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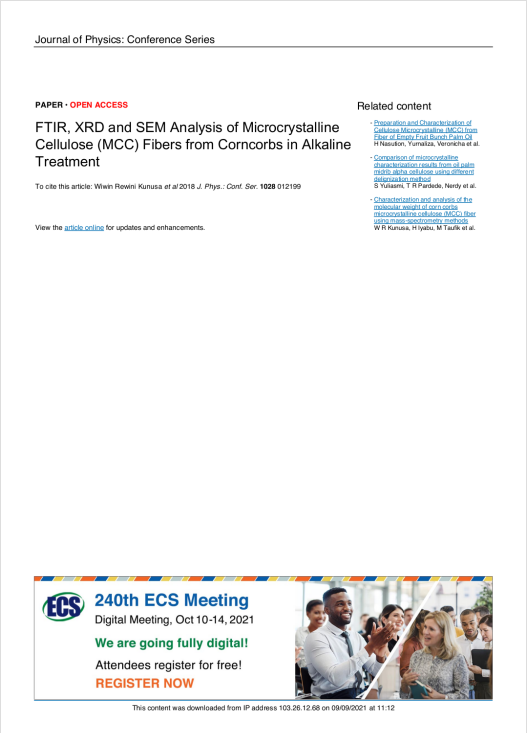

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FTIR, XRD and SEM Analysis of Microcrystalline Cellulose (MCC) Fibers from Corncorbs in Alkaline Treatment

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FTIR, XRD and SEM Analysis of Microcrystalline Cellulose (MCC) Fibers from Corncorbs in Alkaline Treatment

FTIR, XRD and SEM Analysis of Microcrystalline Cellulose (MCC) Fibers from Corncorbs in Alkaline Treatment

Wiwin Rewini Kunusa*, Ishak Isa, Lukman AR Laliyo & Hendrik Iyabu

Department of Chemistry, Gorontalo State University, Gorontalo 96128, Indonesia

* rewinikunusa2014@gmail.com

Abstract. In this paper, we report the isolation method of cellulose, alpha cellulose and microcrystalline cellulose from corncorbs waste in alkaline treatment NaOH 4%,6%,8%,10%,12%,14% and 17% solution. Dewaxing, dehemiselulosa, delignifikasi and bleaching is the stage of cellulose isolation. The MCC particles were extracted by acid hydrolisis with 0.1N HCl. The 4% NaOH concentration provides the highest α-cellulose content (60%) with total yield MCC of 84.53%. SEM, FT-IR and X-ray diffraction analyzes are used to determine of the MCC. FTIR spectra showed that each MCC had -OH group, stretching of C-O on cellulose fiber I and cellulose II. C-0 bonds shows a change in rotation of glucose residue around the glycosidic bond into Cellulose II. SEM analysis showed that the concentration of 6% NaOH has a morphological structure such as cellulose standard. The 8% NaOH concentration yields a CrI value of 98%.

1. Introduction

Corncob is lignocellulosic material consisting of 38.8%±2.5%, 44.4%±5.2%, hemicellulose and lignin cellulose 11,9% ± 2,3%. Cellulose is biodegradable and biocompatible which can be processed into product with economic value. Cellulose is unbranched macromolecule that consist of anhydroglucopyranose units which connected by the β-1,4-glycosidic bond. Each unit of anhydroglucopyranose has three OH groups [1].The original crystalline structure of cellulose polymorphs is known as cellulose I, when dissolved and crystallized in the form of a more stable crystal in a solution called cellulose II. Cellulose II powder is used as an alternative raw material in the field of industrial, pharmaceutical, health and biofuel production [4][20].

In application, degradation of cellulose structures is an important step because it must find a solvent that not reduce polimerization degree (DP) of cellulose [14]. Cellulose fibers are not homogeneous depending on the source of the fiber and solvent quality through opening the pore of the amorphous-crystalline region and breakdown hydrogen bonds [28][15]. Pretreatment technology is requiered in hydrolysis process [22][14] to change the intra-inter chain structure of the molecule by swelling and insertion of the chemical group [24]. Methods of cellulose pretreatment include acetylation, mercerization, peroxidation, benzoyilation, graft copolymerization. Bacterial media is the best method for surface modification of cellulose fibers that have been widely studied [12].

A study of lignocelluloses by various methods has been studied including hydrolysis using the enzyme NovozymesCellic® C-Tec2 and H-Tec+ in 10% NaOH [12] modification of cellulose dissolution in NaOH/ZnO by enzymatic treatment of xylanase and endoglucanase with pulp solubility from 29% -81% [8] the mercerization method using NaOH [6][34][16] KOH [3] NH3 solution [15]

NaOH/ H2O2/hypochlorite [21] NaOH/H2O2 [34] NaOH/NH3 [9] NaOH/Urea [26] (NaOH).NaOH is very effective to breaking lignin and glicosydic bond inlignocellulosic bio-conversion (HOO) and NaOH method [14,6,33] method of microwave oven with use KOH and NaOH 0.75% - 3% [3] NaOH 4%, 8%, 13%, 18% termination of lignin bonding and glycosidic polysaccharides [19-27]. The cellulose structure has a more open surface facilitating the interaction of the fibers [20] 12% -18% NaOH and NH3 forming cellulose II and cellulose-ammonia complexes [1] NaOH 4% cellulose II ± 50% and extractive 72% [21]. NH3 and NaOH altered the crystal-alomorph structure of cellulose II and IIII [8].

Cellulose can be made into MicrocrystalinCelullose or nanocellulose by strong acid hydrolysis [22][32] in the form of white, odorless, tasteless white crystalline powders.[32] Cornstarch cellulose (CPNs) with α-cellulose content 47.27% acording to Avicel® PH 101 [4-29] NaOH 4% yields 47.6% oxidized cellulose with NaClO 7.5 mmol/gr yields 36.5% alkaline cellulose nanofibrils (ACNFs) [11] MCC is synthesized using H2SO4, HCl and HBr [29] MCC is stronger and stiffer than amorphous cellulose or native cellulose [20]. The CRC (CrI) index of sugar palm obtained was 97.5% [17] CrI sample 83.46% [18] Cellulose-based nanoparticles (CPNs) of corncobs with KOH had CrI values 57.28% [2] with H2SO4 values CrI nanocellulose was higher at 60.2% [22]. In this research cellulose, α-cellulose and MCC will be isolated from corncob in alkali treatment with variation of NaOH concentration with 4%, 6%, 8%, 10%, 12%, 14 and 17%. Dewaxing, dehemiselulosa, delignifikasi and bleaching is the stage of cellulose isolation. The MCC particles were extracted by acid hydrolisis with 0.1N HCl ratio 1:2 and refluxed at 80 0C for ± 2 hours. The processing parameters like acid concentration, temperature, time and mechanical force were kept constant. The MCC particles were studied by FTIR, XRD and SEM analysis concentration.

2. Experimental details

2.1. Raw material

Corncob from Milango Village, Pohuwato District, Gorontalo Province, Indonesia. The reagents were of analytical grades, NaOH (reagent grade, 98%), HCl (ACS reagent, 37%), H_2O_2 (technical grade), $K_2Cr_2O_7$, H_2SO_4, Avicel 102 (technical grade), Ethanol (95% pa), Tholuena (95% pa), and distilled water used as solvent.

2.2. Isolation of cellulose

Corncob waste was cut into small pieces ± 2 cm, washed with aquadest and in oven temperature 60 0C-70 0C for 24 hours. The dried sample is blended and in the shaked to obtain corncob powder particles 180 micron by size. Cellulose isolation is a modified procedure [12-14] dewaxing stage: 50 gr of 180 micron corncob powder was soxhleted in ethanol : tholuene (2:1) for 4 hours. Sample was filtered and washed until neutral pH, then it was dried with oven at 60 0C for 8 hours. de-hemicellulose stage: 10 gr of 180 micron corncob powder was dissolved into 200 mL of 4%, 6%, 8%, 10%, 12%, 14% and 17% NaOH solution. The mixture was heated on hot plate at 1000C for ± 2 hours. Then it was filtered and washed until neutral pH, dried with oven at 60 0C. de-lignification and bleaching stage using H2O2 4% for ±1 hour at room temperature. Then sample was filtered and washed until neutral pH. It was oven-dried at 60 0C for 8 hours.

2.3. Production of alpha cellulose

Purification of α-cellulose was done by 5gr of cellulose dissolved in 500 ml of 17.5% NaOH at a temperature of 80^0C for 30 min. Furthermore the precipitate was filtered and washed until neutral pH, and oven-dried at 60^0C for 8 hours [12-14].

2.4. Production of microcrystalline cellulose (MCC)
The preparation of MCC modification of the analytical procedure [9,12,17]. α-cellulose product was hydrolysed with 0.1N HCl ratio 1: 2 and refluxed at 80 ^{0}C for ± 2 hours. The precipitate was filtered and washed with aquadest until neutral pH and oven-dried at 60 ^{0}C for 8 hours.

2.5. Characterization of MCC fiber

2.5.1. Fourier Transform Infrared (FTIR). FT-IR spectroscopy analysis was used to determine the cellulose microcrystalline functional group were carried out using (Shimadzu IR-Prestige21, Instrumen Laboratories Brawijaya University, Malang). Bands were recorded in the region from 4000 to 500 cm^{-1}

2.5.2. X-ray diffraction (XRD). Morphological changes in the crystalline structur of MCC fiber were analyzed using a hight-resolution X-ray difractometer (PANalytical, X´Pert HighScore, at Geologi Laboratories, Bandung). The crystallinity of MCC fiber was determined from X-ray diffraction curves based on the Segal Method (Segal et al, 1959) in Kyoung-Hwa Choi, et al, 2016)[16]. Crystallinity index (CrI) was calculated based on Eq.1

$$CrI\ (\%) = I_{002} - I_{Am}/I_{002}\ x\ 100 \quad (1)$$

were I_{002} is the peak height at 22.4 (2θ) and I_{Am} is the peak height of amorphous cellulose to 19.5 (2θ).

2.5.3. Morphologycal characteristic. SEM was used to examine the microscopic structure and the surface morphologyof MCC fiber. The instrument used was SEM-EDS JEOL JSM-6360LA, Japan, at Geologi Laboratories, Bandung.

3. Result and discussion
Effect of alkalin charge on the isolation of cellulose and α-cellulose levels. NaOH is a cellulose-swelling complex [14], improving particle dispersion to form alkali-cellulose by the reaction of Cel-OH + NaOH ↔ Cel-ONa + H_2O [21][16]. Alkali as a swelling of the cellulose chain (swelling) and penetrating the crystal lattice reacts with the OH group after breaking the hydrogen bonds producing the cellulose-alkali complex [21][24]. At the dewaxing stage, dehemicellulose, delignification, impurities such as extractives, lignin and hemicellulose decreases. Proteins, inorganic salts, fatty acids, resin acids, waxes, tannins and colored compounds are extracted in both solvents and alkalis. As the NaOH concentration increases, more and more short-chain carbohydrates are degraded [18][33]. NaOH 4% gives the highest α-cellulose content (60%). The swelling of inter-crystalline swell is followed by oxidative degradation which largely confined to the amorphous region of fiber ie the degradation of cellulose intercrystalline. Dissolution of extraactive compounds, hemicellulose and lignin molecules due to the breaking of aryl-ether bonds, carbon-carbon, aryl-aryl and alkali-alkali to obtain high purity cellulose. The higher the concentration of NaOH, the lower the content of α-cellulose will be, because strong degradation occurs through intracrystalline swelling, where the reactant penetrates the crystalline region [18][22][33].

Effect of hidrolisis charge on the yield of MCC. NaOH 4% concentration gives the highest yield of MCC (84.53%).The purity of α-cellulose and hydrolysis processes affects the MCC fibril structure. The hydrolysis level is influenced by the structural properties of the biomass substrate, such as cellulose crystallinity, surface area, DP and porosity [9]. During hydrolysis of HCl, Cl^- ions will penetrate cellulose strands and hydrolyze the amorphous regions resulting in highly crystalline substrate with different CrI values [16] Concentration NaOH 10%-17% extensively alters the cellulose molecular structure and supramolecules of cellulose I into cellulose II with decreased crystallinity [9].

3.1. Infrared spectroscopy
FT-IR spectra of the MCC fiber and their intermediate products are shown in Figure 1.

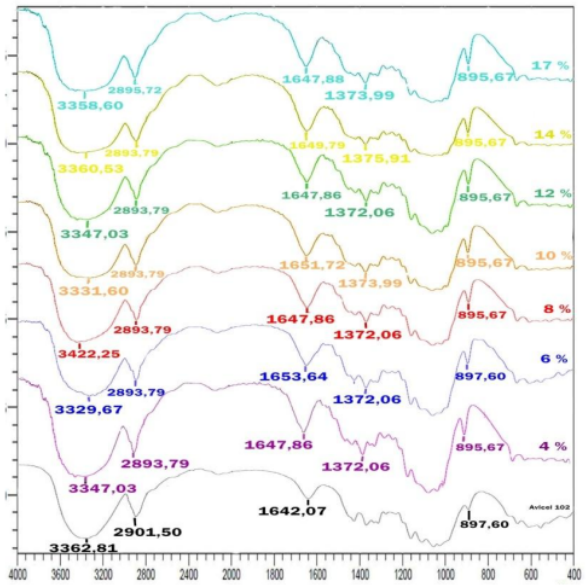

Figure 1. FT-IR Spectra of MCC fiber

Spectra at 4000-2995 cm-1, 2900 cm-1, 1430 cm-1, 1375 cm-1, and 900 cm-1 indicate crystal and amorphous regions. There are structure changing of celluolose I to cellulose II with NaOH 6%, which is showed by stretching OH at 3331 cm-1, 3347 $cm^{-1,}$ 3360 cm-1 and stretching C=O at 1642-1649 cm^{-1}. The characteristics of the glycosidic group of C-O, C-O-C, and C-OH bonds with concentration variations were seen at 1161.83 cm^{-1}. The formation of alkali-cellulose is reinforced by stretching of C-O and C-C groups at 1063-1065 cm^{-1}. For NaOH 4% and 6%, there are sharp peak at 897 cm which typical for β-glycosidic linkages of cellulose I. Cellulose with NaOH ≥6%, showed peak shifts at 998 cm^{-1}, 995 cm^{-1}, 895 cm^{-1}. This is indicate occurrence of cellulose crystal structure II. This peaks correspond to cellulose spectrum [29].

3.2. X-ray diffraction (XRD)

X-ray diffraction spectra of MCC fiber and their intermediate products are shown in Figure 2. The XRD analysis results (Fig.2), gives the amorphic form data at 2θ=14.6^0(4%), 12.1^0(6%), 11.9^0(8%), 19.5^0(10%), 12.2^0(12%), 12.1^0(14%), 12.3^0(17%) and the crystal is read at 2θ=22.4^0(4%), 21.9^0(6%), 21.8^0(8%), 19.8^0(10%), 21.8^0(12%), 21.8^0(14%), 19.7^0(17%). NaOH 4% showed typical diffraction pattern of cellulose I with angle diffraction angle 2θ ie 14.84^0, 20.20^0, 22.63^0. Increasing the concentration of NaOH (≥4%) will decrease intensity and diffraction pattern formed two new crystallinity peaks ie crystal structure II at angle 2θ=$12,5^0$ and 21.68^o [9][31] micro-fibril swelling, degradation and formation of new crystal lattice [33] NaOH 17.5% during the α-cellulose isolation process [5]. The highest crystal structure produced on MCC due to the removal of amorphous alpha cellulose region with acid hydrolysis and 8% NaOH concentration yields a degree of crystallinity (CrI) of 98%. It is interesting to note that the MCC in the present case shows doublet in the intensity of the main peaks corroborating the coexistence of cellulose I and cellulose II allomorphs. The large different spectra between MCC with cellulose and alpha celluloseresults in the appearance of two significant peaks indicating the highly crystalline structure of MCC. High crystallinity indicates an ordered compact molecuar structure, whenthe crystalline cellulose content is high, these two peaks are more pronounced [7].

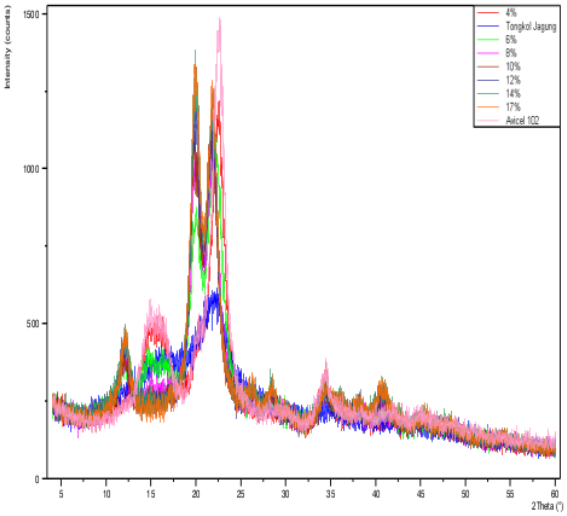

Figure 2. XRD Spectra of MCC fiber

3.3. Scanning Electron Microscopic (SEM)

Figure 3 shows themicroscopic structure and surface morphology of MCC fiber.

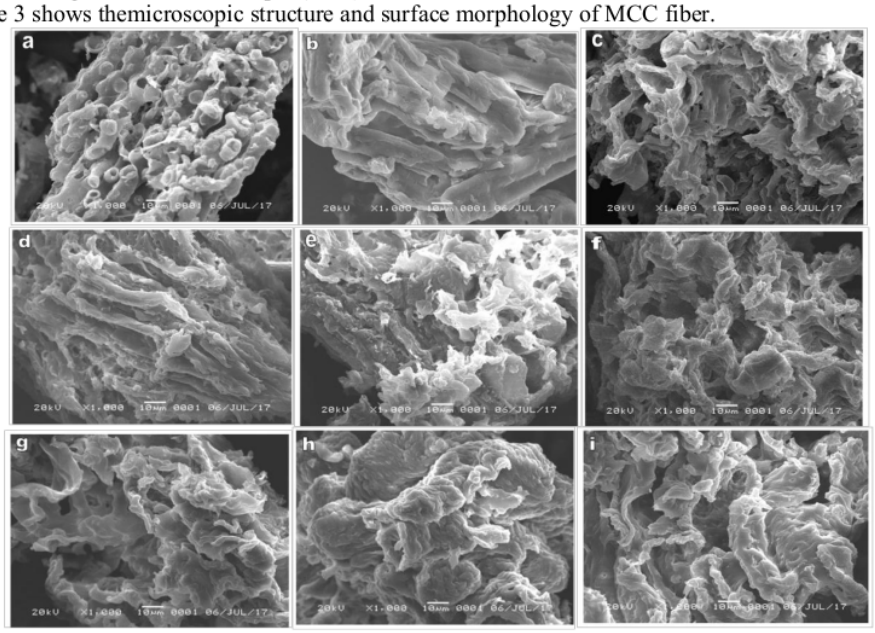

Figure 3. Photo SEM (a) Corncorb, (b) Avixel 102, (c) NaOH 4%, (d) NaOH 6%, (e) NaOH 8%, (f)NaOH 10%, (g) NaOH 12%, (h) NaOH 14%, (i) NaOH 17%.

Micrographic SEM reading (Fig.3) NaOH 6% has a surface structure similarity with Avicel 102. The 8%, 10%, 12%, 14% and 17% NaOH concentrations illustrate the porous (vascularular) texture of the macrofibril shape and the unequal distribution of macrofibrils in cellulose suggest that

mercerization causes fibrillation and fiber damage to small pieces that increase surface area.In this case, after macrofibrils werehydrolyzed and rinsed, the volume of amorphous cellulose was occupied by water molecules that were then removed;the remaining macrofibrils contain large amounts of naked microfibril bundles.

4. Conclusion

The MCC particles were studied by FTIR, XRD and SEM analysis. FTIR spectra showed that each MCC had -OH group at wave number 3422, 3331, 3347, and 3360 cm^{-1}.The C-0 bond at 1635 cm^{-1}, 1642-1649 cm-1 shows the different stretching of C-O on cellulose fiber I and cellulose II and C-0 bonds at 1161.83 and 1063-1065 cm^{-1}. 995-895 cm-1 shows a change in rotation of glucose residue around the glycosidic bond into Cellulose II. SEM analysis showed that NaOH 6% had a standard pore cell surface structure. Acid hydrolysis and NaOH concentration increase the CrI of MCC. I_{002} is the peak height at 22.4(2θ) and I_{Am} is the peak height of amorphous at 19.5(2θ). The highest crystal structure produced on MCC due to the removal of amorphous alpha cellulose region with acid hydrolysis and 8% NaOH concentration yields a degree of crystallinity (CrI) of 98%.

Acknowledgments

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