Tensile Strength and Water Absorption Behavior of Recycled Jute-Epoxy Composites

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ABSTRACT: Recycled natural fibers and biopolymers with sustainable, eco-friendly, and biodegradable properties are receiving increased attention. The moisture absorption and swelling of natural fiber composites adversely influence their mechanical properties and applications. In this research, bio-based epoxy polymers that are reinforced with recycled woven jute fabrics were subjected to water immersion tests in order to study the effect of water absorption on their mechanical and geometrical properties. For comparison, petroleum-based epoxy polymers that are reinforced with new woven jute fabrics were also subjected to the same tests. The effect of fiber percentage on water absorption, thickness swelling, and volume swelling was measured as a function of immersion time. It was observed that water absorption and swelling behavior were higher in bio-based epoxy than in petroleum-based epoxy composites. The stress decreased and the strain increased after water immersion in both composites. However, the rates of change in stress and strain were much more significant for composites made with bioepoxy.

KEYWORDS: Woven jute fabric, bioepoxy, swelling, water absorption, tensile strength, natural fiber composite, biocomposite

1 INTRODUCTION

While synthetic composites have good mechanical properties, there are serious environmental concerns and cost issues with them. For example, the processing temperature of glass and carbon fibers is above 1200°C and glass fibers abrade manufacturing equipment. Moreover, such composites greatly rely on petroleum, which involves consuming nonrenewable energy resources [1]. Growing environmental awareness prompts new research and development on alternative materials in the composites industry. Biocomposites, also called "green composites," are made from bioplastics and natural fibers [2]. Since 1941, when Henry Ford introduced biocomposites from hemp, sisal, and cellulose fibers, much research and development has taken place to advance their properties and expand their applications in aerospace and automotive industries, consumer products, and buildings [3]. Green composites, which are made from plant-derived fibers and crop-derived plastics, are novel materials of the twenty-first century.

Polymers are a class of large molecules composed of many repeated subunits, known as monomers. Epoxy resins, also known as polyepoxides, are a class of reactive prepolymers and polymers which contain epoxide groups. The main raw materials for production of epoxy resin are petroleum-derived materials. In contrast, bioepoxy networks are derived from bio-based materials such as vegetable oils, cardanol, rosin, and sugar [4–7]. Vegetable oils such as linseed oil, soybean oil, castor oil, and pine oil are excellent raw materials for thermosetting biopolymers, as they are inexpensive and abundant [6]. However, because of the low reactivity of aliphatic epoxy groups, any epoxidized oil leads to poorly crosslinked materials with limited thermal and mechanical properties [4].

Natural fibers come from renewable, eco-efficient, and accessible agriculture and forestry feed stock, including wood, wood waste, grasses, flax, jute, hemp, cotton, and sisal. Compared with their synthetic counterparts, natural cellulosic fibers offer primary advantages of: (1) low density, (2) high specific properties, (3) low cost, (4) renewability and biodegradability, and (5) availability [1, 8].

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Cellulosic materials contain hydroxyl groups (-OH), which leads to moisture sensitivity in the resulting composites [9]. This polar group attracts water molecules through hydrogen bonding. Attachment of water molecules to cellulose chain molecules leads to swelling of cell walls, which appears as swelling in plant fibers [10, 11]. As a result, when a cellulosic reinforcing composite is used in a moist environment, the composite absorbs water and swells. The swelling rate depends on temperature and relative humidity. There are three major weaknesses related to applications of cellulosic fibers: (1) The hydrophilic properties of these fibers may lead to poor interfacial adhesion with some hydrophobic matrices. (2) Their swelling tendency could decrease the dimensional stability and mechanical properties of composites. (3) They have low processing and application temperatures due to the degradation of natural fibers at higher temperatures [1].

In general, the moisture diffusion in composites has been shown to be governed by three mechanisms [12–14]. First, water molecules go into micro-gaps between the resin molecular chains. Second, the water molecules are pulled into the interface flaw and gaps between fiber and matrix by capillary force [15]. Finally, water is moved due to capillary transport into micro-cracks in the matrix.

Jute is a type of bast fiber. It can be planted two or three times annually in river flats, depressions, and saline-alkali soils, which are impossible for successful growth of cotton and many other food crops [16]. Jute is the second most common natural fiber (after cotton) cultivated mainly in Bangladesh, Brazil, China, India, and Indonesia [17]. As shown in Table 1, the main chemical constituents of jute fibers are cellulose and hemicellulose. Jute is mainly used for packaging of grain, sugar, coffee, and other food crop, as well as for cement and fertilizers. Jute is a strong fiber, exhibiting brittle fracture, but having a small elongation at break. It has a high initial modulus and small recoverable elasticity [18].

Masoodi and Pillai [10] measured the average diameter of jute fibers when they were exposed to water. It showed that jute fibers swell up to 70% of their initial diameter. When such fibers are placed in composites as reinforcement, the swelling tendency gives rise to an internal pressure inside the composite. The internal stress that comes from the internal pressure may cause micro-cracks in the matrix and failure of interface [10].

Table 1 Typical chemical composition of jute fibers [10].

composition	Percentage (wt %)
α-Cellulose	59–65
Hemicellulose	21–24
Lignin	12–14
Minor Constituents	2

The moisture absorption and swelling of natural fiber composites negatively affect the mechanical properties of composites. Therefore, it is crucial to investigate this behavior and determine how it affects the composite's performance. Such studies provide information about how green composites behave in moist environments. They also enable the exploration of new techniques to overcome this disadvantage in order to promote further development and wider application of natural fiber composites. Several investigations on behavior of composites in moist environments were published during the last two decades. However, nobody has studied the behavior of recycled composites made by bioepoxy and recycled jute fibers. In this experimental research, the behavior of such composites in moist environments has been studied. Both recycled and new jute-epoxy composites were considered in this study to enable comparison.

2 MATERIALS AND PREPARATION

2.1 Materials

In this study, two types of plain woven jute fabrics were used. The first one was new (unused) with an average weight of 323.5 g/m^2 with the number of warp/weft yarns per centimeter of 4/4. The other fabric was used coffee bags with an average weight of 510.5 g/m^2 and number of warp/weft yarn per centimeter of 5/5.

Two different resins were used in this research. One was traditional, petroleum-based epoxy, System 2000 epoxy resin and hardener 2020, produced by Fibre Glast Developments Corporation. The recommended resin-to-hardener ratio was 100:23 by weight [19]. The other type of resin was a bio-based epoxy, Super Sap 100/1000 system, which was supplied by Entropy Resins Inc. As opposed to conventional epoxies that are composed mainly of petroleum-based materials, Super Sap is made from co-products or from waste streams of other industrial processes like wood pulp and biofuel production. The recommended resin-to-hardener ratio was 2:1 by weight [20]. In this paper, the terms 'epoxy' refers to 2000 epoxy resin and hardener, and 'bioepoxy' refers to Super Sap 100/1000 bio-based epoxy resin and hardener, 'jute' refers to new jute fabrics and 'recycled jute' refers to coffee sack fabrics.

2.2 Making Composite Specimens

The hand lay-up method with an aluminum open mold of size 162 mm×135 mm×4 mm was used to make the test samples. First, one layer of mold release was coated on the surfaces of the mold cavity and allowed to dry at ambient conditions. Then the hardener was added to the resin, with appropriate ratio, and the mixture was stirred uniformly for two minutes by hand. Then, the mixture of resin and curing agent was poured into the mold, which could just cover the bottom part of the mold. Then, one or two-layer fabric was placed in the mold and the remainder of the resin/hardener mixture was poured in the cavity until the mold was completely filled.

Six sets of composites with different components and fabric layers were made. They were: (1) pure epoxy, (2) pure bioepoxy, (3) epoxy reinforced with one layer of new jute fabric, (4) epoxy reinforced with two layers of new jute fabric, (5) bioepoxy reinforced with one layer of recycled jute fabric, and (6) bioepoxy reinforced with two layers of recycled jute fabric. Table 2 shows mass percentage of fiber in composite sheets with different fabric layers. The composite sheets were cut to the standard size according to ASTM D 638 [21]. For the swelling and water absorption tests, five specimens from each of six different sets of composite sheets were used after sealing the cutting edges by resin.

3 MEASUREMENTS

3.1 Water Absorption

The initial weight of every specimen was measured and recorded. Then, the specimens were submerged in water and were taken out after certain times to measure the weight after excess water on their surfaces was wiped off. The water absorption percentage was estimated by using the following equation [22]:

$$W_{re}(t) = 100 \times \left(\frac{W_t - W_0}{W_0} \right) \quad (1)$$

Table 2 Mass percentage of fabric in each composite sample.

Number of fabric layers	Jute/epoxy	Recycled jute/ bioepoxy
0	0%	0%
1	8%	12%
2	17.4%	21%

where W_{re} is the relative weight change or water absorption percentage, W_t is the weight at the time t, and W_0 is the initial weight at $t=0$, and t is the soaking time.

3.2 Thickness Swelling

The initial thickness of every specimen was measured and recorded before experiments. The thickness at the upper, middle, and lower parts of each specimen was measured with a digital caliper and the average was recorded as the thickness of the specimen. Then, specimens were submerged in water and were taken out after certain times for future measurements. The percentage of thickness swelling was estimated by [22]

$$T_{re}(t) = 100 \times \left(\frac{T_t - T_0}{T_0} \right) \quad (2)$$

where T_{re} is the percentage of thickness swelling, T_t is the thickness at time t, and T_0 is the initial thickness at $t=0$.

3.3 Volume Swelling

To determine the volume of the specimen, a graduated cylinder filled with water to a known level was used and the specimen was submerged in the water. The change of the water volume inside the graduated cylinder was measured and recorded as the volume of the specimen. And the volume swelling was calculated using following expression [22]:

$$V_{re}(t) = 100 \times \left(\frac{V_t - V_0}{V_0} \right) \quad (3)$$

where V_{re} is the percentage of volume swelling, V_t is the volume at time t, and V_0 is the initial volume at $t=0$.

3.4 Tensile Strength

The tensile tests were performed according to ASTM D 638 [21]. Three standard specimens for each type of composite were used in tensile tests. The experiments were done on a set of samples before water absorption tests and another set of samples after water absorption tests, when the swelling rate reached a steady state. All the specimens were tested at standard testing condition of 20°C and 65% relative humidity. The tensile tests were performed using an Instron 5500R with self-aligning grips. A crosshead speed of 5 mm/min was chosen for all tests. As it was expected, all the specimens broke in the middle of the specimens.

4 RESULTS AND DISCUSSION

4.1 Water Absorption

Five samples from each composite were used, and the average of measured results were used for analysis. Figure 1 shows the water absorption percentage as a function of time. All specimens showed a high rate of water absorption at the beginning of the test, and the rate decreased asymptotically with time and finally reached a steady state. Even pure resin absorbed water, with maximum absorption percentages of 2.03% and 7.66% for pure epoxy and bioepoxy, respectively.

The main raw materials for production of bioepoxy resin, including the Super Sap 100/1000 system that was used in this research, are vegetable oils [20]. Natural plant oils are predominantly made up of triglyceride molecules, which contain aliphatic chains [4]. Because of the low reactivity of aliphatic groups, using triglyceride-based materials leads to poorly crosslinked polymers [4]. Water uptake happens due to the capability of the water molecules to penetrate through the epoxy structure [23, 24]. Since bioepoxy has weaker crosslinked structure, more water molecules can penetrate into the micro-cavities of its network structure. Water uptake of cured thermosets is also attributed to the presence of hydrophilic functional groups, such as hydroxyl or amine, in the network structure [23, 24]. Furthermore, when epoxies are cured using amine-based curing agents, each epoxide ring opened by the amine group creates a hydroxyl group. Most bioepoxy resins have higher hydroxyl concentration than epoxy, which leads to higher water absorption [4]. The water molecules might interact with epoxy molecules by forming hydrogen bonding with hydrophilic groups [10]. In general, the water absorbed by cured polymer can be in two forms: (1) free water which fills the micro-cavities of the network structure, and (2) water molecules that are bonded with polar groups of polymer [24].

Water absorption increased with the increasing fiber content. For the composites made of jute fabric and traditional epoxy, maximum water absorption rate was 5.83% for composite with two-layer fabrics and 3.42% for the composite with one-layer fabric. The water absorption of composites made of recycled jute fabric and bioepoxy followed the same trend. The maximum rate was 13.12% for two-layer fabrics and 10.42% for one-layer fabric reinforced recycled composites. This may be due to the fact that jute fibers absorb more water as compared to the resin [10]. The higher percentage of jute fibers in the composite implies higher percentage of cellulose molecules, which leads to higher water absorption and swelling.

Another important phenomenon is that absorption rate of composite with less fiber loading reached a steady level earlier in time. The pure epoxy reached a steady state after 100 hours. The time for jute/epoxy with one-layer fabric was about 200 hours, and for two-layer composite was 350 hours. Likewise, in recycled jute/bioepoxy, the absorption rate reached a steady level after about 350 hours for one-layer composite and 410 hours for two-layer. This is because less jute fiber means fewer cellulose molecules, which in turn leads to fewer polar hydroxyl groups, which attract water molecules. Moreover, due to the

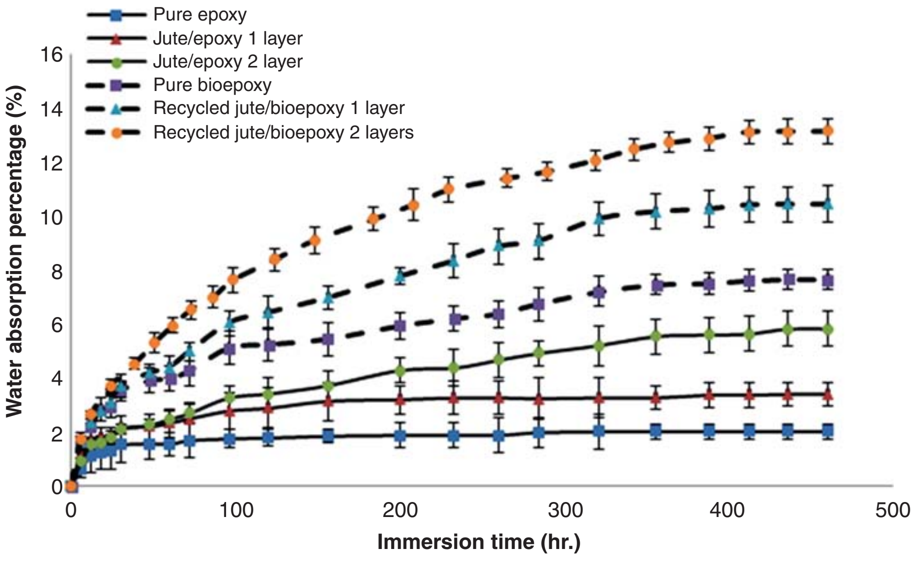

Figure 1 Water absorption as a function of time for the different composites. The scatter bars show 95% confidence intervals [26].

existence of voids, which were caused by weak bonds between the hydrophobic matrix and hydrophilic fibers, water probably filled these voids by capillary action [25].

Figure 2 shows the thickness swelling of six specimens as a function of immersion time. The measured thickness swelling for pure epoxy and pure bio-based epoxy specimens were about 3.67% and 8.26%, respectively. It also shows that adding one layer of jute fabric resulted in 4.66% and 10.45% increases in thickness swelling for epoxy and bio-based epoxy composites, respectively. For composite with two-layer jute fabrics, 6.73% and 12.22% swelling rates were observed for epoxy and bio-based epoxy composites, respectively. Same as with water absorption, the thickness swelling increased with the increasing fiber content.

The change of volume swelling, as shown in Figure 3, follows a trend similar to Figures 1 and 2. The volume swelling for pure epoxy and bio-based epoxy were 3.67% and 7.76%, respectively, after 460 hours. The maximum volume swelling for composites reinforced with one-layer fabric was about 4.16% and 9.95% for epoxy and bio-based epoxy, respectively. At the same time, the increasing of fiber loading to two layers led to 6.23% and 11.31% volume swelling for the aforementioned epoxy matrices. Dimensional variation, which is the result of volume swelling, limits the applications of green composites in moist environments.

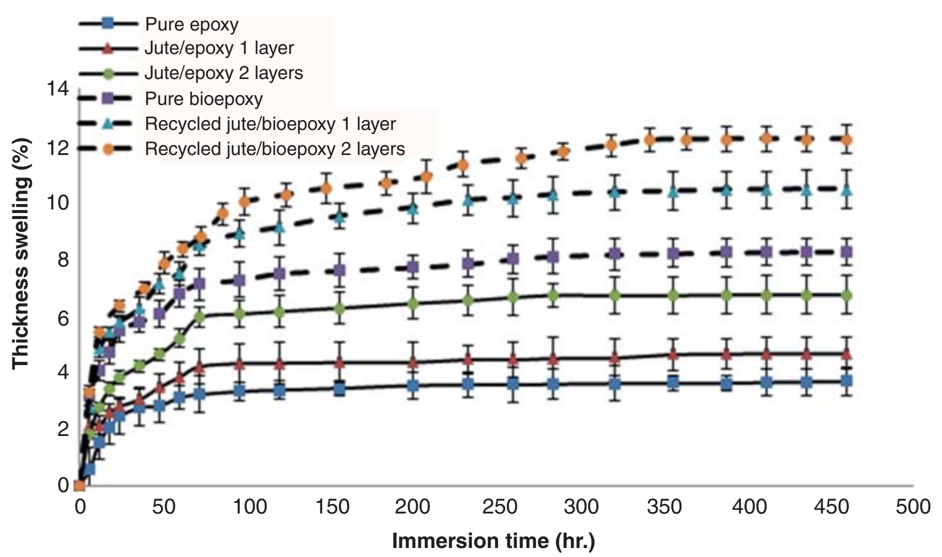

Figure 2 Thickness swelling as a function of time for the different composites.

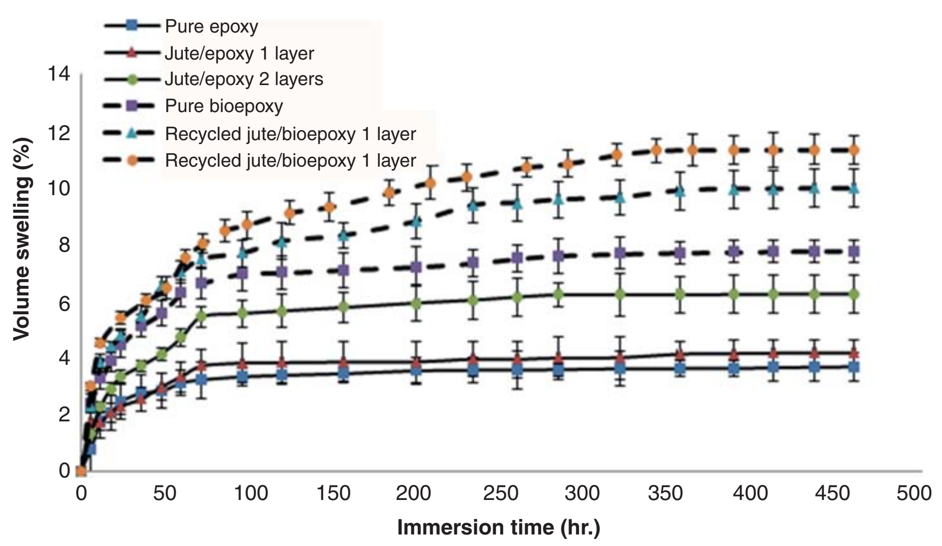

Figure 3 Volume swelling as a function of time for the different composites.

The changes observed in composites with bioepoxy were all greater than those with traditional epoxy. Even the swelling of pure bioepoxy was greater than the epoxy composite with two-layer jute fabrics. That suggests that bio-based epoxy as a matrix plays a crucial role in the water absorption. In the water immersion process, the water molecules come into contact with the matrix first, and they are attracted by bio-based epoxy due to its poor crosslinked structure and presence of hydrophilic functional groups. This explains why the composites with bio-based epoxy have a higher absorption and swelling rate. Moreover, water molecules penetrate into inner phase and get absorbed by jute fibers. On the other hand, jute fibers swell up to 70%, which is much more than the swelling in pure epoxy or bioepoxy; this swelling tendency of jute fibers imposes an internal stress into the matrix and may cause decreased interfacial forces or even cause micro-cracks.

4.2 Mechanical Properties

Figure 4 shows the measured stress-strain curves of jute/epoxy composites before and after immersion. Note that here after immersion means placing them in water for enough time to reach a steady-state swelling percentage. Figures 5 and 6 show the average values of tensile stress and strain at maximum load, respectively. They show that the stress decreases and the strain increases after water immersion. The same trends for stress and strain were observed in recycled jute/bioepoxy composites (Figures 7–9). The internal stress from swelling of fibers may cause micro-cracks in the matrix and failure of the interface, which then results in loss of strength. Water molecules most likely also acted as plasticizer and increased the flexibility of these materials; therefore, the strain increased after the composites were immersed in water [12].

For both jute/epoxy and recycled/ bioepoxy groups, the tensile strength of pure resin was higher than the composite reinforced with fabric. However, the strength of composites reinforced with two-layer fabrics was higher than the composites reinforced with one layer. This phenomenon involves the nature of resin and jute fibers. One difficulty that limits application of natural fiber-reinforced composites is the lack of good adhesion to most polymeric matrices. The hydrophilic nature of natural fibers adversely affects compatibility to hydrophobic matrices, and it may even reduce the strength. On the other hand, the open mold method used in making the composites in this study may have also introduced some air bubbles into the matrix. So, weak interface adhesion and voids existing in the matrix could have led to poor stress transfer and ultimately decreased strength for the composites reinforced with fabrics. However, with the increasing of fiber content, the strength of composites reinforced with two-layer fabrics increased compared with those reinforced with one-layer fabric; this indicates that strength contribution from the fibers exceeds the above-mentioned weaknesses in these composites. It can be predicted that the strength could keep increasing with increasing fiber content.

To study the effect of swelling on mechanical properties of considered composites, their mechanical properties were measured before and after water absorption/swelling tests. Here, 'after swelling' means when swelling reached a steady state. For bioepoxy composite with one layer of recycled jute fabric, the stress decreased 66.3% and strain increased 77.2%

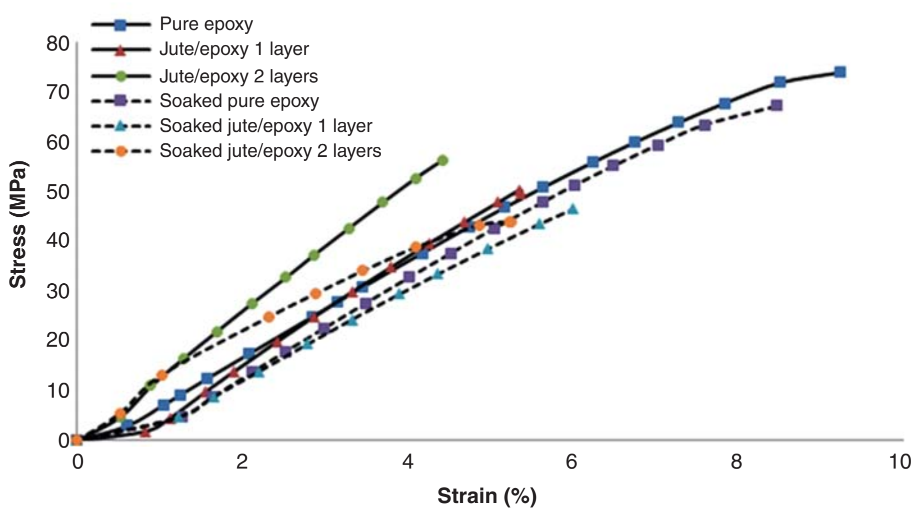

Figure 4 Strain-stress of jute/epoxy composites with different fiber loading before/after swelling.

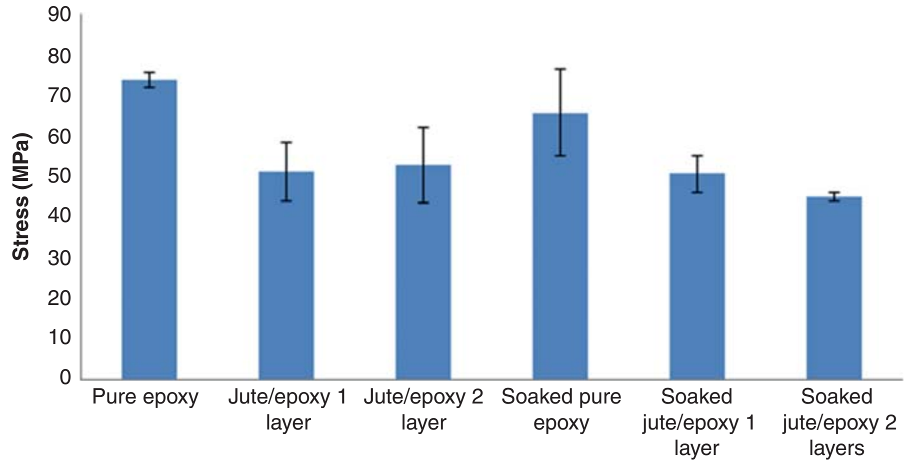

Figure 5 Maximum stress of jute/epoxy composites with different fiber loading before/after swelling.

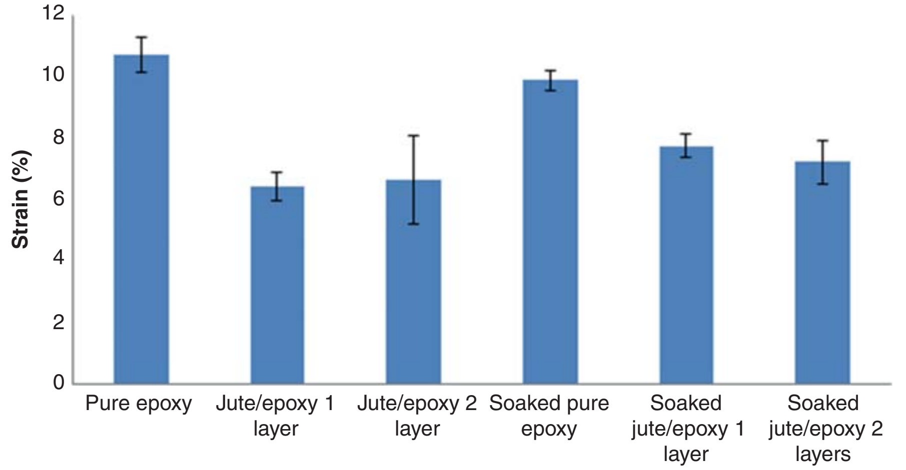

Figure 6 Maximum strain of jute/epoxy composite with different fiber loading before/after swelling.

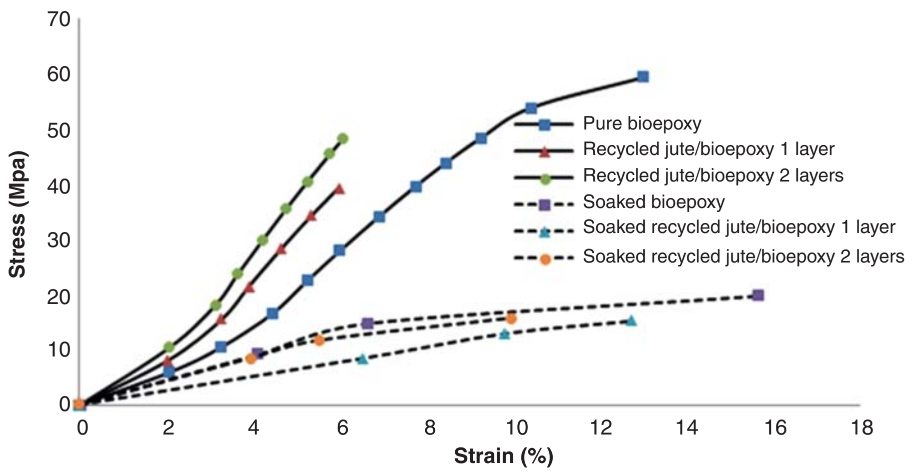

Figure 7 Strain-stress of recycled jute/bioepoxy composites with different fiber loading before/after swelling.

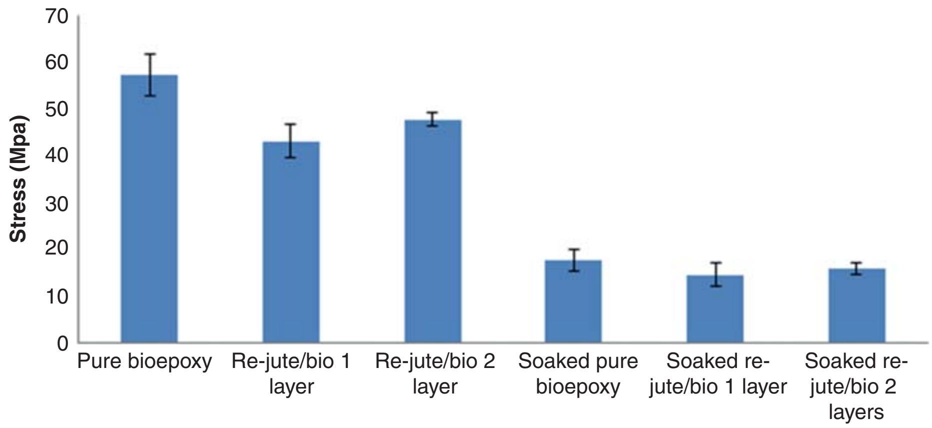

Figure 8 Maximum stress of recycled jute/bioepoxy composites with different fiber loading before/after swelling.

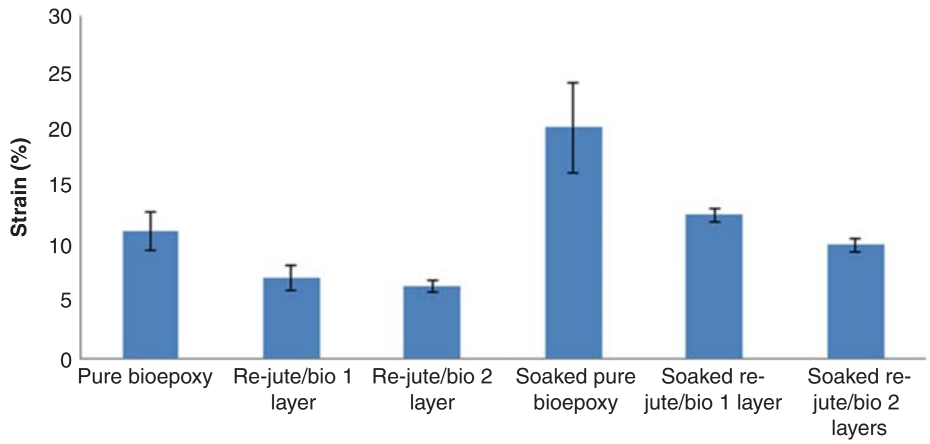

Figure 9 Maximum strain of recycled jute/bioepoxy composites with different fiber loading before/after swelling.

after swelling; for composites with two-layer fabrics, the 66.9% reduction in stress and 56.6% rise in strain were measured. For jute/epoxy composites with one-layer fabric, the maximum rise in stress reduction strain was 1.2% and 20.3%, after swelling. For the same composites, but with two-layer fabrics, the maximum stress reduction strain increases were 14.8% and 8.7%, respectively

When the water absorption reached equilibrium, the bio-based epoxy composites absorbed much more water and had more thickness and volume swelling compared to traditional epoxy composites. Since both bioepoxy and jute fibers absorb water, jute-bioepoxy composites absorb more water than jute-epoxy. Therefore, larger changes in dimensions were observed and the change of tensile strength was greater as well.

One interesting phenomenon is that after about 460 hours of water immersion, all of the specimens of recycled jute/bioepoxy composites became very flexible and bendable; however, specimens of jute/epoxy composite were still stiff. In recycled jute/bioepoxy composite, the water penetrates both fabric and matrix, so the composite becomes flexible. After swelling tests, the effect of increasing fiber content on stress and strain was decreased.

5 SUMMARY AND CONCLUSION

Six different sets of composite specimens using two types of jute fabrics (new and recycled) and two types of epoxy (traditional and bio-based) with different percentage of fibers were made. The water absorption and swelling tests were carried out to study what the water absorption rate was and how it affected the physical and geometrical characteristics of the specimen. Tensile strength tests were also performed and data were studied and compared between specimens before and after water absorption/swelling tests.

All specimens showed a high rate of water absorption and swelling at the beginning of the test, but the rate was reduced asymptotically with time until it reached a steady level. The specimens made by pure traditional resin exhibited water absorption and swelling; this phenomenon was more significant in the bio-based epoxy. Rate of water absorption and swelling increased with the higher percentage of jute fibers in the composite specimens. Both water absorption and swelling data were higher for the bio-based epoxy resin composite than the traditional epoxy resin; even pure bio-based epoxy had higher water absorption rate than the epoxy reinforced with two-layer jute fabrics.

The loss of tensile strength indicated that the hydrophilic nature of natural fibers adversely affects compatibility with hydrophobic matrices. The stress of pure resin was higher than those of specimens reinforced with fabrics. However, stress increased and strain decreased with the increase in fiber content. After swelling tests, the stress decreased while strain increased compared to the values prior to the swelling tests. The internal stress caused by swelling of fibers may cause micro-cracks in the matrix and it may lead to the failure of the interface adhesion. The rates of change in stress and strain were much more significant for bioepoxy specimens compared to the epoxy specimen, as the former absorbs more water. It was observed that the effect of increasing fiber content on stress and strain were higher in dry composites than swelled composites. The water absorption decreased strength of the composites; however, this negative effect was much greater in recycled jute-bioepoxy composites than regular jute-epoxy composites.

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