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# EXPERIMENTAL STUDY OF CELLULOSE EXTRACTION FROM OIL PALM EMPTY FRUITS BUNCHES


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***ABSTRACT***

*Oil palm empty fruit bunches (OPEFB) are one of the non-timber solid wastes produced in the process of making palm oil. Empty palm oil bunches that have been cleaned and then hydrolyzed with 3.5% of $HNO_3$ and 0.03 gram $NaNO_2$ at 90ºC. Then it was dignified by varying the concentration of NaOH 1%, 2%, 3%, 4% and using temperature variations of 40 ºC, 50 ºC, and 60 ºC for 1 hour. Then the cellulose obtained was tested by FTIR test, XRD test, and SEM test, and the yield was calculated. From the results of the FT-IR test, it is obtained that the high wavenumber is 2800-3300 $cm^{-1}$ and the low wavenumber is 500-1400 $cm^{-1}$. The spectrum shows a broad absorption peak located at 2800-4000 $cm^{-1}$ which is a stretch of the –OH group. The highest yield of 90.4% was found at a concentration of 4% NaOH and 40 ºC. The lowest yield 24.88% was found at a concentration of 3% NaOH and 60 ºC. The results of studies show that the concentration of NaOH used affects the yield of cellulose produced.*



***Keywords:*** *Cellulose, Extraction, Concentration of NaOH and Yield*



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***ABSTRAK***

Tandan Kosong Sawit (TKS) merupakan salah satu limbah padat non-kayu yang dihasilkan pada proses pembuatan minyak kelapa sawit. Tandan kosong kelapa sawit yang telah dibersihkan kemudian dihidrolisis dengan $HNO_3$ 3,5% dan $NaNO_2$ 0.03 gram pada 90 ºC. Kemudian didelignifikasi dengan variasi konsentrasi NaOH 1 %, 2 %, 3 %, 4 % dan suhu 40 ºC, 50 ºC, dan 60 ºC selama 1 jam. Kemudian selulosa yang didapatkan diuji dengan uji FTIR uji XRD, , uji SEM, dan dihitung yield nya. Dari hasil uji FT-IR didapatkan bilangan gelombang tinggi yaitu 2800-3300 $cm^{-1}$ dan bilangan gelombang rendah yaitu 500-1400 $cm^{-1}$. Spektrum memperlihatkan puncak serapan yang luas terletak pada 2800-4000 $cm^{-1}$ merupakan peregangan kelompok –OH. Yield yang tinnginya 90,4% terdapat pada konsentrasi NaOH 4% dan suhu 40 ºC. Yield yang terendah 24,88% terdapat pada konsentrasi NaOH 3% dan suhu 60 ºC. Hasil penelitian menunjukkan semakin besar konsentrasi NaOH yang digunakan berpengaruh terhadap yield selulosa yang dihasilkan.



**Kata kunci:** Selulosa, Ekstraksi, Konsentrasi NaOH dan Yield.

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## INTRODUCTION

OPEFB is the largest waste contained in oil palm bunches. The main components of OPEFB waste are cellulose and lignin, so this waste is referred to as lignocellulosic waste [1]. OPEFB contain chemicals such as cellulose 36.59%, lignin 26.53% hemicellulose 24.97 %, and

ash 1.79% [2]. The composition of cellulose which is quite large in oil palm bunches makes this OPEFB has the potential to be used as raw material for the manufacture of -cellulose.

Cellulose is a polymer that is not easily soluble in alkali but easily soluble in acid, while hemicellulose is a polymer that is easily soluble in alkali but difficult to dissolve in acid. Hemicellulose is also not long fibre-like cellulose. So that hemicellulose is easily removed from cellulose by heating with alkali. The results of hydrolysis of cellulose will produce D-glucose, while the results of hydrolysis of hemicellulose will produce D-xylose and other monosaccharides. [3]. Cellulose forms microfibrils through inter and intra-molecular bonds thus providing a soluble structure. Cellulose microfibrils are of 2 types, namely crystalline and amorphous. Hemicellulose is non-crystalline, not fibrous and easy to expand [4].

Cellulose extraction can be carried out by reacting it with a 17.5% NaOH solution at a temperature of 80 $^{o}$C, the cellulose obtained is dissolved in 2% Na2SO3 for 45 minutes, which is then followed by bleaching with calcium hypochlorite and 10% hydrogen peroxide [5].

Pretreatment studies of lignocellulosic materials from coconut leaves were carried out using alkaline and chlorite techniques. This method showed an increase in microfibril content from 0.373 kg/kg to 0.896 kg/kg after chlorite application and alkaline extraction of lignin and hemicellulose. The crystallinity index obtained from XRD and FTIR based on samples before and after treatment were 42.3 and 47.7, respectively. The crystallinity index increased due to the decreasing content of lignin and hemicellulose in this delignification process. From this study, it can be concluded that the diameter of the microfibrils in the range is 10-15μm [6].

The results of the chemical analysis of the fibre showed that most of the cellulose could be regenerated, the percentage of lignin decreased significantly to 18%, and there was an increase in the crystallinity index in the pretreatment with NaOH. At a NaOH concentration greater than 5%, a structural change begins from cellulose I to cellulose II [7].

The delignification process of lignocellulosic OPEFB material can be carried out using 10% NaOH (1:10) and cooking in an autoclave at a temperature of 121 $^{o}$C [8]. The process of extracting cellulose from OPEFB can also be carried out through a strong alkaline hydrolysis process with 15% and 17.5% NaOH at a temperature of 80$^{o}$C and neutralization with NaOCl and bleaching with $H_2O_2$ to obtain a yield of 50.04% with a crystallinity degree of 61.37%, and X-RD shows a peak that appears at an angle of 2θ around 22.5o, this area is a typical peak of the cellulose structure [9].

## LITERATURE REVIEW

Cellulose is a polymer compound having the chemical formula $(C_6H_{10}O_5)n$ which is used by plants as structural polysaccharides. These cellulose polymers consist of hundreds to tens of thousands of bonded D-glucose units [10].

Cellulose is one of the carbohydrates that includes structural polysaccharides, which provide strength to wood and branches for plants. Polysaccharides are easily converted through the hydrolysis process so that cellulose can be converted into monosaccharides. It is estimated that about 1011 tons of cellulose are biosynthesized annually, cellulose accounts for 50% of the free carbon of the earth. Dried leaves are estimated to contain as much as 10-20% cellulose, 50% wood and 90% cotton [11].

Cellulose from OPEFB is obtained through a delignification process. Delignification is a method of separating components from a material using compounds that can be in the form of alkalis or acids. Delignification using alkali will separate the cellulose and other components [9].

Cellulose is an organic compound found in cell walls along with lignin which plays a role in strengthening plant structures. Cellulose consists of long chains of glucose units bound by 1-4 β-glucoside bonds [10]. The structure of cellulose can be seen in Figure 1 below :

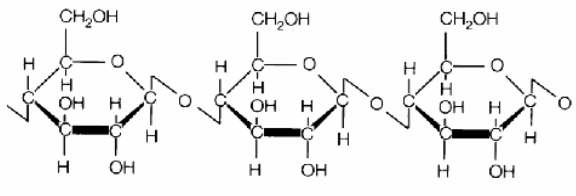

**Figure 1.** Cellulose structure [10].

In its development, cellulose can be used in various ways, for example, the cellulose in empty palm oil bunches has the potential to be used as a renewable energy source in the form of bioethanol. The production of bioethanol is done by removing lignin in order to obtain cellulose which will later be fermented for bioethanol production, but the lignin must be broken down by delignification and sulfonation [12].

Synthesis of polyaniline-cellulose composites using a cellulose matrix derived from empty palm oil bunches through the swelling stage. The polyaniline-cellulose obtained is a semi-conductor because it has a higher conductivity value compared to the composite which was synthesized without going through the initial swelling treatment. Cellulose from empty palm oil bunches can be used as renewable energy in the form of ethanol through simultaneous saccharification-fermentation using cellulase enzymes and yeast Saccharomyces cerevisiae. Cellulose can also be combined with ZnO for the manufacture of bioplastics that are easily degraded by microbes [13].

Cellulose can be converted into cellulose acetate through the cleanse process using -cellulose as the raw material. The reaction steps are activation, acetylation, hydrolysis, neutralization and drying. Isolation of crystalline cellulose nano from alpha cellulose derived from oil palm empty fruit bunches by delignification process using $HNO_3$ [14]. Cellulose can be converted to carboxy methylcellulose (CMC) from -cellulose consisting of two processes, alkalization process and esterification process [15].

Based on the degree of polymerization (DP) and solubility in 17.5% sodium hydroxide (NaOH)n compounds, cellulose can be divided into three types, namely: a) Alpha Cellulose (Alpha Cellulose) is long-chain cellulose that is resistant and insoluble in 17.5% NaOH solution or strong alkaline solution with DP (Polymerization Degree) 600 – 15000. – cellulose is used as an estimator or level of cellulose purity. b) Beta Cellulose (Betha Cellulose) is short-chain cellulose soluble in 17.5% NaOH solution or strong base with Polymerization Degree (DP) ranging from 15 – 90. This beta cellulose can precipitate if the extract is neutralized. c) Gamma Cellulose (Gamma Cellulose) is short-chain cellulose soluble in 17.5% NaOH solution or strong base with a Polymerization Degree (DP) of less than 15. The main content is hemicellulose [16].

## METHOD

Materials needed in this research include OPEFB from PT. Ika Bina Agro Wisesa North Aceh, other materials used were $HNO_3$ solution, NaOH solution, $Na_2SO_3$ solution, NaOCl solution, and $H_2O_2$ solution and $NaNO_2$ solid, for analysis EMSURE® ACS, ISO, Reag. Ph Eur from PT. Rudang Jaya-Medan. The equipment used in this research includes a hot plate of magnetic stirrer, Erlenmeyer, measuring cup, measuring flask, thermometer, and paper frequently.

This study consisted of three stages, namely the preparation of OPEFB raw materials (washing, drying and grinding) extraction of cellulose, and lignocellulosic analysis using the Chesson-Datta method.

OPEFB extraction was carried out by the solid-liquid extraction method using a solution of 3.5% $HNO_3$ and 0.03 gram $NaNO_2$ was heated at 90 $^oC$ for 2 hours. Then it was delignified using a solution of NaOH (1, 2, 3, and 4) % and 2% $Na_2SO_3$ at 40, 50, and 60$^oC$ for 1 hour. Then the cellulose extract is bleached with 1.75 % NaOCl for 30 minutes, then followed with 10 % $H_2O_2$ for 1 hour in the oven, and the cellulose extract obtained was dried.

Lignocellulosic analysis was carried out using the Chesson-data method, first weighing 1 gram of OPEFB as weight (a), then refluxed using 120 ml aquadest at 100 $^oC$ for 1 hour, then

filtered and dried, then weighed in as weight (b). Then the residue was refluxed using 150 ml of 0.5M $H_2SO_4$ solution for 1 hour, the residue was filtered and dried and then weighed as weight (c). The residue was soaked with 10 ml of 72 % $H_2SO_4$ solution for 4 hours, then filtered and dried, then weighed as weight (d), and then the residue was ashed and weighed as weight (e). Then the percent chemical content was calculated using the Chesson-Data method.

## Characterization of Cellulose

### a. Lignocellulosic analysis

This method is used to analyze the chemical content contained in biomass raw materials that exist in nature. The percentage of chemical content contained in the biomass is calculated using the Chesson-Datta method, which is as follows:

(1). Hot water solube (%) $= \frac{a-b}{a} \times 100\%$..... (1)

(2). Hemicellulose (%) $= \frac{b-c}{a} \times 100\%$..... (2)

(3). Cellulose (%) $= \frac{c-d}{a} \times 100\%$..... (3)

(4). Lignin (%) $= \frac{d-e}{a} \times 100\%$..... (4)

(5). Ash (%) $= \frac{e}{a} \times 100\%$..... (5)

### b. *Fourier Transform Infra-Red* (FT-IR)

FT-IR is a tool used for the analysis of chemical compounds. Infrared spectra of a compound can provide an overview of and molecular structure of the compound. The chemical structures of cellulose were characterized on a Nicolet 8700 FT-IR spectrophotometer. FTIR spectra were recorded in the spectral range of 4000–400 $cm^{-1}$.

### c. *X-ray diffraction* (XRD)

The crystal structures were characterized on a Philips PZ1200 X-ray diffraction (X-RD) by using Cu Kα X-rays with a voltage of 40 kV and a current of 30 mA. X-ray diffraction data were collected over an angular range of 0–50 in steps of 0,02$^o$ at room temperature.

### d. *Spektroskopi Scanning Electron Microscope* ( SEM )

SEM is a type of electron microscope that uses electron beams to describe the surface shape of the material being analyzed. Morphology of cellulose analyzed by using SEM-EDX model Oxford INCA 400 voltage 15 KV.

## RESULT AND DISCUSSION

### Yield Cellulose

Table 1 shows the cellulose content obtained from OPEFB using the solid-liquid extraction method. High cellulose content was obtained in the extraction process using 4% NaOH and a temperature of 40 $^o$C for 22.6 grams with a yield of 90.4%. The lowest cellulose content was obtained from the extraction process with 3% NaOH and a temperature of 60 $^o$C as much as 6.22 grams with a yield of 24.88%.

**Tabel 1.** Cellulose is obtained by using the extraction process at various concentrations

| Concentrations of NaOH (%) | Temperature ($^o$C) | Cellulose (gram) | Yield (%) |
|---|---|---|---|
| 1 | 40 | 17.97 | 71.88 |
| | 50 | 12.08 | 48.32 |

| Concentrations of NaOH (%) | Temperature (°C) | Cellulose (gram) | Yield (%) |
|---|---|---|---|
| | 60 | 8.34 | 33.36 |
| 2 | 40 | 17.84 | 71.36 |
| | 50 | 9.54 | 38.16 |
| | 60 | 8.28 | 33.12 |
| 3 | 40 | 20.55 | 82.20 |
| | 50 | 12.90 | 51.60 |
| | 60 | 6.22 | 24.88 |
| 4 | 40 | 22.60 | 90.40 |
| | 50 | 18.75 | 75.00 |
| | 60 | 15.41 | 61.66 |

**Analysis of Lignocellulosic Content**

The method for measuring lignocellulosic content that has been commonly used is the method of Chesson-Datta. This method is a gravimetric analysis of each component after being hydrolyzed or dissolved. The main step of this method is to remove the extractive, then hydrolyze hemicellulose with strong acid without heating, followed by hydrolysis using dilute acid at high temperature. The last part that is insoluble is lignin, the lignin content is corrected by the ash content.

**Table 2.** Chemical composition of OPEFB

| No. | Component | Content (%) |
|---|---|---|
| 1. | Hot Water Solute | 5 |
| 2. | Hemicellulose | 15 |
| 3. | Cellulose | 50 |
| 4. | Lignin | 20 |
| 5. | Ash | 10 |

From Table 2, the analysis data of the lignocellulosic using the Chesson-Datta method, the results of the chemical components contained in robusta empty palm oil bunches are 5% solute content, 15% hemicellulose content, and 50% cellulose content, 20% lignin content, and ash content 10%.

**Effect of Temperature and Concentration of NaOH on Cellulose Yield**

The relationship between the effect of temperature and NaOH concentration on the yield of cellulose produced can be seen in figure 2. Figure 2. shows the decrease in yield when temperature increases at each NaOH concentration. The highest yield is obtained at a concentration of 4% NaOH at a temperature of 40 °C, which is 90.4% and the lowest yield is obtained at a concentration of 3% NaOH at a temperature of 60 °C, which is 24.88%. The graph shows that the higher the concentration of NaOH, the higher the yield obtained. This is because the higher the concentration of NaOH used, the more lignin is decomposed from the cellulose chain, leaving cellulose crystals. These results are in accordance with the research conducted by Zulnazri et al., where the higher the concentration of NaOH used, the less lignin content remains [17]. This is because NaOH can separate lignin from cellulose. According to Zulnazri, high cellulose content can be obtained from OPEFB by extraction using 17.5% NaOH for 2 hour, where the lignin will be completely removed from the lignocellulose chain and leave cellulose crystals [18].

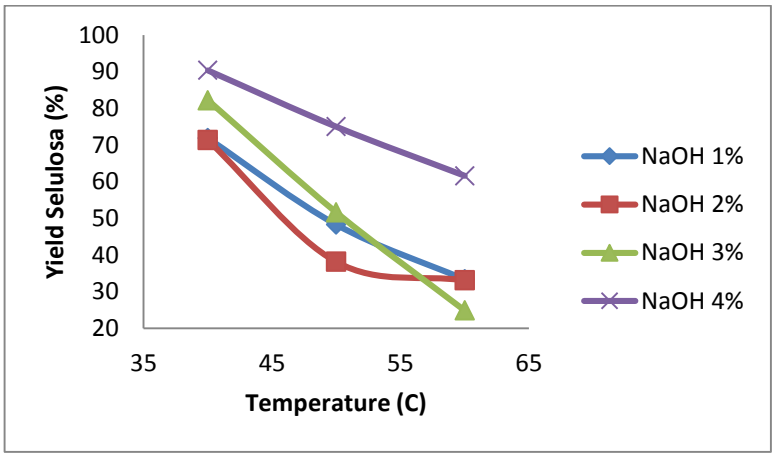


Figure 2. The effect of temperature and concentration on the yield of cellulose

**Analysis of Cellulose Crystal Structure by X-RD**

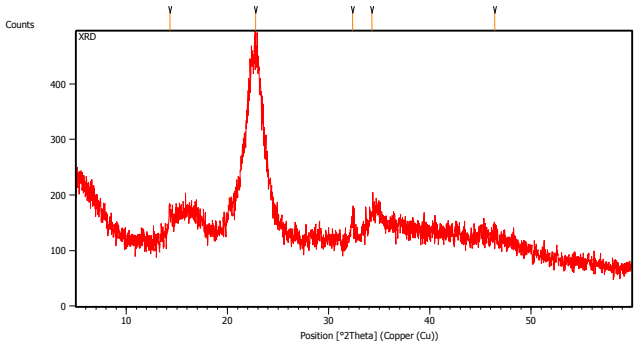


Figure 3. X-RD pattern of cellulose crystal hydrolyzed by NaOH 4%

Figure 3. shows the X-RD pattern of hydrolyzed cellulose from OPEFB with 4% NaOH at 40 ºC for 1 hour. All X-RD patterns show the presence of a high peak appearing at an angle of 2θ around 22.5º, this area is a typical peak of the cellulose structure. It is reported by Zulnazri, that the typical peak of cellulose appears at an angle of 2θ around 22.5º [9]. According to Rosli, the characteristic peak of cellulose crystals is at an angle of 22º-23º [19].

Based X-RD patern fig 3, the crystallinity of the samples cellulose was determined by Segal's method [20].

$$Xc = \frac{\mathrm{I}(crystaline) - \mathrm{I}(amorf)}{\mathrm{I}(crystaline)} \ 100\ \%$$

High crystallinity of cellulose was obtained by hydrolysis using 4% NaOH at 40ºC at 80%. The high crystallinity obtained was due to the deletion of hemicellulose and lignin in the amorphous region leading to the arrangement of the cellulose molecules [21].

**Analysis of Cellulose Functional Groups with FT-IR Test**

The characteristic feature of the FT-IR spectrum of cellulose shown in Fig. 4 displays two main absorption regions, namely the high wavenumber and low wavenumber regions. The spectrum shows a broad absorption peak located around the 3314 $cm^{-1}$ band which is a stretch of the –OH group, and the absorption peak in the 1028 $cm^{-1}$ band is related to the -$CH_2$ group. This is in accordance with the research conducted by Jahan, that -OH group stretching is at around the 1000-1050 $cm^{-1}$ [22]. The absorption peak of 2896 $cm^{-1}$ is the overlap of the -$CH_2$.

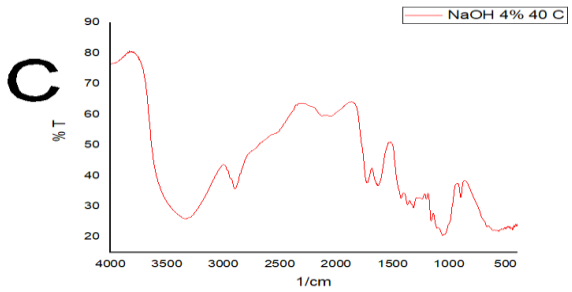


Figure 4. FT-IR spectrum of cellulose

Figure 4. shows an increase in intensity in the 1028 $cm^{-1}$ band which shows the stretching of the C-O-C pyranose ring, which indicates an increase in the value of cellulose crystals. The absorption peak in the 500 $cm^{-1}$ bands is the lowest C-H vibration of cellulose (anomeric vibrations, specific for -glucosides), this is in accordance with research conducted by Li [21].

**Morphological Analysis with SEM**

The morphology and dimensions of the crystal particles were analyzed by scanning electron micrographs (SEM). Cellulose has a crystallin and more regular particle when compared to lignocellulose which looks more random. Figure 5. below shows the morphology of cellulose hydrolyzed at a temperature of 40ºC using 4% NaOH.

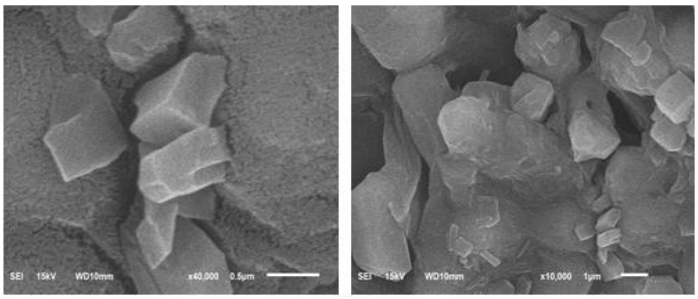


Figure 5. Micrograp SEM of cellulose (a) 0.5µm scale, zoom of 40.000 times, (b) 1µm scale, zoom of 10,000 times**.**

Figure 5 (a) shows the morphology of cellulose with a scale of 0.5 µm and a magnification of 40,000 times. Figure 5 (b) shows the morphology of cellulose with a scale of 1µm and magnification of 50,000 times. Based on Figure 5, the cellulose surface shows the same morphology as a regular sandwich shape.

**CONCLUSIONS**

The process of extracting cellulose from OPEFB can be carried out using a NaOH catalyst through a hydrolysis reaction. The higher the temperature in the hydrolysis process, the lower the cellulose yield obtained. Where in the hydrolysis process with 1% NaOH at temperatures (40, 50 and 60) ºC the yields are 71.88%, 48.32%, and 33.36%, respectively, while in the hydrolysis process with 4% NaOH at a temperature of (40, 50 and 60) ºC yields obtained are 90.40%, 75.00%, and 61.66%, respectively. The cellulose obtained can be synthesized into cellulose nanocrystals that can be applied as biomedical materials.

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