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CELLULOSE EXTRACTION FROM COCONUT COIR WITH ALKALINE DELIGNIFICATION PROCESS

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Abstract. Coconut has been known for its benefits in human life. Coconut coir, as part of coconut which is considered as waste, contains useful components. It contains high cellulose which is could be used in fiber industries. Meanwhile, coconut coir also contains lignin which needs to be separated. In this study, a delignification process was used to remove the brown color on the fiber caused by the lignin content. The delignification process was a pretreatment before the cellulose extraction was carried out. It had been done in the various NaOH concentration (0.5; 8 and 1.5 M), reaction time (1; 1.5; and 2 hours) and the reaction temperature (60,70, and 80 °C). This study aims to determine the cellulose content and the factor that affected the cellulose extraction and the characteristics of the cellulose extracted from the coconut coir. The Chesson Data method and SEM analysis have been used for the characterization of the cellulose. The delignification method known has the potential as a simple and effective method for extracting cellulose from natural materials. The result shows that the optimum cellulose content obtained at 100 mesh coir particle size, 1.5 M NaOH concentration, at 80°C for 1.5 hours was 69.82 %.

Keywords: Cellulose; Coconut Coir; Delignification; Lignin.

1. Introduction

Indonesia is an archipelagic country located in the tropics with favorable climatic conditions and is the second major coconut-producing country in the world after the Philippines [1]. Coir is an industrial waste derived from coconut which contains lignocellulose. In Indonesia alone, coconut coir has not been used properly so coconut coir waste is left alone. Generally, the parts of coconut that are often used are coconut meat and water. Coconut coir is a by-product of coconut fruit which is rich in fiber, coir is usually referred to as waste which is simply piled under coconut stands and then left to rot and dry. The most widely used is for firewood and handicrafts, even though coconut fiber has a useful selling value because it contains cellulose. One way to get cellulose from coconut coir is by extraction. Coconut coir has a percentage of 35% greater than

other components in a coconut fruit. The content contained in coconut fiber consists of cellulose, hemicellulose, and lignin [2]. Numerous methods have been developed to extract cellulose and degrade lignin in coconut fiber. Among them were acid, alkaline, hydrothermal, wet oxidation methods, organ sol ammonia fiber explosion, and the latest ionic liquid pretreatment [3]. Pretreatment of lignocellulose alkaline will selectively remove lignin without degrading carbohydrates. Furthermore, the porosity and surface area of the material can increase so that it will cause enzymatic hydrolysis [4].

In the cellulose extraction process, it is carried out to separate components from other components in a material through an acid or alkaline extraction process, which involves a delignification process that aims to dissolve the lignin content contained in the fiber. The method commonly used in the cellulose extraction process was acid hydrolysis extraction using strong acids such as HCl and H₂SO₄, mainly using shorter reaction times than other processes. In addition, there is also an extraction that involves heat in the process. The existence of heat will speed up the filtering process, and one of them is the reflux method. The reflux was used to extract samples with a rough texture. It can withstand direct heating carried out with a solvent at a certain boiling point, time interval, and amount of heat. The presence of a condenser (coolant). [5]. In this study, coconut coir was extracted using the reflux method using an alkaline solution, NaOH, in the delignification process. The alkali extraction process will produce different cellulose yields depending on the raw materials used [6]. Therefore, this study examines the optimal cellulose content of the cellulose extraction results in coconut fiber using the NaOH alkaline method.

One of the components that make up plant cell walls is cellulose. It has a porous solid material so it can absorb materials around it. Cellulose is divided into three types: alpha-cellulose, beta cellulose, and gamma cellulose as cadmium heavy metal adsorbents [7]. Coconut coir has a moisture content of 5.43%, crude fiber 30.44% [8], cellulose 36.69%, hemicellulose 22.56%, and lignin 42.10% [9]. The initial stage carried out in this coir extraction research was the delignification or pretreatment process, the aim of which was to reduce the levels of lignin content contained in coconut coir so that the cellulose content was easier to obtain [10]. In the delignification process, it functions to facilitate the separation of lignin levels by dissolving the lignin content in the coconut fiber material. [11].

The use of the alkaline delignification method to produce cellulose from coconut fiber is the main focus of this research. This method is the most effective compared to other methods. It is due to the ability to increase the accessibility of cellulose and its ability to remove lignin [12]. Forming cross-links involving xylan and lignin to increase the porosity of lignocellulose. Furthermore, NaOH can also remove the amorphous part of lignocellulose by increasing the internal surface area and degree of crystallinity [13]. Operating parameters such as temperature, NaOH

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concentration, time, and the inherent characteristics of the biomass used can affect the efficiency of pretreatment with alkaline NaOH. This study used the Chesson data analysis method to determine the cellulose content.

2. Methods

2.1. Materials

The main material was coconut coir. The coconut coir was cleaned and dried, then processed with a blender until it became powder. The powdered coconut coir had been sieved into various size particles, which are 60, 80, and 100 meshes. Each was weighed at 7 grams and boiled using distilled water at 100°C for 1 hour. Then it was dried in an oven at a temperature of 45 °C for 24 hours (1 day).

2.2. Delignification and Bleaching Process

Delignification process is a pretreatment process for the bleaching step. This process aims to remove or break the bonds of lignin in cellulose by accelerating the lignocellulose opening its structure so it can be easier to access the cellulose. As much as 5 grams of coconut coir was put into a beaker glass and added with 100 mL alkaline (NaOH) in various concentrations, which are 0.5; 1; and 5 M. The sample that has been soaked with NaOH was put into a three-neck flask for the reflux process with variations in temperature (60; 70; and 80°C) and variations in time (1; 1.5; and 2 hours). Samples that have gone through the delignification process with NaOH were filtered using a vacuum filter then the coconut coir was washed using distilled water until the filtrate was clear.

The delignification process was followed by a bleaching process. This process was carried out to improve color and purity and minimize cellulose degradation in fiber material by removing the levels of lignin. In this research, NaOCl 5% was used as the chemical for the coconut coir bleaching process. The process was carried out at 60°C temperature for 18 minutes. The color will change from dark brown to white, then filter and wash the solids with distilled water until the pH is neutral (reaching 7). The filtrate was dried in an oven at 45°C for 24 hours. Then analyze the content of cellulose, lignin, and hemicellulose using the Chesson Data method [14].

2.3. Analysis and Characterization

To determine the levels of cellulose, lignin, and hemicellulose in this research, the Chesson data method was used [14]. As much as 1.1 grams of the bleached sample was weighed and heated with the addition of 150 ml distilled water. Then, it was filtered and washed with warm distilled water before it dried in the oven at a temperature of 45°C. Afterward, the sample was mixed with 150 ml of 1N H₂SO₄ heated at 100°C for 1 hour, filtered, and washed with distilled water. Then

the coconut coir solids were dried in an oven at 45°C until constant weight. The dried samples were treated with 10 ml 72% for 4 hours. Then mixed with 150 ml H₂SO₄ 1N and refluxed using a three-neck flask at 100°C for 1 hour. After refluxing, the fibers were washed with distilled water and dried in an oven at 45°C. The dried samples were put into the furnace until getting the ash. Then, treated coconut coir was calculated to determine the content of lignocellulosic components using the following calculations:

$$Selulosa\% = \frac{c - d}{a} \times 100\%$$

$$hemiselulosa(\%) = \frac{b - c}{a} \times 100\%$$

Note:

- a. The initial dry weight of coconut coir
- b. Dry weight after heating process with distilled water
- c. Dry weight after heating process with H₂SO₄ 1N
- d. Sample weight after being treated with 72% H₂SO₄
- e. Ash weight of sample

6
3. Results and Discussion

3.1. Cellulose Extraction

The cellulose extraction process consists of delignification and bleaching process. The delignification process uses NaOH as an alkaline solvent which is helpful in dissolving lignin in coconut coir. It supports the separation between lignin and coconut coir. The presence of lignin causes the brown color on the fiber, so it must be treated through a bleaching process. The bleaching process aimed at remnants of lignin compounds to cause color changes by degrading long lignin chains into short lignin chains by adding NaOCl. The bleaching process uses NaOCl, a reactive chemical, to dissolve the remaining lignin in the coconut fiber. The more lignin lost in the coconut coir, the longer the coconut coir lasts on the white color. The bleaching process could increase the purity of the cellulose in the coconut fiber so that the purity of the cellulose content will be higher, and the lignin amount will be in the smallest amount.

In this study, the raw material used as cellulose fiber is coconut coir, carried out through an alkaline delignification process. The delignification process uses NaOH with various concentrations (0.5, 1, and 1.5 M). Figure 1. shows the color difference in the cellulose extraction through the alkaline delignification process and solids through the bleaching process.

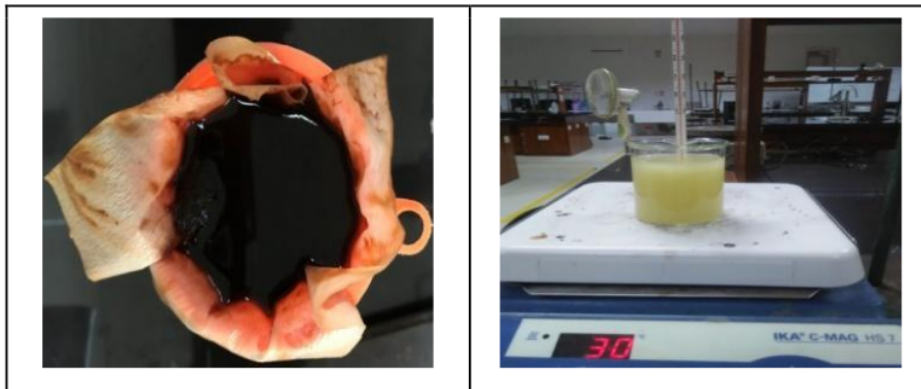


Figure 1. Discoloration of the cellulose extraction after delignification process (left) and bleaching process (right)

The results after delignification with alkaline NaOH pretreatment are dark brown which is affected by the color-causing substances contained in the coconut fiber, namely lignin. These were previously by research of Geng et al (2018), where delignification produces a darker color due to the higher lignin content [15]. Then, the cellulose fiber will be treated in the bleaching process by adding 5% NaOCl at 60 °C. This process caused the fiber's color to change to become white. The NaOCl as the oxidizing agent assists in degrading and removing the brown color from the delignification process. The bleaching process in the cellulose extraction from coconut fiber is affected by several factors. One of them is the temperature of the bleaching chemical. The effective one is in the range of 40-100°C. Furthermore, the reaction time also affects this process, but the cellulose fiber will become more reactive by extending the reaction time. However, if the reaction time is too long, it will cause damage to the cellulose chain in the fiber. Beside of the NaClO₂ temperature exhibited significant impact on the quality of celluloses but also excessive bleaching conditions caused severe oxidation of celluloses and significantly reduced their dimension [16].

3.2 Effect of the particle size on the percentage of cellulose, lignin, and hemicellulose and ash in coconut coir

In this study, various sizes were carried out (60, 80, and 100 mesh) using screening. The delignification process was carried out using NaOH solution because it can damage the lignin structure and separate some hemicelluloses. The factors that affect the delignification process are the sample size, which can affect the porosity so that it affects the delignification process to be carried out [13]. In addition, reducing the sample size will determine the long polymer chains into short ones to facilitate the separation of lignin from cellulose. Figure 2 shows that particle size can affect the degradation process of coconut fiber. The percentage of cellulose in the coconut coir 60,

80, and 100 mesh was 50.00%, 53.63%, and 57.27%, respectively. According to that data, the optimum percentage of cellulose was 57.27 % at 100 mesh because the smaller the particle size, the more cellulose content was obtained. The smaller the size of particles indicated, the broader the surface area. Then, the greater the amount of adsorption can affect the percentage of extraction of cellulose. The smaller the particle size, the bigger potential sample will produce cellulose at higher percentage. So, when carrying out the delignification process, size is one of the main factors that must be considered to produce cellulose in the most optimum conditions [16]. Otherwise, increasing the lignin content that becomes the barrier to the cellulose is getting less. This is influenced because the extraction is carried out by refluxing coconut coir solids through the delignification process using alkaline NaOH.

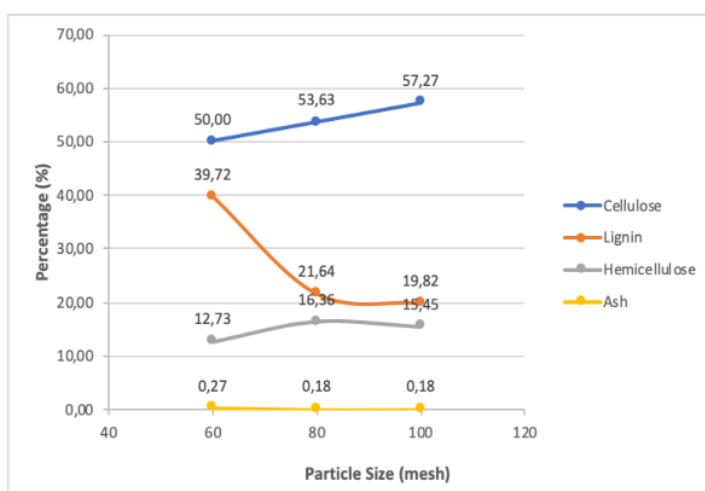


Figure 2. The effect of particle size of coconut coir on the percentage of cellulose, lignin, hemicellulose, and ash.

3.3. Effect of NaOH concentration on the percentage of cellulose, lignin, hemicellulose, and ash in coconut coir

The delignification process was carried out to separate the lignin contained in the coconut coir fiber and to prevent the cellulose from being degraded. The cellulose content from coconut coir was obtained from the delignification, and bleaching process. The delignification process aims to remove lignin content in coconut fiber [15]. The existence of lignin can inhibit the determination process of cellulose. Figure 3 shows the effect of NaOH concentration on the percentage of cellulose, lignin, hemicellulose, and ash. The optimum condition was conducted by adding 1.5 M NaOH solvent with 66.36% cellulose, while the lignin produced was 14.09%. However, at a concentration of 1 M, the cellulose content decreased. Due to the stable open structure of the cellulose, the cellulose molecules were freely dispersed in the solvent (NaOH). High

concentrations of NaOH can dissolve a certain amount of cellulose resulting in a decreased percentage of cellulose [17].

The alkaline in the pretreatment of the delignification process reduces the lignin content in coconut coir. It can increase the cellulose content quickly. The higher the concentration of alkaline in this research was NaOH, the ability of NaOH to reduce lignin levels in coconut fiber increased. Because NaOH acts to separate lignin and some hemicellulose, the percentage of cellulose in coconut fiber can be easier to obtain. Furthermore, the more NaOH is given, the easier the lignin degradation because lignin has a low softening point and melting point. The increase in the cellulose fiber content was caused by some of the lignin dissolved in the delignification process.

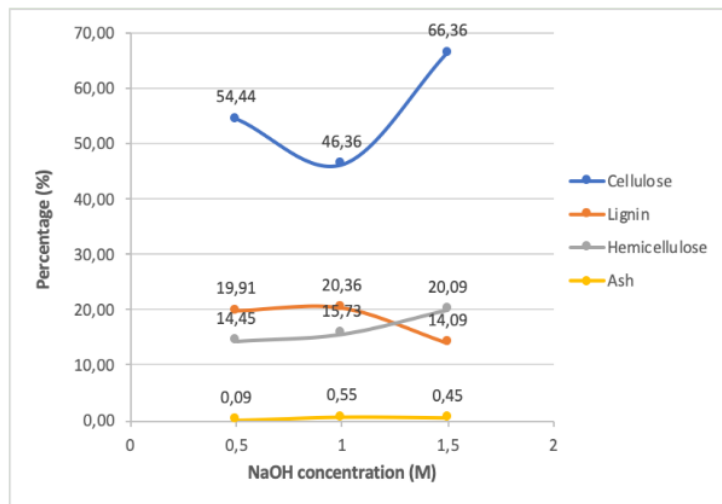


Figure 3. The effect of NaOH concentration of coconut coir on the percentage of cellulose, lignin, hemicellulose, and ash

3.4. Effect of delignification time on the percentage of cellulose, lignin, hemicellulose, and ash in coconut coir

Delignification time was observed due to the optimum delignification process. Variety of time has been carried out on coconut coir samples: 1, 1.5, 2 and hours using 1.5 M NaOH as solvent. Figure 4 shows the effect of delignification time on the percentage of cellulose. It can be seen that the percentage of cellulose obtained decreased in 2 hours. It can be seen that the delignification time is inversely proportional to the results of the cellulose content obtained. At 1 hour delignification time, the cellulose content was 60.00%, and the lignin content was 20.45%. Afterward, it was an increase at 1.5 hours with a cellulose content of 64.55% and lignin content slightly decreased to 11.73%. Then at 2 hours of delignification, there was a decrease in the cellulose content to 60.92%, and the lignin content was obtained by 14.09%.

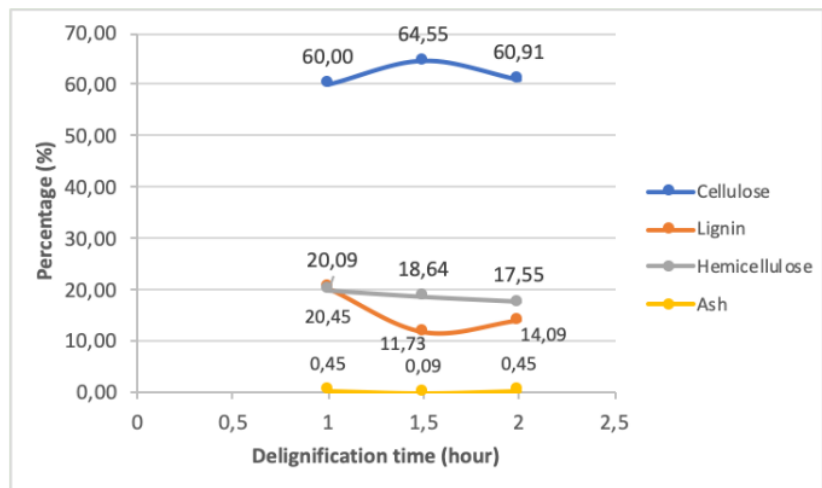


Figure 4. The effect of delignification time of coconut coir on the percentage of cellulose, lignin, hemicellulose, and ash.

The possibility of this can occur because the longer the delignification time will affect more substances that participate and dissolve in the delignification process. Meanwhile, it can affect the cellulose content. As the reaction time increases, the cellulose content will increase until a specific time when cellulose will be obtained at maximum. The contact between the reacting substances can be more prolonged. However, if the time is extended, the growth of cellulose content will decrease. If the reaction time is growing, there will be a breakdown in the cellulose chain. It was affected a lot of the cellulose content will be lost. So, the optimum cellulose content in the delignification process was 1.5 hours.

3.5. Effect of delignification temperature on the percentage of cellulose, lignin, hemicellulose, and ash in coconut coir

Affecting the temperature on the pretreatment process indicates an increasing the content and weight of cellulose while increasing the certain temperature. At high temperatures, it will provide greater energy for the reaction so that the bond-breaking reactions in the lignin and hemicellulose chains go well so that more cellulose bonds can be liberated. Figure 5 shows the effect of varying temperatures at 60, 70, and 80 °C on the percentage of cellulose, lignin, hemicellulose, and ash in coconut coir. It can be seen that the temperature increase is gradual.

The higher the temperature during the cooking process, the higher the cellulose content in the coconut fiber produced. The highest cellulose was obtained at 80°C, which was 67.64%. The lowest cellulose was obtained at 60°C, which was 57.53%. Figure 5 shows that the lignin content in coconut fiber will decrease when the cooking temperature is higher. The increase in temperature

increases the ability of the delignification process of the cooking solution so that more lignin has been dissolved. The lowest lignin was found at 80°C, which was 14.55%. It follows the data obtained that the greater temperature used for the cooking process in the delignification process, the greater the cellulose obtained. On the other hand, the higher the temperature, the lower the lignin content obtained.

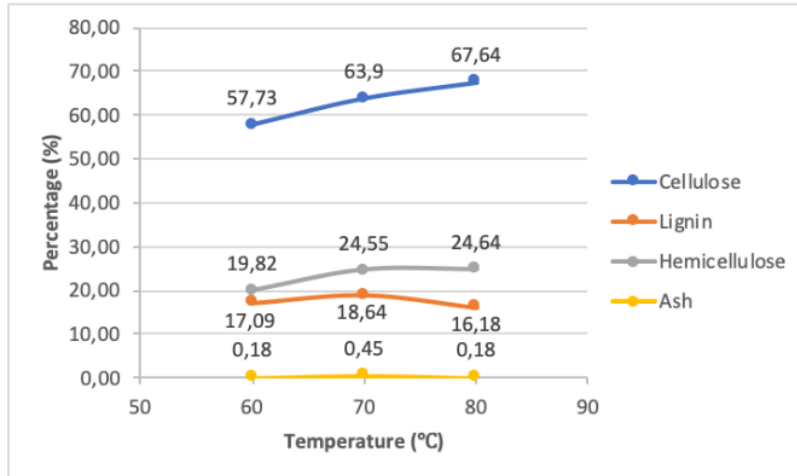


Figure 5. Effect of delignification temperature on the percentage of cellulose, lignin, hemicellulose, and ash in coconut coir

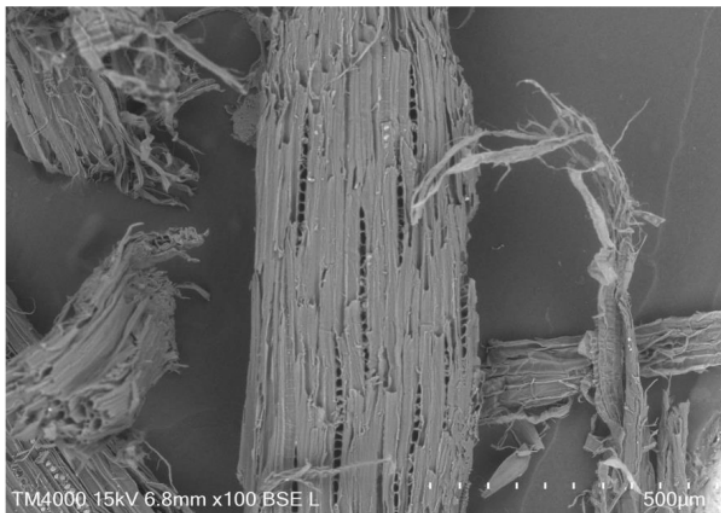


Figure 6. SEM Photo morphology of cellulose from coconut coir.

3.6 Characterization morphology of cellulose

This study showed that the optimum percentage of cellulose was obtained at 69.82%. The most optimum particle size variable is 100 mesh. The smaller the particle size, the greater the

cellulose content obtained. At the NaOH concentration, the optimum result is 1.5 M. Because the higher the NaOH concentration, the more lignin is degraded, so more cellulose is separated from the lignin bonds. High temperatures will provide greater energy for the reaction so that the bond-breaking reaction in the lignin and hemicellulose chains goes well so that more cellulose bonds can be liberated. It is in accordance with the results obtained in the delignification process at an optimum temperature of 80°C and a delignification time of 1.5 hours. Figure 6. shows the morphology of cellulose from coconut coir. The delignification process and activation of NaOH affected the inside fiber's opening. Additionally, the large pores were acquired, torn, and crumbled, so the surface became rough and perforated.

4. Conclusions

Cellulose can be extracted from coconut fiber by the alkaline delignification method. Several factors influencing cellulose extraction in the alkaline delignification process using NaOH are particle size, NaOH concentration, temperature, and delignification time. The optimum cellulose content obtained at 100 mesh coir particle size, 1.5 M NaOH concentration, at 80°C for 1.5 hours was 69.82 %. The alkaline delignification method has the potential as a simple and effective method for extracting cellulose from natural materials, especially biomass.

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