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Research Article
Investigating the Physical Properties of Treated and Untreated Jute Fibre-Polyester Composites

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Abstract
Background and Objective: The jute fibre based composite materials have remarkable advantages of economic and environmental aspects such as low cost, low density, high strength to weight ratio and environmentally friendly. The study was focused on the composites that were prepared using unsaturated polyester as matrix and jute fibre as reinforcement by hand lay-up technique with the aim of the investigating the tensile, bending, thermal properties and water absorbency. **Materials and Methods:** Before fabrication of composites, jute fibres were treated with alkali and bleaching agent commonly known as combined scouring and bleaching. Composite of similar fashion was also prepared using untreated jute fibres. The composites were synthesized at 20:80 fibre-matrix weight percentages. The effect of chemical treatment was examined. The fractured surface morphology of the composites was investigated by Scanning Electron Microscopy (SEM) to comprehend the fibre-matrix interaction. The tensile, bending, thermal properties and water absorbency were studied and the results were graphically showed and explained. **Results:** The treated fibre composites showed higher tensile and bending strength than the untreated one. Both elastic modulus and bending modulus were also found to be higher for treated fibre composites. The treated fibres composites also exhibited lower thermal conductivity and higher thermal resistance. The untreated fibre composites become absorbent and significantly higher water absorption up to 300% and 200% than treated one in both 2 and 24 h water absorbency test. **Conclusion:** The behaviour of treated and untreated composites was explained by removing lignin and impurities from jute fibre that enhanced the better interaction between fibre and matrix. As natural fibres have very little resistance towards the environment, the manufactured composites can be the expedient application of biodegradable composites with auspicious physical properties. Furthermore, due to the simple manufacturing process, quicker processing time and lower manufacturing cost, the composites would be versatile material in the field of engineering and technology.

Key words: Jute, polyester, hand lay-up, tensile, thermal conductivity

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Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Polymer composites have been a subject of increasing interest in many years because of their significantly enhanced mechanical properties and thermal stability versus neat polymers[1]. The cellulose fibers based composite materials have remarkable advantages of economic and environmental aspects such as low cost, low density, high strength to weight ratio, high specific strength and modulus, limited requirements on processing equipment's, gentle to the equipment, soothing to the skin, less health risk, biodegradability, good insulation properties, eases of fibre surface modification and availability[2-6]. The selection criteria of fibre highly depend on their type, application, availability and cost[7-9]. Natural fibre composites have limited applications due to their lower mechanical properties where glass fibres have been taken 95% of the global market[10]. The natural bast fibre, jute is gained from the bark of jute plant and composed of major three components: Cellulose 58-63%, hemicellulose 20-24% and lignin 12-14%[10]. The potential advantage is attributed to its implicit toughness that arises because of the fibres themselves composites, made of reinforcing substances of cellulose strung in a lignin matrix, combined with hemicelluloses directing as interfacial coupling agents[11]. Composites made with jute fibre enhance the mechanical behaviour, increase the thermal resistance[12].

A serious problem of jute is their strong polar character which creates incompatibility with most polymer matrices. High moisture absorption and low processing temperature also the common problem with jute fibre[13-14]. Surface modification, fire resistance, durability are the main challenges to improve the properties of jute reinforced composites[9]. Economically, surface treatments have a negative impact and potentially able to overcome the problem of incompatibility[15]. Chemical treatments can increase the interface adhesion between the fibre and matrix and decrease the water absorption of fibres[16]. They also enhance the bonding strength that increases the fibre surface adhesion to surrounding matrix in the composites[17-18]. Therefore, chemical treatments can be considered in modifying the properties of natural fibres[19]. Unsaturated polyesters are widely used due to their excellent tensile and impact strength, clarity, thermal resistance[20]. As thermosets, the polymers are highly cross-linked which cured by applying heat or applying heat and pressure, and/or light irradiation. Thus the composites give high flexibility, great strength and modulus[21]. Composites made by thermosets are using in running bearings, vehicles, seals, gears, guiding liners, shafts, cams of brake pads of automobiles and aircraft industry[22].

Gowda *et al.*[23] investigated the woven untreated jute fabric reinforced polyester composites, produced by hand layup technique. They measured tensile, compressive, flexural, impact, in-plane shear, interlaminar shear strength and hardness of composites. They concluded that jute/polyester composites have better strength than wood and some plastic composites[23]. Mishra *et al.*[24] studied the mechanical performance of jute-epoxy composites. In the study, they modified the jute card sliver by treated with a liquid detergent solution to free jute batching oil (JBO) from fibre and then bleaching was done for delignification and dewaxing. They observed that the composites made by bleached fibre showed higher tensile strength and unbleached fibre composites showed better flexural properties. The bleached fibre composites also exhibited higher impact strength that unbleached one[25]. Doan *et al.*[12] studied the behaviour of jute-polypropylene composites after modification of matrix by MAHgPP and observed the improvement of adhesion strength with fibre and turned to the mechanical properties of the fibre[12]. Akil *et al.*[26] investigated the effect of water absorption on its mechanical properties of jute fibre reinforced unsaturated polyester composites following different aqueous environments: Distilled water, sea water and acidic solutions at room temperature. A significant increase in the maximum strain was observed to the increase in ductility of jute fibres[26]. Seki[27] investigated on jute polyester and jute epoxy thermoset composites. To improve the adhesion between jute fabric and resin, alkali-treated jute fibres were treated with oligomeric siloxane. The tensile and flexural properties of silane treated jute composites were increased as jute fibre enhanced the inter-laminar shear strength of the composites[27]. Sever *et al.*[28] studied on jute-polyester composites made by hand layup technique. They treated jute fabric with γ-methacryloxypropyltrimethoxysilane (γ-MPS) to improve the adhesion between jute fabric and polyester. By increasing γ-MPS (from 0.3-0.5%), the tensile properties were decreased and the flexural properties were improved[28]. Jawaid *et al.*[29] studied the effect of jute fibre loading on tensile and dynamic mechanical properties of oil palm empty fruit bunch-epoxy reinforced composites. They compared to oil palm empty fruit bunch and jute composites, made by hand layup technique. With increasing jute fibre loading, the tensile properties of the hybrid composite were found to increase[29]. Gopinath *et al.*[30] experimented on mechanical properties of jute fibre reinforced composites with polyester and epoxy resin matrices. Jute fibres were immersed in NaOH solution for 24 h. Jute-polyester and jute-epoxy composites were prepared with treated jute fibre. The jute-epoxy exhibited better tensile and flexural properties than jute-polyester one.

The processing time was very less of jute-polyester, compared to jute-epoxy composites[30]. Since jute-polyester, composites are playing a significant role as an engineering material and are well recognized in present composite epoch[31,32]. Mohammed *et al.*[22] evaluated the mechanical, thermal, energy absorption, moisture absorption, biodegradability, flame retardancy and tribology properties of natural fibre reinforced composites. Composites made from modified fibres by treatment with alkali and coupling agents showed lower moisture absorption and other properties are enhanced due to chemical treatments of fibre[22].

The aim of this study was to improve the physical properties of jute-polyester composites through surface modification of jute fibres by combined scouring and bleaching followed by the evaluation of the tensile, bending, thermal conductivity and water absorbency. In this context, the current study explicates the composites made by the reinforcement of chemically modified jute fibre with unsaturated polyester.

MATERIALS AND METHODS

Materials: Pure hundred percentage Bangla Tossa (BT) jute fibres (*Corchorusolitorious*) was collected from the laboratory of Bangladesh University of Textiles (BUTEX), Dhaka in the form of finisher card sliver. The linear density of fibre and sliver were 4.8 and 86 tex, respectively. Polyester resin and hardener (Methyl ethyl ketone peroxide MEKP) were purchased from Dolphon CC 1105 1K, Malaysia. Scouring agent-sodium hydroxide (NaOH), bleaching agent-hydrogen peroxide (H_2O_2), neutralizer-acetic acid (CH_3COOH), sequestering agent-ethylene diaminetetra acetic acid (EDTA) and peroxide stabilizer-sodium meta-silicate ($Na_2 SiO_2$) were collected from Merck, India.

Chemical treatment (combined scouring and bleaching) of jute fibre: The jute fibres were taken from finisher card sliver to get fibres in open-up, parallel and unidirectional form. Jute fibres were treated with alkali and hydrogen peroxide. The chemical treatment of jute fibres was aimed to increase the adhesion of fibres with polymer matrix which can be obtained by removing impurities e.g., wax, oil, fat, lignin from the fibres[33,34] .

Finisher card slivers were treated by NaOH and H_2O_2 (50%) with a liquor ratio of 1: 50 in a single bath. The fibres were kept immerses in this solution at a temperature of 70-80°C for 45 min. The EDTA-ethylene diamine tetra acetic acid and $Na_2 SiO_2$ were used as sequestering agent and peroxide stabilizer, respectively. The fibres were then washed several times with hot water to remove any NaOH and H_2O_2 sticking to the fibre surface and neutralized with dilute acetic acid CH_3COOH (50%) with a liquor ratio 1: 20 and finally washed again with normal tap water. Final pH was maintained to 6.9±0.1. The fibres were then dried at room temperature for 48 h. The above process was done in such a manner that the uniaxial orientation of fibres was not affected. By this process, the fibres were freed from the jute batching oil, partial de-lignification and de-waxing.

Methods: The fabrication of composites was carried out at the laboratory of Textile Physics Division in the Bangladesh Jute Research Institute (BJRI), Dhaka, Bangladesh. The experimental and other tests were carried out in the laboratory of BJRI, laboratory of University of Dhaka and Ahsanullah University of Science and Technology (AUST) in the prior before March, 2017.

Fabrication of composites: Hand layup technique was used to manufacture the composites board by jute, polyester resin and hardener. At first, Jute fibres were cut by the length of 12 inches. The curing agent was thoroughly mixed with resins and stirred properly for uniform mixing to form paste coat in a jar. Care was taken to avoid the formation of a bubble. Then coat was applied to the maillot paper uniformly. The layer of jute fibre was placed above the resin paste. The resin paste was applied again above the fibre and a steel roller was then used to even out the resin and remove any entrapped air between the fibre and resin. After that, it was covered with a maillot paper and a flat glass plate was placed on the top with a weight of 49 N to ensure a smoother surface and the fabricated composite was allowed to cure at room temperature for 4-5 h. During application of pressure, some amount of mixture of polyester and hardener squeezed out. Care was taken to consider this loss during the preparation of composites. The thickness of laminates was 4.5-6 mm. The gel time of the resin with 1.0% hardener was 5 min at room temperature. Figure 1 presents the manufactured composites.

Tensile test of fibre: The tensile strength of jute fibre was measured in accordance with ASTM D1445-05. This measurement was conducted by bundle fibre strength tester (Fibrostelo, India). Ten samples were tested. Their average and CV% were calculated.

Tensile test of composites: The tensile test of composites was conducted in accordance with ASTM D3039 in the universal

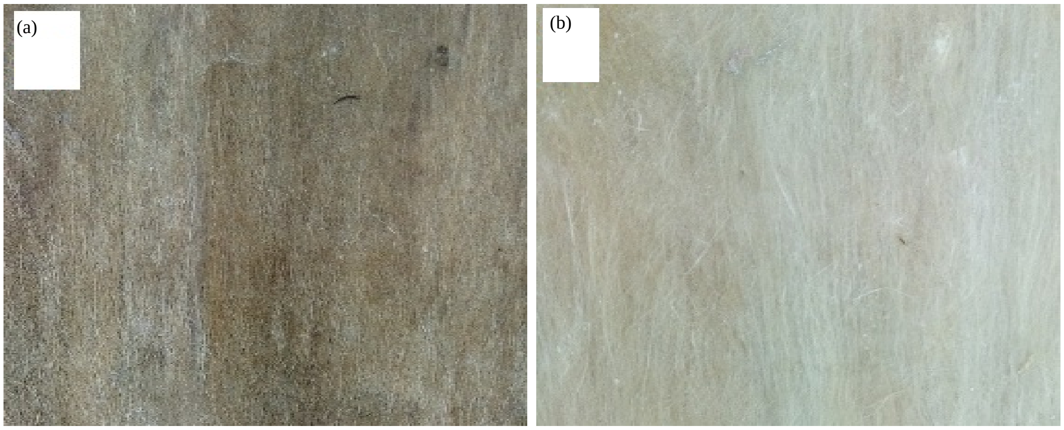

Fig. 1(a-b): Manufactured composites (a) Untreated and (b) Treated

testing machine (Tinius Olsen, USA) with a gauge length of 76.2 mm and crosshead speed of 10 mm min^{-1}. The "dog bone" shaped specimens were 254 mm (10 inches) long having a width of 25.4 mm (1 inch). The stress-strain curve was plotted during the test for the determination of ultimate tensile strength. According to standard, the tensile strength was tested for untreated and treated fibre-polyester composites of 10 samples.

Bending test of composites: The bending strength of composites was conducted to study the behaviour and the ability of material under bending load. The load was applied to the specimen until it was totally broken. The 3-point bending strength test was carried out by a universal testing machine (Tinius Olsen, USA) according to test standard ASTM D790. The specimen dimension was 101.6×12.7×4 mm and the crosshead speed was 10 mm min^{-1}. The tests were conducted for two different materials, five samples each. According to standard, bending strength was tested for untreated, treated fibre-polyester composites of 10 samples.

Thermal test of composites: Thermal conductivity is the intrinsic property of a material which relates its ability to conduct heat. Heat transfer by conduction involves the transfer of energy with in a material without any motion of the material as a whole. Conduction takes place when a temperature gradient existing solid (or stationary fluid) medium. Conductive heat flow occurs in the direction of decreasing temperature because higher temperature equates to higher molecular energy or more molecular movement. The thermal conductivity was measured by Lee's apparatus. The specification of Lee's apparatus were given below:

- Area of the sample in contact with the metallic disc, $A = 0.00971\ m^2$
- Thickness of the sample, $x = 0.005$ m
- Mass of the metallic disc, $m = 0.7915$ Kg
- Heat capacity of the metallic disc, $S = 380$ J/Kg K
- T_2-T_1 = Temperature difference across the sample thickness
- dT/dt = Rate of cooling of the metallic disc at T_2

Thermal conductivity:

$$K = \frac{mS\frac{dT}{dt}}{A\frac{T_2 - T_1}{x}} \tag{1}$$

Thermal resistance:

$$R = \frac{x}{KA}$$

Water absorbency test of composites: The mechanical properties of composites degrade when the composites absorb water. So the water absorbency of jute-composites was investigated to know the degree of degradation. The tests were performed for untreated, treated fibre-polyester composites of 10 samples, according to ASTM D570-95[35]. The specimen dimension was 76.2×25.4 mm.

SEM analysis of composites: The cross-sectional image of composites was observed by the scanning electron microscope. The samples were sputtering with platinum coating through the sputtering machine for 1 h. Then they were placed in the machine. The morphology of the jute-polyester composites with and without treatment was investigated using scanning electron microscope, JS-6490 LV. To make the samples conductive, a vacuum unit sputter coater was used to deposit a thin metallic layer of platinum of 70 nm thickness on the composites at an accelerating voltage of 15 kV.

RESULTS AND DISCUSSION

The properties of jute fibre that were changed due to chemical treatment and the properties of composites made by treated and untreated jute were examined and compared graphically in this section.

Chemical treatment of fibre: After chemical treatment, the weight loss percentage was measured and found 12.8 (average). It was occurred due to removing lignin and impurities from the fibre surface[36]. As well as fibre linear density was also reduced. A change of colour from brown to silvery white was observed due to de-lignification. The surface of fibres now becomes clean with increased micro-roughness which is very much favourable for fibre-matrix adhesion. The linear density of fibre and sliver were reduced to 3.25 and 79 tex, respectively. The hydrophilic nature hinders the effective interaction between fibre and matrix. To enhance the interfacial bonding between fibre and matrix, the fibres were treated with combined scouring and bleaching. It leads to develop a rough surface topography that results in better fibre matrix interface adhesion and an increase in mechanical properties[37]. It also changed the orientation of a highly packed crystalline portion of the fibre and formed an amorphous region in fibre structure. A certain amount of hemicellulose, lignin, pectin and waxes might be removed. It provided access penetration in chemical reactions[38]. Figure 2 presented the fibres used to produce composites.

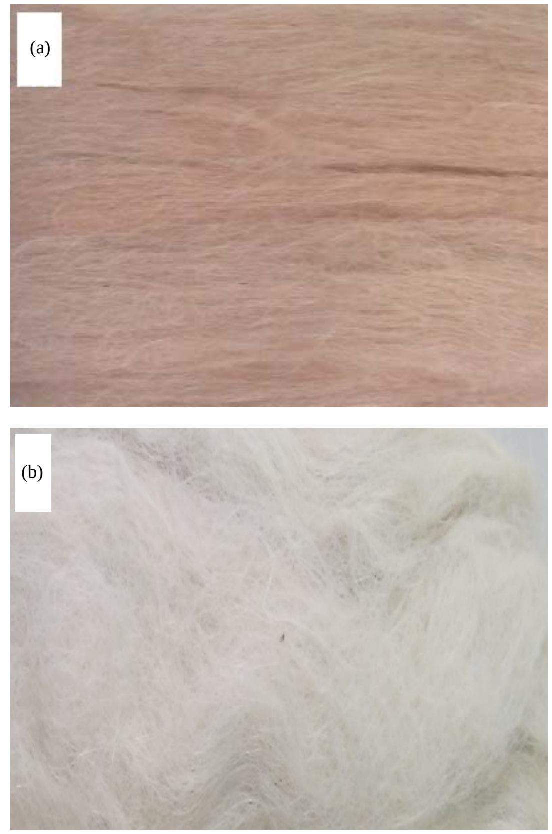

Fig. 2(a-b): Jute fibres (a) Untreated and (b) Treated

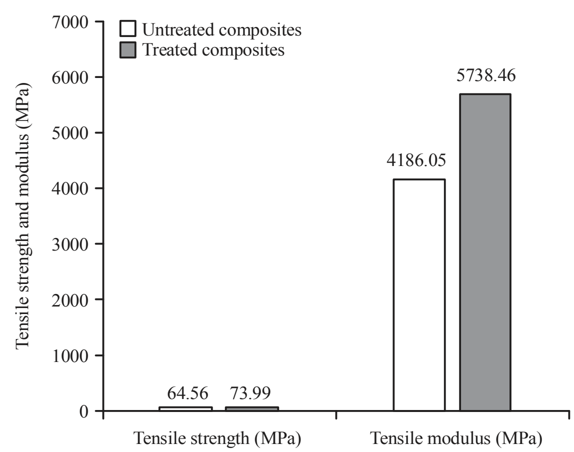

Fig. 3: Tensile strength and modulus of composites

Tensile properties of composites: The tensile behaviour of untreated and treated composites was investigated and shown in Fig. 3 and 4. The superior mechanical properties were found in the case of the treated jute-polyester composite. From the stress-strain curve, it is revealed that both composites demonstrated a brittle mode of deformation with a short initial steep followed by sharply rising stress until fracture. Compared to the untreated composite, treated composite exhibited steeper slopes indicating larger resistance to applying stress. It is attributed to the fact that reduction of lignin and impurities helps to interact fibres and matrix. In addition, the OH^- of NaOH reacts with OH^- and CH_2OH group of the cellulose backbone of jute fibre which increased the effective surface area available for content with the matrix polymer. As a result, jute fibre became more suitable to interact with the polyester matrix. The hydrophilic nature of jute incites to promote the formation of void at the fibre matrix interface that weakens the

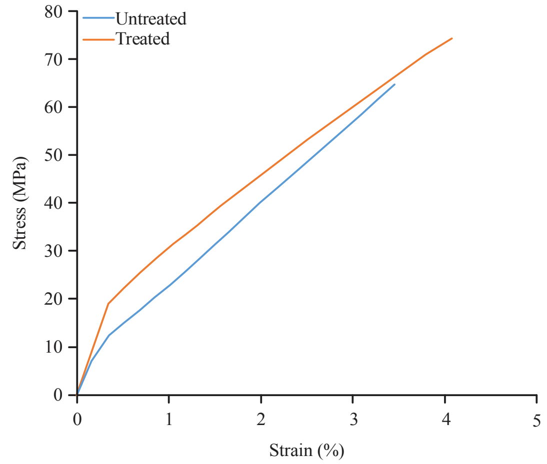

Fig. 4: Stress-strain curve for tensile strength

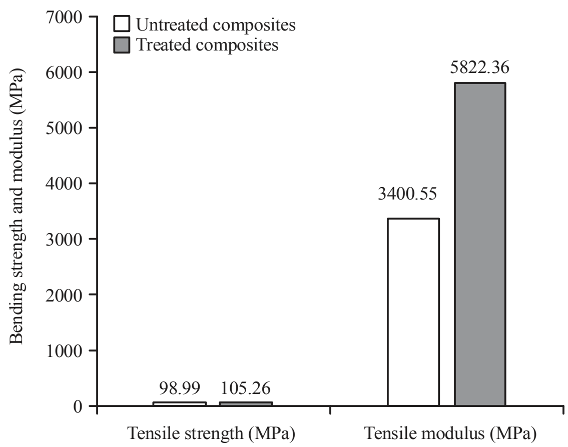

Fig. 5: Bending strength and modulus of composites

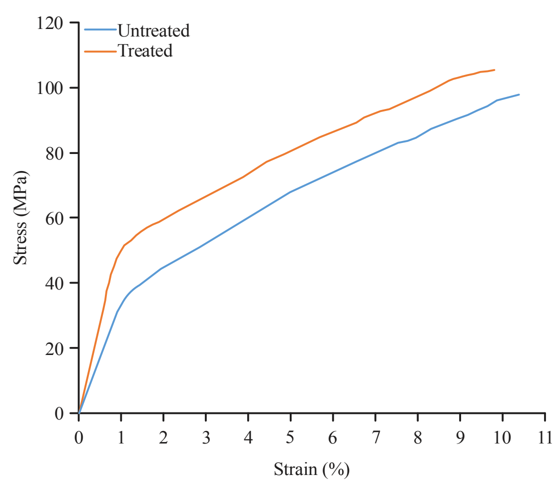

Fig. 6: Stress-strain curve for bending strength

bonds and make the composites of lower tensile properties. With regard to combined scouring and bleaching, the moisture regain capacity of jute fibre is reduced and the suitability with polyester resin has been improved to produce a strong interfacial bond between fibre and matrix[39]. So the treated jute-polyester composite was stiffer than the untreated one. As well as the elastic modulus of treated jute-polyester composite showed a higher value than untreated one.

Bending properties of composites: The bending properties are ascribed as to assess the suitability of a material for structural application. The representative stress-strain curve and bending strength and modulus were shown in Fig. 5 and 6. For both types of composites, the shape of stress-strain curves expressed an initial linear portion, a yield point and region of the high or low slope up to fracture. Both are brittle in nature. The higher tensile strength was obtained for treated composite and lower for untreated one. The fibre surface treatments had an effect on the bending modulus, similarly to the observations of tensile properties. The higher bending strength and modulus were associated with lower work of fracture[40]. This also indicates the increase in the area of contact between the fibre and the matrix were improving the level of adhesion.

Thermal properties of composites: The thermal properties of composites are presented in Fig. 7. It determined the thermal stability of composites. The thermal conductivity of treated composites was found to be lower. Comparing the untreated and treated composites, there was a reduction of fibre weight per unit length (from 4.8-3.25 tex) due to treatment. As the better interaction between fibres and matrix in the treated composites, they might be acted as barriers to the movement of electrons resulting in slowing down in its velocity of heat transfer, which took the higher time to pass through the surface. That might be responsible for lower thermal conductivity and as well as the higher resistance of treated fibre composites. In addition, the absorbance nature and volatiles can cause the increase of the thermal conductivity of composites[41]. So, treated composites showed lower thermal conductivity and higher thermal resistance than untreated composites.

Water absorbency of composites: It is observed that the treated composites showed lower absorbency (%) than untreated one in both 2 and 24 h absorbency test. Due to removing of cementing material and decreasing hydrophilic nature of jute fibres, the wettability of the fibre with resin was increased and there by improved the inter-bonding strength of fibre and matrix. Therefore, the chance of absorption of water reduced. The absorption of water of composites relates to composite properties such as dimensional stability.

Untreated composites absorbed water rapidly than treated one for the hydrophilic character of jute fibre. Water was soaked due to the presence of hydroxyl groups in jute fibre[18]. Figure 8 presented the water absorbency of composites.

SEM (scanning electron microscopy) analysis: The interfacial properties of both untreated and treated fibre composites (Fig. 9a,b) were investigated by Scanning Electron Microscopy (SEM). The fractured surface of both composites during the

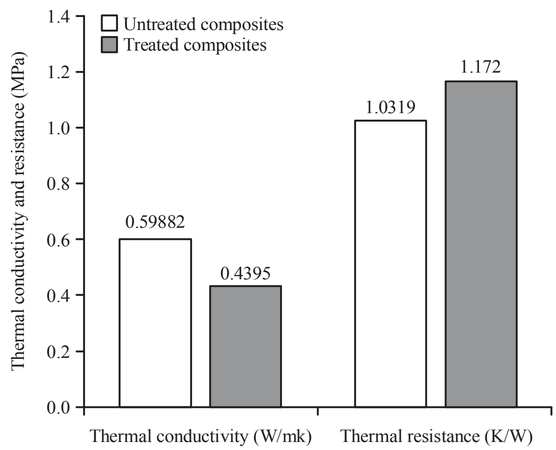

Fig. 7: Thermal properties of composites

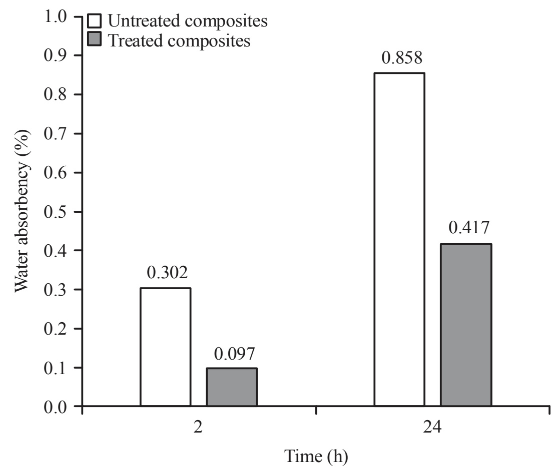

Fig. 8: Water absorbency of composites

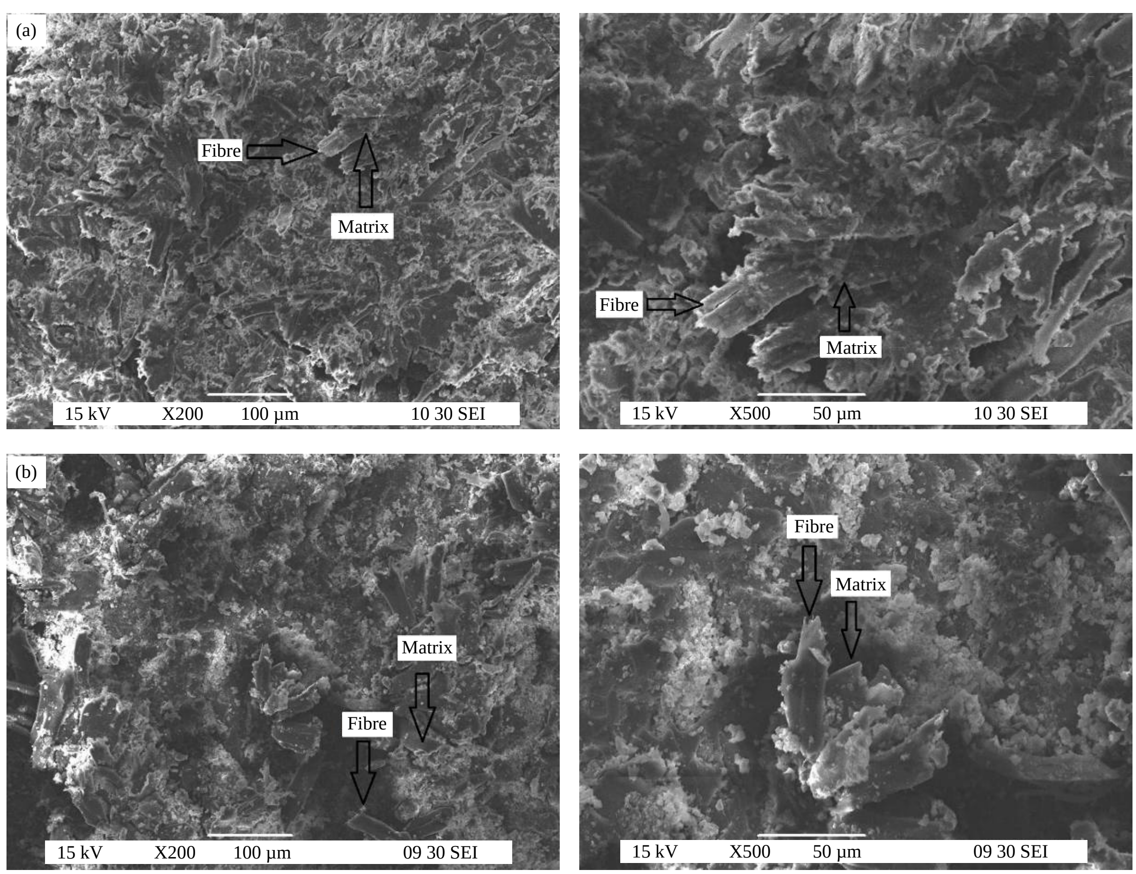

Fig. 9(a-b): SEM micrographs of (a) Untreated jute fibre composites and (b) Treated jute fibre composites tensile test was subjected to SEM. The fibres were easily pulled out from the matrix when stress was applied in untreated composites. The observation indicates that there was a considerable difference in the fibre-matrix interaction between treated and untreated composites. The improved bonding was observed for surface treated with alkali and H_2O_2, jute composites compared to untreated one. This might be facilitated the better mechanical interlocking and fibre-matrix bonding. A few numbers of air gaps were visible in untreated composites. The presence of air voids, fibre-matrix interaction and fibre solidarity are potential factors of reduction of strength of untreated composites. The removal of impurities (wax, fat) and lignin from the fibre surface enhanced the adhesion between fibre andmatrix[13].

From the above discussion, it was executed that the surface modification of fibres by combined scouring and bleaching is more unequivocal and economical way, unlike the conventional surface coating of fibres. It brought the drop in the linear density of fibres and improved toughness and hardness. Both tensile and bending strength of treated composites were higher than the untreated one. The thermal behavior of composites was also improved which may have implications of the materials in the field of heat insulation and sound barrier.

CONCLUSION

Jute-polyester composites were manufactured by untreated and treated fibres as reinforcement. The treated composites showed higher tensile and bending strength than the untreated one. The tensile and bending modulus were also higher in the case of treated composites. The lower thermal conductivity and higher thermal resistance were found for treated composites. Both 2 h and 24 h water absorbency results showed the higher absorbency for treated composites. From the investigation, it is revealed that the treated composites showed better physical properties than the untreated one. Considering the higher strength, lower thermal conductivity and lower water absorbency, the manufactured composites can be employed as shutter door, flooring and electronic device, automobiles as the companies like Daimler Chrysler, Mercedes Benz and Toyota are using composites materials as interior parts and outdoor application like roofing, light weight fishing boats. The distinguished properties of fabricated composites will make a versatile material in the field of engineering and technology along with environmental protection.

SIGNIFICANCE STATEMENT

This study discovers the possible significant effect on composites made by polyester and modified jute fibres with a unique chemical treatment where alkalization and bleaching were combined in a single bath. This study will help the researcher to find the critical area of interaction between the treated surface of the fibre and matrix and the implications of the composites that many researchers were not able to explore. Thus, this paper shows a more economical and less time-consuming technique for manufacturing and application of thermoset composites that can be used as an alternative to synthetic fibre composites.

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