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Extraction and Characterization of Nanocrystalline Cellulose from Sugar Palm Fibres (Arenga Pinnata)

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Abstract

In this work, cellulose was isolated from sugar palm fibre (Arenga pinnata) and converted into sugar palm fibre nanocrystalline cellulose (NCCs) in the hamlet of Medan Sinembah, Indonesia. Alkalization and bleaching were the stages of the therapy process. The cellulose removed with 30 weight percent concentrated sulfuric acid was then used to isolate NCC. Using Fourier transform infrared spectroscopy (FT-IR), the chemical make-up of sugar palm fibre and NCCs was identified. On the basis of structural analysis, X-ray diffraction (XRD) was used. Scanning electron microscopy (SEM) was used for morphological examination, while transmission electron microscopy (TEM) was used to look into NCCs. The outcomes demonstrated that the extracted cellulose underwent bleaching and alkalization processes, respectively, to eliminate lignin and hemicellulose. The diameters of the sugar palm fibre and NCCs were determined 69.025 µm to 6.07±0.14 nm and, respectively.

Keywords: Alkalization, Bleaching, Cellulose, Nanocrystalline Cellulose Sugar Palm Fibre

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INTRODUCTION

In the field of materials science, the separation of nanocrystalline cellulose (NCC) from plant fibres has garnered a lot of interest due to its intriguing inherent characteristics, including its nano-dimensions and large surface area (Islam et al., 2013). In general, "nanocrystalline cellulose" refers to cellulose separation or extraction materials with the extraordinary property of nanoscale structural dimensions. Cellulose is a product of biosynthesis from bacteria and plants. Two essential components that make up the natural fibre structure of natural fibres are lignin and hemicellulose. The branched polysaccharide polymer hemicellulose is made up of different types of sugars, including arabinose, mannose, xylose, glucose, and galactose, whereas the phenolic polymer lignin has strong cross-linking with phenolics. But both lignin and hemicellulose are amorphous polymers. Strong acid hydrolysis is a well-known technique for removing amorphous regions and isolating NCC from natural fibres. separating natural fibres from NCC, etc. When carried out under regulated conditions, acid hydrolysis enables the removal of the amorphous portion of cellulose fibres (Ilyas et al., 2017). NCC isolation serves as a reinforcement in the field of nanocomposites, which has drawn a lot of interest since Favier's study in this area has gotten a lot of attention since Favier et al. (1995) conducted their initial research on it. Research on the creation, or characteristics of naturally occurring nanocrystalline cellulose fibres from arenas is nonetheless hard to come by Favier et al. (1995).

In the tropics, the palm tree, also called Arenga Pinnata, is a well-liked multifunctional tree. This tree is a member of the Palmae family, which comprises roughly 181 genera and

2600 species that are currently recognized (Ishak et al., 2013). The utilization of the use of palm fibre has increased, particularly for diverse technical applications. For instance, it is used for cables that run underground and underwater, to replace geo-textile fibreglass reinforcement used to stabilize soil during road building, and to add reinforcement to polymer matrix composites used in material engineering materials. Numerous studies have demonstrated the huge potential for sugar palm fibres to be employed in a variety of polymer composite applications (Bachtiar et al., 2008). Studying the extraction and characterization of NCC from sugar palm fibre is desirable due to the limits of existing research on the subject.

In this study, sugar palm fibre came from medan sinembah village was chemically processed to extract cellulose and NCCs. X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FT-IR) were used to analyze the chemical composition of the fibres in order to determine the impact of various chemical treatments on them. Additionally, using a scanning electron microscope (SEM) and a transmission electron microscope (TEM) determine the morphological shape of sugar palm fibre.

METHOD

There are two basic ways to remove cellulose fibres from sugar palm fibres: alkalization and bleaching. 30 g of sugar palm fibre are first broken into little pieces and cleaned with tap water to eliminate dust and foreign objects before the alkalization process can begin. The clean fibres were then submerged in 650 mL of an alkalization and bleaching solution in a 1000 mL beaker. The temperature was set at 70°C in a 2000 mL beaker that contained 1000 mL of distilled water, 160 g of sodium hydroxide, was stirred at 700 rpm and for four hours. The degree of alkalization is indicated by the softening of the fibres. The sugar palm fibre sample was brought to pH 7 neutrality.

The bleaching process then began with the neutralized sugar palm fibres, which were swirled once again in a 2000 ml beaker containing 2.5% NaOCl and 1000 ml of distilled water for 30 minutes at 700 rpm and 70°C. This procedure was carried out twice. The degree of bleaching is indicated by the color turning white. The sample was neutralized to pH 7 after filtering. This procedure produced cellulose devoid of lignin and hemicellulose.

The mixture was then agitated once more with H_2SO_4 30% w/b in a ratio of 1:10 of the cellulose amount while rotating at a speed of 450 RPM for one hour at 70°C. The pH meter shows that the residue is acid-free. After that, the cellulose, also known as sugar palm cellulose fibre, was dried in an oven at 30°C. Finally, the cellulose known as sugar palm fibre NNCs was dried in an oven for 24 hours at 100°C.

Fourier transform infrared (FT-IR) Characterization

To identify potential alterations in functional groups found in the sugar palm fibre, Fourier transform infrared (FT-IR) spectroscopy is performed. potential modifications to the functional groups found in fibre at the extraction step. An IR spectrometer (Nicolet 6700 AEM) was used to obtain the material's spectra. The sample's FT-IR spectra (10x10x3 mm) was recorded between 400 and 500 cm-1. The samples of sugar palm fibre and sugar palm NCCs were combined with KBr, and the resulting mixture was then pressed into a thin clear film for analysis.

X-ray diffraction (XRD) Characterization

Using a Rigaku D/max 2500 X-ray powder diffractometer (Rigaku, Tokyo, Japan) fitted with CuK radiation (= 0.1541 nm) over a range of 2Ө 10° to 70°, the X-ray diffraction patterns of sugar palm fiber and sugar palm NCCs were studied. The empirical method was then used to determine the sample crystallinity index.

$$Xc = \frac{I002 - Iam}{I002} x\ 100 \quad (1)$$

Where I_{002} and I_{am} are the peaks of the intensities of crystalline and amorphous materials, respectively.

Transmission Electron Microscope (TEM) Characterization

The creation of nanostructure images of the sugar palm NCCs was accomplished using a transmission electron microscope (TEM) device. For creating TEM pictures, a Philips Technai 20 machine with a 200 kV accelerating voltage and a standard sample holder standard was employed. Sugar palm NCCs were first placed on a copper grid that had been coated with carbon, and they were then left at room temperature. improved TEM nanostructure picture for better resolution.

RESULTS AND DISCUSSION

The sugar palm NCCs fabrication process goes through a chemical treatment process, including alkalisation, bleaching and acid hydrolysis.

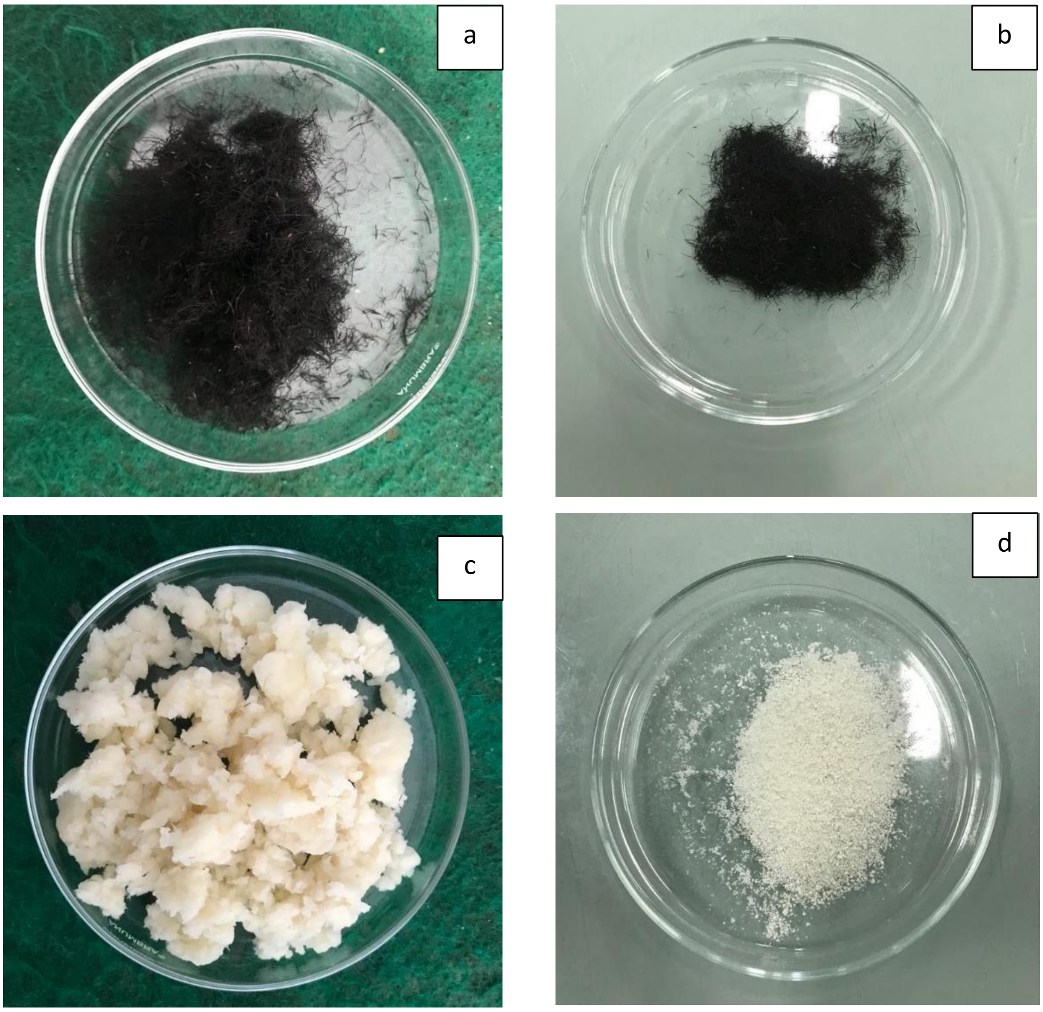

Figure 1. (a) Sugar palm fibre (b) Alkalisation result (c) Sugar palm fibre cellulose (d) sugar palm fibre CNNs

Sugar palm fibre CNNs are made using a chemical technique. In comparison to the mechanical procedure, the chemical process results in sugar palm fibre CNNs that are smaller and more consistent in size. The average diameter of palm sugar CNNs size in this study was determined to be 6.077±0.144 nm. While this was going on, IPB researchers undertook research to make CNNs mechanically from a variety of natural fibres, obtaining sizes of 25.4

nm for TKKS, 161 nm for Bamboo Ampel, and 89.6 for kenaf fibre. Chemical CNNs production does have several drawbacks, especially the longer processing time and the usage of dangerous chemicals in the form of strong acids (Aini, 2016; Bahmid, 2014; Aminah, 2017).

Fourier transform infrared (FT-IR) Characterization

Testing was done using FTIR to determine the amount of cellulose present based on how the chemical bonds that make up cellulose appeared. The cellulose molecule is held together by four different chemical bonds: -CH2 at (1000-650 cm^{-1}), C-O at (1300-1000 cm^{-1}, O-H at (3650-3200 cm-^{1}), and C-H at (3000-2850 cm^{-1}) (Astuti, 2015).

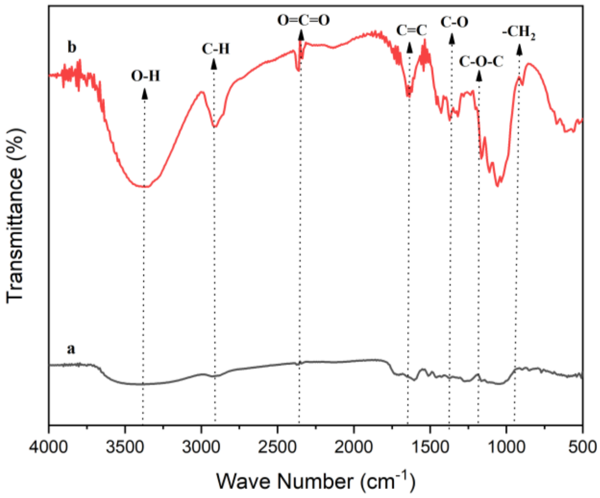

Figure 2. FTIR spectra of sample (a) Sugar palm fibre (b) Sugar palm fibre CNNs

Figure 2 shows the FTIR spectra of sugar palm fibre, and sugar palm fibre CNNs. In general, there is no significant difference between sugar palm fibre and Sugar palm fibre CNNs. The chemical groups of sugar palm fibre and sugar palm fibre CNNs with absorption peaks are presented in table 1.

Table 1. Chemical bond analysis results of FTIR test

No.	**Chemical Bonds**	**Absorption Interval (cm^{1})**	**Palm (cm^{-1})**	**Sugar palm fiber CNNs (cm^{-1})**
1	$-CH_2$*	1000-650	848.68	849
2	C-O*	1300-1000	1056	1373.32
3	C-O-C**	1085-1150	1165	11165
4	C=C**	1680-1600 1600-1475	1512.19	1620.21
5	O=C=O**	2349	2345.44	-
6	C-H*	3000-2850	2931	2900.94
7	O-H*	3650-3200	3387	3371.57

*chemical groups that make up cellulose

**chemical groups that make up non-cellulose components

X-ray diffraction (XRD) Characterization

Tests on sugar palm fibre and sugar palm fibre CNNs show the material is semicrystalline. This can be seen from the test results in Figure 3. To measure the crystallinity of each sample, it can be calculated using equation 1.

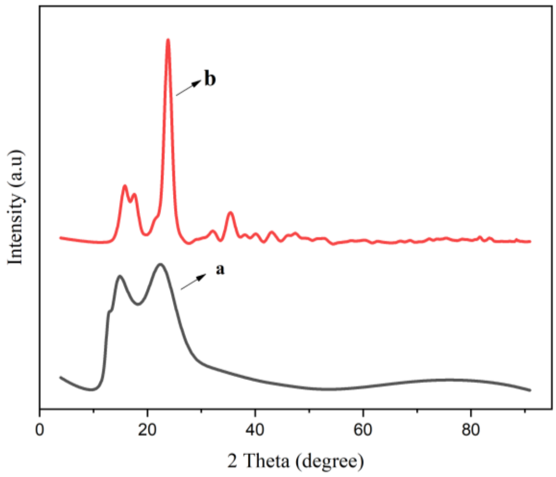

Figure 3. XRD diffraction pattern of sample (a) sugar palm fibre (b) Sugar palm fibre CNNs

Table 2. Crystallinity of each sample

Sample	Amorf Area		Area (002)		I_{cr} (%)
	2θ(º)	I_{am}	2θ (º)	I_{002}	
Sugar Palm fibre	15.1	552.16	22.67	616.13	10.38
Sugar Palm fibre CNNs	15.77	981.72	23.65	1742.67	43,66

The crystallinity value grew as the sugar palm fibre sample chemical treatments were applied to sugar palm fibre CNNs. The decrease in lignin and hemicellulose content in each treatment is indicated by the increase in crystallinity value.

Transmission Electron Microscope (TEM) Characterization

The SEM test was used to observe the morphology of sugar palm fibre against the chemical treatment. SEM test results can be seen in Figure 4.

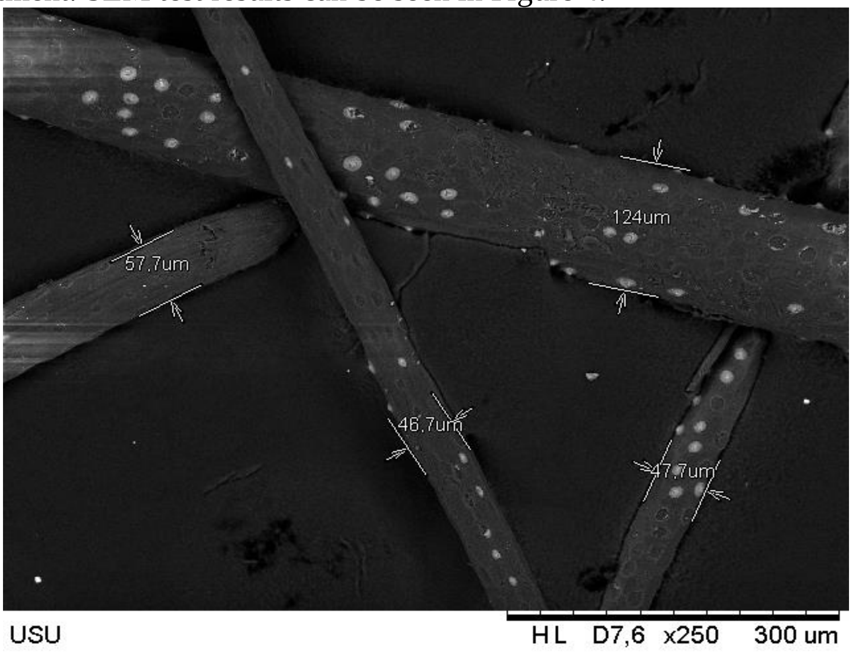

Figure 4. Observation Test Results using SEM for sugar Palm Fibre

According to Figure 4, the longitudinal cross section of the palm fibre has a rough surface topography and pore-like bitnics known as tylosis, which serve to hide cell wall flaws. The findings of the SEM test also show that the diameter of long fibre is approximately 69.025 m (Ilyas et al., 2018).

TEM was used to observe the sugar palm cellulose that had been acid hydrolysed using 30% H_2SO_4 solution whether it had become Sugar palm fibre CNNs. Sugar palm fibre CNNs is observed using TEM, because it can observe in size in the order of nanometers. The TEM test results are shown in Figure 5.

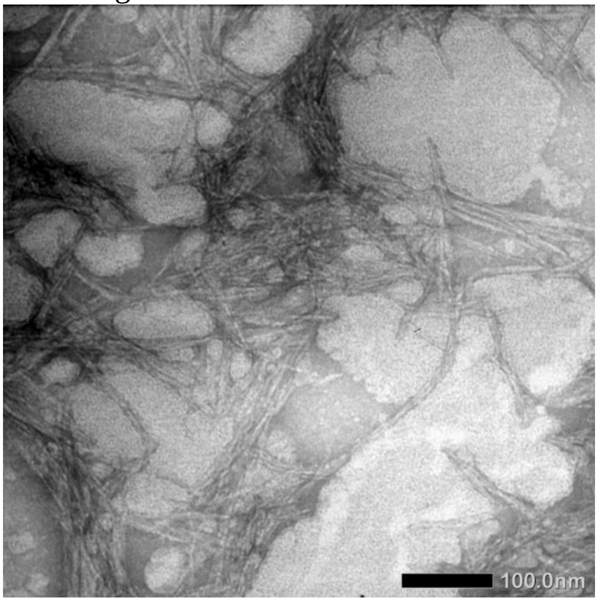

Figure 5. TEM observation test results for sugar palm fibre CNNs

Figure 5 demonstrates how the acid hydrolysis procedure can break down sugar palm fibre CNNs. Because powerful acid is used in the acid hydrolysis process, this is a possibility. By breaking down the fibre cellulose microfibrils into CNN, the acid hydrolysis procedure makes it possible to remove the amorphous areas of the cellulose fibre. By using Image-J and OriginPro 2018 software, the diameter size distribution of CNN is shown in Figure 6.

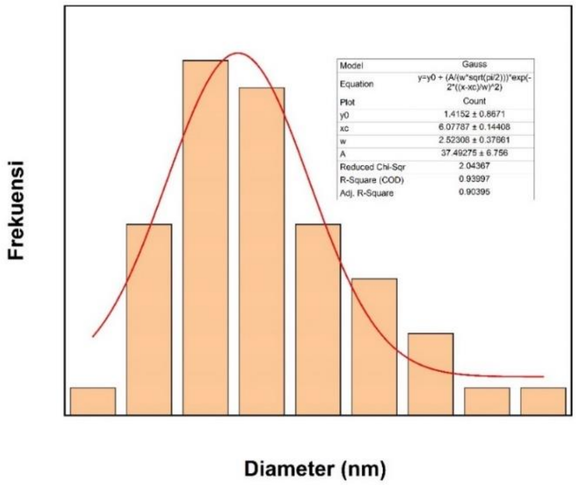

Figure 6. Diameter distribution of sugar palm CNNs

Figure 6 shows the size distribution of sugar palm CNN with the diameter obtained with an average of: 6.07±0.14 nm

CONCLUSION

Using a delignification process (NaClO), sugar palm fiber NCCs were successfully removed and separated from sugar palm fiber employing acid hydrolysis (H_2SO_4), bleaching (NaOCl), and alkalization treatment (NaOH). The FTIR data further demonstrated that lignin and hemicellulose could be efficiently removed using the chemical processes of alkalinization (NaOH) and bleaching (NaOCl). Higher crystallinity was revealed by XRD measurements for sugar palm fiber NCCs hydrolyzed in chemical acids. Overall, the sugar palm fiber's crystallinity increased from 10.38% to 43.66% in NCCs. The creation of sugar palm fiber NCCs can start with sugar palm fiber, according to SEM micrographs, which demonstrated sufficient random dispersion in the solid. The size of the sugar palm fiber NCCs produced was 6.07±0.14 nm, according to TEM images. According to the research, sugar palm fiber can serve as a raw material for the production of nanocrystalline cellulose (NCC).

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