

EXTRACTION CELLULOSE FROM RICE HUSK

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Abstrak. Selulosa dari sekam padi telah berhasil disintesis. Sekam padi merupakan limbah pertanian yang melimpah dan mempunyai kandungan selulosa yang cukup tinggi. Biokonversi sekam padi menjadi selulosa diawali dengan tahapan proses delignifikasi dan bleaching. Selulosa yang dihasilkan dikarakterisasi menggunakan FTIR dan XRD. Spektra IR menunjukkan puncak serapan yang kuat pada $3444,87\text{ cm}^{-1}$ yang dikaitkan dengan getaran peregangan O–H (ikatan hidrogen antar molekul), puncak serapan pada $1033,85\text{ cm}^{-1}$ diidentifikasi sebagai peregangan C–O–C yang merupakan ikatan β -1,4-glikosidik dari selulosa. XRD memperlihatkan ukuran partikel selulosa yaitu 3,44 nm dan memiliki kristalinitas 58,73%. Hasil ini menunjukkan bahwa selulosa dari sekam padi memiliki karakteristik yang baik.

Kata Kunci: Sekam padi, Selulosa, FTIR, XRD

Abstract. Cellulose from rice husk has been successfully synthesized. Rice husk is an abundant agricultural waste and has a high cellulose content. Bioconversion of rice husk into cellulose begins with the stages of delignification and bleaching. The resulting cellulose was characterized using FTIR and XRD. The IR spectra show a strong absorption peak at 3444.87 cm^{-1} which is associated with stretching vibrations O – H (hydrogen bonds between molecules), an absorption peak at 1033.85 cm^{-1} is identified as stretching C – O – C which is β bond -1,4 glycosidic from cellulose. X-ray diffractogram showed that the cellulose obtained had a particle size of 3.44 nm and crystallinity of 58.73%. These results indicated that cellulose from rice husk had good characteristics.

Keywords: Rice Husk, Cellulose, FTIR, XRD

INTRODUCTION

Indonesia is an agricultural country that is known to have abundant biological natural resources, mainly in agriculture. With this abundance of living natural resources, it is not surprising that there are many kinds of agricultural products produced. One of the abundant agricultural waste in Indonesia is rice husk. Rice husk is a by-product produced from rice mills. Based on data from the Central Bureau of Statistics, rice production in Indonesia in the form of milled dry grain was 75.36 million tons. The production continues to increase each year by 4.51 million tons (6.37%). Each kilogram of rice produces an average of 280 grams of the husk or as

much as 20% to 30% of the weight of dry rice milled, so that the accumulation of rice husk waste on average each year is more than 10 million tons (Danarto *et al.*, 2010).

Rice husk is a hard layer which includes kariopsis which consists of two leaf shapes, namely petal husks and crown husks, where in the rice milling process, the husks will be separated from rice grains and become waste material or grinding waste. From rice mills it will produce about 25% of husk, 10% of bran, and 65% of rice (Haryadi, 2006).

Rice husk is a lignocellulosic material derived from agricultural waste, which has an abundant presence. Rice husk is one of the biomass that has the greatest chemical

composition of organic carbon, which is 45% - 50%. The high composition of organic carbon indicates that there is a lot of cellulose in the rice husk (Prabawati and Wijaya, 2008). The main component of rice husk is silica (15-17%) (Leiva *et al.*, 2007; Stefani *et al.*, 2005), other components are cellulose (35%), hemicellulose (25%), and lignin (20%) (Shukla *et al.*, 2013). Due to its high cellulose content, rice husks can be used as a source of cellulose and can be used as a material that has benefits.

Cellulose is a material commonly used in several biological-based applications such as cosmetics and medicines, because it is environmentally friendly, easily recycled, and is one of the renewable materials. Cellulose has a chemical structure consisting of β -1,4 glycosidic. Extracted cellulose is stable, biodegradable, thermal stability (Long *et al.*, 2018), and good crystallinity (Yang *et al.*, 2018). Cellulose also has complex crystalline and amorphous morphological forms. In addition cellulose has interesting properties, such as biocompatible, renewable and can be degraded.

Cellulose from rice husk have been used in many applications including to make cellulose nanoparticles with high fluorescence (Kalitaa *et al.*, 2015), cellulose nanocrystals as reinforce in gelatin hydrogels for drug delivery (Ooia, *et al.*, 2016), cellulose acetate (Das *et al.*, 2014) and using of rice husk cellulose for biomedicine (Shukla *et al.*, 2013). Based on the cellulose content of rice husks, this study was carried out to synthesize and determine the cellulose content of rice husks taken from a rice mill in Makassar, Indonesia.

MATERIALS AND METHODS

Materials

The materials used were rice husk taken from the Maccopa rice mill, Sulawesi Selatan, Indonesia, NaOH, NaOCl, distilled water, and whatmann paper No. 42.

Methods

1. Sample preparation

800 g of rice husk was washed and then dried under the sun. Furthermore, rice husks were ground using a hammer mill to obtain smaller sizes of rice husk. After that, rice husk was sifted with a sieve with a size of 80 mesh to obtain finer rice husk powder.

2. Delignification

A total of 10 grams of rice husk powder was put into a 250 mL beaker and then 12% NaOH was added as much as 100 mL. The mixture was then heated at 80 °C for 3 hours, and stirred with a magnetic stirrer. Next, The mixture was filtered, and the precipitate was washed with distilled water until a neutral pH.

3. Bleaching

The delignification residue was put into a 250 mL beaker, added with 100 mL of 2.5% NaOCl solution, heated at 80 °C, and stirred for 1 hour. The precipitate was filtered and washed with distilled water until a neutral pH value was obtained. The sediment was left for 1 hour and cellulose extract from rice husk was obtained.

4. Characterization

Cellulose was characterized using a FTIR SHIMADZU spectrophotometer, to determine the typical functional groups of cellulose measured at wavelengths between 4000-500 cm^{-1} . The diffractogram was taken from a XRD-7000-Shimadzu with Cu K α radiation filtered high-intensity nickel-monochromatic ($\lambda = 0.15418$) produced at 40 kV and 30 mA. The sample was scanned at a speed of 2°/minute at an angle of 2θ between 10 ° - 50 °.

RESULTS AND DISCUSSION

1. Cellulose Extract

Cellulose extract from rice husk has been obtained by delignification using NaOH and bleaching using NaOCl. The percentage of cellulose obtained from the results of delignification and bleaching was

52.3%. The percentage results obtained were quite large and this showed that lignin and hemicellulose can be degraded quite well, where NaOH in the delignification process can attack and damage the structure of lignin in the crystalline and amorphous parts and separate hemicellulose. Dissolved lignin was marked black in a solution called black liquor (Safaria, 2013). The delignification products were generally white, but the results obtained are still brown. This finding possibly caused by the residual lignin obtained from depolymerization. The remaining chromophore can be removed by bleaching using NaOCl. The bleaching process aims to purify cellulose without a lot of termination of the cellulose chain. The bleaching process will make the color of the pulp brighter or white (Othmer, 1982) and the bleaching results carried out in the study have made the cellulose color of the rice husk obtained brighter and somewhat white.

2. FTIR Characterization

The distinctive feature of the FT-IR spectrum of cellulose is shown in Figure 1. Cellulose rice husk displays two main absorption areas, in the area of high wave numbers ($2800\text{-}3500\text{ cm}^{-1}$) and low wave numbers ($700\text{-}1700\text{ cm}^{-1}$) as has been reported by Haafiz *et al.*, (2013). The FTIR spectrum of rice husk cellulose shows a wide absorption peak located at $3330\text{-}3500\text{ cm}^{-1}$ which is a stretch of the O-H group. The absorption peak in the area of $2800\text{-}2900\text{ cm}^{-1}$ was related to the CH_2 group, with an absorption peak of 2858 cm^{-1} representing overlapping of the CH_2 band (Jahan *et al.*, 2011). Absorption in the area of 1643 cm^{-1} is the vibration of the $\text{C}=\text{C}$ bond of cellulose rice husk. In addition, the absorption peaks in the area of $1300\text{-}1398\text{ cm}^{-1}$ at cellulose aerogels are vibrational bands of C-H and C-O which are related to the aromatic ring of polysaccharides (Yu, *et al.*, 2013). At 1033 cm^{-1} the peak shows a stretching of the C-O-C pyranose ring identified as a $1,4\text{-}\beta$ glycoside bond to cellulose.

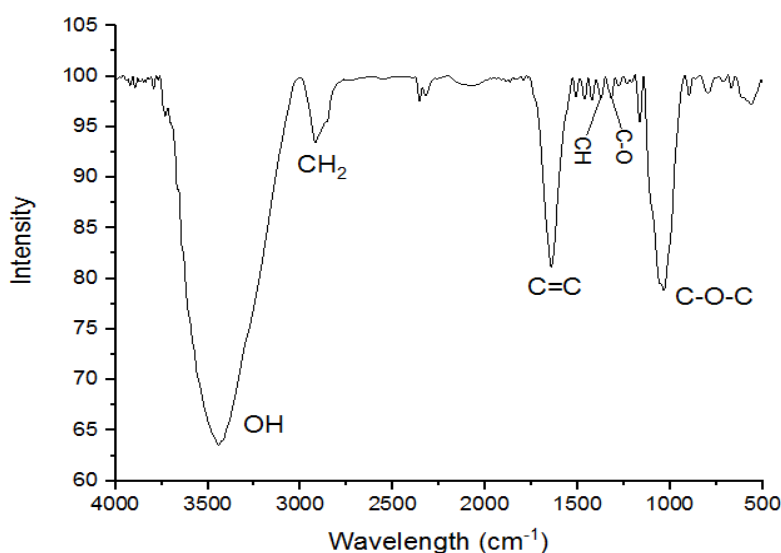


Figure 1. FTIR spectrum of cellulose rice husk

3. XRD Characterization

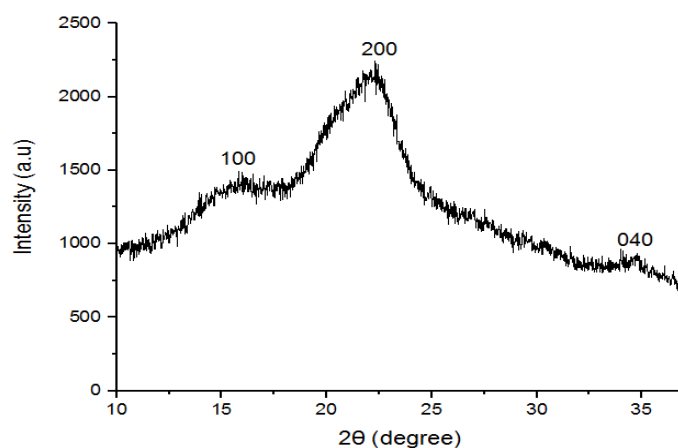


Figure 2. The X-ray diffractogram of cellulose rice husk

The XRD pattern of cellulose is shown in Figure 2. The diffractogram of cellulose from rice husk produced 3 diffraction peaks at angles 2θ of 17.12° , 22.26° , and 34.48° . The three peaks are related to the crystal fields 110, 200 (Huangzeng, *et al.*, 2015 and Chenling *et al.*, 2017) and 040 (Suryanto, *et al.*, 2017). The XRD pattern shows that the highest peak appears at an angle of 2θ around 22.26° , this area is a typical peak of the cellulose structure. The structure of cellulose was shown by diffraction peaks in the range between 22° and 23° and was characteristic of original cellulose (cellulose I) (Le Troedec, *et al.*, 2008).

The crystallinity of cellulose obtained through delignification using NaOH and bleaching using NaOCl reaches 58.73% and amorphous 41,27%. The crystallinity achieved in this study is quite high. The high crystallinity obtained is due to the elimination of hemicellulose and lignin in the amorphous region which leads to fatigue in cellulose molecules (Li *et al.*, 2009). In addition, the crystal size of rice husk cellulose was 3.44 nm.

CONCLUSION

Cellulose synthesized from rice husk gives good results in terms of its characterization using FTIR and XRD. The cellulose produced has a high crystallinity

of 58.73% and a very small crystal size of 3.44 nm.

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