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TENSILE STRENGTH OF SINGLE CONTINUOUS FIBER EXTRACTED FROM *MENGKUANG* LEAVES

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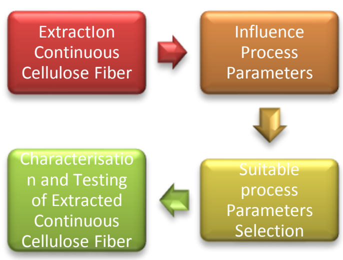
Graphical abstract



Abstract

The focus of this paper is to obtain a continuous cellulose fiber (CCF) from *mengkuang* leaves of the pandanus genus using chemical extraction process and to measure its tensile properties. The higher the concentration of sodium hydroxide (NaOH) and the longer soaking times employed during the alkaline treatment of the *mengkuang* leaves, the higher the cellulose content extracted. The highest tensile strength of 520 MPa was measured for single CCF treated with optimum extraction parameters of 2% NaOH for 60 minutes. Amount of cellulose content of the extracted fiber showed an inverse relationship with the fiber's tensile strength. The removal of lignin and hemicellulose content during extraction process may have caused the reduction in the fiber's tensile strength.

Keywords: Continuous cellulose fiber, extraction, chemical treatment, fiber strength

1.0 INTRODUCTION

Acceptance of natural fiber composites in industrial applications such as automotive and building construction [1], aircraft [2], household apparatus [3] and medical sector [4] has created tremendous research in the related fields. Possessing unique characteristics such as low density with high strength, bio-degradable, less abrasiveness and low cost [5], natural fibers have the potential to replace conventional and synthetic fibers [6]. Despite the advantages of natural fibers, there are several issues related to natural fiber composites. For one, the strength of natural fiber composites do not normally surpass that of synthetic fiber composites due to incompatibility of the polar fiber and non-polar matrix. Another drawback with natural fiber composites is their non-uniform strength due to short and random orientation of fibers.

Significant research has been done on extraction process using single treatment [10] or combination of

Hence, one of the ways to improve the tensile strength of these natural fiber composites is to obtain continuous cellulose fiber that can be directly incorporated in suitable matrix material to produce natural fiber composite. Uni-directional aligned fiber is expected to give more strength to its composite in the longitudinal direction compared to randomly orient short fiber composites. Comparison studies on short, random [7] and continuous, unidirectional composite [8], showed that continuous, unidirectional composite possess better strength. Continuous cellulose fiber (CCF) can be obtained using appropriate extraction technique which must be carefully selected. This is because the quality of the extracted fiber depends on the extraction method and parameters [9]. For examples, mechanical extraction method involving crushing and extraction process using too high reagent concentration may all lead to damage of the fibers.

two or more techniques in order to obtain highly purified cellulose fiber [11]. Obtaining smooth and clean surface of the cellulose fiber will lead to maximum surface area but at the same time results in less adhesion and interlock between the fiber and the matrix. Certain researchers also used mechanical treatment to shorten the cellulose fiber and consequently turned into powder form through mechanical processing [12]. In another work, highly aggressive acid hydrolysis technique was employed to fasten the removal of binding material of the fiber causing swelling and dissolving of cellulose content [13]. Literature also showed that using sodium hydroxide (NaOH) gave promising effect in the extraction process [14] resulting in higher tensile strength of the extracted fiber [15].

Mengkuang is the Malaysian name for Pandanus tectorius. It is also known as screw pine, which is a plant belonging to the Pandanaceae family. These species vary in size and *grow* along mangroves and in tropical jungles. The leaves can be used for weaving. In addition, the leaves yield strong fibers that are used for making rope and weaving hats and mats [16]. Although the leaves are widely used in Asia, very limited studies of the production, composition, or properties of natural cellulose fibers from *mengkuang* leaves have been reported.

The objective of the study was to investigate the influence of extraction process parameters, such as concentration and soaking time, on the yield of cellulose and tensile strength of the extracted continuous fibers. Hence, the optimum extraction process parameters for the treated *mengkuang* leaves could be determined.

2.0 EXPERIMENTAL

2.1 Materials

Mengkuang leaves were obtained in the form of *mengkuang* mat. NaOH for alkali treatment was manufactured by R and M Chemicals, United Kingdom and supplied by Avantis Laboratory Sdn. Bhd. The epoxy used was EpoxAmite® 100 Laminating System with hardener, 102 Medium Hardener both manufactured by Smooth-On Inc.

2.2 Extraction of Continuous Cellulose Fiber

Mengkuang mat was washed thoroughly with distilled water and dried under the sun. It was cut into 12 cm x 3 cm strips and weighed accordingly. Each strip was approximately 0.3 – 0.35 grams. The leaves were treated with 2% up to 10% of NaOH at 170°C for either 60 or 120 minutes. The ratio of the leaves to liquor was 5:300 (g:ml). The leaves were washed with distilled water after each treatment and were dried under the sun for 3 days.

2.3 Single Fiber Characterization and Testing

Visual inspection of the *mengkuang* leaves in terms of structure and texture was carried out after each alkali treatment after the fiber was washed with distilled water and sun dried. The fiber microstructure, before and after treatment was observed using Zeiss SUPRA55VP with an accelerating voltage of 3kV. The images were digitally recorded. The percentage of cellulose fiber were determined by using TAPPI standard; T203. An amount of 25 mL treated *mengkuang* solutions were added with 10.0 ml of 0.5 N potassium dichromate solutions. 50 ml of concentrated H_2SO_4 was added cautiously followed by 50 ml of water after 15 minutes. 2 to 4 drops of Ferroin indicator were then added and titrated with 0.1 N ferrous ammonium sulfate solutions to a purple color. The strength of single fiber was based on ASTM 3822 using Titius Olsen Universal Tensile Machine with 5 kN load cell. The machine was equipped with pneumatic system in clamping, with adhesive layer for better gripers. The crosshead speed was 1.0 mm/min with 50 mm gauge length. Tests were conducted at 55% relative humidity at 23°C.

3.0 RESULTS AND DISCUSSION

3.1 Influence Of Alkali Treatment On Cellulose Content And Tensile Strength Of Fiber

The results of alkali treatment of the *mengkuang* leaves at varying NaOH concentration and soaking time are summarized in Table 1 in terms of the average cellulose content and average tensile strength of the extracted fiber. Cellulose content of the extracted fiber increased as concentration and soaking time of NaOH solution increased. An untreated mengkuang leaf contained 37% cellulose which increased gradually as the NaOH concentration and soaking time increased, reaching up to 72% at 10% concentration and 120 min treatment. This represents 95% increment in the cellulose content indicative of an effective treatment/extraction where most of the binding materials in the fiber such lignin, hemicellulose and pectin were removed [17]. At lower NaOH concentration, it can be observed that the effectiveness of the extraction process is greatly influenced by soaking time rather than NaOH concentration. For instance, treatment number 3 increased the cellulose content from 45 to 53% due to longer soaking time. Increasing the NaOH concentration from 2 to 4% while maintaining 60 min soaking time (comparing treatment 2 and 4) only reached 46% cellulose content, less than that achieved in treatment 3.

Table 1 Cellulose content and tensile strength of extracted continuous cellulose fiber following alkali extraction at varying NaOH concentration and soaking time [a]

Treatment No.	NaOH Concentration (%)	Soaking Time (min)	Avg. Cellulose (%)	Avg. Tensile Strength (MPa)
1	0	0	37 ± 0.6	503 ± 6.8
2	2	60	45 ± 4.1	520 ± 2.8
3	2	120	53 ± 2.9	515 ± 6.1
4	4	60	46 ± 1.5	470 ± 2.5
5	4	120	56 ± 3.5	474 ± 7.6
6	6	60	50 ± 2.1	456 ± 8.7
7	6	120	60 ± 2.5	453 ± 9.4
8	8	60	64 ± 4.5	433 ± 10.6
9	8	120	67 ± 5.5	335 ± 16.9
10	10	60	68 ± 6.4	313 ± 16.0
11	10	120	72 ± 3.5	268 ± 8.4

[a]All experiments were carried out based on TAPPI T203 STANDARD.

However, for NaOH concentration of more than 8%, the influence of soaking time on the cellulose content was less compared to the effect of using high NaOH concentration [18]. The increment in the cellulose content measured gradually decreased with increase in NaOH concentration (8 to 10%). This is attributed to depletion of the binding materials in the fiber itself as most of them have already been removed.

The trend in the average tensile strength of the continuous cellulose fiber after each alkali treatment is opposite to that observed for average cellulose content. As concentration and soaking time of NaOH solution increased, higher cellulose content was measured but tensile strength values were decreasing except at 2% concentration of NaOH. Untreated fiber showed a tensile strength value of 503 MPa which increased to 520 and 515 MPa after treatment at 2% NaOH for 60 and 120 min, respectively. This represents a relatively small, 2-3% improvement in strength. Treating the *mengkuang* leaves at NaOH concentration higher than 2% resulted in continuous decrease of the fiber tensile strength. The strength reduced to a low 268 MPa, a drop of nearly 50% compared to that of the untreated fiber. It was also observed that increasing the alkali concentration has more adverse effect on the tensile strength of the fiber compared to merely increasing the soaking time. This is demonstrated in treatments number 6 to 8. At 6% NaOH concentration, the tensile strength measured were 456 and 453 MPa for 60 and 120 min soaking time, respectively. Increasing the NaOH concentration to 8% reduced the tensile strength further to 433 MPa, even lower than that for combination of 6% concentration and 120 min soaking time. The primary reason for the reduction in tensile strength is due to the removal of the fiber main component which is hemicellulose [19]. The removal of the hemicellulose resulted in formation of new hydrogen bonds between the cellulose chains, changing the crystalline structure of cellulose and orientation of molecular chains [18]. Similar observations on tenacity for jute and cotton fibers were reported elsewhere [20, 21]. In another work, increasing alkali concentration has shown to damage the fiber and show negative effect on the tensile strength of its composite [22]. The initial increase in tensile strength measured for 2% NaOH for both 60 and 120 min soaking time may be attributed to increase in crystallinity index, packing density, molecular orientation of cellulose and removal of amorphous material [23]. The average tensile strength versus the average cellulose content measured following each alkali treatment of the *mengkuang* fiber is plotted in Fig. 1. The plot provides insight into the correlation or relationship between the fiber cellulose content and its tensile strength. Fig. 1 indicates that the cellulose content of the fiber is inversely proportional to its tensile strength with coefficient of determination, R^2 of 0.7132. High cellulose content fiber resulted in lower tensile strength. The removal of the hemicellulose during the alkali treatment has weakened the fiber's strength despite the high amount of cellulose of the extracted fiber. In other words, for cellulose fiber to impart high strength, the presence of hemicellulose is required. In this sense, hemicellulose acted as load transfer matrix to the cellulose fiber and gave strength to the fiber.

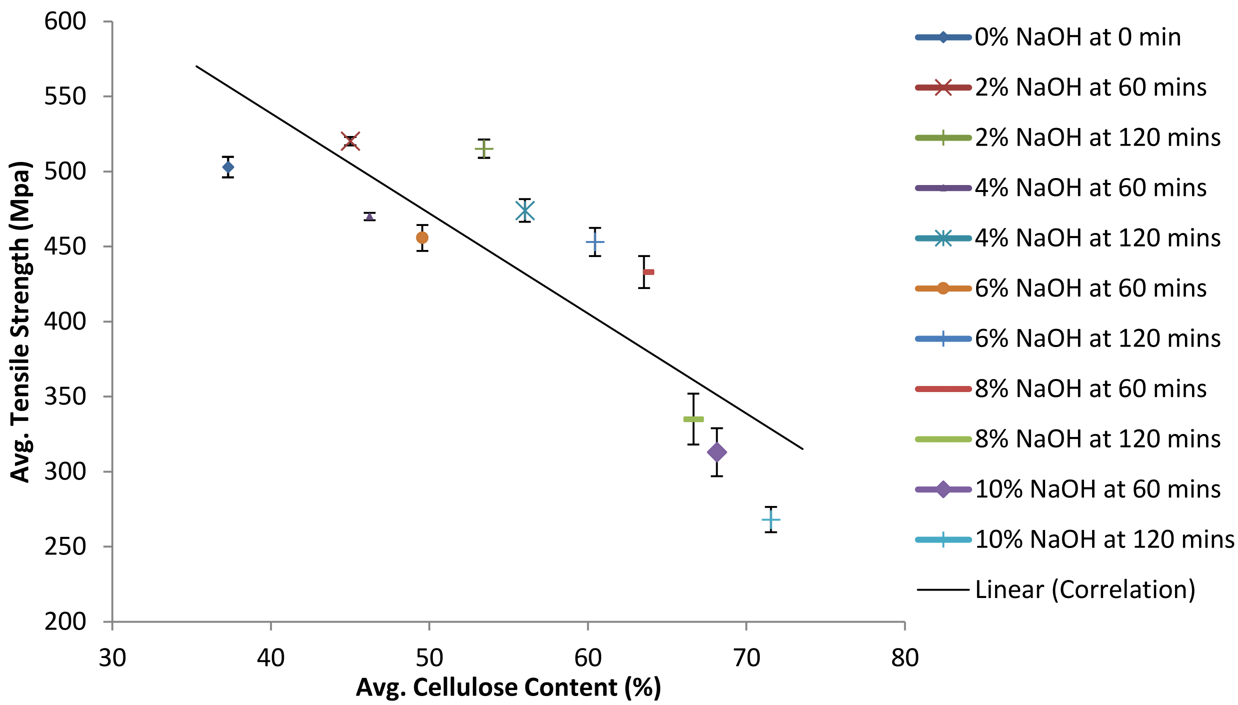


Figure 1 The relationship between cellulose content and tensile strength of continuous single fiber

3.2 Morphology of the Extracted Continuous Single Fiber

Fig. 2 shows the micrographs of the continuous single fiber, before the alkali treatment and after various treatments. Reduction in the extracted single fiber (microfibril) diameter was observed from Fig. 2(b)-(g). The microfibril has shrunk its size as the concentration of NaOH and soaking time increased.

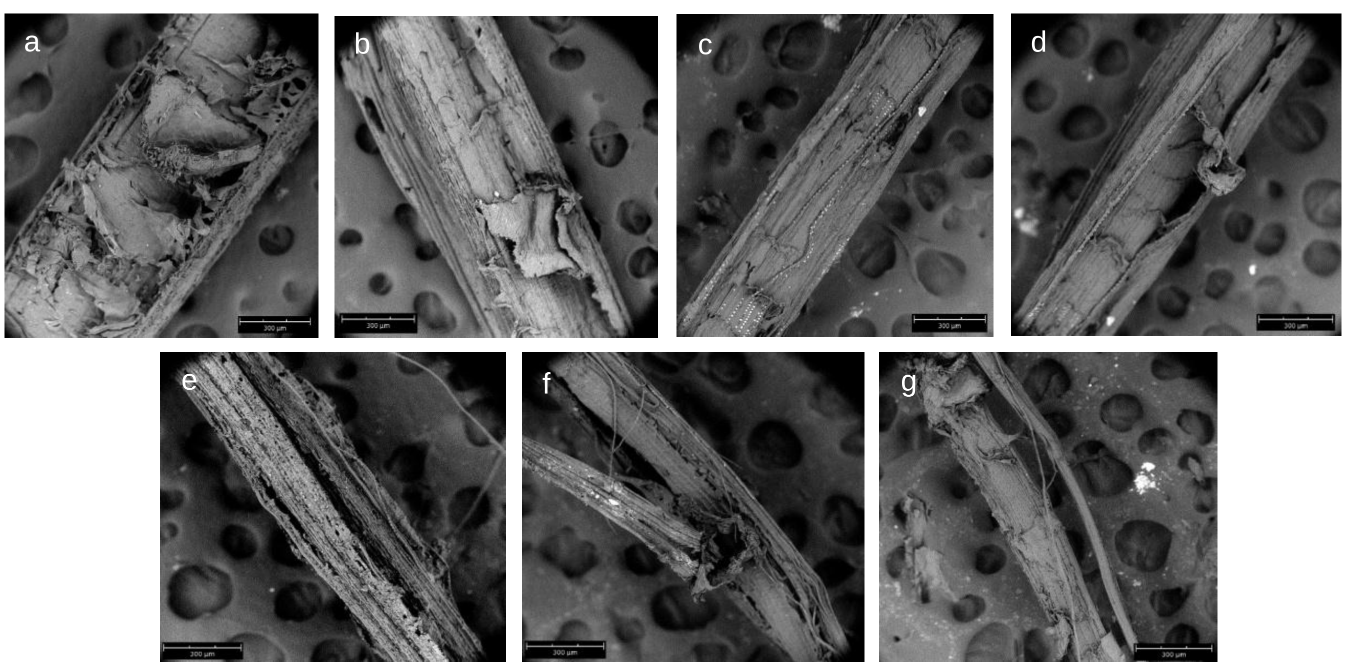
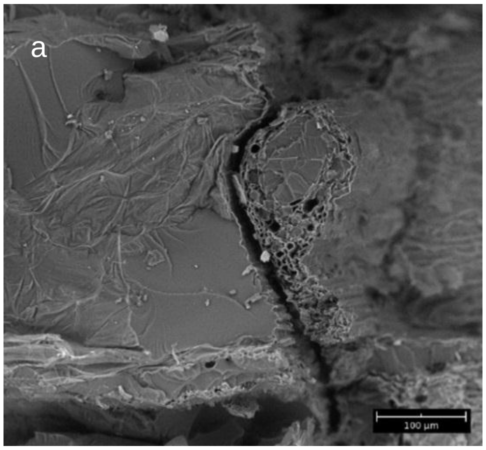


Figure 2 Morphology of single *mengkuang* fiber before and after each extraction process: (a) 0% Treatment (b) 2% NaOH at 60 min (c) 2% NaOH at 120 min (d) 6% NaOH at 60 min (e) 6% NaOH at 120 min (f) 10% NaOH at 60 min (g) 10% NaOH at 120 min (all magnification at 50x)



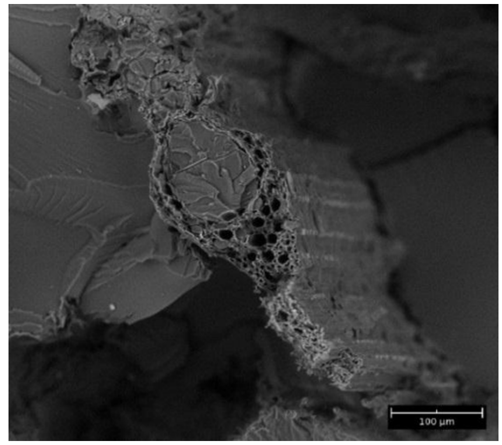


Figure 3 Morphology of the fracture surface of the continuous *Mengkuang* fiber composite. (Magnification of 500x)

Further observation of the micrographs revealed that the surface of the microfibril has become more exposed as the fiber was treated with alkali compared to that of the untreated microfibril, Fig. 2(a). The untreated microfibril's surface was still covered with gummy polysaccharides of lignin, pectin and hemicellulose [23]. The increased surface area of the microfibril due to the removal of the binding materials is expected to provide direct bonding between matrix and cellulose fiber [23] and enhance its composite's tensile strength. In Fig. 2(d), a hole or an opening started to form and fully developed as shown in Fig. 2(e), which was pertaining to treatment at 6% NaOH for 120 minutes.Treating the *mengkuang* leaves at 10% NaOH concentration for 60 min has caused the surface of the microfribils to become rougher as demonstrated in Fig. 2(f). The microfibrils started to separate into elementary fibers and started to bend or curl. Increasing the soaking time to 120 min, Fig. 2(g), has caused complete separation of the microfibrils which were also bending. Similar effects were observed on abaca single fiber treated using alkali at 15 wt% [24]. The removal of the binding material on single fiber has caused the bend to occur which was related to increased microfibril angle (MFA) and longitudinal contraction of microfibrils [25]. The longitudinal contraction of microfribils happened due to decreased end-to-end distance of the chain segments in amorphous region of the cellulose fiber during the alkali treatment [26].

3.3 Selective Study on Extracted Fiber Composite

From the previous results obtained, the highest average tensile strength of the extracted continuous single fiber was achieved during alkali treatment of 2% NaOH for 60 min. The tensile strength was 520 MPa compared to 503 MPa for untreated fiber and 268 MPa for fiber treated at 10% NaOH concentration and 120 min soaking time. In order to further explore the role of the treated fiber on its composite's strength, unidirectional continuous cellulose fiber epoxy composite was fabricated using resin vacuum infusion technique. Only fibers extracted under optimized extraction parameters (2% NaOH and 60 min soaking time) was selected due to its highest strength.

Result showed that the 15 wt% filled composite possess a tensile strength of 44 ± 3.8 MPa. There is more than 92% difference in the value of tensile strength of single fiber and its composite which is commonly attributed to poor compatibility of the epoxy matrix (non-polar) and filler (polar).

Table 2 Comparative study on natural fiber epoxy/resin reinforced polymer composites

Composite	Fiber volume fraction, V_f (%)	Tensile Strength (MPa)	Reference
Short and Random			
Bamboo + Unsaturated polyester resin	14.6	22.4	[7]
Jute + Unsaturated polyester resin	13.7	23.0	[7]
Kenaf + Unsaturated polyester resin	13.1	27.9	[7]
Continuous and Unidirectional			
Mengkuang fiber – epoxy	15	44	Current Study
Kenaf Fiber /epoxy	10	58	[8]
Kenaf fiber/Epoxy	30	124	[8]

A study on similar natural fibers with resin/epoxy reinforced polymer composites in terms of short/random and continuous/unidirectional cellulose fiber was done as summarized in Table 2. However, it is important to note that in all cases hand lay-up technique was employed to fabricate the composites. Composite systems with short and random fiber distribution showed tensile strength values between 22-28 MPa while that using continuous and unidirectional fibers achieved up to 124 MPa (kenaf). Generally, it can be said that composite filled with continuous cellulose *mengkuang* fiber has higher strength than those composites using short and random fibers although still lower than some other type of continuous and unidirectional fiber filled composite.

Morphology of the composite fracture surface was analyzed to give insight on the interaction between the fiber and the epoxy resin. The microstructure of the composite is shown in Fig. 3. As depicted in the micrograph of Fig. 3(a), a visible separation or gap between the fiber and matrix was observed indicating poor bonding of the two. In Fig. 3(b), the epoxy resin is seen to be filling the holes or empty spaces of the fiber although several tiny holes remained unfilled. This may be due to the weakness of the resin vacuum infusion technique to force the matrix to fill up all available holes of the fiber. However, there is no crack detected on the matrix surface indicating effective load distribution over the matrix [27]. For a composite to possess high tensile strength, good matrix-fiber interfacial bond and effective stress transfer from the matrix to the fiber must be achieved [28].

4.0 CONCLUSIONS

In this investigation, it was shown that NaOH alkali treatment was successful in extraction of continuous cellulose fiber from the *mengkuang* leaves. Increasing NaOH concentration and soaking time during the alkali treatment were effective in removing the binding materials of the fiber such as lignin, pectin and hemicellulose resulting in higher cellulose content of the extracted fiber. However, the absence of these binding materials has reduced the extracted continuous cellulose single fiber tensile strength. An inverse relationship was observed between amount of cellulose content and the fiber's tensile strength. The morphology of the single fiber also showed microfibrils disintegration and reduction in fiber diameter which may explain for the lower tensile strength as cellulose content increased for higher NaOH concentration and longer soaking time treatment. However, the highest single fiber tensile strength was achieved by treating the *mengkuang* leaves at 2% NaOH concentration for 60 minutes soaking time. The tensile strength measured was 520 ± 2.8 MPa, only 3% higher than that of the untreated fiber. Upon incorporation of this highest strength fiber in epoxy resin, the composite tensile strength dropped to 44 ± 3.8 MPa due to poor interaction between the fiber and matrix although effective stress transfer was observed from the composite microstructure. Nonetheless, the continuous, unidirectional cellulose fiber composite possessed higher tensile strength compared to other counterparts using short and random fibers.

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