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Fabrication and Characterization of Cellulose Microfibrils from *Pandanus tectorius* (Screw Pine) for Polymer Composite Application

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Research Article

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ABSTRACT

Natural cellulose fiber was extracted from *Pandanus tectorius* (Screw pine) leaves using alkali and combined alkali-bleach treatment. Influence of extraction process parameters on the yield content and mechanical property was evaluated. Optimized process parameter was used in fabrication of a cellulose fiber composite using Vacuum Resin Infusion (VRI) techniques. Chemical composition of screw-pine leaves was determined at different stages of combined alkali-bleach treatment. Structural analysis was carried out by Fourier transform infrared spectroscopy (FTIR). Analysis on morphological structure and tensile strength of the cellulose fiber composite was through scanning electron microscopy (SEM) and universal compressive machine. The results showed that combined alkali-bleach treatment at 4 wt.% of $NaClO_2$ after 8 wt.% of NaOH under alkali treatment resulted in the optimal treatment combination, particularly when soaked for 120 minutes. However, longer soaking time caused damage to the fiber structure. The process parameters all influenced the chemical property, yield content and tensile strength of the cellulose fiber composite. Fiber content of 50 wt.% achieved the maximum tensile strength for the cellulose fiber composite with 28 wt.% composite enhancements for the cellulose fiber composite. The VRI techniques improved the aspect ratio of the cellulose fiber composite after production. The SEM micrograph showed the fibrils of cellulose fiber composite and its deformation. Analysis was carried out to investigate the bonding quality of the cellulose fiber and matrix. As a comparison, a control sample of unfilled epoxy matrix was fabricated

INTRODUCTION

Fabrication of biomass polymer composites have gained attraction from researchers and manufacturers in recent decades courtesy of growing concern for environmental protection, depletion of fossil resources and desires to eliminate or reduce petrochemical consumptions and mitigate pollution [1-3]. Biomass cellulose Fiber composites are widely available in varieties with controllable properties which can potentially substitute the synthetic polymer Fiber composite that is environmentally unfriendly and have some health concerns due to the toxic nature [4,5]. Among the different types of obtainable biomass fibers, the plant based non-wood fibers have demonstrated huge potential for application in polymer composite used from small scale industries to large scale automobile industries [2,6,7].

Pandanus tectorius (Screw pine) belongs to Pandanaceous family that possesses more than 600 known species. The plant can grow up to 14 meters tall and are inhabited in mangroves and in shallow water [8]. The leaves are traditional used in making ropes, mats and hats due to their remarkable strength; they are suitable for other domestics' applications [9]. However, only a few studies were done on screw-pine leaves, although they are famous and widely used in Asia region [10,11].

In a related research, cellulose fiber was successfully extracted from screw-pine leaves in the form of nanocrystal structure, using chemical means [10]. However, the study did not explore the application of the nanocrystal in composite fabrication. Consequently, the effectiveness of the extraction was unascertained. Similarly, Haafiz et al. [1] studied the effects of microcrystalline cellulose (MCC) loading on morphological, thermal and mechanical properties of resulting polylactic acid (PLA) composites from oil palm biomass. The results showed the Young's modulus increased by about 30%, while the tensile strength and elongation at break for composites decreased with addition of MCC. In the work by Deesoruth et al. [12] carry out investigation on an epoxy

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resin vacuum infused in screw-pine Fibers has as a realistic alternative to glass fibre composites. From the comprehensive testing results, alkali-treated fibre composites withstand more load than untreated fibre composites at 5, 10, and 15% (weight basis) fibre loadings. Fatigue test also confirmed that results are significantly better when composites are made from treated fibre.

In another effort, the characteristic of local water hyacinth (WH) Fibers and composites that consist of mixing WH Fibers and unsaturated polyester (UPR) were studied by Hairul et al. The results show that 7% NaOH, 1 hour, treated WH Fibers provided better mechanical properties on UPR matrix composites in comparison with other alkali concentrations. From scanning electron microscopy (SEM) observation, some untreated WH Fibers pulled out from their matrix were observed clearly in fracture surface of composites [13]. Flax fiber polymer matrix (with nano additives) composites were fabricated using Fibers with treated and untreated surface. The chemical structures of the natural Fibers and the compatibility of the matrix material were tested to determine the replicability in synthetic polymer composite. The results concluded that cellulose Flax Fiber demonstrated higher tensile strength, yield content and aspect ratio compared to the synthetic Fiber. However, the nano-additive had adverse effect on the yield content [14]. Several other researches have been carried out on the use of natural Fibers extract as a direct substitute for conventional synthetic polymer composites, [2,4,5,15,16] however, in most of the studies their findings involved different biomass Fibers (Plants, animal and minerals) and process parameter (soaking time, chemical compositions, percentage weight fractions, mixing fractions, temperature etc.). In most of the investigation, many of the studies look at Fiber extract in the form of single cellulose and cellulose composite fabrication. Very fewer attempts are made on extraction of long and durable continuous cellulose Fibers. The aspect ratio impacts the Fiber yield content and mechanical strength positively, yet, not many studied have been conducted cellulose fiber composite for application in polymer industries.

The present study extract cellulose micro fibrils from *Pandanus tectorius* (Screw pine) using chemical technique (combined alkali-bleach treatment) while the fabrication of the cellulose fiber composite is carry out by Vacuum Resin Infusion (VRI) techniques. The micro fibrils are then characterized to evaluate the influence of the process parameter on the yield content and mechanical property for polymer composite application in engineering. The chemical constituents of the untreated, treated cellulose and cellulose composite were determined by chemical analysis. The morphology of the screw pine leaves and cellulose micro fibrils was investigated by scanning electron microscopy (SEM). The structural changes were revealed with a set of spectroscopy methods using Fourier transform infrared spectroscopy (FTIR). Tensile Strength Testing (TST) was carried out on the single cellulose fiber after being treated with combined alkali-bleach and the cellulose fiber composites measured for all samples.

EXPERIMENTAL SECTION

Materials

The main material used in the study is *Pandanus tectorius* (Screw pine) leaves as the main source of cellulose fiber. The fiber was extracted with chemical means using alkali treatment and followed with bleaching to obtain high quality cellulose percentage. The Screw pine leaves were obtained from local shop at Kuala Kangsar, Malaysia. All the processes were assumed to have no appreciable effect on the structural and chemical composition of the fiber.

Chemical Reagents

All chemicals and reagents utilized are of standard quality and were purchased from a reputable supplier. The alkali treatment involved sodium hydroxide (NaOH) diluted to the desired concentrations which were 2–10 wt.% with increment of 2 wt.% concentration. In bleaching process, sodium chlorite ($NaClO_2$) was used and was dissolved from powder to solution of 1, 2 and 3 wt.% concentration. In determining the cellulose content of the extracted fiber, several chemicals were used such as potassium dichromate ($K_2Cr_2O_7$), ferrous (II) ammonium sulphate ($Fe(NH_4)_2(SO4)_2 \cdot 6H_2O$), sulphuric acid ($H_2SO_4$), and sodium hydroxide (NaOH). Each chemical was prepared according to Technological Association of the Pulp and Paper Industry (TAPPI) Standard T_20_3. The epoxy used in the fabrication of the cellulose based polymeric composite was EpoxAmite® 100 Laminating System with hardener, 102 Medium Hardener.

Extraction Processes

Pandanus tectorius (Screw pine) leaves were harvested, cut and washed thoroughly with distilled water and dried under the sun for 24 hours. Then, cut into 12 cm long and 3 cm wide strips and weighed accordingly. Each strip was approximately 0.3-0.35 grams. The leaves were subjected to combined alkali-bleach treatment to enhance cellulose fiber yield content.

The Fibers were treated with 2 wt.% till 10 wt.% of NaOH aqueous solution at 200°C for 60 and 120 minutes, respectively. The ratio of the leaves to liquor was 5:300 (g/mL). The leaves were washed with distilled water after each treatment until the alkalinity indication is removed. Subsequently the Fibers are dried under the sun for 3 days. Selected Fibers extracted under optimum alkali and combined alkali-bleach treatments were further employed for their composite fabrication.

Fabrication of Continuous Cellulose Fiber (CCF)

Vacuum Resin Infusion method is utilised in the extraction of continuous cellulose fiber. Only alkali treated and combined alkali-bleach treated fibres from optimized extraction parameters were used in the fabrication of the cellulose fiber composite.

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The corresponding cellulose fiber was placed on one-sided coated mould with wax in order to avoid fiber stacking on the mould during demoulding process. The cellulose fiber were vertically aligned and arranged into three layers with different arrangement. The arrangements were to avoid gaps between fibres and to reduce porosity. A highly permeable medium of peeling ply and distribution mesh were laid over the surface of the fiber. The whole assembly was enclosed in a vacuum bagging film and sealed with sealant tape. The epoxy resin was 32 injected into the assembly with ratio of epoxy to hardener of 10:3 (w/w) under vacuum pressure. The composite was cured at 70°C for 4 hours inside the oven and naturally cooled down to room temperature. The composite was then demoulded.

Characterization and Measurements

Fourier transform infrared (FT-IR) analysis is performed on a Perkin Elmer 1600 Infrared Spectrometer, with 4000–500 cm^{-1} spectral range; samples were mixed with an analytical proprietary KBr beam splitter. FT-IR spectra of the samples were recorded by Thermo Nicolet's AVATAR 380 at 100 scans, a resolution of 3 cm^{-1}. Nicolet OMNIC 5.01 software was used in determining the transmittance peak at a particular wave numbers. Morphology of samples is observed using Zeiss DSM 950A Scanning Electron Microscopy (SEM). The samples surfaces were coated with gold to avoid charging. The SEM is operated at 25 KeV. The SPA-300 HV atomic force microscopy with SPI 3800 controller is used in performing the analysis of AFM observation for the untreated CF and treated CCFs composite samples with dimensions (0.1 mm x 0.1 mm).

ASTM 3039 standard is used in determining the tensile strength of extracted cellulose fiber composite. Specimen's dimension is 230 mm x 16 mm x 1.6 mm. Extensometer gauge length was 50 mm with 2 mm/min constant crosshead speed. Test was conducted using Zwick/Roell Z005 universal testing machine with 5 kN cell load. The composite was clamped through a set of mechanical springs and mechanical zigzag gripers. All tests were conducted at 55% relative humidity and approximately 23 ˚C temperature.

RESULTS AND DISCUSSION

FT-IR Spectroscopy Analysis

Interaction and phase behaviour of polymer composites have been widely studied using the FT-IR analysis techniques [1,13,17]. Typical FTIR spectra of untreated cellulose fiber, alkali treated cellulose fiber and combine alkali-bleach treated cellulose fiber is shown in **Figure 1**. The alkali treated, comparably as observed in many lignocellulose Fibers, displayed an induced variation on in the physiochemical properties of the Fibers [18]. Whereas, combined alkali-bleach treated showed few variation purportedly due to complete removal of hemicellulose and lignocellulose fibers. Major peaks were observed at 3525 cm^{-1} and 1650 cm^{-1} which represent non-aromatic moieties. Sharp decrease in the intensity of peak around 3500 cm^{-1} for combined alkali treated fiber may be related to complete removal of hemicellulose [18]. The intensity of untreated cellulose fiber decrease around 1450 cm^{-1} for C=O band stretching in the second peak. Similar to combined alkali treated cellulose fiber. The origin of the peaks can be attributed to the carbonyl groups of hemicellulose that is contained in lignocellulose of the polymer components.

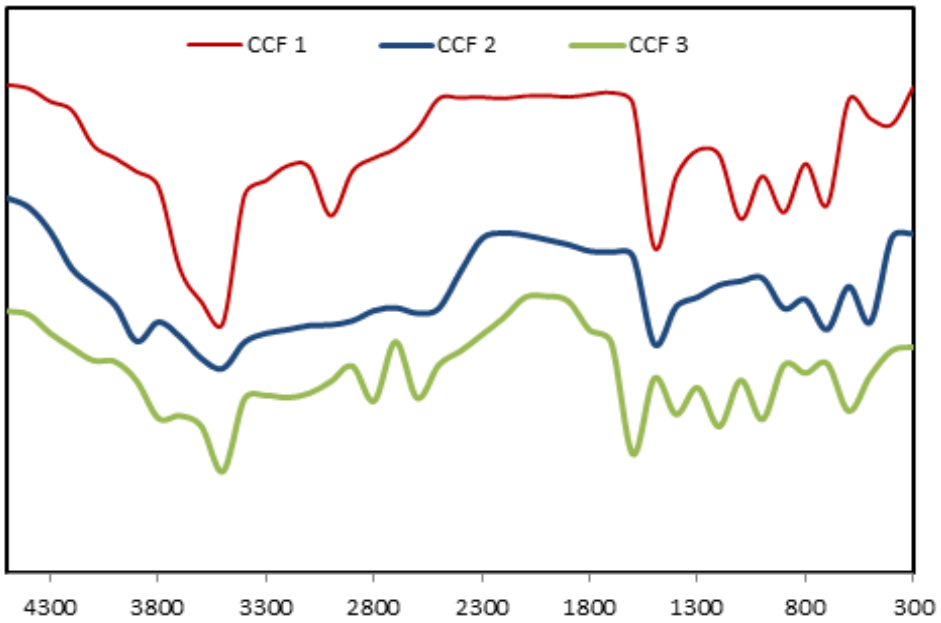

Figure 1. FTIR spectra of screw-pine Fiber (a) untreated cellulose fiber; (b) alkali treated cellulose fiber and (c) combined alkali-bleach treated cellulose fiber. Vertical broken lines represent the characteristic peaks of cellulose and hemicellulose.

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The C-O-H band stretch at 1100 cm^{-1} showed characteristic of phenolic group, however at 1550 cm^{-1} the characteristic stretch bands is comparable to what is obtainable at lignin in the wood [1]. The weakness in the intensity of C=O and C-O-H bands in combined alkali treated CF can be traced to the complete removal of the hemicellulose and lignin from the micro fibrils of the leaves. The characterization of the lignocellulose for the untreated CF and alkali treated CF Fibers have the capability to remove 25% hemicellulose and 44% lignin. The small peaks in the range 1850–1450 cm^{-1} mostly represents the functional groups generally originating from lignin. They may as well arise from cellulose/hemicellulose i.e. peak at 650 cm^{-1} [5].

Morphology Analysis

SEM of fractured surface of the composites is observed to analyse the failure mechanisms and the interaction between different components since the mechanical properties depend on polymer/filler interaction. SEM of fractured cross-sectional surfaces of untreated cellulose fiber, alkali treated cellulose fiber and combine alkali-bleach treated cellulose fiber composites is shown in **Figures 2-4**.

Untreated cellulose fiber composite: Microstructural observation showed absence of fiber during pull out test. This implies poor bonding between fiber and matrix due poor due existence of cementing materials (lignin and hemicellulose) on the Fibers. Micrograph of untreated cellulose fiber is shown in **Figure 2a and 2b**. A non-flat crack overlapped is observe, unfilled void holes depicting poor bonding.

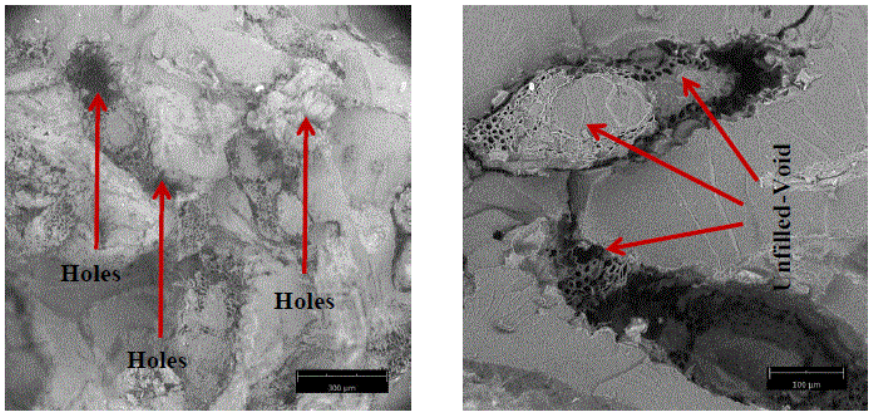

Figure 2. SEM of fractured surface of untreated cellulose fiber Composite Fiber-Matrix a) Crack Formation (200x Magnification) b) Unfilled Holes of the Fiber (500x Magnification).

In addition, the small cracks around the fiber also indicated poor interaction between fiber and matrix due to present of cementing substances. A deep hole was also observed upon removal of the fiber during tensile test. However, there was no crack detected on the matrix surface. This may indicate of effective load distribution and transmission around the matrix [19].

Alkali treated cellulose fiber composite: Figure 3 shows microstructure of the fractured surface of the selected alkali treated cellulose fiber composite. A block structure with filler is formed as observed in **Figure 3**; this can be traced to the hollow structure of the pure screw-pine fiber. The epoxy resin infused and penetrated into the hollow structure. A visible separation or gap is seen in **Figure 3a** which is enlarged in **Figure 3b**.

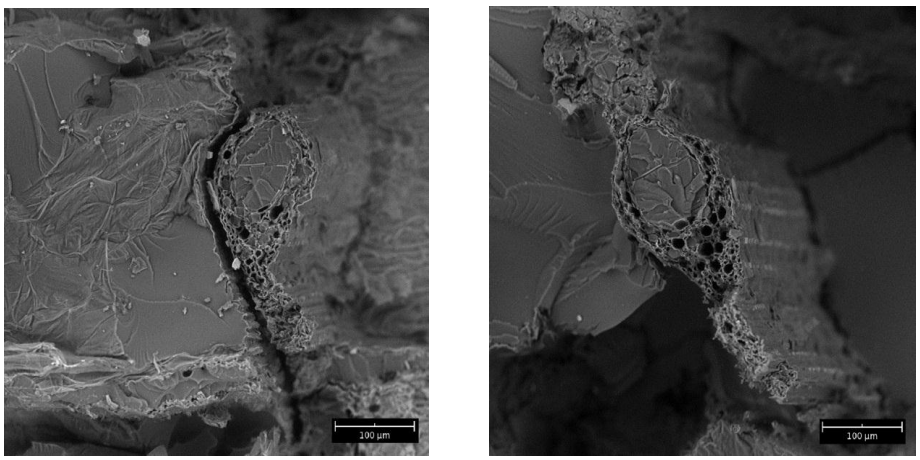

Figure 3. SEM Micrographs of fractured surface of the alkali treated cellulose fiber Composite fiber-matrix bonding (500x Magnification) a) Crack Formation b) Unfilled Holes of the Fiber.

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The gap indicated poor bonding between the fiber and epoxy. A good composite with high tensile strength requires a good matrix-fiber interfacial bond so that an effective stress transfer from matrix to fiber can be achieved [20]. The micrograph of **Figure 3b** also shows several tiny holes that were remained unfilled during the fabrication although most of the fiber structures were filled with epoxy resin. The porosity and void content of the composite created a weaker composite. However, there is no crack detected on the matrix surface. This was an indication of effective load distribution and transmission around the matrix [20].

Combined alkali-bleach treated cellulose fiber composite: Figure 4 show fractured surface of the combined alkali-bleach treated composite. The absence of the rectangular and block structure as seen in alkali treated composite structure is to extensive removal of hemicellulose and lignin due to combine alkali-bleach treatment as observed in **Figure 4**. The interfacial bonding between fiber and matrix was relatively strong and smooth surfaces free of holes were observed. Although, there are still presences of tiny holes observed in the fiber, unfilled by epoxy resin as demonstrated in **Figure 4a and 4b**. Nonetheless most of the epoxy resin wets the surface area of treated fiber rather well. The void space is smaller than in the alkali treated fiber composite. Thus far, the combination of alkali-bleach treatments improved the interaction and bonding of the fiber-matrix. As a result, a higher strength composite is produced.

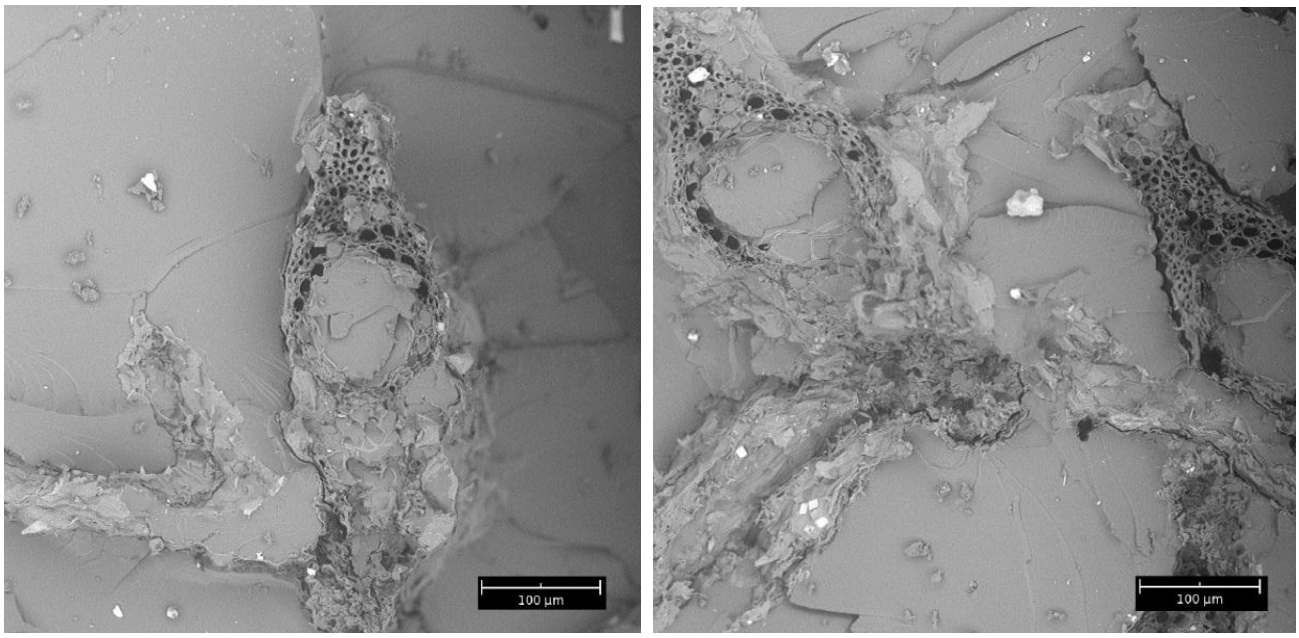

Figure 4. Scanning Electron Micrographs of the Fractured Surface of the Combined Alkali-Bleach Treated Mengkuang Cellulose Fiber Composite (500x Magnification).

The SEM results were consistent with the flexural test results. Similar observations for date palm fibres and thermoplastic starch composite were also reported [21] who conducted alkaline treatment of natural fiber for partial removal of hemicellulose and lignin for bio-composite fabrication.

Mechanical Properties

Alkali Treatment: The samples were all subjected to tensile stress test including the control sample which is 100% epoxy. All other samples contain 15 wt.% of cellulose fiber in the polymer composite. **Figure 5** shows the effects of the alkali treatment on the Fiber polymer composite. The results indicated that tensile strength of the treated cellulose fiber decreased as the NaOH concentration and soaking time increased except at 2 wt.% concentration of NaOH. Tensile strength of the 2 wt.% NaOH treated cellulose fiber increased slightly by 3-4% compared to that of the untreated cellulose fiber. Tensile strengths were 520 MPa and 515 MPa after treatment at 60 and 120 minutes, respectively. The increment in tensile strength may be attributed to increasing crystallinity index, packing density, and molecular orientation of cellulose and removal of amorphous materials [21]. The fibre's tensile strength continued to decrease as the NaOH concentration increased more than 2 wt.%. The lowest tensile strength of 268 MPa was exhibited by cellulose fiber treated at 10 wt.% NaOH. Effect of soaking time on the tensile strength of the treated cellulose fiber was found to be more discernible at higher NaOH concentration, namely 8 and 10 wt.%. Longer soaking time has caused the tensile strength of the cellulose fiber to reduce markedly compared to that soaked for 60 minutes. The difference in the tensile strength for cellulose fiber treated at 8 wt.% NaOH for 60 and 120 minutes was more than 20%.

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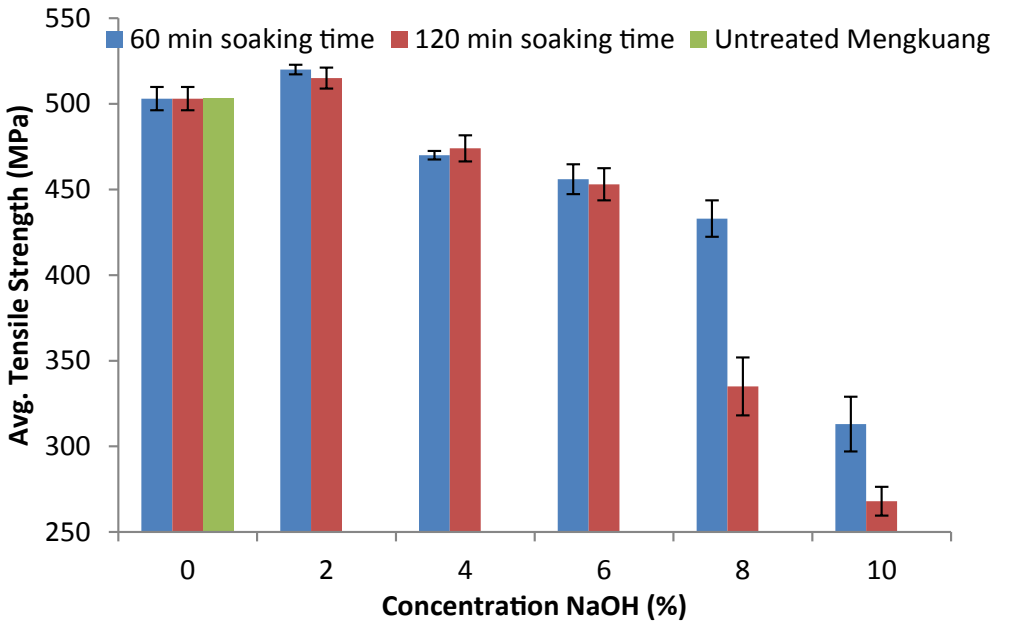

Figure 5. Tensile strength of cellulose fiber treated with varying NaOH concentration and soaking time

Combined alkali and bleaching treatment: Figures 6a and 6b illustrates the effects of combined alkali-bleach treatments on cellulose fiber. The results showed the highest tensile strength was measured for cellulose fiber alkali treated at 2 wt.% NaOH for 60 minutes and bleached at 2 wt.% $NaClO_2$ for 60 minutes.

The tensile strength was 483 MPa. However, the value was 7% lower than that without bleaching treatment. Increasing the bleach concentration and soaking time did not improve on the cellulose fiber's tensile strength. Nonetheless, bleaching at 2 wt.% for 60 minutes of the alkali treated cellulose fiber at 2 wt.% NaOH for 120 minutes showed tremendous increase in the cellulose fiber tensile strength.

A value of 722 MPa was recorded, as shown in **Figure 6b**, an increment of 44% compared to untreated cellulose fiber. This actually represented the highest tensile strength achievable from both alkali and combined alkali-bleach treatment. Hence, 2 wt.% NaOH treatments for 120 minutes, followed by bleaching at 2 wt.% $NaClO_2$ for 60 minutes, were considered as the optimum extraction parameters for screw-pine leaf which yielded the highest tensile strength of the treated cellulose fiber [22-26].

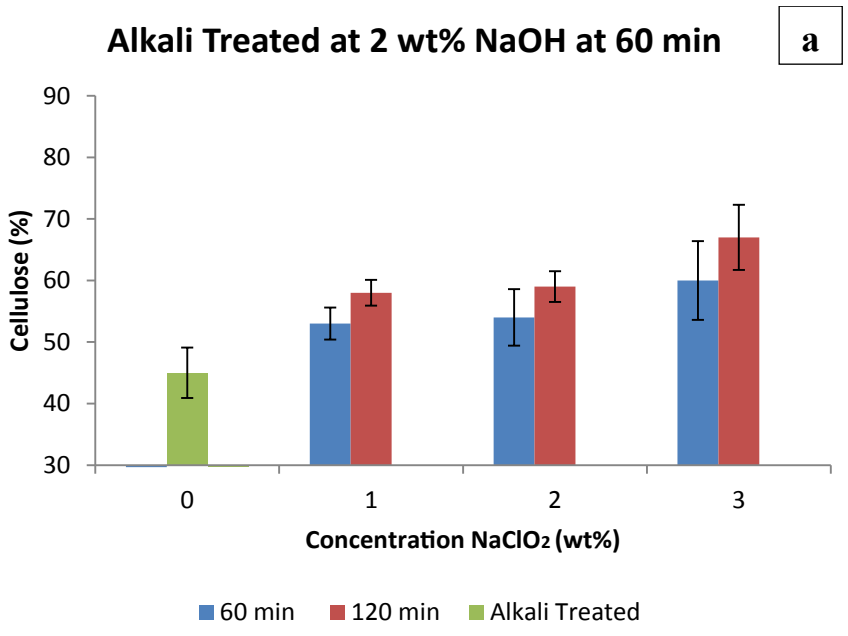

Figure 6a. Influence of $NaClO_2$ concentration and soaking time on tensile strength of cellulose fiber (a) Alkali Treated at 2 wt.% NaOH for 60 minutes.

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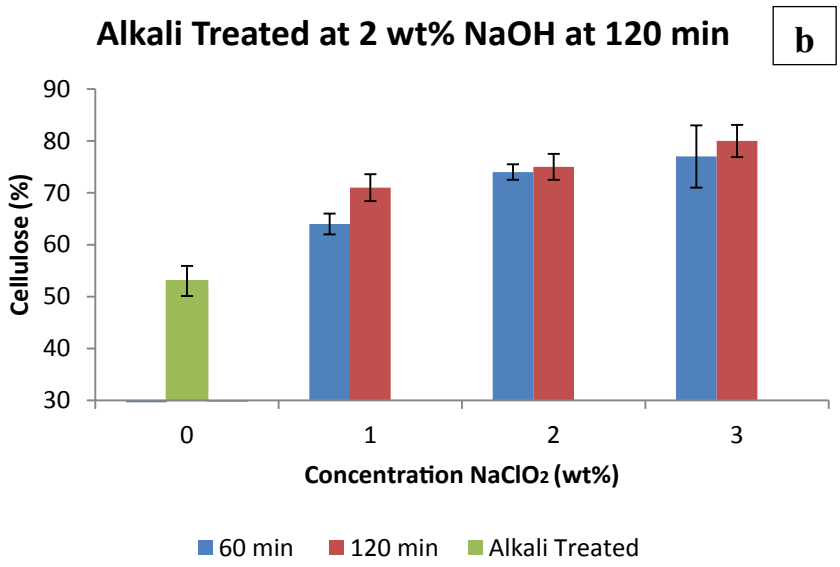

Figure 6b. Influence of $NaClO_2$ concentration and soaking time on tensile strength of cellulose fiber. (b) Alkali Treated at 2 wt.% NaOH for 120 minutes.

Cellulose fiber composite: Figure 7 shows comparison between the average tensile strength of the controlled sample, untreated and treated cellulose fiber composites. The cellulose fiber composites using combined alkali-bleach treated Fibers gave the highest tensile strength. The combined alkali-bleach cellulose fiber composite showed 40% higher tensile strength compared to untreated cellulose fiber composite at 35 2.8 MPa. The alkali treated cellulose fiber composite is 15% higher in tensile strength compared to the untreated cellulose fiber composite. However, tensile strength of untreated cellulose fiber was lower than that of unfilled composite by 5% due to poor adhesion between fiber-matrix bonding. The fabricated cellulose fiber polymeric composite is compared with other relevant research studies from the literature as presented in **Table 1**.

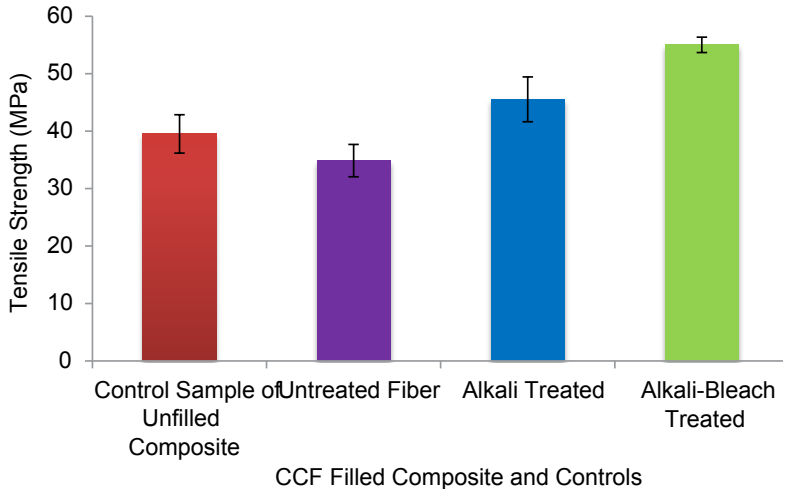

Figure 7. Average tensile strength of the selected cellulose fiber composites

Table 1 Mechanical property of various types of natural fiber reinforced polymer composites including those from the current study.

Table 1. Mechanical properties of some natural Fibers reinforced polymers

Composite	Process	Fiber Fraction (%)	Tensile Strength (MPa)	Reference
Short and Random	Hand lay-up with compression moulding method			
Bamboo +Unsaturated polyester resin		14.6 vol.%	22.4	[22]
Jute + Unsaturated polyester resin		13.7 vol.%	23.0	[23]
Kenaf + Unsaturated polyester resin		13.1 vol.%	27.9	[24]

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Continuous and Unidirectional				
Alkali Treated screw-pine fiber–epoxy	VRI	14 vol.%	45	Current Study
Alkali-Bleached Treated screw-pine fiber-epoxy	VRI	14 vol.%	55	Current Study
Kenaf Fiber/epoxy	Hand lay-up	10 vol.%	58	[25]
Kenaf fiber/Epoxy	Hand lay-up	30 vol.%	124	[26]

Comparing between the short and random composites, continuous and unidirectional fiber reinforced composites is shown in **Table 1**. Normally, short and random composites contain half of the strength of the continuous and unidirectional composites i.e. their composite tensile strength ranges between 22-28 MPa. Whereas continuous and unidirectional fiber reinforced composites, have tensile strength of about 58 MPa for fiber fraction between 10–15%. The composites tensile strength of the continuous and unidirectional fiber can increase up to 124 MPa when the fiber fraction doubles to 30 vol.%. The result for the current study showed alkali treated fiber composite reached a tensile strength 45 MPa and the combined alkali-bleach treated fiber composite reached tensile strength of 55 MPa [11]. Thus implies that the *screw-pine* reinforced fiber composite can be a good substitute fillers in natural fiber composite.

CONCLUSION

The effect of the alkali treated and combined alkali-bleach treated on the micro fibrils of screw-pine cellulose fiber mechanical property and yield content have been studied. Morphological analysis performed on each of the treated fiber samples have shown that the chemical concentration and soaking time have tremendous influence on the fibrils. When the chemical concentration is increased and the soaking time is prolonged, a higher cellulose percentage is obtained. However, roughening of the fiber is observed, particularly on the elementary fiber due to removal of the impurity substances. Increasing the $NaClO_2$ concentration and soaking caused defibrillation to occur. Nonetheless, it is an indicator of complete removal of the hemicellulose and lignin. However, the structure of the screw-pine leaves and their micro fibrils started to distort as the bleaching process parameters increased in concentration and soaking time, likewise defects and porosity level increased. Thus increasing the process parameter will lead to higher cellulose percentage of the fiber, but also caused damage to cellulose fiber structure. Hence, optimum extraction process parameters should be selected to ensure smooth fiber surface (free of cementing materials) and intact micro fibril structure. The optimum parameters occurred at concentration of 2 wt% NaOH and 2 wt% $NaClO_2$. Thus far, natural fiber composite can substitute for synthetic composites which are non-economical and non-environmentally friendly for engineering applications.

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