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by Isnaeni Isnaeni

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Tensile strength and elongation analysis on nano cellulose Film Isolated from Sugarcane Bagasse

M Yuwono¹, B Fauziyah^{2,3,*}, Isnaeni¹ and A Nisak³

¹ Faculty of Pharmacy, Universitas Airlangga, Surabaya, 60286 Indonesia

² Post Graduate Student of Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Universitas Airlangga, Surabaya, 60286 Indonesia

³ Department of Pharmacy, Universitas Islam Negeri Maulana Malik Ibrahim, Malang, 65144 Indonesia

*E-mail: fauziyah@farmasi.uin-malang.ac.id

Abstract. Nano cellulose was successfully isolated from sugarcane bagasse through sulphuric acid hydrolysis of sugarcane bagasse cellulose. Physically, Nano cellulose was transparent and broken white. The crystallinity index of sugarcane bagasse nano cellulose was 80.72%. The particle size of sugarcane bagasse nano cellulose was 225 nm. Delignification process in isolation was successfully showed by releasing peaks in 1239.3 and 1507.7. It show C-O-C vibration of aryl group in lignin and C=C aromatic ring in lignin respectively. Sugarcane bagasse cellulose shows peaks at 1720.2 that represent COOH and hemicellulose carboxylic groups, while the others were not found. The crystallinity index of Nano cellulose was 42.65%. Nano cellulose film prepared in several concentrations (3%, 6%, and 9%). Nano cellulose film also prepared with adding HPMC 2%. Nano cellulose film prepared in 9% concentration was too strict and broken easily. The tensile strength and elongation of Nano cellulose film that prepared in 3% + HPMC 2% and 6% + HPMC 2% were 3.177 Mpa, 10.93% and 3.315 Mpa dan 3.7% respectively.

1. Introduction

Nano cellulose is cellulose that is available in nano size. Similar to cellulose, it is a biopolymer whose availability is abundant in nature and is a very important renewable biomaterial and can be processed into a variety of environmentally friendly polymer products [1]. Nano cellulose is a rigid rod-shaped particle consisting of cellulose chain segments in an almost perfect crystal structure. Nano cellulose has several beneficial properties, including abundance in nature, high tensile strength, high flexibility, and excellent electrical and thermal properties [2]. Besides, Nano cellulose also has an excellent combination of physical, mechanical and biological properties, particularly in its biocompatibility, high biodegradability and low cytotoxicity. Those are making Nano cellulose necessary for designing new biomaterials [1].

Some applications from Nano cellulose include as raw material for paper, paperboard, and packaging. One application for Nano cellulose in the paper and paperboard industry is to increase the bonding strength of paper fibers and have a strengthening effect on paper materials. Nano cellulose has many useful and unique properties, commonly used as fillers or reinforcement in bio composites. It can form emulsions or dispersions and is suitable for use in food products as a thickener or stabilizer. As a medical and hygiene product, Nano cellulose has excellent absorption properties and



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can be used in various products, for example, tissues, nonwoven products, or diapers. Other applications of Nano cellulose are film, painting, cosmetics, automotive, and others [2].

Nano cellulose is a term often used for Nano cellulose made from natural cellulose utilizing acid hydrolysis [1], [3], [4]. The most used acid in hydrolysis can be sulfuric acid [3-6], bromide acid. Compared with sulfuric acid, hydrochloric acid and bromide acid, which do not have much charge on the surface and stable colloidal dispersion, it is more challenging to form Nano cellulose [2].

Based on research, Nano cellulose can convert cellulose from sugarcane bagasse [2]. Nano cellulose has a higher surface area than cellulose, so Nano cellulose can be an alternative solution to make cellulose modified more natural and dissolved in water. It has unique properties such as low density, biodegradable, and excellent mechanical properties. Also, Nano cellulose is more natural to modify and has a high surface area and unique morphology. Nano cellulose with small particle size and high crystallinity has better properties so that the synthesis of Nano cellulose from sugarcane bagasse can be a more environmentally friendly solution because it uses material from unused waste.

2. Methods

2.1. Materials

Sugarcane bagasse sample is a waste from sugar industry located at Malang, East Java, Indonesia. The other reagents used are: sulphuric acid, nitric acid, sodium hydroxide, sodium hypochlorite, hydroxypropyl methyl cellulose (HPMC), aquadest. All chemicals were pro analysis grade.

2.2. Synthesis of Nano Cellulose

Sugarcane bagasse is washed, dried, and then converted to powder using a grinder to obtain sugarcane bagasse powder. The sugarcane bagasse powder is soaked with 3.5% nitric acid with a ratio 1: 4 (w/v) at 90°C for 2 hours, then filtered and washed using distilled water. The next process was delignification using 17.5% sodium hydroxide at 90°C for 5 hour. The final step is bleaching neutral residue using sodium hypochlorite 5%. Sugarcane bagasse cellulose was dried in an oven at 40°C for 24 hours. The cellulose obtained was added with sulphuric acid 50% (1:20) w/v at 45°C, for 30 minutes, then added with aquadest. The colloid suspensions were centrifuged at 3,500 rpm for 30 minutes. The colloids formed were dialyzed with cellulose membranes to neutral pH. The results of dialysis were sonicated and roasted at 45°C for 24 hours.

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2.3. Characterization of Nano Cellulose

2.3.1. Particle Size Analysis. The particle size of Nano cellulose was determined by Microtrac Particle Size Analyzer. The Nano cellulose was diluted with distilled water and inserted into the cuvette. The measurements were done at room temperature with the measurement range from 10 nm until 4,000 nm.

2.3.2. Infrared Spectra Analysis. The infrared spectra of sugarcane bagasse cellulose and nano cellulose were recorded on Fourier Transform Infrared Spectrometer Shimadzu. The sample was measured in the wavelength range from 4000 cm⁻¹ to 500 cm⁻¹.

2.3.3. X-ray Diffraction Analysis. The X-ray diffraction data were collected using X'pert Pro PANalytical. The crystallinity index calculated by using the following calculation. The crystallinity index (CI) was calculated using the equation 1

$$C_I(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100\%$$

2.3.4. Tensile Strength and Elongation Analysis. The Nano cellulose film was made by dissolving 100 mL of Nano cellulose colloid in a distilled water. Solution of Nano Cellulose was stirred at 500 rpm

for 30 minutes. Nano cellulose films were prepared in several concentrations 3%, 6%, and 9% which added 2% HPMC in each varian. The solution was molded and roasted at 60°C for 3 hours. Then, tensile strength and elongation of Nano cellulose film were determined by Texture analyzer.

3. Results and Discussion

3.1. Synthesis of Nano Cellulose

Sugarcane bagasse was hydrolysed by nitric acid. Nitric acid destruct ester bonding between lignin and hemicellulose and cleave hydrogen bonding between lignin and cellulose. Nitric acid convert lignin into nitro lignin form, which can be dissolved in the base [7]. The influence of electrons on the nucleus of the guasil group of lignin during the hydrolysis process causes the electrophile to be strongly oriented to the para or ortho position of the OH group causing a break in bonding with the side of the propyl chain, where the final unit of lignin will split from the remaining lignin macromolecules into dissolved form [8]. Sodium hydroxide release lignin and hemicellulose while α -cellulose is obtained [7]. The change of sugarcane bagasse to cellulose is given in figure 1. Sodium hypochlorite remove chromophore component, which absorbs unbleached light, especially by the remnants of the degraded functional lignin group [9].

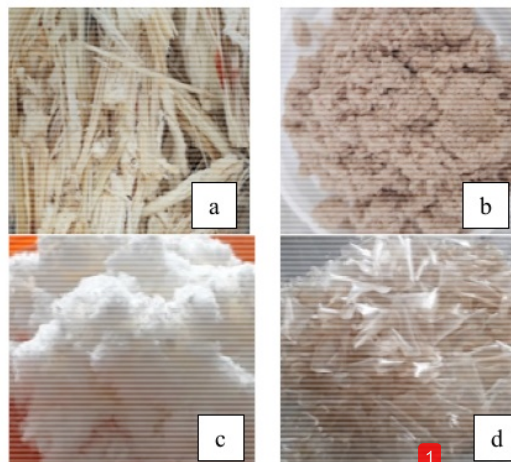


Figure 1 a. sugarcane bagasse; b. sugarcane bagasse powder; c. sugarcane bagasse cellulose; and d. sugarcane bagasse Nano cellulose

Sulphuric acid degrade hydrogen bonding in the weakest region of cellulose (amorphous region). The amorphous regions that have been reduced make the crystallinity of cellulose increase and produce Nano crystalline cellulose [2]. Hydrolysis process with sulphuric acid is given in figure 2 [10].

3.2. Particle Size Distribution

The particle size of Nano cellulose which obtained from sugarcane bagasse is 225 nm. These results are following the previous studies [3], [11], which state that sugarcane bagasse nano cellulose has range size between 10-350 nm.

3.3. Infrared Spectra Analysis

Infrared spectra analysis was used to show the degradation of lignin from lignocellulose biomass sugarcane bagasse through analysis of its functional groups of sugarcane bagasse, sugarcane bagasse cellulose and sugarcane bagasse nano cellulose.

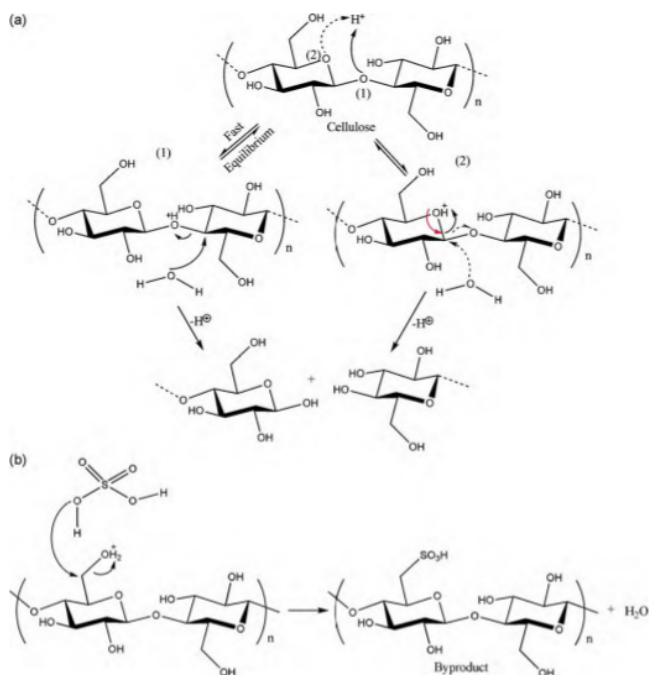


Figure 2. Hydrolysis process of cellulose with sulphuric acid [10].

From the FTIR spectra (Figure 3), all samples have peaks in the area 3,326.6-3,336, which showed an O-H stretching vibration bond. All samples show same peak in 28,994.3-2,896.1 which showed the presence of C-H stretching vibration bonds. The peak of 1,602.8-1,647.5 shows the O-H bond of cellulose which can absorb water. The absorbance in 1313.9-1367 area show the -O- to form cellulose. The presence of C-O-C pyranose ring stretching vibration showed in 1,019.4-1,030.6.

The important peak to show the difference between sugarcane bagasse with cellulose and nanocellulose is peak in 1,239.3 and 1,507. It shows C-O-C vibration of aryl group in lignin and C=C aromatic ring in lignin respectively. Sugarcane bagasse cellulose shows peaks at 1,720.2 that represent COOH and hemicellulose carboxylic groups.

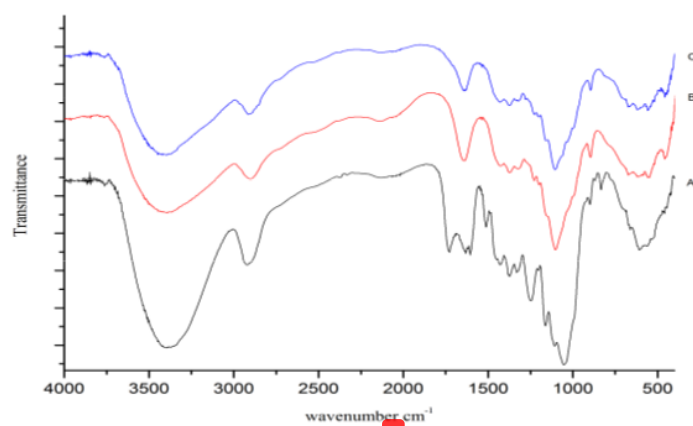


Figure 3. The infrared spectra of sugarcane bagasse (a), sugarcane bagasse cellulose (b), and sugarcane bagasse nano cellulose (c)

3.4. X-Ray Diffractogram Analysis

Figure 4 shows the intensity of sugarcane bagasse nano cellulose higher than sugarcane bagasse cellulose. Both of graphs show three diffraction peaks, they are : 14.8°, 16.8°, and 22.5°. The crystallinity indeks of sugarcane bagasse cellulose and nano cellulose are 63.97% and 80.27% respectively.

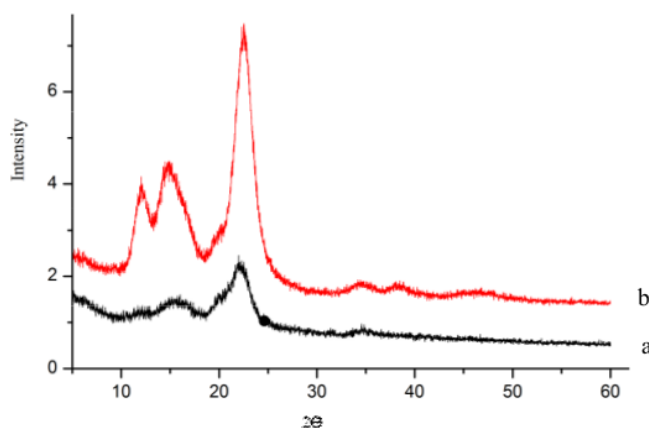


Figure 4. XRD Graph of a. sugarcane bagasse cellulose and b. sugarcane bagasse nano cellulose

3.5. Tensile strength and elongation Analysis

The results of the tensile strength and elongation test of Nano cellulose film 3% + HPMC 2% and 6% + HPMC 2% were 3,177 MPa, 10.93% and 3,315 MPa and 3.7%, respectively. It shows that the more Nano cellulose content, the more hydrogen bonds are formed between the same polymers, so that it requires higher energy to break the bonds but can break at a low strain [11]. The addition of HPMC, which shows comparable results without the addition of HPMC but has higher tensile strength and elongation value due to the addition of HPMC adds mechanical strength from the film Nano cellulose

film also prepared with adding HPMC 2%. Nano cellulose film prepared in 9% concentration was too strict and broken easily. The tensile strength and elongation of Nano cellulose film that prepared in 3% + HPMC 2% and 6% + HPMC 2% were 3.177 Mpa, 10.93% and 3.315 Mpa dan 3.7% respectively.

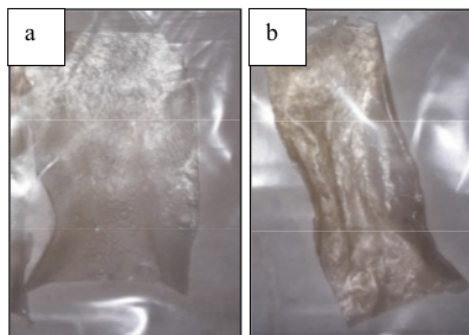


Figure 5. Nano cellulose film a. Nano cellulose 3%+HPMC 2% and b. Nano cellulose 6%+HPMC 2%

4. Conclusion

Nano cellulose was successfully isolated from sugarcane bagasse through sulphuric acid hydrolysis of sugarcane bagasse cellulose. Physically, Nano cellulose was transparent and broken white. The particle size of sugarcane bagasse nano cellulose was 225 nm. all samples have peaks in the area 3326.6-3336, which showed an O-H stretching vibration bond. All samples show same peak in 28994.3-2896.1 which showed the presence of C-H stretching vibration bonds. The peak of 1602.8-1647.5 shows the O-H bond of cellulose which can absorb water. The absorbance in 1,313.9-1,367 area show the -O- to form cellulose. The presence of C-O-C pyranose ring stretching vibration showed in 1,019.4-1,030.6. Delignification process in isolation was successfully showed by releasing peaks in 1,239.3 and 1,507.7. It show C-O-C vibration of aryl group in lignin and C=C aromatic ring in lignin respectively. The crystallinity indeks of sugarcane bagasse cellulose is 80.72%. Sugarcane bagasse cellulose shows peaks at 1720.2 that represent COOH and hemicellulose carboxylic groups, while the others were not found. The crystallinity index of Nano cellulose was 42.65%. Nano cellulose film prepared in 9% concentration was too strict and broken easily. The tensile strength and elongation of Nano cellulose film that prepared in 3% + HPMC 2% and 6% + HPMC 2% were 3.177 Mpa, 10.93% and 3.315 Mpa dan 3.7% respectively.

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