

Production and Characterisation of Cellulose and Nano-Crystalline Cellulose from Kenaf Core Wood

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Natural fibers such as kenaf have been studied extensively as a reinforcing phase and received major attention recently due to their renewability, biodegradability, and high strength comparable to other synthetic fibers. In this study, nano-crystalline cellulose (NCC) was produced from kenaf core wood using the acid hydrolysis method. Kenaf core was alkali treated with a 4 wt% of sodium hydroxide solution and subsequently bleached using sodium chlorite in acidic buffer. The resulting white, bleached kenaf core was hydrolyzed in 64 wt% sulfuric acid (H₂SO₄) to obtain NCC. The resulting NCC suspension was characterized using X-ray diffraction (XRD), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), Fourier transform infrared (FTIR) analysis, and scanning transmission electron microscope (STEM). Hydrolysis with highly concentrated H₂SO₄ further increased the crystallinity of bleached kenaf core cellulose and reduced the dimension of cellulose to nano scale. FTIR results showed that with each subsequent treatment, hemicellulose and lignin were removed, while the chemical functionalities of cellulose remained after the acid hydrolysis treatment. XRD peaks shown by bleached kenaf core were characteristic of cellulose I, which was reaffirmed by the DSC results. The diameters of NCC obtained from kenaf core were found to be in the range of 8.5 to 25.5 nm with an average aspect ratio of 27.8.

Keywords: Kenaf core; Cellulose; Nano materials; Nano-crystalline cellulose

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INTRODUCTION

Kenaf (*Hibiscus cannabinus* L., Malvaceae), an herbaceous dicotyledonous plant, consists of outer bast which resembles softwood fibers and a woody core which resembles hardwood (Pande *et al.* 2000). Kenaf plant can grow very fast, reaching a height of more than 3 m in 3 to 4 months (Villar *et al.* 2009; Webber III and Bledsoe 2002). The annual yield of kenaf (whole stem) ranges between 12 and 30 tons ha⁻¹, which is three times the maximum yield of *Pinus radiata*, depending on cultivars, soil type, climate, *etc.* (Villar *et al.* 2009). Kenaf core comprises 60 to 65 wt% of the whole stem of kenaf plant (Pande *et al.* 2000; Villar *et al.* 2009). Kenaf plant is largely used for its bast fibers, which are superior in mechanical strength as compared to kenaf core.

In 2009, designation of the International Year of Natural Fibers highlighted the importance of natural fibers and their impact towards people. Natural fibers have a good mechanical strength comparable to synthetic fibers and most importantly, are renewable and sustainable. In recent years, a particular natural fiber derivative received major attention for its superior mechanical properties *i.e.* cellulosic nano-fibers (Dufresne 2010; Eichhorn *et al.* 2010). They are most notably used as a reinforcing phase in composites and can also be used in a wide range of applications, such as drug delivery excipient,

transparent paper, iridescent film, aerogels, *etc.* (Beck *et al.* 2010; Jackson *et al.* 2011; Nogi *et al.* 2009; Sehaqui *et al.* 2010).

In this study, kenaf core wood was used to produce nano-crystalline cellulose (NCC) using the acid hydrolysis method. Kenaf bast is known to be higher in length and mechanical strength and therefore more suited for high mechanical strength application such as reinforcement in composites (Ishak *et al.* 2010), cordage, making ropes, paper pulp (Villar *et al.* 2009), *etc.* Kenaf core wood was chosen because of its limited usage compared to bast fibers. On the other hand, NCC from kenaf core wood source has not been prepared and characterized yet. The produced NCC have been characterized using X-ray diffraction (XRD), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), Fourier transform infrared (FTIR) analysis, and scanning transmission electron microscopy (STEM).

EXPERIMENTAL

Materials

Kenaf core powder of V36 cultivar was obtained from Rice and Industrial Crops Research Center (MARDI), Malaysia. Sodium hydroxide (NaOH), sodium chlorite (NaClO₂), and sulfuric acid (H₂SO₄) were purchased from Merck, Acros, and JT Baker, respectively.

Alkali Treatment and Bleaching

Five-month-old kenaf core wood was ground and sieved to obtain kenaf core powder (80 micron). It was then rinsed once with distilled water and strained using a cloth filter to remove impurities such as sand. Alkali treatment was conducted in NaOH solution of 4 wt% at 80 °C for 3 times. Alkaline treated fibers were subjected to NaClO₂ bleaching of 1.7 wt% in acetic acid buffer at 80 °C for 4 times. The resulting fibers were strained and washed with distilled water and cloth filter until it reached neutrality. The kenaf core wood powder was then oven dried at 105 °C. Then it was deemed as bleached kenaf core.

Acid Hydrolysis

Bleached kenaf core wood was hand ground before acid hydrolysis. The ground product was hydrolyzed in pre-heated H₂SO₄ (64 wt%) (Beck-Candanedo *et al.* 2005). The high concentration of acid was removed through centrifugation at 10,000 rpm for 10 min and repeated until the solution was turbid. The resulting NCC suspension was dialyzed using cellulose membrane in deionized water until the suspension reached a pH of around 5. The NCC suspension was freeze-dried to obtain NCC powder.

Analysis

Chemical Analyses

Kenaf core powder, alkaline-treated kenaf core, and bleached kenaf core were analyzed for Klason lignin and holocellulose content using standard methods described in TAPPI T222 om-99 and Wise *et al.* (1946), respectively.

X-ray diffraction (XRD)

Kenaf core powder, alkaline treated kenaf core, bleached kenaf core, and NCC were analyzed using XRD (Bruker AXS D8 Advance), Cu_{Kα1} from 2θ = 5° to 40° with a step size of 0.0250°.

The crystallinity index (CrI) was calculated using Segal's method. Segal's method is an empirical method to quickly determine the relative crystallinity. Crystallinity index was calculated using the following equation.

$$\text{CrI (\%)} = [(I_{002} - I_{AM}) / I_{002}] \times 100 \% \quad (1)$$

Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA)

DSC and TGA (Mettler Toledo) analysis was conducted for bleached kenaf core and NCC. Samples were heated at a rate of 10 °C/min from room temperature to 600 °C under N₂ flow for DSC. For TGA, samples were heated at a rate of 10 °C/min from room temperature to 600 °C under N₂ gas to analyze its thermal stability.

Fourier transform infrared (FTIR) Analysis

FTIR (Perkin-Elmer Spectrum 400) analysis was conducted for kenaf core powder, alkaline treated kenaf core, bleached kenaf core, and NCC in the range of 4000 to 650 cm⁻¹.

Scanning transmission electron microscope (STEM) Analysis

The dimensions of nano-crystalline cellulose were evaluated using STEM, Hitachi SU8000 at 30 kV. A drop of the diluted suspension of NCC was dropped onto a lacey carbon coated copper grid. From the micrographs, the diameters of NCC were calculated with the aid of integrated computer software. At least 60 measurements were taken.

RESULTS AND DISCUSSION

Chemical Analysis

Table 1 shows the proximate chemical composition of kenaf core powder, alkaline treated kenaf core, and bleached kenaf core. The holocellulose content of the alkaline treated kenaf core was found to be higher than kenaf core powder due to the removal of hemicellulose after alkali treatment, whereas the holocellulose in kenaf core powder consists of cellulose and hemicellulose. After alkali treatment, a majority of lignin fraction in sample still remains, therefore the Klason lignin is expected to be higher than that of the kenaf core powder. After repeated bleaching, the Klason lignin in bleached kenaf core was found to be 0.13%, whereas the holocellulose portion was 99.10%. High lignin content in sample is known to impede the acid hydrolysis process (Kumar *et al.* 2009); therefore low lignin composition is desirable for the production of NCC using acid hydrolysis.

Table 1. Holocellulose and Lignin Contents of Kenaf Core Powder, Alkaline Treated Kenaf Core, and Bleached Kenaf Core

Sample	Holocellulose (%)	Klason lignin (%)
Kenaf core powder	80.26	23.58
Alkaline treated kenaf core	72.14	24.93
Bleached kenaf core	99.10	0.13

XRD

Figure 1 shows the XRD intensities of kenaf core wood powder, alkaline treated kenaf core, bleached kenaf core, and NCC. The peaks obtained from the XRD intensities were the characteristic peaks for cellulose I. The (002) peak of samples shifted slightly

towards higher angles with more intensive treatment, which is consistent with kenaf bast (Kargarzadeh *et al.* 2012) and other sources of cellulose. The crystalline peaks of cellulose are more profound with increasing treatment with the emergence of a doublet of (101) and (10 $\bar{1}$). Both bleached kenaf core and NCC conform to peaks normally shown by cellulose such as Avicel PH-101, a type of microcrystalline cellulose (Park *et al.* 2010).

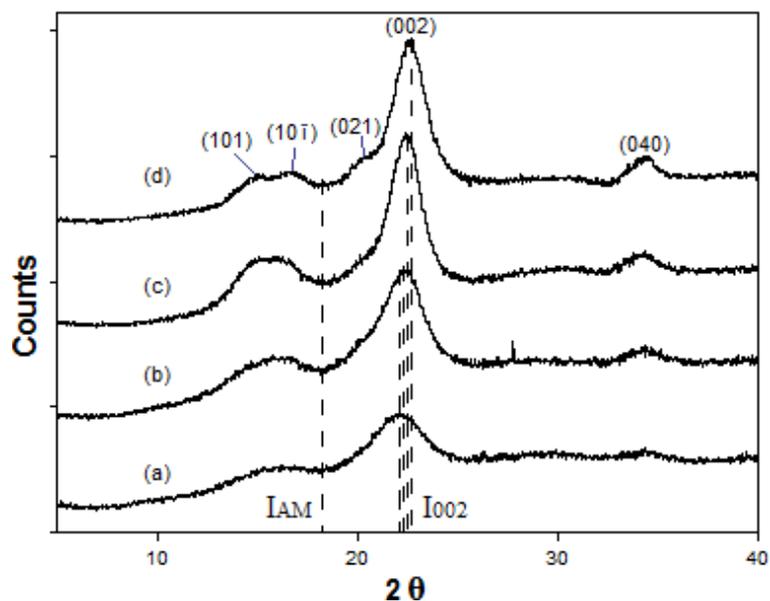


Fig. 1. XRD Intensities of (a) kenaf core wood powder, (b) alkaline treated kenaf core, (c) bleached kenaf core, (d) NCC

The crystallinity index (CrI) of kenaf core increased after alkali treatment due to the removal of amorphous hemicellulose. A combination of low NaOH concentration and relatively low temperature did not alter the cellulose structure, as shown by XRD results. Bleaching the alkaline-treated fibers further increased the CrI by removal of lignin. Further treatment using concentrated sulfuric acid increased the CrI from 48.1% for kenaf core powder, to 75.0% for NCC samples, which can be attributed to the removal of amorphous hemicellulose, lignin, and cellulose; the surface chains of remaining celluloses (NCC) may recrystallize causing an increment in CrI. On the other hand, aggregation of the products after treatment may also contribute to higher crystallinity (Leppänen *et al.* 2009). The NCC produced in this study was freeze-dried; hence, the effects of recrystallization and aggregation were reduced.

FTIR

Figure 2 shows the spectra for (a) kenaf core wood powder, (b) alkaline-treated kenaf core, (c) bleached kenaf core, and (d) NCC. The successive chemical treatments are expected to induce chemical changes.

The absorption peak at 1734 cm^{-1} is attributed to the absorption of acetyl and ester groups of hemicellulose in kenaf (Jonoobi *et al.* 2010) and rice husk (Johar *et al.* 2012). This peak disappears after alkali treatment. This shows that alkali treatment is capable of removing hemicellulose, which is amorphous (Gibson 2012). The absorption peak at 1593 cm^{-1} is attributed to the aromatic symmetric stretching, while the absorption peak at 1503 cm^{-1} is attributed to aromatic asymmetric stretching from lignin (Atalla and Agarwal 2010; Yan *et al.* 2009). These peaks disappeared after bleaching was conducted, thus confirming the removal of lignin. Both peaks were not observed for bleached kenaf core and NCC. The absence of designated hemicellulose and lignin peaks confirms the

earlier XRD results, which showed an increment of CrI due to amorphous nature of hemicellulose and lignin (Gibson 2012). Meanwhile, the absorption peaks at 2900 cm^{-1} , 1640 cm^{-1} , and 1160 cm^{-1} are attributed to aliphatic alkyl, adsorbed water on fibers, and ether linkages from pyranose ring (Morán *et al.* 2008; Yang *et al.* 2007). These peaks can be observed throughout the final product, NCC, and are the characteristic peaks for cellulose. No significant differences can be observed for bleached kenaf core and NCC, which indicates that the NCC structure remains unchanged after acid hydrolysis treatment.

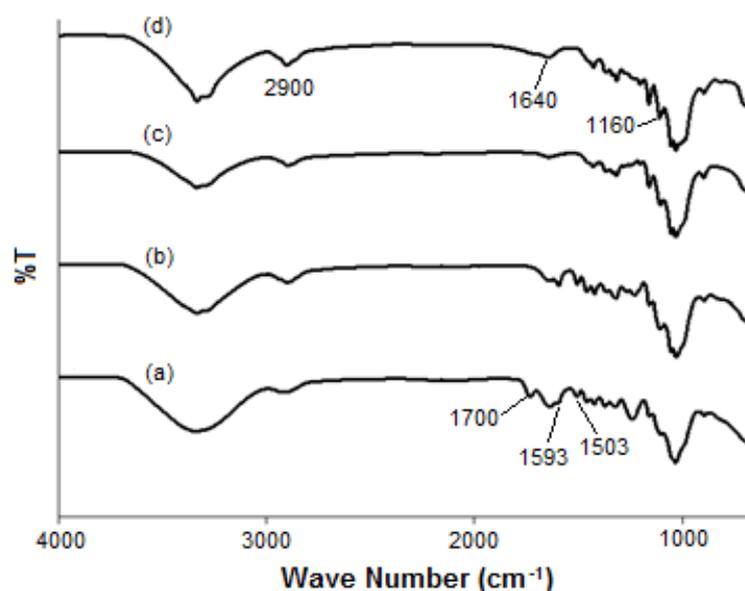


Fig. 2. FTIR spectra for spectra for (a) kenaf core wood powder, (b) alkaline treated kenaf core, (c) bleached kenaf core, (d) NCC

DSC and TG Analysis

The DSC thermograms of bleached kenaf core (cellulose), sulfated NCC, and unsulfated NCC are shown in Fig. 3. The NCC (sulfated NCC) obtained after dialysis was neutralized with NaOH until neutrality to obtain unsulfated NCC so as to increase its thermal stability (Kargarzadeh *et al.* 2012).

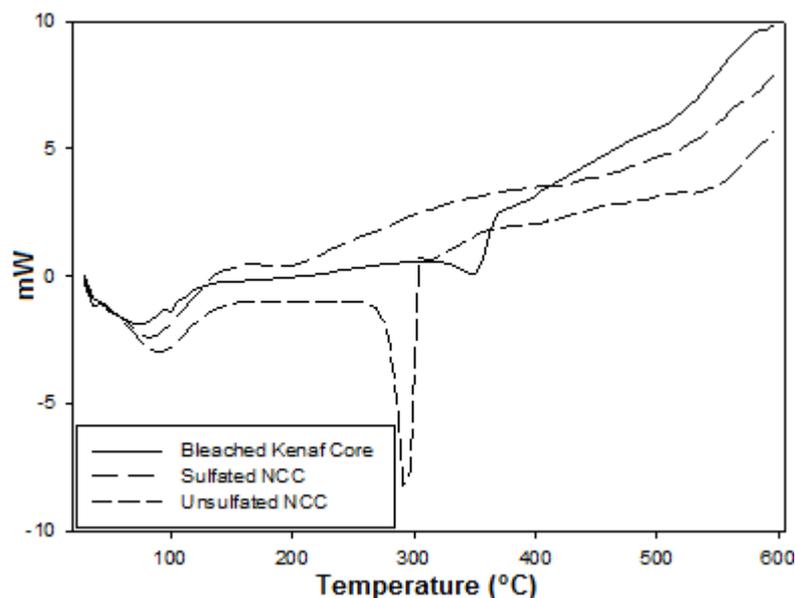


Fig. 3. DSC thermograms for the bleached kenaf core and NCC (sulfated and unsulfated NCC)

The first endothermic peak, which can be attributed to the loss of water, was observed in the range of 32 °C to around 130 °C for bleached kenaf core, whereas for the sulfated and unsulfated NCC sample, the first endothermic peak was observed at the range of 32 °C to around 140 °C. The area of this first endothermic peak for sulfated NCC and unsulfated NCC is larger than that of the kenaf core wood powder. This may be due to the smaller size and larger surface area, leading to greater water absorption. A second endothermic peak can be observed at around 350 °C for bleached kenaf core. The same result was obtained for commercial cellulose by Sigma-Aldrich from a previous study (Yang *et al.* 2007). A small endothermic peak was observed at 200 °C for sulfated NCC, but no significant endothermic peaks were observed at 350 °C for the sulfated NCC sample. This may be due to the presence of remaining sulfated groups on the surface of sulfated NCC that act as a flame retardant which lowered its degradation temperature (Kargarzadeh *et al.* 2012; Roman and Winter 2004). By neutralizing the sulfated NCC associated with low degradation temperature, the degradation temperature increased to 298 °C for unsulfated NCC. Low degradation temperature of sulfated NCC was not observed, while the degradation profile exhibited is the same with bleached kenaf core, albeit with a lower degradation temperature.

TG and DTG thermograms for the bleached kenaf core and NCC samples are shown in Fig. 4. The initial weight loss for both bleached kenaf core and NCC at around 100 °C can be attributed to vaporization of water. For the bleached kenaf core, at higher temperature, the weight loss is slow and then varies sharply. The maximum weight loss occurred at 347 °C; at 356 °C only 23.8% of sample weight still remained, which shows a similar tendency with the earlier DSC results. This value also corresponds well with commercial cellulose by Sigma-Aldrich (Yang *et al.* 2007) and Avicel (Rhim *et al.* 2010). For temperatures higher than 400 °C, a residue of 17.6 % still remained. On the other hand, for TG and DTG thermograms of the sulfated NCC, the maximum weight loss happened at a lower temperature, *i.e.*, 180 °C, compared to the bleached kenaf core. Around 12.9 wt.% of solid residue remained at 600 °C for the sulfated NCC, whereas for the bleached kenaf core, only 0.1 wt.% was left. This may be attributed to the remaining unwashed sulfated groups present on the sulfated NCC, which lowered its degradation temperature and served as flame retardants (Kargarzadeh *et al.* 2012; Roman and Winter 2004). The remaining sulfated NCC sample decomposed slowly with no significant DTG peak until the end. The absence of sulfate on unsulfated NCC increases its degradation temperature at maximum weight loss from 180 °C to 305 °C. The low degradation temperature (<200 °C), characteristic of NCC produced from sulfuric acid was not found if neutralization is employed.

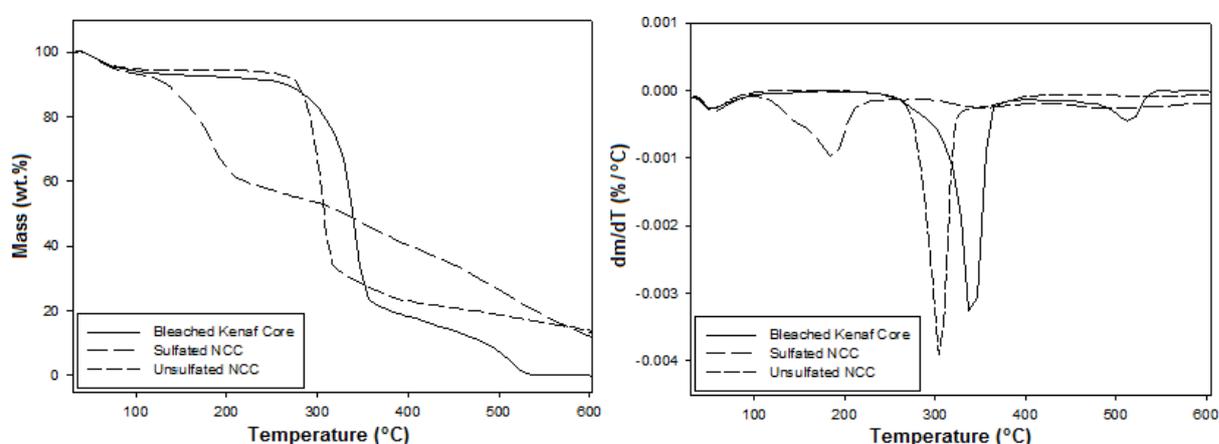


Fig. 4. (a) TG curves and (b) DTG curves for bleached kenaf core, sulfated NCC, and unsulfated NCC

STEM

Figure 5 shows the morphology of NCC observed by STEM. Panel (a) shows a single particle of NCC and (b) and (c) show agglomerated NCCs. Hydrolysis with high acid concentration can produce NCC (Bondeson *et al.* 2006; Dong *et al.* 1998). After hydrolysis, bleached kenaf core gave long strands of product. STEM images clearly show that the diameter of the acid hydrolyzed bleached kenaf core is in nano scale (< 100 nm).

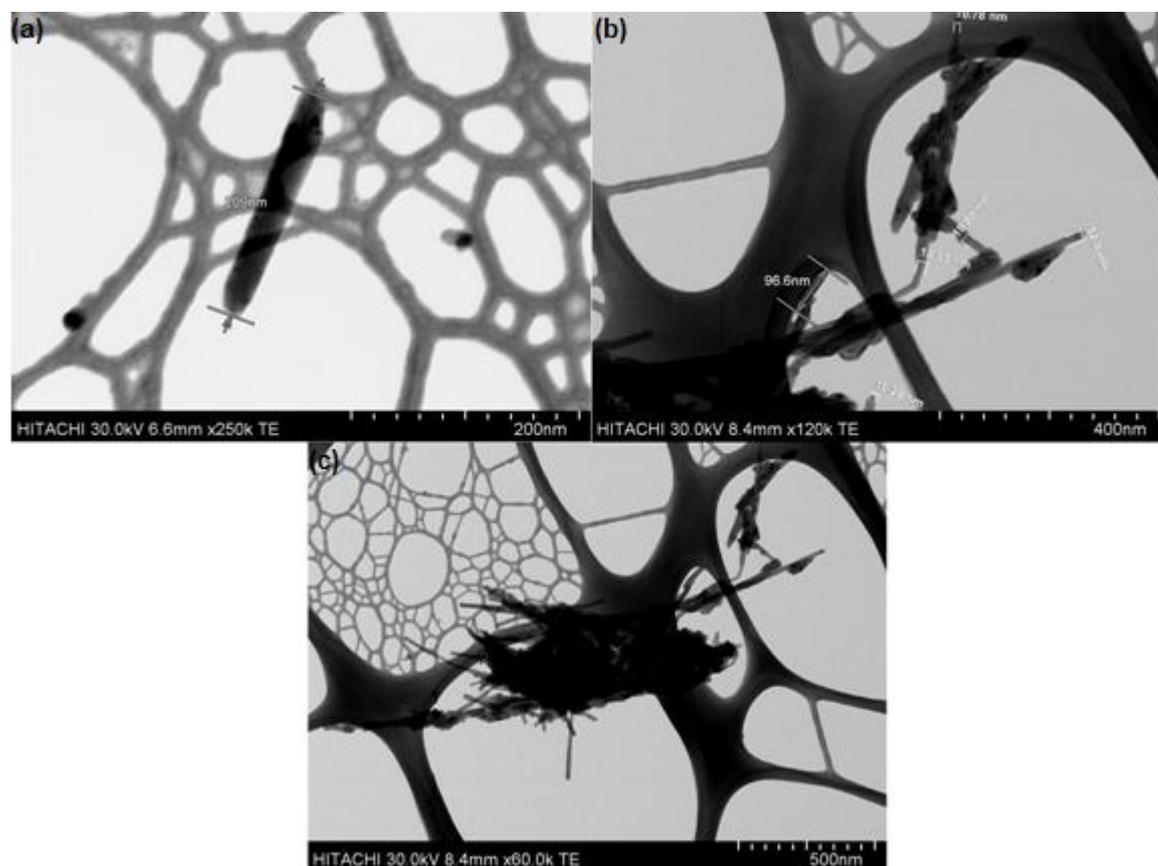


Fig. 5. (a) Single NCC on lacey copper grid, (b) Agglomerated NCCs, and (c) Another agglomerated NCCs showing a long feature

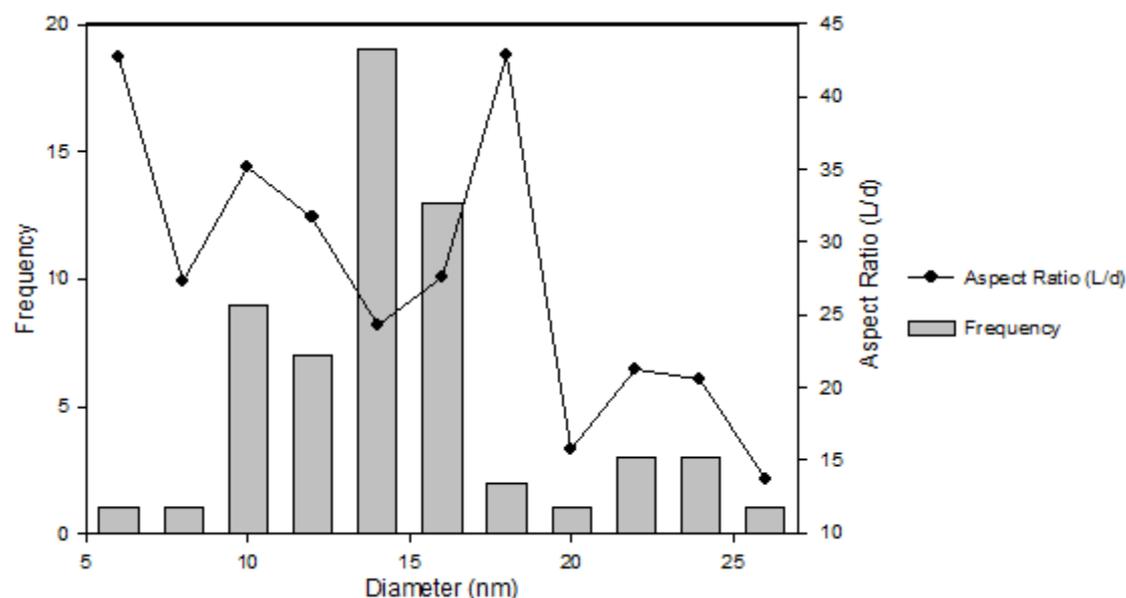


Fig. 6. Distribution of kenaf core NCC diameters and its aspect ratio obtained from STEM images

The STEM images showed rod-like cellulose, unlike the whisker-like cellulose (Eichhorn *et al.* 2010) normally shown by transmission electron microscopy (TEM). Agglomerates can be found due to sample preparation by water evaporation, inducing the formation of agglomerates. Sixty measurements of NCC diameter and length were performed. The mean diameter of NCC sample was 13.9 nm in the range of 5.4 to 25.9 nm with a standard deviation of 4.2. The distribution of diameter and aspect ratio of the NCC is plotted in Fig. 6. One noticeable quality of the NCC produced from kenaf core in this study is its high aspect ratio. Table 2 shows the aspect ratio of some NCC and its method of production. The average aspect ratio of kenaf core NCC was 27.8, which is much higher than that of kenaf bast.

Table 2. Aspect Ratio of NCC from Different Sources and its Processing Condition from Hydrolysis of Sulfuric Acid

Sample	Aspect Ratio (L/d)	Concentration of acid	Temperature	Time (minute)	References
Tunicin	50 - 200	55 wt.%	60	20	(Anglès and Dufresne 2000)
Wheat straw	45	65 wt.%	25	60	(Helbert <i>et al.</i> 199
Ramie	28	65 wt.%	55	30	(Alloin <i>et al.</i> 2011)
Black spruce	23.3	64 wt.%	45	45	(Beck-Candanedo <i>et al.</i> 2005)
Rice husk	10 - 15	10 M	50	40	(Johar <i>et al.</i> 2012)
Bagasse	13	65 wt.%	45	45	(Bras <i>et al.</i> 2010)
Cotton linter	11 - 12	65 wt.%	45	45	(Roohani <i>et al.</i> 2008)
Cotton	10.7	6.5 M	45	75	(Martins <i>et al.</i> 201
Kenaf bast	13.2	65 wt.%	45	40	(Kargarzadeh <i>et a</i> 2012)
Kenaf core	27.8	64 wt.%	45	45	(This study)

CONCLUSIONS

1. NCCs were successfully produced from a kenaf core source and were found to have an average diameter of 13.9 nm with high aspect ratio of 27.8 and CrI of 75.0 %.
2. Mild alkali treatment and subsequent sodium chlorite bleaching produces bleached kenaf core wood cellulose, which is comparable to commercially available cellulose from XRD and thermal analysis.
3. The thermal stability of sulfated NCC is lower than bleached kenaf core due to remaining sulfate from sulfuric acid hydrolysis; however, its thermal stability can be increased by neutralizing it.
4. Acid hydrolysis yield was determined to be *ca.* 40% while the overall yield of NCC production from kenaf core wood was found to be 15.7%.

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