos nucifera* or coir fibers can be obtained from the tissue surrounding the seed of coconut palm. They are composed of cellulosic fibers with hemicelluloses, pectin and lignin as a bonding material. Fibers used in this work (coir) have low cellulose and hemicelluloses; they are stiff and tough fibers due to high content of lignin [11, 12]. Likewise, they have high microfibrillar angle contrasted with other common natural fibers. The mechanical performance of composites of natural fibers depends on many

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parameters such as the volume fraction of fibers, length, shape, arrangement, fiber polarity and the interfacial bonding with the polymer matrices [13, 14].

Along with a number of benefits of natural fibers as reinforcements, there are some limitations on using natural fibers in composite preparation such as the lack of adequate adhesion between fibers and the matrix, hydrophilic nature as they contain strongly polarized hydroxyl and poor thermal stability [1, 15].

Natural fibers are axiomatically incompatible with some of polymer matrices, such as polypropylene (PP) and polyvinyl chloride (PVC), as these matrices have hydrophobic nature. This shortcoming leads to weak interfacial bonding with polymer matrices which, as a result, lead to debonding of fibers and failure in the end use production [16]. The interface between reinforcing fibers and the matrix plays a critical role in determining the mechanical performance of green composite. Generally, the properties of natural fiber composites are unequivocally connected with the nature of the natural fibers and their compatibility with polymer matrices [17, 18].

Hence, it is very important to reduce the moisture absorption and hydrophobic character of natural fibers by making a proper surface treatment to enhance fiber compatibility with different resin matrices [19–21]. There are a number of chemical treatments that can be used to improve the compatibility between matrix and fiber and increase the functionality of natural fibers. Alkali treatment is the most used chemical treatment for natural fibers to remove wax and oil covering some parts of these fibers and to increase the roughness of fiber surfaces that lead to better interlocking of fibers with the host polymer [16].

GU [22] used alkali treatment, NaOH solution, with concentrations varied from 2.0 to 10% separately on freshly brown laminated coir fibers to study their tensile behavior. He reported an improvement in coir fiber adhesion with polypropylene after the chemical treatment.

He also reported a decrease in tensile strength values as the concentration of NaOH increased. One of the main conclusions was stated that as the concentration of NaOH is higher than 8%, the adhesion of the fiber with the matrix is improved.

In similar procedure conducted by Karthikeyan et al. [23], NaOH was used for treating freshly retted long combed coir fibers. They made various concentrations (2.0, 4.0, 6.0, 8.0 and 10) % at ambient conditions for 10 days. The treated fibers were reinforced epoxy resin to study the mechanical properties. The treatment with 6% NaOH provided the best result in impact strength, while higher alkali concentrations reduced fiber strength and consequently the impact strength.

Anyakera [24] has conducted that coconut palm fibers are promising alternative candidates to replace synthetic

fibers for reinforcing a thermoset polymer that was polyester resin. The latter can be used for many applications such as building construction. He reported that surface treatment to coconut fibers improve the mechanical properties of the composites.

Potadar and Kadam [25] published their comparative study between groundnut shells and coir fibers as natural reinforcing agents for epoxy. They investigated the effect of grain size of the natural reinforcing agents on the flexural strength of the resulted composites. They confirmed the positive effect of the two types of natural reinforcing agents, as well as the grain size of fibers, on the flexural strength of the resulted composites compared to the neat polymer. Epoxy/coir fibers with grain size of 1.0, 1.5 and 2.0 mm, respectively, scored higher values of flexural strength compared to epoxy/groundnut shells with similar grain size.

In this work, different grain sizes of coir fibers are reinforced epoxy resin. The mechanical performance for the resin and the resulted composites, represented by impact and flexural strength, is reported with a special focus on the failure mechanism. Also, the morphology of the fibers and the composites is investigated. The main aim of this study is to design composites by selecting abundant materials with adopting a cost effective approach of manufacturing. The aforementioned investigation possibly helps in failure prediction approach for systems designed by employing these abundant materials.

2 Experimental part

2.1 Materials

Coconut shell purchased from the local market, Baghdad, Iraq, was used as material for reinforcement, while epoxy resin (Sikadur-52) obtained from commercial chemical store in Iraq was used jointly with hardener to serve as a matrix. The mix of mass ratio between the resin and its slow hardener was 2:1 which means 800 gm of the resin mixed with 400 gm of the hardener. The density of the epoxy resin was 1.12 g/cm³.

2.2 Preparation of composite

The preparation of the coir shell involved washing with soap and detergent, cleaning using dried cloth, drying in sunlight for 3 days then in oven (Mettler Germany) operating at a temperature of 60 °C for 3 h, followed by crushing with hammer. The dried coir fibers were then soaked in sodium hydroxide (NaOH) solution with 2.0% w/v concentration for 3 h to remove minor impurities. After completing the soaking process, the fibers were taken out and

washed in tap water again, and then dried by adopting the same aforementioned procedure. Subsequently, the coir fibers were crushed to a smaller size by using a hammer. Finally, fibers were grinded using a coffee mill and sieved through the meshes varied from 0.075 to 1018 μm to obtain fine uniform shapes and to get different particles' size. These different finely sieved particles were employed as reinforcement material in the epoxy polymer matrix. The hand layup process was employed for preparing the specimens. Before the start of the molding process, the mold surface was cleaned and oiled with a suitable mold-releasing agent which was necessary to ensure an easy removal of the specimens from the used mold.

For preparing composite material, 10.0 wt% of fibers was put together into small beaker and mixed by using mechanical stirrer and low heat of 30 $^{\circ}\text{C}$ for 10 min in low speed (100 rpm) to ensure that air bubbles were not formed. Afterward, hardener was added to the mixture and continuous stirring was resumed at same low speed until the mixture started to be thick. The mixture then poured in the mold and left to be cured for 24 h at room temperature which was 25 $^{\circ}\text{C}$. Then, the specimens were

removed from the mold and inserted in oven for 3 h at 60 $^{\circ}\text{C}$ to complete the cross-linking process of epoxy (Fig. 1).

3 Characterization

3.1 Flexural strength

Flexural strength test was performed on the samples of size (100 $\times$ 10 $\times$ 5) mm according to ASTM D 790 standards. In this three-point bending test, load was applied at three points in which the top, middle and bottom layers of the samples were subjected to compression, shear and tension forces, respectively. The test specimens were placed between two jaws with 80 mm span length between them. A load of 500 N was applied with a crosshead speed of 2.0 mm/min was set for this test. Three composite specimens were tested for each sample and average result was reported.

Fig. 1 Steps of coir treatment and composite preparation

3.2 Impact strength

Impact strength test was performed using Charpy impact machine with a hammer of 2.0 J to obtain the fracture of specimens. The size for each specimen was $(55 \times 10 \times 5)$ mm according to the standard of ISO179. The samples were cut using a circular saw. Three samples for each grain size were tested at ambient conditions, and the average result was reported.

3.3 Scanning electron microscopy (SEM)

Morphology of particles, distribution of fibers in the matrix and fracture surfaces of some samples are observed using SEM (TESCAN MIRA3, Czech Republic). Those composite samples of 250 μm grain size were imaged to investigate the distribution of fibers in the matrix and to be correlated with the mechanical properties. The samples are mounted onto SEM holder using double-sided electrically conducting carbon adhesive tapes to prevent surface charge on the specimen when exposed to electron beam.

4 Results

4.1 Flexural strength

Mechanical properties have great importance as an indispensable tool for investigating the behavior of natural fiber polymer composites. The mechanical behavior for the natural fiber polymer composites is influenced by many factors such as orientation of fibers and their distribution in the host polymeric material, the nature of the fibers used to reinforce the polymer and the interphase region. Flexural strength is considered as one of the most widely spread mechanical tests used to assess the mechanical performance of the composites which are employed in structural applications [26].

Flexural test is performed in order to evaluate the efficiency and improvement in mechanical characteristics of composite after changing the grain size of natural fibers. The importance of this test derived from the fact that this test is a combination of the compressive and tensile strengths. Consequently, flexural modulus is directly associated with the evaluation of the deformation of the composite materials in bending mode [27]. Flexural strength of epoxy records 60.792 MPa, as given in Table 1. When fibers reinforced the matrix, the values of flexural strength are increased because of the chemical treatment of fibers that enhances the interfacial bonding interaction between fibers and matrix [28].

Results indicate that the flexural strength is firstly increased with the increment in grain size. The grain size

Table 1 Average flexural and impact strength of neat polymer and composites' specimens

Specimens	Grain size (μm)	Flexural strength (MPa)	Impact strength (KJ/ m^2)
Epoxy (PA)	–	61.11 ± 0.45	5.21 ± 0.81
Composite (CB)	950	103.57 ± 18.86	5.45 ± 0.32
Composite (CC)	750	67.2 ± 10.82	6.68 ± 2.23
Composite (CD)	250	73.30 ± 5.66	8.69 ± 1.12

is become more compactly filled with narrow distance between particles in the epoxy resin and the shape of particles are become more irregular [29]. The increment in the values of the flexural strength associated with the highest grain size can be attributed to the increment in the stiffness of the composite due to the mobility reduction of the polymeric chains as the grain size is going higher [30]. Bello et al. [31] confirmed that the presence of reinforcements in the matrix led to the improvement in flexural strength values as the composite was stiffened and the flexural deformation associated with flexural strength was delayed. On the other hand, the poor mechanical performance revealed by the flexural strength associated with lower grain size can be ascribed to the crack formation at the interphase region, poor stress transfer from the fibers to the matrix and weak interfacial bonding. All the aforementioned lead to a weak structure that lead to a poor mechanical performance related to lower grain size [30]. The grain size of 750 μm showed the lowest value of flexural strength compared to values related to other grain sizes according to uneven geometrical patterns for the fibers. The distorted geometrical features for fibers inside the brittle resin may play a crucial role in the decrement of the values of flexural strength findings [32]. This explanation was adopted in the literature by Ameh et al. [33]. This behavior is in contrast to what has been concluded in the literature by Seth et al. [34] who emphasized that the smaller the grain size, the superior the properties due to good compaction, little porosity and efficient stress transfer. The relationship between the obtained results of the flexural strength and the grain size is illustrated in Fig. 2.

4.2 Impact strength

The coconut shell particles have considerable effect on the strength, hardness and impact energy of composite [35].

The impact strength is a measure of the fracture toughness of materials when they are subjected to an impact load. The impact properties of composites are considerably influenced by the nature of the constituent materials, fiber/matrix interface, construction and geometry of the composite, and they are also relying on the test

Fig. 2 Flexural strength of coir fiber epoxy composites

Fig. 3 Impact strength of coir fiber epoxy composites

conditions [36]. Impact strength is increased according to the enhancement in the elasticity of the composites which led consequently to an improvement in matrix deformability [8]. In general, natural fibers play a central role in the impact resistance of fiber-reinforced composites as they interact with the crack formation and act as stress transferring medium [36].

In the current study, the impact strength of coir epoxy composites with different grain size is greater than pure epoxy material as shown in Fig. 3. It is observed that all the composite specimens are showing improvement in the impact strength due to good adhesion between the fibers and matrix which is responsible for the good resistance to crack propagation [4].

From the data given in Table 1, the impact strength decrease as the grain size increase. The diminishing in impact strength is due to the inability of the reinforcements to block the crack propagation resulting in reduction in the impact strength [8].

At 250 μm fiber grain size, the impact strength gives the highest value followed by 750 μm of grain size. This is probably due to fiber–matrix surface interaction efficient in stress-absorbing capacity. Aruniit et al. [37] confirmed

that the impact strength was improved for unsaturated polyester as the reinforcing agent, which was alumina tri-hydrate, had smaller grain size [37].

It should be mentioned that Hassan et al. [38] reported that impact strength of oil palm empty fruit bunch fiber reinforced with polymer composites decreased with increasing of fiber size [38]. Mohamed et al. confirmed that the bigger fiber grain size of 500 μm had a negative effect on the impact strength compared to 125 μm fiber grain size reinforced high-density polyethylene blended with natural rubber. The aforementioned findings were confirmed for a range of weight fractions (0, 10, 20 and 30) wt % for different grain sizes of 125, 250 and 500 μm. As the grain size of mengkuang fiber-reinforced thermoplastic natural rubber went higher, the values of impact strength were decreased. The authors attributed this behavior to the weak adhesion between the fibers and the matrix that impeded the efficient stress transfer [39].

4.3 Scanning electron microscopy SEM

The morphology of modified surface for coir fiber is shown in Fig. 4 micrographs (a) and (b). The typical micrograph for coir fiber cross section, which was not reached in this work, includes lacunas and lumens. Bigger lacuna can be found in thicker fibers, while lumens have length and width of few millimeters, so their role in wetting with polymers is marginal. In addition, coir fiber cross section, in general, is circular as shown in the micrographs and this result has been confirmed in the literature [40, 41]. The morphology of coir fiber in this work is different as compared with the clarified typical ones which explained previously in the past lines. Amorphous shapes of circular and non-circular voids with numerous numbers of microfibrils can be seen in the obtained micrographs of coir fiber, quite similar morphology of coir fiber to what has been clarified in this work reported by Yan et al. [42]. Geethamma et al. [43] had confirmed the presence of voids over the coir fibers' surface as a result of alkali treatment which led to the removal of the protrusions detected over the untreated coir fiber surfaces. It should be mentioned that the reason behind the alkali treatment for coir fibers is to prevent segregation and floating for fibers in the epoxy, improving the wettability for these natural reinforcement agents with the matrix and guaranteeing the uniform dispersion of the fibers in the polymer [44].

Figure 5a shows the SEM image of pure epoxy as a general view. It can be seen that the image is featureless which reflects the nature of epoxy represented by image taken for a cross section. This result was confirmed by Yao et al. [45]. b and c show the fracture surface of the neat epoxy which reflects the fact of smooth nature of the crack region. The presence of the crack lines gives an indication

Fig. 4 SEM image (a–b) of the morphology of coir fiber cross section

about the low ductility of the epoxy. This outcome was confirmed by Li et al. [46]. The brittleness of the epoxy resin is confirmed with the presence of these obvious crack lines. Furthermore, the crack lines are decreased and started to disappear with the heterogeneous phase of the composite materials (as the coir fibers reinforced the resin). Micrographs d and e show the distribution of coir fibers in the matrix. The uniform dispersion of the fibers in the matrix is obtained. These images show no segregation for the fibers from the matrix which indicates a good wettability between the reinforcing agent and the host matrix. Such good wettability leads to better mechanical properties and this is achieved throughout the impact strength for the imaged sample as the grain size of 250 μm showed the highest impact strength compared to other composite samples of different grain sizes. The fractography of coir fiber epoxy composites shows a rough fracture surface related to the roughness of the coir fibers. The roughness of the fibers is intimately associated with the alkali treatment and that roughness was consequently led to better mechanical interlocking between fibers and the matrix. Micrograph f confirms the interlocking between the matrix and the reinforcing agent and shows the interphase region and adhesion between the fiber and the matrix which interprets the good mechanical properties of impact and flexural strength obtained in the current study. Alkali treatment was also led to uniform dispersion of the fibers in the polymeric matrix [41]. In addition, Prasad et al. 1983 explained that poor wettability leads to the formation of

voids in the structure of the composites. Voids could not be seen in the structure which clarifies the reason behind the good mechanical properties obtained for the different composites. Micrographs g–i show the deformation in the fracture surface of the composite. The images are confirming the occurrence of deformation in the fracture surface of the composite which indicate the fact of the presence of strong adhesion between natural fibers and the matrix which is already shown in image f. Similar interpretation was adopted by Sin and Kumar.[47].

5 Conclusion

The mechanical performance of flexural and impact strength was significantly improved after reinforcing epoxy with chemically treated coir fibers. The grain size played a major role in such an improvement as the obtained results showed better mechanical performance in flexural strength with composites reinforced with a higher grain size. The situation is completely different in case of impact strength as the highest value was reported for the lower grain size of the fibers. SEM images for different fracture surfaces of the polymer and its composites confirmed the homogeneous distribution of the fibers in the polymer as well as the deformation that occurred to the composites and the neat polymer.

Fig. 5 SEM images of: **a** general view of the neat epoxy. **b** and **c** fracture surface of the neat epoxy. **d** and **e** distribution of coir fibers in the matrix. **f** Interphase and adhesion between a coir fiber and the matrix. **g–i** fracture surface of epoxy coir composites

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Compliance with ethical standards

Conflict of interest The author(s) declare that they have no competing interests

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