Study of Crystallinity Extract Cellulose from Corn Stalk Fiber

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Abstract. Study of crystallinity Extract Cellulose was carried out. Cellulose extracted by Corn Stalk Fiber using the ‘'Peracid−Hydroxide−Hypochlorite (P−H−H)' method and characterized with FTIR and XRD. IR spectra showed strong absorption at 3415.93 cm^{-1} which is attributed to O−H stretching vibration (intermolecular hydrogen bonds), vibrations at 1028.06 cm^{-1} is assigned for C−O−C stretching which appears almost simultaneously with the vibration located at 993.34 cm^{-1} (Indicate β−1.4−Glycosidic bond by Cellulose). The particle size of Cellulose which is 20.95 nm and has a crystallinity phase of 66.18%. These results indicate that extract cellulose by the P−H−H method has good material crystallinity.

Keywords: *Cellulose, Corn Stalk Fiber, Crystallinity.*

1 Introduction

Cellulose is the most abundant chemical compound in nature, especially as a constituent of plant cells [1]. Cellulose content in fibrous plant parts, which is as a constituent of 40−50% Wool, 80% hemp, and 90% cotton fiber. Cellulose has a chemical structure consisting of β−1.4 glycosidic bonds [2].

Cellulose which has very compatible, biodegradable, good thermal stability, stable chemical properties, and very cheap [3] to be used as an efficient renewable material, has good crystallinity, and has mechanical properties [4]. Cellulose−based materials, namely nanocrystal cellulose [5][6][7] which have been developed into materials, such as aerogels [8][9][10].

Cellulose was extracted by corn stalk fibers from agricultural waste products in the North Maluku, Indonesia. we used 'Peracid−Hydroxide−Hypochlorite (P−H−H) method, contained in a document of the United States Patent **4,400,237** [11], to get a high percentage of extract cellulose. Characterization with FTIR and XRD to determine the functional group, crystal size, and crystallinity phase of extract cellulose.

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2 Material and Methods

2.1 Material Preparation

Prepared 2 kg of corn stalk fiber which has been cutting with a size of 1−3 cm, dried in an oven at a temperature of 100 ºC for 16 hours to remove the moisture content in the fiber of the corn stalk to obtain a constant sample weight. Then mashed with a blender and sifted using an 80 mesh sieve to obtain powder of corn stalk fiber.

2.2 Cellulose extraction

2.2.1 Delignification

As much as 5 grams of fiber powder of corn stalks are put in a 250 ml cup glass with 0.5 mL of acetic acid added and then heated using a hot plate at 70ºC while stirring with a magnetic stirrer for 1 hour. Then the addition of 6% NaOH as much as 200 mL the temperature is increased to 80ºC for 1,5 hours while stirring. Filtering well done using filter paper, then the precipitate is washed with distilled water until a neutral pH is measured using a pH meter.

2.2.2 Bleaching

Putting the residue into a 250 mL beaker then adding it with a 0.4% NaOCl solution of 150 mL and heating it at 40ºC while stirring for 3 hours. The precipitate is filtered and washed with Aquabides until a neutral pH value obtained. The precipitate is left for 1 hour and taken as cellulose extract.

2.3 Characterization

The extract Cellulose was characterized using *FTIR SHIMADZU spectrophotometer*, to find out the typical functional group of cellulose start at a wavelength on 4000 cm^{-1}–400 cm^{-1} and crystallinity is characterized using *XRD-7000 - Shimadzu Scientific Instruments*, at wavelength between the angle theta (θ) 10^0– 40^0.

3 Results and Discussion

3.1 Extract Cellulose

Extract Cellulose has been obtained using the method of *Peracid−Hydroxide−Hypochlorite* (*P−H−H*). The percentage of cellulose was obtained by 89% which showed that Lignin and hemicellulose were degraded well in the delignification and bleaching process. Acetic acid and NaOH can increase the release of lignin from cellulose and reduce the degradation process in the cellulose chain [12][11].

3.2 FTIR Characterization

FTIR characterization based on the cellulose structure shown in Figure 1.

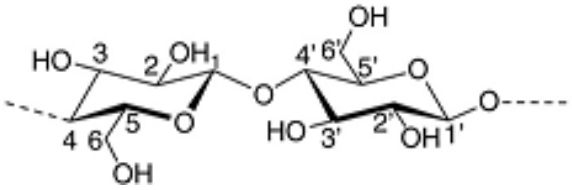

Figure 1. Chemical structure of cellulose. (Kang, H. et al., 2015 on [13])

Cellulose has an absorption area, which is a functional group area at a wavelength of 3500 cm^{-1}−1500 cm^{-1}, fingerprint region at a wavelength of 1500 cm^{-1}−1000 cm^{-1}, and Aromatic region at a wavelength <100 cm^{-1}.

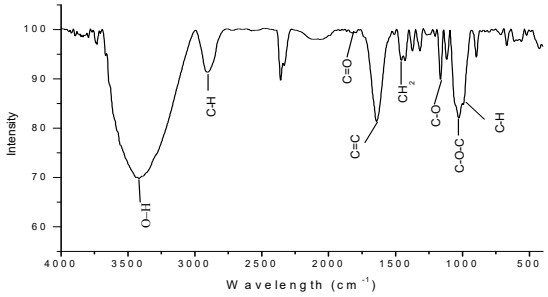

Figure 2. FTIR Cellulose spectra from corn stalk fibers

Table 1. Type of bonding and vibration mode derived from FTIR spectra of Cellulose in figure 2.

Wavelength (cm^{-1})	Type of bond	Vibration mode
3415.93	O−H	stretching vibration
2092.87	C−H	Bending vibration
1730.15	C=O	stretching vibration
1643.35	C=C	Streching ring
1456.26	C−H	CH_2 deformation vibration
1165	C−O	various bands
1028.06	C−O−C	stretching vibration
993.34	C−H	Aromatic plane bending

Indicate β−1.4−Glycosidic bonds on cellulose in the wavelength region of 1028.06 cm^{-1} which is simultaneous with a wavelength of 993.34 cm^{-1}. Stretching vibration at wavelength 3415.93 cm^{-1} shows the interaction between molecules in hydrogen bonds in the chemical structure of cellulose.

3.3 XRD Characterization

The XRD spectra of cellulose have a characteristic that is determined based on the muller index (hkl) that appears at the peak, on 110, 110, 200 [14]., 110,110,102, and 004 [15] Characteristics of cellulose crystals are triclinic and monoclinic [16]. XRD spectra below in figure 3.

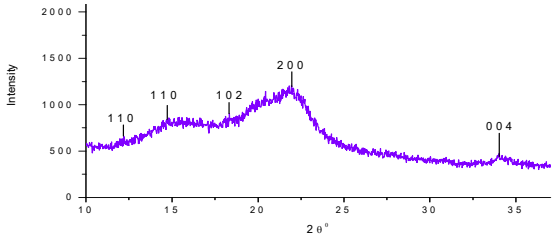

Figure 3. Spectra of XRD Cellulose from corn stalk fiber

Miller indices (hkl) show peaks 110, 110, 102, 200, 400 with angles theta (θ) 12.16º, 14.75º,18.36º, 21.96º, 33.95º in figure 3. The small diffraction peaks indicate areas of high crystallinity [17].

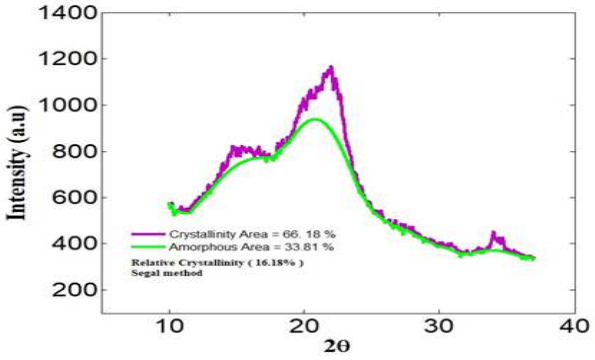

Figure 4. Interpretation of MATLAB to the crystallinity of cellulose

Table 2. Crystal size, crystallinity, Amorphous

Crystallitesize (nm)	Crystalinity (%)	Amorphous (%)
20.95 nm	66.18	33.81

Crystallinity and Amorphous cellulose areas shown in Table 2 are calculated using the MATLAB program (Operating by Department of Physic, Hasanuddin University, Makassar, Indonesia) on figure 4. Relativity of crystallinity with the Segal method is 16.38%. Crystallinity Relative has a low percentage indicating intermolecular force in the cellulose structure is a very strong.

4 Conclusion

We studied of crystallinity of cellulose and characterization with FTIR and XRD. MATLAB program used to determine crystallinity and amorphous on cellulose. These result we have a percentage of cellulose is very high and has been good crystallinity properties.

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