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Cellulose Extraction from Castor Shell

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Abstract

Cellulose extracted from biomass sources is considered the future feedstock for many products due to the availability and eco-friendly nature of cellulose. In this study, castor shell (CS) was evaluated as a potential source of cellulose. The cellulose was extracted after pretreatment process was done on the CS. The pretreatment process began with the removal of other extractives from CS, then an alkaline treatment, bleaching process with hydrogen peroxide, and followed by a mixture of acetic and nitric acids. CS cellulose was analysed by infrared absorption spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray diffraction (XRD), and thermogravimetric analysis (TGA). The result showed that the overall process was adequate to produce cellulose with high purity and crystallinity from CS waste.

Keywords: Castor shell, cellulose, hemicelluloses

Introduction

The use of cellulose-based waste materials such as rice straw [1], sugarcane bagasse [2], orange peels [3], and apple pomace [4] as a source of energy is increasing nowadays. Cellulose-based waste materials are abundant, renewable, and eco-friendly in characteristics.

One of the agriculture wastes that can be used as a source of cellulose is castor shell, which is part of the castor plant known as *Ricinus communis*. The castor seed is the source of castor oil and castor shell is a residue from the castor oil extraction process. Usually after the seed removal from the capsule, the shell will become accumulated waste. The shell constitutes of around 30% of the weight of a castor seed. Most castor shells are either discarded to the land thus causing soil pollution, or simply burned in the fields thus leading to air pollution.

Castor shell is plant fibre with the main components including cellulose, hemicelluloses, and lignin. Cellulose is the most abundant renewable polymer and usually used as raw material for the production of, among others, pharmaceuticals, paper, fibreboard, ropes, sails, and timber for housing [5] [6]. It is the structural component of the primary cell wall of green plants. Normally, 33% of all plant matter is made of cellulose. The cellulose content of cotton is 90% and of wood is 40-50%.

India, which is the largest exporter of castor oil with over 88% share in the global exports, produces the largest share of castor seed worldwide, accounting of more than 75% of the production, followed by China

(10-12%) and Brazil (6-7%). The global castor seed production, which was 1.59 million tons in 2010-2011, increased dramatically in 2011-2012 due to the record production in India. The mass production of castor seed will eventually discard enormous castor shells. This is an opportunity for researchers to develop some end products from the waste.

The castor shell is one of the plant fibres with the main components including cellulose, hemicelluloses, and lignin. Cellulose is the most abundant renewable polymer that is usually used as a raw material for the production of, among others, pharmaceuticals, paper, fibreboard, ropes, sails, and timber for housing [5] [6]. Cellulose is the structural component of the primary cell wall of green plants. Normally, 33% of all plant matters are cellulose. The cellulose content of cotton is 90% and for wood is 40-50%.

In the present work, the use of castor shell as a new source to obtain cellulose through extraction using the delignification and bleaching was proposed.

Experiment

Materials

Castor shell was obtained from Casa Kinabalu, Sarawak. Hexane (296090-100ML Sigma) obtained from FKKSA lab was used for dewaxing process. Sodium hydroxide (R&M Chemicals) and hydrogen peroxide (Merck) were used to bleach the castor shell. Nitric acid (0587-100ML Amresco) and acetic acid (Amresco,

0714-500ML) were used for purification purpose. Commercial cellulose (Sigma, S5504-500G) was used to compare it with the product. All solvents and reagents were of analytical grade.

Procedure

The raw material (CS) was cut into small pieces, then dried in oven and ground to 0.4 mm (40-60 mesh sieves). Ground castor shell was submitted to a dewaxed process with hexane in a Soxhlet apparatus until the solvent was clear. The process took about 6 h. The dewaxed process was based on a procedure adapted from the dewaxing of rice husk as described by [7]. The castor shell was then put into autoclave with 5% aqueous NaOH at 121 °C for 30 min. The dewaxed castor shell was bleached with 2% H₂O₂ at pH 11.8 for 12 h at 48 °C using water bath. To purify the cellulose, 66 mL of 80% acetic acid and 6.6 mL of 70% nitric acid were added successively to 2 g of castor shell, then heated over the oil bath at 120 °C for 30 min. The treated mixture was cooled and washed with 20 mL of 95% ethanol, 20 mL distilled water, and again 20 mL of 95% ethanol to remove extraction breakdown products and traces of nitric acid. Finally the cellulose was dried in an oven at 60 °C until constant weight.

Results and Discussion

Characterisation of the cellulose
Spectroscopic characterisation



Fig. 1. Photos of CS (a) before treatment and (b) after the treatment (cellulose)

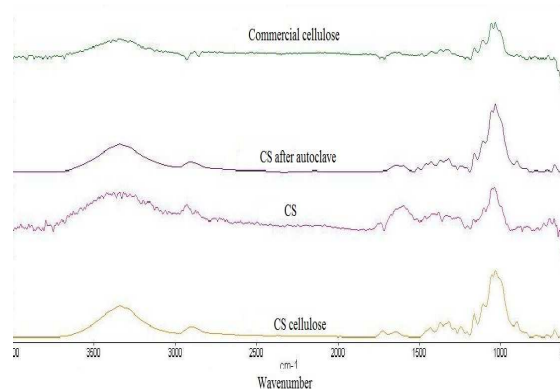


Fig. 2. FTIR spectra for castor shell, castor shell after autoclave, castor shell cellulose and commercial cellulose within the range from 4000 to 800 cm⁻¹.

Figure 1 shows the physical aspect of the original castor shell and the cellulose at the end of experiment. Great effectiveness of the process was observed because the colour of cellulose obtained was white. The bleaching process was expected in this state. The sample was analysed using a Fourier Transform IR spectrometer. The FTIR was used since it was an easy method to obtain direct information on chemical changes occurred. The spectrometer also showed the chemical structure of each compound. Fig. 2 shows the FTIR spectra of the castor shell, castor shell after autoclave, castor shell cellulose, and commercial cellulose. The castor shell cellulose was comparable to commercial cellulose [8]. All samples showed two absorbance regions. The first one was at high wavenumbers, corresponding to the range of 1800–600; and the second one was at lower wavenumbers, corresponding to the range of 4000–2800.

Thermogravimetric analysis (TGA)

Figure 3 shows the thermal degradation pattern of the commercial cellulose, castor shell, and castor shell cellulose. All samples showed dehydration occurring at 150 °C according to the figure showed. The mass loss of water was determined from 35 °C to 150 °C. The percent of mass loss was 2.88 wt% for commercial cellulose, 5.96 wt% for castor shell, and 6.55 wt% for castor shell cellulose. The effective temperature of the castor shell started from 185 °C. The figure also shows that castor shell cellulose showed higher thermal stability than the castor shell itself because some components had been removed at lower temperature. The peak of crude castor shell was the widest among the samples due to the decomposition of hemicelluloses and lignin. The commercial cellulose and castor shell cellulose decomposed in single step, thus proving the absence of hemicelluloses and lignin. This finding was in

accordance with the result by Simone et al. (2011). The maximum rate of decomposition of castor shell cellulose occurred at 329 °C while the commercial cellulose showed decomposition at 332 °C. The commercial cellulose showed higher temperature than the castor shell due to greater crystallinity of cellulose in commercial cellulose according to the research before. However, the size of crystal and the atmosphere in which the experiment was conducted (in nitrogen or air) also affected the peak of temperature, according to [9].

At the end of the experiment, at the temperature of 700 °C, castor shell showed the highest residual mass among the three samples. Determined from the TGA curves, the residual mass of castor shell was 36.3%, which was quite considering the high silica content of castor shell. The residual mass for castor shell cellulose was 21% and the least residual mass was the commercial cellulose (7.5%).

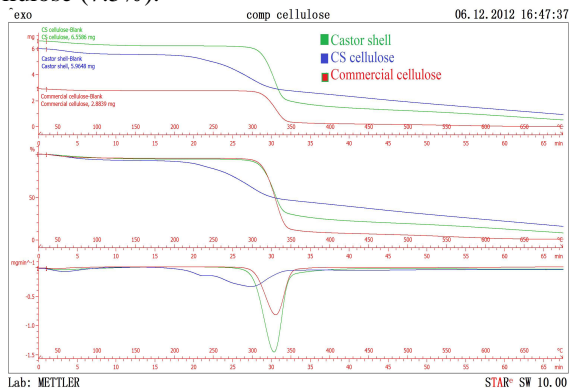


Fig. 3. TGA curves for commercial cellulose, castor shell, and castor shell cellulose.

X-ray Diffraction

Figure 4 shows that the major crystalline peak occurred at 22 °C for each sample. For commercial cellulose, the crystalline peak was at 22.6 and for castor shell cellulose was at 22.7. Based on Segal method, the peak height method was used to calculate the crystallinity of the samples as shown below:

$$X_{CR} = \frac{I_{200} - I_{AM}}{I_{200}} \times 100$$

where I_{200} is the height intensity of the 200 peak at 22.6, which represents both crystalline and amorphous material and I_{AM} , which represents amorphous material only, is the lowest intensity between the 200 and 110 peaks [7]. Crystalline fraction of wood is given only by cellulose because the other two main wood components namely hemicellulose and lignin are amorphous [10].

The crystallinity index of castor waste cellulose (calculated by Segal formula) was approximately 70% while that of commercial cellulose was estimated as

91%. For comparison, the crystallinity index of other samples, as reported in the literature, was found to be around 66% for potato tuber cellulose, 68% for rice straw cellulose, and 71% for wood cellulose (Abe & Yano, 2009). It can be concluded that the procedure employed in this study for cellulose extraction from castor shell was adequate for obtaining samples with high crystallinity. Highly crystalline fibres and fibril aggregates could be more effective in achieving higher reinforcement for composite materials (Cheng, Wang, Rials, & Lee, 2007). In addition, it can be seen in Fig. 4b that CS cellulose can be classified as cellulose I, since there is no doublet in the intensity of the peak at $2\alpha = 22^\circ$. A similar finding was reported by Morán et al. (2008) for sisal cellulose extracted by other procedures.

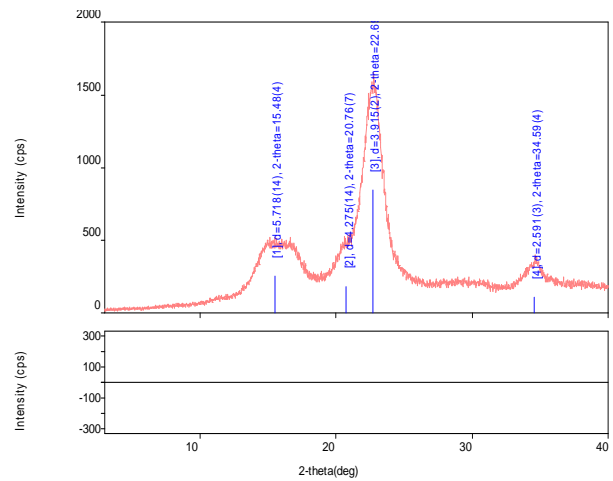


Fig. 4. a) Commercial cellulose

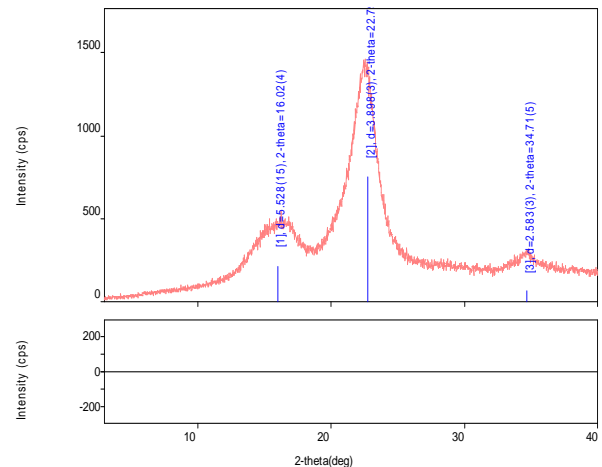


Fig. 4. b) Castor shell cellulose.

Conclusion

Castor shell is an abundant and largely unused agricultural waste. The present study reveals the potential of castor shell as a source of cellulose, using an environmentally friendly extraction method to avoid pollution due to the huge amount of residues from castor oil industry. The utilisation of castor shell for cellulose production is undoubtedly a sustainable. TGA analysis was done under nitrogen and showed high-mass residence for castor shell at 700°C, which can be said, was due to the high silica content of the material. The analysis result supported that the product was cellulose, i.e., the lignin and hemicelluloses had been removed from the castor shell

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