EXTRACTION AND CHARACTERIZATION OF CELLULOSE FROM WASTEPAPER IN UNIVERSITY OF CYBERJAYA

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Abstract: Previous studies on university waste have shown that paper products constitute a large proportion of the solid waste generated. Hence, to reduce paper wastage, wastepaper can be reused for the extraction of cellulose for other application such as biomaterial for the production of biodegradable products. This study is aimed to extract and characterize cellulose from wastepaper collected in University of Cyberjaya (UoC). Pre-treatment with sonicator, alkaline and bleaching treatments were employed for the extraction of cellulose. The functional group present in the extracted wastepaper cellulose (WPC) was determined using fourier transform infrared spectroscopy (FT-IR). Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were carried out to characterize its thermal stability. The hydration property of WPC was evaluated by water retention capacity (WRC). All the characterization analyses of WPC were compared to commercialized microcrystalline cellulose (MCC). 53.1% of WPC was successfully extracted with some extent of lignin and its purity was supported by the FT-IR findings where the functional groups of cellulose were detected. In thermal analysis, WPC has lower thermal stability than MCC due to its lesser crystalline structure. WPC has higher WRC than MCC because of its bigger particle size. The results obtained in this study reflect the effectiveness of the methods applied in extracting WPC. Extraction of WPC for the utilization in biomaterial production is definitely a practical way to reduce paper waste.

Keywords: Cellulose, Extraction, Wastepaper

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INTRODUCTION

Cellulose is one of the most abundant naturally occurring polymers and it is the largest organic carbon source on earth. With high annual production of ~1.5 $\times 10^{12}$ tons, cellulose attracted the world to be considered as the major source of raw materials (Rojas, 2016). It has been widely known that cellulose has numerous beneficial properties such as hydrophilicity, non-toxicity, potential as a sorbent, facile chemical modification, good mechanical properties and safe disposability (Suhas et al., 2016). These properties lead to diverse applications of cellulose as one of the most important commercial raw materials. Cellulose has been used in the papermaking industry since the Chinese invented the process back then. As cellulose can easily react with strong bases and acids, other products can be produced by chemical process. As instance, rayon, a type of fabric and cellophane, a transparent sheet of film are made using numbers of processes that involve acid bath. Cellulose is also extensively used in the manufacturing of other organic products such as ethanol and methanol. In food processing, cellulose has always been used as food additive as a thickening agent and to add bulk in food products. Other than that, it has also been used widely in pharmaceuticals for different applications (Kamel et al., 2008).

Nowadays, paper products constitute a large proportion of solid waste generated by higher education institutions. This rising environmental issue is due to the academic and research endeavors paper and other paper products such as printing paper, newspaper and boxes (Smyth, Fredeen, & Booth, 2010). Generally, paper is made up of up to 87% cellulose (Palmisano & Barlaz, 1996). Due to its abundant cellulosic content that is beneficial for various applications, wastepaper has the potential as a source material for the extraction of cellulose. The extraction of cellulose from wastepaper would provide an alternative to paper recycling and possibly reduce the impact of by-products arising due to paper recycling (Danial et al., 2015).

EXPERIMENTAL METHODS

Extraction of cellulose from wastepaper

The extraction method of WPC used was adapted and modified

from Danial et al., 2015. 30 g of shredded wastepaper collected in University of Cyberjaya (UoC) was placed in an ultrasonic generator. The ultrasonication was conducted for 40 min at 55 ˚C followed by stirring for 80 min at 55 ˚C. The alkaline treatment was carried out with 5% sodium hydroxide (NaOH) at 80 ˚C for four hours. Then, bleaching treatment was conducted with 2% sodium hypochlorite (NaOCl) at 80 ˚C for four hours. The process included continuous stirring. The slurry was the filtered and washed with distilled water until neutral pH was achieved. The resulting material was then freeze dried for 24 hours and finally blended into powder form before being analyzed. The extract yield is expressed as a percentage of extract weight after drying to the total weight of wastepaper following the equation below:

$$\text{Extract yield (\%)} = \frac{\text{Weight of dried product (g)}}{\text{Weight of wastepaper (g)}} \times 100\%$$

Fourier Transform Infrared Spectroscopy

The functional groups present in WPC and MCC were determined by FT-IR spectroscopy. FT-IR spectra were recorded using potassium bromide (KBr) discs on a (Nicolet iS10, Thermo electron, USA) FT-IR spectrometer. KBr pellets containing 1% of each material was prepared. The scanned range was 4000 – 400 cm^{-1}.

Thermal Analysis of Extracted Cellulose

The thermal properties of WPC and MCC were investigated by TGA and DSC on a simultaneous thermal analyzer (METTLER TOLEDO/TGA DSC 1). Samples weighing 5.0 mg for WPC and 5.3 mg for MCC were used. Each sample was heated from 30 °C to 600 °C at a temperature rate of 20 °C/min and flow rate of 10 mL/min under argon.

Water Retention Capacity

The method to study the WRC of WPC and MCC was adapted from Tochampa & Kongbangkerd, 2014. In a 15 mL centrifuge tube, 0.5 g of each sample was mixed with 7.5 mL distilled water. The slurry was allowed to stand for 10 min, and then centrifuged at 2000 × *g* for 15 min by centrifuge machine (Hettich EBA 280). After centrifugation, the supernatant was drained and the wet sample precipitate was weighed. The procedure was repeated thrice to get the average result. The result was expressed as gram of water per gram of the sample. The WRC value was calculated using the equation below:

$$\text{Water Retention Capacity} = \frac{\text{gram of water}}{\text{gram of the sample}}$$

RESULTS AND DISCUSSION

Extraction Yield of Cellulose

The WPC yield was 53.1% of starting material by weight. The application of sonication performed the mechanical action on the cell walls enhanced the accessibility and extractability of hemicellulose and lignin components. It solubilized the hemicellulose and lignin that are mainly present on the outer surface whereas the long cellulose chains were found in the inner parts of the fibers and hence, are difficult to dissolve (Sun & Tomkinson, 2004). The alkaline treatment removed hemicellulose in the pulp of the wastepaper due to oxidation of the bonds between the components by the oxygen radicals generated from NaOH (Ünlü, 2013). Generally, crystalline cellulose is embedded in hemicellulose and the alkaline treatment expanded the hemicellulose matrix, causing the intermolecular hydrogen-bonding to weaken. The brownish solution produced during the process indicates that the lignin and hemicellulose were extracted out from the wastepaper into the solvent.

The bleaching treatment removed colored substances and due to decomposition of NaOCl that oxidizes coloring matter, thus destroying it to simpler compounds, which are then solubilized and washed out from the material, resulting in whiteness (Abdel-Halim, 2014). At the end of the process, the color of WPC turned from greyish to white and it has a fluffy powdery texture.

Spectroscopy Studies

FT-IR spectra of WPC and MCC are shown in Figure 3.1 and Figure 3.2 respectively. Despite minor differences between them, the FT-IR spectra of both WPC and MCC show the general characteristic spectrum of cellulose. Both samples present two main absorbance regions. The first region is in the range 600 – 1700 cm^{-1} whereas the second region is in the range 2800 – 3600 cm^{-1}. The common functional groups and corresponding bands for each component in cellulose were studied and compared in previous literature (Cem Pang, Fun Chin, & Yih, 2011; Hii, Yeap, & Mashitah, 2014; Morán et al., 2008). The peaks at 3331.44 cm^{-1} and 3331.36 cm^{-1} are the absorption peaks of –OH in WPC and MCC respectively. The stretching vibrations of –CH are represented by the peaks between 2890 cm-1 and 2900 cm-1 in both spectra.

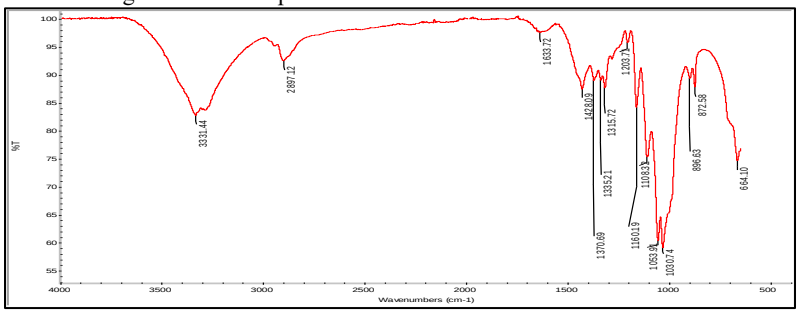

Figure 3.1 FT-IR spectra of WPC.

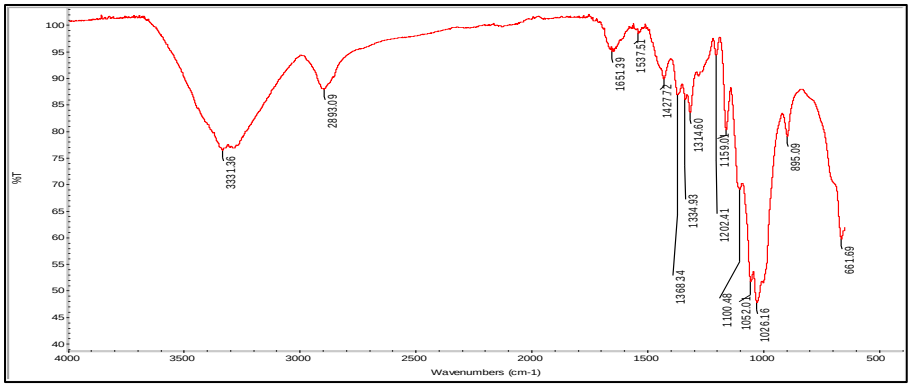

Figure 3.2 FT-IR spectra of MCC.

The presence of bands at 1633.72 cm^{-1} in WPC and 1651.39 cm^{-1} in MCC indicate that there are -OH bending of adsorbed water. Although both samples had undergone drying process, the remaining water molecules are difficult to extract due to the strong hydrogen bonding between the interaction of cellulose and water (Hasan & Sauodi, 2014; Łojewska, et al., 2005; Morán, et al., 2008).

The band observed at 1573.51 cm^{-1} in the MCC spectrum, ascribed to C=C aromatic vibrations which indicates the presence of lignin (Łojewska et al., 2005). Lignin is also composed of O-CH_3 group, which represented by bands at ~1430 cm^{-1} (Morán et al., 2008). Both WPC and MCC show bands at 1428.09 cm^{-1} and 1427.72 cm^{-1} respectively. Thus, it is evident that there is lignin residue remaining after the extraction process.

The absence of bands at 1765 – 1715 cm^{-1} in both WPC and MCC spectra indicates the absence of the functional group C=O for ketone or carbonyl. This indicates that there is no remaining of hemicellulose. Hence, it suggests the effectiveness of alkaline treatment in removing hemicellulose. The similar trend of FT-IR spectra in case of WPC and MCC further supports the successful extraction of cellulose from wastepaper.

Thermal Properties

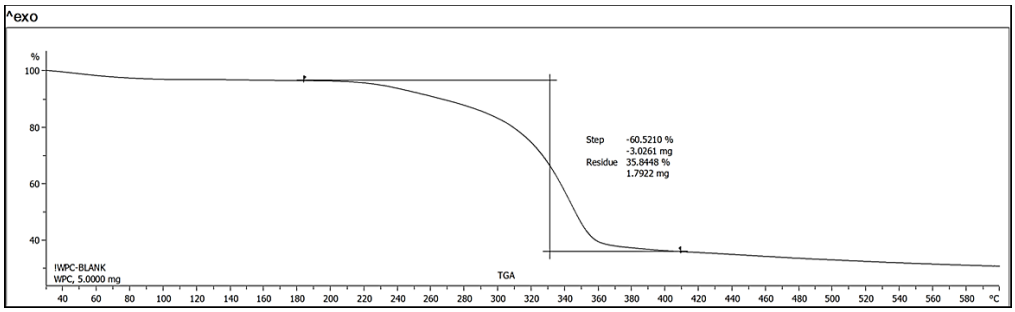

Figure 3.3 TGA curve of WPC.

The TGA curves in Figure 3.3 and Figure 3.4 show that both WPC and MCC follow the similar degradation patterns.

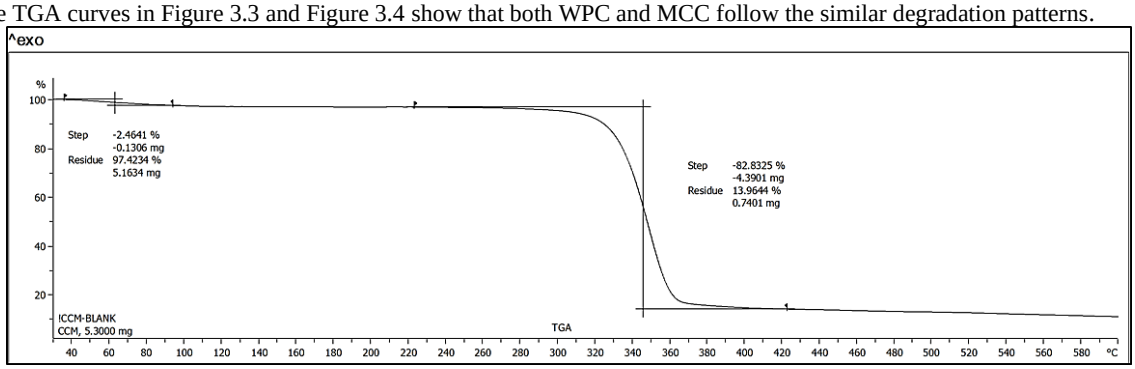

Figure 3.4 TGA curve of MCC.

Both TGA curves for WPC and MCC show small weight loss in the range of 30 °C to 100 °C due to the evaporation of humidity contained or the loss of remaining low molecular weight compounds. The onset of weight loss of the main degradation occurs at about 190 °C for WPC and 220 °C for MCC. WPC shows a maximum rate of weight loss at 330 °C

whereas it occurs in MCC at about 15 °C higher (345 °C). At 550 °C, the residue remained for WPC was 31%, which is higher than MCC that is only left with 11% of residue.

These data indicate that WPC begins to decompose at a lower temperature but left more residues as compared to MCC, which started to decompose at a higher temperature but left lesser residues. Different temperatures of decomposition occur due to the differences in the chemical components in a material. Based on a literature on thermal analysis, cellulose starts to decompose at 315 °C and continues up to 400 °C whereas the decomposition of hemicellulose starts at 220 °C and persists until 315 °C. In lignin, the decomposition starts below 200 °C and remains above 700 °C. The wide temperature range in decomposition of lignin is caused by the various activities of chemical bonds that made up the structure (Yang et al., 2007). Hence, the difference in the thermal decomposition property of WPC as compared to MCC is probably due to the remaining lignin present in WPC.

DSC was used to determine the reduction in crystallinity and decomposition of WPC and MCC. Based on Figure 3.5, early endothermic peak at 30 °C to 70 °C indicates the removal of moisture during the heating of WPC. For MCC, the process of moisture removal occurred at 30 °C to 95 °C as shown in Figure 3.6. These data are supported by the TGA curves as the water evaporation occurred around these ranges of temperature.

WPC also shows an endothermic peak at 342.35 °C while MCC shows a sharp endothermic peak at 347.63 °C. These peaks are corresponding to the fusion of the crystalline part. The lower temperature for the peak to occur in WPC is due to the increase in the amount of amorphous cellulose, which has poorer thermal resistance than crystalline. It is also can be caused by the reduced length of the cellulose crystallite in WPC. The peak difference indicates that MCC has wider molecular weight and size distribution of cellulose crystal as compared to WPC (Morán et al., 2008; Mwaikambo & Ansell, 2002).

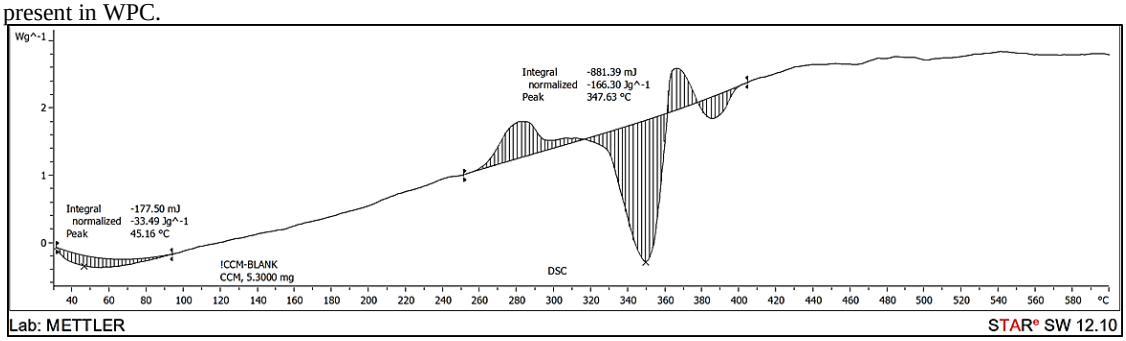

Figure 3.5 DSC curve of WPC.

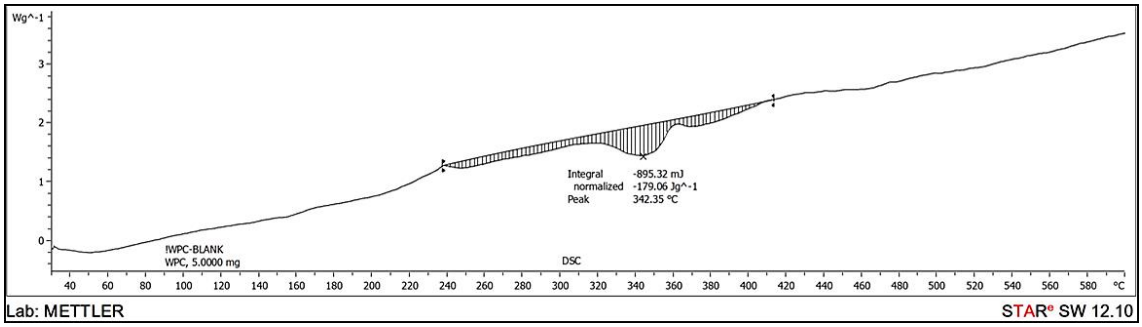

Figure 3.6 DSC curve of MCC.

Water Retention Capacity

The WRC of WPC and MCC were determined to characterize their hydration properties. Based on the results obtained in Table 3.1, WPC has a higher WRC (2.17 g water/g dried sample) than MCC (1.93 g water/g dried sample).

Table 3.1. WRC values for WPC and MCC.

Cellulose sample	WRC (g water/g dried sample)			Average WRC (g water/g dried sample)
	1	2	3	
WPC	2.19	2.14	2.18	2.17
MCC	2.11	1.72	1.96	1.93

A previous study done by Tochampa & Kongbangkerd, 2014, found that the WRC of cellulose extracted from banana peels was higher than the commercial cellulose that has smaller particle size. Furthermore, Cadden, 1987, also found that reduced particle size of cellulose from rice bran resulted in lowered water absorption. The smaller particle size of cellulose has lower bulk, packed and hydrated density properties than the larger particle size. This is because reducing the particle size will damage the fibrous network of the pores that retain water in the cellulose structure resulting in lower water absorption (Cadden, 1987; Tochampa & Kongbangkerd, 2014).

WRC is closely related to many applications of cellulose such

as in stabilizing water based formulations and keeping a surface wetted. Hence, WPC can be modified to enhance its WRC for these kinds of applications to replace the use of synthetic additives and contribute to greener products.

Conclusions

WPC was successfully extracted from wastepaper by applying the combination of physical pre-treatment and low concentration chemical methods. By means of FT-IR, TGA, DSC, and WRC, it was possible to characterize the obtained WPC and analyze the presence of other remaining components. High amount of WPC (53.1%) was successfully extracted with some extent of lignin. From the FT-IR spectra of WPC, general characteristic spectrum of cellulose was observed. In thermal stability, TGA curve shows that WPC is less stable than MCC. The DSC curve of WPC gave significant endothermic peak at a lower temperature that indicates its poorer crystallinity as compared to MCC. However, WPC has a higher WRC value than MCC.

With abundance of wastepaper, extraction method of WPC from wastepaper could be innovated and improved as a proper practice in order to reduce environmental waste. This study can be a good platform to provide more scientific evidences in the suitability of WPC to be utilized as biomaterial for the production of more eco-friendly products.

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