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Article

Effect of Mimosa Grafting on Anatomy, Chemical Composition and Tensile Properties of Bamboo Fiber

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Abstract. The current study presents systematic and improved methodologies to characterize elementary bamboo fiber grafted with mimosa. For that, control and grafted bamboo culm anatomy was observed under scanning electron microscopy (SEM), chemical analysis was conducted and tensile properties were measured. During surface analysis, smoother surface was found for grafted fiber compared to the control fiber. No significant change in chemical composition was observed after grafting. In this experiment with the increase of span length the value of tensile strength has decreased but the young modulus has increased. The tensile strength of grafted fiber was higher than the control fiber. This is because fiber of larger span length has much larger surface area having more flaws and surface defects, which make the chance of failure larger .Thus it can be concluded that mimosa grafting seems to be useful for stronger control fiber with more reliable results.

Keywords: Mimosa, strain to failure, α-cellulose, SEM.

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1. Introduction

In the context of challenging environmental issues and a global energy crisis, bio-based materials are attracting increasing levels of research interest, from both academia and industry, because of their numerous advantages: renewable resource usage, low cost, biodegradability, and so on [1, 2]. The development of biodegradable polymers has been a subject of great interest in materials science for both ecological and biomedical perspectives. Increasing awareness of the environmental damage caused by plastic materials over the last few decades has led to research aimed at producing eco-friendly versions of those plastics [3, 4]. For that polymer matrix composites, such as carbon or glass fiber reinforced plastics have been widely used in industry since they have high strength and modulus [5, 6]. Hemp, flax, sisal, jute etc have been identified as attractive materials for the reinforcement of thermoplastic polymers. They are cheap, abundant and renewable, and have good specific properties due to their low densities [7-10]. Beside them, bamboo fiber can also be used as a potential reinforcement in composite. Bamboo is an abundant natural resource in Asia, and its overall mechanical properties are comparable to those of other fibers. Bamboo has a great potential to be used as a reinforcing agent in polymeric composites. Such composites can be successfully used as consumer durable goods and for less-intensive load-bearing application. Furthermore, bamboo grows to its mature size in only 6–8 months, whereas wood takes about 10 years. Shao et el. [11], Papadopoulos et el. [12] and Kongkeaw et el. [13] worked on bamboo fiber for composite preparation. In natural fiber, presence of hydroxyl and polar groups lead to weak interfacial bonding between fibers and relatively hydrophobic polymers. This is due to pendant hydroxyl and polar groups in various constituents of fiber. As a result hydrophilic natural fibers absorb a large amount of water in the composite leading to failure by delamination from the matrix. Adequate adhesion across the interface can be achieved at desirable levels by better wetting and chemical bonding between fiber and matrix. To make good use of biofiber reinforcement in composites, fiber surface treatment can carried out to obtain an enhanced interface bonding between hydrophilic bamboo fiber and hydrophobic polymer matrixes. Besides such treatments will decrease the moisture absorption and hydrophilic character of bamboo fibers. Surface modification is therefore necessary to obtain better performance of the resulting composite. Kushwaha et el. used silane as interfacial coupling agents to form stable covalent bonds with both the mineral fiber surface and the resin [14]. Chen et al. used cardanol as grafting agent in preparation for better performance of composite. In their experiment isocyanate silane and amino silane were used to modify the surface of bamboo fiber [15, 16]. In present research mimosa was used for grafting the surface of bamboo fiber. Grafting reaction is a practical technique to improve its dyeability, compatibility and hydrophobicity in economical way conducting chemical modification or polymerization reaction. Grafting enhanced the commercial viability and cost effectitiveness of the material. Man's relationship with plant polyphenols is ancient. Mimosa is vegetable polyphenols tannis, which converted organic substance into stable materials, raised the denaturation temperature, resistance to putrefaction by micro-organisms in the environment. Chemically, mimosa is a polyphenol named 3,4-dihydroxy-2[[(3,4,5-trihydroxybenzoyl)oxa]oxan-2-yl]methol 3,4,5 trihydroxybenzonzte with molecular weight 36346866 g/mol having chemical formula $C_{27}H_{24}O_{18}$. In mimosa there are 11 H-bond donor which have been shown in Fig. 1. Mimosa can scavenge carcinogenic and mutagenic oxygen free radical which will be bonded with hydroxyl of cellulosic fiber. As a result cellulose can be able to stabilise against putrefaction, rendering it resistance to biochemical degradation [17, 18]. In present research control fiber was grafted with mimosa and its effect on anatomical, chemical composition and tensile properties were evaluated. It seemed that grafting process can enhance the mechanical properties of bamboo, which in turn can be used for preparing better bio-fiber reinforced composites.

2. Experimental Method

2.1. Materials

Control fiber was extracted from culms of bamboo plants. The fiber was extracted by steam exploration out of the *Bambosa vulgaris* species, which is available in Bangladesh. Before test, control fiber was visually selected in order to verify the absence of defects along the length of the fiber. Mimosa was purchased from local market in Bangladesh. All chemicals used in present research were of analytical grade. Chemical structure of mimosa is shown in Fig. 1.

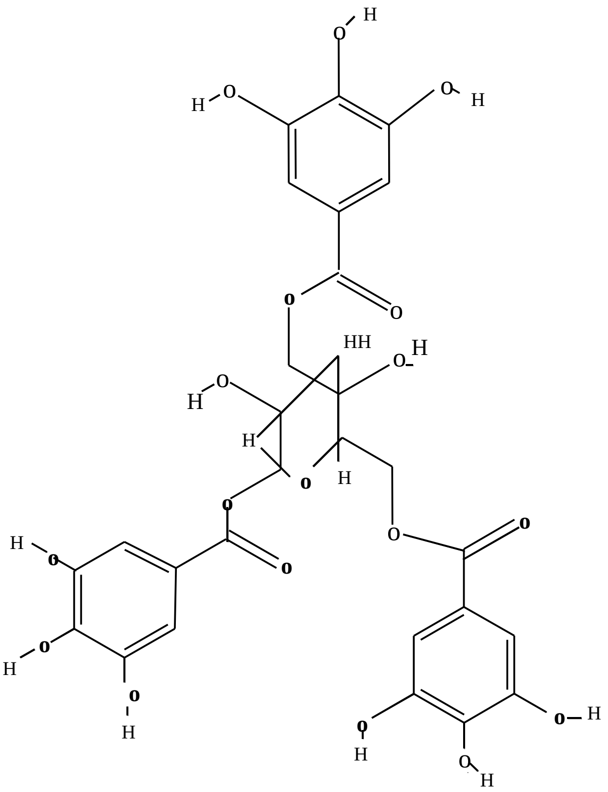


Fig. 1. Chemical structure of mimosa [19].

2.2. Chemical Treatment of Bamboo Fiber

The control fibers were initially collected and cleaned. For chemical treatment, a solution of 8% mimosa with few drops of H_2SO_4 was prepared to maintain initial pH of 4-4.5. Bamboo fiber was taken into the prepared solution and shaken for 3 hours. After 3 hours reaction, the fibers were washed properly in distilled water. Fibers were then dried and stored in dry container.

2.3. Morphological Study

The anatomy of bamboo, both control and mimosa treated, culm and cell was observed using scanning electron microscope (XL 30 Philips, Netherland). Diameter of the fiber used for tensile test was also measured from SEM. The samples were made conductive by applying a gold coating using sputtering technique. Thin gold coating caused the electrons to interact with the inner atomic shells of the sample.

2.4. Chemical Analysis

In order to determine chemical composition (holocellulose, alpha-cellulose, Klason lignin, hot water extractives and ash) of control and grafted fiber sample, the internodes of each height were cut into small strips with razor blade. The strips were small enough to be placed to ground in a Wiley mill. The material was then placed in a shaker with sieves to pass through a no. 40 mesh sieve (425-µm), yet retained on a no. 60 mesh sieve (250-µm). The resulting material was then placed in glass jars for chemical analysis. All tests were conducted using ASTM standards [20-25].

2.5. Tensile Test

Tensile test of control fiber was carried out using an Instron universal testing machine (Model no3369) by varying span length (5mm, 10mm, 15mm, 25mm and 35mm) of both control and grafted fiber. The sample preparation is shown in Fig. 2. The cross-head speed and load cell used were 1mm/min and 200N respectively. Samples which had broken near the edge of the clamps were excluded from the analysis. The isolated fibers were conditioned at 20 ± 2°C and 65 ± 2% relative humidity before temporarily fixed on the paper frame with adhesive tape. A droplet of glue was applied on the centre of both sides of the hole along the length of card.

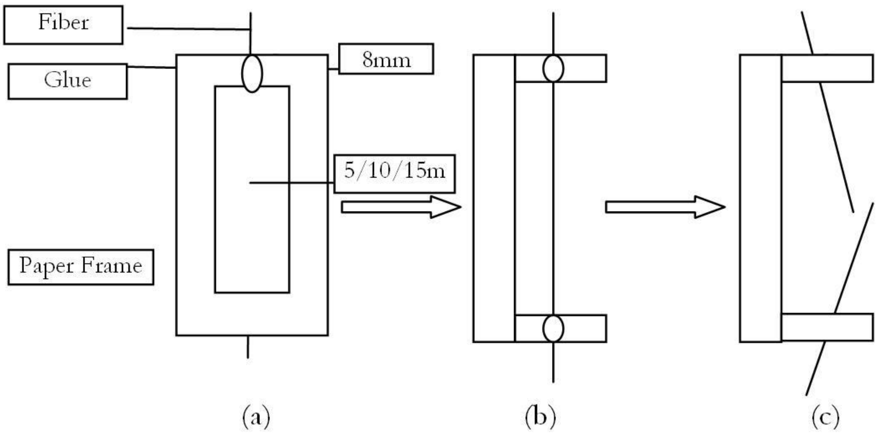


Fig. 2. Set-up of single fiber testing: (a) specimen mount; (b) test specimen on the paper frame; (c) broken fiber.

The tensile strength was calculated using following formulas:

Tensile Strength, $$\sigma = \frac{F_{max}}{A} \tag{1}$$

where F_{max} is maximum force and A is cross sectional area.

The Young's Modulus and strain to failure was measured from the stress-strain curve. During tensile test, some slippage portions always occur. Large span length minimizes the slippage portion more compare to smaller ones. Thus Young's modulus found was higher for larger which was not desired. So correction was required for Young's modulus values. Young's modulus was corrected by using the following steps:

$$\frac{\Delta l_{total}}{\text{Test length}} = \frac{\Delta l_{fiber}}{\text{Test length}} + \frac{\Delta l_{nonfiber}}{\text{Test length}} \tag{2}$$

where Δl_{total} is the measured displacement of the clamps, Δl_{fiber} is the elongation of the fiber and $\Delta l_{nonfiber}$ is the displacement caused by slippage and test set up compliance.

A correction process was adopted to determine the non fiber displacement. The fiber displacement Δl_{total} was measured and at a certain stress in the linear part of the stress-strain curve was calculated. (Δl_{fiber}/test length) was calculated by dividing the same chosen stress by the estimated the e-modulus for an infinity long test length, in other word the extrapolated modulus from an Young's modulus in function of (1/span length) curve at (1/span length)=0.

$$\frac{\Delta l_{non\ fiber}}{\text{test length}} = \frac{\alpha F}{\text{test length}} = \frac{\sigma A \alpha}{\text{test length}} \tag{3}$$

where F is the load put on the fiber, A is the cross-sectional area, σ is the stress (in the linear portion of the stress strain curve) α the factors that estimated the influenced of slippage and the test setup compliance. So for every tested fiber α can be calculated:

$$\alpha_i = \frac{\Delta l_{total,i} - \Delta l_{fiber,i}}{F_i} \tag{4}$$

All α-values were plotted in function of span length and by linear regression, an estimated of the α-value for each test length can be determined ($\alpha_{\text{test length}}$) for each type of fiber. With this estimated value for α the corrected strain can be calculated:

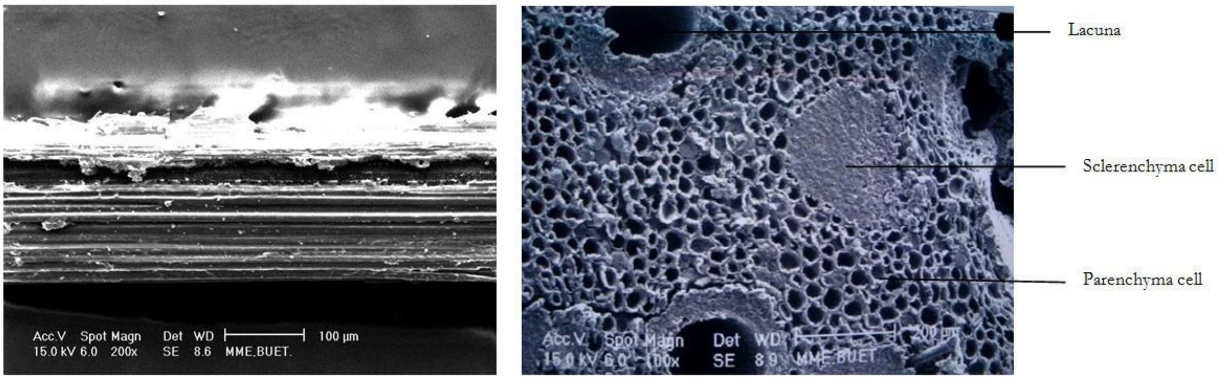
$$\text{Corrected Strain} = \frac{\Delta l_{fiber\ i}}{\text{test length}} = \frac{\Delta l_{total,i}}{\text{test length}} - \frac{\alpha_{\text{test length}} F_i}{\text{test length}} \tag{5}$$

Young's modulus was finally determined by drawing the stress in function of corrected stress-strain curve.

3. Results & Discussion

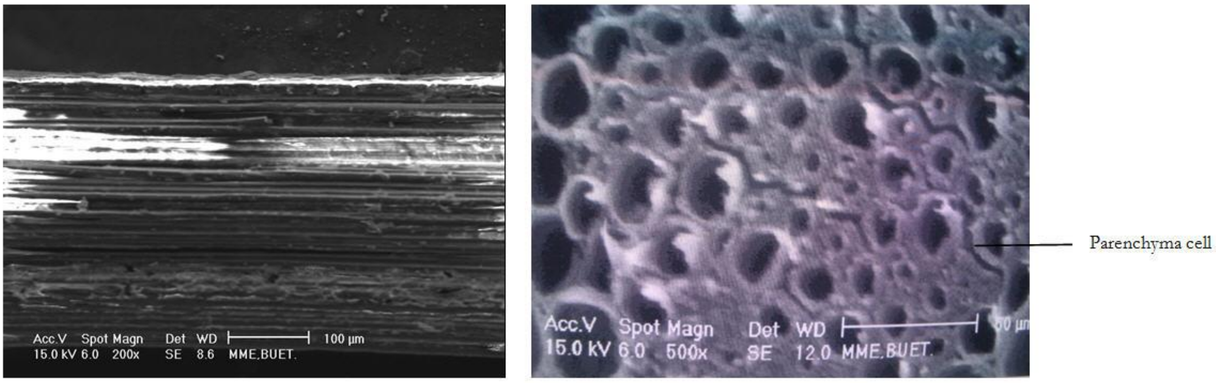
3.1. Morphological Study

In present research, anatomy and cell wall was observed under SEM. Fig. 3(a) and Fig. 3(b) represent the surface and cross-sectional area of control fiber respectively, while Fig. 4(a) and Fig. 4(b) represent the surface and cross-sectional area of grafted fiber respectively. The inner layer is wrapped by sclerenchyma cells. In lacuna, elements in bamboo are easily permeable. The structure of a bamboo culm transverse section characterized by numerous vascular bundles embedded in the sclerenchyma tissue and parenchymatous ground tissue. The parenchyma cells are mostly thin-walled and connected to each other by numerous simple pits. Pits are located predominantly on the longitudinal walls. But the sclerenchyma cells are thick-walled. Sclerenchyma generally forms packages of fibers below vascular bundles and around the vascular bundle. Sclerenchyma surrounding the first cycle of peripheral vascular bundles are not interrupted by interfascicular parenchyma. Surface of control and grafted fiber was under the SEM. The surface of the grafted fiber sample was smoother compared to the untreated bamboo fiber. When the bamboo fiber was grafted with mimosa, in the transverse section, parenchymatous ground tissue was found thicker than the control fiber. As there is no significant change in chemical composition so the change was may be in the cellulosic chain. May be mimosa have been deposited in the cellulose chain. As mimosa have 11 H- donor for that they formed the covalent bond with cellulose -COOH to form cellulose stronger.



(a) (b)

Fig. 3. SEM of (a) cell wall and (b) cross-sectional area of control bamboo fiber.



(a) (b)

Fig. 4. SEM of (a) cell wall and (b) cross-sectional area of mimosa grafted bamboo fiber.

3.2. Chemical Analysis

The results of chemical analysis of control and grafted bamboo fiber are listed in Table 1. In this processes 14.03% mimosa was grafted. Li worked on *Phyllostachys pubescens* bamboo and found 5.14% hot water solubility, 22.11% lignin, 70.84% holocellulose, 47.30% α-cellulose, 1.94% ash [26]. Ghoshal et al. worked on bamboo fiber and found 60% cellulose and 32% lignin [16]. During present chemical analysis, alpha cellulose was found to be 50.23±.18% indicating that homopolysacsride consist of β-D-glucopyranose linked together by β,1-4-linkage. Each cellulose monomer contained three hydroxyl groups that were able to form hydrogen bond for crystalline packing governing the tensile properties of cellulose of bamboo [27-30]. No significant change in chemical composition was observed after grafting.

Table 1. Chemical composition of original and mimosa treated bamboo sample.

Sample	Hot Water Soluble %	Moisture %	Lignin %	Holocellulose %	α-cellulose %	Hemicellulose %	Ash %
Original bamboo	5.07±0.06	12.33±0.56	23.91±0.58	67.08±0.17	50.23±0.18	16.85±0.09	1.38±0.05
Mimosa treated bamboo	4.30±0.03	11.15±0.55	22.41±0.32	67.67±0.12	50.93±0.18	16.94±0.09	0.47±0.02

3.3. Tensile Properties

The tensile strength of control single fiber for different span length is shown in Fig. 5, while Fig. 6 shows average the tensile strength of control and grafted single fiber. Tensile strength decreased with increase in fiber span length. This is because fiber of larger span length has much larger surface area having more flaws and surface defects, which make the chance of failure larger. However the tensile strength of mimosa treated fiber was higher than control fiber. The corrected and uncorrected Young's modulus values for both control and mimosa grafted fiber are shown in Fig. 7 and Fig. 8 respectively. Uncorrected Young's values increased with increase in span length. This may be due to slippage and test setup. The slippery portion is higher for smaller span length compared to higher span length. The uncorrected extrapolated Young's modulus for infinitely long fiber was 35.2 GPa for control fiber and 44.45 Gpa for grafted fiber. Strain to failure against span length for control and grafted fiber are shown in Fig. 9 and Fig. 10 respectively. With increasing the fiber length, the strain failure decreased due to more weaknesses and earlier break.

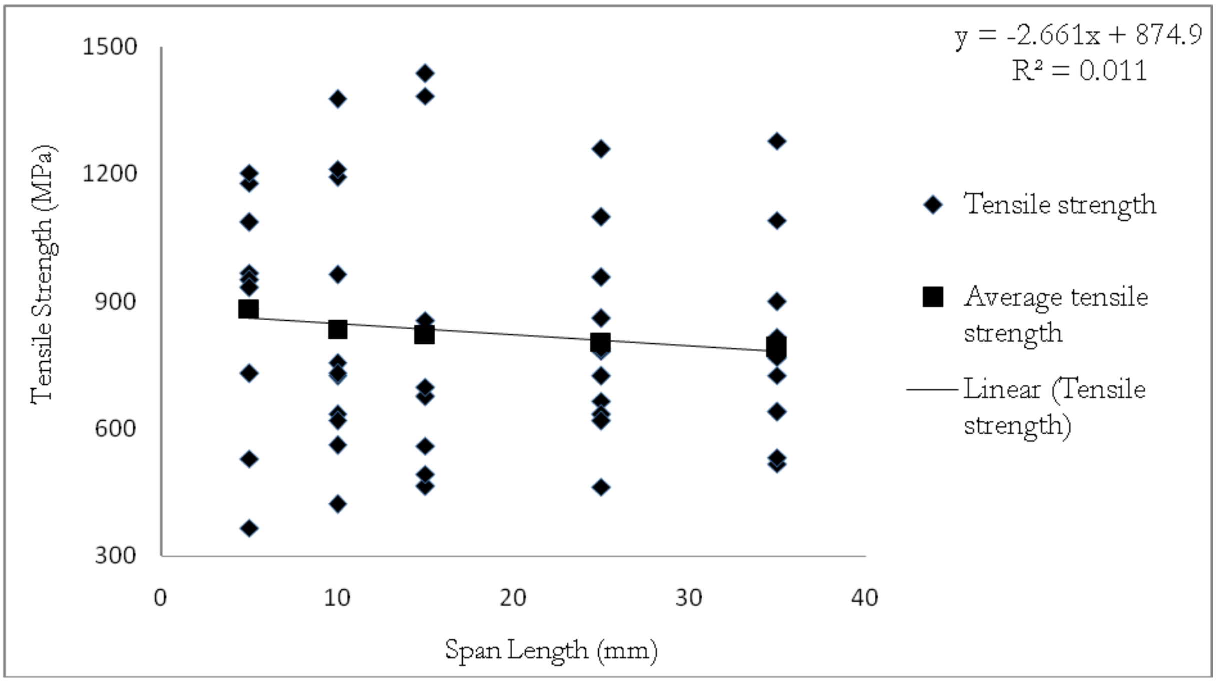


Fig. 5. Tensile strength as a function of fiber span length for control fiber.

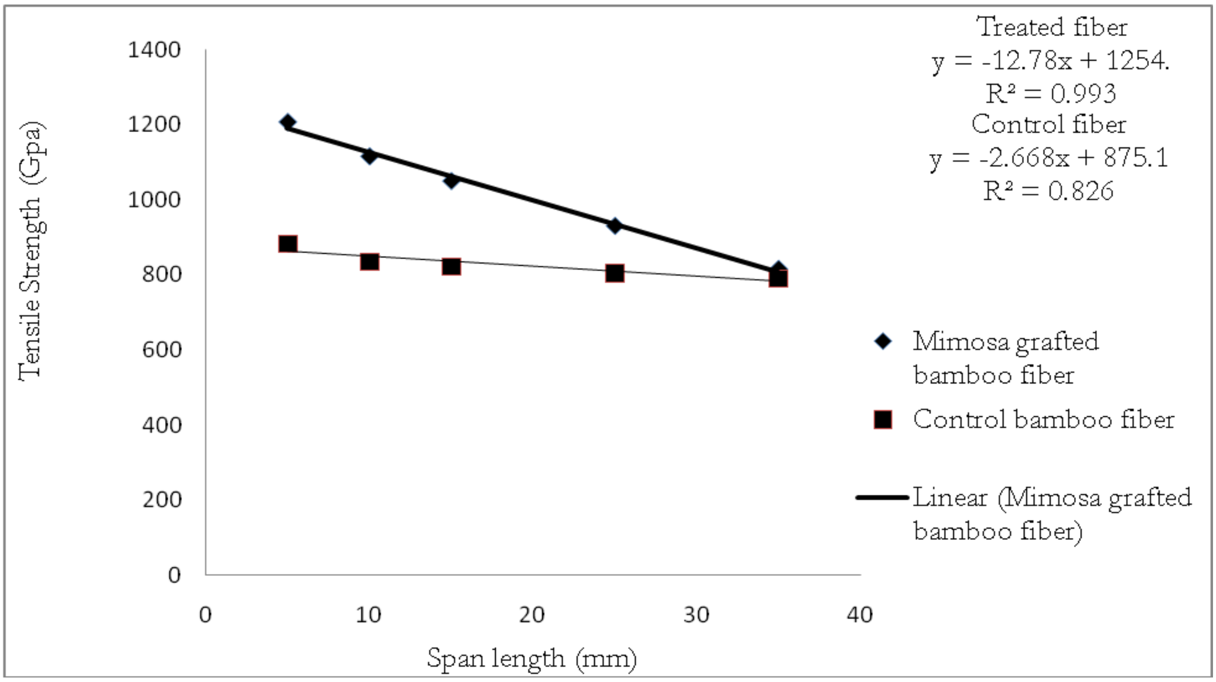


Fig. 6. Tensile strength as a function of fiber span length for control fiber and grafted fiber.

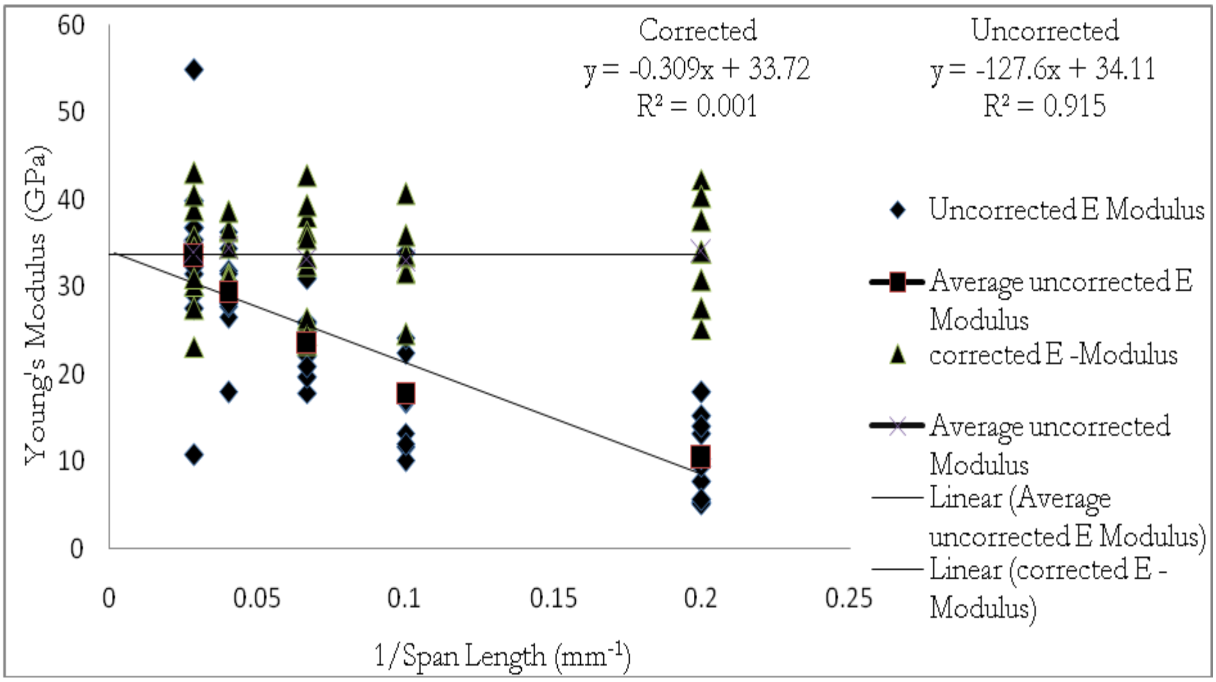


Fig. 7. Uncorrected and corrected Young's modulus vs span length for control fiber.

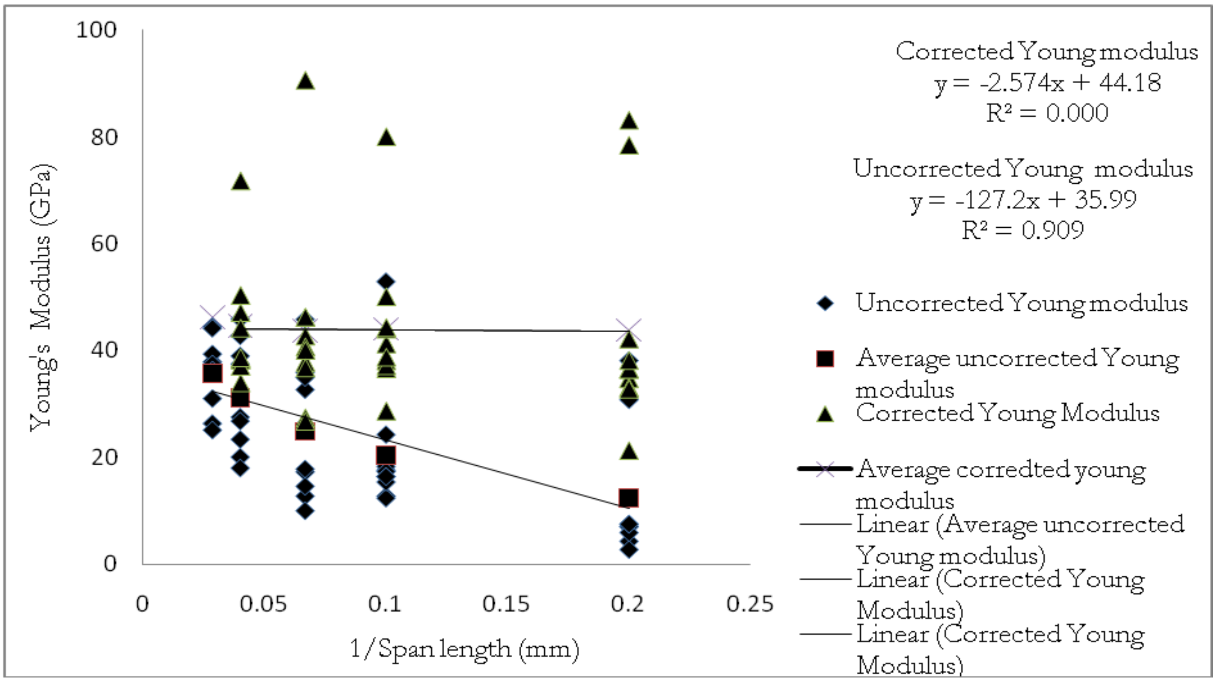


Fig. 8. Uncorrected and corrected Young's modulus vs span length for grafted fiber.

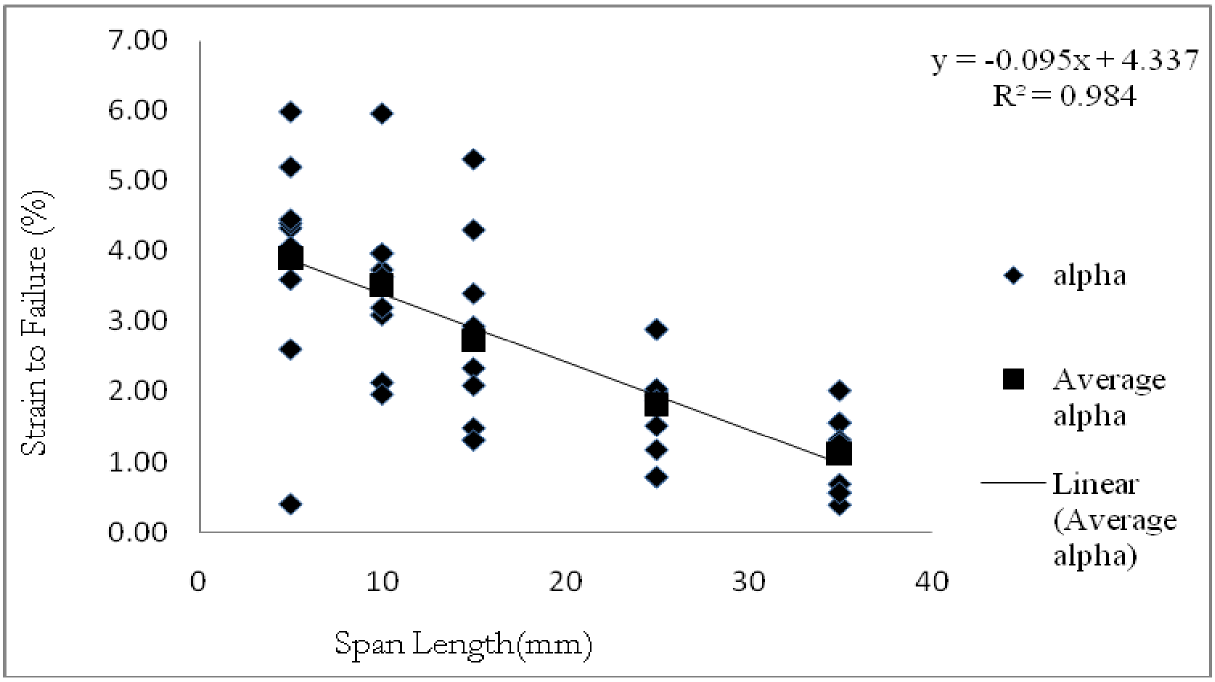


Fig. 9. Strain to failure as a function of span length for control fiber.

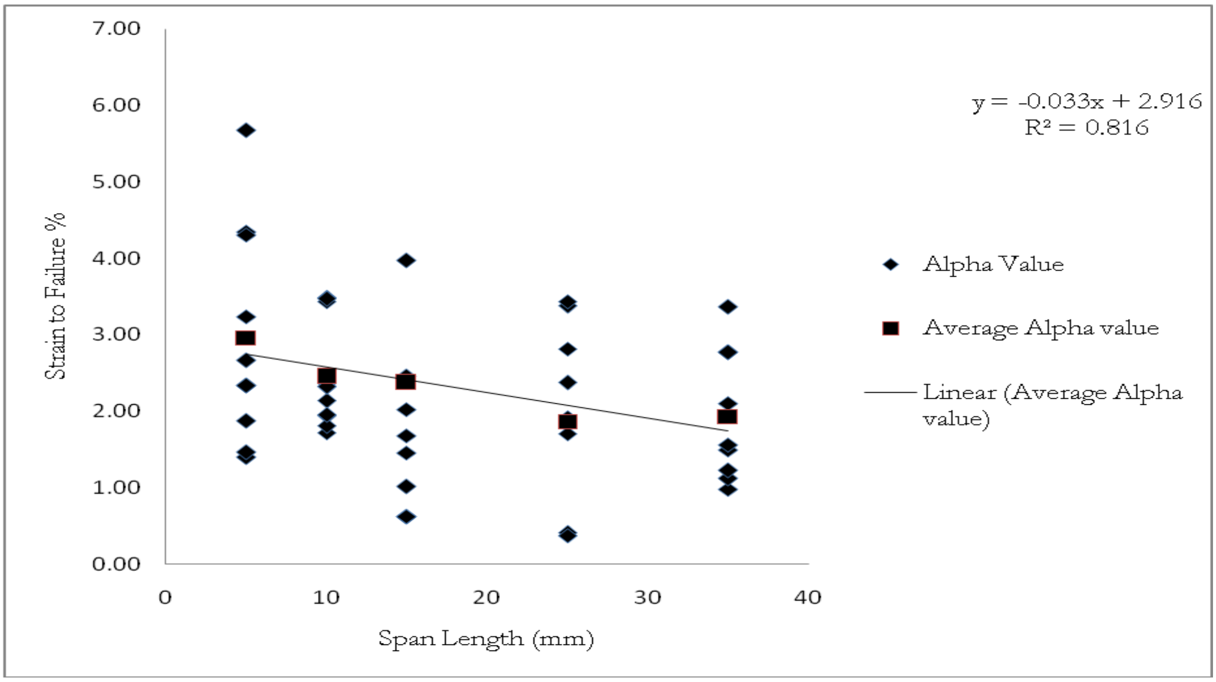


Fig. 10. Strain to failure as a function of span length for grafted fiber.

From SEM, it was observed that the anatomical features directly affect physical and mechanical properties of bamboo. To understand the properties, it is important to study its anatomical characteristics. In circular cross section, most bamboos are found hollow. Bamboo culm comprises about 40% fibers. Mimosa might react with cellulose *via* hydrogen bonds resulting in covalent bond. May be for this fixation reaction, 2-4% mimosa did not removed even in organic solvent. As a result cellulose did not revert to raw condition. In Fig. 1, the proximity of the aromatic nuclei in the mimosa structure means that free radical oxidative bond rearrangement can take place easily. As a result mimosa can react with cellulose to form regular arrangement. Fiber percentage is higher, which contributed to its superior slenderness and strength. Most fibers have a thick poly-lamellate secondary wall. Fibers in bamboos are either grouped in bundles or sheaths around the vascular bundle. This gives the high tensile strength to the bamboo fiber. For that, tensile strength of different span length fiber was measured. It was found that tensile strength decreased with increasing the fiber span length due to multicellular structural flaws, which broke the fiber easily. Uncorrected Young's modulus was dependent on span length. As Young's modulus should be independent of span length, a correction for Young's modulus values was conducted. After calculation it was found that that modulus was independent of span length. [18].

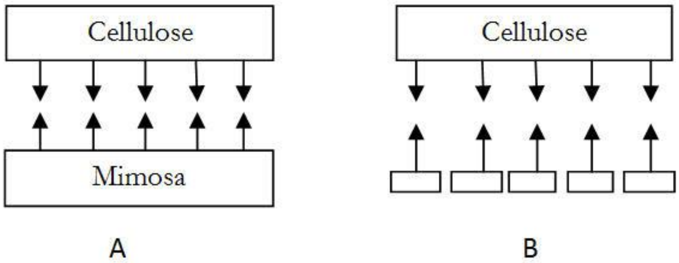


Fig. 11. Symmetric bonding diagram of cellulose and mimosa, A is more stable than B.

In longitudinal direction, tensile strength and Young's modulus was dependent on the cell wall, which was assumed to positively correlated to the content of cellulose and lignin. In grafting of mimosa, it makes bond with cellulose with 11-H of mimosa. Mimosa is a bigger molecule containing several molecular active reactive sites which are attached with cellulose. A symmetric representation of grafting of mimosa and cellulose has shown in Fig. 11. As mimosa is a long chain aromatic molecule aggregating for that this mimosa is containing several reactive groups may lead to a more stable compound. This compound is more stable compared to equivalent number of small ones each having one reacting group. Symmetric representation is shown in Fig. 11.

Figure 12 represents the possible attaching way of mimosa and cellulose. Cellulose fibers are crystallizing in that way. May be for this reason cellulose fiber is showing more tensile strength and Young's modulus is bamboo fiber. From present study it can be concluded that chemical composition and anatomy are correlated to tensile properties.

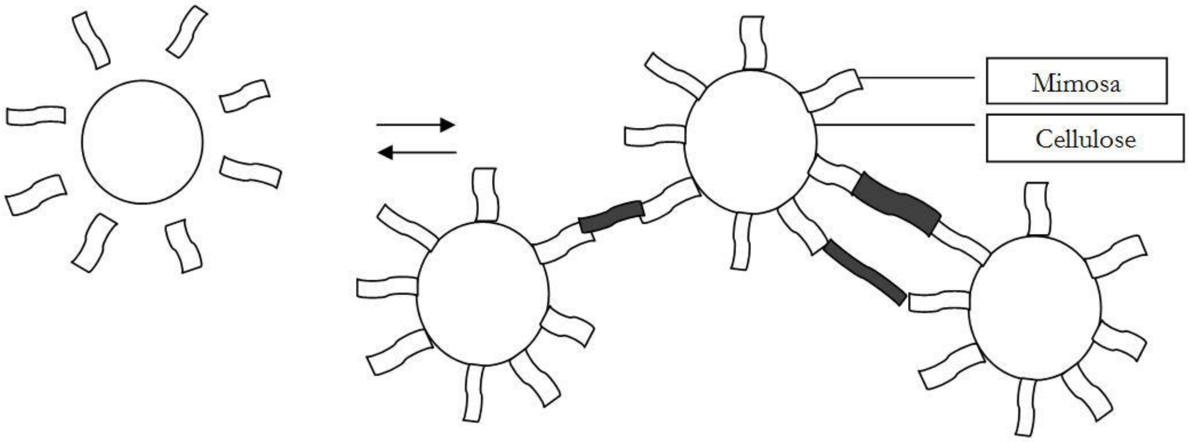


Fig. 12. A possible attaching way of bonding of mimosa and cellulose.

4. Conclusions

Depending on anatomical and mechanical properties, grafted fiber can be used for making light weight composites. The Young's modulus of grafted fiber was found to be high compared to other control fibers. The sclerenchyma cell played dominant role on cell properties of meso bamboo. This may be due to the interactions between the components in bamboo. It was observed that mimosa has played as tannins in bamboo fiber. Having higher molecular weight of mimosa, it may be become to a stable cellulosic compound. The tensile strength and Young's modulus increased, which is the desirable properties of reinforcement in composite when the bamboo fiber was grafted with mimosa. It can be concluded that the control fiber grafted with mimosa can be used as biodegradable grafting agent to obtain desirable result for composite.

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