International Journal of Technology (2012) 1: 16-23
ISSN 2086-9614

MECHANICAL PROPERTIES AND MICROSTRUCTURE OF METROXYLON SAGO FIBER TREATED BY SODIUM HYDROXIDE

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(Received: May 2011 / Revised: October 2011 / Accepted: December 2011)

ABSTRACT
In the present study, natural fibers located in thick outer woody rinds of the metroxylon sago (MS) tree were investigated. The investigation focused on measuring the mechanical properties and observing the microstructures of MS fibers before and after treatment with 5% sodium hydroxide. A scanning electron microscope was used to observe the microstructure of MS fiber, and the results showed that there was a decrease in fiber diameter after mercerization. A porous structure in the cross-section area of untreated fibers was clearly seen, and it was highly compressed after mercerization. The strength of MS fiber increased significantly after it was treated by 5% NaOH solution for two hours. The average ultimate strength of untreated MS fiber was recorded as 46 MPa; treatment with sodium hydroxide resulted in a significant increase in average ultimate strength to 163 MPa. Additionally, the elastic modulus of treated fiber was greater than that of untreated fiber.

Keywords: Mechanical properties; Microstructure; Natural fibers

1. INTRODUCTION
Many metroxylon sago plants grow well in tropical countries such as Indonesia. As reported by Singhal et al. (2008), wild sago palms grow in Indonesia in an area roughly estimated as more than 7 million ha in Sumatera, Papua, and Kalimantan. Sago palm is a species of the genus metroxylon belonging to the Palmae family; it reaches a maximum height of 25 m and a diameter of 40 cm. The most useful section of the sago palm tree is the trunk, where starch can accumulate until the flowering stage. Sago starch is an ingredient in various food products such as sago meal, noodles, biscuits, and desserts (Singhal et al., 2008). Additionally, sago starch can be used to produce adhesive material for paper, textiles, and plywood. Starch in the sago trunk is generally obtained from extraction of the pith, which is enclosed by an outer woody rind that is approximately 50 mm thick and light lengthways so that the soft pith can be separated using a sharpened knife.

The woody rind usually becomes waste, but an abundance of fibers is typically embedded in such waste bark. Therefore, we were interested in researching the sago palm fiber that lies in the woody rind as potentially useful material for the reinforcement of a natural fiber composite. Many investigations of the sago palm have been conducted; the majority focuses on sago starch (Karim et al., 2008; Nawang et al., 2001; Sopade & Kiaka, 2001). Karim et al. (2008) reported the effects of alkali on the pasting properties of sago starch, as well as modifications in gel properties on cooling and storage.

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Singhal et al. (2008) also intensively reviewed the industrial production, processing, and utilization of products derived from the sago palm. Fibers of palm sago belong to a group of cellulose-based natural fibers, which includes jute, coir pineapple, oil palm, banana, etc. Many methods have been developed to improve the mechanical properties of natural fibers (Abral et al., 2009; Bachtiar et al., 2008; Vallo et al., 2004; Cao et al., 2006). However, an investigation of properties of MS fibers treated with alkali was not found in references. It is well known that the mechanical properties of natural fibers can be improved by chemical treatment with sodium hydroxide during mercerization (Gassan & Bledzki, 1999; Vilay et al., 2008). As reported in some literatures, the treatment of natural fibers with mercerization resulted in an increase in tensile strength and tensile modulus of the fibers (Gañán & Mondragon, 2005; Rong et al., 2001). Therefore, the objective of this study was to investigate the properties of palm sago treated with 5% sodium hydroxide solution due to common alkali concentration in order to observe any significant effects on natural fiber properties (Symington et al., 2009). The objective of MS fiber research is to obtain important information about the characteristics of sago palm fiber treated by alkali solution.

2. METHODOLOGY

2.1. Resource of fibers

Sago palm used in this study was obtained locally from Padang, Sumatera Barat, Indonesia. Outer woody rind of sago has typically been considered useless after the spongy inner pith has been taken for animal food. Therefore, interesting fibers obtained in this investigation were taken from the same trunk cortex of the palm sago; we observed that the outer woody rind, approximately 50 mm thick, enclosed a spongy inner pith containing a high proportion of starch. First, the rind had to be cleaned from residual pith with a steel wire brush so that the fibers were easily pulled out from the tree bark. Then, the fibers were rinsed with water many times to remove residual impurities. The cleaned fibers were subsequently dried for 48 hours using low humidity air from a dehumidifier. Dried fibers were classified as untreated candidates for further testing.

2.2. Treatment with 5% sodium hydroxide

Untreated fibers were immersed in a 5% sodium hydroxide solution at room temperature for two hours; they were additionally neutralized several times with fresh water for three hours to remove any traces of alkali solution until a neutral pH was obtained. The cleaned fibers were then dried continuously by using low humidity air produced by a dehumidifier for 24 hours. The dried fibers were classified as treated.

2.3. Measuring fiber length and diameter

Diameters of the untreated and treated fibers were measured to assess any changes in fiber dimensions due to the effect of alkali treatment. Pictures of fiber diameter were taken by using the optical microscope; further, fiber diameter was measured. Diameters of the fibers were needed in calculations of fiber cross-sectional areas to measure strength. The lengths of fibers were measured to calculate fiber strain by using a vernier caliper with an accuracy of 0.01 mm.

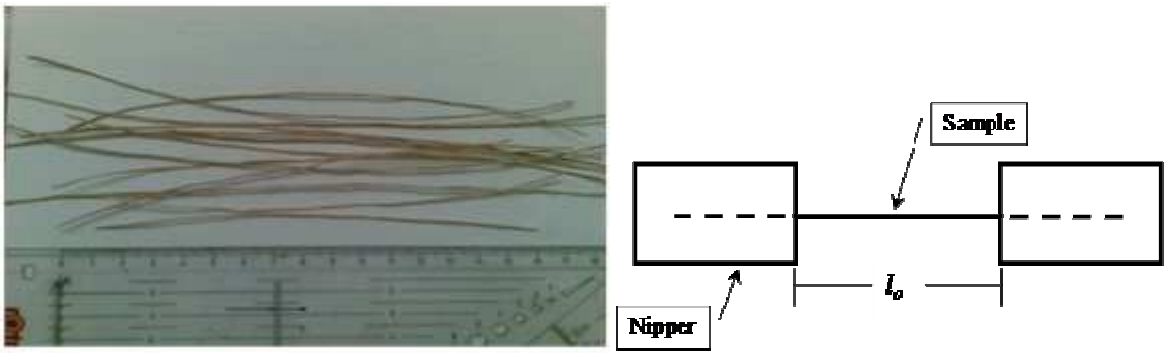
2.4. Scanning electron microscope (SEM)

SEM apparatus used in the research was a product of LEO 420i (Leica Electron Optics, Cambridge Instruments Ltd., Cambridge, UK). The samples were initially vacuumed to ensure that no residual impurities affected the investigation results.

2.5. Tensile testing

Figure 1a shows some samples of MS fibers. Each untreated and treated fiber diameter was

measured in five different locations to obtain the average diameter used to calculate fiber strength. Figure 1b exhibits the schema of tensile testing in which MS fiber was clamped in a tensile machine; initial length was *lo*=70 mm. Tensile testing of MS fibers was carried out using a tensile machine produced by Com-Ten Industries (USA). The applied load was measured using a micro-load cell attached to the machine. Tensile testing included subjecting sago palm fiber to a tensile speed of 5 mm/min at room temperature. In order to measure the stress-strain diagram, the apparatus was equipped with data acquisition software developed under the ANSI C programming language. Average diameter and initial length lo were input to the software that had been installed on an integrated computer, so that both a force-deflection curve and a stress versus strain diagram could be obtained automatically and precisely from the apparatus.



(a) (b)

Figure 1 (a) Some MS fiber samples (b) Scheme of tensile testing while MS fiber was clamped in tensile equipment

3. RESULTS AND DISCUSSION

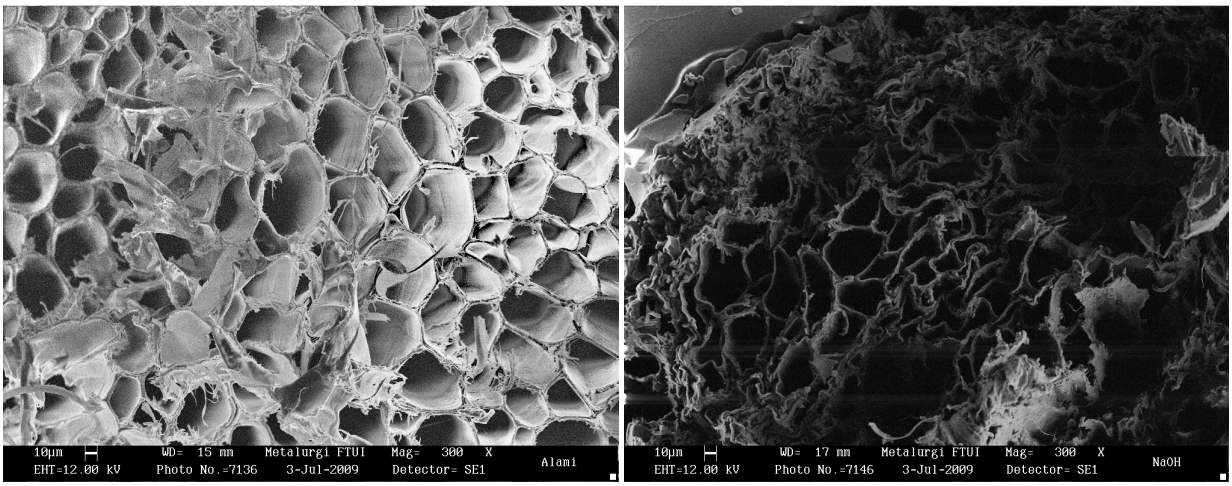
3.1. Cross-section of the fibers

For this study, 30 pieces of prepared fibers were evenly divided into two groups: untreated fibers and treated fibers. The average diameter of the untreated fibers was measured at 1.07 mm, while the diameter for treated fibers was 0.86 mm. The decrease in fiber diameter shows the effect of alkali treatment, which collapsed the cellular structure of the sago palm fibers. Some published references report fiber shrinkage of cellulose material following chemical treatments, such as sodium hydroxide treatments (Vallo et al., 2004; Cao et al., 2006). The average diameter of fibers processed by alkali treatment was significantly lower than that of untreated ones.

3.2. SEM micrograph of the MS fibers

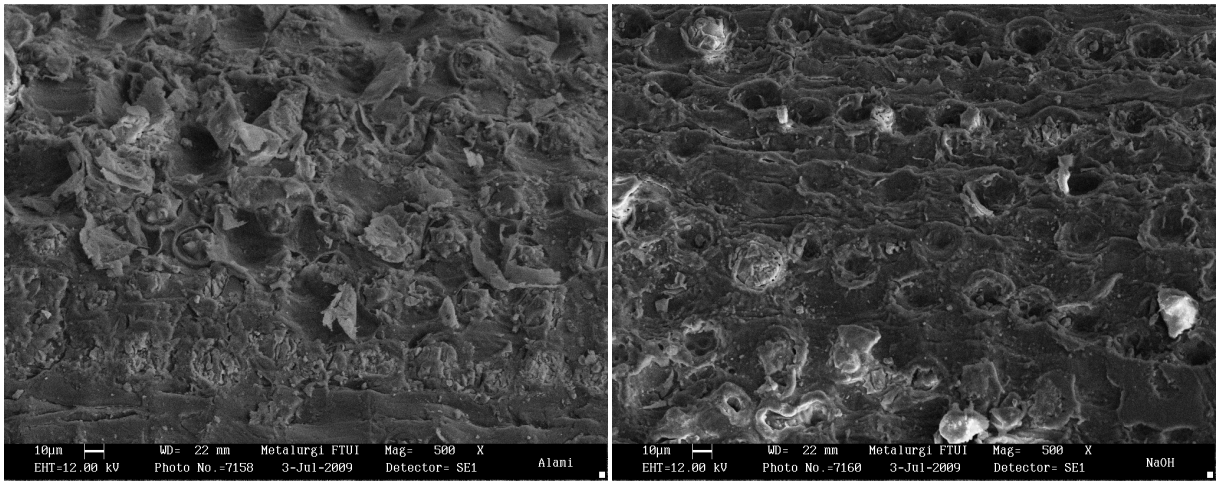
Figure 2 reveals SEM micrographs of cross-sections of untreated and treated MS fibers after tensile testing. As shown in Figure 2a, MS fiber is characterized by more hollow structures consisting of multi-cell walls than other natural fibers (Cao et al., 2006). Multi-cell walls were packed together; between them, the middle lamella that largely consists of pertinacious substances was found (Robin et al., 2009). Similar appearance was presented also by cellular structure of untreated bagasse fiber (Vilay et al., 2008). Figure 2b shows the condition of the cellular structure of MS fiber treated with a solution of 5% sodium hydroxide for two hours. In the photograph, the individual cells of such fibers collapsed after mercerization and appeared more compacted following treatment with sodium hydroxide. According to Bledzki and Gassan (1999), the compacting of cellulose affects the mechanical properties of the fibers, enhancing tensile strength and Young's modulus of the fibers. Figure 3 presents the surface morphology of untreated and treated MS fibers, and it is clear that the surface of virgin natural fiber (Figure 3a) differs significantly from that of treated MS fiber (Figure 3b). Untreated MS fiber shows a

relatively smooth surface and the treated one exhibits a rougher surface. According to Gañán and Mondragon, (2005), untreated natural fiber contains natural and artificial impurities. For impurities still seen on the untreated MS fiber surface, removing this layer chemically with alkali exposed a rougher surface (Vallo et al., 2004). As presented in Figure 4, the ultimate strength of all treated fibers was evidently higher then that of untreated ones.



(a) (b)

Figure 2 SEM micrograph of fracture surface after tensile testing of (a) untreated and (b) treated MS fibers (mag. 300X)



(a) (b)

Figure 3 SEM micrograph of the surface of MS fibers: (a) untreated and (b) treated

3.3. Effect of alkali treatment on mechanical properties

Changes to mechanical properties of lignocellulosic material due to mercerization have been well known. Using a NaOH solution is one of the usual methods for improving tensile strength and modulus elasticity of natural fiber. In this study, NaOH solution was applied in the treatment of MS sago fibers to study the effect of alkali on their behaviors. Figure 4 displays maximum strength for 30 pieces of MS fiber before and after chemical treatment. The strength values of all treated MS fibers are higher than those of the untreated ones. All untreated fibers present similar values in ultimate strength. Further, treated MS fibers exhibit more fluctuations in tensile stress values.

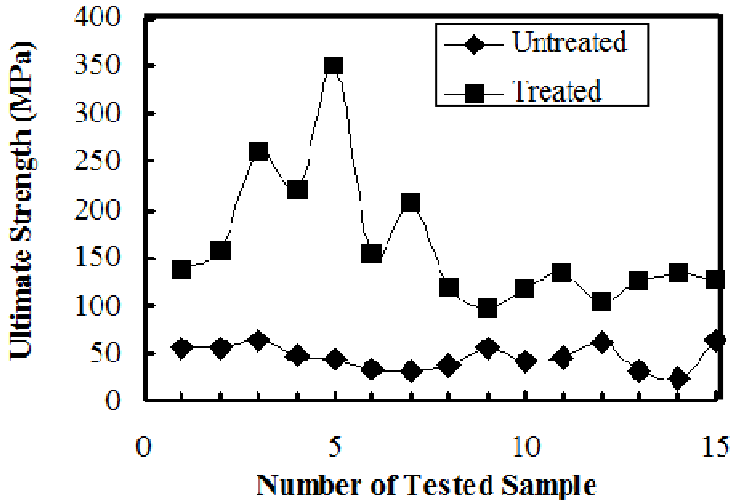


Figure 4 Tensile strength of MS fibers before and after sodium hydroxide treatment

3.3.1. Stress-strain characteristic

3.3.1.1. Untreated MS fiber

Figure 5 presents the characteristics of the stress-strain curve for MS fibers. Ultimate strength of material is usually obtained from the maximum point in a stress-strain graph. In Figure 5, two kinds of characteristics are shown based on whether MS fibers were treated or untreated. Based on the curve, untreated MS fiber shows the stress-strain curve in straight linear form. All of the tested MS fibers show no evidence of plastic deformation areas on the stress-strain curve, indicating that untreated stress-strain MS fiber behaves like an elastic material (Abral, 2009). For untreated fibers, as shown in Figure 5, ultimate and fracture strength lie in a similar point that indicates no necking area, as presented in a stress-strain curve of low carbon steel. The average ultimate strength for 15 pieces of MS fiber tested without alkali treatment was measured as 46 MPa.

The usual method for measuring the elastic modulus from a stress-strain curve was carried out by determining the slope of the stress-strain axis (Figure 5). The elastic modulus for the untreated MS fibers was estimated as 3 GPa. A purely elastic characteristic revealed by untreated MS fibers was possibly affected by existing hemicelluloses and lignin surrounding the microfibrils of MS fiber. Hemicellulose between untreated microfibrils was applied as a bonding agent and restricted slippage to an extent (Goda et al., 2006). Therefore, the untreated MS fibers behave like a highly elastic material, as shown in Figure 5.

3.3.1.2. Treated MS fiber

Conversely, a form of stress-strain behavior of treated MS fiber was presented in Figure 5. Compared to the stress-strain graphic of untreated MS fiber, it is clearly seen that treated MS fiber presents elastic and plastic deformation. The elastic stage was shown by a straight line that moves from point zero on the stress-strain axis. After achieving the maximum value in the elastic region, the MS fiber then enters what may be considered the plastic zone. Similar characteristics were also observed in ramie fiber, as researched by Goda et al. (2006). Hence, there is no necking point seen in Figure 5; the ultimate and fracture strengths lie in the same point without exhibiting a necking level. Similar behavior of the stress-strain curve was exhibited by the 15 pieces of treated MS fibers tested in the current study. The average fracture strength of MS treated fibers was measured at 163 MPa, representing an average tensile strength improvement of 254% compared to that of untreated fibers (46MPa). Clearly, there

was a significant increase of MS fiber strength after alkali treatment. In order to measure the elastic modulus, the slope between the stress-strain axis was determined by making a straight line from zero up to the maximum point in the elastic region. Compared to the elastic region for untreated MS fiber, there was a significant improvement in the slope in the elastic area, meaning that the Young's modulus of MS fiber increased due to alkali treatment. From the 15 samples observed, the average Young's modulus was estimated to be 50 GPa.

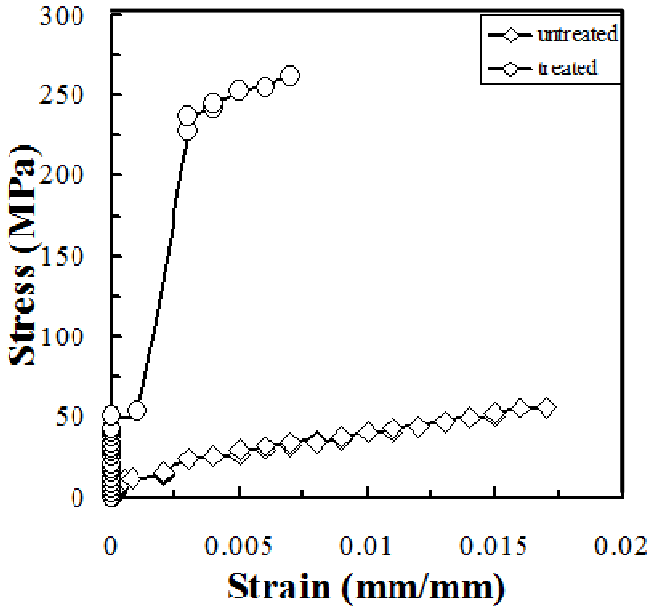


Figure 5 Stress-strain curve of MS fibers before and after sodium hydroxide treatment

Increases of the elastic modulus and tensile strength after mercerization were affected by changing the microstructure and chemical composition in MS fiber (Gassan & Bledzki, 1999). Changes in cellulose crystallinity and orientation of molecular chains of MS fiber improved the strength and elastic modulus of treated MS fiber (Sreekala et al., 2001). Similar results were observed on other cellulosic material treated by chemical solution (Rahman & Khan, 2007). As reported previously by Gañán and Mondragon (2005), there was improvement in fique fiber tensile strength after mercerization due to changes in the microfibrillar angle by the load application. Because of alkali treatment, the crystallinity region decreased in size and, conversely, the amorphous section in the microfibril increased (Goda et al., 2006; Gassan & Bledzki, 1999). Elimination of microvoids as presented in the SEM micrograph (Figure 2b), increased fiber homogeneity and enhanced the tensile strength of natural fiber treated chemically (Rahman & Kahn, 2007). Since the elimination of microvoids reduces the diameter of MS fibers, as explained in Section 3.1 above, treated MS fiber is thereby strengthened significantly.

4. CONCLUSION

The present study has provided some understanding of the alkali effect on the fiber of metroxylon sago palm fiber. Alkali treatment of 5% sodium hydroxide solution on palm sago fibers changed their mechanical properties and microstructures. The untreated MS fiber behaved as a purely elastic material; meanwhile, treated fibers revealed not only an elastic area but also a plastic zone. The average diameter of treated fibers decreased in comparison to the untreated ones. Cellulose shrinkage was evident in the SEM micrograph, and the cellulose of untreated MS fiber exhibited structures that were more porous, while cellulose in treated MS fiber was destroyed and/or compacted after mercerization. The change in microstructure was

attributed to mercerization resulting in increased mechanical properties of MS fiber. From tensile testing of the untreated sago palm fiber, the ultimate strength was recorded at only 46 MPa. After fibers were treated with 5% NaOH solution, the tensile strength of the fibers significantly increased. Of 15 samples of treated sago palm fibers, all exhibited increased tensile strength over that of the untreated fibers. Thus, the average ultimate strength of 163 MPa for the treated fibers was obtained. Treated fibers showed tensile strength improvement of 254% compared to untreated fibers. Additionally, the elastic modulus of MS fibers increased significantly after alkali treatment. Therefore, the abundant MS fiber, which has previously been discarded as waste, may be used as a valuable alternative reinforcement material for natural fiber composite.

5. ACKNOWLEDGEMENT

The authors are grateful to Directorate General of Higher Education, Ministry of National Education (Dikti), Indonesia for the financial support of this research. We are also appreciative to all Mechanical Engineering students of Universitas Andalas who have assisted in completion of the present work.

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