

OPTIMIZATION OF PULPING PROCESS OF BETEL NUT FIBER USING CENTRAL COMPOSITE DESIGN FOR THE PREPARATION OF CELLULOSE FIBER*

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Abstract

Betel nut fiber was used to isolate cellulose fiber by four processing steps such as alkali hydrolysis using sodium hydroxide (pulping), bleaching using hydrogen peroxide, acid hydrolysis using sulphuric acid and homogenization. In the pulping of biomass, Central Composite Design of Response Surface Methodology was used to optimize the yield of cellulose. Three variables such as alkali concentration, alkali volume and cooking time were considered as influencing factors on the yield of holocellulose, alpha cellulose and hemicellulose during alkali pulping process. The maximum yields of holocellulose, alpha cellulose and hemicellulose by % wt were 94.33 ± 0.76 , 80.17 ± 1.89 , and 13.83 ± 1.75 under the optimum conditions. Cellulose fiber was analysed by thermogravimetric analysis (TGA), Fourier-transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), and X-ray diffraction (XRD). The XRD results revealed high crystallinity of both the cellulose fiber and cellulose pulp, while the TGA thermograms indicated that the alkali and acid treatment completely removed lignin and hemicelluloses from the betel nut fiber. FTIR results reveal that the peaks in the range between 1200 and 1300 cm^{-1} were not observed in pulp and cellulose fiber which indicated the removal of hemicelluloses and lignin. SEM microphotograph showed that mercerization and acid hydrolysis lead to fibrillation and breakage of the fiber into smaller pieces which promote the effective surface area available for contact.

Keywords: Cellulose Fiber, Central Composite Design, Response Surface Methodology, Holocellulose, Alpha Cellulose, Hemicellulose

Introduction

The disposal of agricultural waste is becoming a problem especially in the intensive growing areas. The unmanaged agricultural waste in the plantation causes terrible odor and other decay-related problems. Agricultural waste and by-products are rich sources of cellulose and hemicelluloses. Lignocellulosic fibers from agricultural waste act low cost and more environmentally friendly replacement for wood in polymer composite. In the last few years, cellulose fibers have been a trendy research subject due to their high strength and stiffness combined with low weight and biodegradability (Liew *et al.*, 2015). Cellulose fibers are widely used in the paper and packaging products, automotive, furniture, sporting goods, electronics, pharmacy and cosmetic (Dungani *et al.*, 2016). The betel nut fiber is a strong fibrous material covering the endosperm and constitutes about 60-80% of the total weight and volume of the areca fruit. The husk of betel nut fiber is composed of 55.82% cellulose, 34.28% hemicelluloses, 6.82% lignin, 1.80% moisture content and 1.28 % ash content. This highly cellulosic material is being used as a fuel and fabricated as value added products such as cushion, handcrafts and non-woven fabrics. Surface modifications of natural fibers by various chemical treatments are one of the most significant areas in the field of technical utilization of natural fiber reinforced polymer composites in various industrial sectors (Dhanalakshmi *et al.*, 2015). Cellulose is physically and chemically surrounded by hemicellulose and lignin, with the links forming a lignocellulose matrix (Galiwango *et al.*, 2019). Cellulose was extracted by industrial pulping processes. Most other constituents of the cell walls are broken down or solubilized and removed (Mohammadi *et al.*, 2011). The pulping processes are categorized as mechanical, thermal, semi-chemical or wholly chemical methods. Chemical pulping accomplished the degradation of lignin and

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* Best Paper Award Winning Paper in Industrial Chemistry (2020)

hemicelluloses into small water-soluble molecules which can be washed away from the cellulose fibers. Chemical methods include Kraft, sulphite, soda, and organosolv pulping processes (Azeez, 2018). During pulping, the reagents attack the lignin and hemicellulose on which the macromolecules are converted into low molecular weight that are soluble in the liquor. Pulping is not enough to eliminate all lignin and hemicellulose residue; therefore, additional bleaching process can be used (Pereira *et al.*, 2011). Acid-alkali treatment is the most effective method for individual cellulose isolation from the complex structure of lignocellulose (Galiwango *et al.*, 2019). Combination of sulphuric acid hydrolysis and homogenization methods to isolate cellulose nanofiber is reported to be convenient and has minimum effect on fiber properties (Chaturbhuj *et al.*, 2016). Sulphuric acid hydrolysis mainly removes the amorphous regions of the cellulose (Zhang *et al.*, 2019)

In this study, cellulose fiber was prepared from betel nut fiber. Conversion of non-woody biomass to cellulose fiber was conducted through pulping by alkali hydrolysis and bleaching followed by acid hydrolysis and homogenization.

Materials and Methods

Materials

Betel nut fiber was brought from betel nut farm, Thandwe Township, Rakhine State. Analar grade BDH products – acetic acid, hydrogen peroxide, sodium chlorite, sulphuric acid and commercial grade sodium hydroxide were purchased from Golden Lady Store – Chemical Trading, No. (114), 28th street, Pabeden Township.

Preparation of Cellulose Fiber

Sodium hydroxide was used as cooking liquor for conversion of betel nut fiber to pulp. Response Surface Methodology was used to optimize the maximum yield of alpha cellulose content in pulp with the process variables such as alkali concentration, alkali volume and hydrolyzing time during cooking process as shown in Table (1). Central Composite Design was chosen and (17) experimental runs were conducted as shown in Table (2).

Betel nut fiber was dried in sunlight about one week. Dried betel nut fiber was ground by using a grinder. Powder 50 g was treated with 464 mL of 48 % (w/v) sodium hydroxide for 4 hr for pulping process. After alkali treatment, it was washed with tap water and bleached with 30 % (v/v) hydrogen peroxide for one hour. It was then washed with purified water in order to remove excess hydrogen peroxide. After that, it was soaked in 10 % (v/v) acetic acid at room temperature for half an hour to remove excess sodium hydroxide and washed with water. The resulting pulp was dried in an oven at 100 °C for 6 hr. Dried pulp powder 30 g was treated with 600 mL of 20 % (w/v) sulphuric acid solution for 1 hr at 45 °C. After acid hydrolysis, it was soaked in sixfold of cold water to stop the reaction. After that it was washed with purified water until it was neutralized. It was then homogenized using ultra-turrax for 30 min. The resulting cellulose fiber was dried in an oven at 100 °C for 6 hr.

Table 1 Variables in Experimental Design for Pulping

Variables	Levels	
	Lower	Upper
Alkali Concentration% (w/v)	40	50
Alkali Volume (mL)	450	550
Hydrolysing time (hr.)	3	5

Table 2 Experimental Runs According to Central Composite Design for Pulping of Betel Nut Fiber

Run Order	Alkali Concentration (% w/v)	Alkali Volume (mL)	Hydrolysing Time (hr)
1.	50.00	550.00	5.00
2.	40.00	450.00	3.00
3.	45.00	415.91	4.00
4.	45.00	500.00	4.00
5.	36.59	500.00	4.00
6.	50.00	450.00	5.00
7.	45.00	500.00	5.68
8.	53.41	500.00	4.00
9.	45.00	500.00	4.00
10.	45.00	584.09	2.32
11.	45.00	500.00	3.00
12.	50.00	450.00	3.00
13.	40.00	550.00	5.00
14.	40.00	450.00	3.00
15.	50.00	500.00	3.00
16.	40.00	550.00	5.00
17.	45.00	500.00	4.00

Characterization of Cellulose Pulp and Cellulose Fiber of Betel Nut Fiber**Scanning Electron Microscopy (SEM)**

Morphological study of raw material, cellulose pulp and cellulose fiber was conducted using JSM-5610, Jeol Ltd., Japan, scanning electron microscopy at the Universities' Research Center, University of Yangon.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was carried out to examine the changes in the functional groups in raw material, cellulose pulp and cellulose fiber. FTIR-8400, SHIMADZU, Japan was used to obtain the spectra of each sample. FTIR analysis was carried out at the Universities' Research Center, University of Yangon.

X-Ray Diffraction (XRD)

XRD analyses were performed for raw material, cellulose pulp and cellulose fiber using Regakuminiflex 600 Detax, Tokyo, Japan. XRD analysis was carried out at the Department of Physics, University of Yangon.

Thermogravimetric Analysis (TG-DTA)

Thermal stability of raw material, cellulose pulp and cellulose fiber were analyzed using Pyris Diamond TG-DTA High Temp.115V thermal analyzer DTG-60H. TG-DTA analysis was carried out at the Universities' Research Center, University of Yangon.

Determination of Alpha Cellulose Contents

Alpha cellulose was determined according to (ASTM 1104-56, 1978) and (TAPPI T 203 cm-99 (TAPPI 1999b)). Holocellulose, alpha cellulose and hemicellulose contents in resulting pulp were determined. Determination of holocellulose content was carried out according to the modified method of (Wise *et al.*, 1946). Pulp 2 g was added to in a 250 mL Erlenmeyer flask. Pulp was dissolved in a solution of 1.5 g of sodium chlorite (NaClO₂) in 100 mL distilled water and 5 mL of 10% (v/v) acetic acid. The flask was placed into a water bath and kept at 70 °C for 30 min with frequent stirring (Wise *et al.*, 1946). Furthermore 5 mL of acetic acid and 1.5 g NaClO₂ until 6 g of NaClO₂ had been added, after which, the flask was further heated for 30 min after the last addition of NaClO₂. It was then cooled to 10 °C. It was washed with acetone and allowed to be air-dried. Finally, the residue was transferred into desiccators and weighed until a constant weight was obtained.

Alpha-cellulose content of pulp was determined according to TAPPI T203 cm-99 (TAPPI 1999b). The holocellulose containing crucible was placed in a Syracuse watch glass, which contained water to a depth of 1 cm and 3 mL of 17.5 % (w/v) NaOH were added to each crucible. After 5 min, additional 3 mL of 17.5 % (w/v) NaOH solution was added. The contents were allowed to stand for 35 min, a total of 42 min contact time. After washing with 60 mL of distilled water, 5 mL of 10 % (v/v) acetic acid was added and 5 min later, the alpha-cellulose was washed with distilled water 60 mL, and then finally with acetone 20 mL. After washing, it was oven-dried to a constant weight, and the alpha-cellulose content calculated based on oven-dried weight. The hemicellulose content was calculated by subtracting the weight of alpha-cellulose from that of holocellulose (TAPPI 1999b).

Results and Discussion

Pulping by alkali treatment with 17 experimental runs was conducted according to the experimental design. The second-order polynomial quadratic regression equations are stated in Eqns. (1) to (3). By solving these equations in matrix notation using MATLAB, the predicted and actual yield of celluloses are shown in Table (3). As can be seen in the table, high content of holocellulose (94.33±0.76) and alpha cellulose (80.17 ± 1.89 % (w/w)) have resulted. It is indicated that delignification and opening the structure of biomass was related to produce the alpha cellulose. The increase of alpha cellulose during the pulping process indicated the content of lignin and hemicelluloses decrease in the pulping and lignin was almost completely removed during bleaching (Soliman *et al.*, 2017). The low level of alpha cellulose content indicates that the cellulose structure might be damaged (Kopania *et al.*, 2012). Alkali treatment has a positive effect on the mechanical properties and interfacial adhesion of various types of natural fibers (Lazim *et al.*, 2014). During chemical pulping, lignin and a large proportion of the hemicelluloses were removed and it opened up the fiber bundles. In addition, the high content of alpha cellulose indirectly indicated no damage of cellulose structure and cellulose content is the equal fiber content of pulp (Khalil *et al.*, 2006). In soda pulping, NaOH tend to disperse to form alkali cellulose by the reaction of Cel-OH + NaOH → Cel-O-Na+H₂O (Kunusa *et al.*, 2018). Three dimensional surfaces and contour plots were designed to investigate the interaction among the variables and to determine the optimum condition of each factor for maximum alpha cellulose content. The plots are shown in Figure 1 (a), (b) and (c).

$$\begin{aligned} \text{Holocellulose Content (\%)} &= +77.11 - 0.19x_1 + 1.09x_2 - 6.59x_3 + 0.94x_1x_2 - \\ &0.31x_1x_3 - 1.06x_2x_3 + 2.16x_1^2 + 2.25x_2^2 + \\ &3.58x_3^2 \dots\dots\dots \text{Eq}^n \ 1 \end{aligned}$$

$$\begin{aligned} \text{Alpha Cellulose Content (\%)} = & +63.13-2.17x_1+ 1.27x_2 -6.02x_3+0.31x_1x_2- \\ & 2.56x_1x_3-0.19x_2x_3+2.25x_1^2 +2.07x_2^2 + \\ & 3.75x_3^2 \dots\dots\dots \text{Eq}^n \ 2 \end{aligned}$$

$$\begin{aligned} \text{Hemicelluloses Content (\%)} = & +13.98+2.05x_1- 1.98x_2-0.57x_3 +0.63x_1x_2 + \\ & 2.25x_1x_3 - 0.88x_2x_3-0.086x_1^2 +0.18x_2^2- \\ & 0.17x_3^2 \dots\dots\dots \text{Eq}^n \ 3 \end{aligned}$$

Table 3 Yield of Cellulose from the Pulping of Betel Nut Fiber under Optimum Conditions

Sr. No.	Alkali Concentration % (w/v)	Alkali Volume (mL)	Hydrolysing Time(hr)	Composition of Pulp	Predicted Yield % (w/w)	Actual Yield % (w/w)
1	44.40	475.60	3.08	Holocellulose	80.17	94.33±0.76
2	47.60	463.62	4.10	Alpha Cellulose	63.84	80.17±1.89
3	45.84	491.18	4.66	Hemicelluloses	13.88	13.83±1.75

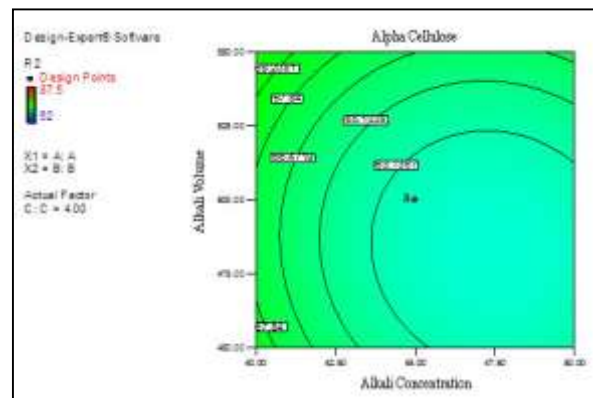
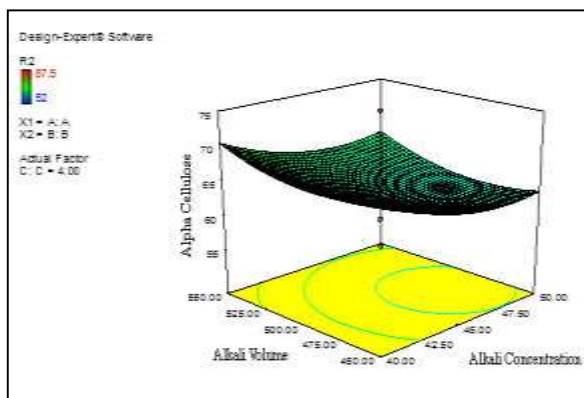


Figure 1 (a) 3-D Surface and Contour Plots of Alpha Cellulose Content for Betel Nut Fiber as a Function of Alkali Concentration and Alkali Volume

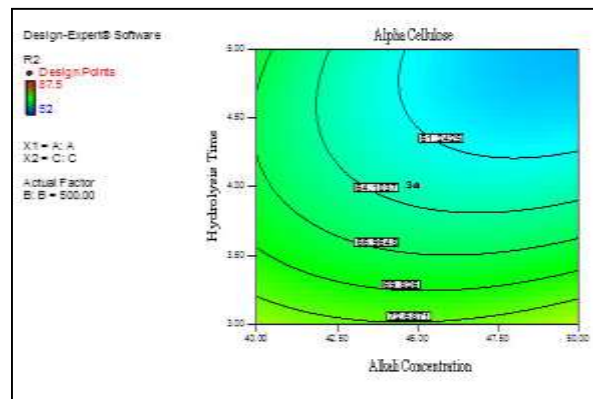
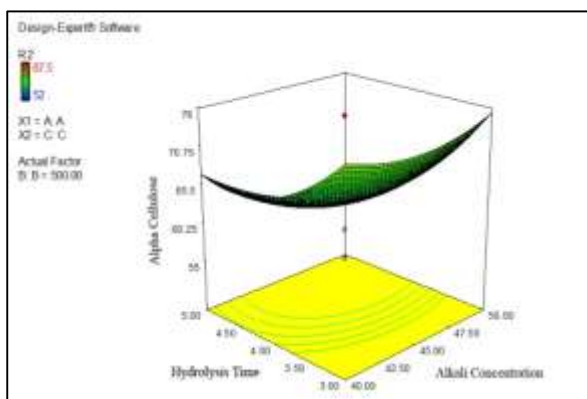


Figure 1 (b) 3-D Surface and Contour Plots of Alpha Cellulose Content for Betel Nut Fiber as a Function of Alkali Concentration and Hydrolysing Time

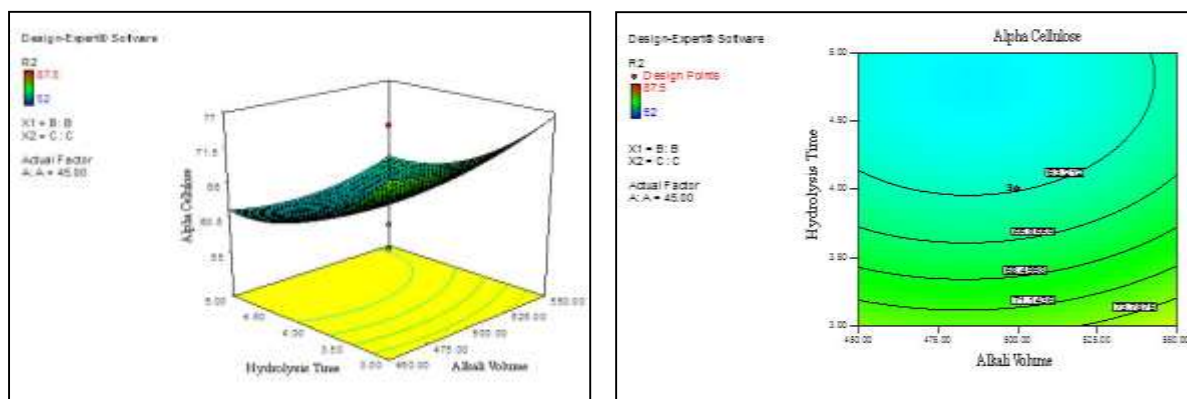


Figure 1 (c) 3-D Surface and Contour Plots of Alpha Cellulose Content for Betel Nut Fiber as a Function of Alkali Volume and Hydrolysing Time

After alkali and acid treatment, lignin and hemicelluloses were broken down and cellulose was separated. So the changes in structure of raw material, pulp and cellulose fiber were observed. Figure (2) represents the FTIR spectra of the raw material, cellulose pulp and cellulose fiber. All the samples exhibited a broad band in the region 3500 cm^{-1} - 3200 cm^{-1} that indicates the free O-H stretching of O-H groups in cellulose molecules. The prominent C-H stretching band was found at 2887.53 cm^{-1} , 2893.32 cm^{-1} , 2895.25 cm^{-1} and 2926 cm^{-1} . The peak at 1647.26 cm^{-1} for betel nut fiber and 1635.69 cm^{-1} in pulp were observed due to the -OH bending of absorbed water. The water molecule in the cellulose is very difficult to extract due to the cellulose-water interaction. The moisture was slightly absorbed in the space left vacant from the removal of hemicelluloses and lignin due to the alkali treatment. The higher moisture content is probably due to the higher cellulose content (Sofla *et al.*, 2016). Lignin presented characteristic peak in the range 1200 cm^{-1} - 1300 cm^{-1} corresponding to the aromatic skeletal vibration. These peaks are associated with C-O stretching of hemicelluloses and lignin. These peaks were not observed in pulp and cellulose fiber which indicated the removal of hemicelluloses and lignin. The high contents of alpha-cellulose and holocellulose were obtained as a result of the delignification and refining treatments, the opening of the lignocellulosic structure of the pulps, causing hemicelluloses hydrolysis and cleavage of lignin-hemicelluloses bonds, resulting in the removal of most of the hemicelluloses and almost all of the pulp lignin (Guimaraes *et al.*, 2015). The alkaline cellulose is formed by stretching C-O and C-C group at around 1060 cm^{-1} . It was observed that these stretchings were found in the spectra of cellulose fibers. Beta glucosidic linkages between glucose units in cellulose presented the peak at around 900 cm^{-1} . The appearance of the signal at 896.93 cm^{-1} and 895.50 cm^{-1} were observed in pulp and cellulose fiber which is a typical structure of cellulose. The presence of this peak showed the increase in the percentage of cellulosic components after removal of non-cellulosic materials by chemical treatments (Wicaksono *et al.*, 2013). After the chemical treatment, the absorption bands at 1730 cm^{-1} , 1620 cm^{-1} , 1595 cm^{-1} and 1512 cm^{-1} were not observed on the spectrum of pulp and cellulose fiber. The bands at 1512 cm^{-1} and 1250 cm^{-1} were drastically reduced in FTIR spectrum of the pulp and cellulose fiber which indicated that most of the lignin was removed. After acid hydrolysis and homogenization treatment, the spectra of cellulose fiber were fairly close to that of the pulp.

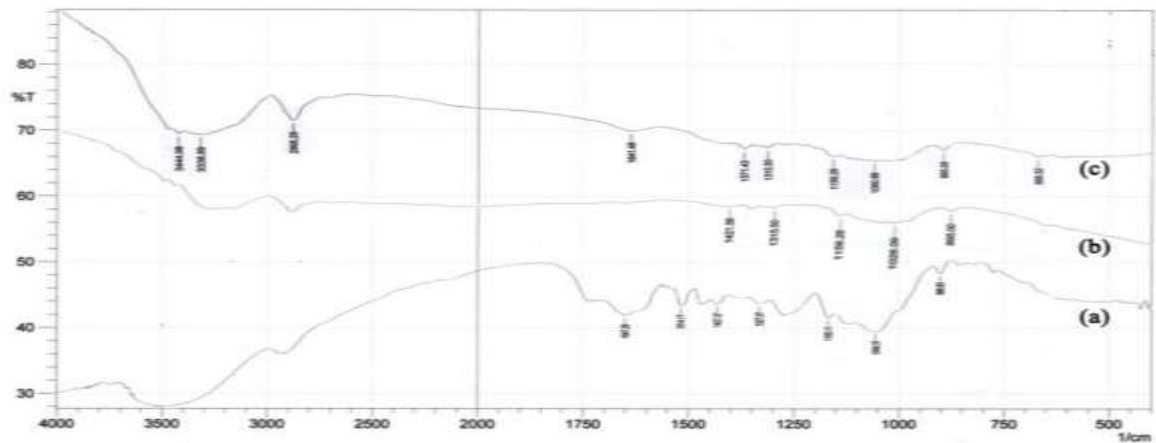


Figure 2 FTIR Spectra of (a) Betel Nut Fiber (b) Its Cellulose Pulp and (c) Its Cellulose Fiber

Figure (3) reveals the SEM images and represents the structure and shape of fiber matrix in betel nut fiber, cellulose pulp and cellulose fiber. The raw fiber was uneven and tangled with lignin binder. The raw fiber is composed of individual cells that are bound together by cemented components of lignin and hemicelluloses. The diameter of the betel nut fiber was found to be around 86.14 μm . After soda pulping, the resulted fiber had smooth and clear surfaces. It was observed that the surface of the fiber look smoother than the raw fibers due to the removal of the bundles. The bleaching process decreased the fiber diameter because of the removal of lignin. The bleaching treatment modified the surface of the fibers and it became smooth (Ireana *et al.*, 2014). After pulping and bleaching process, the fiber diameter for betel nut fiber further reduced to 9.92 μm respectively. Specifically, alkali treatment brought about the leaving pit-like pores on betel nut fiber. This was due to the removal of waxy layers from the surface (Lazim *et al.*, 2014). Acid treatment of pulp could also weaken the structure of aggregate fibers and defibrillation occurred (Wicaksono *et al.*, 2013). It can also be seen that the cellulose fibers were smoother than the raw material and pulp. The size of the cellulose fiber was found to be 8.01 μm .

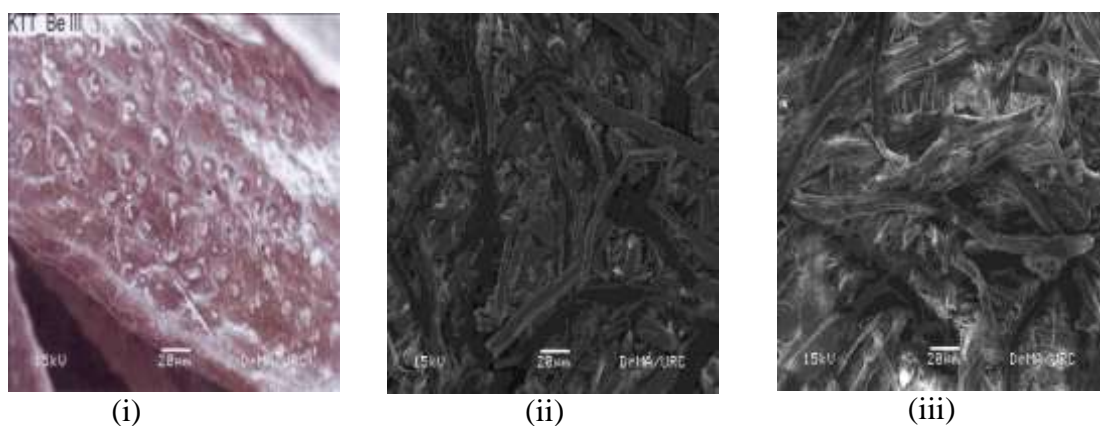


Figure 3 SEM Images of (i) Betel Nut Fiber (ii) Its Cellulose Pulp and (iii) Its Cellulose Fiber

Figure (4) shows the XRD patterns of the raw, pulp and cellulose fiber of the betel nut fiber. These were possible to observe a major diffraction peak for 2θ ranging between 22° and 23° , which corresponds to cellulose (002) crystallographic planes (Pereira *et al.*, 2011). The spectra corresponding to the untreated betel nut fiber showed wider diffraction pattern observing at 2θ ranging 22° . It was found for 2θ ranging between 12.32° and 22.30° for cellulose pulp and cellulose fiber of betel nut fiber. The high intensity peak associated with pulp and cellulose fiber was due to the removal of lignin and hemicelluloses with the consequent increase in the cellulose

index. The sharp diffraction peak is an indication of high crystallinity degree in the structure of the treated fibers (Alemdar and Sain, 2008). In diffraction pattern the narrower peaks indicates crystalline part of the material while the amorphous part of fibers presents as wider peaks. The Bragg angles of 16° , 22° and 35° are characteristics of cellulose and the peak corresponding to crystalline region that is located in a Bragg angle 2θ of 22° (Fonseca *et al.*, 2015). Cellulose consists of both amorphous and crystalline regions whereas lignin and hemicelluloses display a completely amorphous structure. The sharp diffraction pattern was observed for the pulp and cellulose fiber. The structure of hemicelluloses was easily hydrolysed while the structure of cellulose was not easily broken by acid hydrolysis. Therefore, more sharper diffraction pattern was found in cellulose fiber than in pulp.

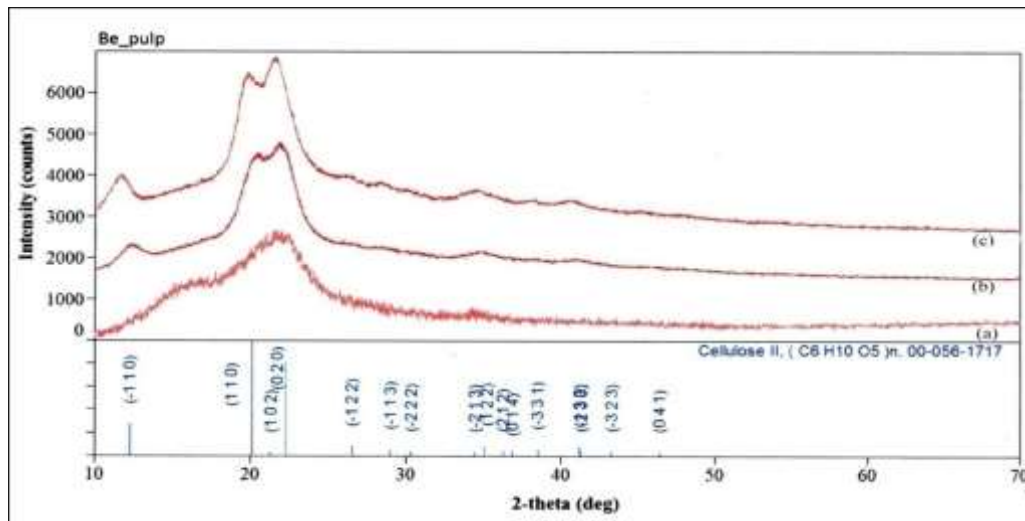
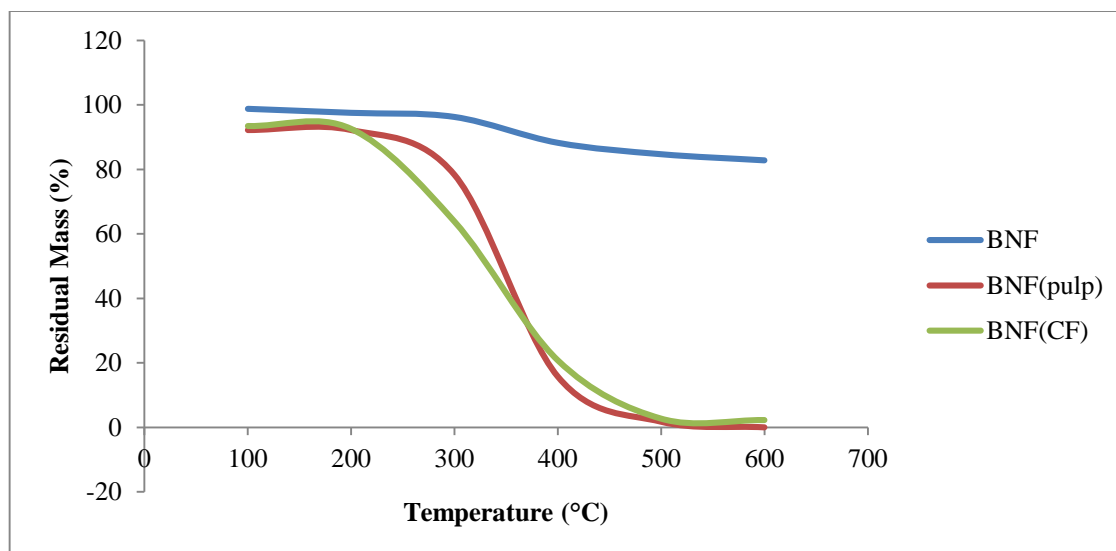


Figure 4 X-Ray Diffraction Analysis of (a) Betel Nut Fiber (b) Its Cellulose Pulp and (c) Cellulose Fiber

The resulting TGA curves as shown in Figure (5) indicated that small mass of loss was found at around 100°C due to the evaporation of absorbed moisture. According to the results, thermal stability of betel nut fiber is greater than that of pulp and cellulose fiber. Because betel nut fiber has higher lignin content. Lignin is different from hemicelluloses and cellulose, because it is composed of three kinds of benzene-propane units, being heavily cross-linked and having very high molecular weight. The thermal stability of lignin is thus very high, and it is difficult to decompose (Poletto *et al.*, 2014). The curves of the raw material, cellulose pulp and cellulose fiber showed several different decomposition stages, which indicated the presence of different components. The temperature range where maximum mass loss rate occurred was related to the decomposition of hemicelluloses ($225\text{--}325^\circ\text{C}$), lignin ($250\text{--}500^\circ\text{C}$) and cellulose ($305\text{--}375^\circ\text{C}$) as reported (Guimaraes *et al.*, 2015). Betel nut fiber showed a lower original decomposition temperature about 220°C due to the presence of hemicelluloses and lignin. Pulp degradation began at a temperature approximately 330°C due to the removal of hemicelluloses and lignin after chemical extraction. Bleaching increased the thermal stability of the pulp (Guimaraes *et al.*, 2015). Therefore, TG-DTA curves obtained for cellulose fiber revealed difference in thermal behavior in comparison to that of the pulp and raw materials due to the presence of sulfate groups, which promote dehydration reactions that release water and which can influence cellulose degradation reactions (Corradine *et al.*, 2016).



BNF = Betel Nut Fiber
 BNF (pulp) = Cellulose Pulp of Betel Nut Fiber
 BNF (CF) = Cellulose Fiber of Betel Nut Fiber

Figure 5 Thermogravimetric Analysis Graph of Betel Nut Fiber, Its Cellulose Pulp and Its Cellulose Fiber

Conclusion

In this study, cellulose fiber was isolated from betel nut fiber through four steps, including alkali treatment, bleaching, acid hydrolysis and homogenization. Acid and alkaline conditions degrade lignin and hemicelluloses preserving the cellulose. After acid hydrolysis and homogenization, the spectra of cellulose fiber were fairly close to that of the pulp. The size of the cellulose fiber was observed to be 8.01 μ m. The sharp diffraction pattern was observed in cellulose fiber than in pulp and raw materials. TG-DTA analysis expressed that cellulose fiber revealed difference in thermal behavior in comparison to that of the pulp and raw materials due to the presence of sulfate groups.

Acknowledgements

I would like to express my gratitude to the Myanmar Academy of Arts and Science for allowing me to submit this article.

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