Research Progress in Mechanical and Manufacturing Engineering Vol. 3 No. 1 (2022) 393-402
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RPMME

Homepage: http://publisher.uthm.edu.my/periodicals/index.php/rpmme
e-ISSN : 2773-4765

Effect of Tensile Strength on Treated Bamboo Fiber with Alkali Treatment Compared to Untreated Bamboo Fiber

Norhamizi Arief Abdullah[1], Mohd Yussni Hashim[1]*,

[1]Faculty of Mechanical and Manufacturing Engineering,
Universiti Tun Hussien Onn Malaysia, Batu Pahat, 86400, MALAYSIA

*Corresponding Author Designation

DOI: https://doi.org/10.30880/rpmme.2022.03.01.041
Received 15 Nov. 2021; Accepted 15 April 2022; Available online 30 July 2022

Abstract: The study systematically explored the effect of alkali concentration and soaking time on the microstructure and tensile properties of single-cellulosic. Alkali concentration (w/v%), immersion time (minute) and immersion temperature (°C) were used as a parameter in alkali treatment conditions optimization determination. Furthermore, the fiber surface morphology and composite breakage microstructure were also investigated experimentally using a scanning electron microscope. The single-cellulosic bamboo fiber was immersed in 6 wt.% a NaOH solution for soaking times of 12, 24, and 48 h. The alkali concentration and soaking time significantly affected the fiber properties. These aspects fit the new worldwide requirements for the use of renewable and sustainable resources, which warrant the further development of BFC. The tensile properties of the fiber increased after each alkali treatment. The alkali concentration and soaking time significantly affected the fiber properties. The highest tensile strength herein was 290 MPa for the single-cellulosic fiber that was soaked for 12 h in 6 wt.% NaOH. Comparatively, the tensile strength of the single-cellulosic bamboo fiber that was soaked for 24 h in 6 wt.% NaOH was 170 MPa. The tensile modulus of the single-cellulosic fiber was 30 GPa after soaking in 6 wt.% NaOH for 12 h, indicating that a strong alkali treatment negatively affected the stiffness and suitability for use of the fibers in applications. The topography of the fiber surface became much rougher after the alkali treatments due to the removal of hemicellulose and other surface impurities. The alkali treatments substantially changed the morphology of the fiber surface, suggesting an increase in wettability.

Keywords: Bamboo Fiber, Alkali Treatment, Tensile Test

1. Introduction

Bamboo is known as one of the most attractive bio-fibers because that has several advantages, such as small environmental load, renewability, rapid growth, and relatively high strength compared to other natural fibers (e.g., jute and cotton). Asian countries such as China and India produce over 80% of the worldwide availability of bamboo fiber. Bamboo fiber is a regenerated cellulosic fiber produced from bamboo. Starchy pulp is produced from bamboo stems and leaves through a process of alkaline

**Corresponding author: yusni@uthm.edu.my*

hydrolysis and multi-phase bleaching. Further chemical processes produce bamboo fiber. It is softer than cotton, with a texture similar to a blend of cashmere and silk. Because the cross-section of the fiber is filled with various micro-gaps and micro-holes, it has much better moisture absorption and ventilation. Moisture absorbency is twice than that of cotton with extraordinary soil release.

The introduction should describe general information on the subject matter area of study. It is usually arranged in such a manner to gradually bring to focus the specific motivations of the current study, the research questions, the problem statements, the hypotheses, the objectives, as well as the expected outcome.

Cellulose, hemicelluloses, and lignin are the three major chemical compositions of bamboo, and they are closely associated in a complex structure [1]. They contribute about 90% of the total bamboo mass. The minor components are pigments, tannins, protein, fat, pectin and ash. Others include resins, waxes, and inorganic salts. These constituents play an important role in the physiological activity of bamboo, and they are found in cell cavities or special organelles [2]. The chemical composition of bamboo is known to be similar to that of wood, but bamboo has a higher content of minor components compared with wood [4]. There is variation in the chemical composition of bamboo depending on its age. Notably, cellulose content decreases with an increase in the age of bamboo [5].

Bamboo has one of the most favourable combinations of low density and high mechanical strength, that is, it has high particular stiffness and strength. According to several authors,21–25 bamboo fiber bundles are distributed densely in the outer region of the culm wall and sparsely in the inner region, and also concentrated in the upper part of the culm compared with the base.

Sodium hydroxide, also known as lye and caustic soda, is an inorganic compound with the formula NaOH. It is a white solid ionic compound consisting of sodium cations Na^+ and hydroxide anions OH^-.Sodium hydroxide is a highly caustic base and alkali that decomposes proteins at ordinary ambient temperatures and may cause severe chemical burns. It is highly soluble in water, and readily absorbs moisture and carbon dioxide from the air. Sodium hydroxide is used as a treatment to modify of natural-based fillers or reinforce hybrid composites. This treatment can be used to modify hydrophilic properties and thus improve the dimensional stability of composites.

The alkaline treatment or mercerization is a chemical treatment in which the natural fibers are immersed in a known concentration of aqueous sodium hydroxide (NaOH) for a given temperature and a time. Chemical treatment was commonly used is sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2) and calcium hydroxide [6]. The alkaline treatment modifies the surface of fibers by removing a certain rate of lignin, hemicellulose, wax, and oils covering the external surface of natural fibers [7]. The partially removed lignin wax and hemicellulose enhance the matrix–fiber interface and ensure good adhesion between the matrix and natural fibers. If the treatment parameters are not optimized, the mercerization can cause fiber defibrillation, pore formation, and fiber embrittlement [8, 9]

The addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide [10]:

$$(\mathrm{Fiber} - \mathrm{OH} + \mathrm{NaOH} \rightarrow \mathrm{Fiber} - \mathrm{O} - \mathrm{Na} + \mathrm{H_2O}) \quad \text{Eq 1}$$

2. Materials and Method

This section explains in detail the equipment and materials used in the experimental work. It is covered with bamboo fiber and sodium hydroxide pallet.

2.1 Tensile strength

The specified requirement is referred to as the dimension according to the American Society for Testing and Materials (ASTM) standard. Therefore, the tensile test specimen follows the ASTM C1557-03 as shown in Figure 1 for each treatment condition [44]. The preparation of specimen is selected based on single fiber testing due to a common type of test specimen from randomly bast and bundle fiber used for the tensile test.

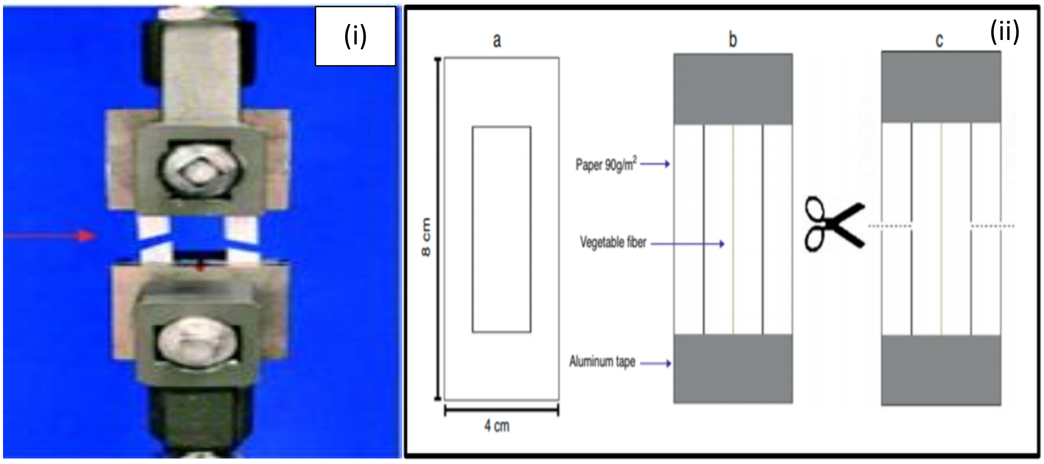

Figure 1 (i) ASTM C1557-03 specimen grip, (ii) Preparation of specimen for the tensile test: (a) paper for fixing the fibers, (b) fibers fixed in the paper and (c) detail of the cutting of the paper before the test.

Bamboo fibers are fixed into a paper tab as the frame to hold the fiber by binding using aluminum and double-sided tape. These specimens are similar to studies conducted by Ernestina (2013) but different in terms of gauge length (20 mm instead of 50 mm) [4]. The schematic diagram with dimensions as per ASTM standard is shown in Figure 3.2, while the tensile test specimen is mounted on the paper tab.

Table 1 Dimension of the tensile test specimen

Details	Dimension (mm)
Length (L)	80
Width (W)	40
Thickness (T)	90 g/m^2
Gauge length	20
Discontinuous line (centre)	40
Position of fiber (centre)	20
Aluminium and double-sided tape	30 (both side)

2.2 Surface morphology

The surface morphology of bamboo fiber at various alkali treatment conditions was examined using a scanning electron microscope (SEM), model: JEOL - JSM6380LA as shown in Figure 3 to Figure 5. The bamboo fiber was randomly cut into appropriate lengths and placed into SEM specimen stub.

2.3 Fiber Density

Both conditions treated and untreated were tested to get the density. It was randomly selected and cut into short fibre to get the density. This experiment is run using Mettler Toledo analytical balance (Model XS64) to measure weight in the air and weight in the water. This experiment applied the Archimedes principle which is the upward buoyant force that is exerted on a body immersed in a fluid is equal to the weight of the fluid that the body displaces.

3. Result and discussion

The impact of alkali treatment conditions on bamboo physical properties was investigated to achieve the first research objective. Three bamboo fiber physical properties at various soaking times were selected as responses, which are fiber density, fiber diameter, fiber cross-section area, and fiber morphology. The experimental results were discussed in next following sub-section.

3.1 Fiber density

The untreated bamboo fiber had a higher density than alkali-treated bamboo fiber, because it may contain soluble substances from fiber, such as lignin, hemicellulose, oils, waxes, and impurities. Lignin, soluble substance, and other impurities are normally removed after the alkali treatment [9, 10].

Table 2 The density of untreated and alkali-treated bamboo fiber-based on [56]

Bamboo fiber Density Changes (g/cm^3)	Time (Hours)	Temperature Level (°C) Room temperature (±27) Alkali Concentration (w/v 6%)	References
Average	untreated	1.158	[8]
	12	0.91	[10]
	24	0.77	[10]
	48	0.67	[10]

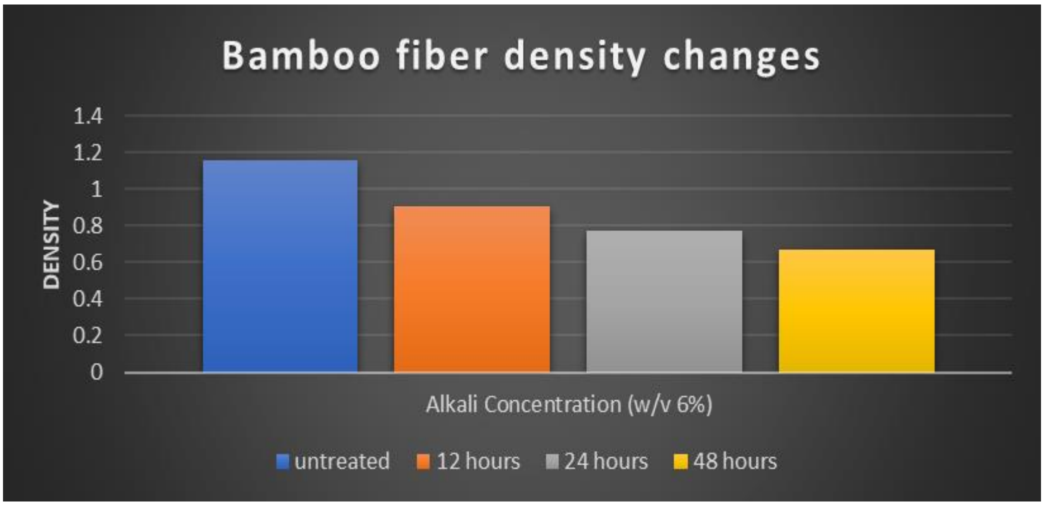

Figure 2 Value of fiber density measured between untreated and after treated with various alkali treatment condition.

3.2 Fiber morphology

The surface morphology of the bamboo fiber after various alkali treatment conditions was studied by scanning electron microscope (SEM, ZEISS/Carl ZEIS). The fractured surface of the tensile test samples was used with the SEM observations on an instrument operated at an accelerating voltage of 20 kV [7].

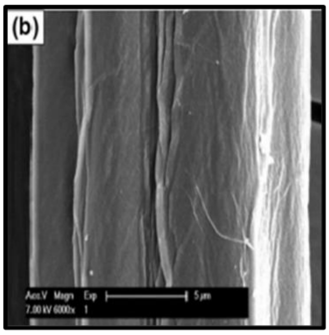

Figure 3 SEM images of the surface morphology of individual bamboo fiber treated with 6% NaOH for 12 hours [7]

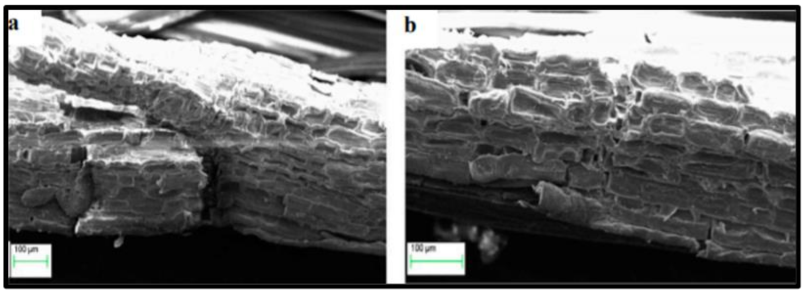

Figure 4 SEM images of the surface for 24 h in 6 wt.% NaOH showing in (a), and (b)

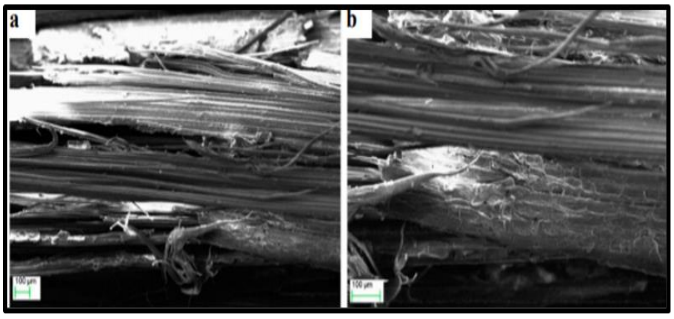

Figure 5 SEM images of the surface for 48 h in 6 wt.% NaOH showing in (a), and (b)

An excessive alkali treatment and soaking time can damage the strength of single cellulosic bamboo fiber strips, suggesting that these variables are also unsuitable for treating bamboo fibers as shown in Figure 6. The decrease in strength for higher alkali concentrations can also be attributed to the partial removal of cellulose. Hence, excessive alkali treatment caused damage to the mechanical properties of single bamboo fiber and was deemed unsuitable for treating bamboo fibers. To conclude, 6 wt% NAOH treated bamboo fibers were more suitable for the fabrication of bamboo fiber/epoxy composites, which was also supported by a single fiber tensile test [9].

3.3 Fiber diameter

The bamboo fiber was selected and separated from the bundle fiber and finally separated into single fibers. The bamboo fiber diameter was measured at 4.5 mm intervals, along its length, using a Leica Stereo Microscopic video analyser 2000. The mean diameter of single bamboo fiber strips was measured from the average of ten fibers, in which the diameter for every fiber was the average value of the diameter measured ten times at 4.5 mm intervals along the fiber length. The measurement was replicated three times for each treatment condition [6].

Table 3 Value of fiber diameter changes measured between untreated and after being treated with various alkali treatment conditions

Bamboo fiber diameter changes (µm)	Time (Hours)	Temperature Level (°C) Room temperature (±27) Alkali Concentration (w/v 6%)	References
Average	untreated	140.82	[9]
	12	124.83	
	24	120.81	[10]
	48	112.82	

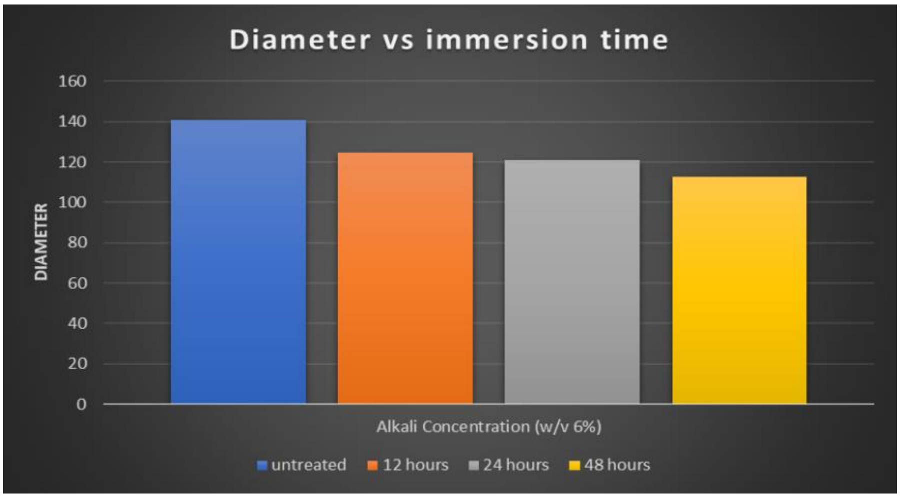

Figure 6 bar chart of fiber diameter changes measured between untreated and after treated with various alkali treatment condition.

Table 3 shows the decrement pattern of bamboo fiber diameter after treated at various alkali treatment conditions. Similarly, at 12- and 24-hours immersion time, the percentage of single bamboo fiber diameter decrement was 124.83 and 120.81. The fiber diameter decreased with the increment of immersion duration. For the standard deviation value, the range was small and it was about 10 ~ 20. From the bar chart (Figure 6), it was clearly understood that at constant immersion temperature and immersion time, an increment of alkali concentration reduced the diameter of bamboo fiber.

4.4 Fiber tensile test

The mechanical properties of bamboo fibers are affected by many factors, such as growth environment, growth years, and fiber extraction method [9]. Therefore, it is necessary to measure the mechanical properties of bamboo fibers used in this study. Considering the severely unstable mechanical properties of bamboo fibers, a single fiber tensile test was carried out for 3 specimens to achieve a valid average. The results were summarized in Table 4 and Table 5.

Table 4 Tensile strength of the single-cellulosic bamboo fiber strips subjected at 6% NaOH alkali concentrations, for various soaking times.

Bamboo fiber tensile strength (MPa)	Time (Hours)	Temperature Level (°C)	References
		Room temperature (±27) Alkali Concentration (w/v 6%)	
Average	0	262	[50]
	12	290	
	24	170	
	48	275	[61]

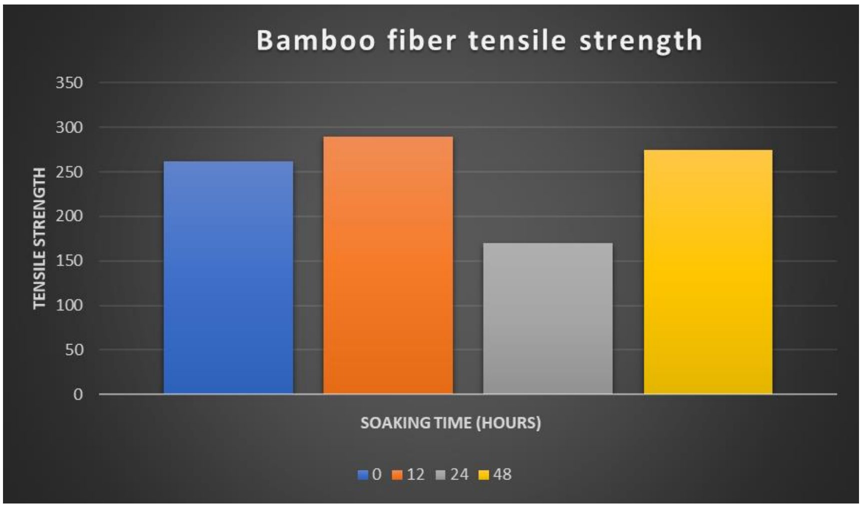

Figure 7 Bar chart of tensile strength of the single-cellulosic bamboo fiber strips subjected at different 6% NaOH concentrations, for various soaking times

The tensile property of alkali-treated fiber increased with the soaking time due to the elimination of impurities and cementing substances from the interfibrillar region of the fiber surface. However, excess delignification at 48 h did not yield an improvement in the tensile property. Instead, a reduction in ultimate tensile strength (81 %) and modulus (42 %) were observed at 48 h of soaking time, which could be attributed to an excessive elimination of lignin that bonded the cellulose together, which weakened the fiber structure [40]. The tensile strength of the composites is improved by the NaOH treatment. However, too high of alkali concentration can cause an excess removal of covering materials from the cellulose surface, which results in weakening or damaging of the fiber structure [59].

4.5 Fiber young modulus

As shown in Figure 9, the trend of the tensile modulus is similar to that of the tensile strength, and the tensile modulus increased smoothly up to the 6 wt.% concentration; moreover, the effect of the treatment time seemed to be significant. The optimum increase in the tensile modulus was achieved at a 6 wt.% concentration and 12 h of soaking [50].

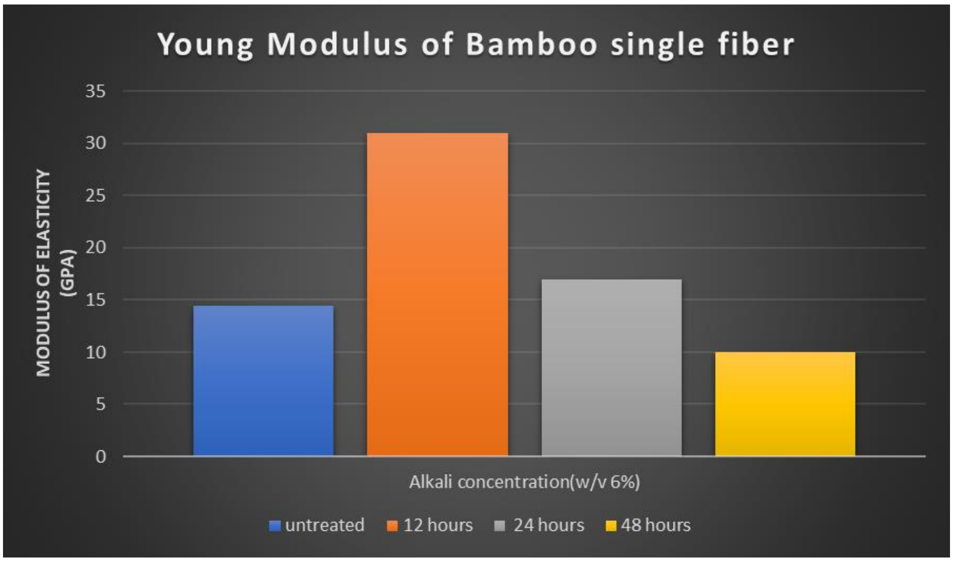

Figure 9 Bar chart tensile modulus of the single-cellulosic bamboo fibres strips subjected at 6% NaOH concentrations, for various soaking times.

However, after 6 wt.% NaOH treatment, the tensile strength and the Young's modulus of bamboo fibers increased by 38% and 14%, respectively according to [6]. According to Refs. [8, 9] moderate alkali treatment could effectively remove the hemicellulose and lignin in bamboo fibers, so that cellulose crystallinity increased, which usually improved both fiber tensile strength and modulus. For higher concentrations of NaOH treatment, the bamboo fibers remained at a certain tensile strength, but there was a significant decrease in Young's modulus, which was also found in other relevant studies [9].

Table 5 Tensile modulus of the single-cellulosic bamboo fiber strips subjected at 6% NaOH concentrations, for various soaking times.

Bamboo fiber Modulus of elasticity (GPa)	Time (Hours)	Temperature Level (°C) Room temperature (±27) Alkali Concentration (w/v 6%)	References
Average	untreated	14.48	[51]
	12	30	[50]
	24	17	[50]
	48	10	[64]

4. Conclusion

The study systematically explored the effect of alkali concentration and soaking time on the microstructure and tensile properties of single-cellulosic. Alkali concentration (w/v%), immersion time (minute), and immersion temperature (°C) were used as a parameter in alkali treatment conditions optimization determination. The single-cellulosic bamboo fiber was immersed in 6 wt.% NaOH solutions for soaking times of 12, 24, and 48 h. The alkali concentration and soaking time significantly affected the fiber properties.

In this study, 6 wt.% NaOH treated bamboo fibers were proven to be optimal for the fabrication of bamboo-fiber composites by a single fiber tensile test and scanning electron microscopy (SEM). The results showed that the optimum tensile strength was obtained at 6 wt.% alkali concentrations/12 h soaking time. The optimum tensile modulus (31 GPa) was shared between the water retting condition and 6 wt.% alkali concentrations/12 h soaking time. Insufficient chemical soaking time decreased the tensile properties.

Acknowledgment

The authors would also like to thank the Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia which has supported the accomplishment of the research activity.

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