The Fabrication of Cellulose Acetate Fiber based on Empty Fruit Bunches (EFB) using Electrospinning Technique

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ABSTRACT

The cellulose acetate fiber is widely applied in various fields. This study elaborates the fabrication of cellulose acetate fiber based on cellulose acetate is synthesized from cellulose contained in empty fruit bunches (EFB). The cellulose extraction process is carried out through the following five stages: preparation, acid hydrolysis, delignification, pulping, and bleaching. The second step of the synthesis process of cellulose acetate is carried out through three stages, including activation, acetylation, and hydrolysis. Then, the next step of fabricating cellulose acetate fiber was carried out by using the electrospinning method by varying the distance from the tip of the needle to the collector of 9, 11, and 13 cm. The cellulose and cellulose acetate were characterized using FTIR, SEM, examining its water and cellulose content. The cellulose acetate fiber was characterized using FTIR, optical microscope, examined for acetyl content, and degree of substitution. The average content of α-cellulose obtained from this study was 95.33%. SEM micrograph shows that after the extraction process, it was seen that the surface of the fiber tended to wrinkle due to the loss of waxy layer on its surface area. These indicate that the extraction process was succeeded in removing most of the hemicellulose, lignin, and pectin content. The yield of cellulose acetate obtained from this study was 405.4% (yield of 100% immersion will produce 8.47 g of cellulose acetate). The average diameter of the fiber with the needle tip distance to the collector of 9, 11, and 13 cm, are 1.428, 1.271, and 1.194 m, respectively.

1. INTRODUCTION

Cellulose acetate is a natural polymer derived from cellulose which has a microfibril structure and excellent solubility in various types of non-polar solvents. Cellulose acetate has unique properties such as easy to decompose, so that this material becomes an environmentally friendly material, has a good level of transparency and high tensile strength, is resistant to heat, has low water absorption, and low fabrication costs. These properties make cellulose acetate widely applied in various fields, such as water sensors [1] in [EMIM]OAc, which was dry–wet spun into water. The cladding layer on the cellulose core was produced by coating a layer of cellulose acetate, dissolved in acetone, using a filament coater. The chemical and optical properties of both regenerated cellulose and cellulose acetate were studied from cast films using ultraviolet–visible and Fourier-transform infrared spectroscopy measurements. Regenerated cellulose film was

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observed to absorb UV light, passing the visible light wavelengths. Cellulose acetate film was observed to pass the whole light wavelength range. The mechanical strength and topography of the prepared optical cellulose fiber were investigated through tensile testing and SEM imaging. The mechanical performance of the fiber was similar to previously reported values in the literature (tensile strength of 120 MPa, bioplastic [2], and drug delivery systems [3]. The use of cellulose acetate in various fields uses fiber as the basic material.

One of the common techniques used to produce cellulose acetate fiber is electrospinning [4]. This method has advantages in producing fiber, among others such as the production process is relatively fast, simple, and the cost is affordable. The quality of the cellulose acetate fiber produced is influenced by three main factors, namely; solution characteristics, control variables (voltage, distance from spinneret tip to collector, flow rate, and needle tip design), and environmental factors (temperature, humidity, air velocity) [5].

Lee *et al*. [6]i.e., the composition of the solvent; the concentration of the polymer; and the solubility of the solvent and the polymer. Also, we incorporated polyvinylpyrrolidone (PVP have studied the effect of solvent composition, polymer concentration and solvent solubility mixed with polymer on the morphology of cellulose acetate nanofiber. Cellulose acetate nanofiber has also been successfully fabricated by Ertas *et al.* [7] polybenzoxazine based cross-linked cellulose acetate nanofibrous membrane exhibiting enhanced thermal/mechanical properties and improved adsorption efficiency was successfully produced via electrospinning and thermal curing. Initially, suitable solution composition was determined by varying the amount of the benzoxazine (BA-a by varying the amount of benzoxazine resin (BA-a), cellulose acetate (CA) and citric acid (CTR) to obtain a uniform nanofibrous membrane through electrospinning for water treatment applications. One of the strategies for low cost of cellulose nanofiber production focuses on chemical-assisted treatments, including acid hydrolysis with H_2SO_4 and cellulase pretreatment. These methods enable fibrillation by cleaving the cellulose chains in the amorphous regions. Nevertheless, the yields based on cellulose of acid hydrolysis with H_2SO_4 are low at only 25–35% [8]. A number of these studies used commercial cellulose acetate as the basic material to produce cellulose acetate fiber. The use of cellulose acetate produced from less-than-optimal utilization of biomass such as empty fruit bunches (EFB) as the main material for the fabrication of cellulose acetate nanofibers is interesting to analyze further. This is due to the EFB contains large amounts of cellulose about 40 to 60% [9]. In the current study, we explore the potential of cellulose acetate fiber based on cellulose acetate synthesized from cellulose contained from EFB in a new and important usage mask application. The distance between the tip of the needle and the collector will be varied in this study to produce optimal cellulose acetate nanofiber.

2. EXPERIMENTAL SECTION

2.1 Materials

Raw materials EFB from PT. Swadaya Indopalma, Palembang, Sumatera Selatan, Nitric acid (HNO_3, Merck), Sodium Hydroxide (NaOH, Merck), Sodium Sulfite (Na_2SO_3, Merck), Hydrogen Peroxide (H_2O_2, Merck), Acetic Acid (CH_3COOH, Merck), Sulfuric Acid (H_2SO_4, Merck), Acetic Anhydride, Ethanol, Hydrochloric Acid (HCL, Merck), Potassium dichromate, Acetone, and Dimetil Asetamida (DMAC, Merck).

2.2 Methods

2.2.1 Cellulose Extraction

The cellulose extraction process is carried out through five stages, namely preparation, acid hydrolysis, delignification, pulping, and bleaching. The preparation process is carried out by cleaning the EFB to remove the remaining dirt and oil. Then, the EFB are dried using sun heat radiation for 4 hours. The average value of the EFB water content obtained is 8.933%. After this process, the water content analysis will be carried out. The next process is acid hydrolysis by adding 1000 ml of 3.5% HNO_3 (v/v) into 75 grams of dry EFB and then heating at 90 °C for 2 hours [10]. The sample was then filtered, and the dregs were

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washed using aqua DM and pure water until the pH was neutral and dried at 100 °C for 3 hours. The acid hydrolyzed pulp was then delignified by adding 200 ml of NaOH 2% (w/v) and 2% Na_2SO_3 (w/v) each and then heated at 50 °C for 1 hour. The sample was filtered again, and the dregs were washed until the pH was neutral and dried using an oven at 100 °C for 3 hours. The dregs from the delignification were given the pulping treatment. The pulping process was carried out by adding 300 ml of 17.5% (w/v) NaOH into the pulp and then heated at a temperature of 80 °C for 3 hours. After completing this process, the sample is dried, and the dregs are washed until the pH is neutral. The dregs were dried at a temperature of 100 °C for 3 hours. The last process is bleaching using H_2O_2 10% (v/w) as much as 300 ml which is added to the pulp resulting from the pulping process. This process takes place at a temperature of 60 °C for 15 minutes. The results obtained through this process will then be treated with FTIR (Thermo scientific Nicolet iS-10) characterization, morphology using SEM (JEOL JSM-6510), examining the water and cellulose content.

2.2.2 Determination of Moisture Content (MC)

Moisture content was determined by using the following equation;

$$MC=\frac{m_1-m_2}{m_1}x100\% \quad (1)$$

Where MC is the moisture content of the sample, m_1 is the dry mass of the sample, and m_2 is the real-time mass of the sample at a set relative humidity (RH) [11].

2.2.3 Determination of Yield of Cellulose

The yield of cellulose (%) obtained was calculated by Equation (2):

$$Yield=\frac{\text{Weight cellulose}}{Weight\ EFB}x100\% \quad (2)$$

2.2.4 Determination of Cellulose Content (α-cellulose)

20 mL of (17,5% concentration) NaOH solutions added to 1 g of cellulose, and the mixture for 15 minutes. After that, the samples were added with 25 ml of aqua DM and then mixed for 1 minute. After this process was completed, the samples were filtered and then washed. The residue was added to 40 ml of CH_3COOH 10% and leave it for 5 minutes. The result of this process is filtered and the residue is dried at 105 °C. The cellulose content was calculated according to equation (3):

$$\alpha\text{-cellulose}\ (\%)=\frac{W_2-W_0}{(1-MC)W_1}\ x\ 100\% \quad (3),$$

where W_0 is the initial weight of material, W_1 is added weight with component W_0 as initial weight of material, W_2 as weight after the sample has been dried in oven, and MC is moisture content.

2.2.5 Cellulose Acetate Extraction

The synthesis process of cellulose acetate is carried out through 3 stages, namely, activation, acetylation, and hydrolysis. The activation process was carried out using 50 ml of CH_3COOH 100% (v/w) to which H_2SO_4 was added. The solution mixture was put into 10 grams of extracted cellulose, then stirred until homogeneous and let it rest for one hour at room temperature. After that, the samples were added $(CH_3CO)_2O$ 100% (v/w) and CH_3COOH 100% (v/w) respectively as much as 50 ml and 20 ml and then heated for thirty minutes at a temperature of 50 °C. After this process was completed, the sample was added with 100 ml of 70% CH_3COOH (v/w) which had been added with concentrated H_2SO_4, then stirred at 350 rpm at 50 °C for 3 hours. The filtrate formed from this process was then given 50 ml of aqua DM gradually while stirring. A white precipitate will begin to form, then 500 ml of aqua DM is added. The result of this process is filtered and the residue is washed to reach the neutral pH and then dried at 55 °C for 24 hours. The samples produced from this process were characterized using FTIR (Thermo scientific

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Nicolet iS-10), examined for moisture content, acetyl content, and degree of substitution.

2.2.6 The Fabrication of Cellulose Acetate Fiber

The process of fabricating cellulose acetate fiber using the electrospinning (Bio ES 02) method begins with dissolving 15% (w/v) cellulose acetate using acetone and dimethylacetamide in a ratio of 2:1 (v/v) while stirring it until homogeneous. The homogeneous cellulose acetate solution was put into a 10 ml syringe and then placed on top of the syringe pump. The solution was flowed into the spinneret at a flow rate of 1.5 L/h. The metal tip of the spinneret is then connected to the positive pole of a high-voltage power source. The mica-coated aluminum foil is connected to the negative pole which is used as a fiber collector formed during the electrospinning process. This process is carried out by varying the distance from the tip of the needle to the collector at 9, 11, and 13 cm with an electric voltage of 10 kV at room temperature. The resulting cellulose acetate fiber was then treated in the form of characterization using an optical microscope (Meiji) to see the morphological structure of the sample.

3. RESULT AND DISCUSSION

3.1 Characteristic of Cellulose

Figure 1 shows the photograph of cellulose extracted from EFB which were previously carried out by 5 stages (preparation, hydrolysis, delignification, pulping and bleaching). Those processes aimed to remove the lignin content that remains in the fiber of EFB. The yield of cellulose produced in this study was about 32.89%, this result was slightly lower than that conducted by Dewanti *et al*.[12], which was about 34%. This can be explained due to the obtained low level of cellulose purity that was not well treated by 17.5% (v/w) NaOH. Therefore, this causes the obtained cellulose still contains other types of β-cellulose dan γ-cellulose**.**

Figure 1. The photograph of Cellulose Extracted from EFB

The cellulose produced from this study contains a moisture content of 5.6%. This can be achieved due to the higher temperature used by Bahmid during the drying, thus it accelerates better water evaporation process [13]. The moisture content is important in the cellulose acetate synthesis due to the hydroxyl bonds in cellulose are tend to easily bind O-H in water than acetyl groups. However, the average water content in the range of 4-7% is considered to follow the standard of the average for the cellulose acetate synthesis process. Therefore, low water content is

Table 1. The results of calculates average content of α-cellulose

W_1 (g)	W_2 (g)	W_3 (g)	M (%)	-Cellulose (%)	Average (%)	Standard Deviation	%Error
36.42	1.0	37.33	5.6	96.39	95.33	0.86	0.61
36.42	1.0	37.32		95.33			
36.42	1.0	37.31		94.27			

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expected to facilitate the fiber substitution process properly. In addition, the acetylation process is a reversible reaction, so that high water content can cause the cellulose acetate formed to return to cellulose. The results of calculated average content of α-cellulose are shown in Table 1. It obtained from this study was about 95.33%.

This high value of α-cellulose content indicates that the cellulose obtained has a high level of purity so that it can be used as a raw material for the synthesis of cellulose acetate. The high of α-cellulose in this work was caused by the use of proper sodium hydroxide during the soaking process and made it easier to reduce the hemicellulose in EFB.

Figure 2. shows the results of functional group analysis of EFB before and after extraction (cellulose) process.

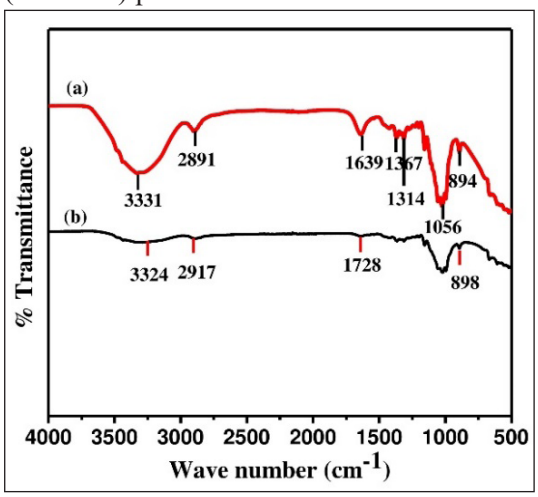

Figure 2. FTIR spectra of: (a) EFB, (b) Cellulose

The FTIR plot of raw EFB after the extraction process (Fig. 2a) shows an increase in absorption at a wave number of 3331 cm-1, which is the O-H bond that forms cellulose. This finding is supported by the results of a study conducted by Salim et al. [14], that found stretching vibrations of the O-H group at a wave number of 3000 – 3750 cm-1. Another study conducted by Furthermore, there was an increase in absorption at a wave number of 2891 cm-1, which was identified by C–H stretching vibration. Salim et al. [14] also showed the presence of C–H stretching vibration at a wave number of 2899 cm-1. Wang et al. [15] have succeeded in showing the formation of cellulose which was confirmed by the peak appearance at a wave number of 2900 cm-1 that corresponds to the C–H stretching vibration. The peak formed at the wave number of 1639 cm-1 was identified as the water adsorption area. These results are also confirmed by the results of research conducted by Guo et al. [16] and Salim et al. [14] for each sample of mulberry bark and Leucaena leucocephala bark, respectively. Peaks with low intensity were formed at wave numbers 1367 cm-1 and 1314 cm-1, which corresponded to the CH3 groups of cellulose and C-O stretching vibration, respectively [17,18]. The peak with a wave number of 894 cm-1 shown by the FTIR graph on the sample after extraction is known as C-H rocking vibrations from the cellulose. These results are supported by the results from Salim et al. [12]. Thus, based on the FTIR results, it was shown that cellulose was successfully extracted from EFB. However, there is still a small amount of lignin residue, which is indicated by the appearance of a peak at the wave number of 1056 cm-1 that corresponds to guaiacyl type units.

Figure 3. shows the SEM micrograph of EFB before and after extraction (cellulose) process. The SEM of EFB (Fig. 3a) shows that the fibers are still bonded to each other, and some smooth and flat surface is formed due to the fiber surface is coated with wax. After the extraction process, the morphology of cellulose (Fig. 3b) was more wrinkled due to the loss of the waxy layer on the fiber surface. These results indicate that the extraction process succeeded in removing most of the hemicellulose, lignin and pectin.

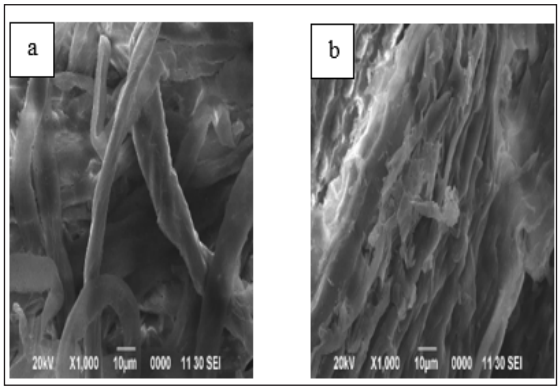

Figure 3. SEM micrograph (1000x) of: (a) EFB and (b) Cellulose

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3.2 Characteristics of Cellulose Acetate

Figure 4. shows the photograph of cellulose acetate from cellulose which was previously carried out through three stages (activation, acetylation, and hydrolysis).

Figure 4. Photograph of Cellulose Acetate

The number of cellulose acetate that we obtained from this work was 34.34 g which was obtained from yield of 405.4% (yield of 100% immersion will produce 8.47 g of cellulose acetate). The acetylation process causes the hydroxyl group (O-H) to be replaced with an acetyl group (CH3CO) which has a heavier density. This causes an increase in the final weight value of the cellulose acetate produced. In addition, the increase in the final weight of cellulose acetate is influenced by the hygroscopic nature of cellulose acetate. The moisture content of cellulose acetate obtained from this study was about 65.66%. The moisture content of cellulose acetate is very high because it is used for not optimal drying the production process. Meanwhile, cellulose acetate produced from this study has an acetyl content of 38.77% and a substitution degree of 2.34.

The absorption range of stretching O-H groups on the cellulose spectrum appears in the absorption region between 3000 - 3750 cm-1. The results of the FTIR analysis showed that the cellulose spectrum before the acetylation process had stretching O-H functional groups that appeared at around 3331 cm-1 and after the acetylation process had stretched O-H functional groups that appeared at around 3394 cm-1. This is because the intensity of the absorption peak of the hydroxyl group decreases while the intensity of the absorption peak of the acetyl group increases.

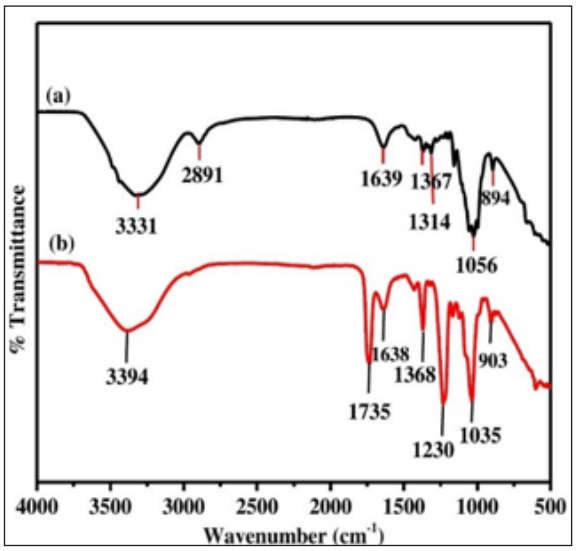

Figure 5. FTIR spectra of: (a) Cellulose and (b) Cellulose Acetate

The results obtained are in accordance with the research that has been done previously by He et al. [19].

The results of functional group analysis using FTIR also showed an absorption peak of the carbonyl group C=O (1870 - 1540 cm-1) and the C-O ester group from the acetyl group (1320 - 1210 cm-1) at wave numbers 1735 cm-1 and 1219 cm-1. This indicates the formation of cellulose acetate compounds through a sharp peak at a wave number of 1735 cm-1 and a decrease in the intensity of the O-H group due to being substituted by acetyl group. Cellulose with a high level of purity can produce cellulose acetate with good quality. The purity level of cellulose is indicated by the high value of α-cellulose and the presence of a distinctive peak in the proper IR spectrum of cellulose.

3.3 Morphological Properties and Diameter of Cellulose Acetate Fiber

Figure 6 shows an optical micrograph of cellulose acetate fiber given the variation treatment of electrospinning in the distance between the tip of the needle to the collector about 9, 11, and 13 cm. Based on the picture, it can be seen that the cellulose acetate fiber has been successfully formed. However, the resulting fiber is not much. This is possibly influenced by the produced high-water content of the cellulose acetate. Meanwhile

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based on the calculation, the obtained average diameter of the cellulose acetate fiber are 1.428; 1.271; and 1.194 μm for the needle tip distance to the collector at 9, 11, and 13 cm, respectively.

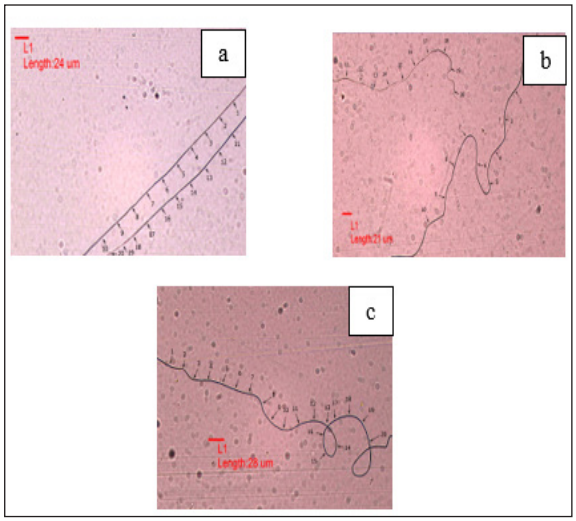

Figure 6. Optical Microscope photograph (550x) of Cellulose Acetate Fiber by the variation treatment in the distance between the tip of the needle to the collector about (a) 9, (b) 11, and (c) 13 cm.

These results indicate that the farther the distance between the needle tip to the collector, the smaller the diameter of the fiber size. This happens due to the distance between the needle tip to the collector affects the evaporation process of the cellulose acetate solution. The solution will have a longer time to evaporate at longer distances, thus it will provide an increased elongation process and can produce thinner fibers.

4. CONCLUSION

Cellulose acetate fiber has been successfully synthesized from raw EFB. The cellulose acetate fiber fabrication process starts from the extraction of cellulose from raw EFB. The average content of α-cellulose obtained from this study was 95.33%. This high value of α-cellulose content indicates that the cellulose obtained has a high level of purity so that it can be used as raw material for the synthesis of cellulose acetate. Based on SEM micrograph, after the extraction process, some wrinkled fiber surface was shown due to the loss of the waxy layer. These results indicate that the extraction process succeeded in removing most of the hemicellulose, lignin and pectin. The number of cellulose acetate that we obtained from this work was 34.34 g which was obtained from yield of 405.4% (yield of 100% immersion will produce 8.47 g of cellulose acetate). Based on optical micrograph, the cellulose acetate fiber has been successfully formed. However, the resulting fiber is not much. This is possibly influenced by the high-water content of the produced cellulose acetate. The average diameter of the fibers are 1.428; 1.271; and 1.194 μm at the needle tip distance to the collector at 9, 11, and 13 cm, respectively.

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