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Simultaneous Effect of Ultrasonic and Chemical Treatment on the Extraction of Nanocellulose from Sugarcane Bagasse

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Abstract

The focus of this study was the simultaneous effect of ultrasonic and chemical treatment on the extraction of nanocellulose from sugarcane bagasse. Ultrasonic waves can accelerate the dispersion process of nanocellulose particles so that extraction runs faster and is environmentally friendly. The bagasse was treated by chemical treatment with ultrasonic waves, and then the nanocellulose was prepared using acid hydrolysis with ultrasonic waves. The effect of ultrasonication was investigated. The crystallinity of sugarcane bagasse, cellulose, and nanocellulose was analyzed by X-ray diffraction. Based on the diffractogram, there was an increase in the crystallinity of nanocellulose. The chemical composition of extracted cellulose and nanocellulose was analyzed by Fourier-transformed infrared spectroscopy. The results of the analysis showed that lignin and hemicellulose were removed from the bagasse during the extraction process. The analysis results also showed that the breaking of intramolecular hydrogen and glycosidic bonds occurred during the hydrolysis process. The morphology of bagasse, cellulose, and nanocellulose was analyzed by Scanning electron microscopy. While the particle size of nanocellulose was analyzed by the Particle Size Analysis instrument. The average size of nanocellulose particles was 132.67 nm.

1. Introduction

Nanocellulose is a new cellulose material characterized by improved crystallinity, aspect ratio, surface area, and increased dispersion and biodegradability [1, 2, 3]. Nanocellulose particles can be used as polymer reinforcing fillers, composites, biodegradable material, membrane reinforcement, thickener for dispersions, and drug carrier media and implants [4, 5, 6]. Recently, nanocellulose has received much attention because of its application in various fields of science [7]. Nanocellulose has many hydroxyl groups. Because of that, it has a stable structure in water [8]. Various methods of nanocellulose extraction have been developed [9]. Camargo *et al.* [5] succeeded in making bagasse nanocellulose using the enzymatic hydrolysis method. In contrast, Saputri *et al.* [10] made nanocellulose from bagasse with a more straightforward technique, through a blending technique using a household blender [11, 12]. The chemical synthesis method can be done by hydrolysis [13]. Several studies have used nanocellulose as the base material for bio nanocomposites because nanocellulose can be obtained from renewable and biodegradable natural materials [14, 15]. Various methods of nanocellulose extraction have been developed, until now, the discovery of an environmentally friendly, energy-efficient, and cost-effective nanocellulose isolation method remains a challenge. Therefore, it is necessary to research the extraction of nanocellulose-based antibacterial materials that are environmentally friendly, energy-efficient, and cost-effective.

Nanocellulose can be extracted from agricultural waste, one of which is bagasse [16, 17]. Nanocellulose extraction methods from bagasse that have been successfully carried out include the high-pressure mechanical method, the enzymatic hydrolysis method [18], the acid hydrolysis method [15, 16], and the ultrasonication method [17]. Each method has its weaknesses. The high-pressure mechanical method

requires considerable energy. The enzymatic hydrolysis method requires many costs, and the extraction time is longer. The acid hydrolysis method is less environmentally friendly because of high concentrations of acid, while the ultrasonic method is environmentally friendly. However, Nanocellulose from acid hydrolysis is better than that of ultrasonication. Based on this, this study used a combination method of acid hydrolysis with ultrasonication.

The resulting nanocellulose then characterized its physical properties using several instruments. The crystallinity of sugarcane bagasse, cellulose, and nanocellulose was analyzed by X-ray diffraction (XRD). The chemical composition of extracted cellulose and nanocellulose was analyzed by Fourier-transformed infrared spectroscopy (FT-IR). FT-IR analysis was used to verify the removal of lignin and hemicellulose during the cellulose extraction process. The morphology of bagasse, cellulose, and nanocellulose was analyzed by Scanning electron microscopy (SEM). While the particle size of nanocellulose was analyzed by Particle Size Analysis (PSA) instrument. Analysis data of XRD and FT-IR results was performed using Origin Pro software.

2. Methodology

This research was conduct in three stages, including sample preparation, nanocellulose extraction, and nanocellulose characterization.

2.1. Material and Instrumentation

Sugarcane bagasse gathered in Sidoarjo (East Java, Indonesia) was used in this study. The chemical reagent use was NaOH (Merck), NaClO (Merck), H_2SO_4 (Merck), Filter paper (Whatman No.44), demineralized aqua (Bratachem). The equipment uses glasses, Oven (DGG 9053A), ultrasonicator, analytical balance (Ohaus px224/E), centrifuge, Sputtering Hitachi MC1000. The functional group of nanocellulose was characterized using Fourier transformed-infrared spectroscopy (Portable Benchtop FT-IR spectroscopy Cary 360). The crystallinity of nanocellulose was observed using X-Ray Diffraction (XRD Philips Analytical). Morphology and atomic composition were observed by Scanning Electron Microscopy (SEM HITACHI FLEXSEM 1000), and nanocellulose particle size was analyzed using a Particle size analyzer (PSA Horiba Scientific SZ-100).

2.2. Extraction of Nanocellulose from Sugarcane Bagasse

Mandal and Chakrabarty [19] method was used in this study to modify simultaneous ultrasonication during the extraction process. Clean and dry sugarcane bagasse (SB) was cut into small pieces and crushed into a more delicate powder, then dried in an oven at 60°C for 16 hours. The dry bagasse was put into the Erlenmeyer flask, then 250 mL of 17.5% w/v NaOH was added. The mixture was ultrasonicated for 2 hours at 70°C. Furthermore, the mixture was filtered. Then, the residue was bleached by a 0.7% v/v NaClO solution. The mixture was sonicated for 2 x 1 hour. The residue produced during the bleaching process was washed with distilled water. Then, the residue was dried in an oven at 60°C to obtain cellulose (C-SB). A total of 5 grams of C-SB was reacted with 25 mL of 45% H2SO4. Acid hydrolysis was carried out with the aid of an ultrasonicator at a temperature of 70 ° C for 90 minutes. After that, the mixture was neutralized with 28 mL of 0.5 M NaOH and washed with distilled water until the pH was neutral. Then the mixture was centrifuged to obtain nanocellulose (NC-SB). Then, the NC-SB was dried at 60°C until constant weight [19].

2.3. Lignocellulose Analysis

Lignocellulose analysis was used to determine the levels of lignin, cellulose, and hemicellulose in C-SB. This lignocellulose analysis used the Chesson Method (1981). A total of 1 gram of sample (constant weight) was put into a three-necked flask, and 150 mL of distilled water was added. The sample was then refluxed for 2 hours at a temperature of 100°C. After that, the sample was filtered and washed with distilled water until the filtrate volume was 300 mL and pH 7. Then, the sample was dried in an oven at 80°C to a constant weight. The resulting residue is a hot water soluble (a). The dry residue (a) was put into a three-neck flask and added 150 mL of 0.5 M H2SO4 then refluxed at 100°C for 2 hours. After that, the residue was filtered and washed with distilled water until the filtrate volume was 300 mL and pH 7. The residue was dried to a constant weight, and the dry residue was weighed (b) so that the weight was lost as hemicellulose was obtained. The dry residue (b) was put back into the three-neck flask and added 10 mL of 72% (v/v) H2SO4, then soaked for 4 hours at room temperature. After that, the solution was diluted by adding 150 mL of 0.5 M H2SO4 and refluxed at 100°C for 2 hours. The residue was then filtered and washed with distilled water until the filtrate volume was 400 mL and pH 7. The residue was dried to a constant weight, and the dry residue was weighed by analytical balance (c) so that the weight lost as cellulose was obtained. Meanwhile, the final residue weight is lignin (d) [20, 21].

2.4. Material Characterization

Nanocellulose characterization was carried out the see the composition, functional group, morphology, and degree of crystallinity. The chemical composition of SB, C-SB, NC-SB was characterized using portable benchtop FT-IR Cary 360 to verify lignin and hemicellulose removal. This type of FT-IR does not require sample preparation and does not damage the sample so that the sample can be reused for other characterizations. SEM characterized the morphology of materials. The degree of crystallinity was analyzed through XRD data. The degree of crystallinity was determined through equation (1), the origin pro software determined the XRD diffraction area.

$$\%\text{crystallinity} = \frac{area\ of\ crystalline}{Total\ area} \times 100\% \quad (1)$$

2.4.1. Particle Size Analysis

The dry sample was weighed as much as 0.5 grams. Then the sample was put into a test tube containing 10 mL of demineralized aqua. Furthermore, the mixture is ultrasonicated at 40°C to form a homogeneous mixture. Then, the mixture was analyzed for particle size using the

PSA Horiba scientific instrument at 25 °C with a scattering angle of 90° [22].

2.4.2. Sample Preparation for SEM Analysis

Samples were prepared with gold plating for SEM analysis. A total of 1 gram of dry sample was coated with gold (Au) for 90 seconds with a constant current of 18 mA. Furthermore, the samples were analyzed by SEM [23].

3. Results and Discussion

3.1. Nanocellulose extraction from Sugarcane Bagasse

Nanocellulose extraction from sugarcane bagasse consists of 3 stages, the first stage is alkali treatment, the second stage is bleaching, and the third stage is acid hydrolysis. In this study, all these stages are carried out by ultrasonication. The first stage, Alkali treatment or delignification using NaOH, aimed to remove some lignin monomers, hemicellulose, mineral salts, waxes, pectin, and ash sugarcane bagasse. NaOH was used in this stage to increase the purity of α-cellulose. α-cellulose has insoluble in 17.5% NaOH [24]. This process produced light brown powder (Figure 1b). In the second stage, bleaching treatment using 0.7% NaOCl to bleach the fiber by removing the content of hemicellulose, lignin, and impurities to obtain white cellulose (Figure 1c), NaOCl can oxidize cellulose specifically in its amorphous zone [25]. The third stage was acid hydrolysis using 45% H_2SO_4. Strong acid can remove the amorphous zone of the cellulose chain, so the isolation of the crystalline zone of cellulose can be carried out [14]. Sulfuric acid hydrolysis in cellulose is a heterogeneous process. Acid diffuses into cellulose fiber and cuts the glycosidic bond in cellulose polymers [10, 26]. During the acid hydrolysis process, some of the hydroxyl group (-OH) on the crystalline surface area will turn into sulfate group ($-OSO_3$) [10]. After the hydrolysis stage, the material's color changed from white to brown (Figure 1d). This color change indicates that any charring of cellulose takes place due to the use of a high concentration of H_2SO_4.

Furthermore, the acid hydrolysis product is weighed with a digital analytical balance. Based on the weighing results, 60 grams of bagasse produced 6,890 grams of acid hydrolysis residue. The residue of the acid hydrolysis process was assumed as nanocellulose of sugarcane bagasse (NC-SB) to confirm that the residue of acid hydrolysis was analyzed their lignocellulose content and was characterized by SEM, PSA, XRD, and FT-IR.

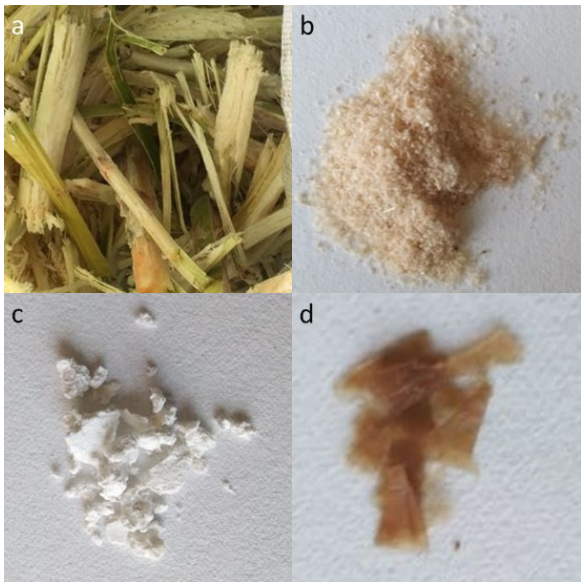

Figure 1. a) Sugarcane bagasse (SB); b) SB after alkali treatment; c) Cellulose (C-SB); d) Nanocellulose (NC-SB)

3.2. Analysis of Lignocellulose and % Moisture Content

Analysis of lignocellulose content aims to determine the levels of lignin, hemicellulose, cellulose in α-cellulose (C-SB). The Chesson-data test results showed that the cellulose content in C-SB was 51.27%, the lignin content was 22.33%, the hemicellulose content was 8.49%. The moisture content of C-SB and NC-SB was found to be 1.47% and 3.85%. The moisture content increases slightly after acid hydrolysis treatment. This is due to three free hydroxyl groups in cellulose that can enhance the rate of moisture absorption [21, 27].

3.3. Material Characterization

3.3.1. SEM Characterization

SEM carried out the morphological analysis, and Figure 2 is a micrograph of sugarcane bagasse before and after treatment. The surface of SB was smoother than NC-SB. This is due to the existence of the outer non-cellulosic layer on SB, such as pectin, lignin, wax, and hemicellulose, which is acted as cementing material to holds the fibers in bundles (Figure 2a). After delignification and bleaching treatment (Figure 2b), there is a removal of the outer non-cellulosic layer on SB, acting as cementing material. The surface morphology of C-SB is not smooth. There is a fiber of cellulose. After ultrasonication in the presence of H_2SO_4 and the drying process, the samples tended to be self-assembled into fibrillated fiber (Figure 2c). The dimension of fiber was decreased because of the removal of the amorphous cellulose region. The NC-SB surface was eroded by acid hydrolysis. Moreover, the erosion of NC-SB may be caused by heat and exited species during ultrasonication. Ultrasonication in the presence of acid and ionic liquid medium hydrolyzed the amorphous region of cellulose up to a certain extent. Some portions of the cellulosic fragment were completely broken to yield soluble oligo mono-saccharides [28].

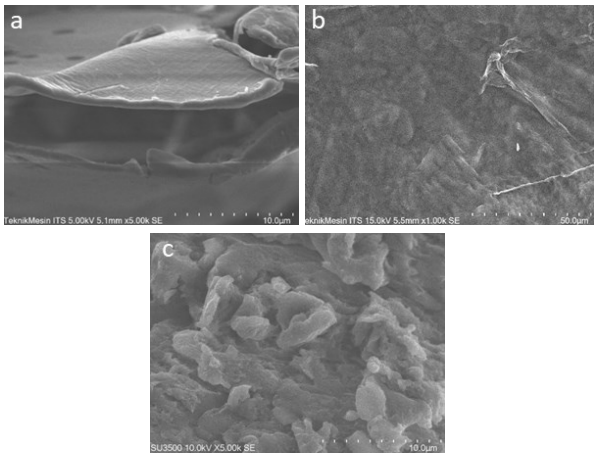

Figure 2. a) Sugarcane Bagasse (SB); b) α-cellulose (C-SB); c) Nanocellulose (NC-SB)

3.3.2. Particle Size Analysis

NC-SB was analyzed their particle size by Instrument Horiba Scientific SZ-100. The result showed particle have polydisperse distribution form, its means that particle has many sizes. The number probability frequency curves of PSA data are multimodal (Figure 3). These curves indicate the particle shape is asymmetry, and there is particle aggregation [29]. This PSA interpretation was supported by SEM analysis (Figure 2c). The average particle size of NC-SB was found to be 132.6 nm.

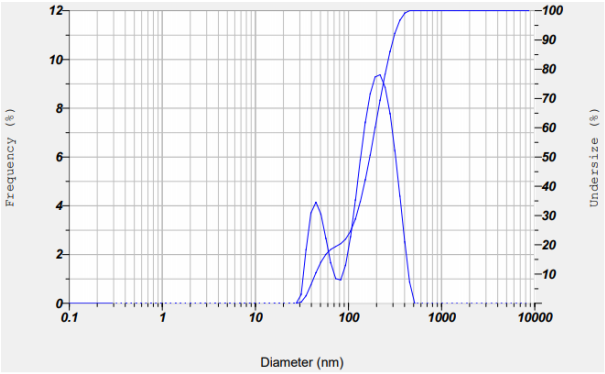

Figure 3. The number probability frequency curves of PSA data

3.3.3. Analysis FT-IR

FT-IR analysis aims to see the functional groups at each stage of nanocellulose. FT-IR analysis aims to see the functional groups at each stage of nanocellulose extraction. Changes in infrared absorption at each stage of the extraction treatment indicated a change in the composition of the bagasse cellulose. Figure 4 shows the FTIR spectra obtained for sugarcane bagasse at different stages of treatment. As present in SB, the FTIR band at 1238 c-1 has indicated the C-O out-of-plane stretching vibration of the aryl group in the lignin monomer. At 1742 cm^{-1} in SB, the FTIR band was the carbonyl group (C=O) stretching vibration of the acetyl and uranic acid ester groups from hemicellulose, pectin, or the ester linkage between the ferulic and p-coumaric acid, carboxylic group of lignin or hemicellulose [30]. The intense absorption band at 1512 cm^{-1} at SB has indicated the aromatic C=C vibration in-plane symmetrical stretching vibration of the aromatic ring in lignin [31]. These three bands are only found in the SB band. This result indicated that the delignification process was successful at the alkaline treatment stage.

Overall, the FTIR spectrum of C-SB dan NC-SB has a similar peak in some waves. The differences between both FTIR spectrum were slightly intensity changes in the peaks. All the spectra have broadband in the region of 3300 cm^{-1} to 3400 cm^{-1}, indicating the O-H stretching vibration of the OH group in cellulose. The absorbance band around 2900 cm^{-1} indicated C-H stretching, and the absorbance peak in the region between 1630 cm^{-1} to 1650 cm^{-1} reflects the O-H bending of absorbed water [30]. The band around 1048 cm^{-1} reflects the C-O-C pyranose ring skeletal vibration, and the increase in the intensity of this peak showed an increase in the crystallinity of the samples [32]. The band around 895 cm^{-1} reflects the β-glycosidic linkage between the anhydroglucose units in cellulose [30].

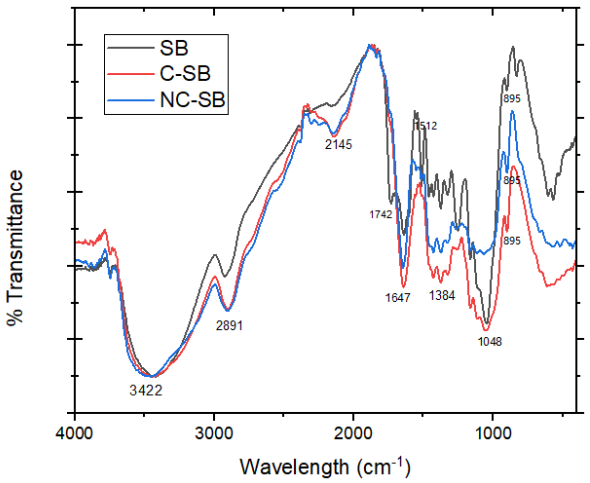

Figure 4. FT-IR Spectra of sugarcane bagasse (SB), Cellulose (C-SB), Nanocellulose (NC-SB)

3.3.4. XRD Analysis

Figure 5 showed the XRD patterns for sugarcane bagasse at different stages of treatment. All XRD patterns showed peaks around 2θ of 16° and 22°, indicating the typical cellulose I structure. The only difference is a slight intensity change in the peaks, indicating some change in the crystallinity index of samples. The most defined peak at 16° and 22° is the NC-SB peak, while the C-SB peak at 16° is more defined than the SB peak. The NC-SB peak at 16° was more defined than the SB peak. This is indicated that acid hydrolysis and ultrasonic wave can increase the crystallinity index of cellulose. During the acid hydrolysis, hemicelluloses and lignin were dissolved, and the remaining crystallin area was isolated. The particle can increases peak intensity and gives narrower crystalline peaks [33]. Ultrasonication in the presence of acid hydrolysis medium effectively dissolved lignin and hemicellulose from SB. %Crystallinity for SB, C-SB, and NC-SB was found to be 68%; 77.2%; 92.62%.

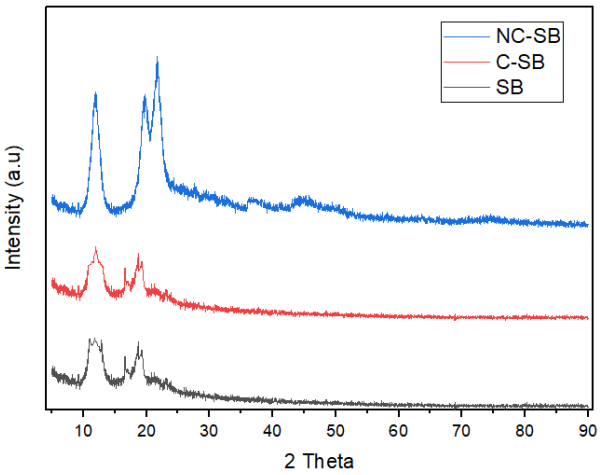

Figure 5. Diffractogram SB, C-SB, and NC-SB

4. Conclusion

Based on this research, it can be concluded that the simultaneous ultrasonic at nanocellulose extraction from sugarcane bagasse by acid hydrolysis method has the effect of %crystallinity of nanocellulose. In this study, nanocellulose with high %crystallinity was successfully extracted from sugarcane bagasse. The FTIR spectrum showed the broadband at 3300-3500 cm^{-1} found to be the vibration of the cellulose OH group. The band around 895 cm^{-1} reflects the β-glycosidic linkage between the anhydroglucose units in cellulose. %crystallinity of nanocellulose reached up to 92.62%. The average particle size of nanocellulose was found to be 132.6 nm. However, the use of high concentrations of sulfuric acid can cause the charring of cellulose. Therefore, it is necessary to conduct further research related to the effect of the concentration of acid used in the extraction of nanocellulose.

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