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Micro-Fibrillated Cellulose Fabrication from Empty Fruit Bunches of Oil Palm

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Abstract. Micro-fibrillated celluloses (MFCs) are made from oil palm empty fruit bunches (EFB). EFB is processed through several stages of the process, including washing, alkalization, and bleaching to remove impurities, lignin, and hemicellulose. Each treatment stage was characterized by differential scanning calorimeter (DSC) and thermogravimetric (TGA) analysis. Morphological analysis was characterized using Scanning Electron Microscope (SEM). The process results show that MFC has an average length and thickness of 450 and 80 microns for coarse fibers respectively, averaging 50 and 5 microns for fine fibers, respectively. Fibrillation fibers appear on the surface of fibers which are treated using alkalization and bleaching processes. The TGA results showed a decrease in weight occurred at a temperature of 40 to 109 °C for the first stage of the heating process and at a temperature of 247 to 382 °C for the second stage. The decrease in fiber weight is caused by evaporation of water content and degradation of cellulose compounds at each stage. The glass transition temperature of MFC was obtained at 236 °C. The thermal stability of cellulose from fibers treated using alkalization and bleaching processes proved the formation of cellulose crystals. Removal of lignin and hemicellulose is shown by the absorption of O-H and C-C bonds in FTIR spectroscopy. From these results, it is stated that micro-fibrillation cellulose is formed well through a series of processes given.

Introduction

Cellulose is one of the most important renewable materials for human life. Cellulose is crystalline, hard fibrous which is widely used in the textile, paper, sanitary wares, and packaging industries [1]. Cellulose-based materials can be used with the presence or absence of lignin and hemicellulose.

At present, cellulose had been produced using mechanical [2], Biological [3], and chemical method [4]. Chemical methods prefer to be used in cellulose fabrication because the product yields are higher than mechanical methods that require much energy. Cellulose production using biological methods is less preferred because of the long process. Making cellulose by chemical methods can eliminate lignin and hemicellulose quickly so that the fibrillation process runs faster to cellulose fibrillation. The fibrillation process on cellulose through this chemical method can also provide different characteristics and is better for cellulose fibrillation produced.

Microfibril cellulose generally has a fibrous cellulose structure as thick as 2-10 nm with a length of several tens of microns, varying depending on the origin [5]. Usually, MFC consists of aggregates of cellulose microfibrils [6]. Cellulose microfibrils undergo transverse division along the amorphous region due to treatment, both mechanical, biological, and/or chemical. This transverse division makes the MFC aggregate have strong bonds between the components [7].

Indonesia is one of the largest palm oil producers in the world. The palm oil industry produces waste, one of which is oil palm empty bunches (EFB). According to Pattanamanee et al., [8], EFB waste produced globally reaches 40 million tons. So far, EFB is only used for fertilizers because it has high organic and inorganic content [9]. In addition, EFB is also used as fuel through an incineration process [10]. Seeing this, the utilization of EFB waste is still not high. EFB contains much fiber has excellent potential to be developed into micro-fibrillated cellulose material, which has a higher economic value than as a raw material for fertilizer and combustion processes.

In addition to EFB, several factories have developed as raw materials to make MFC. Yuanita, et al. [11] investigated the manufacture of MFCs from palm oil fibers. Kumar et al. [12] have researched the production of MFC from bagasse and straw fiber. Meanwhile, Ismojo et al. [13] have made MFC using sorghum raw materials. Therefore, the above has reinforced the estimate that EFB can be used in making MFC.

In this study, MFC was made from EFB fibers through several stages, including the alkalization and bleaching process. The alkalization process is used to break down lignin and hemicellulose and to remove impurities. This process also aims to increase resistance to water so that the fiber has hydrophobic properties. After the alkalization process, the fiber goes through a bleaching process which aims to (i) whiten the color of MFC and (ii) fibrillate the surface of the cellulose fiber. Scanning Electron Microscopy (SEM) was used to examine EFB fiber morphology before and after treatment and to prove the formation of MFC. Thermogravimetric (TGA) and Differential Scanning Calorimeter (DSC) analyzes were carried out to prove cellulose crystal formation and removal of lignin and hemicellulose as impurities, as well as Fourier-transform infrared spectroscopy (FTIR) analysis.

Materials and Method

Materials. Empty fruit bunches of oil palm waste were obtained from an oil palm plantations in Ciseeng, West Java, Indonesia. Sodium hydroxide and hydrogen peroxide were purchased from Merck (Darmstadt, Germany).

Micro-fibrillated Cellulose Fabrication. Before use, oil palm empty bunches fibers were dried under the sun (29-32 °C) for three days. After drying, the fiber was broken down and crushed using a crusher then filtered with a 40 mesh sieve until the average size was 3 mm. The alkalization process was carried out by immersing 1 g of crushed fiber in 35 mL of 4% sodium hydroxide solution at 70 °C for 3 hours while continuing to stir for the product to be homogeneous. The bleaching process was carried out by dipping the alkaline fiber into hydrogen peroxide at room temperature for two hours, and then the fiber was dried under 50 °C overnight to produce micro-fibrillation cellulose.

Characterization

Scanning Electron Microscopy (SEM). SEM was used to analyze the morphology of MFC, which was formed after treatment with alkalization and bleaching. SEM micrographs were taken using a Field-Emission Scanning Electron Microscope (FE-SEM) FEI Inspect F50 (Oregon, USA). The sample was prepared by dispersing dry MFC powder on a two-sided conductive adhesive tape and coated with carbon to make it more conductive.

Thermal Stability Analysis. Simultaneous Thermal Analysis STA-6000 from Perkin Elmer (Massachusetts, USA) was used for DSC-TGA analysis simultaneously of the initial fiber, after alkalization, and after bleaching, under nitrogen flow at a rate of 10 mL/min. It was calibrated against melting points and fusion enthalpies of indium and silver. Samples (9-10 mg) were heated from 40 to 500 °C at a rate of 10 °C/min.

Fourier Transform Infra-Red (FTIR) Spectroscopy. Spectrum Two™ Infrared Spectrometer Perkin Elmer (Massachusetts, USA) was used to determine the chemical bonding of treated EFB micro-fibrillated cellulose.

Results and Discussion

Morphology Analysis. Figure 1 shows the micrographs of the fibers before processing and after being processed through alkalization and bleaching. In untreated fibers, the fiber surface was relatively smooth, and some impurities are still found. The average fiber length was around 500 microns for both crude fiber and fine fiber. Whereas in fibers treated with alkalization and bleaching to form MFC, the surface of the fiber looks different where cellulose fibers are clearly visible on crude fibers. Lignin and hemicellulose then dissolved during the alkalizing and bleaching process. Coarse fiber had an average length of 400 microns, and fine fibers had a length between 50-100 microns. This proves that fiber treatment using alkalization and bleaching can reduce fiber size.

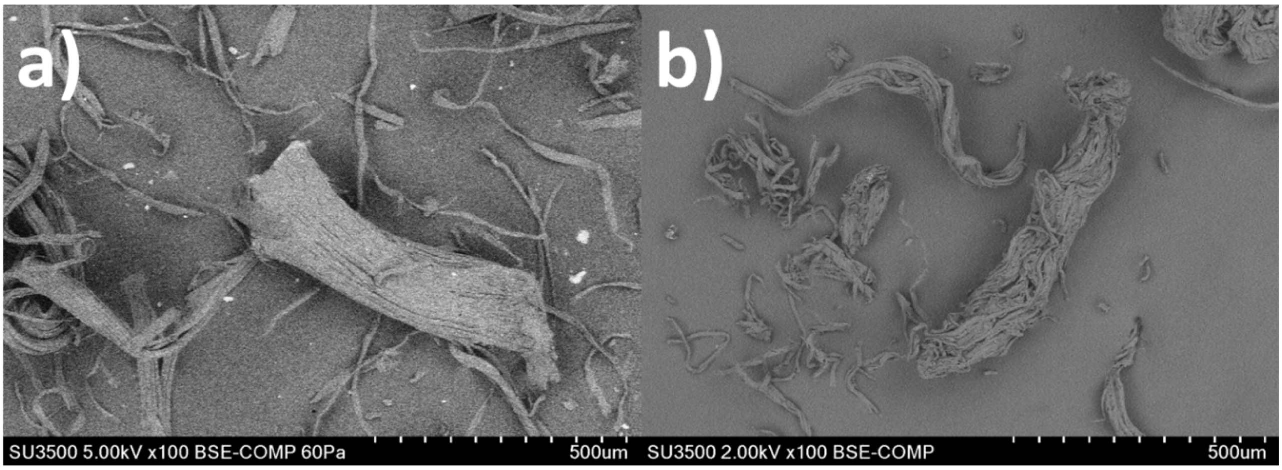

Figure 1. SEM images of a) un-treated fiber and b) fiber which treated using alkalization and bleaching process.

Thermal Stability Analysis. Investigating the thermal properties of each treatment process in MFC production from oil palm empty bunches is essential to do. Figure 2a shows the TGA for fiber samples (i) before treatment, (ii) after alkalization, and (iii) after bleaching. All TGA curves show a decrease in weight in two stages. In the fiber sample before treatment, the weight loss from 40 to 136 °C (first stage) indicating the drying process that causes evaporation of water contained in the sample. Then the weight loss dramatically at temperatures around 220-347 °C (the second stage) due to the degradation process of the components such as cellulose, hemicellulose, lignin at the same time. In fiber samples after the alkalization process, the first stage of weight loss occurred from 40 °C until the temperature reached 116 °C, and the second stage occurred at 237-357 °C. Whereas in the fiber sample after the bleaching process, the first stage of weight loss occurred until temperature achieved 109 °C and the second stage at 247-382 °C. The increasing decomposition temperature after the alkalization and bleaching process was caused by the crystallization of cellulose, which has better thermal stability than the initial fiber [14,15]. The crystalline of cellulose causes fibers through the process of treatment (alkalization and bleaching) to have high thermal stability compared to alkalinized fiber, and fiber without treatment [16].

The DSC thermograms from heating fiber samples (i) before treatment, (ii) after alkalization, and (iii) after bleaching are shown in Figure 2b. Thermograms of fibers after alkalization process had Tg of 238 °C, higher than fibers without treatment and fiber after the bleaching process which had

Tg around 235 and 236 °C, respectively. All thermograms had two melting peaks. In general, the melting peak occurs due to melting and recrystallization process of the crystalline phenomenon during the heating process [17]. In the initial fiber (without treatment), the second peak appeared around 340 °C. In the fiber after the alkalization process and after bleaching, the second peak appeared when the temperature was 353 and 369 °C, respectively. The interaction between cellulose crystals can slow down the melting process of cellulose.

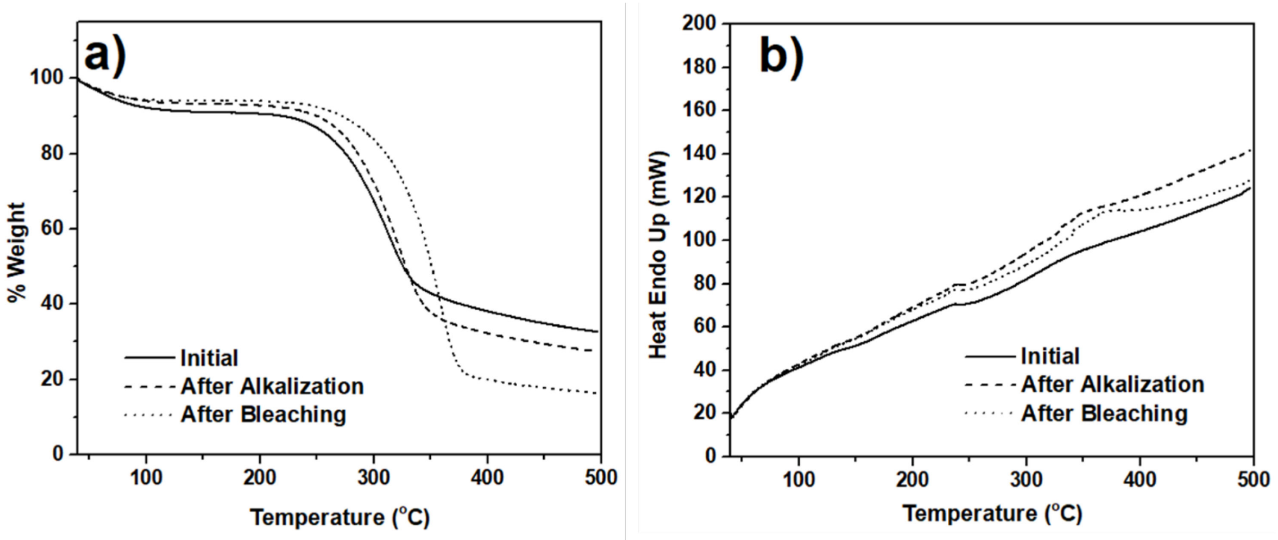

Figure 2. a) TGA and b) DSC curves of MFC samples; the heating rate of 10 °C/min.

FTIR Analysis. The width of the relative absorbance peak at 2895.88 cm^{-1} occurs because of the presence of CH_2 groups in the MFC sample [18]. This band also represent the crystallinity degree of cellulose. While high and sharp peak intensity at 1029.24 cm^{-1} showed an increase in cellulose content which has a skeletal ring C-O-C pyranose. The peak at 892.60 cm^{-1} was small but sharp, representing the glycosidic deformation of C_1-H, which was related to β-glycoside bond between anhydroglucose units in cellulose [19].

A prominent peak at 1639.08 cm^{-1} seen in the spectrum illustrates the presence of esters of the carboxylic and p-coumaric groups or acetyl groups and uronate esters of lignin and/or hemicellulose [20]. The peak at 1510.02 cm^{-1} represented the aromatic C-C stretch of the lignin aromatic ring [21]. The small absorbance at 1639.08 cm^{-1} and about 1510.02 cm^{-1} showed that hemicellulose, pectin, and lignin in oil palm empty bunches fibers were effectively removed by leaving cellulose as the main component. The peaks at 3334.49 cm^{-1} were related to O-H bonds which shows the degradation of hydrogen bonds between lignin and carbohydrates or with cellulose.

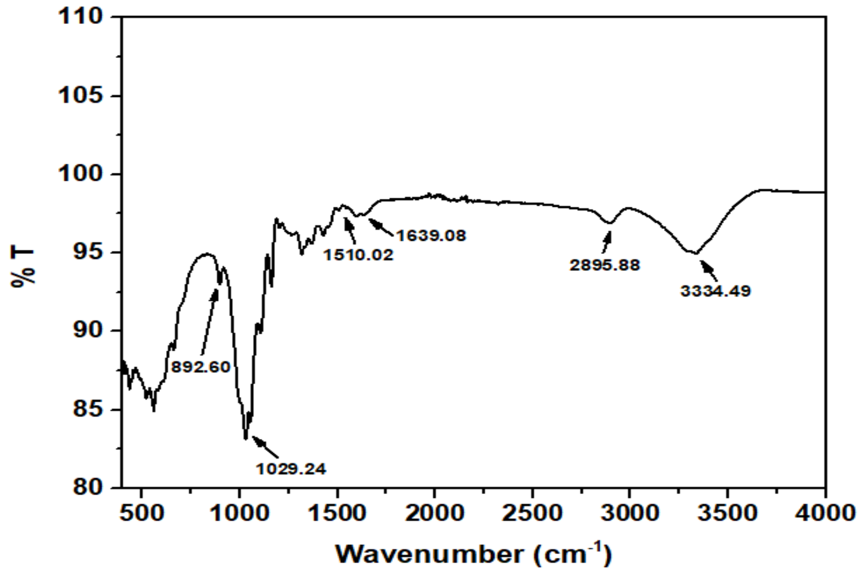

Figure 3. FTIR spectra of cellulose sample after alkalization and bleaching process.

Summary

Chemical treatments such as alkalization and bleaching process had been carried out for the fabrication of micro-fibrillated cellulose from oil palm empty bunches. The morphology of the fiber changes drastically after undergoing an alkalization and bleaching process in which lignin and hemicellulose are removed and make fibrillated fibers clear. Fibers treated with alkalization and bleaching have better thermal stability than the initial fibers without treatment, and an alkalization treatment with degradation temperature was 247-382 °C. This proves the presence of cellulose crystals will slow down the decomposition of fiber material. Two peaks were also seen at temperatures 236 and 369 °C during DSC analysis, also proved the existence of the produced cellulose crystals. FTIR spectra strengthen the analysis of the formation of cellulose crystals and the elimination of lignin and hemicellulose from micro-fibrillated cellulose produced from these oil palm empty bunches, where the O-H and C-C bonds had relatively small peak spectrum intensities.

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