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## Isolation and Characterization of Cellulose from Banana Stems using Microwave Heating

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### Abstract

During each day of harvest, wasted banana stems are obtained in large quantities. These stems are composed mainly of 74.37% cellulose which is a very important raw material. This study aims to isolate cellulose from banana stems using liquefaction, delignification and bleaching processes with a microwave at power variations of 450, 600 and 800 W. The results showed that the highest cellulose content of 86.43% was obtained at 800 W for 14 minutes. Meanwhile, the fourier-transform infrared spectroscopy (FTIR) analysis result did not show a peak at wavenumber 1519 cm<sup>-1</sup> which is the specific peak for lignin but showed a peak for cellulose at wavenumber 898 cm<sup>-1</sup>. Furthermore, XRD analysis of crystallinity showed a typical diffraction peak of cellulose at 22.5° with a degree of crystallinity of 56.8% while, morphological analysis with SEM showed that the sizes of the cellulose fibers produced varied, ranging from 5 to hundreds of micrometers and visible fibrillary fibers

**Keywords:** Banana stem, microwave, cellulose

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### 1. INTRODUCTION

Bananas have been widely planted by farmers because of its numerous benefits and high nutritional value. In Indonesia, there are 13 varieties of banana with extensive uses such as leaves, heart, tubers and roots which are currently been used by the community (Erawan *et al.*, 2019). Based on data obtained from the Central Statistics Agency and the Directorate General of Horticulture (2019), Indonesia produces 7 million tons of banana from 107.683 ha harvest area with a productivity of 67.46 tons/ha.

The use of banana trees has focused more on the fruit and leaves, other parts such as the stem have not been properly utilized. Banana stems are waste product obtained in large quantities during harvest, which makes disposal difficult (Raju *et al.*, 2019). Therefore, the utilization of these stems into a more useful material will increase its added value. Studies have been previously carried out on the use of banana stems, such as electrodes

for supercapacitors (Taer *et al.*, 2018), nutraceutical (Ramu *et al.*, 2017), biofilters (Sulastris and Rahmidar, 2016), antioxidants (Budi *et al.*, 2015), paper (Malinen *et al.*, 2014) and natural coagulants for wastewater treatment (Alwi *et al.*, 2013).

Banana stem contains 3% nitrogen (N), 0.40% sulfur and 0.25% phosphorus with protein content 8.98% being the highest (Rochana *et al.*, 2017). The largest component contained in banana stem is cellulose, which account for 74.37% of its total composition (Lange *et al.*, 2018). Therefore, the large quantities of banana stem waste produced have high cellulose fiber content which is the main source in producing cellulose.

Cellulose provides protection and support for cells and tissues. It is composed of D-glucose subunits which are linked to each other due to the presence of  $\beta$ - (1 $\rightarrow$ 4)-glycoside bonds. Cellulose is most commonly found in stalks, stems, or all woody parts (Lehninger 1993) of plants and also present in

the cell wall. Furthermore, it has been widely applied in many fields including biomedicine (Du *et al.*, 2019), packaging industry (Yadav dan Chiu, 2019) and as a drug carrier (Weyell *et al.*, 2019).

Various methods are used to isolate cellulose including enzyme hydrolysis (Mukwaya *et al.*, 2015), alkaline treatment (Szyma *et al.*, 2017) and the combination of liquefaction, alkali and chlorite treatments (Xie *et al.*, 2016). The methods mentioned above use conventional heating except for the combination of liquefaction, alkaline and chlorite treatments. Meanwhile, heating with microwaves reduce the time of the insulation process and provide more efficient heat (Xie *et al.*, 2016; Sudiana *et al.*, 2017).

The use of microwaves as a substitute for conventional heating has been carried out in several studies including lignin isolation (Zhou *et al.*, 2017), bioconversion of lignocellulose to glucose (Sudiana *et al.*, 2017) and isolation of cellulose from bamboo (Xie *et al.*, 2016).

It was discovered that heating with microwaves increase the rate of the reaction. This is because microwave energy directly interacts with the reaction mixture, causing a more efficient and faster heating process which requires less energy. Meanwhile, conventional heating requires more energy (Sweygers *et al.*, 2018). Based on the study carried out by Xie *et al.*, (2016), it was reported that heating using microwaves increase the percentage of cellulose content and reduce lignin content. Therefore, they are very effective for fast isolation of lignin and short heating with high lignin yield (Zhou *et al.*, 2017).

From previous studies carried out in relation to the isolation of cellulose from banana stems, none has been able to utilize microwave heating. Therefore, in this study, the isolation of cellulose from banana stem was carried out by utilizing microwave heating in all treatments, which include liquefaction, delignification and bleaching. An evaluation was also carried out on microwave power and cellulose content.

## 2. MATERIALS AND METHODS

### Tools and Materials

The tools used in this study include Microwave Oven (ME731K / XEU Solo, 20 L), oven and glassware. The materials used include glycerol (Bratachem), methanol p.a

(Merck), NaOH (Merck), H<sub>2</sub>O<sub>2</sub> 30% (Merck) and 96% H<sub>2</sub>SO<sub>4</sub> (Merck). The banana stems used were those of the Uli banana species (*Musa paradisiaca sapientum*) aged 15 months which were obtained from the Plantation Center for Tropical Horticulture Studies, IPB University.

### Banana Stem Preparation

The banana stems were cut into small pieces and left to dry in the sun. The dried stems were mashed and sieved with a sieve size of 70 mesh, to obtain a 70 mesh stem powder.

### Isolation of Cellulose

The isolation of cellulose involved the following processes, which include liquefaction, delignification and bleaching. The liquefaction process was carried out based on the method of Xie *et al.*, (2016) by putting 10 g of banana stems, 150 mL of solvent (glycerol: methanol = 2: 1) and 35 mL of 1.75% H<sub>2</sub>SO<sub>4</sub> into a beaker. The mixture was heated in the microwave for 3 minutes, filtered, neutralized with methanol and dried in an oven at 105°C. Furthermore, the delignification and bleaching processes were carried out using Purwaningsih method (2012) with modifications to the heating process i.e. from conventional heating to heating using microwaves. The residue was added to 4% NaOH and heated in the microwave for 3 minutes. Furthermore, the sample was filtered using a vacuum pump and the resulting residue was washed with distilled water to a neutral pH (pH 7). The residue was placed in an oven at 50°C to dry until a constant weight was obtained. Afterwards, 5% H<sub>2</sub>O<sub>2</sub> solution was placed in a sample container, heated in a microwave for 4 minutes, filtered and the precipitate washed with distilled water to neutral pH. The treatment with the peroxide solution was repeated twice, simultaneously. Meanwhil, the bleached product was placed in an oven at 60°C to dry until a constant weight was obtained. The power variation on the microwaves consisted of 450, 600 and 800 W. In this stage, the results obtained were characterized.

### Functional Groups with Fourier Transform Infrared (FTIR) Spectroscopy

Cellulose isolates were recorded through a Perkin Elmer Spectrum One FTIR

spectrometer using KBr pellets with a wavenumber between  $4000\text{ cm}^{-1}$  to  $450\text{ cm}^{-1}$ .

### Surface Morphology with SEM (Scanning Electron Microscope)

Morphological observations of cellulose isolates were carried out on a Quanta 650 electron microscope with a 10 kV voltage of 10 kV with 50 times magnification.

### Crystallinity Analysis with X-Ray Diffractometer

The resulting cellulose was analyzed using a wide-angle X-ray diffraction (Bruker D 5000).

Data were generated by a diffractometer with Cu K radiation ( $\lambda = 1.54\text{ \AA}$ ) at 40 kV and 30 mA over an angle range of  $2\theta = 10\text{--}40^\circ$  and a step time of 2.0 seconds. The crystallinity index (CrI) was determined using the Segal *et al.*, (1959) equation, namely:

$$\text{CrI (\%)} = \frac{I_c}{I_c + I_a} \times 100\%$$

Where  $I_c$  is the crystalline intensity and  $I_a$  is the amorphous intensity.

## 3. RESULTS AND DISCUSSION

### Effect of Microwave Power on Cellulose Content

The isolation of cellulose was carried out in three processes, namely liquefaction, delignification and bleaching. Microwave heating was used to replace conventional heating. In conventional heating, delignification process takes 30 minutes and bleaching takes 4 hours, making the total

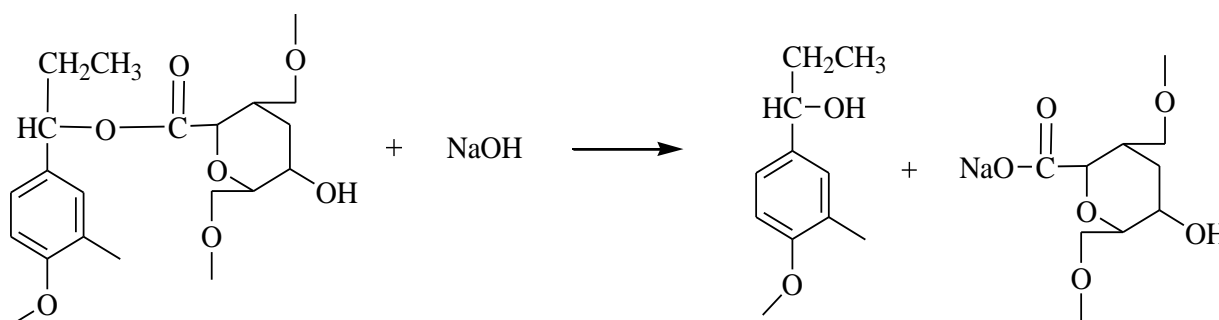
amount of time needed 4 hours 30 minutes (Lange *et al.*, 2018). Meanwhile, heating using microwaves takes 3 minutes for the delignification process and 4 minutes for the bleaching, making the total amount of time needed 7 minutes.

Isolation of cellulose from banana stems was carried out by heating using microwaves in the liquefaction, delignification, and bleaching processes with a total processing time of 14 minutes. In the liquefaction process, an organic solvent with an acid catalyst is used which causes damage to the ester bond between carbohydrates and lignin and the lignin depolymerization. Meanwhile, delignification and bleaching processes were carried out to completely remove lignin and hemicellulose, some of which are still present in the residue (Xie *et al.*, 2014).

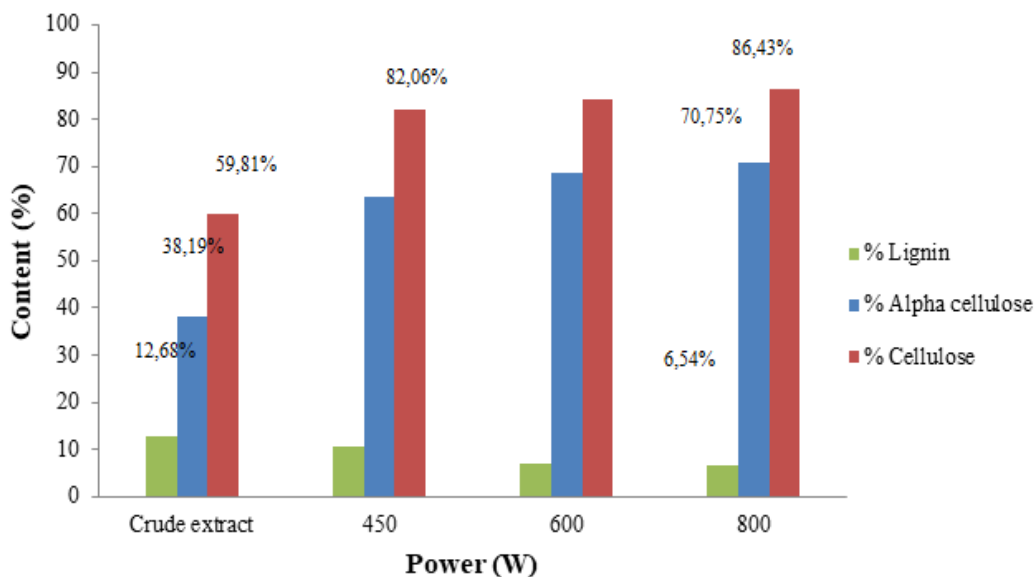
Purwaningsih (2012) reported that NaOH solution removes lignin by 60%. Meanwhile, figure 1 shows that NaOH damages the ester bond between cellulose and lignin. Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) was used in the bleaching process.  $\text{H}_2\text{O}_2$  in an alkaline solution increases the pH of the reaction therefore, making it useful in dissolving most of the hemicellulose as seen in equation (1) below:



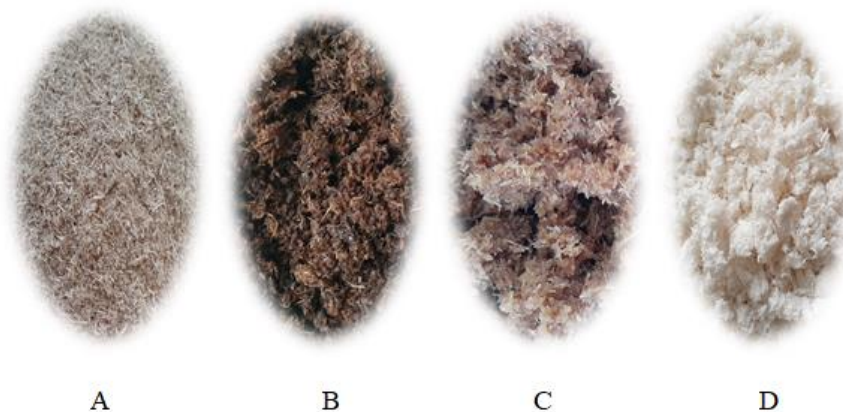
The reaction between  $\text{H}_2\text{O}_2$  and base produces peroxide anions. These peroxide anions ( $\text{HOO}^-$ ) play an important role in the removal of lignin chromophore groups (Sun *et al.*, 2004).



**Figure 1.** Lignin removal reaction with NaOH (Tong dan Hamzah, 1989)



**Figure 2.** Effect of microwave power



**Figure 3.** (A) Crude extract of banana stems (B) after liquefaction (C) after delignification (D) after *bleaching*

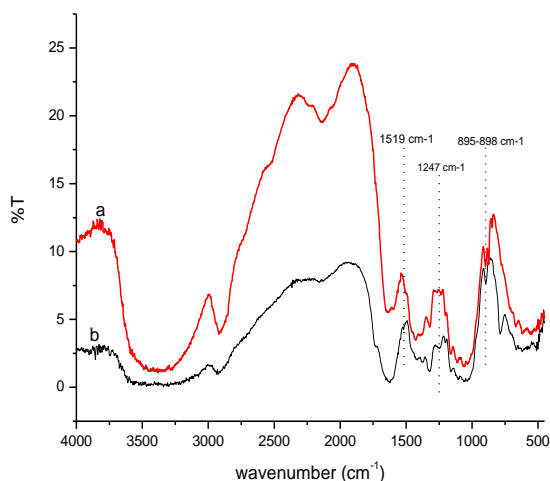
In this study, variations in microwave power were carried out, namely 450, 600 and 800 W. The results of cellulose content are shown in Figure 2, which showed increase cellulose content and a significant decrease in lignin content with increasing microwave power. At a power of 450 W, the cellulose content was 82.06% and when the power was increased to 800 W, the cellulose content increased to 86.43%. This was because heating using microwaves have high efficiency and selectivity to biomass treatment (Zhou *et al.*, 2017). Heating using microwaves produces internal heating in which microwave energy interacts directly with the molecules causing a rapid rise in temperature due to dipole rotation and ion conduction. The mechanism of microwave heating is based on the high rotation of polar molecules in order to experience a collision which converts kinetic

energy into heat. Increased power causes an increase in energy and temperature which damages the fiber and causes lignin to degrade (Sweygers *et al.*, 2018; Zhou *et al.*, 2017; Sudiana *et al.*, 2017).

Figure 3 shows the changes that occurred in the three processes of isolation, which includes liquefaction, delignification and bleaching. After liquefaction, the crude brown banana stem extract turns brownish-black. Meng *et al.*, (2018) reported that the brownish-black color is due to the formation of chromophores such as lignin derivatives. Delignification produces a brown color, which indicates that lignin has not completely disappeared. Furthermore, bleaching was carried out to completely remove lignin and whiten it, indicating that most of the non-cellulosic components and impurities are missing.

### Characteristics by FTIR Spectroscopy

The infrared spectra of crude extract and cellulose are presented in Figure 4. The FTIR spectrum shows that the presence of wave numbers 1519 and 1247  $\text{cm}^{-1}$  in the crude extract, whereas in cellulose it only produces absorption of functional groups at 1235  $\text{cm}^{-1}$  which is a typical lignin peak. The loss of the wavenumber 1519  $\text{cm}^{-1}$  indicates that cellulose was successfully extracted, which is supported by a decrease in lignin content present in the sample. Based on the study carried out by Ndruru *et al.*, (2019) and Meng *et al.*, (2018), the typical peak of lignin is at the wave number 1521  $\text{cm}^{-1}$  and the wave number 1253  $\text{cm}^{-1}$  is the stretch of the carbonyl unit which represents lignin vibrations.



**Figure 4.** FTIR spectrum (a) Cellulose of Banana stem and (b) Crude extract

The wavenumbers 898  $\text{cm}^{-1}$  and 895  $\text{cm}^{-1}$  in cellulose and crude extract, respectively are typical peaks of cellulose. Ndruru *et al.*, (2019) reported that the typical peak of cellulose at the wavenumber 896  $\text{cm}^{-1}$  is known to be a C-O-C bond between anhydroglucose units as a characteristic of the  $\beta$ -(1.4)-glycosidic bond. Some of the typical signals commonly found in cellulose include stretching C-O at 1052-1032  $\text{cm}^{-1}$  and the vibrational stretching of the C-O-C pyranose ring at 1161  $\text{cm}^{-1}$  (Meng *et al.*, 2018). The polymorphic type of cellulose may be determined by the presence of wavenumbers 1428 and 898  $\text{cm}^{-1}$ . Based on the study carried out by Ndruru *et al.*, (2019), wavenumbers 1420 and 896  $\text{cm}^{-1}$  are polymorphic types of cellulose I. Therefore, it is concluded that the type of cellulose used in this study is

polymorphic cellulose I. Resume of functional group absorption is seen in Table 1.

**Table 1.** FTIR analysis functional group absorption on crude extract and cellulose

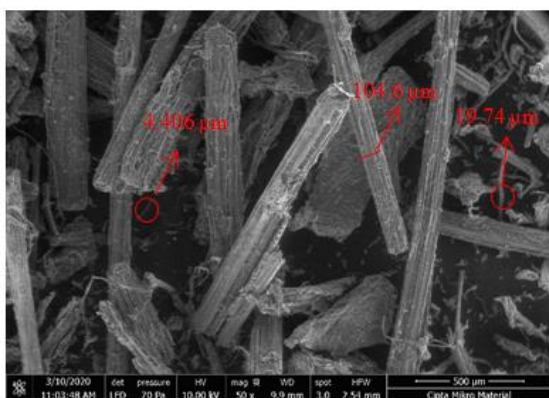
Wavenumber $\text{cm}^{-1}$		Functional groups
Crude extract	Cellulose	
3300	3379	Hydrogen bonds to the OH group in cellulose
2922	2918	Saturated C-H
1519	-	C=C in the lignin aromatic ring
1623	1633	O-H bending in cellulose
1247	1235	C=O stretching
1161	1161	C-O-C vibrational stretching of the pyranose ring
895	898	$\beta$ -(1.4)-glikosidik

### Cellulose Morphology

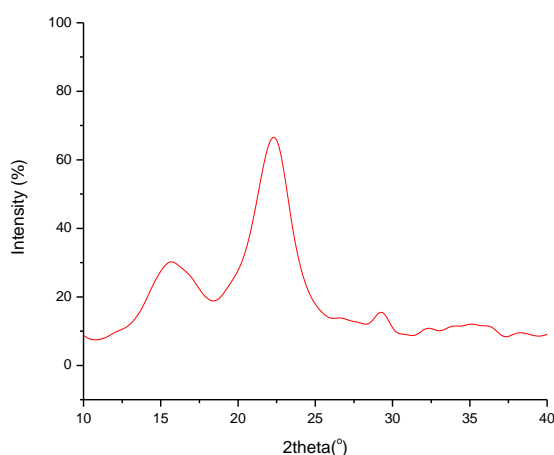
Scanning electron microscopy (SEM) was used to examine the surface morphology of cellulose. Figure 5 shows the morphology of cellulose resulting from the study. Based on the results of SEM analysis after chemical treatment, it was discovered that the banana stem fiber had a rough surface. Meanwhile, based on the study carried out by Li *et al.*, (2015) after liquefaction, the surface became rougher compared to before liquefaction, which showed that most of the hemicellulose was removed. However, a large quantity of small pieces was still present. Meng *et al.*, (2018) made use of banana stem raw materials and reported that the small pieces were lignin and inorganic mineral deposits. In this study, the size of cellulose fibers produced varied, ranging from 5  $\mu\text{m}$  to hundreds of micrometers. The same result was also produced in the study carried out by Faradilla *et al.*, (2016) with banana stem raw materials, where the size of the cellulose produced varied from less than 50 to hundreds of micrometers. The XRD diffractogram (Figure 6) presents the X-ray diffraction pattern of cellulose which shows the highest peaks at intensities of 32, 30 and 14 with angles of  $2\theta$  of 22.5°, 21.9° and 23.5°, respectively. According to Li *et al.*, (2015) and Hayati *et al.*, (2017), it is the characteristics of cellulose show two main peaks, namely 22° and 22.5°. In this study, the



resulting cellulose structure which is in form of crystals with a degree of crystallinity of 56.8%, indicates that the resulting cellulose polymer has a large crystal area compared to the amorphous region, therefore making it stronger and useful as raw material for packaging. This is in accordance with the study carried out by Faradilla *et al.*, (2016) using banana stems as a source of cellulose to be applied as a packaging material, where the cellulose diffractogram showed a peak of 22.2°, with a degree of crystallinity of 57%.



**Figure 5.** Cellulose morphology of banana stems



**Figure 6.** Cellulose XRD diffractogram

#### 4. CONCLUSION

From the results obtained, it was concluded that the isolation of cellulose from banana stems assisted by microwave effectively removes lignin in a shorter time i.e. 14 minutes with a power of 800 W, producing cellulose of 86%. Based on the fiber morphology which showed a hough surface, the sizes of microfibrils produced varied. The results of FTIR spectrometer

analysis showed that cellulose is characterized by wavenumber 898  $\text{cm}^{-1}$ , namely  $\beta$ -(1.4)-glycosidic and lignin is characterized by wavenumber 1519  $\text{cm}^{-1}$ . Furthermore, XRD analysis showed that the highest peak obtained at an angle of  $2\theta$  were 22.5°, 21.9° and 23.5° and the degree of crystallinity produced in this study was 56.89%.

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