

EXTRACTION AND CHARACTERISATION OF CELLULOSE FROM THE RESIDUE OF OIL PALM EMPTY FRUIT BUNCH-XYLAN EXTRACTION

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ABSTRACT

Cellulosic solid residual, which is the by-product of oil palm empty fruit bunch (OPEFB)-xylan extraction process, was evaluated in terms of cellulose recovery by eco-friendly approach. The OPEFB-fibre was initially treated with alkaline for xylan extraction. The resulting solid residue was dried before treated with mild concentration of bleaching agents of 20% (v/v) formic acid and 5% (v/v) hydrogen peroxide at 85°C for the extraction of cellulose. α -cellulose content of $84.16 \pm 0.4\%$ was achieved. Fourier transform infrared spectroscopy (FTIR) analysis showed that the products obtained have the standard cellulose structure and functional group. The crystalline nature of the extracted cellulose, as proven by X-ray diffractometer (XRD) has crystallinity index and crystalline size of 54.4% and 5.5 nm, respectively, whereas the thermogravimetric analysis (TGA) indicates the sample purity in terms of thermal stability at 370°C by comparing the results to commercially available cellulose. Meanwhile, scanning electron microscope (SEM) observation showed that the extracted cellulose morphology is in the form of separated fibrils. In terms of impurities, trace elemental analysis showed the presence of phosphorus (P) as the major element but the quantity of fluorine (F), chlorine (Cl), bromine (Br), iodine (I) and sulphur (S) detected on the cellulose was insignificant.

Keywords: cellulose, eco-friendly extraction, oil palm empty fruit bunch, xylan extraction residue.

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INTRODUCTION

Oil palm empty fruit bunch (OPEFB) is the most abundant biomass generated by the mills after extracting the oil from fresh fruit bunch (FFB). In 2018, 485 palm oil mills in Malaysia processed 118.9 million tonnes of FFB generating approximately 9.16 million tonnes (dry weight) of OPEFB (Kushairi *et al.*, 2019). The abundant quantity of OPEFB generated in the oil palm mills provides huge resources for conversion into value-added products, as the OPEFB is inexhaustible, renewable,

biodegradable, recyclable and a derivatisable biopolymer. Lignocellulosic material (LCM) in biomass waste composed of about 37.1% cellulose, 39.9% hemicellulose, 18.6% lignin and 3.1% extractives (Megashah *et al.*, 2018). Each of these lignocellulosic components can be extracted and exploited for the production of fine chemicals, especially from cellulose and hemicellulose.

In previous study, a method was developed to produce the xylooligosaccharides (XOS) from OPEFB fibre at laboratory scale (Noorshamsiana *et al.*, 2015). In this method, hemicellulose was extracted from OPEFB-fibre using alkaline solution and precipitated into xylan as starting material for the production of XOS using enzymatic method. XOS is one of the most promising prebiotic products which is extensively studied and proven to

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stimulate microflora growth in the gastrointestinal tract (Manderson *et al.*, 2005; Parajó *et al.*, 2004). In fact, XOS gains much attention as it possesses many health benefits, particularly in prebiotic activities besides being food additive and health products (Gupta *et al.*, 2012; Seema and Arun, 2011; Moure *et al.*, 2006; Alonso *et al.*, 2003). The solid residue generated from xylan extraction using alkaline solution has yet to be explored. The solid residue is in fact a LCM which consists of cellulose, lignin, as well as some traces of hemicellulose. Cellulose is another value-added product from natural fibre and it is useful in food and pharmaceutical applications. Cellulose is the most abundant polysaccharide, comprising about 44%-64% of plant materials (Mansor *et al.*, 2019; Nazir *et al.*, 2013; Dhillon *et al.*, 2013; Nawirska and Kwasniewska, 2005; Wanrosli *et al.*, 2004; McKendry, 2002) and OPEFB-fibre contains about 40%-65% of cellulose (Megashah *et al.*, 2018; Zakaria *et al.*, 2015; Adelaet *et al.*, 2014; Chang *et al.*, 2014; Ching and Ng, 2014; Khalil *et al.*, 2008; Law *et al.*, 2007). Cellulose is a natural long-chain polymer that indirectly contributes to the human food cycle. This polymer has versatile uses in many industries, such as veterinary food, pulp and paper, and fibre for clothing. In addition, cellulose has a semisynthetic derivative which is extensively used in pharmaceutical and cosmetic industries.

Cellulose can be extracted from natural fibres via chemical and mechanical methods. Acidified sodium chlorite ($\text{NaClO}_2 + \text{H}_3\text{O}^+$) is commonly used as the standard reagent for cellulose delignification and extraction from wood materials (Szymańska-Chargot *et al.*, 2017; Hook *et al.*, 2015; Brendel *et al.*, 2000; Leavitt and Danzer, 1993; Loader *et al.*, 1997; Wise *et al.*, 1946). Chlorite (ClO_2^-) produces chlorine radical which is reactive and fragments the LCM into highly toxic organochlorine. The cellulose extraction from OPEFB-fibre involves pretreatment with toluene/methanol (2:1, v/v) (Sun *et al.*, 1999) and ethanol/benzene (1:2) (Fahma *et al.*, 2010), with further delignification using chlorite to prepare microcrystalline cellulose (Rosnah *et al.*, 2009). However, the existing procedures produce low yields as well as neither environment-friendly nor energy efficient. Therefore, more focus should be placed on eco-friendly methods in cellulose extraction by utilising green treatment that reduces or eliminates hazardous chemicals, using safer solvent and safer reaction conditions. Organic acids such as acetic acid and formic acid are eco-friendly reagents, less corrosive and effective for the pretreatment of lignocellulosic biomass, and they provide a more stable medium in the aqueous phase for monosaccharide than sulfuric acid (Marzialetti *et al.*, 2011). A mixture of hexane/ethanol and water is used to remove wax from the rice husk, followed by autoclaving and ultrasound treatments to delignify and purify cellulose using hydrogen

peroxide and acetic acid (Simone *et al.*, 2012). The cellulose extraction from OPEFB using hydrogen peroxide with ultrasound-assisted alkali extraction produced cellulose with 49% yield (Nazir *et al.*, 2012). The use of low concentrations of formic acid (20%, v/v) and 30% hydrogen peroxide (10%, v/v) at 85°C to extract cellulose from OPEFB produced 64% (w/w) cellulose, which is the highest ever reported (Nazir *et al.*, 2013). The development of a novel process that uses ionic liquid followed by alkaline method to extract cellulose from OPEFB fibre resulted in 93% α -cellulose recovery (Norzita and Lani, 2014). From the literature, most studies only focused on cellulose extraction from raw OPEFB fibre. This research focused on cellulose recovery from the byproduct of hemicellulose extraction from OPEFB.

This study aims to extract cellulose from the by-product of hemicellulose extraction using green method in order to solve the disposal problem of the process as well as to create another value added products from the OPEFB-fibre. Thus, the use of bleaching agents in mild concentration to recover cellulose from the residue of OPEFB-xylan extraction is out of concern for environmental protection. The characterisation of the product obtained was carried out by fourier transform infrared spectroscopy (FTIR), X-ray diffractometry (XRD), thermogravimetric (TG), elemental analyser and scanning electron microscopy (SEM).

MATERIALS AND METHODS

Materials

OPEFB-fibres were obtained from Biomass Plant, Biomass Technology Unit, MPOB-UKM in Bangi, Malaysia. The treated and dried OPEFB fibre was ground and stored in the cold room prior to use. The hemicellulose extracted from OPEFB fibres using alkaline solution and precipitated into xylan was the starting material for the production of XOS using enzymatic method (Noorshamsiana *et al.*, 2015). The resulting solid residue from the xylan extraction process was further treated for cellulose recovery. The cellulose standard derived from spruce with >99% purity (FLUKA) was used as reference. All chemicals and solvents used are of reagent grade and were used as received.

Cellulose Recovery from the Solid Residue of Xylan Extraction

Cellulose extraction was adapted and modified from the method developed for OPEFB-fibres (Nazir *et al.*, 2013). The solid residue was dried in the oven at 50°C to a constant weight before extracting. Two grams of solid residue were soaked in a mixture of 100 ml 20% formic acid (CH_2O_2) and 100 ml 5%

hydrogen peroxide (H_2O_2). The mixture was then placed in the shaking water bath (WiseBath, Daihan Scientific; 45 litre) at $85^\circ C$ for 2 hr. After hydrolysis, the samples were filtered with grade 4 filter paper (Whatman, England) and the supernatant washed with 10% CH_2O_2 before being washed with distilled water several times. The supernatant was resuspended with 10% H_2O_2 and 10% NaOH at pH 11 and $60^\circ C$ for 90 min. The product obtained was then filtered, washed with distilled water and dried in oven at $60^\circ C$ before being characterised. The α -cellulose content was determined by TAPPI T203 method.

Fourier Transform Infrared Spectroscopy (FTIR)

The infrared spectrum of the extracted cellulose was recorded using Perkin-Elmer Spectrum Frontier FTIR Spectrometer with wavelength in the range of $4000-650\text{ cm}^{-1}$. The spectrum was compared with standard cellulose.

X-ray Diffraction (XRD)

The phase behaviour of standard cellulose and extracted cellulose was elucidated by using an X-ray diffractometer (D8-Advance Bruker-AXS Germany). The peak measurement was conducted by taking 2Θ value between 5° and 60° with $0.04^\circ\text{ min}^{-1}$ step size. The crystallinity index (C_{ir}) was calculated based on Segal method (Segal *et al.*, 1959) as follows:

$$C_{ir} = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad \text{Equation (1)}$$

where I_{002} is the maximum peak intensity of crystalline fractions located at $2\Theta = 22.0^\circ - 22.5^\circ$, whereas I_{am} is the low peak intensity in the amorphous region at $2\Theta = 16^\circ$, which corresponds to the crystallographic plane.

Crystallite size (ϵ) is used to indicate the quality of the crystals. It was determined using the Scherrer's equation (Patterson, 1939) as follows:

$$\epsilon = \frac{k\lambda}{\beta \cos \Theta} \quad \text{Equation (2)}$$

where k is the constant of 0.91, λ is the x-ray wavelength, β is the full width at half maximum (FWHM) peak intensity and Θ is the Bragg's angle.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) of the standard cellulose and extracted cellulose were performed using a Perkin Elmer Pyris 1 thermogravimetric analyser. About 10 mg to 20 mg of

each sample was analysed continuously from $30^\circ C$ to $600^\circ C$ at $20^\circ C\text{ min}^{-1}$ heating rate. The analyser was purged with nitrogen, followed by 20 ml min^{-1} oxygen flow to prevent any thermo-oxidative degradation.

Scanning Electron Microscope (SEM)

Morphological studies of the extracted cellulose and standard cellulose were carried out with a scanning electron microscope (SEM), Hitachi S-3400 N. A piece of dry sample was put on the SEM sample lens, then the lenses were placed in a vacuum chamber to avoid obstruction and contamination by other particles. The lenses helped to direct electrons towards the sample. The electron imprint was converted into three-dimensional image. Elemental analysis was performed using an energy-dispersive X-ray (EDX) coupled with SEM. When electron beam penetrated the sample, the produced X-ray was collected, analysed and detected by silicon drift detector (SDD) and the X-ray map recorded.

Elemental Composition Analysis

The elements in the cellulose samples were analysed using CHN628 Series Elemental Determinator. The instrument is used to determine nitrogen, carbon/nitrogen, and carbon/hydrogen/nitrogen composition in organic matrices by utilising combustion technique.

Trace Element Content

Cellulose samples were analysed for nitrogen, sulphur and halogen content using NSX-2100 Automatic Combustion Unit (Model AQF-2100 series).

RESULTS AND DISCUSSION

Eco-friendly Extraction of Cellulose from the Solid Residue of OPEFB-xylan Extraction Process

The solid residue generated from OPEFB-xylan extraction was treated with mild bleaching agents of 20% (v/v) formic acid and 5% (v/v) hydrogen peroxide at $85^\circ C$, which yielded $82.69 \pm 3.9\%$ cellulose with α -cellulose content of $84.16 \pm 0.4\%$. Formic acid was used to remove hemicellulose, leaving the solid residue to have lesser hemicellulose with high cellulose content (Goldmann *et al.*, 2017; Li *et al.*, 2015). Hence, more cellulose was obtained in the treated product. The major contributor of cellulose extraction was formic acid because it is very stable in aqueous medium (Nazir *et al.*, 2013). A stable formic acid provided an alternative for the interaction and dissolution of non-crystalline

part in cellulose. This helped to remove the non-lignin parts, hemicellulose and lignin by converting the hemicellulose into salt and lignin was converted to carboxylic acid. Hemicellulose and lignin were removed as water-soluble fraction while the insoluble crystalline cellulose was left suspended in the aqueous medium. A study by Li *et al.* (2015) found that formic acid is useful to remove hemicellulose, swells cellulose fibres, and releases cellulose nanocrystals (CNC). The findings showed that formic acid does increase the amount of hemicellulose extracted from biomass fibre, leaving behind the cellulose component. Furthermore, it was found that more cellulose can be recovered using formic acid at low temperature. The low temperature of 85°C favours hemicellulose hydrolysis rather than cellulose hydrolysis, resulting in more cellulose to be retained in the solid residue (Goldmann *et al.*, 2017). It was also observed that cellulose recovery decreased with the increase in hydrogen peroxide concentration. This is due to the presence of H⁺ ions that help to strengthen the microfibrillated lignin-cellulose structure, stiffness and stability (Lindman *et al.*, 2010).

Chemical Characterisation

Fourier transform infrared spectroscopy (FTIR). The extracted cellulose was analysed with FTIR and compared with standard cellulose (FLUKA), to determine the composition of functional group and confirm its chemical structure.

FTIR analysis showed that the spectrum of the OPEFB-based cellulose extracted using eco-friendly reagents was identical to that of standard cellulose, suggesting the presence of similar backbone and functional groups in both samples (Figure 1). The

O-H bond which indicates the presence of -OH groups on the glucose unit is characterised by the broad band found in the range of 3200-3400 cm⁻¹ (Norzita and Lani, 2014; Moran *et al.*, 2008; Khalil *et al.*, 2001). In this study, the O-H bond of the extracted cellulose and standard cellulose was found at 3327.84 cm⁻¹ and 3330.34 cm⁻¹, respectively. The medium peak at 2891.61 cm⁻¹ for extracted cellulose and 2893.33 cm⁻¹ for standard cellulose reflects the C-H bonds. Meanwhile, the peak at 1023.84 cm⁻¹ for extracted cellulose and 1052.33 cm⁻¹ for standard cellulose is assigned to the C-O-C linkage of the anhydrous repeating units. The peaks at 1735 cm⁻¹ and 1375 cm⁻¹, which represent wax and hemicellulose, respectively were not present in the extracted cellulose, indicating both components were removed during bleaching. From literature, the FTIR spectral peaks representing lignin at 1248 cm⁻¹ and 1037 cm⁻¹ were not observed in the extracted cellulose (Ching and Ng, 2014; Nazir *et al.*, 2013; Simone *et al.*, 2012). It was observed that the extracted cellulose had lower transmittance than standard cellulose. This may be due to the compound that exists in the extracted cellulose causes an increase in absorptivity, which leads to lower transmittance percentage. The extracted cellulose has similar structure and functional groups to that of standard cellulose thus confirming that the product was cellulose.

X-ray diffraction (XRD). The X-ray diffraction (XRD) patterns of the extracted cellulose and standard cellulose are shown in Figure 2. The diffractogram (diffraction pattern) shows the presence of crystalline and amorphous fractions in both samples.

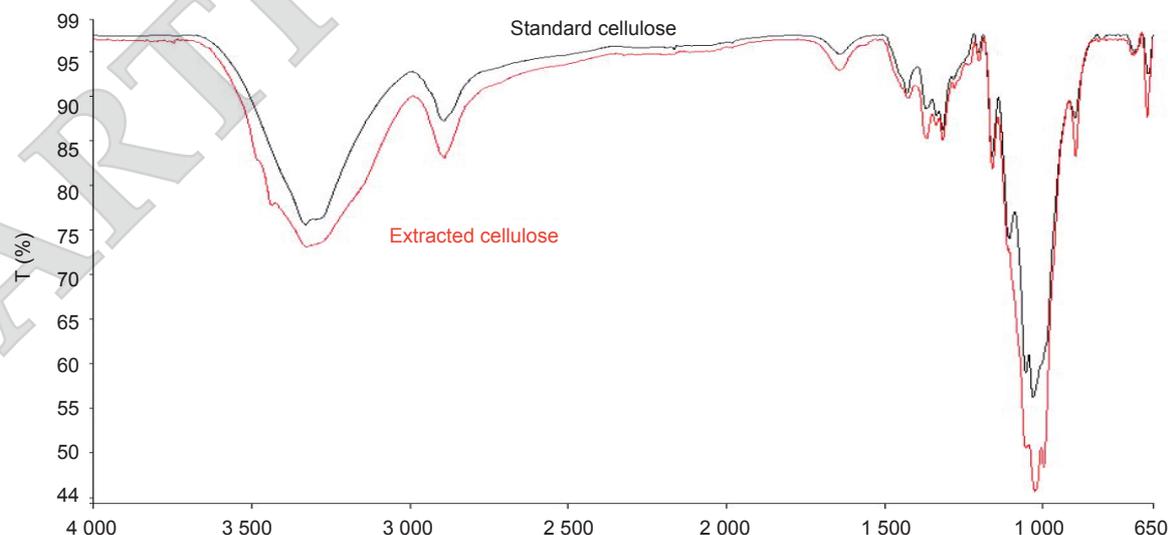


Figure 1. Fourier transform infrared spectroscopy (FTIR) spectra of extracted cellulose and standard cellulose.

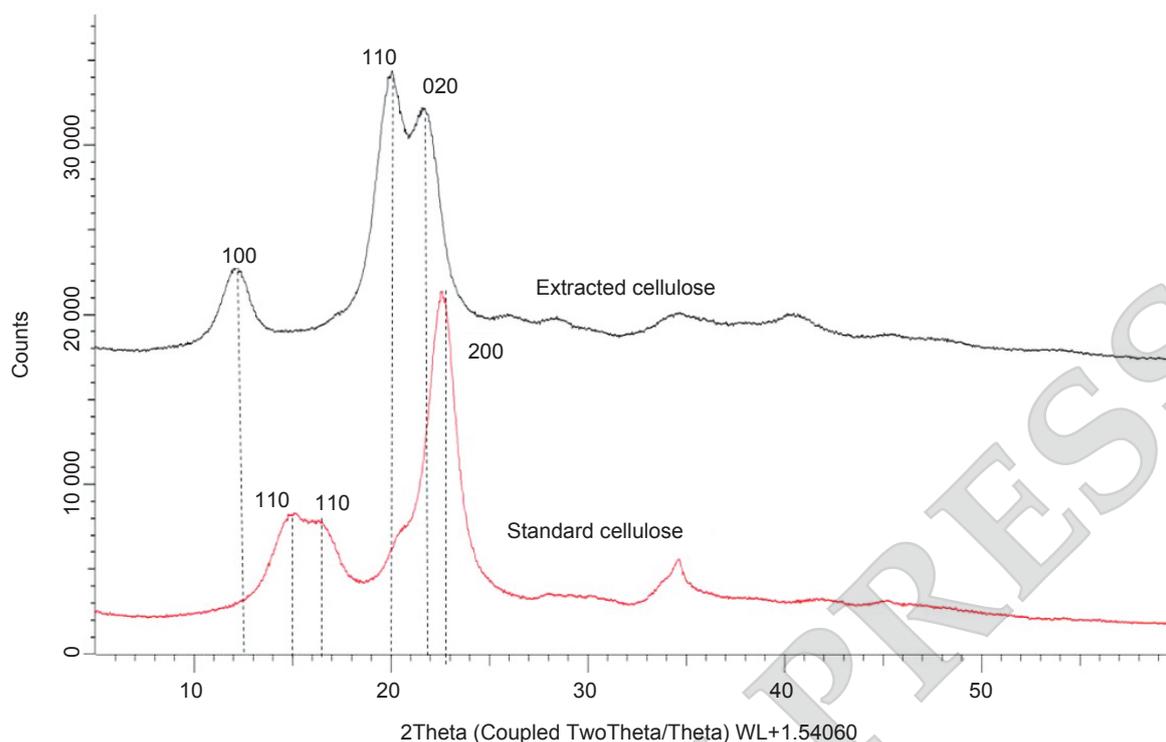


Figure 2. X-Ray diffraction (XRD) patterns of cellulose extracted from the residue of OPEFB-xylan extraction and standard cellulose derived from spruce wood.

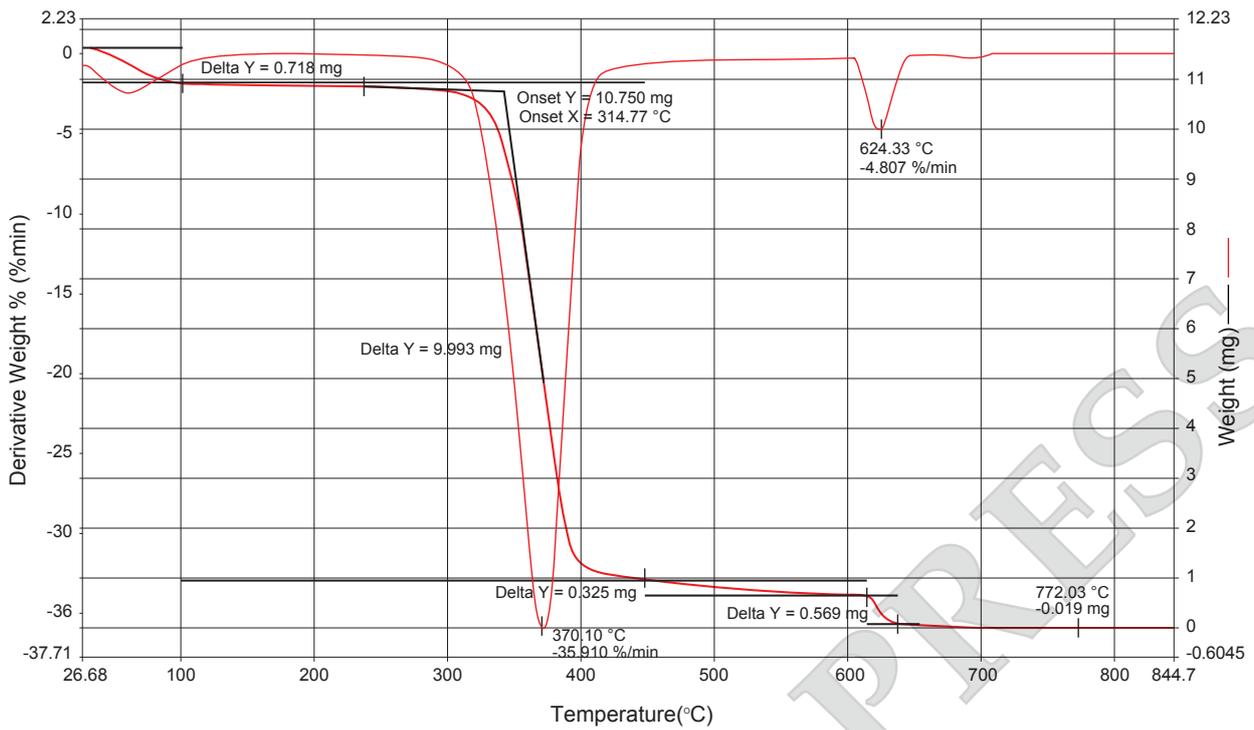
It is observed from Figure 2 that the standard cellulose has diffraction peaks around $2\Theta=20^{\circ}$ - 24° while the extracted cellulose has diffraction peaks at $2\Theta=18^{\circ}$ - 24° . The standard cellulose crystallinity index (C_{lr}) and crystalline size (ϵ) were 71.9% and 6.0 nm, respectively, whereas for the extracted cellulose, they were 54.4% and 5.5 nm, respectively. For comparison purposes, the crystallinity indexes of cellulose derived from native OPEFB fibres in the previous studies were 82.4% (Megashah *et al.*, 2018), 70% (Nazir *et al.*, 2013), 69% (Jonoobi *et al.*, 2011), 63% (Norzita and Lani, 2014) and 55% (Fahma *et al.*, 2010). In general, the structures of the natural cellulose fibres are classified as type I, II, III and IV. It is observed that the standard cellulose is classified as cellulose type I as it has no doublet in the main peak of $2\Theta=23^{\circ}$ (Norzita and Lani, 2014; Bhatnagar and Sain, 2005). Meanwhile, the cellulose derived from the solid residue of OPEFB-xylan extraction using mild bleaching agents is categorised as type II. This is based on the crystalline peak that appears as a doublet at $2\Theta=20^{\circ}$ and $2\Theta=22^{\circ}$, demonstrating the transformation of native cellulose from cellulose type I to cellulose type II.

Thermogravimetric analysis (TGA). The thermal behaviour and thermal stability of the samples obtained were studied using TGA to determine the changes in mass when temperature is increased over time.

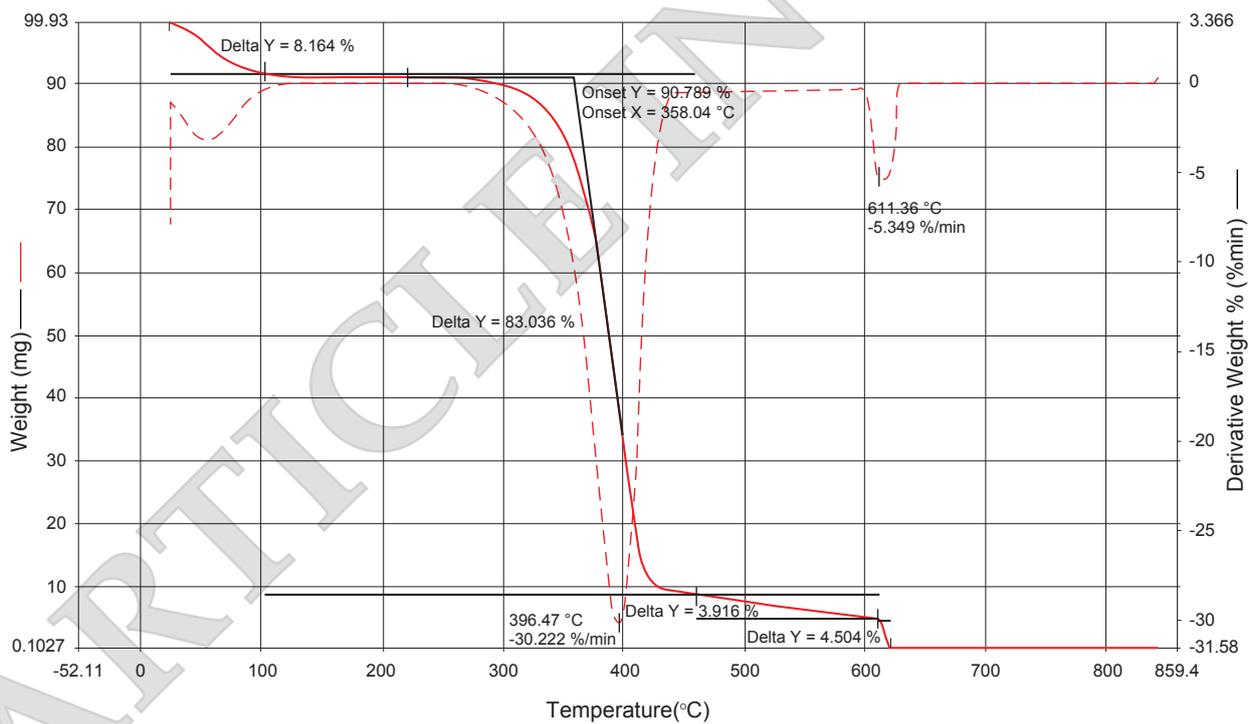
TABLE 1. THERMAL ANALYSIS OF CELLULOSE

Samples	T _{50%} (°C)	T _{max} (°C)
Extracted cellulose	341.77	370.10
Standard cellulose (FLUKA)	358.04	396.47

Figures 3a and 3b show the thermogram of the extracted cellulose and standard cellulose, respectively. It was observed that both cellulose samples exhibited single step degradation in the temperature range of 200°C-400°C, indicating that the degradation was due to one main component. Weight loss was observed at around 100°C, due to the loss of moisture in both samples. Looking at Table 1, it was observed that the standard cellulose was degraded by half at higher temperature and has higher maximum thermal decomposition compared to the extracted cellulose. The findings suggest that standard cellulose has better thermal stability than extracted cellulose. This is because the thermally unstable component in the extracted cellulose contains celluloses from different sources. The standard FLUKA cellulose (purity >99%) is isolated from spruce wood, while the extracted cellulose is from the empty fruit bunch fibres. In addition, the cellulose extraction for these two samples are different, whereby, the standard cellulose is extracted using chemical method, whereas the



(a)



(b)

Figure 3. Thermogravimetric profile of (a) extracted cellulose (b) standard cellulose.

cellulose in this study was extracted using eco-friendly reagents. Also, crystallinity is important in determining thermal stability. Lower crystalline region in the extracted cellulose results in lower thermal stability (Megashah *et al.*, 2018; Nascimento *et al.*, 2015; Poletto *et al.*, 2014).

Scanning electron microscope (SEM). Surface morphology of the solid residue from the OPEFB-xylan extraction is depicted in Figure 4, whereas the surface morphology of the extracted cellulose and standard cellulose are depicted in Figures 5 and 6, respectively.

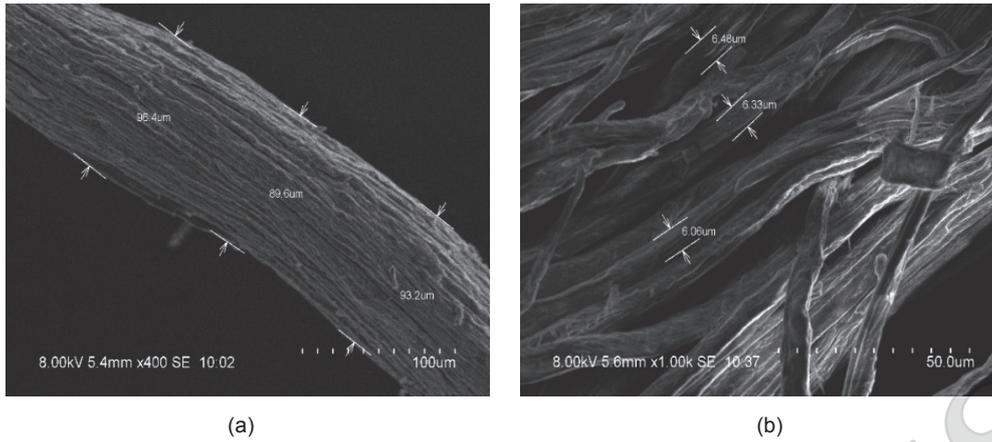


Figure 4. Scanning electron microscope (SEM) micrograph of the OPEFB-xylan extraction solid residue; (a) 400X magnification, (b) 1000X magnification.

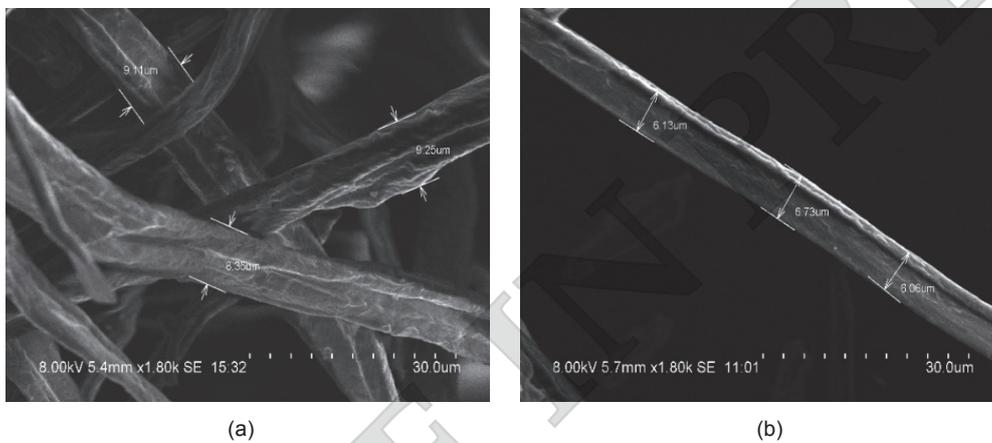


Figure 5. Scanning electron microscope (SEM) micrograph of the extracted cellulose; (a) 1800X magnification, (b) 1800X magnification.

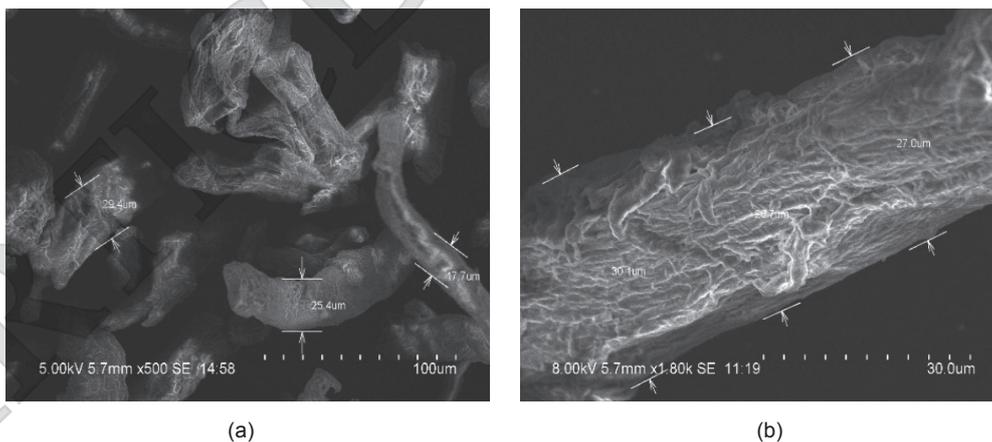


Figure 6. Scanning electron microscope (SEM) micrograph of the standard cellulose; (a) 500X magnification, (b) 1800X magnification.

Figure 4a shows that the solid residue of OPEFB-xylan extraction is a lignocellulosic composite with compact fibril packing, having average diameter of $93.07 \pm 2.78 \mu\text{m}$, suggesting incomplete removal of lignin and hemicellulose (Megashah *et al.*, 2018; Nazir *et al.*, 2013). Complete microfibrillation with

microfibrils in the fibre bundle with diameter of $6.29 \pm 0.17 \mu\text{m}$ is observed in Figure 4b. The extraction of hemicellulose (xylan resource) from native OPEFB fibre via alkaline treatment generates solid residue consisting of cellulose, lignin while the remaining hemicellulose results in compact

fibrillated structure. From *Figure 5a*, the microfibrils of the extracted cellulose have average diameter of $8.90 \pm 0.39 \mu\text{m}$, indicating the disruption of lignocellulosic structure. The fibre bundles were separated into individual cellulose microfibrils, indicating that lignin and hemicellulose were removed through bleaching (Megashah *et al.*, 2018; Sofla *et al.*, 2016; Zeng *et al.*, 2014). As depicted in *Figure 5b*, there was an individual cellulose microfibre with a diameter of 6.31 ± 0.30 . *Figures 6a* and *6b* show incomplete microfibrillation as compared to solid residue and extracted cellulose morphologies due to the presence of non-fibrillated structure. There are uniform microdimensional crystal bundles and smooth surface, indicating that it was in the crystalline form. This supports the XRD results that standard cellulose has higher crystallinity index than extracted cellulose. The smooth surface indicates there is no inorganic materials or impurities on the standard cellulose.

Elemental Analysis

Chemical compositions analysis. As shown in *Table 2*, oxygen was detected as the main element in both samples besides carbon and hydrogen. Nitrogen was also detected but the value was insignificant.

No sulphur was found in both samples. The carbon (C), hydrogen (H), oxygen (O), and nitrogen (N) composition in the extracted cellulose was similar to standard cellulose.

Trace elements analysis. The existence of impurities in samples indicates the quality of the product. The quantity of trace elements and halogen content in both samples is tabulated in *Table 3*.

From the findings, no bromine was detected in both samples. The concentration of chlorine in the extracted cellulose (0.21%) was comparable to the standard cellulose (0.22%). It is deemed negligible as the amount is below 1.0%. Fluorine (F) (0.01%), iodine (I) (0.13%), and sulphur (S) (0.04%) were also detected but in insignificant quantity. Concentration of phosphorus (P) was highest in both extracted and standard celluloses, at 1.20% and 1.40% respectively. P comes from soil and fertiliser. Fertiliser is used in the oil palm plantation when the available P in the soil is less than 15 mg kg^{-1} (Sundram *et al.*, 2019; Afandi *et al.*, 2016; Goh and Rolf, 2003; Adiwiganda, 2002; Rankine and Fairhurst, 1999). P is essential for plant growth and also important for root growth in the early growth stage of the plant. Therefore, the presence of P in the OPEFB-based cellulose is higher than other elements.

TABLE 2. CHEMICAL COMPOSITION OF THE EXTRACTED AND STANDARD CELLULOSE

Components	Chemical composition (%)	
	Extracted cellulose	Standard cellulose
Carbon (C)	39.58±0.11	41.66±0.49
Hydrogen (H)	6.01±0.09	6.60±0.11
Oxygen (O)	54.35±0.24	51.73±0.60
Nitrogen (N)	0.06±0.04	0.01±0.00
Sulphur (S)	Not detectable	Not detectable

Note: Data reported are the means of three replicate analyses of independent samples.

TABLE 3. CONTENTS OF TRACE ELEMENTS AND HALOGEN IN THE EXTRACTED AND STANDARD CELLULOSE

Elements	Concentration (%)	
	Extracted cellulose	Standard cellulose
Fluorine (F)	0.01±0.0	0.01±0.0
Chlorine (Cl)	0.21±0.01	0.22±0.01
Bromine (Br)	Not detectable	Not detectable
Iodine (I)	0.13±0.01	0.14±0.01
Sulphur (S)	0.04±0.01	0.07±0.01
Phosphorus (P)	1.20±0.17	1.40±0.01

Note: Data reported are the means of three replicate analyses of independent samples.

CONCLUSION

Cellulose with α -cellulose content and crystallinity index of $84.16 \pm 0.4\%$ and 54.4% , respectively was successfully recovered from the OPEFB-xylan extraction residue using green method of 20% formic acid and 5% hydrogen peroxide. The results from FTIR, TG, and elemental analyses showed that the extracted cellulose has similar chemical properties and characteristics of standard cellulose. With the recovery of all OPEFB components, disposal issue can be solved, as well as creating value-added products from OPEFB-fibre.

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