Experimental Investigation and Analysis of Mercerized and Citric Acid Surface Treated Bamboo Fiber Reinforced Composite

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Abstract: Mercerization or NaOH fiber surface treatment is one of the most popular surface treatment processes to make the natural fibers such as bamboo fibers compatible for use as reinforcing material in composites. But NaOH being a chemical is hazardous and polluting to the nature. This paper explores the possibility of use of naturally derived citric acid for bamboo fiber surface treatment and its comparison with NaOH treated Bamboo Fiber Composites. Untreated, 2.5 wt\% NaOH treated and 5 wt\% citric acid treated Bamboo Fiber Composites with 5 wt\% fiber content were developed by Hand Lay process. Bamboo mats made of bamboo slivers were used as reinforcing material. Mechanical and physical characterization was done to compare the effects of NaOH and citric acid bamboo fiber surface treatment on mechanical and physical properties of Bamboo Fiber Composite. The experiment data reveals that the tensile and flexural strength was found to be highest for citric acid and NaOH treated Bamboo Fiber Composite respectively. Water absorption tendency was found more than the NaOH treated Bamboo Fiber Composites. SEM micrographs used to analyze the morphology of fracture surface of tensile test specimens confirm improvement in fiber-matrix interface bonding due to surface treatment of bamboo fibers.

Keywords: Mercerization, Citric Acid, Bamboo Fiber, Interface Adhesion, Characterization.

1. INTRODUCTION

Metals are the most widely used material for various purposes. But the need for lighter and stronger material grew stronger in next few decades and the composites started gaining importance: [1]. Presently the composites are used in structural and engineering applications confidently due to its advantages over metals such as less cost, high strength to weight ratio, customizable property etc.: [2]. Natural composites were being used abundantly during ancient times: [1]. But since past few decades due to rapid development and industrialization, synthetic fibers like glass fibers and carbon fibers gained popularity. Rising concern for depleting non-renewable resources and environmental circumstances have led the researchers to again explore the possibilities of use of natural fibers as reinforcing material in composites: [3]-[7].

Generally, the PFRCs are used as cheap, light and eco-friendly reinforcements but in
applications having no structural role. However, the recent research shows that high cellulose (~60-80%) content fibers have the potential to be used in structural applications: [8]-[10].

Bamboo is strong and stiff due to its lower micro fibrillar angle with the fiber axis and thicker cell walls compared to other natural fibers: [11]. Due to this reason Bamboo is also called “nature’s glass fiber”: [12]. The specific tensile strength and specific gravity of bamboo are found to be less than those of glass fibers but being cheap resource, it makes a demanded resource to be used as a reinforcing material: [13],[14]. The unidirectional nature of natural bamboo fiber over the other natural fibers allows good result reproducibility and constant mechanical properties: [15]. Natural bamboo fiber consists of around 60-70% cellulose, lignin (~32%) and hemicellulose matrix which holds these cellulose fibrils together: [16]-[19]. Hemicellulose is a cell wall polymer having hydrophilic nature because of which it has the maximum tendency to absorb water or moisture which later leads to undesirable effects such as swelling of fiber and presence of voids. This causes poor mechanical properties, dimensional stability of the composite and increases chances of microbial attacks: [9],[20]. Whereas Lignin shows hydrophobic nature, acts as a binding agent which provides compressive strength, stiffness to the fiber and also is resistant to alkalis: [9],[21],[22]. Due to the hydrophilic and hydrophobic nature of fiber and matrix respectively, the interfacial bonding formed is weak. This proves to be the weak point while transferring applied load from matrix to the fiber: [23].

With the help of suitable surface treatments, wettability and interface adhesion can be increased by removing hemicellulose, wax, oil and other impurities: [14],[24]. Also the natural fibers contain hydroxyl or few other oxygenated groups that absorb moisture through H-bonding. Due to availability of many reactive groups, modification of cell wall is possible using proper surface modifier to increase the usage of natural fibers as reinforcement: [25].

Mercerization or NaOH treatment is one of the most widely used surface treatment process. It has also delivered remarkable results by improving the interface adhesion. From the studies, it has been seen that such treatment processes improved fiber-matrix interfacial bonding resulting to improved mechanical properties such as hardness, tensile strength, impact strength etc.: [26]-[30]. But as we want to move towards sustainable development and low cost processing of fiber, surface treatment chemicals should be cheap, user friendly and less hazardous. Citric acid being a naturally derived weak organic acid can be used for surface treatment of bamboo fibers. It is available in citrus fruits naturally and can be commercially produced by fermenting glucose or glucose- and sucrose-containing materials: [31],[32]. Besides its uses in foods, beverages and pharmaceuticals, it also acts a good crosslinking agent for plant fibers, paper, wood, starch and natural elastomers. Articles and research papers on citric acid as a bonding material is available but literature related to citric acid as surface modification material is hardly found. Researchers have reported that citric acid can be used as a natural adhesive as the ester linkages are formed by carboxyl groups of citric acid and hydroxyl groups of wood: [33]-[35]. One of the researchers used citric acid for treatment of Kenaf fibers and found that citric acid treated fibers show less fiber surface damage. Tensile strength of citric acid treated fiber composite decreased with increase in fiber loading compared to NaOH treated fiber composite. But percentage elongation
of Citric acid treated fiber composite was found to be much higher than the NaOH treated fiber composite: [36]. However, the mechanical properties of citric acid treated bamboo fibers have not been studied yet. As citric acid is cheaper than NaOH and derived from eco-friendly process, replacement of NaOH with citric acid in fiber surface treatment can lower the processing cost with eco-friendly substitute. Hence, this paper will explore the effects of citric acid treatment of bamboo fibers on bamboo fiber composites and its comparison with mercerized bamboo fiber composites.

2. EXPERIMENTAL

2.1 Materials

Woven bamboo fiber mats were used as reinforcing material. Bamboo mats of Dendrocalamus strictus species of 300 x 300 mm were obtained from the rural bamboo handicraft worker.

Thickness and width of the slivers used in bamboo mats were kept 0.6±0.15 mm and 5±1 mm respectively. 2D and 3D Weaving pattern of acquired mats are represented by Fig. 1 and 2 respectively. The mats were washed in water to remove dirt and other water soluble impurities and dried at 30°C for 24 h. Then the dried mat was stored in air tight polybag until its use.

![Fig. 1 2D weaving pattern](image1)

![Fig. 2 3D weaving pattern](image2)

Epoxy Resin was used as matrix in manufacturing composites. Standard epoxy resin Araldite CY-230 is a colorless liquid resin having high dimensional stability, mechanical, chemical and adhesion property with good chemical and atmospheric resistant property. Araldite HY-951 was used as hardener with epoxy resin in 1:10 ratio by weight. Araldite HY-951 is a yellowish green color liquid.

2.2 Fabrication of Composites

Composites with 5 wt% fiber content measuring 200 x 140 x 3.6 mm were fabricated using Hand Lay process. Composites were fabricated for untreated, citric acid treated and NaOH treated BFC. Glass mold was used along with Perspex sheets as releasing agent. Layers of resin and mat were laid one by one till desired thickness builds up. A roller was used to squeeze out any trapped air bubbles. The mold was kept under uniform load for 48h at room temperature. Post curing, the Perspex sheet was removed just by pulling with minimal efforts. The composite panel was checked for any defects like bubbles entrapment of gaps due to lack of resin.

2.3 Sample Preparation

For tensile, flexural and water absorption testing, sample shapes were marked on the
composite panels and samples were cut out from it. Jig saw machine with fine teeth blade was used for sample cutting purpose. Table I shows the nomenclature used for untreated and treated fiber composite specimens.

Table I: Nomenclature of the specimens

| Specimen | Specification                  |
|----------|--------------------------------|
| U        | Untreated BFC                  |
| C        | 5 wt% citric acid treated BFC  |
| N        | 2.5 wt% NaOH treated BFC       |

2.4 Characterization of Composites

2.4.1 Tensile and Flexural Testing

Tensile and Flexural Characterization on BFCs were performed as per ASTM D638-02a and ASTM D790-02 respectively on Universal Testing Machine (Model: INSTRON 4467) equipped with 30KN load cell.

Five specimens were cut out from BFC panel and tested for each type of composite at 75 mm gauge length and a crosshead speed of 1 mm/min. All the specimens were conditioned for 24 h in a room at 23°C and 50% RH, prior to the testing. The ultimate tensile strength, tensile failure strain, Young’s modulus were measured from the stress strain curve and flexural strength, flexural modulus and flexural strain were obtained from the load-displacement graph.

2.4.2 Water Absorption Testing

Water absorption test was conducted as per ASTM D570-98 to observe the water absorption tendency of the untreated and treated fibers and its effect on the composites. Five specimens of 50 x 13 x 3.5 ± 0.2 mm were prepared and weighed to the nearest 0.001g with electronic weighing machine having a least count of 0.001g. Each specimen was completely immersed in distilled water at 25°C.

2.4.3 Scanning Electron Microscope

Scanning Electron Microscope (Model: JEOL) was used to observe the fiber-matric interface of cross section of the tensile test specimens and evaluate the fracture behavior to correlate it with the tensile strength. The specimens being non-metallic, to enhance the conductivity, the specimens were coated with palladium using a sputter coater for 60 seconds at a current of 20 mA using Auto Fine Coater (Model: JEOL JFC – 1600). We can obtain clear images at higher magnification when compared to optical microscopes.

3. Result and Discussion

Results from tensile and flexural test of untreated and treated BFC are presented in Fig. 3 and 4 respectively. The entire tensile and flexural test specimen failed in the region of gauge length. NaOH and citric acid treated BFC were found to have 13.38% and 20.43% higher tensile strength respectively when compared to untreated BFC and decreasing trend in Young’s Modulus can be seen from untreated to treated BFCs. Citric acid treated BFC shows highest tensile strength and lowest Young’s Modulus. Whereas, NaOH and citric acid treated BFC were found to have 43.58% and 15.15% higher flexural strength respectively when compared to
untreated BFC. Fig. 4 shows that NaOH treated BFC have highest flexural strength. It indicates that the fiber surface treatment improves the load bearing capability and the stiffness reduces.

Fig. 3: Chart for Tensile test results  
Fig. 4: Chart for Flexural test results

The improvement in tensile and flexural properties of the treated BFC is due to the fiber surface treatment. Higher tensile strength and lower flexural property of citric acid treated BFC compared to NaOH treated BFC shows that the mechanical bonding is dominant in case of force acting parallel to the fiber surface and weak when force acting perpendicular to the fiber surface. The fiber surface treatments change the fiber surface chemistry and clean the fiber which results to increased wettability, interface adhesion and reduced water absorption tendency. This can be confirmed by weight gain (%) by water absorption test.

In Fig. 5, it is observed that the weight gain (%) is a function of time. The water absorption of NaOH and citric acid treated BFC were found to be lower than untreated BFC. The NaOH treated BFC highest resistance to water absorption. However, irrespective of fiber surface treatment, water absorption increased with time in different rates. The maximum weight gain (%) obtained for untreated, Citric acid treated and NaOH treated bamboo fiber mat are 19.85, 15.57 and 14.02 respectively.

Treated fiber absorbs less water as they contain contracted cellulose walls due to which water absorption is also less. The reduction in water absorption tendency in case of treated BFC shows that the fibers hydrophilic property is reduced and interface bonding has increased: [37,38]
Due to NaOH treatment, certain amount of hemicellulose, lignin, wax, inorganic salts and oils covering the surface of natural bamboo fiber is removed and few changes occur in surface of natural bamboo fiber which provides better flexural property to the NaOH treated BFC such as fiber surface topography is roughened which provided additional sites for chemical and mechanical interlocking, Hydrophilic property of the natural bamboo fiber is reduced, hence making the fiber more resistant to moisture and better interface adhesion, polymeric bonds of the cellulose converts to monomers exposing short length crystallites: [39–45].

Similarly, citric acid treatment cleans the fiber surface by removing fewer amounts of hemicellulose, lignin and more amounts of wax, inorganic salts and oils. Due to its weak acidic property, it doesn’t roughen the fiber surface much. The natural binding property of citric acid acts as a binder to the matrix because of which higher tensile properties are obtained in citric acid treated BFC. The stronger bonding between the fiber and matrix is due to formation of ester linkages between hydroxyl groups of fiber surface and anhydride produced due to dehydration of citric acid during the reaction: [46].

Study of SEM micrograph explains the effects of surface treatment on fibers and the reason for change in mechanical properties. The failure pattern indicates that the failure is matrix dominated in all the cases. The SEM micrograph of untreated bamboo fiber composite in Fig. 6 a) shows occurrence of fiber pull-out and presence of gaps in between interface. Fig. 6 b) shows the SEM micrograph of untreated fiber bundle surface containing hemicellulose, wax, oil and other impurities on its surface. This explains the incompatibility of untreated fibers with the matrix which leads to early failure of the composite.

Fig. 6: SEM micrograph of fracture surfaces of Tensile tested specimen. a) Fiber pull-out and gap at interface; b) Fiber surface containing hemicellulose, wax and other impurities

Fig. 7: SEM Photomicrograph of fracture surfaces of tensile tested NaOH treated BFC specimen. Fiber pull-out occurred from between the fiber bundle

Fig. 8: SEM Photomicrograph of fracture surfaces of tensile tested NaOH treated BFC specimen. Fiber peeling observed on the pull-out fiber surface
Fig. 9: SEM micrograph of fracture surfaces of tensile tested Citric acid treated BFC specimen. a) and b) Fiber pull-out occurred from between the fiber bundle

Whereas in case of treated bamboo fiber composite the interface adhesion is higher which results to higher mechanical properties. SEM micrograph show similar observations for NaOH and Citric Acid treated BFC as shown in Fig. 7, 8 and 9. Unlike the NaOH treated BFC as shown in Fig.7 and 9, citric acid treated fibers didn’t get brittle after treatment and hence the stress from the matrix was transferred to the fiber more effectively and the fibers were able to sustain more load resulting to more superior strength. As shown in Fig. 8, fiber peeling can be observed on the pull-out fiber bundle surface. The parts of fibers can also be seen sticking to the fiber pull-out part of the matrix. This indicates that post treatment; the fiber-matrix interface adhesion has increased due to removal of hemicellulose, wax oil and other impurities from the fiber surface. Improvement of fiber-matrix interface adhesion leads to better mechanical strength. As shown in Fig. 7 and 9, alike in NaOH treatment, due to the presence of fiber bundles, effect of citric acid treatment was more on outer fibers than the middle ones and the resin was in contact with only the outer fiber of the fiber bundle. The weak natural bonding between the outer and inner fiber of the fiber bundle lead to fiber pull-out from the middle of the fiber bundle.

4. CONCLUSION

1. The tensile and flexural strength increased for treated BFC. Citric acid and NaOH treated BFC resulted to highest tensile and flexural strength respectively.
2. Compared to NaOH treated BFC, citric acid treated BFC shows better interface bonding when force applied parallel to the fiber surface and weak when force applied perpendicular to the fiber.
3. Surface treatment of fibers reduces the hemicellulose, wax, oil and other impurities contents from the fiber surface. Unlike mercerization, citric acid treatment didn’t make the fibers brittle and fiber surface rough.
4. Surface treatment of NaOH and citric acid treated fibers improved the wettability of fiber and the fiber-matrix interface was stronger than the fiber-fiber interface.
5. Citric acid treatment can be a replacement to NaOH treatment which will result to low processing cost and eco-friendly.
6. Due to use of slivers containing fiber bundle, the surface treatment was more pronounced on the outer fibers of the fiber bundle and the bonding of the resin was possible only with the outer fibers of the fiber bundle.

7. Treated BFCs show less water absorption tendency. NaOH and Citric Acid treated BFC show minute difference in water absorption tendency.

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