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CHARACTERIZATION OF BIOPOLYMER NANOCELLULOSE FIBER FROM CORN COB WASTE (*ZEA MAYS L*) USING CHEMICAL-MECHANICAL TREATMENTS

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ABSTRACT

This study aims to determine the highest cellulose purity based on variations in NaOH concentration and to characterize nanocellulose fiber from corn cob waste. Nanocellulose fiber was obtained using chemical-mechanical treatments. Chemical treatment consists of pretreatment, de-lignification, and bleaching stages. While mechanical treatment consists of stirrer and ultrasonication methods, The results showed that the highest cellulose purity was 75.32% using 60% NaOH. In addition, FTIR test results showed that lignin and hemicellulose compounds were successfully removed during chemical treatment, and functional groups from the typical structure of cellulose, which are OH, C-H, and C-O-C groups, with their respective wavenumbers, did not show significant changes during mechanical treatment. Nanocellulose fibers have been successfully formed. It was evident from the morphology of the fibers, which had an average diameter of 120.57±14.05 nm. Additionally, the nanocellulose fiber has excellent colloidal stability with a potential zeta value of -72.5 mV. Based on these results, biopolymer nanocellulose fiber has been successfully obtained from corn cob waste through chemical-mechanical treatments.

Keywords: Cellulose Purity, Characteristics, Chemical Treatment, Colloidal Stability, Mechanical Treatment, Zeta Potential.

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INTRODUCTION

Corn production has increased because it can adapt to various agroclimatic conditions. Therefore, the rapid growth of corn production results in an increase in corn cob waste. Approximately 40% of corn grain produced goes to waste, including corn cobs, stalks, and husks. In most cases, corn kernels are used as fodder or compost.[1-2] Many people burn corn cobs waste using a quick and easy procedure. The incomplete burning of leftovers will release significant quantities of dangerous pollutants into the environment, such as SO_2, CO, and NO_x, which can pollute the air and accelerate global warming.[3] So there needs to be good handling to overcome these impacts. Previous research has provided formulations for using waste corn cobs as a material for medicine, bioenergy, and nanocomposites.[4] This is because various elements and chemicals can be found in corn cobs, even when both are extracted and used directly for the extraction process to get complex products. These elements and chemical compounds include carbon (42.90%), hydrogen (6.00%), nitrogen (1.00%), oxygen (50.10%), aluminum (4.44%), calcium (2.09%), iron (1.06%), potassium (2.20%), magnesium (1.49%), sodium (1.14%), silica (10.06%) and protein (5.20%).[5-7] In addition, corn cobs contain a fairly high lignocellulose content that can be used in the fiber and composite industry consisting of cellulose (45.68%), hemicellulose (41.03%), and lignin (6.38%).[8] Cellulose is the primary

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component of lignocellulose biomass, odorless, hydrophilic, biodegradable, water-insoluble material obtained from plants, animals, or bacteria, and a cellulosic extract or processed substance with nanoscale structural dimensions is referred to as "nanocellulose" in most cases.[9] The research and implementation of nanocellulose fiber have piqued industry interest over recent decades due to its promising qualities and capabilities, such as inherent biocompatibility, renewability, natural abundance, biodegradability, cost-effectiveness, and environmental friendliness.[10] The compounding of nanocellulose fiber into the xylan or lignosulfonates matrix provided excellent mechanical properties of the films with Young's modulus (1.08-3.79 GPa) and tensile strength (12.75-14.02 MPa).[11] Membranes from nanocellulose films, such as MFCs, CNFs, and CNCs (or NCC), frequently have exceptional qualities such as great mechanical strength and low thermal expansion coefficient that make them desirable materials for packaging.[12-14] Therefore, in this study, nanocellulose fiber was obtained by utilizing the cellulose content of corn cob waste. Several studies have been done to produce nanocellulose fiber from corn cob waste, and there are still not many researchers who study the characteristics at each stage of isolation, both physically and chemically. In addition, the method in this study is also not used very frequently, especially in mechanical treatment by combining stirring and ultrasonication stages. Therefore, this study aims to determine the highest cellulose purity based on variations of NaOH concentration and to characterize nanocellulose fiber from corn cob waste using chemical-mechanical treatments.

EXPERIMENTAL

Materials

Nanocellulose fiber was produced from Bisi-18 corn cob waste collected from the Aik Ampat Village area, Gerung District, West Lombok Regency, Indonesia. The tools and materials used include Fourier Transform Infrared (FTIR) type Perkin Elmer Spectrum Two (USA), Scanning Electron Microscope (SEM) type Jeol JCM-7000 (Japan), Zeta Potential Analyzer type Nano Particle Analyzer Horiba SZ-100 (USA), ultrasonic cleaner PS-30A (China), sodium hydroxide pearl (NaOH) 60% (w/v) (technical grade) and sodium hypochlorite (NaOCl) 12% (technical grade).

Preparation

Before the isolation of cellulose, corn cob waste powder was prepared first through several stages including washing, cutting, drying, grinding, and sieving. The ground-up powder was homogenized by sifting it through a sieve with a 100-mesh. A variation of the treatment test was first done to determine the purity of cellulose. Variation tests were carried out by varying the concentration of NaOH at the de-lignification stage, namely by 40%, 50%, 60%, 70%, and 80%. The highest concentration of NaOH with cellulose purity was then used in the nanocellulose fiber isolation process.

Isolation of Cellulose

The cellulose isolation process consists of three stages: pretreatment, de-lignification, and bleaching. The pretreatment stage was carried out by adding 5 grams of corn cob powder with distilled water heated at 100 ºC for 3 hours. The pretreated solids were then dried at 70 ºC for 3 hours. The de-lignification stage was carried out by dissolving the pretreatment powder in 60% NaOH solution (w/v) and heating at 100ºC for 3 hours. After that, it was filtered to separate the solids. The next stage was bleaching, the residue from de-lignification was purified using 100 mL of 12% NaOCl solution heated for 1 hour 30 minutes. After the cellulose precipitate was filtered and neutralized to pH 7, it was dried at 50 ºC for 4 hours. The powder produced from each stage of isolation identified its characteristics both from physical and chemical properties: yield, lignocellulose content, and the presence of functional groups.

Isolation of Nanocellulose Fiber

The isolation of nanocellulose fiber was carried out using mechanical treatment through the stirring and ultrasonication stages. The stirring stage was carried out by adding 0.5 grams of pure cellulose powder to 80 mL of distilled water and stirring for 4 hours at 1000 rpm speed. The suspension was continued at the ultrasonication stage which was carried out for 6 hours. To obtain solid nanocellulose fiber, the suspension was centrifuged for 10 minutes and the resulting solid was dried at 50 ˚C for 4 hours. The nanocellulose fiber powder resulting in this stage was then characterized using FTIR, SEM, and Zeta Potential Analyzer.

Characterization

Several parameters were identified including functional groups, morphology, fiber size distribution, and colloidal stability. Functional groups were carried out using FTIR Perkin Elmer Spectrum Two at wavenumbers 400-4000 cm^{-1}. In addition, morphological characterization and fiber size distribution were carried out using SEM JCM-7000 Jeol type. Characterization was carried out at magnification variations of 6000, 9500x, and 10000x, 2 μm scale, WD 12.8 mm, signal type of SED, and at a voltage of 5.0 kV. Colloidal stability was carried out using a Zeta Potential Analyzer with a dispersing medium of distilled water. The results of all characterizations were then processed using some software including Microsoft Excel, Image-J, and Origin Pro 8.5.1. In addition, the effects of five variations in NaOH concentration on cellulose purity and yield percentage were conducted by using the ANOVA One-way analysis approach, with hypotheses of P value < 0.05 meaning there are significant differences in data of cellulose purity and yield percentage from variations in NaOH treatment.

RESULTS AND DISCUSSION

Identification of Cellulose Characteristics

The powder produced from each isolation stage identified its properties in terms of organoleptic, yield, lignocellulose content, and functional groups of cellulose. Shape, color, and odor are the organoleptic characteristics that were identified. The shape (powder) and odor (odorless) were consistent at each stage, but the powder's color had different characteristics (Fig.-1). At the pretreatment stage, the powder's color was slightly brighter than before treatment. It became more brownish after the de-lignification stage, which indicates that lignin and hemicellulose compounds have been successfully dissolved using a 60% NaOH solution (w/v).[15] The bleaching stage was completed to create pure cellulose powder. Using a 12% NaOCl solution, the remaining lignin and hemicellulose were satisfactorily purified with a noticeable color change to whiter. The test's results were consistent with calculations of yield percentages that show that each stage of the isolation has a lower yield than the previous one, as seen by the drop in powder mass from up to 5 grams to 0.77 grams. The yield percentage of the three stages are 81.00%, 30.40%, and 15.40%. The more material lost during the stage, the smaller the yield value produced.[16]

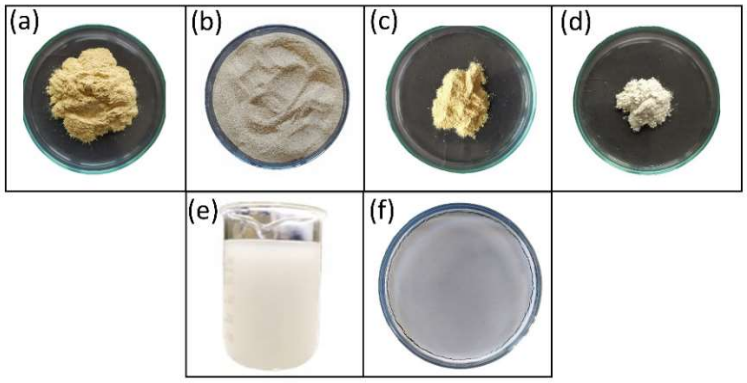

Fig.-1: Powders (a) Untreated, (b) Pretreatment, (c) De-lignification, (d) Bleaching, and (e) Suspension of Nanocellulose Fiber (f) Dried Nanocellulose Fiber

The presence of lignocellulose was identified in every stage of isolation. The lignocellulose content before chemical treatment was also tested as a baseline reference for assessing changes in lignocellulose content during the isolation stage. As well as the percentage of yield, lignin, and hemicellulose content also decreased. This is due to the implementation of chemical treatment during isolation that can remove lignin and hemicellulose compounds.[17] The initial lignin content of 7.11% decreased to 3.48%, while hemicellulose whose initial content was 16.12% to 4.69%. The results differ on cellulose content, which increases at each stage of the isolations. Cellulose content increased from 40.95% to 75.32% (purity of cellulose) after chemical treatment. The cellulose produced in this study has a fairly high enough level of purities.

Table-1: Lignocellulose Content Test Results

Stages	Test Parameters	Reference[18]	Sample (%)
Untreated	Cellulose	32.60	40.95
	Lignin	14.40	7.11
	Hemicellulose	48.88	16.12
Pretreatment	Cellulose	-	52.28
	Lignin	-	7.04
	Hemicellulose	-	3.95
De-lignification	Cellulose	-	68.73
	Lignin	-	1.83
	Hemicellulose	-	1.79
Bleaching	Cellulose	29.60	75.32
	Lignin	11.40	3.48
	Hemicellulose	47.55	4.69

Identifications of cellulose characteristics at each stage of the isolations were also strengthened by knowing the emersion of functional groups. Identification was carried out by analyzing the absorption band of the FTIR spectrum (Fig.-2). Several patterns of absorption bands change at each stage of isolation. At the pretreatment stage, the absorption band intensity occurs very much at wavenumber range of 1000-1750 cm^{-1} and decreases in the de-lignification and bleaching stages, which indicates the elimination of leftover lignin and hemicellulose because of a non-cellulose compound absorption band at wavenumbers 1500-1750 cm^{-1}.[19] Additionally, a significant change in absorption peak occurred at wavenumber 1732.01 cm^{-1} which was an acetyl and uronic ester group.[20] The peak absorption of the group is lost in the de-lignification and bleaching stages which shows that the acetyl xylan group was almost completely lost through the chemical treatment used (Table-2).

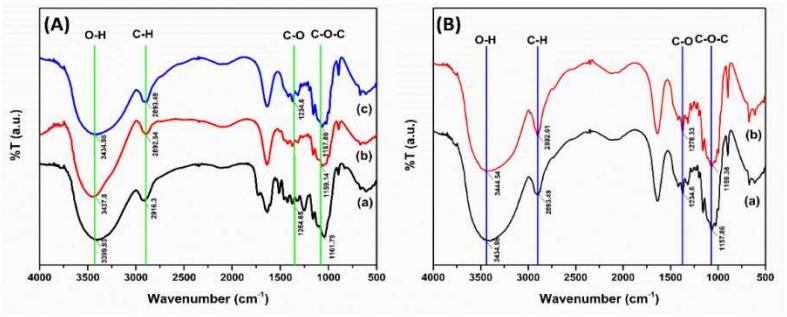

Fig.-2: FTIR Spectrum (A) Each Isolation Stage (a) Pretreatment (b) De-lignification and (c) Bleaching, (B) Comparison FTIR Spectrum (a) Cellulose (b) Nanocellulose Fiber

The existence of a typical cellulose functional group in this study was shown by the appearance of absorption bands in the O-H group (3399-3435 cm^{-1}), C-H stretching vibration functional group (2892-2917 cm^{-1}), C-O-C stretching (1157-1162 cm^{-1}), stretching vibration from the C-O group (1233-1256 cm^{-1}) and the presence of $-CH_2$ bending (1417-1427 cm^{-1}) that shows crystalline peaks of cellulose.[21] After IR spectra analysis at each isolation stage, the OH group with a wavelength range of 3399.39 cm^{-1} experienced a shift in absorption peak width to 3437.8 cm^{-1} at the de-lignification stage. These data were also reinforced by symmetrical C-H stretch absorption peaks of the CH_2 and CH_3 groups in the wave range 2917 cm^{-1} to 2892 cm^{-1}. The bleaching stage increases the uptake of symmetrical C-H stretching of the CH_2 and CH_3 groups to 2893 cm^{-1} and strengthens the presence of cellulose.

Table-2: Results of Wavenumber and Functional Group Analysis of Each Isolation Stage

Functional Groups	Wavenumber (cm^{-1})				Reference[22]
	A	B	C	D	
-OH group	3399.93	3437.8	3434.98	344.54	4000-2995
C-H stretching vibration	2916.3	2892.54	2893.49	2892.61	2890
Acetyl and uronic ester	1732.01	-	-	-	1730-1700
H-O-H stretching	1637.72	1638.21	1638.81	1638.26	1640

Carboxyl group stretching	1606.26	-	-	-	1613
C=C stretching aromatic	1515.2	-	-	-	1450
-CH_2 bending	1426.82	1424	1417.3	1419.62	1430
-OH phenolic group bending	1376.62	1375.26	1371.7	1375.25	-
-OH bending vibration	1320.64	1318.05	1317.9	1316.48	-
C-O stretching vibration	1254.65	-	1234.6	1278.33	1215
C-O-C stretching	1161.79	1159.14	1157.86	1159.38	1170-1082
Glycosides of cellulose	898.13	896.06	896.25	895.72	700-900

In Table-2, A is pretreatment, B is de-lignification, C is bleaching, and D is isolation of nanocellulose fiber. At the pretreatment stage, strong absorption at wavenumber 1637.72 cm^{-1} indicates stretching of the H-O-H group in the amorphous region. At the de-lignification and bleaching stages, there is an increase in absorption intensity indicated by a wavenumber shift of 1638.81 cm^{-1}. This suggests that in amorphous regions water molecules are absorbed by crystalline cellulose fibers, and also indicate the formation of crystalline peaks in the resulting cellulose powder.[23] The presence of typical cellulose functional groups shows that the chemical treatment used in this study has succeeded in removing lignin and hemicellulose compounds by not damaging the typical structure of cellulose at each stage. This result also shows conformity with the yield data obtained and the lignocellulose content test results in Table-1.

Table-3: Anova Test Results on Treatment Variations

Treatment Variations	Yield (%)	Cellulose Purity (%)
NaOH 40%	12.05	69.52
	12.35	69.26
NaOH 50%	13.95	74.24
	14.25	74.49
NaOH 60%	15.60	75.52
	15.20	75.12
NaOH 70%	15.45	69.52
	15.75	69.26
NaOH 80%	14.20	66.69
	14.40	67.24
P value	0,000107549	0,0000020227

The effect of five variations of NaOH concentration on the yield percentage and cellulose content was tested and the results can be seen in Table-3. After One-way ANOVA analysis, the variation of treatment with yield percentage data has a P value of 0.000107549 (<0.05), which indicates a notable divergence in yield percentage from the variation of treatment. In addition, there is a significant difference in the level of cellulose purity from variations in NaOH concentration with P value = 0.0000020227 (<0.05). The data shows that the composition of NaOH affected the yield percentage and the purity of the cellulose produced. Based on some of the results above, the best treatment for making nanocellulose fiber was with a composition of 60% NaOH.

Identification of Nanocellulose Fiber Characteristics

Nanocellulose fibers have been successfully isolated using mechanical treatment through stirring and ultrasonication. The isolation results of nanocellulose fiber are shown in Fig.-1. Three parameters were identified: functional groups, morphology, and fiber diameter distribution. The results of FTIR spectrum analysis are shown in Fig.-2, and the wavenumber and functional group between cellulose and nanocellulose fiber are shown in Table-2. The absorption intensity of the two absorptions has the same similarity in the wavenumber in each functional group that appears (no significant change occurs). In nanocellulose fibers, the typical constituent groups of cellulose experience an increase in absorption intensity compared to cellulose powder. Among them the O-H group increased to 3444.54 cm^{-1}, the CH_2 group 1419.12 cm^{-1}, the C-O stretch to 1278.33 cm^{-1}, and the C-O-C group increased in intensity to 1159.38 cm^{-1}. In addition, the absorption band at wavenumber 895.72 cm^{-1} of the nanocellulose fiber spectrum was sharper than the cellulose spectrum, which indicates improved crystallinity growth and reinforces that the isolated nanofibers' major component was cellulose. Another essential characteristic is the morphology and

fiber diameter distribution of nanocellulose fiber. The morphology generated consists of fibers with uneven patterns and various diameters (Fig.-3). After analysis using the Image-J application, the diameters of fiber were mostly in the size range of 50-150 nm with an average of 120.52±14.05 nm. The smallest fiber diameter obtained was 45.62 nm, while the largest was 287.93 nm. The diameter of the nano-sized obtained fibers shows a good nano-fibrillation. The mechanical treatment applied in this study can weaken hydrogen bonds between nanofibers. Van der Waals forces, which maintain relatively weak borders between bound microfibrils, are destroyed in the presence of strong enough mechanical oscillation power produced by ultrasonic waves. In addition, sonication time can impact the particle size of materials. As a result, micro-sized cellulose fibers can gradually split in the axial direction and disappear into nanofibers.[24,25] Evenly distributed nano-sized fibers can be observed qualitatively by decreasing suspension transparency after mechanical treatment (Fig.-1). Although the applications of the chemical-mechanical method succeeded in isolating nanocellulose fiber, the average diameter size obtained was quite high (>100 nm). One of the challenges is the scalability of the tools available. High-specification ultrasonication is necessary to achieve a smaller and more consistent dispersion of fiber diameter sizes. To achieve the required outcomes, this process must be further modified.

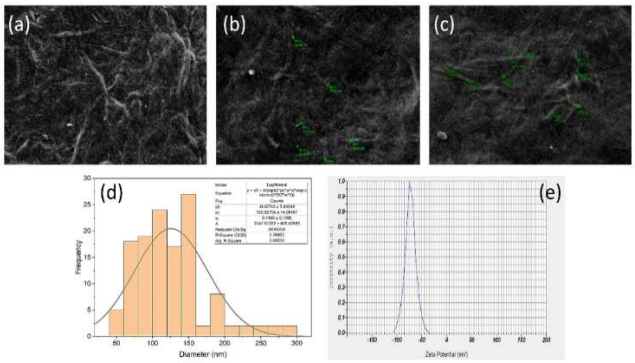

Fig.-3: Morphology of Nanocellulose Fiber at Magnification of (a) 6000x, (b) 9500x, (c) 10000x, (d) Diameter Distribution of Nanocellulose Fiber, and (e) Distribution of Potential Zeta Value

In addition to some of the parameters above, a potential zeta was also characterized to identify the colloidal stability of nanocellulose fiber. The results of the potential zeta test are shown in Fig.-3. The potential zeta value obtained was quite high (>±60 mV). This result showed that the nanocellulose fiber produced has excellent colloidal stability (no agglomeration or merger of colloids into larger sizes). It also shows the stable surface charge and polarization properties of nanocellulose fiber particles and reflects the good electrical potential of the particles.[26]

CONCLUSION

Based on the results of research that has been carried out, the highest level of cellulose purity was obtained in 60% NaOH concentration, which was 75.32%, powder color changes occurred during the isolation stage with a final yield percentage of 15.40%. In addition, FTIR test results showed that lignin and hemicellulose compounds were successfully removed during chemical treatment, and functional groups from the typical structure of cellulose, which are OH, C-H, and C-O-C groups, with their respective wavenumbers, did not show significant changes during mechanical treatment. The results of the SEM test showed the formation of morphology in the form of fibers with an average diameter of 120.57±14.05 nm. The nanocellulose fiber obtained has excellent colloidal stability with a potential zeta value of -72.5 mV. Biopolymer nanocellulose fiber has been successfully obtained from corn cob waste using chemical-mechanical treatments.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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