# Utilization of Peroxide Bleached Sugar Palm (Arenga pinnata) Fibre Waste into Cellulose Nano Crystal

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ABSTRACT

Sugar palm (Arenga pinnata) fibre (SPF) waste is a side product of sugar palm starch production and needs to be processed to avoid environmental pollution. Since the SPF has high cellulose content, it can be beneficial if it is valorized into high-value products such as cellulose nanocrystal (CNC). The CNC production from SPF was initiated by cellulose production by using an environmentally friendly peroxide bleaching as elementary chlorine free bleaching method. The CNC production was conducted via sulfuric acid hydrolysis at a temperature of 40°C, solid/liquid ratio of 1:10, and hydrolysis time of 45, 60, 75, and 90 minutes. The same functional groups were observed in all CNC samples, including the appearance of the ester sulfate group. The decrease in yield and crystallinity index (CrI) as the hydrolysis time was observed. These phenomena were caused by the degradation of the crystalline structure of cellulose and the formation of the ester sulfate group. The measurement of CNC diameter size was carried out by using the scanning electron microscopy (SEM) technique. The CNC diameter was below 100 nm which indicated the nanoparticle formation was observed at CNC produced at hydrolysis times of 75 and 90 minutes. In conclusion, CNC production was successfully produced from peroxide bleached SPF which is more environmentally friendly than the conventional method using chlorite bleached cellulose. Furthermore, it is needed to optimize the production of SPF CNC in further research.

## 1. INTRODUCTION

Sugar palm (*Arenga pinnata*) is a multipurpose plant whose products mainly consist of fruit, sugar, and starch (Lempang, 2012). Various types of waste are generated during the production of sugar palm, *e.g.*, dregs and fibre (Firdayati & Handajani, 2005). These wastes can be utilized rather than released into the environment. By recycling its large by-product, a great source of highly valued material for other sectors was provided. This avoids the loss of a large quantity of untapped biomass as well as environmental concerns in the long term (Padam *et. al.*, 2014).

The sugar palm dregs can be utilized as raw material for bioethanol production (Dayatmo & HS, 2015), while the fibre waste can be used to produce various composite products (Huzaifah et al., 2017; Musa et al., 2021). The utilization of sugar palm fibre (SPF) waste was already conducted by many researchers. Direct utilization or simple pretreatment (*e.g.*, alkaline pretreatment) brought out SPF as filler for composites material such as lightweight brick, concrete, and biodegradable

polymer (Huzaifah et al., 2017; Musa et al., 2021). Converting SPF to cellulose was a choice because cellulose was known to be a versatile material that can be used in bioplastics, composites, membranes, and fibres (Wang et al., 2016). Thus, the production of cellulose is a promising way to utilize SPF.

The next attractive option is converting cellulose into its micro or nano form. Microcrystalline cellulose (MCC) is commonly used in pharmaceutical, food, and beverage industries due to its chemical activity, non-toxicity, and hygroscopicity (Trache et al., 2016). The nanoform of cellulose consists of cellulose nanocrystal (CNC) and cellulose nanofibre (CNF). CNF was mainly produced through mechanical disintegration, while CNC was produced via acid hydrolysis reaction (Shak et al., 2018). Both types of nanocellulose (NC) have a high surface area, low density, good mechanical strength, and low coefficient of thermal expansion which make them very useful for various applications (Xie et al., 2018).

Market demand for NC is expected to increase with the potential largest utilization in packaging and paper applications (de Assis et al., 2017; Shatkin et al., 2014). Cowie et al. (2014) also predicted the market competition between CNC and CNF utilization in several sectors. Between these two types of NC, CNC is relatively easier to produce. Common inorganic acids, e.g., sulfuric and hydrochloric acid can be used as hydrolysis agents in the CNC production process (Perumal et al., 2022). Furthermore, the latest techno-economic analysis conducted by Rosales-Calderon et al., (2021) showed that CNC fabrication via acid hydrolysis is competitive technically and economically.

Sulfuric acid is widely used to produce CNC since its product has good suspension stability due to the negative charge formed on the CNC surface (Xie et al., 2018). Through the sulfuric acid hydrolysis method, CNC has been produced from some biomass, such as rice straw, rice husk, and garlic straw (Johar et al., 2012; Kallel et al., 2016; Xu et al., 2018). CNC also has been fabricated from SPF waste through the sulfuric hydrolysis process (Ilyas et al., 2018). The process involved cellulose extraction from SPF biomass via alkaline pretreatment and bleaching process. Unfortunately, this process still comprised a chlorine-based bleaching method which is harmful to the environment. In contrast to acid or alkaline pretreatment, alkaline peroxide pre-treatment was performed at a more moderate setting (concentration, temperature) and atmospheric pressure, while still successfully removing lignin from a variety of agricultural leftovers (Ho et al., 2019).

Studies of peroxide-base bleaching treatment on SPF prior to cellulose microcrystal production were carried out by Saputro et al. (2017) and Lubis et al. (2018). To the best of our knowledge, the application of peroxide treatments on SPF followed by CNC production has not been conducted. Therefore, according to our previous study on cellulose extraction from SPF using peroxide-based bleaching treatment (Fitriana et al., 2020), in this study we intend to develop CNC production from SPF using more efficient operating condition, e.g., lower temperature and higher solid/liquid ratio than a study carried out by Ilyas et al. (2021). Moreover, the effect of hydrolysis time on the production and characterization of CNC was also studied.

## 2. METHODS

### 2.1. Materials

Cellulose extracted from sugar palm fibre waste (C-SPF) was obtained from our previous work through alkaline and peroxide treatments with a hydrogen peroxide concentration of 15% (Fitriana et al., 2020). Sulfuric acid (95-97%, Merck) and distilled water were used as received.

### 2.2. Cellulose Nano Crystal Fabrication

The production of cellulose nanocrystal (CNC) was referred to previous work by Ilyas et al. (2018) with modification. Sulfuric acid at a concentration of 60% was used as a hydrolyzing agent and was pre-cooled prior to use. Sugar palm fibre cellulose(C-SPF) was mixed with a cold sulfuric acid solution at a solid/liquid ratio of 1:10 followed by stirring with a magnetic stirrer at 200 rpm. The temperature was then gradually increased to 40°C. Different hydrolysis times (45, 60, 75, and 90 minutes) were chosen to evaluate the effect of reaction time.

The hydrolysis process was stopped by adding distilled water to the mixture. The centrifugation procedure was conducted to separate CNC from sulfuric acid (5000 rpm, 10 minutes, 5°C). The suspension was then separated from the supernatant. The procedure was repeated five times followed by freezing the suspension overnight at a temperature of -20°C. Freeze drying of the frozen CNC suspension was then

conducted at a temperature of -55°C for 20 hours. Each sample was given an NC-hydrolysis time code (NC-45, NC-60, NC-75, NC-90) and weighted to calculate the yield of each process.

$$\%Yield = \frac{m_{C-SPF} - m_{CNC}}{m_{C-SPF}} \; x \; 100 \qquad (1)$$

where $m_{C-SPF}$ was the mass of SPC cellulose and $m_{CNC}$ was the mass of freeze-dried CNC. Furthermore, all samples were then stored in a sealed plastic bag until next used.

### 2.3. Characterization

The functional group analysis was conducted with Fourier Transform Infrared (FTIR) method (F. Jiang & Hsieh, 2013). FTIR spectra of cellulose from sugar palm fibre (C-SPF) and CNC of SPF (NC-45, NC-60, NC-75, NC-90) were recorded with Shimadzu IR Prestige-21 FTIR spectrophotometer. The sample was mixed with KBr powder to form a transparent pellet. Spectra analysis was conducted at a wavenumber of 4000-400 cm$^{-1}$.

X-ray diffraction (XRD) spectra were collected using PANalytical X'pert3 Powder with Cu Kα radiation ($\lambda$ = 1.5406 Å). Diffractograms were scanned on 2θ value from 10° to 80° at step size 0.02°. The crystallinity index (CrI) was calculated based on the equation proposed by Segal, Creely, Martin, & Conrad (1959).

$$CrI = \frac{I_{002} - I_{am}}{I_{002}} \; x \; 100 \qquad (2)$$

This equation shows the relative value of CrI as a function of crystalline region maximum intensity ($I_{002}$) and amorphous peak intensity ($I_{am}$). The value of $I_{002}$ is 2θ at around 22.6° (Ilyas et al., 2018) and $I_{am}$ at around 18° (Segal et al., 1959).

The surface morphology of CNCs was observed with the scanning electron microscopy (SEM) method using Hitachi SU3500 at 5 kV acceleration voltage. A diluted CNC suspension was prepared and then the suspension was dripped into a carbon tape that stuck on a copper grid. The suspension was allowed to dry at room temperature for several days (Chen et al., 2017). Samples were coated with gold prior to analysis. Magnification was set at 500-25000 based on each sample condition to measure the diameter of the CNC particle.

## 3. RESULT AND DISCUSSION

### 3.1. Yield of Sugar Palm Cellulose Nano Crystal

The yield of sugar palm cellulose nanocrystal (CNC) at various hydrolysis times was presented in Table 1.

**Table 1.** The yield of sugar palm cellulose nanocrystal as a function of time

| Time (min) | Yield (%) | |
| --- | --- | --- |
| | This research | A study by Ilyas et al. (2021) |
| 30 | - | 33.51 |
| 45 | 34.80 | 29.01 |
| 60 | 11.47 | 13.12 |
| 75 | 1.94 | - |
| 90 | 1.76 | - |

The other operating conditions were temperature of 40°C, solid/liquid ratio of 1:10 and sulfuric acid concentration of 60% with the raw material was peroxide-bleached sugar palm fibre (SPF). Meanwhile, Ilyas et al. (2021) conducted resemblant research but used sugar palm cellulose (SPC) obtained by chlorite bleached SPF with slightly different operating conditions at the temperature of 45°C and solid/liquid ratio of 1:20. Both results showed that the yield was inversely correlated with hydrolysis time.

Isolation of CNC from biomass generally consists of several steps, e.g., delignification, bleaching, and acid hydrolysis processes (Phanthong et al., 2018). Since cellulose consists of the crystalline and amorphous region, the removal of the amorphous region has occurred during the acid hydrolysis (Kim et al., 2015). However, the degradation of the crystalline region might happen during extended hydrolysis time, resulting in a decrease in CNC yield (Ilyas et al., 2021) as observed in this study.

The effect of hydrolysis time also revealed a drastic decrease in CNC yield to 1% at 75 and 90 minutes. This result was in agreement with previous research conducted by Ditzel et al. (2017) at a hydrolysis time of 90 minutes on the raw material of peroxide bleached pine wood and corn cobs, which yielded 2.3 and 6.0% CNC, respectively. Another study on CNC production of chlorite bleached rice straw also resulted in a CNC yields of 4.83 and 6.43% (Lu & Hsieh, 2012). There is no difference in CNC yield between chlorite and peroxide bleached biomass as raw material. Thus, the utilization of the peroxide bleaching method was preferred to the chlorite-based process because it is more environmentally friendly (Qasim et al., 2020), and this method can be utilized to produce cellulose from biomass as a raw material for CNC.
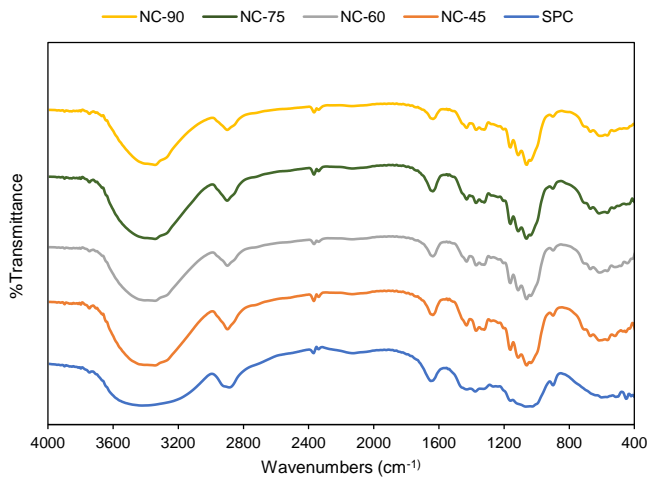
**Figure 1.** FTIR spectra of sugar palm cellulose (SPC) and sugar palm cellulose nanocrystal at various hydrolysis times (NC-45, -60, -75, -90)

### 3.2. Functional Group Analysis

The FT-IR spectra were carried out to characterize the chemical structure by identifying the functional group on the sample. Figure 1 shows the FTIR of the nanocellulose from the sugar palm (*Arenga pinnata*) fibres (NC) with various hydrolysis times. The bands near 3348 and peaks at 2900 were assigned to stretching vibration of OH, and aliphatic saturated C-H (Wang et al., 2017). The peaks at 1427 and 1319 cm$^{-1}$ were assigned to the deformation vibrations bending and wagging of CH$_2$ (Zhao et al., 2019), respectively. The absorption peaks of 894 cm$^{-1}$ were associated with the β-glycosidic linkage of cellulose (Zhao et al., 2018). There was an increase in intensity at an absorbance peak of 897 cm$^{-1}$ in sugar palm cellulose (SPC) compared to NCs indicating enhancement purity of cellulose. The peak at 1635 cm$^{-1}$ was attributed to the hydroxyl bending of absorbed water (Jiang et al., 2011).

It is noted that nanocellulose from the sugar palm fibres (NC) showed stronger and lower bands in OH stretching absorption than that of SPC. In addition, the nanocellulose had a stronger peak in OH stretching absorption than SPC. This result indicated that free hydroxyl groups were exposed on the surface of the nanocellulose due to the sulfuric acid hydrolysis. Furthermore, the absorption peaks that appeared at 1157 cm$^{-1}$ and 609 cm$^{-1}$ in NC-45, NC-60, NC-75, and NC-90 were assigned to the COS and S=O stretching vibrations, respectively. This finding reveals that the interaction between
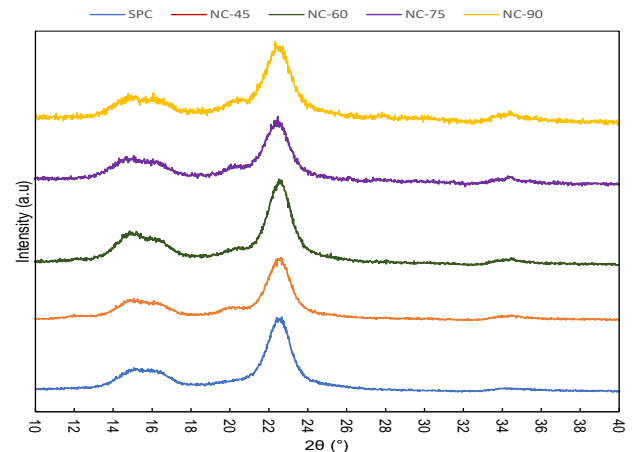


**Figure 2.** XRD diffractogram of sugar palm cellulose (SPC) and sugar palm cellulose nanocrystal at various hydrolysis times (NC-45, -60, -75, -90)

sulfuric acids and hydroxyl groups of nanocellulose generates acidic ester sulfate groups that are exposed on the surface of NCs (Meng et al., 2017).

### 3.3. Crystallinity Analysis

The crystallinity of sugar palm nanocellulose (NC) was observed by the X-ray diffraction (XRD) method. The XRD diffractogram was presented in Figure 2. All samples had similar cellulose type I plane patterns of (010) and (002) which were indicated by a peak at around 15° and 22.6°, respectively. These results are in line with the study of Ilyas et al. (2021). The results also showed the appearance of the lattice plane (021) at around 20° and this result is in accordance with the study of Park et al. (2010) after the conversion SPC to NC by sulfuric acid hydrolysis. Another lattice plane of (040) was also observed in all SPC cellulose nanocrystal diffractograms. This phenomenon was also observed in a previous study with the raw material of chlorite-bleached SPC and garlic straw (Ilyas et al., 2018; Kallel et al., 2016).

Meanwhile, the decrease of CrI during acid hydrolysis was also found in some previous studies with pine wood, corn cobs, and oil palm biomass as the raw material (Ditzel et al., 2017; Haafiz et al., 2014). This circumstance was caused by the degradation of the cellulose crystal phase (Teixeira et al., 2011). This was also probably due to the production of cellulose sulfate ester by the esterification reaction during sulfuric acid hydrolysis (Lu & Hsieh, 2010) which caused the decrease of CNC CrI (Haafiz et al., 2014; Tang et al., 2013).

**Table 2.** The crystallinity index of sugar palm cellulose nanocrystal as a function of hydrolysis time

| Time (min) | Crystallinity Index (%) | |
| --- | --- | --- |
| | This research | Previous study by Ilyas et al. (2021) |
| 0 | 87.9 | 76.0 |
| 30 | - | 81.0 |
| 45 | 86.1 | 85.9 |
| 60 | 85.8 | 83.5 |
| 75 | 81.8 | - |
| 90 | 78.0 | - |

### 3.4. Morphological Analysis

The scanning electron microscopy (SEM) image size of NC is shown in Figure 3. The results of morphological observations showed that an increase in the hydrolysis time leads to a decrease in the nanocellulose diameter. Diameter measurements revealed that there would be some variation in fibre diameter for different treatments. A decrease in diameter was observed with increasing hydrolysis time. This phenomenon could be due to a longer reaction time during the hydrolysis treatment of the fibres, which removed amorphous regions from the nanofibres. Moreover, a longer hydrolysis duration may aggravate the nanocellulose structure (length and diameter). This is in line with the research of Ilyas et al. (2021) that the longer the hydrolysis time will produce smaller and shorter nanocellulose.

Unfortunately, the length of the nanocellulose cannot be determined because the fibres are stacked and overlap. This is most likely caused by the formation of interfibrillar hydrogen bonds (Deepa et al., 2015). SEM images also showed that after treatment, the diameter of sugar palm CNC was less than 100 nm at hydrolysis times of 75 and 90 minutes. The diameter size of NC-75 and NC-90 ranged from 22-66 nm. This result was in agreement with another study by Ilyas et al. (2021) which had CNC with an average diameter size of 25-37 nm based on a Field Emission Scanning Electron Microscopy (FESEM) image. According to Xie et al. (2018), CNC diameter size should be below 100 nm. Thus, based on SEM images NC-45 and NC-60 were not in the CNC size range but still in the size range of the cellulose microcrystals.

Meanwhile, a study by Ilyas et al. (2021) also showed the difference between diameter size measured by FESEM and Transmission Electron Microscopy (TEM). Their result showed an average diameter between 8-13 nm measured by TEM, which meant half to one-third compared to FESEM



(a)                                (b)

(c)                                (d)
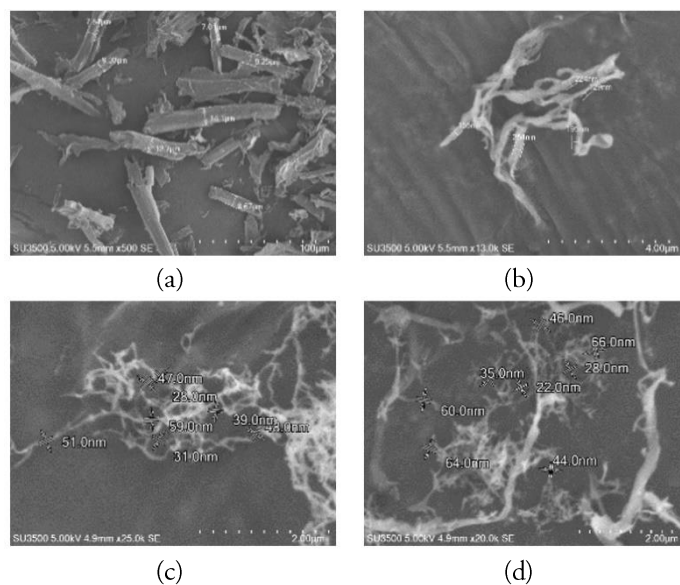
**Figure 3.** SEM image of sugar palm cellulose nano crystal at various hydrolysis time (a) 45; (b) 60; (c) 75; (d) 90 minutes.

measurement. Since the diameter of NC-60 was in a range of 129-254 nm according to SEM, it is suggested to measure by TEM in further studies

### CONCLUSION

The production of cellulose nanocrystal (CNC) from peroxide bleached sugar palm fibre (SPF) waste was accomplished. All CNC produced by hydrolysis times of 45, 60, 75, and 90 minutes had similar functional groups and a high crystallinity index. The CNC yield decreased with increasing hydrolysis time. CNC with a diameter below 100 nm was obtained at hydrolysis times of 75 and 90 minutes. These results showed that the preparation of SPF cellulose by peroxide bleaching method followed by sulfuric acid hydrolysis could be an environmentally friendly alternative method to produce CNC. Furthermore, by involving the yield and crystallinity index results, further study on the hydrolysis time between 45 and 75 minutes can be performed to optimize not only the diameter size of CNC but also the yield and crystallinity index.

### ACKNOWLEDGEMENT

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