Extraction and Characterization of Cellulose Derived from Oil Palm Empty Fruit Bunch

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Abstract
Cellulose derived from oil palm empty fruit bunch (OPEFB) is an alternative low cost of absorbent. Cellulose possesses significant functional hydroxyl groups which available for modification. Alkaline pre-treatment and bleaching process was done to produce high purity of cellulose fiber (Cell-OPEFB). The functional groups and crystallinity in Cell-OPEFB was identified by Fourier Transformation Infrared Spectroscopy (FTIR) and X-Ray Powder Diffractometer (XRD) respectively. The topological properties were examined by Field-Emission Scanning Electron Microscopy (FESEM), while the elemental composition was analyzed by Energy Dispersion X-Ray Spectroscopy (EDX). The thermal composition was determined by TG/DTG. FTIR spectrum represented the characteristic peaks of cellulose at approximately 3450 cm^{-1}, 2900 cm^{-1}, and 1060 cm^{-1} associated with O–H, C–H and C–O stretching, respectively. The XRD result showed type 1 cellulose with main peaks at $2\theta = 15º\text{-}16º$ and $2\theta = 21º\text{-}22º$ which represent (110) and (200), respectively. FESEM morphology shows that the surface of Cell-OPEFB appeared as a separate fibril with a smooth surface while EDX analysis results presented that fundamental of Cell-OPEFB was element C followed by element O. TGA analysis showing thermal stability for Cell-OPEFB achieved at 350ºC.

Keywords: Oil palm empty fruit bunch (OPEFB), cellulose, cellulose derived from OPEFB, natural cellulose, natural fiber.

1.   Introduction

Cellulose is a durable and strong natural materials [1]. Cellulose properties consisting of fibrous polymers, resistant and tough to dissolve in water bring it as an important character in maintaining the structure of plant cell walls. Besides, cellulose is also used as an alternative to nondegradable fossil fuel-based polymers because it is a natural polymer which is biodegradable, biocompatible, and renewable properties [2]–[6].

Cellulose is formed by condensation process, and the structure of cellulose (Figure 1) is contains of monomers joined together through the glycosidic oxygen bridges [2]. Cellulose with a molecular formula $(C_6H_{10}O_5)n$, is consists of β-1,4-linked glucopyranose units that constitute a linear homopolymer with high–molecular-weight, and the position of every unit monomer is exactly at 180° to its neighbours [2], [7]. Available natural polymers on the earth mostly are the cellulose [1], [2], [4], [6], [8]–[11] which is appear in various types of plants, and play as important base for the formation of cell wall. Mostly, cellulose can be obtained from plant-based substances such as grasses, water plants and agricultural residue [3], [12].

Cellulose is commonly classified into four crystal categories that is cellulose I, cellulose II, cellulose III and cellulose IV [7], [13]. According to literature, natural cellulose is classified as cellulose I and mostly extracted from plant, wood, cotton, and straw. Cellulose II is the cellulose that has been regenerated from natural cellulose through alkaline treatment and recrystallization with anti-parallel aligned chains. Regenerated cellulose has lower molecular weight, polymerization degree and crystallinity compared to natural cellulose; besides the entanglement is also less. On the other hand, cellulose I or cellulose which has been treated by using ammonia liquid will produce another type of

cellulose which is III1 or III2 and both products are classified as cellulose III. Lastly, cellulose IV is the cellulose that is generated from III1 or III2 through heating with glycerol to gain IV1 or IV2 [7].
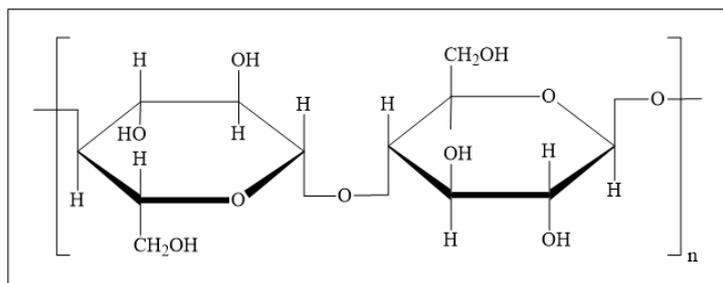

Figure 1. Chemical structure of cellulose

Cellulose is a natural polysaccharide which abundantly available in the earth [1], [3], [12]. A lot of researchers have derived cellulose from various fibers and plants, such as banana yarns, wood, potato peel waste, coconut husk fibers, oil palm empty fruit bunch, mengkuang leaves and kenaf fiber [12]. Other than plants, cellulose is also appear in a most kind of living species; for example in fungi, algae, bacteria, and part of sea animals such as tunicates [1], [2].

One of the known sources of cellulose is from oil palm empty fruit bunch (OPEFB). Asian country becomes the most country that produce palm oil production which cover more than 85% of world palm oil with Malaysia is the second largest country after Indonesia [11]. Malaysia has produced more than 39% of global palm oil production and 44% of global palm oil export to promote the local economic circumstances. About 150 kg of fresh fruit bunch (FFB) was produce per year for each plant and the weight of FFB is around 10-40 kg, depending on the number of fruits that are still intact in the bunch [9].

However, there is a problem that the industry has not yet overcome which is the solid and liquid waste management. The major contributor to the organic solid and liquid waste in Malaysia was from palm oil industry [12]. More than 70% of waste can be produced during harvested palm oil process [11]. Among the problems that occur including the formation of waste from empty fruit bunch, fresh fruit bunch, palm fronds, oil palm trunks, along with palm oil mill sludge, palm oil mill effluent and emission of CO_2. From the total amount of waste presented, OPEFB waste contribute 9% from the total waste.

Cellulose extracted OPEFB fiber has attracted growing interest because of their unique characteristics, including low cost, lower density, high specific strength, good thermal properties, and biodegradable. Malaysia is currently accounts for 39% of world palm oil production and 44% of world exports, hence large quantity of the biomass is generated. Furthermore, it has the highest composition of cellulose compared to coir, corn, bagasse, and kenaf fiber.

OPEFB was generated during extraction of palm oil from FFB [14], [15]. During palm oil processes, it will generate a total of 77.2 million tonnes of biomass in wet and dry basis, where 19.8 million tonnes on wet basis while 6.93 million tonnes on a dry basis, which is OPEFB. This makes OPEFB the highest wastes from palm oil industries [14]. Researcher have reported that OPEFB waste contains 40-65% cellulose, 15-25% hemicellulose and 12-20% lignin composite which can be utilized further to multiple application in different types of field [16], [3], [15], [17]. Since the high value in cellulose which been extracted from OPEFB and plenty sources in Malaysia, therefore the extraction of cellulose from OPEFB are very interesting to be examined and developed.

Cellulose extraction method can be various, such as sulfuric acid hydrolysis, grinding, TEMPO-mediated oxidation, cryocrushing and enzyme-assisted hydrolysis [12]. The most effective way to get a higher purity of cellulose is by chemical treatment. Many researchers had use alkaline treatment methods then followed by chlorite bleaching to extract cellulose from plants [12]. Alkaline treatment is used to dissolve the lignin layer and the excess pectin and hemicellulose [12], [15]. Study conducted by researchers found that alkaline treatment is only capable of eliminating part of hemicellulose and lignin. Further chemical treatment is required to obtain higher content of α-cellulose which is through the bleaching process [12]. Chlorite bleaching serves to extract holocellulose from raw cellulose fiber. This process helps the removal of most of the lignin contained in the fibers, which bring to defibrillation [12].

2. Methodology

2.1. Materials

Raw OPEFB used in this study was obtained from Tai Tak Palm Oil Mill, Kota Tinggi, Johor, Malaysia. Sodium hydroxide and ethanol were purchased from Merck, while toluene was purchased from J.T. Baker. Next, sodium chlorite was purchased from Acros Organics and acetic acid was purchased form QReQ. Lastly, palmitoyl chloride was purchase from Sigma-Aldrich and stearic acid was purchased from ChemPur®.

2.2. Preparation of OPEFB

Pre-treatment and bleaching process is needed to produce high purity of cellulose fiber. This process will remove hemicellulos, lignin and other extractives such as waxes and ashes. Raw OPEFB obtained was washed with 1% dish detergent to remove any impurities and visible dirt (mud, sand etc.) adhering on the surface. Next OPEFB waste was washed with tap water until dark brown water turned to colourless. Then it was dried in the sun for several days until it has parched. Next, the dried sample were cut into small pieces by using grinder. The OPEFB fine fibers was immersed with distilled water to eliminate dust, forming a sludge mixture. The sludge then was filtered out with filter paper to remove excess water. The process was repeated for three times and the remaining OPEFB fiber was dried overnight at 353K. Dried OPEFB fiber were kept for cellulose extraction at further step.

2.3. Preparation of Cell-OPEFB

2.3.1 Alkaline treatment

Firstly, OPEFB fiber was immersed in toluene-ethanol mixture with ratio of 2:1 and was continuously stirred for 6 hours. Then, the OPEFB sludge was separated from the toluene-ethanol mixture by using filtration and the remaining sample was dried overnight at 353 K. Next, OPEFB fiber was digested with 0.1 M sodium hydroxide (NaOH) solution with continuously stirred for 4 hours at temperature 353 K. The alkali treatment promotes the removal of hemicellulose, lignin, waxes, and pectin and produces rough surface topography [15], [18], [19]. Lignin is the most difficult component of biomass to be degraded due to its complex structure, high molecular weight, and high insolubility. Lignin is linked by carbon–carbon and ether bonds to form tri-dimensional network associated with the hemicelluloses polysaccharides inside the cell wall [19]. In addition, alkali treatment leads to breakdown of the fiber bundles into smaller fibers, reduces fiber diameter, and increases aspect ratio [18]. This increases the effective surface available for wetting by the matrix material. Alkali-sensitive hydrogen bonds existing among the fibers break down, and new reactive hydrogen bonds form between the cellulose molecular chains. Due to this, hydrophilic hydroxyl groups were partially removed, and moisture resistance property was improved. Then, OPEFB sample was washed with distilled water until it reached neutral pH to ensure NaOH residue was fully removed from the sample.

2.3.2 Bleaching process

OPEFB fiber was go through a bleaching process using a mixture of sodium chlorite ($NaClO_2$) and acetic acid (CH_3COOH) with ratio of 1.3:10 (v/v), stirred for 4 hours at 353K. Chlorite bleaching has the function of extracting holocellulose from raw cellulose fiber and helps to remove most of the lignin present in the fibers, which leads to defibrillation Next, the fiber was washed sufficiently with 0.1 M NaOH followed by distilled water until the pH of the fiber became neutral. Subsequent alkali treatment process was benefit to removes additional hemicellulose from the treated fiber [12] while washed well with distilled water to eliminate remaining chemicals [18]. Treated fiber was dried overnight at 323 K and the resulting product is known as Cell-OPEFB.

2.5. Characterization of Cell-OPEFB

2.5.1 Fourier Transform Infrared (FTIR) Spectroscopy

Determination of chemical structure of Cell-OPEFB were performed by using Perkin Elmer FTIR Spectrometer. Analysis performed at a resolution of $8cm^{-1}$ with accumulation of 5 scans in the mid IR region between 400-4000 cm^{-1}. Cell-OPEFB were mixed homogeneously with KBr powder. The ratio between sample and KBr is 1 mg sample/100 mg KBr approximately. The finely ground mixture then was being pressed under 8 tons pressure for 5-10 seconds to produce about 1 cm2 transparent disk. FTIR spectrum was generated in line with to the vibration to which the samples were exposed, and their % transmittance intensity were plotted against the wavelength of light (cm^{-1}).

2.5.2 X-Ray Diffraction (XRD)

The XRD (X-ray diffraction) (Rigaku SmartLab Diffractometer) was used to check the crystal structure of the samples. Analysis was using Cu-Kα radiation with λ=1.5418Å at 40 kV and 30 mA in the range

of $2\theta = 3 - 90^o$. The crystallinity index (I_c) was determined using Segal empirical method [3], [20] which can be expressed as equation $I_c\ (\%) = (I_{002} - I_{AM})/I_{AM}\ x\ 100$; where, I_{002} represents both crystalline and amorphous materials while I_{AM} represents the amorphous material.

2.5.3 Field Emission Scanning Microscopy (FESEM)

Field-Emission Scanning Microscopy (FESEM) model Hitachi SU8020 was used to observe the surface morphology features and elemental distribution of the samples. Sample was mounted on a double-sided carbon tape or an aluminium or copper stub. The sample is sputter coating with carbon vapor and finally with gold. The replica of the sample is taken on a copper grid and examined under electron microscope with suitable magnification. SEM micrographs reveal surface modification, fractured surfaces, cross-sectional area of the fibers, arrangement of the fibrils, adherence of minerals (silicon, potassium, calcium, magnesium, aluminium), porosity, fiber compactness, etc.

2.5.4 Energy dispersive X-ray (EDX)

Energy dispersive X-ray (EDX) Oxford Instrument model Xman-N was used to observe the elemental distribution of the sample. EDX data were gained using a micro-analytical unit that able to detect the minor different in identifying element content. An accelerating voltage of 20kV was apply with a scan time of 60s per sampling area were performed during the analysis. EDX analysis area is related directly to the FESEM morphological examination at 12.0k magnification and at least three areas were analysed for the given depth of each sample.

2.5.5 Thermogravimetric analysis (TGA)

TGA instrument (model TGA Q500 V20.13 Build 39) was used to determine the thermal stability of the samples. Around 4-6 mg sample was used during the measurement. All measurements were performed under a nitrogen atmosphere with a gas flow of 10 mL min^{-1} by heating the material from room temperature to 700∘C at a heating rate of 10∘C min^{-1}. During the pyrolysis process, the system will automatically collect data.

3.   Results and Discussion

3.1. FTIR Analysis

FTIR spectrum for Cell-OPEFB was shown in Figure 2. The spectrum represented the characteristic peaks of cellulose at approximately 3450 cm^{-1}, 2900 cm^{-1}, and 1060 cm^{-1} associated with O–H, C–H and C–O stretching, respectively. Elsewhere, peaks as around 1430 cm^{-1}, 1370 cm^{-1} and 1330 cm^{-1} also present, which are associated with the bending vibrations of –CH$_2$, C-H, and C-O respectively [6], [11], [12], [21]. The peaks observed at 895 cm^{-1} and 1060 cm^{-1} are attributed to the C-H rocking vibrations and the C-O stretching of the carbohydrates, respectively [12]. C-H vibration peak was observed at around 2900 cm^{-1} and [16], [17], [21], [22] and a relatively narrow band at around 3400 cm^{-1} which attributed to OH of cellulose [21].
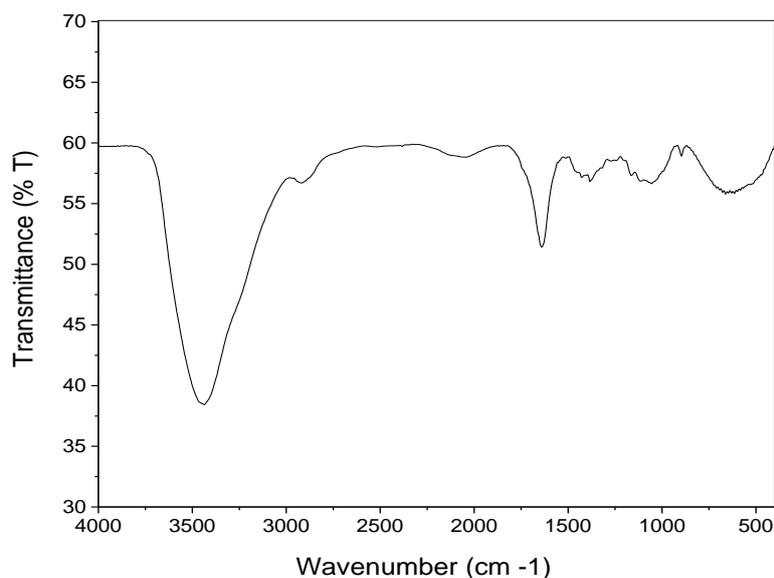

Figure 2. FTIR Spectrum of Cell-OPEFB.

3.2 XRD Analysis

Figure 3 illustrates the XRD pattern of cell-OPEFB. Two main peaks were observed from the XRD patterns, i.e., 2θ = 15 to 16° and 2θ = 21 to 22°, representing (110) and (200), respectively. The main peaks for the Cell-OPEFB occur around 16° and 22° 2θ, which represents the characteristic peaks of cellulose type I [12], [20], [23]–[25]. These intensity peaks at (110) and (200) correspond to the amorphous and crystalline peaks of cellulose, respectively. Similar result has been reported by previous researchers, Voronova et al. (2012), which been exhibit higher intensities at position (200) than at position (110), due to cellulose particles possess high surface energy and easier forming aggregates. The diffraction peak intensity of Cell-OPEFB was higher at 21-22°, which is may due to the removal of amorphous region in cellulose [26]. The increase of the peak intensity at 2θ = 22° signifies the transformation of the nature of the material from amorphous to crystalline [3]. From the analysis result, the crystallinity index shown by Cell-OPEFB was 75.51%.
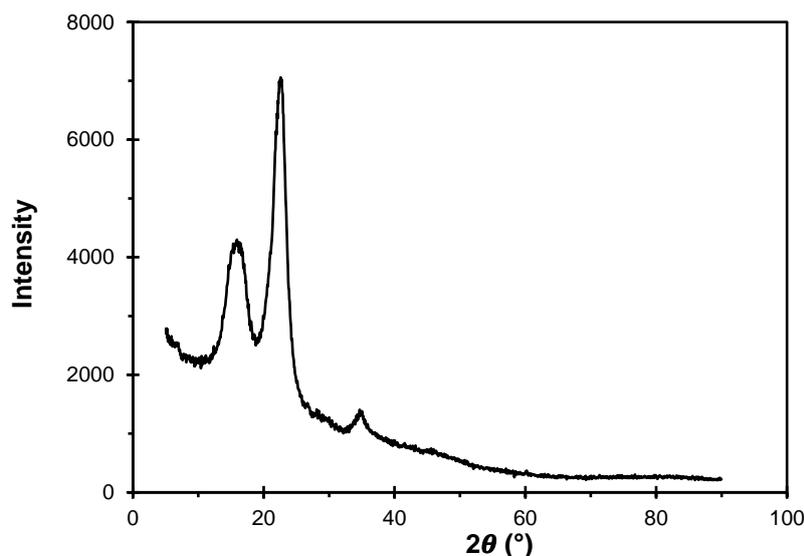

Figure 3. XRD diffractogram of Cell-OPEFB

3.3 FESEM and EDX Analyses

The surface morphologies of extracted cellulose with different magnification is presented in Figure 4. The surface of Cell-OPEFB appeared smooth [5], [15], [27]. The Cell-OPEFB sample demonstrate the fibril was isolated and separately from each other, signifying the removal of wax, hemicellulose and lignin was completed [28]. The outer surface shows a mixture of smooth surfaces along with scars, most likely due to the elimination of inorganic particles for instance silica and metal components. This was supported by EDX spectra (Figure 5) which showed an abundant of carbon (69.1%) and oxygen (30.3%) as the largest element in the sample [28].
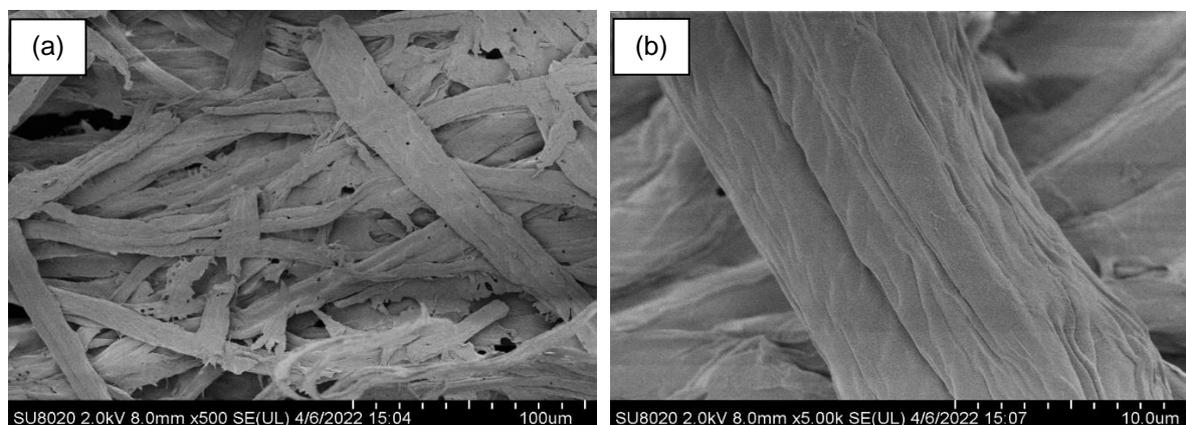

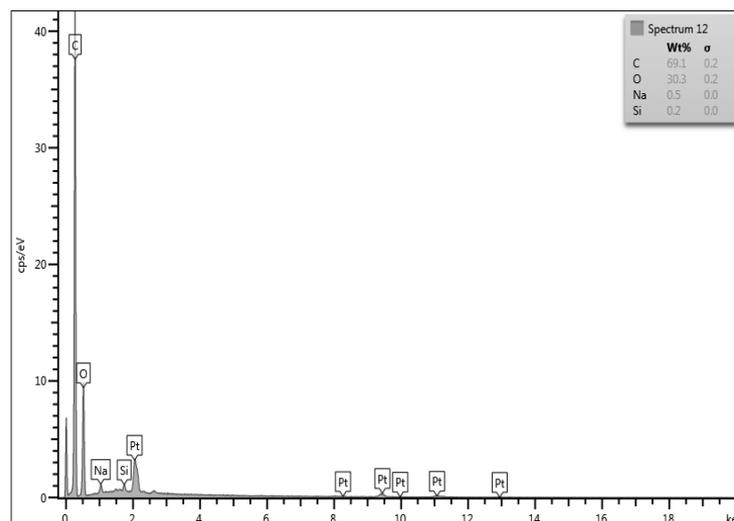**Figure 4.** FESEM images (a) and (b) of Cell-OPEFB at different magnification.

Figure 5. EDX result of Cell-OPEFB

3.4 TGA Analysis

Figure 6 shows the thermal degradation graph for Cell-OPEFB sample. Overall, three-step process were taking place during the process. The first step showing the water losses from the sample. From the figure, thermal decomposition for Cell-OPEFB was started at temperature 250 ºC before turning out to be stable at 350 ºC [28]. At this stage, the samples quality had undergone thermal decomposition which is most of the quality loosed during for this stage. The weight loss of recorded for Cell-OPEFB was 80 wt%. The observation found that there almost no water quality loss occurred before the sample began thermal decomposition showing that cellulose didn't absorb water easily [27]. Finally at the last phase, the decomposition of Cell-OPEFB will become slowly and the sample will start to carbonize and finally the sample was destroyed. [28]
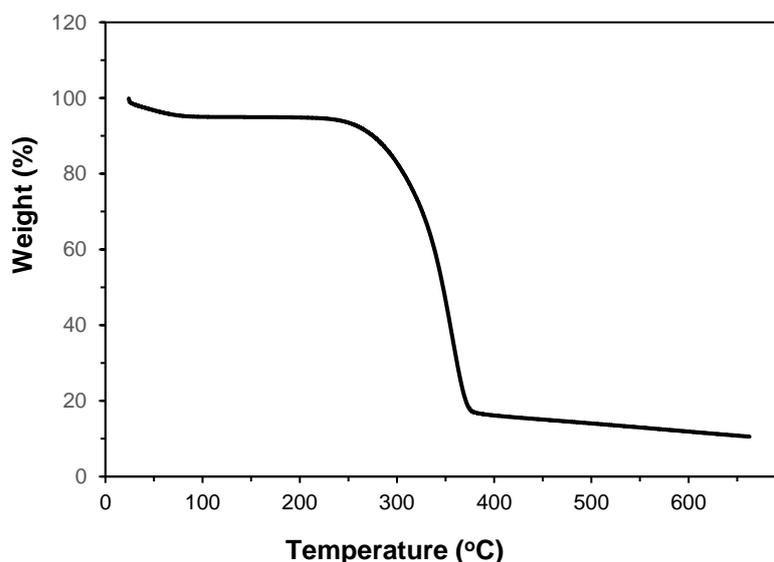

Figure 6. TGA curve of Cell-OPEFB

Conclusion

The study shows that nature cellulose (Cell-OPEFB) was successfully extracted from oil palm empty fruit bunch by using esterification method. High purity of cellulose was obtained through chemical pre-

treatment using alkaline treatment and bleaching with sodium chlorite to remove non-cellulosic substances in natural fibres. Cell-OPEFB shaped basic crystalline structure of cellulose after confirmed by FTIR and XRD analysis while the crystal structure of Cell-OPEFB belonged to type I cellulose was proven by the XRD analysis results. TGA analysis showing thermal stability for Cell-OPEFB achieved at 350°C whereas FESEM observations show that extracted cellulose morphology appears as a separate fibril with a smooth surface.

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