Research Article

Production and Characterization of Alpha Cellulose Derived From Rice Straw (*Oryza sativa L.*)

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ABSTRACT

The production of alpha cellulose from rice straw has been done. Alpha cellulose was prepared by maceration of rice straw with ethanol, delignification with 3.5 % sodium hydroxide, extraction of alpha cellulose with 17.5% sodium hydroxide, and bleaching with hydrogen peroxide 20%. In this study, the physicochemical properties of alpha cellulose were evaluated and compared to previous studies. The physicochemical properties include morphology by Scanning Electron Microscopy (SEM), crystallinity by X-ray Diffraction (XRD) analyses, functional groups by Fourier Transform Infrared (FTIR) Spectroscopy, loss on drying, pH and organoleptic. Results showed the SEM image shows the morphology of cellulose fibres, the sample was semi-crystalline with crystallinity index 78 %, the functional groups present in the sample are the same as the functional groups in alpha cellulose, loss on drying 8 is % w/w, pH 7 is and the organoleptic are white powder, odourless and tasteless. The yield of alpha cellulose produced is 30.11 % w/w.

Keywords: Rice straw, alpha cellulose, delignification, bleaching.

INTRODUCTION

Rice straw is the most significant agricultural waste in Indonesia; it has been utilized in the production of bioethanol, microcrystalline cellulose, lactic acid. However, the utilization is still limited, most of them are burned.[1, 2, 3] Contents of sulfur, nitrogen, chlorine to lead the formation of gaseous pollutants such as SO_2, NO_x, N_2O, HCl, and to some extent, dioxins and furans. These pollutants can pose a health hazard.[4]

In general, rice straw (*Oryza sativa*) and other lignocellulose sources contain about 32-47% cellulose, 19-27% hemicellulose and 5-24% lignin. Cellulose is a polymer of β-glucose with β-1-4 bonds between glucose units. Cellulose found in wood, cotton, hemp and other plants. Cellulose is an organic compound found in the cell walls together lignin role in cementing the structure of plants. Wood cellulose generally ranges from 40-50 %, whereas in cotton almost 98 %.[4, 5]

Cellulose consists analytically of three fractions, alpha, beta, and gamma. Alpha cellulose is widely used in the paper industry.[6] Up-scale production has obtained microcrystalline cellulose at the optimal hydrolysis time for 60 minutes using 2.5 N HCl at a temperature of 100 °C from rice straw. Microcrystalline cellulose from rice straw is not significantly different from Vivacel by organoleptic inspection, identification, pH, and dissolved substances in water, solubility, drying shrinkage, apparent specific gravity, starch test, and have as well as the infrared spectrum. In testing the density of compressed, Hausner ratio and Carr's index, there is a noticeable difference between the microcrystalline cellulose from rice straw and Vivacel.[7] The objective of this research is to utilize the rice straw to produce alpha cellulose and to evaluate the alpha cellulose obtained.

MATERIALS AND METHODS

Plant Material

Rice straw was collected from a rice field in Padang, West Sumatera, Indonesia on March 2017. It was cut into chips and dried.

Chemicals

Ethanol, sodium hydroxide, hydrogen peroxide, and distilled water were purchased from PT Bratachem, Indonesia.

Methods

The method of this study was adopted from research done by Ngozi *et al.*,[8] combined with research by Oyeniyi and Itiola.[9] Briefly, about 100 grams of rice straw powder was macerated with ethanol for 24 hours. The biomass was then soaked in hot water and dried.

Delignification and extraction of alpha cellulose

Dry rice straw was heated in 3.5 % sodium hydroxide for two hours at a temperature of 100 °C, then it washed with distilled water until pH 7 and dried in the oven. The material was further treated with 17.5 % sodium hydroxide for one hour with a temperature of 100 °C and washed with distilled water until pH 7 and dried in the oven so that alpha cellulose was obtained.

Bleaching

Bleaching was done by using 20 % Hydrogen peroxide (H_2O_2) for two hours, then washed with distilled water and dried at 60°C for one hour and powdered again, then stored.

Evaluation procedures

For morphological properties, samples were bonded with stable metal palladium, cleaned with a blower, and coated with gold and palladium in a pressurized machine 1.492 x 10^{-2} atm. Samples were subsequently put into a particular room and then irradiated with electron beam powered 10 kV. The sample emits secondary electrons and electrons that bounce can be detected by a detector which is then amplified by an electrical circuit that produces an image on a CRT (Cathode Ray Tube). Shooting is done after selecting a specific part of the object (sample) and the desired magnification to obtain a good and clear photo. Crystal analysis was performed using XRD (Rigaku Miniflex 600) with a voltage of 40 kV, the power of 600 W, the electric current 15 mA and Cu radiation source at an angle 2ɵ = 0 – 70^{o}.[10]

Other characterization of alpha cellulose include organoleptic examination, identification, measurement of pH, determination of substances soluble in water, measuring the solubility in a mixture of ammonia-copper (II) sulphate, determination of drying shrinkage, determination of real density and compressed density, determination of Carr's index and Hausner ratios, starch test, and determination of FT IR spectra.[11, 12, 13]

RESULTS AND DISCUSSION

Rice straw was collected from rice field, dried and ground by the grinder. The result of rice staw powder was shown in Figure 1. Alpha cellulose obtained from 100 grams of rice straw powder was 30.11 grams (30.11%), and then it was bleached by hydrogen peroxide (see Figure 2).

Figure 1: Rice straw powder

Figure 2: Alpha cellulose before (left) and after (right) bleaching process

The production of alpha cellulose begins with the pre-treatment by maceration in ethanol, which is useful for dissolving polar and non-polar compounds of rice straw, such as fat, wax, carbohydrates (monosaccharides), amino acids and other compounds. Then it is washed with hot water which aims to dissolve insoluble compounds in ethanol such as pectin and so on. In this case, it may also develop of cellulose cells or to remove the rest of methanol so it is expected that the impurity compounds can be removed.

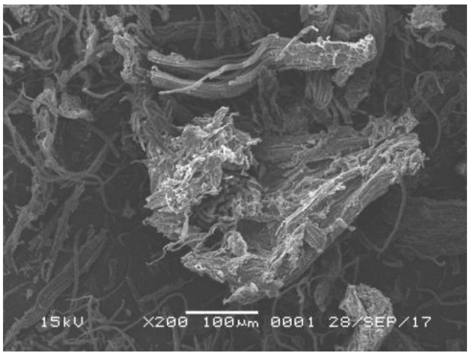
The delignification process using 3.5 % sodium hydroxide aims to break the lignin bond. Removal of lignin residue is enhanced by the bleaching process using 20 % hydrogen peroxide. Biomass was cooked in 17.5 % sodium hydroxide, to obtain alpha cellulose, since the cellulose components, beta and gamma cellulose is dissolved in this solution, while the alpha cellulose is not dissolved in 17.5 % sodium hydroxide.

Alpha cellulose in its application is often used as a marker of the purity of a cellulose material. Alpha cellulose has the highest purity level. Alpha cellulose > 92% are eligible for use as the primary raw material for propellant manufacture, while the lower cellulose is used as raw material in paper and cloth industry.

The organoleptic characteristics of alpha cellulose were eligible according to British Pharmacopeia, except for loss on drying which was more than 7% of its weight.[13] The organoleptic test conducted on alpha cellulose showed that the sample was a white powder, odorless, and tasteless. Determination of pH was done by shaking 5 g alpha cellulose with 40 mL of water for 20 minutes and centrifuged. The supernatant was measured by its pH, and the resulting pH was 7. The sample was dried at 105 ^{o}C for 3 hours to determine water content in the sample, resulting 8 % loss of its weight.

SEM analysis

The morphological property was investigated by using Scanning Electron Microscopy (SEM) with a voltage of 15 kV. Figure 3 shows the morphology of cellulose fibers with a magnification of 200X and 750X. At specific points, cellulose fibers were 5-10 µm in diameter.



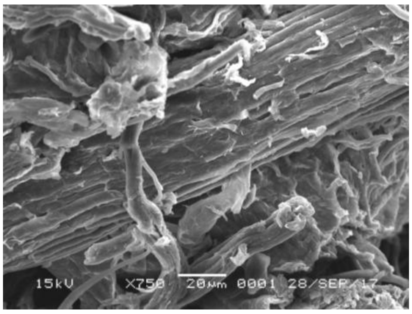


Figure 3: SEM images of Alpha cellulose produced with a magnification of 200X and 750X

XRD Analysis

To analyze the crystallinity of the alpha cellulose obtained in this work, diffractometric X-ray measurement was carried out. The crystallinity index (CrI) of cellulose was calculated using the equation:

$$\%ICr = \left(1 - \left(\frac{I_{am}}{I_{002}}\right)\right) \times 100$$

Here, I_{am} was the minimum intensity of diffraction of amorphous material, I_{002} was the maximum intensity of diffraction of the lattice peak.[14] The crystallinity index of the alpha cellulose obtained was 78 %.

In general, the polymer material of alpha cellulose is semi-crystalline. It means that alpha cellulose contains the amorphous part beside the dominant crystalline parts. The X-ray diffractogram of crystalline polymers produces sharp peaks; while the amorphous polymers tend to produce a widened or blunt peak (see Figure 4). There is a typical pattern of a diffractogram, on the 2ɵ value of 22.5 contained a sharp peak that shows the nature of the crystalline, and on the 2ɵ value of about 18 the blunt and widened peak or valley show amorphous nature.

The sharp peak of XRD spectrum can be attributed to the scattering of crystallinity while their diffuse background is attributed to this order. Therefore, the alpha cellulose should be considered to be semi-crystalline. The x-ray diffraction spectrum (Figure 4) is blurring, perhaps due to the presence of non-cellulosic amorphous material such as fiber extractives. Crystalline index of obtained alpha cellulose was 78%. After pre-treatment of cellulose pulping in the cellulose source, the crystallinity may vary from 74 to 91 %.[15, 16]

FTIR analysis

FTIR is a method of infrared spectrophotometry that can be used to identify unknown samples, determine sample quality, and determine the number of components in a mixture.[17, 18] Figure 5 shows the infrared spectrum of alpha cellulose derived from rice straw, and the functional groups present in the sample were listed in Table 1.

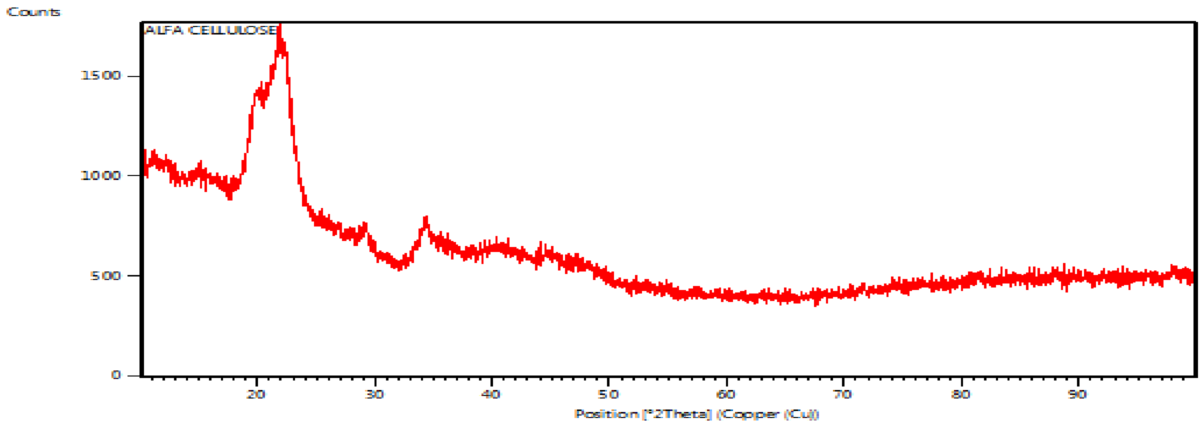


Figure 4: X-Ray Diffraction spectrum of Alpha cellulose produced

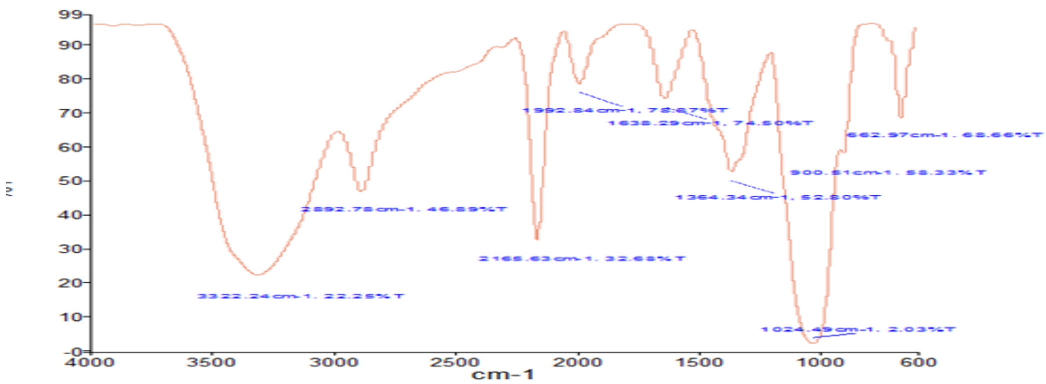


Figure 5: FTIR spectrum of alpha cellulose produced from rice straw

Table 1: Functional groups of alpha cellulose produced from rice straw base on FTIR spectra

Wave number (cm^{-1})	Transmittance	Functional groups
3322,24 cm^{-1}	22,25 %	O-H stretching vibration
2898,78 cm^{-1}	46,89 %	C-H stretching vibration
2165,63 cm^{-1}	32,68 %	Alkyne (C≡C)
1992,84 cm^{-1}	78,67 %	C-H bending
1638,34 cm^{-1}	74,5 %	H_2O absorption
1364,34 cm^{-1}	52,8%	C-H bending
900,51 cm^{-1}	58,33 %	C-O stretch and deformation

FTIR may be used to determine whether resins, lignin, and hemicelluloses have been removed, leaving purified alpha cellulose. A peak near 1725 cm^{-1} and 1735-1731 cm^{-1} are not present in alpha cellulose. In this FTIR spectrum, peaks near those wavelengths did not exist. Therefore, the sample can be considered as free from resins, lignin, and hemicelluloses.

CONCLUSION

From this research can be known that rice straw is a potential source of alpha cellulose. In addition to its high cellulose content, it can also add value to the use of rice straw waste that is not widely utilized. The results of alpha cellulose characterization show that alpha cellulose has good physicochemical properties, except for loss on drying that does not meet the requirements.

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