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Effect of H₂SO₄ concentration on cellulose isolation from palm empty fruit bunches.

N Nilawati¹, R Rahmi^{1,*}, L S Desiyana²

¹Chemistry Department, Syiah Kuala University, Banda Aceh, Indonesia

²Pharmacy Department, Syiah Kuala University, Banda Aceh, Indonesia

E-mail: rahmi@fmipa.unsyiah.ac.id

Abstract. Isolation of cellulose from palm empty fruit bunches had been conducted. Isolation was conducted with H₂O₂ 30% and H₂SO₄ with various concentrations (20, 30, 40, and 50%). Hydrolysis and bleaching processes were performed for 90 minutes. The obtained cellulose was characterized by Fourier Transform Infrared (FTIR), X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM). FTIR analysis confirmed the typical bands of cellulose in the sample. XRD patterns showed increasing H₂SO₄ concentration on hydrolysis process increased crystallinity of cellulose. However, at H₂SO₄ concentration more than 40% the crystallinity of cellulose reduced. It was due to the reduction of crystalline part of cellulose.

1. Introduction

Indonesia is a country that has the largest palm oil plantation in the world. In 2015, Indonesia has an area of 6,735,300 hectares of oil palm plantations spread over 22 provinces with 31.07 million tons of palm oil production per year. Oil palm plantations in Indonesia are expected to continue to rise, as it is a potential producer of vegetable oils. This palm oil processing industry produces a very large amount of solid waste. Several types of solid waste were palm empty fruit bunches, coir, mud, palm shells, etc., Palm empty fruit bunches are the largest solid waste. The average production of palm empty fruit bunches is 22% to 24% of the total weight of fresh fruit bunches processed at the palm oil industry.

Palm empty fruit bunches are composed of several important components that can be utilized and processed into other materials with more economical value. The constituent components include cellulose, lignin, holocellulose, hemicellulose, water and other extractive substances. One of the most important components of palm empty fruit bunches that can be converted to be another valuable product is cellulose. Cellulose is a natural polymer that can be used as raw material for bioplastics [1-4], bioethanol, and food packaging materials. Palm empty fruit bunches contains 38,76% or about 37,50% of cellulose with fiber content reach 72,67%.

* Corresponding Author: rahmi@fmipa.unsyiah.ac.id



In order to obtain pure cellulose from raw material, some methods can be applied such as chemical method, mechanical method and biological method. One of chemical methods is strong acid hydrolysis. This method is the most commonly method for cellulose purification where strong acid easily hydrolyze the amorphous part of the cellulose fiber and hemicellulose.

In our previous work [5], cellulose had been isolated from palm empty fruit bunches using HCl. However, the crystallinity of cellulose obtained was very low. Crystallinity is very important to show the level of purity of cellulose and it will affect the mechanical properties of cellulosic products [6]. Rashid (2017) reported that cellulose isolation process from cotton wool using different concentrations of acid resulted cellulose with different crystallinities [7]. Therefore, in order to study the effect of acid concentrations on cellulose isolated from palm empty fruit bunches, the hydrolysis process was conducted with several concentrations (20, 30, 40 and 50%) of H_2SO_4 in this work. The obtained cellulose was characterized by XRD, FTIR and SEM.

2. Materials and Methods

2.1. Materials

Palm empty fruit bunches were collected from Aceh. Sulfuric acid and hydrogen peroxide were analytical grade.

2.2. Isolation of cellulose

Palm empty fruit bunches were washed with distilled water and dried. Dried palm empty fruit bunches were cut into small pieces. The small pieces of palm empty fruit bunches were hydrolyzed using H_2SO_4 with various concentrations (20, 30, 40, and 50%). Where 3g of the small pieces of palm empty fruit bunches were added into beaker glass containing 26 mL of H_2SO_4 and stirred for 90 min. The hydrolysis process was stopped by adding frozen distilled water to the mixture. The mixture was filtered and rinsed several times until neutral pH was reached. After hydrolysis process, the hydrolyzed sample was bleached with H_2O_2 30% for 1h at room temperature. The obtained cellulose was dried in a oven for 6 h at 40°C .

2.3. Characterization

a. Fourier Transform Infrared (FTIR)

FTIR analysis was performed using Cary 630 Fourier Transform Infrared Spectrometer (Agilent Technologies). The sample was placed in a set holder and spectral scanning was conducted in a wave number ranged from 4000 to 600 cm^{-1}

b. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis was done to determine the crystallinity of cellulose. XRD patterns were recorded using the Shimadzu XRD-700 Series X Ray Diffractometer. It was operated at 40 kV and 30 mA producing $\text{CuK}\alpha$ with $\lambda = 0.154\text{ nm}$ using a step size of $0.02^\circ/\text{minute}$ in the range of $2\theta = 10-70^\circ$. The crystallinity of cellulose can be determined using a formula :

$$C = \frac{I_{200} - I_{\min}}{I_{200}} \times 100\% \quad (1)$$

I_{200} was found between scattering angles of $2\theta = 22^\circ$ and 23° . $I_{\min}$ was obtained between $2\theta = 18^\circ$ and 19° [6]

c. Scanning Electron Microscopy

SEM analysis was conducted to determine the morphology of cellulose. Cellulose was affixed to the set holder with double adhesive, and coated with gold or palladium metal in a vacuum. Micrographs were obtained with JSM-6510A/JSM-6510LA (Analytical/Analytical low vacuum SEM) at 500 $\times$ magnification.

3. Results and Discussions

3.1 XRD

In order to study the crystallinity of cellulose obtained from palm empty fruit bunches, the XRD analysis was performed. The results were shown in Figure 1. Figure 1 showed typical XRD patterns of cellulose at $2\theta=15^\circ$ and 22° . Figure 1(a) was XRD pattern of palm empty fruit bunches without hydrolysis process. Figure 1(b), 1(c), 1(d) and 1(e) were XRD patterns of cellulose isolated from palm empty fruit bunches using H_2SO_4 20, 30, 49 and 50% , respectively. Compared with hydrolyzed cellulose, the XRD pattern of palm empty fruit bunches without hydrolysis process showed broader peaks. It indicated the crystallinity was lower than hydrolyzed cellulose. Increasing H_2SO_4 concentration increased the crystallinity of cellulose. However, the crystallinity of cellulose decreased at H_2SO_4 concentration 50%. The increasing in crystallinity was due to the reduction of some components contained in the sample such as lignin, pectin and the amorphous part of cellulose during hydrolysis process. Cellulose contains amorphous and crystalline part. However, in high acidic concentration the cellulose will be decomposed and resulting lower crystallinity.

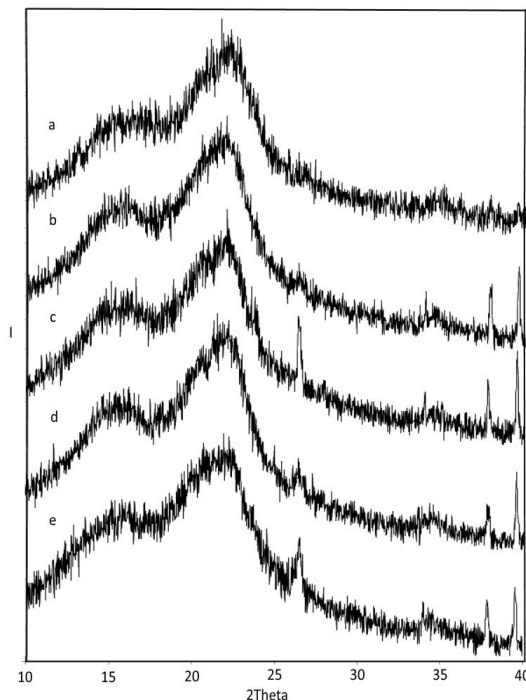


Figure 1. XRD patterns of cellulose without hydrolysis (a), hydrolyzed with H_2SO_4 20% (b), 30% (c), 40% (d) and 50% (e).

Table 1. The yield and crystallinity of cellulose isolated from palm empty fruit bunches (initial weight was 3g)

Cellulose	Yield (g)	Crystallinity (%)
H ₂ SO ₄ 20%	1.564	61
H ₂ SO ₄ 30%	1.532	63
H ₂ SO ₄ 40%	1.512	66
H ₂ SO ₄ 50%	1.492	65

Hydrolysis at high concentration of acid caused the chain opening of cellulose that change the arrangement of polymer chains and cellulose was more easily degraded. The reduction of these components resulting reduction of the yield of cellulose obtained as shown in Table 1. The increasing H₂SO₄ concentration decreased the yield of obtained cellulose. Based on Figure 1 and Table 1, the highest crystallinity of cellulose was obtained at H₂SO₄ concentration of 40%.

3.2 FTIR

FTIR analysis was conducted in order to study the functional groups contained in the cellulose. The results were shown in Figure 2. Figure 2 showed FTIR spectra of palm empty fruit bunches (a) and cellulose isolated from palm empty fruit bunches (b).

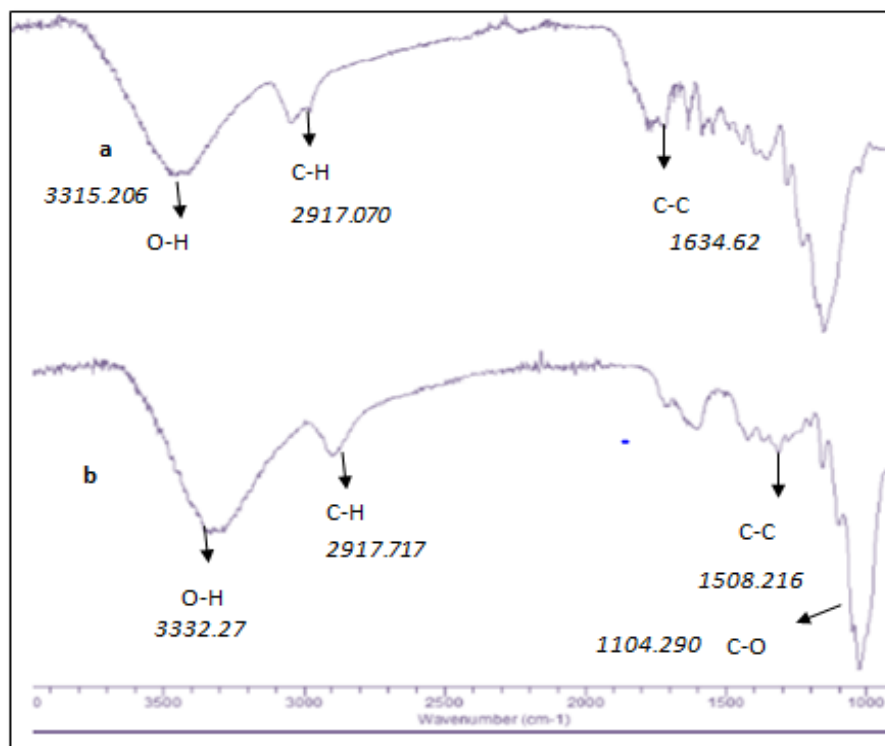
**Figure 2.** FTIR Spectra of (a) palm empty fruit bunches (b) cellulose isolated from palm empty fruit bunches

Figure 2b showed absorption band at wave number of 3332.273 cm⁻¹ attributed to -OH vibration. The absorption band of -OH vibration was also found in FTIR spectrum of palm empty fruit bunches with lower wave number (3315.206 cm⁻¹). It was due to the removal some components of palm empty fruit bunches after hydrolysis process. Absorption bands at wave

numbers $1300\text{--}1400\text{ cm}^{-1}$ in FTIR spectrum of cellulose can be assigned to -O- which linked with carbon chain in the cellulose. The absorption bands at 2917.717 and 1023.053 cm^{-1} corresponded to -CH_2 and C-O vibrations, respectively. These results showed typical FTIR spectrum of cellulose. The results obtained in accordance with research conducted by Isroi [8].

Compared with FTIR spectrum of cellulose isolated from palm empty fruit bunches, the FTIR spectrum of palm empty fruit bunches showed some differences. The differences were due to the reduction of lignocellulosic component after hydrolysis process. The result was in agreement with FTIR spectrum of cellulose isolated from palm empty fruit bunches using alkaline [9] and cellulose isolated from sugarcane bagasse [10].

3.3. SEM

The morphological observations were performed using Scanning Electron Microscope (SEM). SEM images of cellulose isolated from palm empty fruit bunches were shown in Figure 3 (magnification $500\times$) and 5 (magnification $2000\times$).

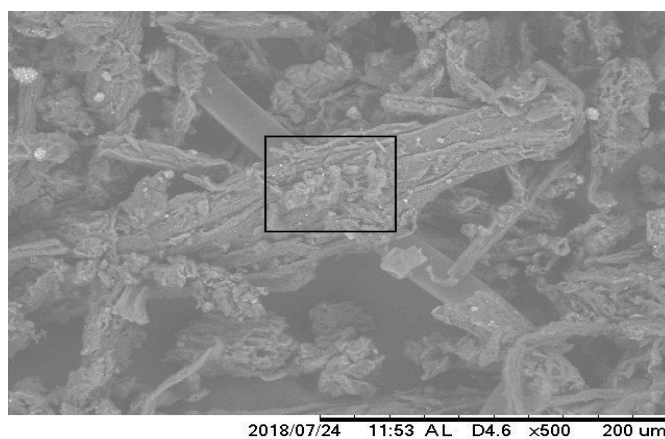


Figure 3. SEM image of cellulose isolated from from palm empty fruit bunches at $500\times$ magnification.

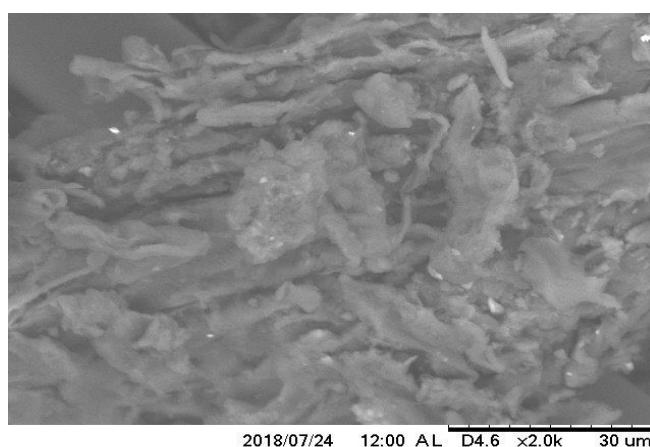


Figure 4. SEM image of cellulose isolated from palm empty fruit bunches at $2000\times$ magnification.

Figure 3 showed the existence of various particle sizes of cellulose. SEM of cellulose particles showed that they are irregular in shape and polydisperse. The different in particle size was found due to hydrolysis process that could reduced the particle size.

SEM images also showed amorphous and crystalline part of cellulose as shown in Figure 3. At magnification 2000x showed amorphous part of cellulose. Figure 3 and 4 indicated the cellulose has been successfully isolated from palm empty fruit bunches. The results are consistent with results reported by Xie [11] for cellulose isolated from bamboo using microwave liquefaction combined with chemical treatment and ultrasonication.

4. Conclusions

Isolation of cellulose from palm empty bunches with variation of H_2SO_4 concentration has been successfully conducted. FTIR analysis confirmed the functional groups contained in cellulose isolated from palm empty fruit bunches. XRD analysis showed crystallinity of cellulose influenced by H_2SO_4 concentration used in hydrolysis process. The highest crystallinity was found at H_2SO_4 concentration of 40%. SEM images confirmed the morphology of cellulose isolated from palm empty fruit bunches with different particle sizes due to hydrolysis process. These results indicate palm empty fruit bunches can be used as a potential source of cellulose.

5. References

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