

Effect of Water Absorption on the Mechanical Properties of Alkaline Treated Bamboo and Flax Fiber Reinforced Epoxy Composites

Ravikantha Prabhu^{1,*}, Sharun Mendonca¹,
Rudolf D'Souza¹ and Thirumaleshwara Bhat²

¹Department of Mechanical Engineering, St Joseph Engineering College, Mangaluru 575028, India

²Department of Mechanical Engineering, Shri Madhwa Vadiraja Institute of Technology and Management, Udupi 574115, India

(*Corresponding author's e-mail: prabhuravikantha@gmail.com)

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Abstract

Untreated and alkaline treated bamboo and flax fiber reinforced epoxy composites are processed using a hand layup process. The effect of alkaline treatment on the mechanical properties of the composites has been analysed. Alkaline treatment of the fiber has enhanced the mechanical properties of the developed composites. Composite reinforced with 5 % NaOH treated fiber show better performance when compared with untreated fiber reinforced composites. Alkaline treatment of the bamboo and flax fiber with 5 % NaOH has improved the hardness by 3.57 and 2.43 %, tensile strength by 47 and 20.72 % and flexural strength by 7.36 and 13.85 % in bamboo and flax fiber reinforced composites, respectively. The increase in the percentage NaOH in the alkaline treatment of the fibers resulted in weakening of fiber resulting in a drop in the properties of the developed composites. Water absorption tests of the developed composites were conducted as per ASTM D570 by immersion in distilled water at room temperature. The influence of water absorption on mechanical properties of developed composites is also examined. The quantity of water absorption and diffusion coefficient are reduced with alkaline treatment of fiber. Mechanical properties of the composite were found to decrease by the water absorption, which can be controlled by alkaline treatment of fiber and thereby reducing water absorption rate and improve the mechanical properties of the composites.

Keywords: Bamboo fiber, Flax fiber, Epoxy resin, Alkaline treatment, Water absorption, Mechanical properties

Introduction

Natural plant fibers such as bamboo and flax are emerging as the promising and environmentally friendly alternative for synthetic fibers for the following reasons. The production of fiber consumes less non-renewable energy resources compare to synthetic fiber production and thus reduces environmental pollution. To get the equivalent performance of the composite the volume and weight of the natural fiber required are more compared to synthetic fiber which in turn reduces the volume and weight of the polymer matrix required in the production of the composite, which reduces the energy usage in the production of the polymer. The density of the natural fiber is less than manmade fibers which enhances the fuel efficiency and minimizes emission in the application phase (Example: Automobile application). The use of natural fiber reinforced composites made of lower mass of base polymer matrix compared to synthetic fiber reinforced composites results in reduced carbon emissions when the composite is incinerated. natural fiber reinforced composites offers great environmental benefits such as a decrease in dependence on non-renewable material/energy sources, lesser emissions of a pollutant, lesser emissions of greenhouse gas, improved energy recovery and a satisfactory biodegradability of the components at the end of life.

Reinforcing natural fiber in polymer resin is highly beneficial because it helps to improves the strength and toughness of the polymer. However, high moisture absorption rate and lack of interfacial adhesion between the polymer and natural fiber made natural fiber reinforced composites less attractive for the same applications [1]. This problem can be overcome by surface treatment of the fiber. Modification of the fiber surface can be done by a physical method or a chemical method [2,3]. The

physical method of surface treatment of the fiber involves stretching of fiber, thermal treatment of fiber [4], electric discharging of fibers and calendaring of fibers [5] to name a few. These techniques modify the surface properties and improve the surface bonding of the fiber with a polymer matrix to a certain extent.

The chemical treatment of the fiber surface facilitates efficient bonding between polymer matrix and fiber surface thereby improves the mechanical and thermal properties of the developed composite [6]. Chemical treatment of the natural fiber is done using alkali [7], silane [8], benzylation [9], steric acid [10], permanganate [11] and maleated coupling agents [12]. Alkaline treatment of the fiber increases the fiber surface roughness, which results in improved mechanical interlock between fiber and matrix [13]. Silane coupling agent used in the silane treatment of the natural fiber penetrates the pores on the surface of the fiber and mechanically interlocked coating is developed on the surface [14]. Similarly, peroxide treatment [15], benzylation (using benzoyl chloride) [15] and acetylation (using acetic acid and acetic anhydride) [16] improve the surface quality of the fiber and the bonding between polymer matrix and fiber. Chemical treatment of natural fiber using an aqueous alkaline solution is the most commonly used method to modify the fibers when used with polymer resins. Alkaline treatment using aqueous NaOH solution promotes ionization of the hydroxy group in the natural fiber to alkoxide Eq. (1) [17].



The compatibility of the epoxy matrix with the fiber improves with the increase in the hydrogen groups in the fiber. Thus, alkaline treatment of the natural fiber affects the degree of polymerization, cellulosic fibril and the extraction of lignin and hemicellulosic compound directly [18].

Alkaline treatment of the bamboo fiber results in the removal of amorphous weak hemicellulose components from the surface of the fibers responsible for enhancing the flexural strength of the bamboo fiber reinforced hybrid composite [19]. Alkaline treatment of the flax fiber improved longitudinal property (both strength and modulus) by 40 % or more [20]. Alkaline treatment of jute with 5 % NaOH solution for a given period improves crystallinity in jute fiber, alkaline treatment improves modulus by 12, 68 and 79 % after 4, 6 and 8 h of treatment, for 35 % fiber reinforced composite. For the same composite, with 4 h of treatment flexural strength improved by 20 % [21]. A similar chemical treatment has been tried for the treatment of flax fiber [22]. Polyester composite reinforced with 5 % NaOH sisal fiber has better tensile strength than the 10 % NaOH treated composites. After a certain percentage of alkali in the chemical treatment of fiber, the tensile properties of the composite drastically decrease [23]. Alkaline treatment of raffia palm fiber enhanced the microhardness and extension at the break at all fiber loads, compared to untreated fiber composite [24]. Alkaline treated in a 5 % concentration jute fiber reinforced vinyl ester composite has defect concentrations of 18.5 and 28.5 % when fibers are soaked for 4 and 8h respectively compared to untreated fiber with 30 % defect [25]. Hot alkaline treatment flax fiber using NaOH solution at 160 °C, increased bending strength by 28.8 % and tensile strength by 8.6 % [26]. Hildebrand solubility parameter (HISP) increases with pressure and relation with temperature is linear [27]. Soaking of bamboo fibers for duration of 48 h in 10 % NaOH concentration solution produce a composite with the highest ultimate tensile and modulus [28].

The present work mainly focused on finding the effect of alkaline treatment of bamboo and flax fibers on the water absorption properties of composites and effect of water absorption on the mechanical properties of developed composites.

Materials and methods

Materials

In the present research work, epoxy resin Araldite LY556 (Reaction product: bisphenol A-(epichlorhydrin)) and hardener Aradur HY951(Low viscosity aliphatic amine) were used for the fabrication of the composites (supplied by Herenba Instruments and Engineering, Chennai, India). Epoxy resin systems are the largely used matrix material for low- temperature application (usually below 200 °F (93 °C)) because of their good chemical resistance, superior property of adhesion to the fibers and dimensional stability.

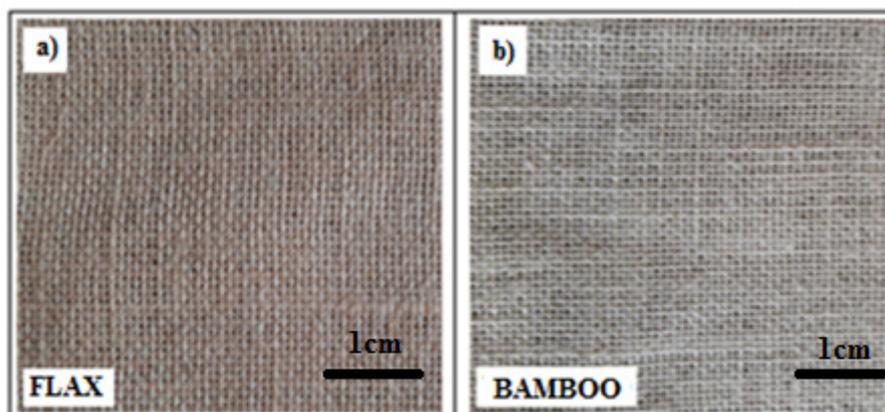


Figure 1 Digital image of (a) flax fiber mat (b) bamboo fiber mat.

In this study, bidirectional ($0^\circ / 90^\circ$) roving flax fiber mat (**Figure 1(a)**) and bamboo fiber mat (**Figure 1(b)**) have been used as the reinforcing material (supplied by Go Green Products Ltd., Chennai, India). The average thickness of each bamboo mat is about 1.0 mm. Properties of the bamboo and flax fibers used in the present work are listed in **Table 1**. Initially, the bamboo fiber mat was cut into the dimension of $300 \times 300 \text{ mm}^2$. The mat is immersed in alkaline solution (5 and 10 % NaOH) for 30 min, followed by rinsing with fresh water and then with distilled water, to take away remaining traces of NaOH from the surface of the fiber mat. Finally, the fiber mat was dried at room temperature for 48 h. The mats are preheated in an oven at 80°C for 1 h before composite preparation to eliminate moisture content present.

Table 1 Properties of bamboo and flax fibers.

Fiber	Property	
Bamboo	Density (g/cc)	1.12
	Fiber diameter (mm)	0.6 - 1.0
	Tensile strength (MPa)	180
	Youngs Modulus (GPa)	14.2
	Elongation at the breakage (%)	14
	Break force (N)	6.1
Flax	Density (g/cc)	1.52
	Fiber diameter (mm)	0.8 - 1.2
	Tensile strength (MPa)	348 - 580
	Youngs Modulus (GPa)	19.8
	Elongation at the breakage (%)	2.9
	Break force (N)	8.2

Fabrication of composites

Fabrication of the composite is done using a simple hand layup process followed by light compression. Mild steel mold plates of dimension $300 \times 300 \text{ mm}^2$ are used for the fabrication of the composites. The fabrication process begins with cleaning the metal mold plates by removing any sort of dirt or grease present on the surface. Polyethylene sheets are placed on the metal plates and a thin layer of wilon wax is uniformly applied on the surface of the sheet for easy removal of the composite plate. To the epoxy resin weighed quantity of hardener (in the ratio of 1:10) was added with constant stirring. Fiber mats of dimension $300 \times 300 \text{ mm}^2$ are used. Epoxy resin is uniformly applied on the surface of the fiber mat using the brush. For uniform spreading of resin, a hand roller was used. The application of laminate

layers is continued until the required thickness of the composite is achieved. The digital image of 5 % NaOH treated epoxy/flax and epoxy/bamboo composite plates are presented in **Figures 2(a)** and **2(b)**, respectively. Material designation for the developed composite materials is given in **Table 2**.

Table 2 Material designation for the developed composite material.

Designation	Composition
UB	Epoxy+ Bamboo (Untreated)
TB5 %	Epoxy+ Bamboo (Treated with 5 % NaOH)
TB10 %	Epoxy+ Bamboo (Treated with 10 % NaOH)
TB15 %	Epoxy+ Bamboo (Treated with 15 % NaOH)
UF	Epoxy+ Flax (Untreated)
TF5 %	Epoxy+ Flax (Treated with 5 % NaOH)
TF10 %	Epoxy+ Flax (Treated with 10 % NaOH)
TF15 %	Epoxy+ Flax (Treated with 15 % NaOH)

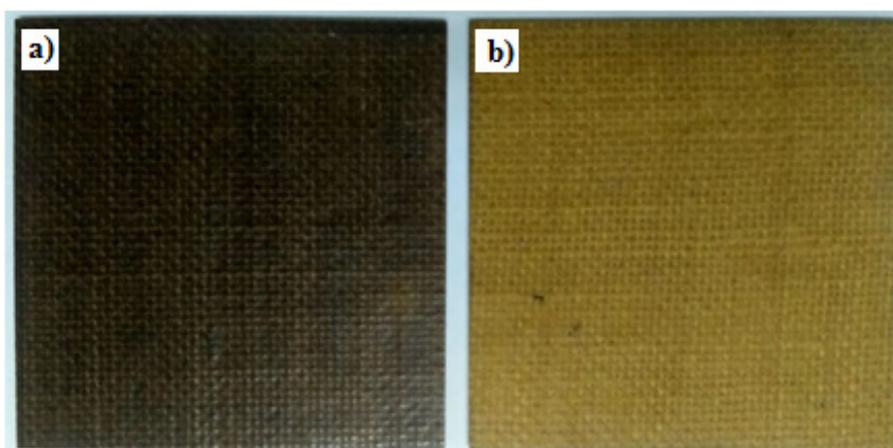


Figure 2 Digital image of 5 % NaOH treated (a) epoxy/flax composite (b) epoxy/bamboo composite.

Characterizations of composite materials

Water absorption test

Water absorption tests of the developed composites were conducted as per ASTM D570 by immersion in distilled water at room temperature for a long time period to reach a stable equilibrium or saturation point. The samples were taken out after each time interval and water particles are wiped out from the surface of the specimen using filter paper. Samples are weighed immediately within 30 s to avoid error due to evaporation, using an electronic balance (AY220 type, ± 0.1 mg accuracy). After weighing, the sample is immersed in the water bath to permit water absorption until the saturation point is reached after 135 days. The weight difference of the specimen gives the value of the water absorption. The water content percentage (M_t), is calculated using Eq (2)

$$M_t(\%) = \frac{W_t - W_0}{W_0} \times 100 \quad (2)$$

where W_0 - initial weight of the test sample, W_t - weight of the sample at time 't'.

The water absorption behavior of the sample kept in the water bath can be studied as Fickian behavior. Therefore, Eq. (3) has been used [29,30].

$$\frac{M_t}{M_\infty} = 4 \left(\frac{Dt}{\pi h^2} \right)^{\frac{1}{2}} \quad (3)$$

where D - diffusion coefficient, h - sample thickness, M_t - water content at time t and M_∞ - equilibrium water content.

Mechanical test

The hardness value of the developed composite was measured as per ASTM D785-08 (Reapproved 2015) standard using Rockwell's hardness tester. Tensile tests were conducted using a JJ LLOYD universal testing machine (UTM) of 1 - 20 kN capacity. The tests were conducted as per ASTM D3039-08 standard. Each time the flat specimen of size $150 \times 12.5 \times 3 \text{ mm}^3$ was subjected to uniaxial load at both ends. For fixing each of the properties, 5 specimens of each type were tested. In each of the cases, the gauge length was kept as 50 mm and the crosshead speed was maintained at 2 mm/min. The flexural properties were tested using the same machine with special three-point bending attachments. Testing was conducted as per ASTM D790-10 standard. The bending test was performed on a flat specimen of size $90 \times 12.5 \times 3 \text{ mm}^3$ at a crosshead speed of 2 mm/min considering a beam span of 50 mm. Izod impact testing was carried out on an unnotched specimen of dimension $60 \times 12.5 \times 3 \text{ mm}^3$ using a pendulum type impact testing machine (Ceast make) as per ASTM D256-10 standard.

Result and discussion

Water absorption

The percentage of weight gain due to water absorption as a function of the square root of time for, untreated, 5 % NaOH, 10 % NaOH and 15 % NaOH treated bamboo and flax fiber reinforced composite due to immersion in distilled water for 135 days at room temperature is shown in **Figure 3**.

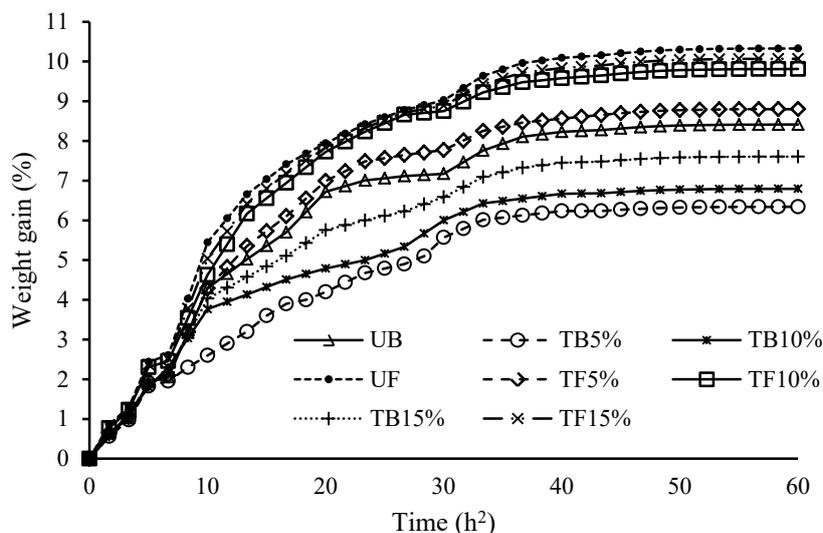


Figure 3 Water absorption behavior.

It has been observed that water absorption is minimum for 5 % NaOH treated fiber reinforced epoxy composite. Water absorption rate increases rapidly initially till it reaches saturation state and thereafter there will not be much increase in the rate of water absorption, following a Fick-ian diffusion process. The hydrophobic nature of the fibers and greater fiber and matrix interfacial area increases water absorption by the composite [31]. The maximum water absorption at saturation point and the diffusivity values are given in **Table 3**.

Table 3 Maximum water absorption and diffusivity of bamboo and flax fiber composite.

Material composition	Water absorption at saturation point, M_{∞} (%)	Diffusivity, $D \times 10^{-6}$ (mm ² /s)
UB	8.4	2.6947
TB5 %	6.4	1.8798
TB10 %	6.8	2.1749
TB15 %	7.4	2.0456
UF	10.4	2.7652
TF5 %	8.8	2.6478
TF10 %	9.8	2.7619
TF15 %	10.2	2.7864

From **Table 3** it can be noted that water uptake and the diffusion coefficient decrease with the alkaline treatment of the fiber. Composite with bamboo and flax fiber treated with 5 % NaOH exhibits very low diffusion of water compared with untreated, 10 % and 15 % NaOH treated sample.

Effect of water absorption on mechanical properties

The impact of water ingestion on the mechanical properties of untreated and alkaline treated epoxy/bamboo and epoxy/flax composites was examined after placing specimens in distilled water at room temperature for 135 days and properties of these were compared with samples of the same composite kept in dry condition.

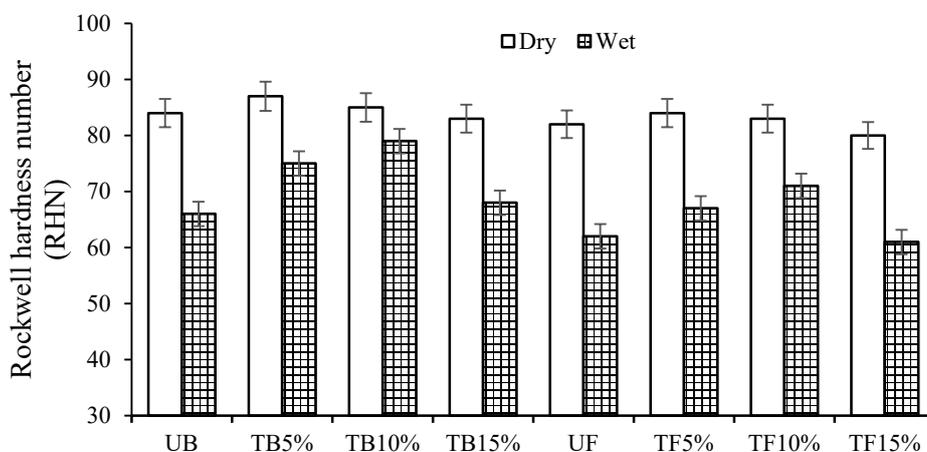
**Figure 4** Rockwell hardness of dry and wet samples.

Figure 4 represents the impact of water absorption on the hardness values. In all developed bamboo and flax reinforced composite samples, hardness is found to be reduced. This may be due to the weakening of interface bonding between the fiber and matrix due to water absorption [32]. In the hardness test of flax fiber composite, indentation depth is more for water-immersed specimens compared to the dry sample. In untreated bamboo and flax fiber reinforced composite, hardness decreased by 21.42 and 24.39 %. In 5 % NaOH treated fiber reinforced composite hardness decreased by 13.79 and 20.23 %. This improvement in hardness is due to a decrease in water absorption with the alkaline treatment of the fibers.

Table 4 Tensile properties of dry and wet samples.

Material composition	Tensile strength (MPa)		Tensile modulus (GPa)		Elongation at break (%)	
	Dry	Wet	Dry	Wet	Dry	Wet
UB	46.8±0.6	38.32±0.5	2.75±0.02	2.26±0.03	5.42±0.05	5.06±0.04
TB5 %	70.2±0.7	62.76±0.8	3.07±0.03	2.84±0.05	5.56±0.06	5.24±0.06
TB10 %	68.8±0.5	58.92±0.6	3.01±0.04	2.89±0.06	5.48±0.08	5.16±0.05
TB15 %	62.1±0.4	53.46±0.5	2.96±0.03	2.72±0.04	5.43±0.07	5.10±0.04
UF	108.1±1.0	91.25±1.5	4.76±0.06	3.82±0.05	6.26±0.09	5.56±0.07
TF5 %	130.5±1.5	112.95±2	5.82±0.05	5.21±0.07	6.89±0.07	6.16±0.05
TF10 %	127.9±2.0	108.25±1.6	5.71±0.08	5.39±0.04	6.62±0.04	5.92±0.08
TF15 %	112.8±1.5	102.4±1.2	5.62±0.06	4.86±0.03	6.42±0.05	5.86±0.06

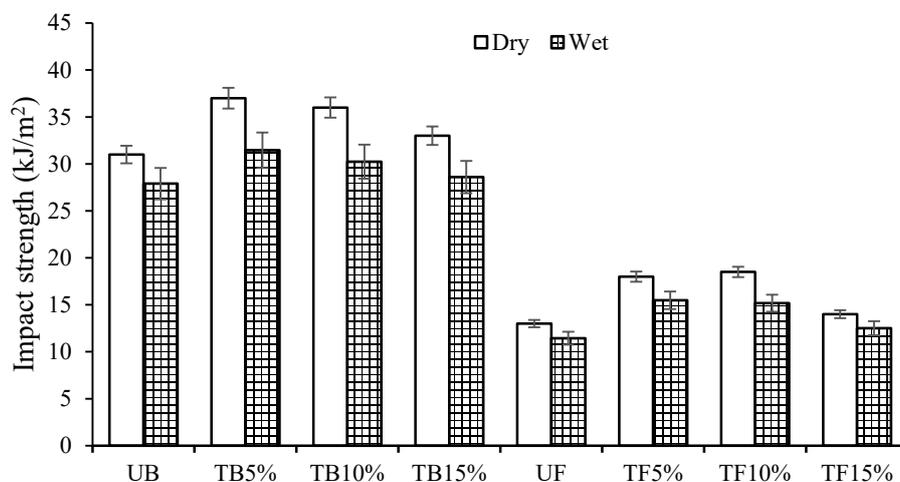
The tensile properties of untreated and alkaline treated samples under dry and wet conditions are presented in **Table 4**. Tensile properties of the bamboo and flax fiber reinforced composite increases with alkaline treatment of the fiber. The composite reinforced with 5 % NaOH treated bamboo and flax fibers given best tensile properties and further increase in the NaOH% resulted in weakening of the fiber resulting in decrease in the properties of the composite. Similar results are obtained in case of Polyester composite reinforced sisal fiber [23]. The tensile properties of the developed composites have been decreased with water aging for both untreated and treated conditions. In untreated bamboo and flax fiber reinforced composite, tensile strength decreased by 18.12 and 15.58 %, respectively and in 5 % NaOH treated fiber reinforced composite tensile strength decreased by 10.59 % and 13.45 %, respectively. In untreated bamboo and flax fiber reinforced composite, tensile modulus decreased by 17.81 and 19.74 % respectively and in 10 % NaOH treated fiber reinforced composites tensile modulus decreased by 3.98 and 5.61 %, respectively. In untreated bamboo and flax fiber reinforced composite, percentage elongation at breakage decreased by 6.64 and 11.18 %, respectively and in 5 % NaOH treated fiber reinforced composite percentage elongation at breakage decreased by 5.75 and 10.21 %, respectively. This improvement in tensile strength is due to a decrease in water absorption with the alkaline treatment of the bamboo and flax fiber.

Table 5 presents the effect of alkaline treatment on the flexural properties of dry and wet epoxy/bamboo and epoxy/flax composites. The composite reinforced with 5 % NaOH treated bamboo and flax fibers given best flexural properties and further increase in the NaOH% resulted in weakening of the fiber resulting in decrease in the properties of the composite. From the **Table 5**, it can be noted that soaking the test sample in distilled water has reduced the flexural strength of the untreated bamboo and flax composite by 20.08 and 16.07 %, respectively. With alkaline treatment of fiber with 5% NaOH, the flexural strength has been affected by 9.85 and 9.77 %, respectively. The flexural strength of the fiber is affected by the water absorption and alkaline treatment of fiber has reduced water absorption rate and helped to retain the flexural strength of the composite. Similarly, the flexural modulus of the untreated bamboo and flax fiber reinforced sample is reduced by 12.07 and 2.21 %, respectively and with the alkaline treatment of the fiber the flexural modulus has been affected by 8.30 and 1.88 %, respectively.

Table 5 Flexural properties of wet and dry samples.

Material composition	Flexural strength (MPa)		Flexural modulus (GPa)	
	Dry	Wet	Dry	Wet
UB	72.10±1.0	57.62±1.5	2.65±0.04	2.33±0.04
TB5 %	77.41±1.5	69.78±1.0	3.13±0.07	2.87±0.06
TB10 %	75.85±2.0	65.79±2.0	3.06±0.06	2.93±0.05
TB15 %	74.25±1.5	60.85±1.0	2.82±0.05	2.64±0.0
UF	109.89±3.5	92.23±2.5	4.51±0.09	4.41±0.08
TF5 %	125.12±3.0	112.89±3.0	5.31±0.09	5.21±0.07
TF10 %	122.61±2.5	110.28±3.5	5.24±0.08	5.23±0.06
TF15 %	114.46±2.0	104.36±2.5	5.12±0.07	5.12±0.05

Figure 5 represents the effect of alkaline treatment on the impact strength of dry and wet epoxy/bamboo and epoxy/flax composite. It is observed that the impact strength of the wet sample has been reduced compared to the dry sample. Impact strength of the wet samples of untreated bamboo and flax fiber reinforced composites has been reduced approximately by 10.11 and 12.14 %, respectively due to soaking in distilled water, whereas the impact strength of 5 % NaOH treated fiber reinforced composite is reduced by 9 % due to soaking in distilled water, this is due to the improved water resistance property composites reinforced with alkaline treated fibers.

**Figure 5** Impact strength of wet and dry samples.

Conclusions

Epoxy/bamboo and epoxy/flax composites are processed and the effect of alkaline treatment and effect of water absorption on mechanical properties of composite has been evaluated. Alkaline treatment of fiber has enhanced the mechanical properties of the developed composite. The composite treated with 5 % NaOH gives the best set of properties among all the tested composites. Alkaline treatment of the bamboo and flax fiber with 5 % NaOH has improved the hardness by 3.57 and 2.43 %, tensile strength by 47 and 20.72 % and flexural strength by 7.36 and 13.85 % in bamboo and flax fiber reinforced composites respectively. Alkaline treatment of the fiber is seen to reduce the magnitude of maximum water uptake and diffusion coefficient. Mechanical properties of wet samples after water absorption are found to be less than the dry ones. Alkaline treatment of the fiber thus has reduced the impact of water absorption on mechanical properties.

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