

## Comparison of cellulose properties from different agriculture biomass

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**Abstract.** Cellulose is one of the most abundant molecules on the planet that presence in all plant cell walls on Napier grass and bamboo. Cellulose has been extensively used in industries like textile, food, composites, and pharmaceuticals, due to its outstanding properties. This research project aims to obtain a yield of cellulose from Napier grass, bamboo and Kenaf fiber by manipulating the operating condition of alkaline treatment. The extracted cellulose fiber was then being characterized to identify its yield, chemical composition, and crystallinity of the fiber by applying moisture content, chemical analysis, FTIR and X-ray Diffraction. As the temperature of the alkali reaction increases, the cellulose yield decreases due to the formation of purified cellulose after chemical process. Cellulose content increased after the fibers were water retted. The results were then validated by using FTIR analysis, which the amount of hemicellulose and lignin content decreased as the concentration, temperature, and time increases after treatment and the peak intensity band for lignin and hemicellulose reduces which indicate the removal of these two components. XRD analysis showed a declining trend for its crystallinity index as the temperature elevates. The crystallinity of the treated fibers did not affect the crystalline structure of the cellulose and that water retted fibers have higher crystallinity index.

### Introduction

The development of cellulosic biomass materials has received a great deal of attention in researching their utilization towards relevant applications. According to Sonali Mohapatra et al., biomass is a renewable organic substance and form of sustainable energy that originates from various resources that give multiple benefits economically and environmentally [1]. In the last three decades, biomass energy has been the most widely used renewable energy source after hydropower (Annual Energy Review, 2016). Biomass is a cheap, safe, and easily accessible resource that minimizes the world's major pollution, emissions of greenhouse gases. Thus, cellulosic biomass-based materials caught the attention industries and policy makers to further research and transform them into value-added products. Lignocellulosic biomass is a type of polymer that is renewable in which is derived from various sources such as agricultural residues, forestry residues, and energy crops. Cellulose, lignin, and hemicellulose are complexly bundled in

the structure of the cell wall of the lignocellulosic biomass. One of the compositions of the lignocellulosic biomass, which is cellulose, specifically has been by far the most abundant organic polymer

Cellulose is a polymer made up of glucose units joined together by condensation polymerization. Plant-based cellulose fiber has many unique benefits such as high heat resistance, being renewable, and inexpensive [2]. Hence, cellulose is used in a wide range of products such as synthetic products, thickening agents in shampoo, cosmetics, plastics, and even fabrics. However, various sources of cellulose are utilized in different applications. Generally, the cellulose that is isolated from hardwood fibers such as bamboo is commonly used as a raw material for the pulp and paper industry to make paper, fiberboard, sanitary pads, and other related products. Furthermore, cellulose fibers can be extracted from a hardwood plant in different ways to achieve high purity of cellulosic fiber. Barbash and Yashchenko states that parameters and conditions in the extraction process immensely affect the physical and mechanical properties of cellulose [3]. The statistic of the global cellulose market by end-use industry displayed that paper and pulp industry encompassed the largest share of the global cellulose market while closely followed by textiles, chemical synthesis and construction [4]. Due to the concern of sustainability and waste issue created by the synthetic materials, natural resources have become an attractive alternative instead of the synthetic which led to further demand for cellulose-based materials.

In this study, cellulose was extracted using the alkaline extraction method with necessary pretreatments to isolate unbleached cellulose fiber from bamboo, Napier grass and Kenaf with varying alkali concentration, temperature, and time of treatment to observe the properties of the extracted cellulose.

## Materials and Methods

### Materials

Napier grass Pakchong (NG) were obtained from Bota, Perak. Napier grass leaves and roots were chopped off and their stems were cleaned using damp cloth to remove dirt. They were then chopped into pieces having the length of approximately 15 cm. Next, the chopped stems were sun-dried for 8 hours before oven-dried for 12 hours at temperature of 105°C. The dried stems were then granulated using granulator and afterwards grinded into 500 microns using a power mill. The grinded Napier grass were then sieved into sizes of 300 µm and 500 µm.

2 types of bamboo were used in this project. Bamboo 1 (*Schizostachyum brachycladum*) (BL) was collected from Kampung Gajah, Perak and Bamboo 2 (*Bambusa Vulgaris*) (BM) used was obtained from Kampung Pintu Pandang, Gopeng, Perak. Bamboo culms were collected in poles of approximately 30cm and they were cleaned with a damp cloth to remove dirt. The bamboo poles were stored in the cold storage room at 10°C before being chopped into small pieces at an approximate length of 5 cm. The bamboo pieces were let to sun-dried for 8 hours and then oven-dried using biomass ovens for 48 hours at 105 °C. The dried bamboos were then granulated using a granulator to an approximate size of 3-5mm, grinded and sieved into 250µm using biomass power mill.

Fresh Kenaf was obtained from Lambor, Perak, Malaysia and the stems were separated from the leaves. The stems from the Kenaf were chopped into small pieces to be around 5cm with a machete for the extraction of fibers from the grass stem internodes. The fibers were then separated into non-water retting (NRK) and water retting (WRK) method. The fibers from Kenaf were water retted by soaking in a bucket of water for 7 days. It was then washed with distilled water followed by drying in the oven at temperature 105°C for 24 hours. The dried samples were then granulated with a granulator followed by grinding by a power mill and sieved into a particle size of 250 microns using a sieve shaker. The samples were then stored separately in polyethylene bags based on the type of feedstocks and retting method for 3 days.

Acetic acid, ethanol, sodium bisulfite, sodium hydroxide pellets, nitric acid, sodium chlorite and toluene used were of analytical grade and purchased from Avantis Laboratory Supply.

#### *Extraction of cellulose process*

Similar processes were done to extract cellulose, but some methods were modified according to each sample. For Napier grass, the methodology was adopted from Reddy et al [5] with a few modifications. NG and BL were dewaxed using Soxhlet extraction apparatus using ethanol: toluene (2:1, v/v) for 6 hours. The samples were then dried for 24 hours at 105 °C. The samples were delignified and bleached using 0.7 % sodium chlorite, acidified using acetic acid at 100 °C for 2 hours. The samples were then filtered and washed with 2 % sodium bisulfite and water. The filtrate was treated with 17.5 % sodium hydroxide solution at 25 °C for 45 minutes. After alkali treatment, it was filtered, washed with 10 % acetic acid and distilled water to neutralize the solution. It was then acid hydrolyzed using 80 % acetic acid and 70 % nitric acid (10:1 ratio) at 120 °C for 20 minutes. The samples were then cooled, washed with 95 % ethanol and water and dried at 105 °C until constant weight. The experiment was repeated using different alkali treatment temperature at 35 °C.

For BM, Soxhlet extraction was used onto 5.0 g of grinded sample using 95% ethanol for 4 hours to remove extractives. The sample was treated according to Fitriana et al. [6] with some modifications. The extractive- free bamboo was then mixed with 50ml aqueous sodium hydroxide (NaOH) solution at 5wt% and 10wt%. The solution obtained was incubated in a shaker flask at 35°C and 45 °C. This incubation process was done in an incubator for 16 hours at 100 rpm. The solid residue of the incubated sample which was the cellulose fibers were filtered using the gravitational filtration method. The solid residue will be washed thoroughly with 100ml deionized water and dried at 45 °C in an oven to remove water until constant weight.

For Kenaf sample, Soxhlet extraction was also used to remove extractive similar to NG and BL. The extractive-free kenaf was then dried at 105 °C and let cooled in a desiccator. It then underwent digestion with 400 mL of hot water at 100 °C for 3 hours and washed with 100 mL of hot water followed by 50 mL of ethanol. The subsequent alkali treatment procedures were adopted from Kommula et al. [7] with some modifications. The extractives-free samples were treated with 5 % (w/v) NaOH solution for 4 hours at temperature of 25°C. A liquor ratio of 30:1 was maintained for the alkali hydrolysis to remove the hemicellulose, lignin, and surface impurities. The extractives-free samples were treated with 5 % and 15 % (w/v) sodium hydroxide, NaOH solution for 4 hours and 6 hours, and at temperature of 25 °C and 35°C. Next, the fibers were neutralized using 1% (w/v) acetic acid solution. The solid residue was filtered, washed with distilled water, and dried for 24 hours at 100 °C. The dry solid residue was soak in a 4% (w/v) NaOH solution at 50°C for bleaching purpose with constant stirring at 200 rpm. Then 30% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) to the same amount of NaOH was added by drops and stirred continuously for 1 hour. The samples were then filtered, washed, and dried until constant weight.

#### *Moisture Content Analysis*

The procedure for conducting the experiment was based on ASTM D4442 with a few changes. The following Eq. 1 used to calculate the moisture content:

$$\text{Moisture content}(\%) = \frac{W_2 - W_3}{W_3 - W_1} \times 100\% \quad (1)$$

where  $W_1$  is the weight of crucible and cover,  $W_2$  is the weight of the sample with crucible and cover and  $W_3$  is the weight of dried sample with crucible and cover.

### *Yield analysis*

Yield analysis was done to observe each sample at different treatment conditions which indicate the fibre quality from the extracted biomass. The yield is calculated based on the ratio of extracted cellulose weight over the initial weight of sample used. Eq. 2 is used to calculate the yield percentage.

$$\text{Yield (\%)} = \frac{W_c}{W_b} \times 100\% \quad (2)$$

where  $W_c$  is dry weight of extracted cellulose after the treatment process and  $W_b$  is dry weight of bamboo sample after Soxhlet extraction.

### *Fourier Transformed Infrared (FTIR) Spectroscopy Analysis*

FTIR with Attenuated total reflectance (ATR) was used to evaluate the pretreated sample. Changes in the intensity of functional group of the pretreated sample are determined and analyzed based on the FTIR spectrum. The spectra were recorded in the frequency range of 4000-400  $\text{cm}^{-1}$  with a resolution of 1  $\text{cm}^{-1}$ . The expected functional groups that shall present were C-H, C-O, and O-H.

### *X-ray Diffraction (XRD) Analysis*

The pretreated samples are scanned in XRD at  $10^\circ \leq 2\theta \leq 40^\circ$  at an exposure time of 1 seconds with a step size of  $0.02^\circ/2\theta$ . The crystallinity index for cellulose is calculated using the empirical method in Eq. 3 below

$$\text{Crystallinity index, CI (\%)} = \frac{I_{002} - I_{AM}}{I_{002}} \times 100\% \quad (3)$$

where  $I_{002}$  is main crystalline plane in which the intensity of the crystalline peak at the maximum  $2\theta$  at  $22.5^\circ$  and  $I_{AM}$  refers to amorphous fraction in which the intensity at the maximum  $2\theta$  at  $16^\circ$ .

## **Results and discussion**

### *Moisture content*

The average moisture content of each sample was shown in Fig. 1. It is important that the moisture content is kept under 5 %. Higher moisture content contributes to less lignocellulose content per weight of sample since much of the weight is contributed by the weight of water molecules.

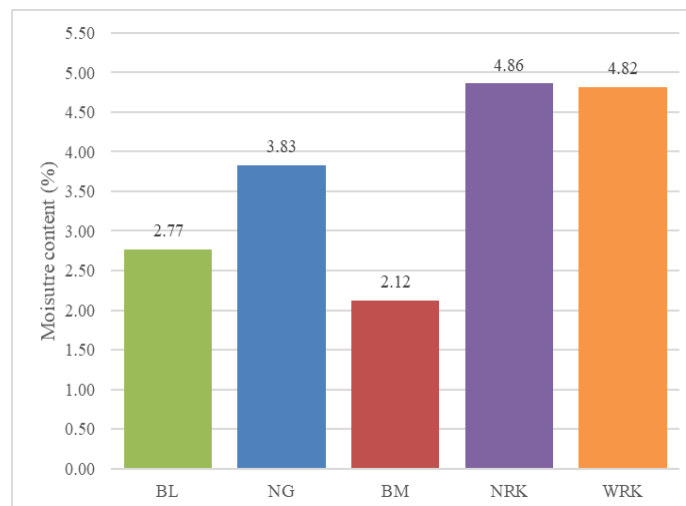


Figure 1: Moisture Content % against the type of sample

### Yield Analysis

The yield of each set is shown in Fig. 2.

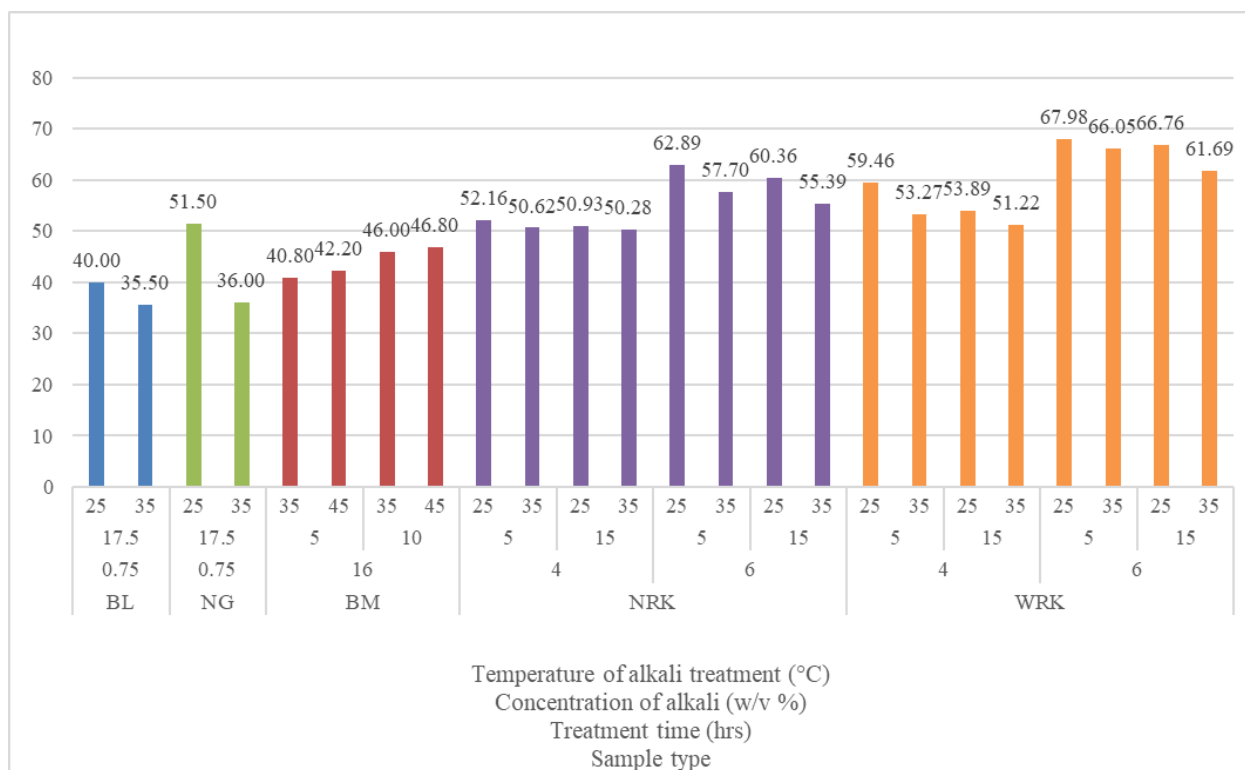


Figure 2: Yield of Cellulose (%) against Temperature, Concentration of alkali, Treatment Time, and Sample type

#### Bamboo BL and Napier grass

When comparing between BL and NG, NG seems to have higher extracted cellulose. It is seen that the yield decreases as the temperature of alkali treatment increases. The decline in NG is much more significant than BL. This is due to the enhanced corrosive nature of sodium hydroxide at higher temperature [8]. This also causes the extracted samples to be finer and easily distinguishable. Using high temperatures for alkaline conditioning, problems such as the formation of toxic compounds and loss of sugars have been reported [9].

#### Bamboo BM

For BM, it is found that the yield of cellulose increases with both increment of concentration and temperature. It is significant that treatment conditions using 10 wt.% of NaOH at 45 °C have the highest yield percentage. This shows a similar trend of increases in cellulose yield along with the temperature. However, the increase of incubation temperature beyond 45 °C no longer shows significance in the yield of cellulose. Therefore, it can be concluded that the optimum temperature for BM using alkaline treatment is at 45 °C.

#### Kenaf

It is found that water retted samples of Kenaf produces higher extracted lignin. The objective of water retting in this experiment was to allow water to penetrate the portion of central stalk and burst the outer layer through swelling the inner cells. This process enhances the absorption of moisture as well as bacteria that decomposition. This allows the substances surrounding the fibers such as cellular tissues and gummy substance to rot away while also separating the fibers from the

stem. Water retting also removed substances surrounding the fibers such as pectin and other impurities thus increasing the proportion of cellulose within the fibers.

Increase in temperature and concentration causes the yield to decrease. Similar to NG and BL, this is due to the more intense disruption from the alkali. The time effect improved the yield where 6 hours produces more cellulose than 4 hours. Longer reaction time is found to improve component penetration and better in removing the hemicellulose and impurities resulting in higher yield.

#### *FTIR analysis*

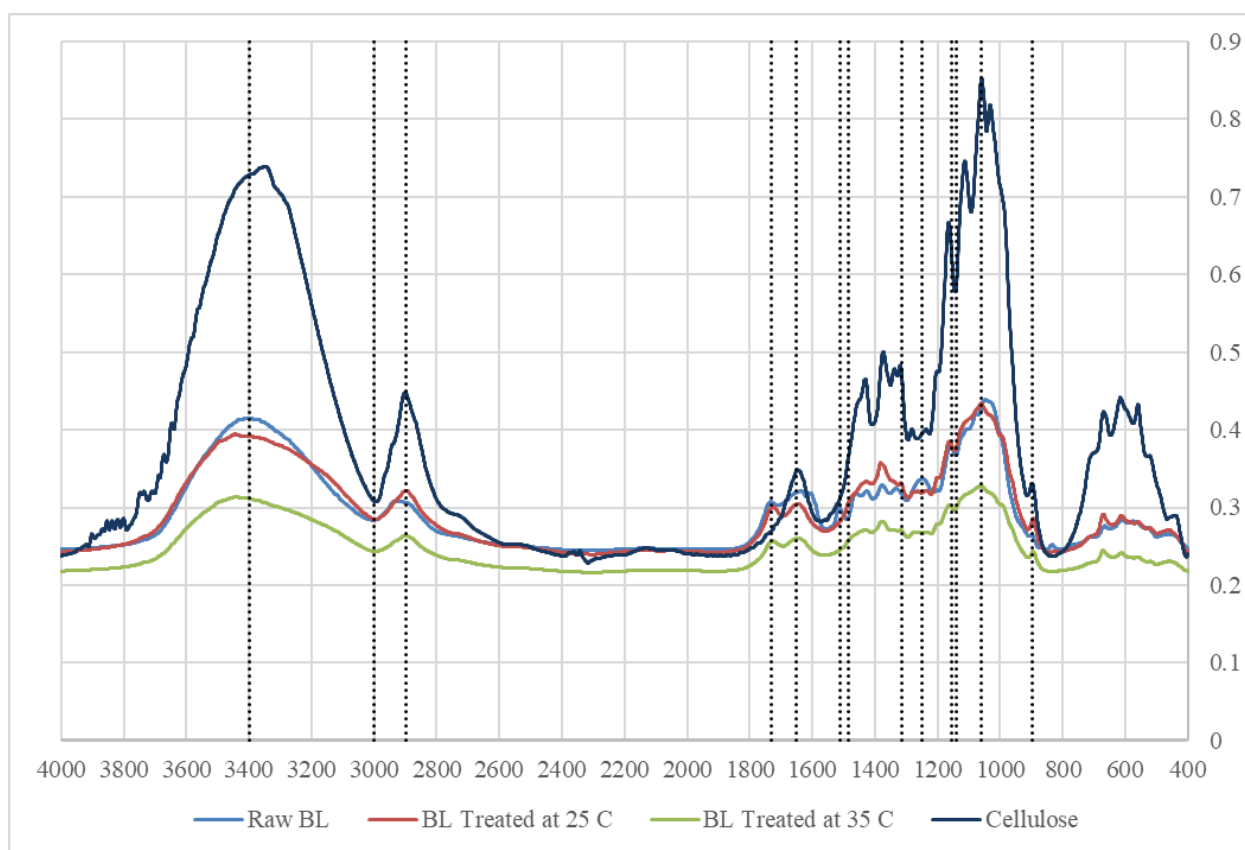
Characterization of functional group and chemical composition were analyzed using FTIR analysis. For cellulose, there are several functional groups that should be observed such as hydroxyl, carbonyl, methoxyl, carboxyl, aromatic and phenolic. Assignment of functional group to FTIR spectrum wavenumber are listed in Table 1 below.

*Table 1: Assignment of functional group to FTIR spectrum wavenumber [10-12]*

Wavenumber (Cm <sup>-1</sup> )	Assignments	Band assignment
3600-3100	O-H	Stretching vibration of alcoholic and phenolic OH groups involve in hydrogen bonds
2960-2820	C-H	-CH <sub>2</sub> , -CH <sub>3</sub>
1770-1685 1680-1650	C=O	Conjugated p-substituent carbonyl and carboxyl
1600-1500, 1430-1420	Aromatic skeletal	Aromatic ring vibrations
1515-1511	C=C	Aromatic skeletal breathing with C-O stretching
1470-1450, 1370-1360	C-H	C-H deformations methyl and methylene
1427-1423	C-H	Aromatic skeletal vibrations combined with C-H in-plane deformation
1375-1397	O-H C-H	Phenolic OH and aliphatic C-H in methyl groups
1170-1150	C-H	Aromatic C-H in-plane deformation in the guaiacyl ring
1145-1140	C-H	Aromatic C-H in-plane deformation in the Syringyl ring
1035-1025	C-O, C-H	Aromatic ring and primary alcohol

#### *Bamboo BL and Napier grass*

The spectra of raw BL, treated BL and pure commercial cellulose are shown in Fig. 3. For BL, it is found that the FTIR spectra after alkali treatment at 25 °C is almost similar to the untreated sample in which the only slight decrease in absorbance intensity is seen. This shows that the treatment is not as effective to produce cellulose. At this temperature, the alkali might not penetrate the lignocellulosic matrix in which only the surface is degraded and causes the yield to be only at 40.0 %. When the temperature was increased, a prominent impact is observed where the absorbance spectra is much lower. This means that the increase in temperature breaks much of the chemical bonds. The intensity at 1730 cm<sup>-1</sup> decreased for after alkali treatment which clearly showed hemicellulose removal after the chemical treatment. The band near 1514 cm<sup>-1</sup> and 1250 cm<sup>-1</sup> which indicated the aromatic skeletal vibration and C-O stretch of ester group was significantly reduced as compared to the raw sample. Also, it is worth noting that the peak near 897 cm<sup>-1</sup> was presented as the C-H group in cellulose is noticeable after treatment which shows that cellulose is obtained.



*Figure 3: FTIR Spectrum for Bamboo in terms of its absorbance*

The spectra of raw NG, treated NG and pure commercial cellulose are shown in Fig. 4. The spectra for NG after alkali treatment is much distant when compared to raw NG. Similar to BL, increase in temperature causes the spectra to be shifted downwards indicating much of the chemical bonds are broken at higher temperature. Looking at the peaks at 1250 cm<sup>-1</sup>, 1514 cm<sup>-1</sup> and 1730 cm<sup>-1</sup>, the peaks seem to diminish suggesting delignification is successful. The peaks at 897 cm<sup>-1</sup> for both treated samples are more prominent as compared to the untreated sample and it is similar to the peak at pure cellulose. This shows that cellulose is well extracted from the NG samples.

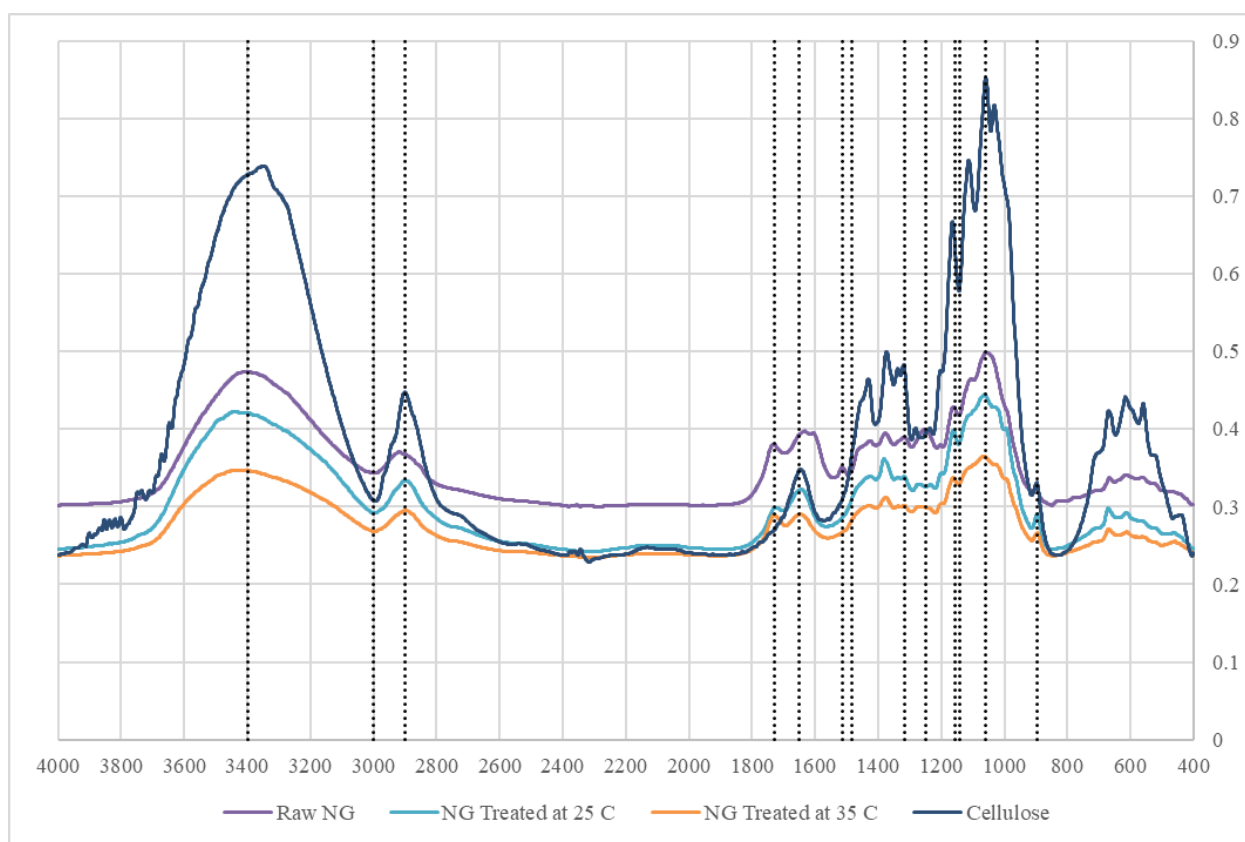


Figure 4: FTIR Spectrum for Napier Grass in terms of its absorbance

#### Bamboo BM

The spectra for untreated BM, treated BM and pure commercial cellulose is shown in Fig. 5. It is noticeable that all 4 conditions of treatments show a similar trend of the spectrum. The C=C double bond stretching at  $1513\text{ cm}^{-1}$  which attributes to the aromatic ring group in lignin had totally disappeared in the treated samples. This corresponds to the removal of lignin from the alkaline treatment process. The O-H bending group at  $1650\text{ cm}^{-1}$  still shows dominance in the peak after the treatment. This is because, the O-H bending is assigned as the interaction with water on the molecular surface [13]. Thus, hydrophilic cellulose strongly interacts with water and remains the O-H bending. Cellulose structure does not dissolve in water, but it can absorb a large quantity of water which causes it to swell. Similar to BL and NG, the peaks at  $897\text{ cm}^{-1}$  which indicates the presence of  $\beta$ -glycosidic linkages which associates with the C-H group are visible after alkali treatment [14]. The bamboo samples that were treated with alkaline treatment method using NaOH indicates that the extracted cellulose samples were mainly compromised of cellulose structure.



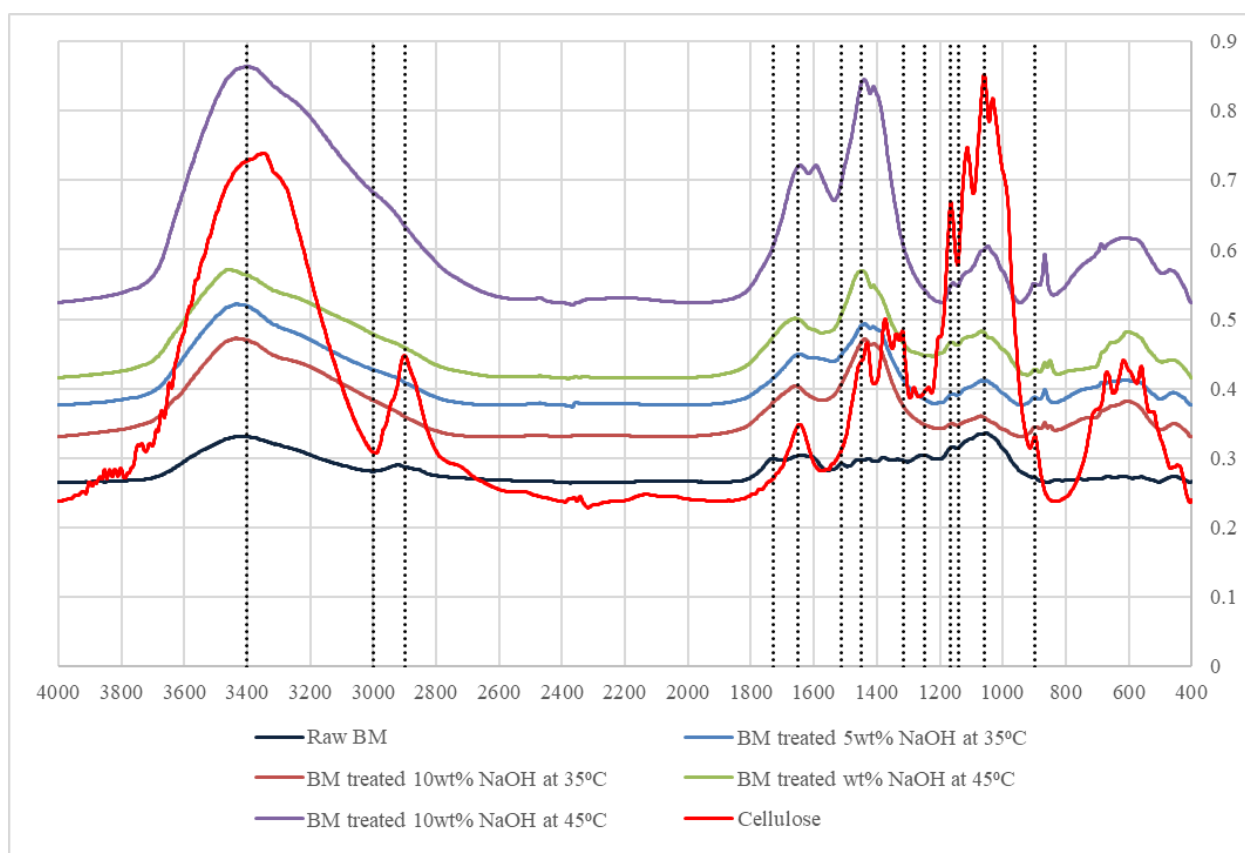


Figure 5: FTIR Spectrum for Bamboo BM in terms of its absorbance

### Kenaf

The spectra of NRK, WRK and Kenaf samples after alkali treatment are shown in Fig. 6, 7, 8 and 9. When comparing the NRK and WRK before alkali treatment, it is seen that the peaks are almost similar but differing in absorbance values. The biggest difference between NRK and WRK is at  $3339\text{ cm}^{-1}$  where WRK produces much higher absorbance. This is due to the swelling effect after the sample are soaked in water and accumulation of water molecules.

After alkali treatment, the FTIR spectra appear to be lower when comparing with before the treatment. It can be seen that the treated WR-Kenaf have higher absorbance than treated Kenaf following the same trend of untreated WR-Kenaf and Kenaf. This is due to the water retting process removing the surrounding substance around the fibers such as pectin and gummy substances thus increasing the quality of the fibers resulting in increasing amplitude intensity of treated WR-Kenaf fibers than treated Kenaf fibers. similar absorption bands and the band at  $1240$  and  $1735\text{ cm}^{-1}$  can be seen to decreases as compared to the untreated fibers. This shows the C-O stretching of acetyl of hemicellulose and C=O stretching vibration in ester groups of hemicellulose to decreases. As for the absorption band at  $1505\text{ cm}^{-1}$  shows the C=C stretching vibrations in aromatic rings of lignin still having a peak. This shows that the lignin component was not fully removed during the treatment. Although traces of lignin component were present, the absorption band at  $1505\text{ cm}^{-1}$  of treated fibers were lower than the untreated fibers. can be seen that for concentration of 15% NaOH have higher absorbance than concentration 5% NaOH that have lower absorbance. As for the 5% NaOH, the soaking time of 6h have lower absorbance as compared to soaking time of 4h. As for K 15% 4h, the absorption bands do not follow the trend of the other treated fibers though it shows the decreasing in peak at  $1505\text{ cm}^{-1}$  indicating full removal of lignin.

This can be considered pure cellulose although a replication of FTIR analysis could uncover the reason on different trend.

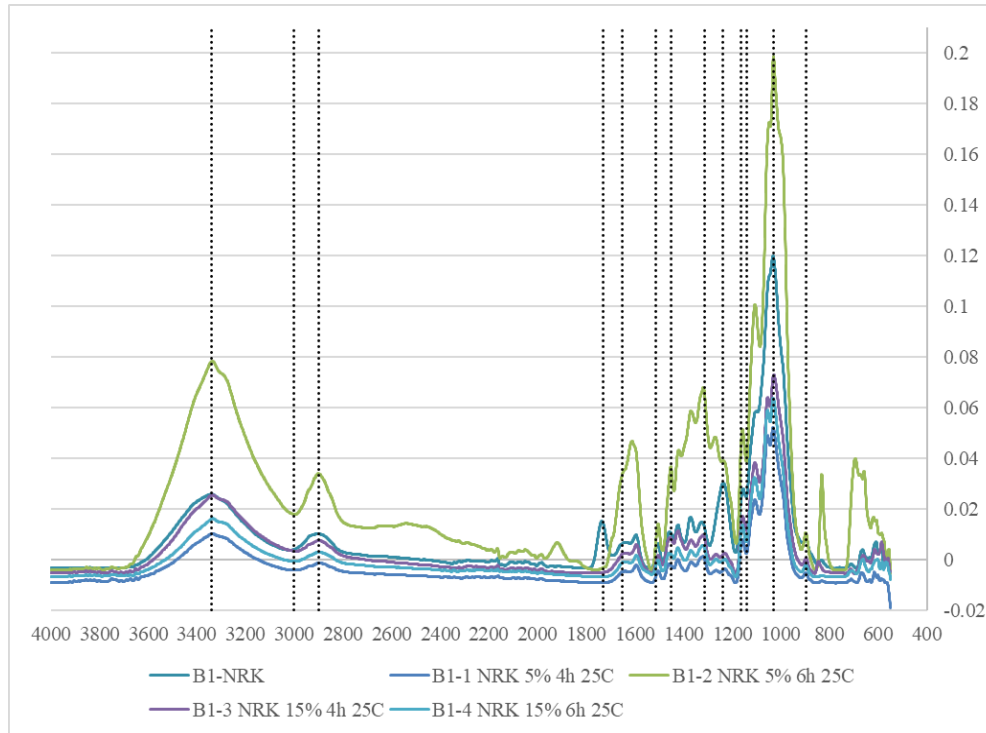


Figure 6: FTIR Spectrum for NR-Kenaf at 25 °C in terms of its absorbance

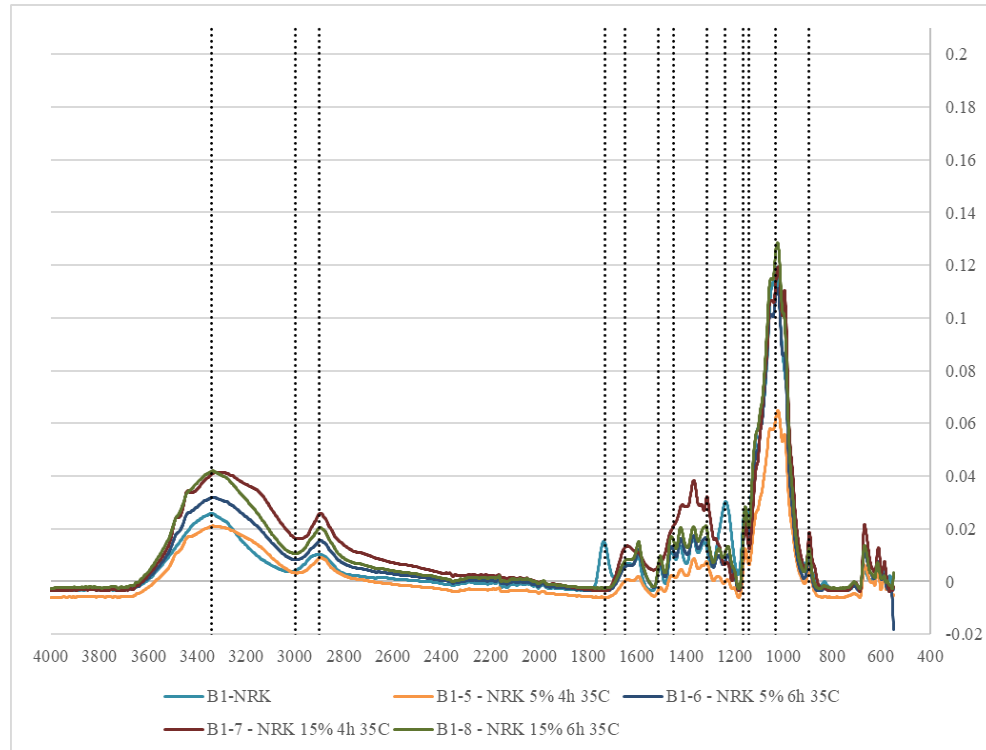


Figure 7: FTIR Spectrum for NR-Kenaf at 35 °C in terms of its absorbance

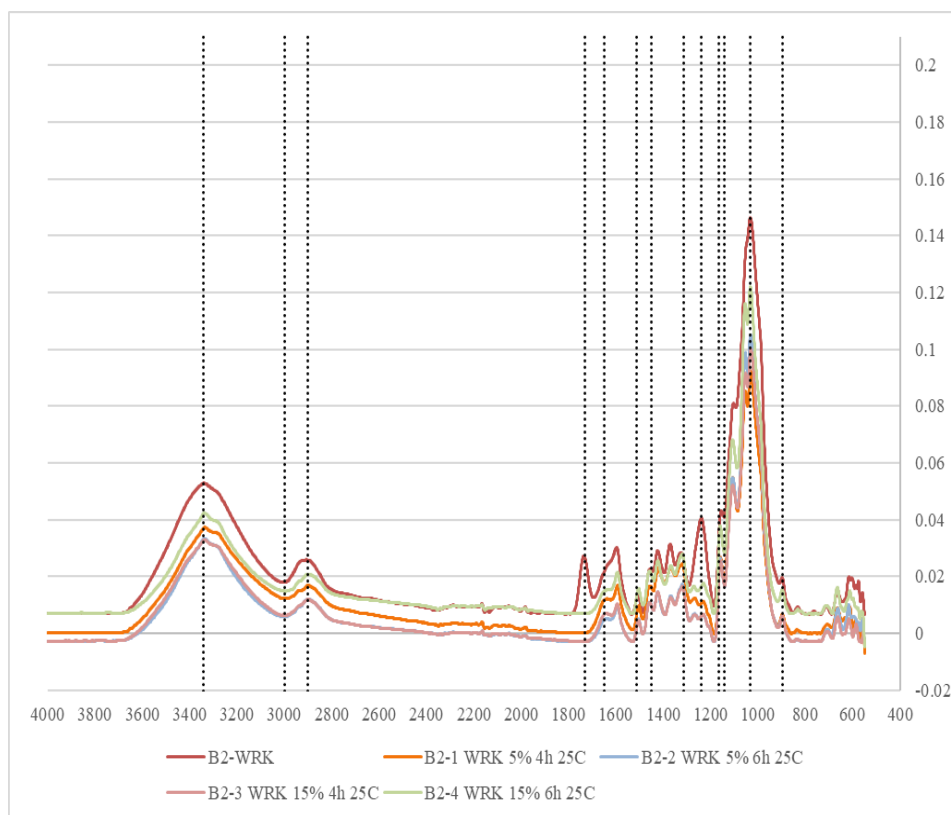


Figure 8: FTIR Spectrum for WR-Kenaf at 25 °C in terms of its absorbance

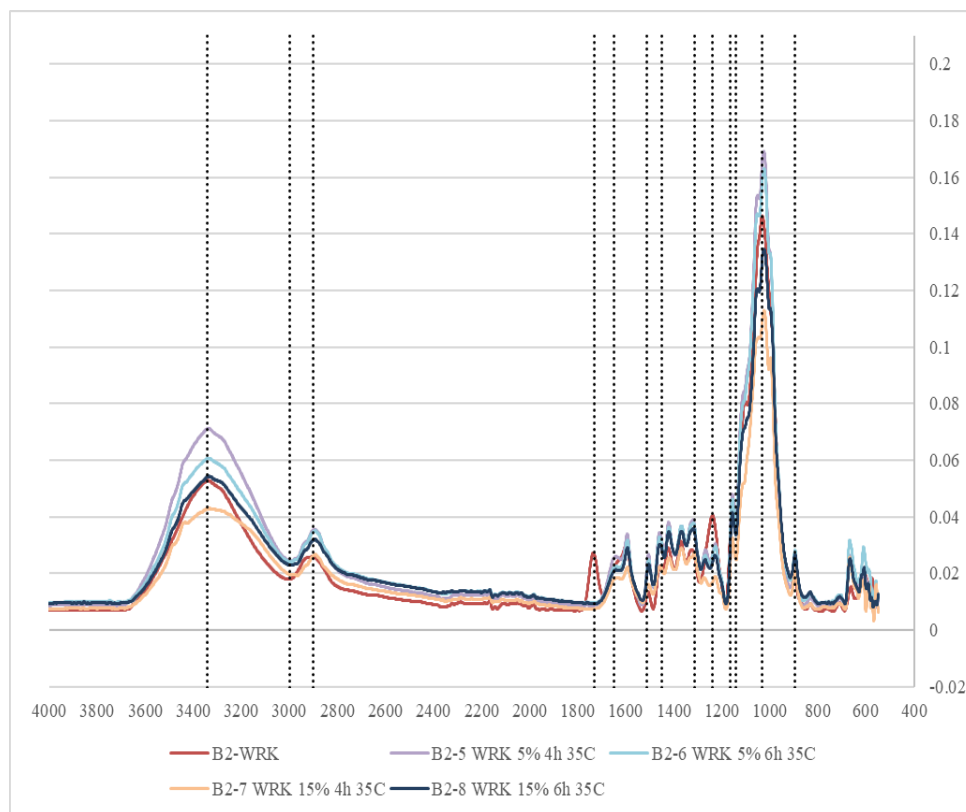


Figure 9: FTIR Spectrum for WR-Kenaf at 35 °C in terms of its absorbance

### *XRD Analysis*

The calculated crystallinity indices for the samples are shown in Fig. 10.

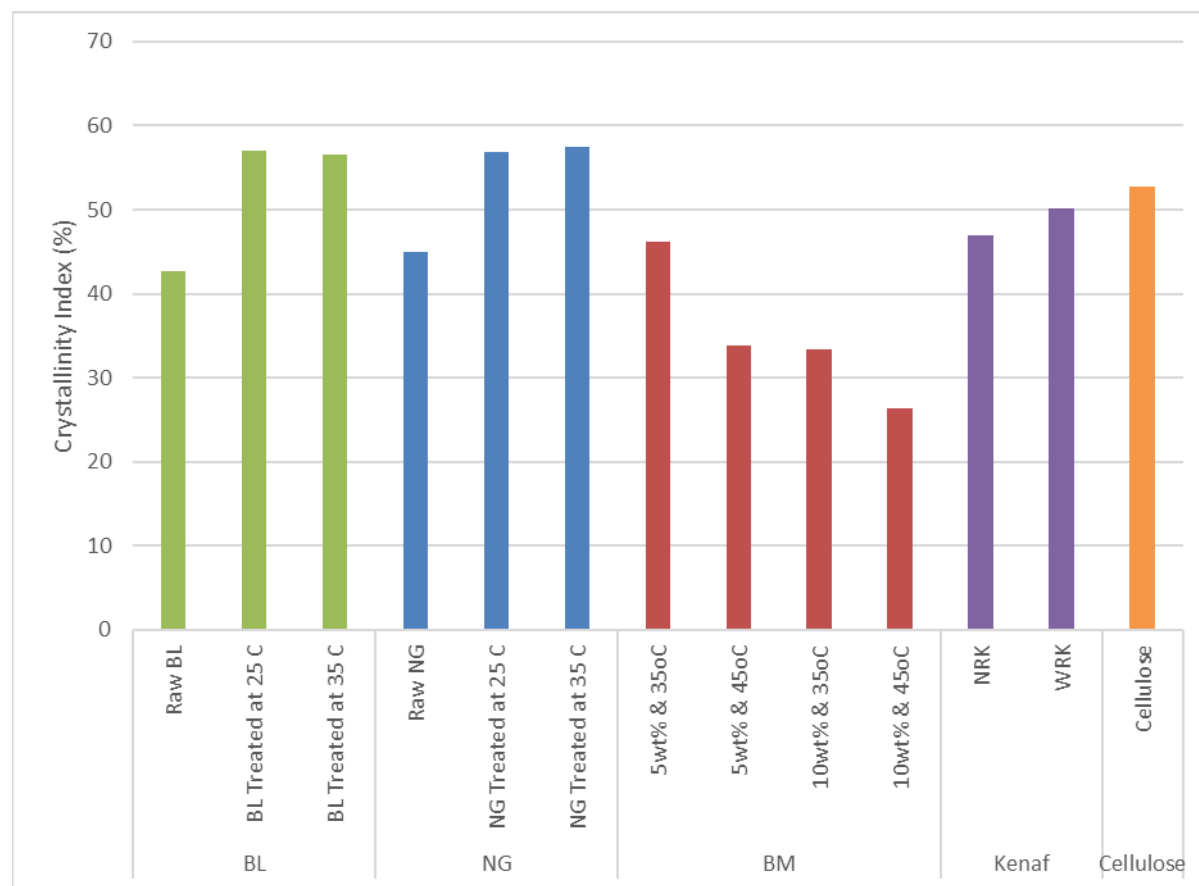
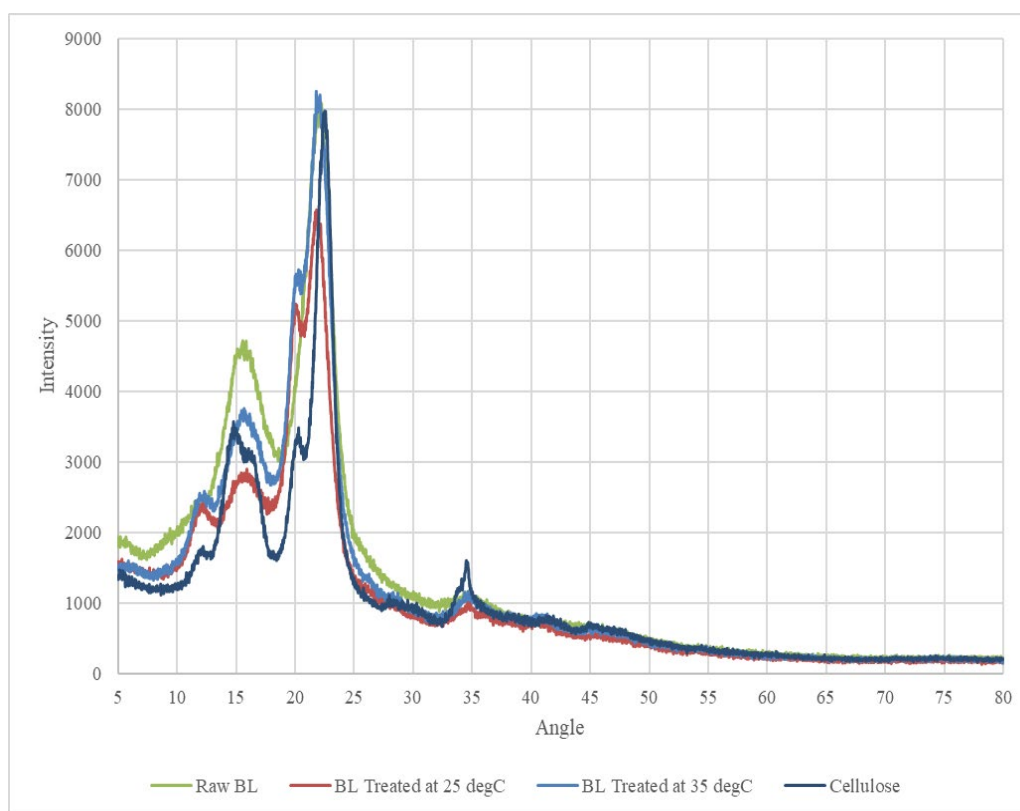


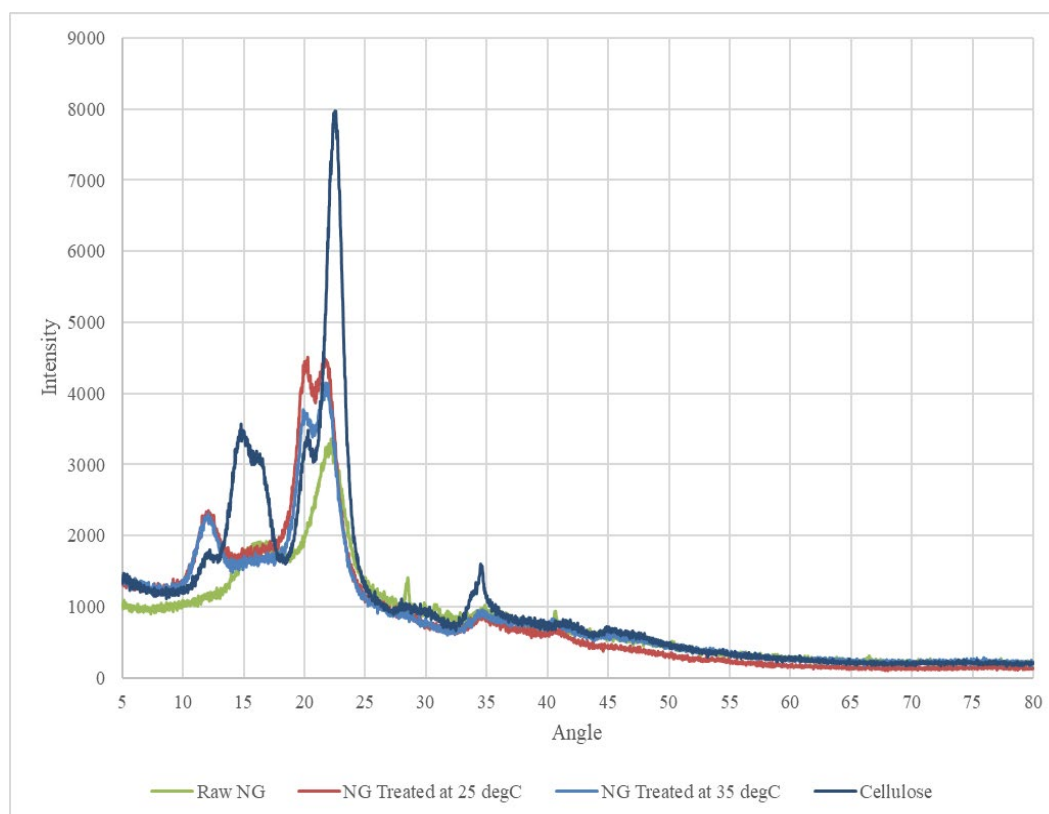
Figure 10: Crystallinity Index of Samples

### *Bamboo BL and Napier grass*

For BL, the XRD spectra is shown in Fig. 11. The crystallinity index (CrI) shows that the alkali treatment increases the CrI. When comparing between the samples treated with different temperature, the change is not significant with regards to CrI. Fig. 12 shows the XRD spectra for NG. Based on the crystallinity results for NG, the CrI seems to also increase with increase in alkali treatment temperature. The increasing in crystallinity index was due to the removal of lignin and hemicellulose during the chemical process as compared to the raw samples where lignin and hemicellulose were still present in the samples. The treated fibers appeared to be more crystalline than the untreated ones since the loss of their amorphous constituent happened after treatment [12].



*Figure 11: XRD Spectra for BL*



*Figure 12: XRD Spectra for NG*

### Bamboo BM

From Fig. 13, the cellulose crystallinity diffraction pattern for all 4 different conditions shows a similar trend. It is significant that cellulose sample treated with 10 wt.% of NaOH at 45 °C have the highest crystallinity index at 61% and 5 wt.% of NaOH at 35 °C have the lowest crystallinity at 52%. Besides, an untreated sample of bamboo is observed to have a crystallinity index of 45.57%. The crystallinity index of bamboo cellulose pulp (Bambusa Vulgaris) is reported to be 51.48% which was produced by the mechanical defibrillation process of 0.3% anthraquinone ( $C_{14}H_8O_2$ ) per unit solid mass. Thus, this crystallinity index obtained was within the range of bamboo cellulose fiber. The crystallinity index obtained also clearly reflects accordingly to the absorbance (%) in the FTIR analysis in Fig. 5. Therefore, the treated cellulose sample for 10 wt.% of NaOH at 45 °C is the closest to the analytical grade of pure cellulose which associates with the highest crystallinity index and absorbance (%). This result explains that the concentration of 10 wt.% of NaOH as well as the incubation temperature of 45 °C is preferred for a higher crystallinity index of cellulose from bamboo.

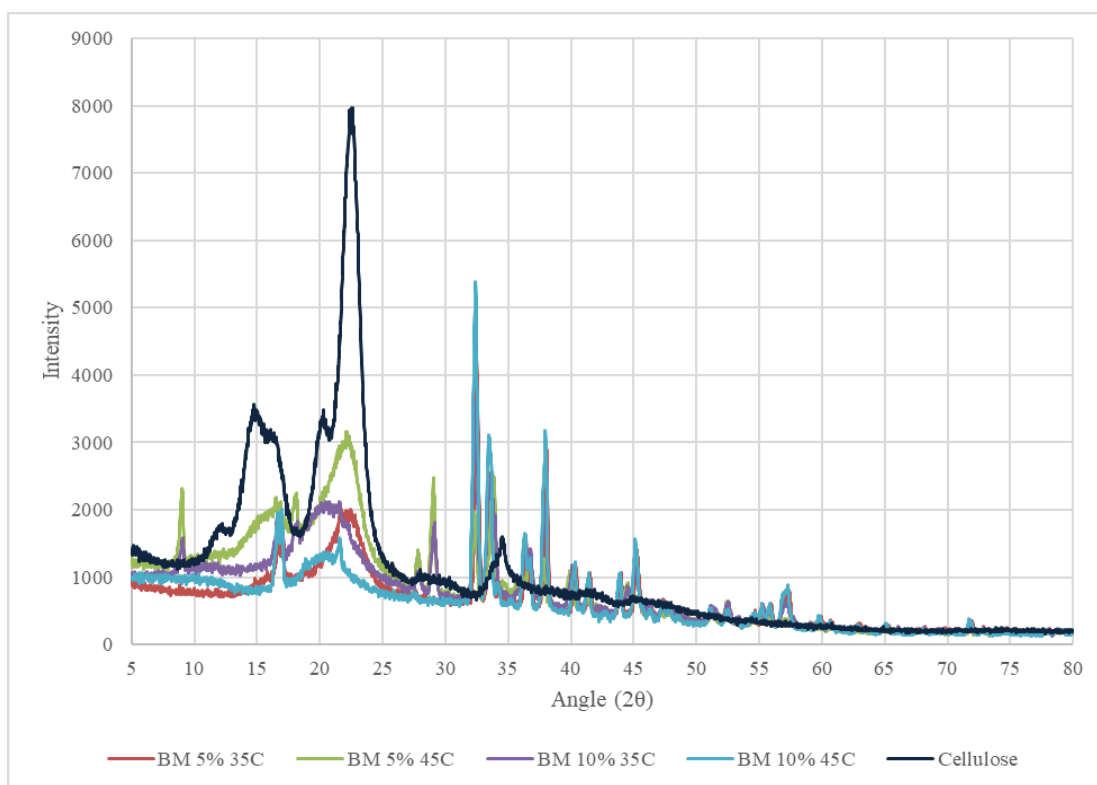
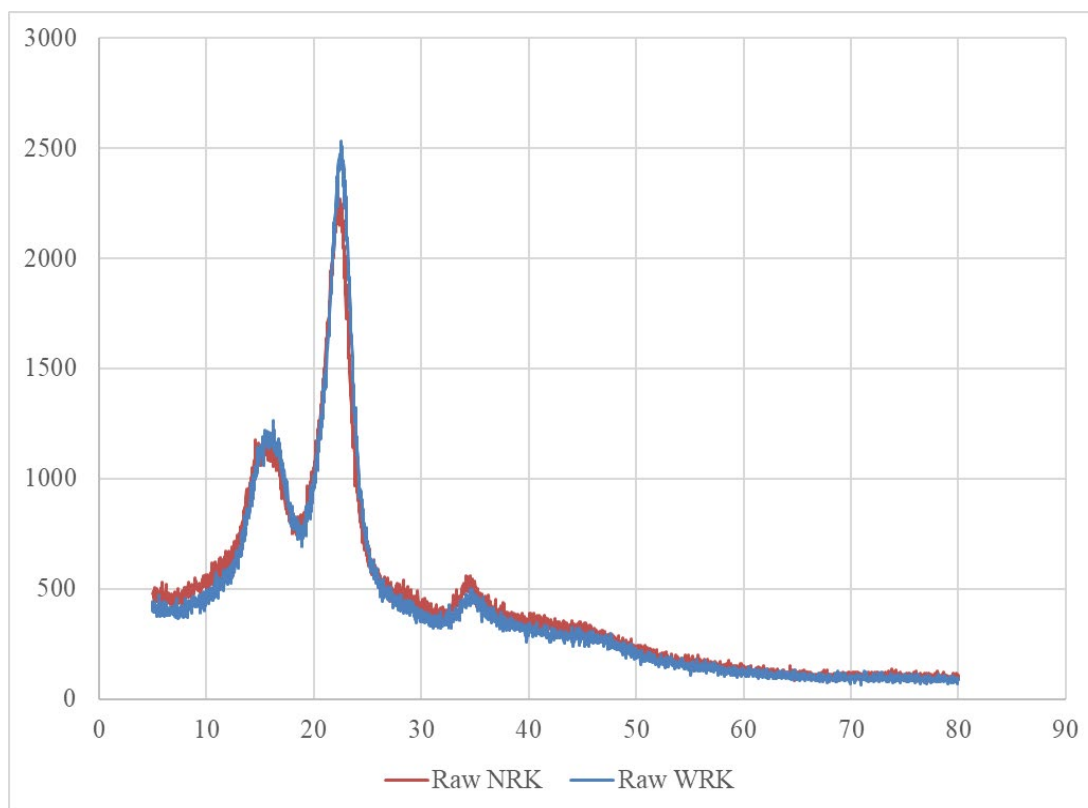


Figure 13: XRD Spectra for BM

### Kenaf

Fig. 14 shows the XRD spectra of alkali-treated NR-Kenaf (ATNR-Kenaf) fibers and alkali-treated WR-Kenaf (ATWR-Kenaf) fibers at constant parameters during the alkali treatment and bleaching process. A similar diffraction pattern can be seen for both the alkali-treated NR-Kenaf and alkali-treated WR-Kenaf. The diffraction for WR-Kenaf have a higher peak at 22.53° showing higher intensity as compared to a lower peak intensity of NR-Kenaf. The peak at 22.53° was presumed to be cellulose while the peak at 16.23° was presumed to be the presence of hemicellulose and lignin results in an amorphous phase. It can also be seen that the retting process does not affect the reformation of cellulose crystalline structure. The removal of amorphous components, pectin, hemicellulose, and some lignin were aided by the retting, alkali treatment, and bleaching process.

Crystallinity index has been calculated and presented into Fig. 10. As shown in the table, the CrI of ATNR-Kenaf and ATWR-Kenaf were 47.01% and 50.12% respectively. It shows that the treated WR-Kenaf have a higher CrI than the treated Kenaf at a difference of 3.11%. The water retting process removes the pectin allowing the alkali treatment and bleaching process to easily remove amorphous components, hemicellulose, and lignin. The treated Kenaf does not utilize water retting resulting in the alkali treatment and bleaching process to remove pectin, amorphous components, hemicellulose, and lignin by the process itself which results in lower CrI for non-water retting.



*Figure 14: XRD Spectra for Kenaf*

## Conclusion

The variation of parameter of cellulose extraction is to quantify and evaluate the extraction via alkali treatment. The feedstocks were varied, and different concentration, temperature and treatment time were applied to optimize the extraction factor and the extracts were analyzed for its properties. The moisture content before alkali treatment was checked so that it is well-below 5 % in order to not compromise the chemical process. The yield of cellulose decreases with increasing alkali concentration and temperature while increasing the time would improve the yield. Of all the feedstocks, cellulose was able to be extracted best from Kenaf. Based on the FTIR analysis, it is evident that extractive removal and delignification was successful in removing lignin, hemicellulose, and extractives. After analyzing the crystallinity indices of the samples, it was found that increasing the temperature and concentration also decrease the crystallinity of cellulose, but the changes of temperature are less significant towards the crystallinity. The extracts were assessed with analytical pure cellulose for comparison and much of the aspects of cellulose were exhibited in the samples which shows that the process done is effective, but more optimizations are needed. This would unlock the potential of utilizing and commercialize agricultural biomass feedstock for production of cellulose and also towards other component extraction.

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