Extraction and Characterization of Cellulose from Jackfruit Rind *(Artocarpus Heterophyllus)*

Wahida Abdul Rahman[1*], Noor Aishatun Majid[2], Siti Fatimah Abd Razak[1]

[1,2]Faculty of Applied Sciences, Universiti Teknologi MARA Cawangan Perlis, Kampus Arau, 02600 Arau, Perlis, Malaysia
Corresponding author: *wahida811@uitm.edu.my

Received: 21 November 2018
Accepted: 11 April 2019
Published: 30 June 2019

ABSTRACT

In this study, jackfruit cellulose was extracted by undergoing two steps; bleaching and alkaline treatment. The first step was the production of holocellulose by using bleaching method that removed the lignin from the jackfruit rind (JR). Next step was converting the holocellulose into cellulose by using alkaline treatment at room temperature. The percentage of cellulose extracted from JR in this study was 40% which was higher than the previous research. The characterizations of extracted JR cellulose were investigated by using Fourier Transform Infrared Spectrometer (FTIR) and Scanning Electron Microscopy (SEM). FTIR confirmed that the removal of non-cellulosic components in the jackfruit fibre and the micrograph by SEM displayed a rough surface indicated the presence of the cellulose fibre. Based on this study, JR has the potential to be used as a reinforcement element in composite materials.

Keywords: *alkaline treatment, bleaching process, cellulose, holocellulose, jackfruit rind*

INTRODUCTION

Cellulose can be found in plant cell wall mostly that forming the structural fibre of plants and it gives strength because it is the most abundant organic compound, for example, stem, leaves, and branches. It is organized into microfibril in the cell wall, interrupted by hemicellulose and surrounded by a lignin matrix [1]. For example, cellulose can be extracted from potatoes, rice husk, wood pulps, durian rinds, and jackfruit rinds. Jackfruit rinds (JR) are always discarded since they are not edible but the rinds are a good source of cellulose and it can be extracted by using suitable extraction method.

Jackfruit or *Artocarpus heterophyllus* is occasionally found in Pacific island home gardens and commonly found in Southeast Asia. It is well known as an aromatic, juicy and flavourful fruit that can be eaten fresh or preserved. The fruit has many benefits from the seeds until the wood chips yield. For example, the seeds can be boiled, roasted, eaten like chestnut or cooked in other dishes, the wood chips can be used as a dye to give colour to the robes of Buddhist priest but the rind is always discarded [2]. The rind contained cellulose up until 27.75% depending on the type of extraction [3]. The aim of this study is to extract cellulose from JR by using bleaching treatment and alkaline treatment and to characterize the cellulose contents.

EXPERIMENTAL

Materials

The raw material used for this study is jackfruit rind (JR) waste collected from the jackfruit stalls. The chemicals and reagents used in this study were; ethanol (C_2H_5OH, 95%, ACS reagent, Sigma Aldrich), acetic acid (CH_3COOH, 99%, Reagent Plus, Sigma Aldrich), sodium hypochlorite (NaClO, 10% available chlorine), Technical Grade, Sigma Aldrich), and sodium hydroxide (NaOH, Sigma Aldrich).

Sample Preparation

In this study, the raw materials (JR) was collected from jackfruit stalls in the night market around the area of Arau, Perlis. The JR was then washed with tap water, cut into small size and air dried. After completely dried, JR was treated by blanched with an equal amount of 95% ethanol at 80^0 C and dried at 55^0 C until the weight of the JR was constant according to Koh *et al.* (2014) [4] with a slight modification. Next, JR was grounded into powder-like and stored in an airtight container for further analysis.

Extraction of Cellulose from Jackfruit Rind (JR)

According to Penjumras *et al.* (2014) [1] with a slight modification, JR's first step of extraction of cellulose was chlorination or bleaching treatment. 20 g of the pre-treated JR was rinsed with tap water to remove any particulate matters. The sample was soaked into 640 mL of distilled water. Then, it was transferred into the water bath at 70^0 C. 4 mL of CH_3COOH and 7.2 mL of NaClO were added in the beaker every hour for the duration of 5 hours of bleaching where the

lignin was separated completely from the fibre. Samples are then washed and rinsed with tap water until the yellow colour of the sample turned clear which indicated the presence of holocellulose and the odour of chlorine was eliminated. Next step was to convert the holocellulose into cellulose. 80 mL of 17.5% NaOH solution was added into the holocellulose.

Next, for every 5 minutes intervals, another 40 mL of 17.5% NaOH solution was added into the mixture up to 3 times. The solution was left to stand for 30 minutes and 240 mL of distilled water was added into the solution. The solution was to let stand for another 1 hour before filtering. An 800 mL of 8.3% NaOH solution was added into the cellulose for 5 minutes and then filtered and rinsed with distilled water. The alkaline water was neutralized by adding 120 mL of 10% CH_3COOH for 5 minutes. Then, cellulose was filtered, washed, rinsed with distilled water until residue free from acid and the JR cellulose was dried in a vacuum oven at 100^0 C. The weight of dried cellulose was taken, stored in an airtight container, and the percentage of cellulose was calculated as following [5]:

$$\text{Cellulose } (\%) = \frac{W_2 \text{ (Weight of the cellulose), g}}{W_1 \text{ (Weight of holocellulose), g}} \times 100 \qquad (1)$$

Characterization of Jackfruit Rind (JR) Cellulose

Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) Spectroscopy

The raw JR and extracted JR cellulose was analyzed by using Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) Spectroscopy. The FTIR spectrum was performing within wavenumber from 400 cm^{-1} to 4000 cm^{-1}.

Scanning Electron Microscopy (SEM)

The morphology study of the JR cellulose was analyzed by using Scanning Electron Microscopy (SEM). The sample was coated with gold before being analyzed.

RESULTS AND DISCUSSION

The cellulose composition in JR was 40% as shown in Table 1 after calculated by using Eq. (1). The percentage of extracted JR-cellulose was higher than the research from Antony *et al.* (2017) [3] that used a gravimetric methodology to determine the composition of cellulose, starch, and pectin in their samples. Figure 1 shows the image of extraction stages from raw JR until cellulose was obtained.

Table 1: Percentage of Extracted JR-cellulose (%)

Weight of holocellulose (g)	Weight of cellulose (g)	Cellulose (%)
7	2.8	40

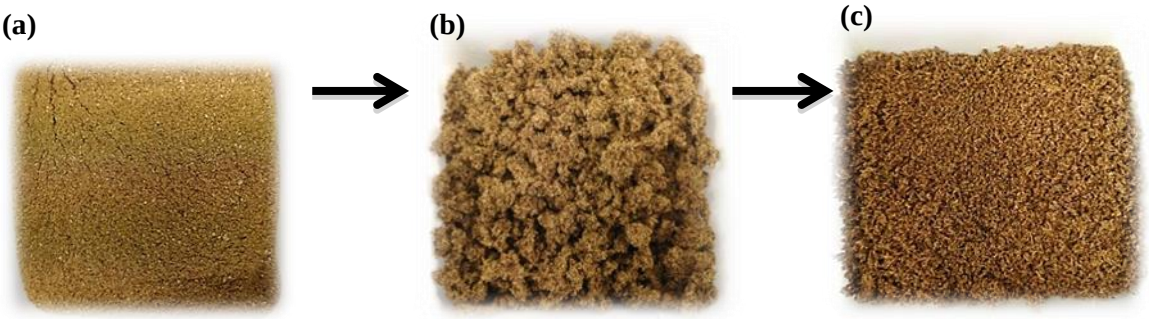

Figure 1: Physical appearance of a) JR Raw; b) JR Holocellulose; c) JR cellulose

ATR-FTIR Spectroscopy Analysis

ATR-FTIR is necessary because it can provide the chemical composition of the material. Peaks exhibited by the spectrum represent the functional groups exist in the material that has been analyzed. These functional groups depicted chemical bonding in the material that can be confirmed based on the previous study of the related samples. Figure 2 shows the ATR-FTIR spectra for a) JR cellulose and b) JR raw. The strong and broad bands near 3500-3000 cm^{-1} were observed in both spectra, which corresponded to O-H stretching vibrations of a hydroxyl group in cellulose molecules as well as intramolecular and intermolecular hydrogen bonds [1, 6-9]. The peak was narrower for extracted cellulose, which indicated that extracted cellulose contained more -OH group than the raw jackfruit rind [1].

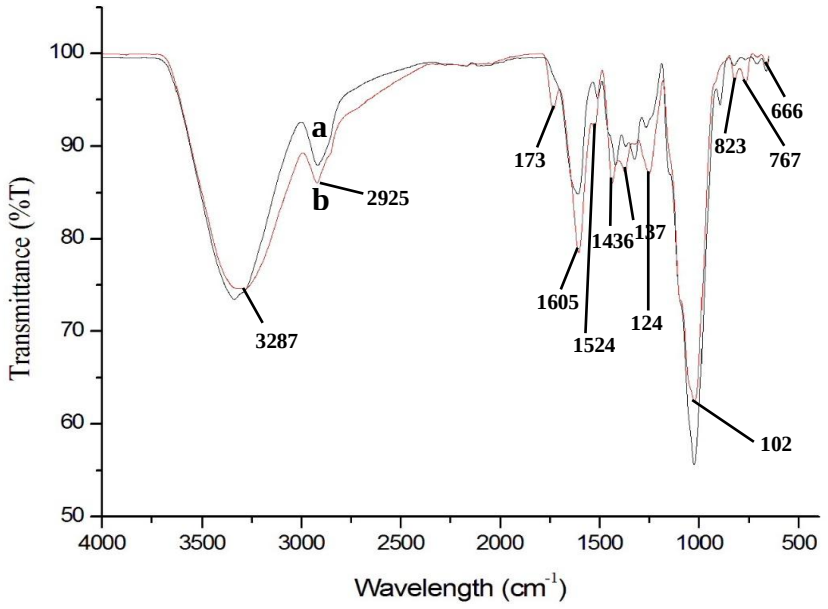

Figure 2: ATR-FTIR spectra for a) JR cellulose and b) JR raw

Peaks presented around 3000-2800 cm^{-1} was attributed from C-H stretching vibrations from some methylene groups of polysaccharides [3, 10]. The peak presented at 1730 cm^{-1} corresponded to the carbonyl groups (C=O) due to the presence of acetyl ester and carbonyl aldehyde groups of hemicellulose and lignin. This peak disappeared completely in the JR cellulose because of the removal most of the hemicellulose and lignin during extraction process [11]. However, there was still a trace of lignin due to the existence of a peak at 1605 cm^{-1} [1]. The peaks at 1524 and 1436 cm^{-1} represented the aromatic C=C ring stretching and C-H deformation in methyl, methylene and methoxyl groups of lignin. The intensity of these peaks in JR cellulose decreased because of removal of the lignin during the extraction process.

The peak around 1350-1300 cm^{-1} in spectra gave a characteristic of CH_2 wagging vibration in cellulose [1, 11]. A peak at 1248 cm^{-1} represented –COO vibration of acetyl groups in hemicellulose. The intensity of this peak decreased in JR cellulose [11]. A sharp peak at 1023 cm^{-1} represented C-O-C pyranose ring skeletal vibration in cellulose and its intensity increased in JR cellulose [1, 3, 11]. The peak at 823 cm^{-1} is referred to glycosidic C-H rocking vibration which was reported to be the characteristic of cellulose structure [1, 3, 11].

Table 2: Comparison of peak absorption in samples at different stages of treatment

Wavenumber (cm^{-1})			
JR Raw	**JR Cellulose**	**Functional Groups**	**References**
3287	3336	-OH stretching cellulose	Sun *et al.* (2004), Yan *et al.* (2009); Tawakkal *et al.* (2010); Mandal *et al.* (2011); Pejumras *et al.* (2014)
2925	2918	-CH stretching methylene groups	Antony *et al.* (2017); Ma *et al.* (2016)
1605	1607	Stretching of aromatic hydrocarbons of lignin	Pejumras *et al.* (2014)
1373	1323	-CH_2 wagging vibration of cellulose	Pejumras *et al.* (2014); Uma *et al.* (2012)
1023	1022	-C-O-C- vibration in cellulose	Pejumras *et al.* (2014); Antony *et al.* (2017); Uma *et al.* (2012)
823	892	-CH rocking vibration in cellulose	Pejumras *et al.* (2014); Antony *et al.* (2017); Uma *et al.* (2012)

SEM Analysis

Morphology of the JR cellulose was analyzed by using the SEM instrument that gave information about the surface morphology as shown in Figure 3.

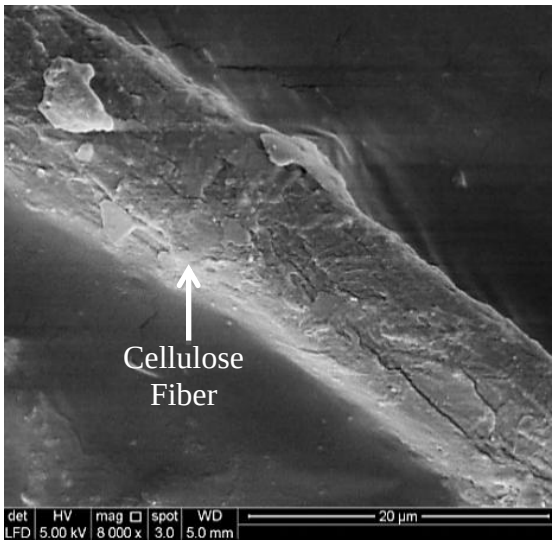

Figure 3: SEM image of JR cellulose
at 8000x magnification

The rough cellulose surface shown by the SEM image was due to the removal of holocellulose and lignin that covered the surface of the jackfruit rind. This microstructural characteristic is comparable to the previous study by Rahman *et al.,* (2016) [5] that used durian rind as raw material.

CONCLUSIONS

The cellulose was successfully extracted from the jackfruit rind (JR) by using bleaching and alkaline treatment at 40%. The cellulose was characterized by using ATR-FTIR and SEM instrument. From SEM analysis, the JR cellulose fibre gave a rough surface indicating the removal of holocellulose and lignin from the surface of the jackfruit rind.

ACKNOWLEDGMENTS

This research was partially funded by Universiti Teknologi MARA Cawangan Perlis, Kampus Arau, Perlis, Malaysia. The authors are also grateful to the management for providing adequate laboratory facilities in the execution of this study.

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