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Characteristics of crude cellulose from palm (*Elaeis oleifera*) midrib by sodium hydroxide pretreatment

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Abstract

The purpose of this research was to determine the effect of concentration of sodium hydroxide (NaOH) on physicochemical characteristics of cellulose powder extracted from palm midrib that was underutilized optimally. Cellulose extraction was done using a solution of NaOH with concentrations of 9, 12, 15, 18%. Cellulose powder obtained was analyzed water content, cellulose content, yield, lightness, functional groups, and crystallinity. Cellulose powder extracted with NaOH 15% had water content 9.3% (w.b), cellulose content 89.63% (d.b), yield 37.32% (d.b), lightness value 90.83, and crystallinity 31.57%. NaOH had the ability to break lignocellulose bond so that cellulose will be free from lignin. The results of FTIR spectra analysis of commercial cellulose and cellulose from palm midrib extracted with NaOH 15% solution showed the same pattern of the functional group.

1. Introduction

The midrib was part of the palm oil plant that was underutilized optimally, even its presence was often regarded as a plantation waste (Ferdiansyah *et al.,* 2017). One of the efforts that had been made to utilize palm midrib was making it as a mixture of animal feed. This utilization was still limited, in terms of application and economic value. Another alternative in terms of utilization of palm midrib was needed so that it would produce more economically valuable products and had various applications.

The process of delignification, bleaching, and purification in palm midrib sample will give white alpha-cellulose (Sebayang and Sembiring, 2017). From 75 grams of palm oil, midrib powder will give 19.77 grams of pure alpha-cellulose (it was assumed to be 26.36% of the initial mass). Such high levels of cellulose were a potential that palm midrib can be further processed into a product of higher economic value and useful in applications. Cellulose was a polymer that is found in the cell wall of plants. In the cell wall, cellulose was bound to other substances such as lignin and hemicellulose. To get a pure cellulose it was necessary to do the extraction process first. Cellulose could be extracted from palm midrib by extraction process using a basic NaOH solution. NaOH had the ability to disrupt the lignin structure of the biomass (Brodeur, 2011). The purpose of this research was to determine the effect of the concentration of NaOH on some characteristics of cellulose powder extracted from palm midrib

2. Materials and methods

2.1 Materials

Palm midrib used in the research was derived from oil palm plantation in Donomulyo District, Malang, East Java, Indonesia. NaOH (technical grade), aquadest, NaCl, acetic acid, NaOCl, and Na-metabisulfite were used for cellulose extraction process. Ethanol, acetic acid, NaOH, and aquadest were used for cellulose characterization. The equipment used for the preparation of palm midrib powder were grinding machine and sieve. For the process of extraction of palm, midrib cellulose, Erlenmeyer 1 L, and water bath equipped temperature control was used. The oven equipment, desiccators, weighing bottles, porcelain cups, furnaces (Heracus Instruments M-110), Fourier-transform infrared spectroscopy (FT-IR, Shimadzu Prestige-21), X-Ray diffractometry (XRD, Mac Science MXP3) were used for characterization.

2.2 Preparation of palm midrib powder

Powdering palm midrib started with cutting and

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drying of the palm midrib in dryer cabinet of 50°C, the pieces of dried palm midrib were milled by using a grinding machine and then sieved to 60 mesh size.

2.3 Cellulose extraction of palm midrib

Cellulose extraction of palm midrib was extracted from the palm midrib with NaOH solution with several concentrations (9, 12, 15, 18%) temperature of 100°C for 3 hours that aimed to dissolve the lignin components. The solids were lagged after washing with clean water from residual of NaOH, immersed with distilled water mixed with NaCl and acetic acid. Washing process and the solids were lagged could be done by bleaching with NaOCl 12% and Na metabisulphite of 60°C for 3 hours, then washed with clean water until the pulp (cellulose) obtained odorless hypochlorite. Cellulose was obtained then dried using an oven.

2.4 Characterization of palm midrib cellulose

Palm midrib cellulose was characterized based on the water content, yield, cellulose content, lightness, crystallinity and functional groups. Methods used for analysis were cellulose content (Browning, 1967), water content (ASTM D-1439-94 (ASTM, 1994)), lightness analysis (Rossel *et al.,* 2009). Structural analysis was performed using FTIR and X-Ray Diffraction (Lii *et al.,* 2002). FTIR used to functional groups analysis and X-Ray Diffraction used to crystallinity analysis.

2.5 Statistical analysis

This research used factorial design. The Data obtained were analyzed by Variant Analysis (ANOVA) followed by real difference test DMRT (Duncan Multiple Range Test) with confidence interval 5%.

3. Results and discussion

3.1 Characteristics of palm midrib powder

This research used the main raw material, palm midrib, that had been dried and made into powder with a size of 60 mesh. The powder making process aimed to expand the surface of the material so that it will be easier in the process of cellulose extraction. The palm midrib powder was characterized to determine the cellulose content and the characteristics of the cellulose. The characterization results of the palm midrib powder are shown in Table 1.

Table 1. Characteristic of palm midrib powder

Characteristic	Content
Water Content (% w.b)	7.51
Ash Content (% d.b)	2.95
Cellulose Content (% d.b)	70.78

In this study, comparison difference was observed. Palm midrib had a moisture content of 9.7% (d.b) and cellulose content of 35.88% (d.b) (Caesari *et al.,* 2014). The difference in the characteristics of oil palm midrib occurred due to differences in the place of growth and varieties of oil palm. The chemical composition of palm midrib depended on the nutrient content of the plant and the absorptive properties of the plant.

3.2 Effect of NaOH concentration on water content of cellulose

The cellulose extracted from palm midrib had an average water content of 8% - 9% (w.b). Figure 1 shows that the cellulose extracted using 15% NaOH concentration had the highest water content of 9.3% (w.b). But the results of statistical analysis showed that the concentration of NaOH did not affect the water content of cellulose.

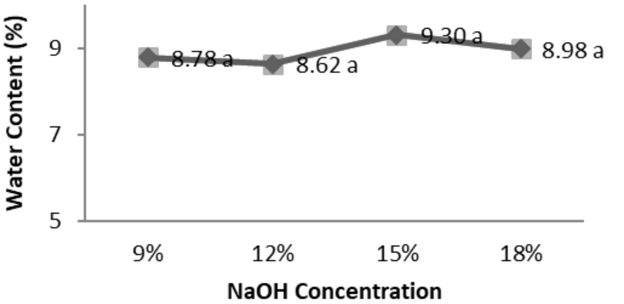

Figure 1. Effect of NaOH concentration on water content of cellulose

3.3 Effect of NaOH concentration on yield of cellulose

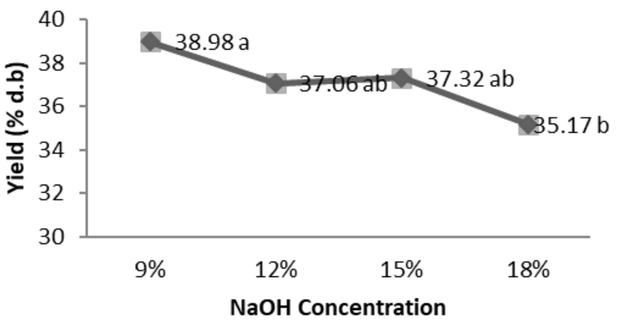

Figure 2. Effect of NaOH concentration on yield of cellulose

Increasing the NaOH concentration caused the yield to decrease in the process of cellulose extraction from palm midrib. The decrease in yield of the cellulose powder occurred due to NaOH breaks the lignin structure that bound cellulose fibers and dissolve them to pulp form, thus reducing the lignin. Brodeur *et al.* (2011) stated NaOH had been extensively studied for many years, and it had been shown to disrupt the lignin structure of the biomass, increasing the accessibility of enzymes to cellulose However, the decrease of the cellulose yield with increasing NaOH concentration may

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cause the increase of the cellulose purity when the lignin component is reduced. The effect of NaOH concentration on cellulose yield on palm midrib cellulose powder is displayed in Figure 2.

3.4 Effect of NaOH concentration on cellulose content

Different concentrations of NaOH also affected cellulose content, where increased NaOH concentration led to elevated cellulosic content. This was due to the increased ability of NaOH to break the lignocellulose bond so that cellulose will be free from lignin. The highest cellulose content was at the concentration of 15% NaOH. However, concentration of 18% NaOH tend to decrease cellulose content due to NaOH degrades cellulose by damaging the structural crystals of cellulose. The use of an alkali causes the degradation of ester and glycosidic side chains resulting in structural alteration of lignin, cellulose swelling, partial decrystallization of cellulose, and partial solvation of hemicellulose (Brodeur *et al.,* 2011). The effect of NaOH concentration on cellulose content on palm midrib cellulose powder is shown in Figure 3.

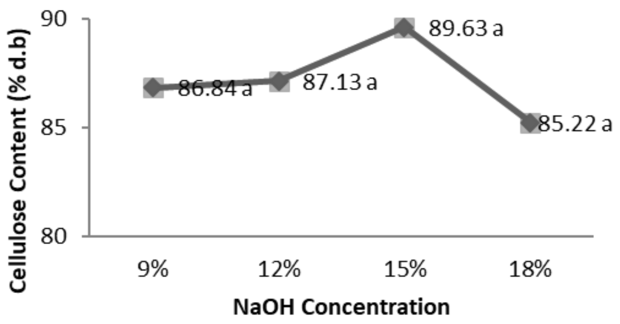

Figure 3. Effect of NaOH concentration on cellulose content

3.5 Effect of NaOH concentration on cellulose lightness

The NaOH concentration affected the lightness of the extracted cellulose with the pattern as shown in Figure 4. The higher concentration of NaOH led to the increase of lightness caused by the ability of NaOH to break the bond between cellulose with lignin. The non-cellulose compounds cause the brown color of the material (Fengel and Wegener, 1995). The decrease in lightness at higher NaOH concentrations was due to the high concentration of the base of the solution so that the pigment removal process in the cellulose fiber was inhibited (Kuntari, 2006). Extremely high NaOH concentration caused the process of saturation, so the removal process of lignin was not optimal. Figure 5 shows the visual appearance of cellulose.

3.6 Effect of NaOH concentration on cellulose crystallinity

The crystallinity change occurring in the polymer was affected by the extraction conditions such as the NaOH concentration. Figure 6 showed the effect of NaOH concentration on cellulose crystallinity. The higher NaOH concentration caused the crystallinity to decrease. Crystallinity decreases due to the intermittent hydrogen bonds and intra-molecules of cellulose due to the extraction process with NaOH. As a result, the amorphous regions become more than the crystalline regions, so the percent crystallinity reduced. Cellulose with low crystallinity levels will be easier to process into cellulose derivative products.

The crystallinity of palm midrib cellulose extracted with 15% NaOH was 31.57%. Based on the previous research conducted by Adinugraha *et al.* (2005), the authors stated that NaOH influence on cellulose crystallinity. Other research mentioned banana stem cellulose extracted using NaOH 8% had a crystallinity of 49.27% (Adinugraha *et al.*, 2005), pineapple crown cellulose extracted using 12% NaOH had a crystallinity of 36% (Suzana, 2009), pod husk cacao cellulose extracted using 12% NaOH had a crystallinity of 27.14% (Hutomo *et al.,* 2012), and water hyacinth cellulose extracted using 15% NaOH had a crystallinity of 24.36% (Ventola, 2013).

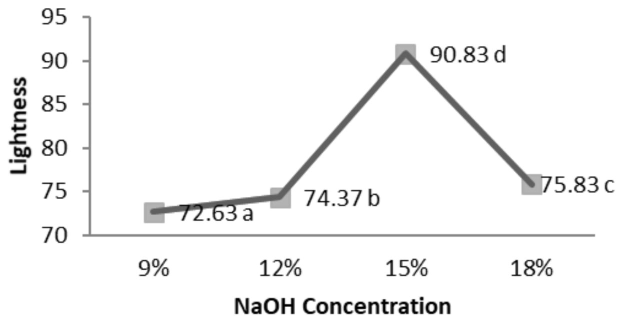

Figure 4. Effect of NaOH concentration on cellulose lightness

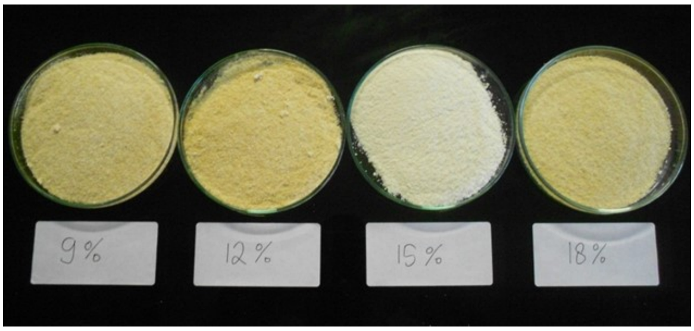

Figure 5. Cellulose powder extracted with various cancentrations of NaOH

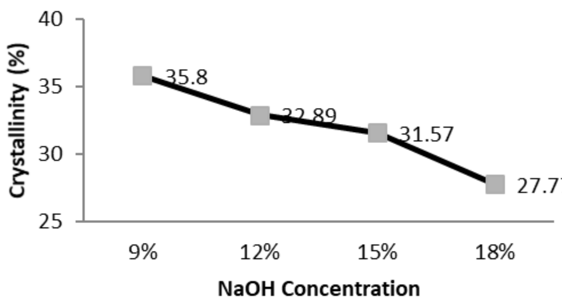

Figure 6. Effect of NaOH concentration on cellulose crystallinity

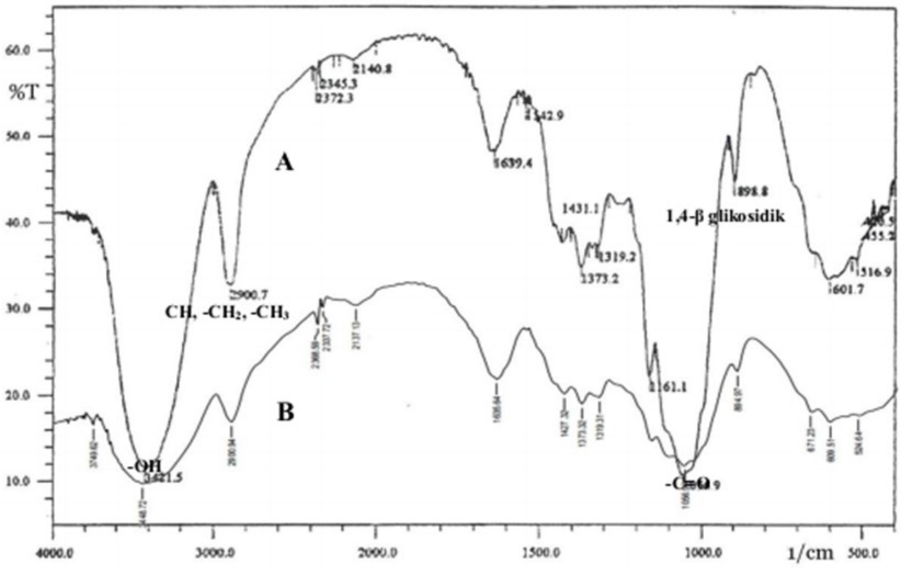

Figure 7. FTIR spectra of commercial cellulose (A) and palm midrib cellulose (B)

3.7 FTIR spectra of cellulose

The results of FTIR spectra of Sigma commercial cellulose and palm midrib cellulose that extraction with 15% NaOH showed the same pattern. The -OH group will appear as an in-plane deformation at the wave number between 3,448 - 3,410 cm^{-1} where in the OH group of cellulose derived from palm midrib in a wave of 3,448.72 cm^{-1} (Meenakshi *et al.,* 2002). The C-O group as stretching vibration was indicated on the wave number 1,064-1,056 cm^{-1}, on the palm midrib cellulose peak at the wave number 1,056.99 cm^{-1}. At the number of 3,448 cm^{-1} wave looks very sharp peak which was characteristic of cellulose. The peak shows stretching vibration and hydrogen bonds intra-molecular -COH. The wave number 2,924 - 2,893 cm^{-1} was the peak of the -CH and $-CH^2$ groups of cellulose. The wave number 894 cm^{-1} shows a 1.4-β bond of cellulose. The results of commercial cellulose FTIR spectra and palm midrib cellulose can be seen in Table 2. FTIR spectra for commercial cellulose Sigma and palm midrib cellulose with 15% NaOH is as shown in Figure 7. The 15% NaOH concentration was the best treatment. It was based on the parameters of high lightness levels as a result of the double bleaching process.

Table 2. The results of FTIR spectra of commercial cellulose sigma and palm midrib cellulose

Numbers of waves (cm^{-1})		Cluster of Bonds
Sigma Commercial Cellulose	Cellulose Oil Palm	
3421	3448	OH stretching
2900	2900	CH streching CH_2 & CH_3 group
1373	1373	OH inplane bonding
1056	1056	C-O asymmetry bridge bonding
898	894	1,4-β glycosidic

4. Conclusion

The concentration of NaOH influenced the water content, cellulose content, yield, lightness, functional groups, and crystallinity of cellulose powder extracted from palm midrib. Cellulose powder extracted with NaOH 15% had water content 9.3% (w.b), cellulose content 89.63% (d.b), yield 37.32% (d.b), lightness value 90.83, and crystallinity 31.57%. NaOH had the ability to break lignocellulose bond so that cellulose will be released from lignin. The results of spectra FTIR analysis of commercial cellulose and cellulose from palm midrib extracted with NaOH 15% solution showed the same pattern of functional group.

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