# Effect of Preparation Conditions on Cellulose from Oil Palm Empty Fruit Bunch Fiber

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The effects of chlorite bleaching on the properties of cellulose derived from oil palm empty fruit bunch (OPEFB) fiber were investigated in this work. Cellulose was extracted from oil palm empty fruit bunch fiber *via* chlorite bleaching, alkali treatment, and acid hydrolysis. Cellulose was extracted by varying the bleaching duration, which corresponded to 4, 8, or 12 h. Fourier transform infrared (FTIR) analysis showed that the lignin and hemicellulose were significant removed after the bleaching process, whereas no spectral differences were observed in the samples with the increase of bleaching durations. The main removal of the lignin and hemicellulose components had occurred during the bleaching process. There was only slight additional removal of lignin and hemicellulose during the further extraction process with alkali and acid hydrolysis. The peaks at 1740 $cm^{-1}$ and 1246 $cm^{-1}$ which represent hemicellulose and lignin, respectively, were not present in the final extracted cellulose.The cellulose yield contents did not increase with the increasing of bleaching duration from 4 h to 12 h. X-ray diffraction (XRD) analysis revealed that the crystallinity and the 200 peak of OPEFB had increased after the bleaching process. Analysis of the visible light transmittance of cellulose, after a bleaching duration of 12 h, demonstrated the highest transmittance due to the disintegration of the fibers. By increasing the bleaching duration, the temperature at 50% weight loss of OPEFB increased, suggesting that the thermal stability of cellulose had improved.

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## INTRODUCTION

Cellulose is the most abundant renewable organic material produced in the biosphere. Currently, the production of cellulose is estimated to be over 7.5 x $10^{10}$ tons per year (Habibi *et al.* 2010). Many factors play important roles in the properties of cellulose, *e.g.*, internal fiber structure, chemical composition, microfibril angle, cell dimensions, and defects, which vary from plant to plant (Siqueira *et al.* 2010). Cellulose, hemicellulose, and lignin form the major constituents of the natural fibers and may vary depending on the plant's age, growth conditions, weather effect, testing methods used, and soil conditions. Cellulose can be obtained from numerous resources, such as plants, animals, bacteria, and some amoebas (Rosa *et al.* 2010; Chen *et al.* 2012). Cellulose is initially found in an amorphous state, bonded tightly with crystalline domains through both the inter-molecular and intra-molecular hydrogen bonding in the cellulose plant fiber (Lee *et al.* 2009).

Various methods can be used to extract cellulose, such as grinding, cryocrushing, TEMPO-mediated oxidation, sulfuric acid hydrolysis, and enzyme-assisted hydrolysis (Habibi *et al.* 2010; Rosa *et al.* 2010; Siqueira *et al.* 2010; Chen *et al.* 2012). Chemical

treatment is the most effective way to obtain a higher purity cellulose. A combination of chlorite bleaching, alkali treatment, and acid hydrolysis is the most common process used to extract cellulose from the plants (Habibi *et al.* 2010; Liu *et al.* 2010; Rosa *et al.* 2010; Siqueira *et al.* 2010; Chen *et al.* 2012; Kargarzadeh *et al.* 2012; Sheltami *et al.* 2012; Zaini *et al.* 2013). Chlorite bleaching has the function of extracting holocellulose from raw cellulose fiber. This process helps to remove most of the lignin present in the fibers, which leads to defibrillation. Kargarzadeh *et al.* (2012) had reported that the diameter of the bleached kenaf bast fibers had become much smaller than that of the raw fibers. Alkali treatment is a process used to solubilize the lignin and the remaining pectins and hemicelluloses. Acid hydrolysis is used to degrade amorphous cellulose. Many researchers have obtained cellulose from different fibers and plants, such as wood (Araki *et al.* 1998; Beck-Candanedo *et al.* 2005), banana (Cherian *et al.* 2008), yarns (Liu *et al.* 2010), coconut husk fibers (Rosa *et al.* 2010), potato peel waste (Chen *et al.* 2012), mengkuang leaves (Sheltami *et al.* 2012), bamboo (He *et al.* 2013), and kenaf fiber (Kargarzadeh *et al.* 2012; Zaini *et al.* 2013).

The extraction and characterization of nanocellulose from oil palm empty fruit bunch (OPEFB) has previously been reported (Fahma *et al.* 2010; Jonobi *et al.* 2011; Nazir *et al.* 2013; Mohamad *et al.* 2013). Generally, the extracted OPEFB fiber contains about 40 to 65% cellulose, 15 to 25% hemicellulose, and 12 to 20% lignin, with a moisture content of about 10 to 15%, and it has the highest composition of cellulose compared to coir, corn, bagasse, and kenaf fiber (Sun *et al.* 1999; Fahma *et al.* 2010, 2011; Nazir *et al.* 2013). The extracted cellulose yield is affected by the preparation condition such as the type of chemical, concentration used, duration and temperature of hydrolysis treatment (Chen *et al.* 2009; Li *et al.* 2009). Rosa *et al.* (2010) reported the effects of the bleaching period on the properties of cellulose derived from coconut husk fibers. The same authors also reported that the residual lignin content can be controlled by the bleaching stage. Table 2 shows the chemical composition obtained from various plants by using different chemical treatments. Most of the researchers had extracted cellulose from plants by using alkali treatment and followed by chlorite bleaching (Cherian *et al.* 2008; Fahma *et al.* 2011; Kargarzadeh *et al.* 2012; Sheltami *et al.* 2012; Rosli *et al.* 2013; Zaini *et al.* 2013). The researchers reported that alkali treatment was only able to remove hemicellulose and lignin partially. Higher content of α-cellulose can only be obtained by further chemical treatment with bleaching process.

**Table 1.** Chemical Composition of Untreated and Treated Cellulose Fibers from Various Sources

| Fiber source | Condition of fiber | α-cellulose (%) | Hemicellulose (%) | Lignin (%) | References |
|---|---|---|---|---|---|
| Banana | Untreated | 64.0 | 18.6 | 4.9 | Cherian *et al.* 2008 |
| | Alkaline treated | 82.4 | 14.0 | 3.6 | |
| | Bleached | 95.9 | 0.4 | 1.9 | |
| Mengkuang leaves | Untreated | 37.3 | 34.3 | 24.0 | Sheltami *et al.* 2012 |
| | Alkaline treated | 57.5 | 15.5 | 22.6 | |
| | Bleached | 81.6 | 15.9 | 12.5 | |
| Agave angustifolia | Untreated | 67.0 | 25.2 | 6.3 | Rosli *et al.* 2013 |
| | Alkaline treated | 88.0 | 3.9 | 6.0 | |

However, there is still a lack of reports and thorough investigation on the effect of bleaching duration on the properties of the treated OPEFB fiber and final derived cellulose. In this study, the cellulose was extracted from OPEFB fiber through chlorite bleaching, alkali treatment, and acid hydrolysis treatment. The chlorite bleaching was conducted for various durations (4h, 8h, and 12h). The effect of various treatment durations on the visible light transmittance and thermal properties of the treated fiber and derived cellulose from OPEFB fibers are studied and compared.

## EXPERIMENTAL

### Materials

The OPEFB fibers were purchased from Sabutek Sdn. Bhd., Malaysia. Poly(vinyl alcohol) (Kuraray-PVA-220S, > 87% partially hydrolyzed) was supplied by Kuraray Co., Ltd., Singapore. Sulfuric acid ($H_2SO_4$) (95-97%), acetic acid glacial and sodium hydroxide (NaOH) were purchased from Friendemann Schmidt Chemicals (Malaysia). Sodium chlorite ($NaClO_2$) (80%) was purchased from Fisher Scientific (M) Sdn. Bhd. All chemicals were of reagent grade and used without purification.

### Methods

*Extraction of cellulose*

The OPEFB (5 g) was treated with a mixture of 1.5 g of sodium chlorite ($NaClO_2$) and 150 mL of distilled water. Then, 8 to 10 drops of glacial acetic acid were added at 70 °C. The OPEFB was bleached for various durations, *i.e.*, 4, 8, or 12 h. Bleached fibers were filtered with cold distilled water four times, and the samples were labelled EFB-4B, EFB-8B, and EFB-12B, respectively.

Bleached fiber was mixed with 6 wt% sodium hydroxide (NaOH) at room temperature for 24 h, and further heated at 80 °C for 2 h to remove the remaining hemicellulose and lignin that was present. The resultant cellulose was washed with distilled water until a pH of 7 was reached, and labelled EFB-4BT, EFB-8BT, and EFB-12BT. The composition of the raw fiber (EFB) and treated fibers (EFB-4B, EFB-8B, EFB-12B, EFB-4BT, EFB-8BT, and EFB-12BT) were estimated according to the ASTM standards. Holocellulose content was determined based on ASTM D1104-56. 4.0 g of the sample was treated with 2.0 mL of acetic acid and 5.0 g of sodium chlorite at 70 °C for 4 h. After that, the suspension was filtered and dried until constant weight was achieved. The remaining sample was then further used for determining the α-cellulose content according to ASTM D1103-60. In this step, 2.0 g of holocellulose was treated with 50 mL of 17.5% sodium hydroxide solution. Then, the suspension was again filtered and dried until the constant weight was achieved. The lignin content was measured according to ASTM D1106-96. About 2.0 g of sample was mixed with 15 mL of 12% $H_2SO_4$ at a temperature of 20 °C for 2 h. The suspension was diluted to 3% concentration of $H_2SO_4$ before boiled for 4 h. Lastly, the mixture was filtered and dried to constant weight.

*Sulfuric acid hydrolysis*

The resultant cellulose (*i.e.,* EFB-4BT, EFB-8BT, and EFB-12BT) was added to a 64 wt% of sulfuric acid ($H_2SO_4$) solution for 2 h at a temperature of 45 °C. The suspension was washed with distilled water and centrifuged at 6000 rpm for 10 min, until it reached a pH of 7 (Rosa *et al.* 2010; Chen *et al.* 2012). The final concentration of the suspension was

diluted with distilled water to 1 wt%, and ultrasonically treated for 5 min. The suspensions were then dried at 40 °C for approximately 24 h until a constant weight is achieved. The cellulose after the hydrolysis process was labelled EFB-4BTA, EFB-8BTA, and EFB-12BTA.

*Characterization*

Chemical bonding of OPEFB was performed using a FTIR Spectrum 400 (Perkin Elmer, USA). The FTIR scan range was analyzed between 2000 and 500 cm$^{-1}$, with a scan rate of 32 unit scans. The crystallinity of the OPEFB was characterized by a X-ray diffractometer (XRD-D5000, Siemens; Germany) using CuKα radiation, with a wavelength at 1.5460 Å. The intensity data were collected over a range of $2\theta$ range from 10° to 50°, with a scan rate of 0.05°/s. Optical transmission properties of cellulose and EFB suspension were carried with a UV-visible spectrometer (Varian Cary 50, USA) in the wavelength range of 400 to 700 nm. Thermogravimetric weight loss of the OPEFB fibers and its derived celluloses was studied using a Mettler Toledo TGA/SDTA851 thermogravimeter (Mettler Toledo Coro., Switzerland). For the thermal decomposition behavior, the samples were heated from a temperature range of 30 to 600 °C, with a scan rate of 20 °C/min. A sample of 5 to 10 mg was used for each run, and changes in weight were recorded.

## RESULTS AND DISCUSSION

### Extraction of OPEFB cellulose

The chemical composition of treated and untreated OPEFB fiber at different stages of treatment is shown in Table 1. The raw OPEFB (EFB) consisted of 36.7% of α-cellulose, 35.8% of hemicellulose, and 18.6% of lignin. After 4 h of chlorite bleaching (EFB-4B), the α-cellulose content had been increased around 80%, whereas hemicellulose and lignin were reduced to 14% and 3.5%, respectively. However, the further increase of chlorite treatment period only slightly reduced the content of hemicellulose and lignin from the treated fiber. It was found that there were no significant changes in the content of α-cellulose, hemicellulose, and lignin when the chlorite treatment duration was increased from 4 h to 12h (Table 2).

**Table 2.** Chemical Composition of Treated and Untreated OPEFB Fiber

| Sample | α-cellulose (%) | Hemicellulose (%) | Lignin (%) |
|---|---|---|---|
| EFB | 36.7 | 35.8 | 18.6 |
| EFB-4B | 80.2 | 14.3 | 3.5 |
| EFB-8B | 81.5 | 13.9 | 3.0 |
| EFB-12B | 82.4 | 13.2 | 2.6 |
| EFB-4BT | 81.9 | 13.5 | 3.1 |
| EFB-8BT | 82.5 | 12.8 | 2.8 |
| EFB-12BT | 83.4 | 11.9 | 2.6 |

Table 2 also illustrates that there was no significant changes in the content of α-cellulose, hemicellulose, and lignin for the treated fiber after alkali treatment (EFB-4BT, EFB-8BT, and EFB12BT). This result indicates that the bleaching process plays an important role to remove both hemicellulose and lignin simultaneously. Based on the chemical composition analysis, it clearly shows that the chlorite bleaching removed a

majority of the hemicellulose and lignin from OPEFB (Alemdar and Sain 2008). Subsequent alkali treatment process only slightly removes additional hemicellulose from the treated fiber (Rosli *et al.* 2013).

The final stage with 2 h acid hydrolysis slightly reduced the amount of lignin and the hemicellulose fraction. The cellulose yield produced from this treatment was 58.5% (w/w) on a dry weight basis. The cellulose content was different from that reported by other researchers, such as 44.4% (Sun *et al.* 1999), 60.6% (Wanrosli *et al.* 2004), 49% (Nazir *et al.* 2012), and 64% (Nazir *et al.* 2013). This differential on the cellulose contents might be due to the different origins and hydrolysis conditions (De Menezes *et al.* 2009; Li *et al.* 2009).

## FTIR Spectra

Figure 1a shows the FTIR spectra of OPEFB at different stages of chemical treatment and its derived celluloses after multiple bleaching durations. The peaks around 1370 to 1430 $cm^{-1}$ and 1330 $cm^{-1}$ in the spectra represented the units of -$CH_2$ and C-H of cellulose, respectively (Cherian *et al.* 2008; Fahma *et al.* 2010; Kargarzadeh *et al.* 2012; Nazir *et al.* 2013; Zaini *et al.* 2013). Both the peaks at around 1640 $cm^{-1}$ and 1210 $cm^{-1}$ correspond to the bending vibrations of the OH groups of cellulose (Alemdar and Sain 2008; Kargarzadeh *et al.* 2012). The peak at 1165 $cm^{-1}$ arises due to the asymmetrical deformation of the C-O-C band (Kargarzadeh *et al.* 2012; Zaini *et al.* 2013). The peaks observed in all spectra 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 (Alemdar and Sain 2008; Kargarzadeh *et al.* 2012; Nazir *et al.* 2013).

The peak at 1740 $cm^{-1}$ can be seen in the EFB FTIR spectra, which occurs due to the C=O stretching of the acetyl and uronic ester groups of the hemicellulose ester. The peak located at 1740 $cm^{-1}$ also represents the carbonyl ester linkage of the carboxylic groups of ferulic and p-coumaric monomeric lignin (Cherian *et al.* 2008; He *et al.* 2013; Nazir *et al.* 2013). The peaks observed at 1246 $cm^{-1}$ and 1514 $cm^{-1}$ are attributed to a C-O-C bond of the aryl-alkyl ether and the C=C stretching from the aromatic ring of lignin, respectively. These two peaks were completely absent from the spectra of the bleached fibers (EFB-8B), alkali-treated fibers (EFB-8BT), and OPEFB cellulose (EFB-8BTA). The disappearance of these peaks (1740 $cm^{-1}$, 1246 $cm^{-1}$, and 1514 $cm^{-1}$) indicates that most of the hemicellulose and lignin were dissolved during the chlorite bleaching stage. No significant changes were observed in the spectra that were attributable to the alkali treatment of sample EFB-8BT and the further process with acid hydrolysis (EFB-8BTA). Previous studies had reported the same observation that the removal of lignin and hemicellulose mainly occur during the bleaching process (Alemdar and Sain 2008; Sheltami *et al.* 2012).

Many previous reports have considered the effects of an alkali treatment prior to a bleaching process (Fahma *et al.* 2011; Kargarzadeh *et al.* 2012; Sheltami *et al.* 2012; Santos *et al.* 2013; Zaini *et al.* 2013). According to these sources, the three peaks at 1740 $cm^{-1}$, 1246 $cm^{-1}$, and 1514 $cm^{-1}$ were decreased after alkali treatment, and then further vanished after bleaching process. In the present study, the bleaching process was carried out before alkali treatment. Results showed that the bleaching process had successfully removed all the major hemicellulose and lignin composition from the raw fiber. Thus, the further alkali treatment in this study did not play an important part in the further extraction process, as shown in Figure 1a.

A comparison of the FTIR spectra between derived cellulose from various bleaching durations is illustrated in Figure 1b. No behavioral difference was found between these three spectra, indicating that there was no changes of chemical composition in the bleached sample after various bleaching times from 4 h to 12 h. Variations in the bleaching duration did not show any effectiveness in the removal of lignin or hemicellulose. The same finding also had been reported by Rosa *et al.* (2010).
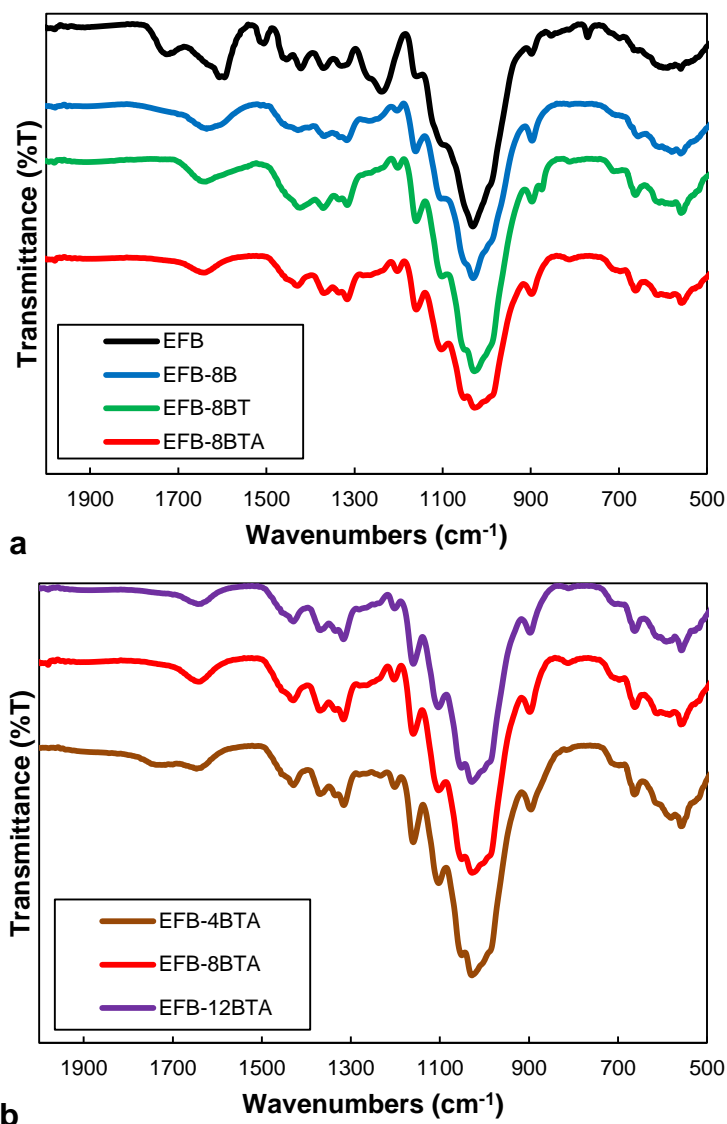


**Fig. 1.** FTIR spectra of (a) untreated OPEFB, treated OPEFB, and its derived extracted cellulose and (b) cellulose derived from OPEFB with different bleaching durations (4 h, 8 h, and 12 h)

## X-Ray Diffraction

The XRD spectra of OPEFB and its derived celluloses are shown in Fig. 2. Two main peaks were observed from the XRD patterns, *i.e.*, $2\theta = 13$ to $14°$ and $2\theta = 21$ to $22°$, representing (110) and (200), respectively. These intensity peaks at (110) and (200) correspond to the amorphous and crystalline peaks of cellulose, respectively (Nazir *et al.* 2013). The appearance of (110) and (200) revealed the existence of the cellulose I type (Fahma *et al.* 2010).
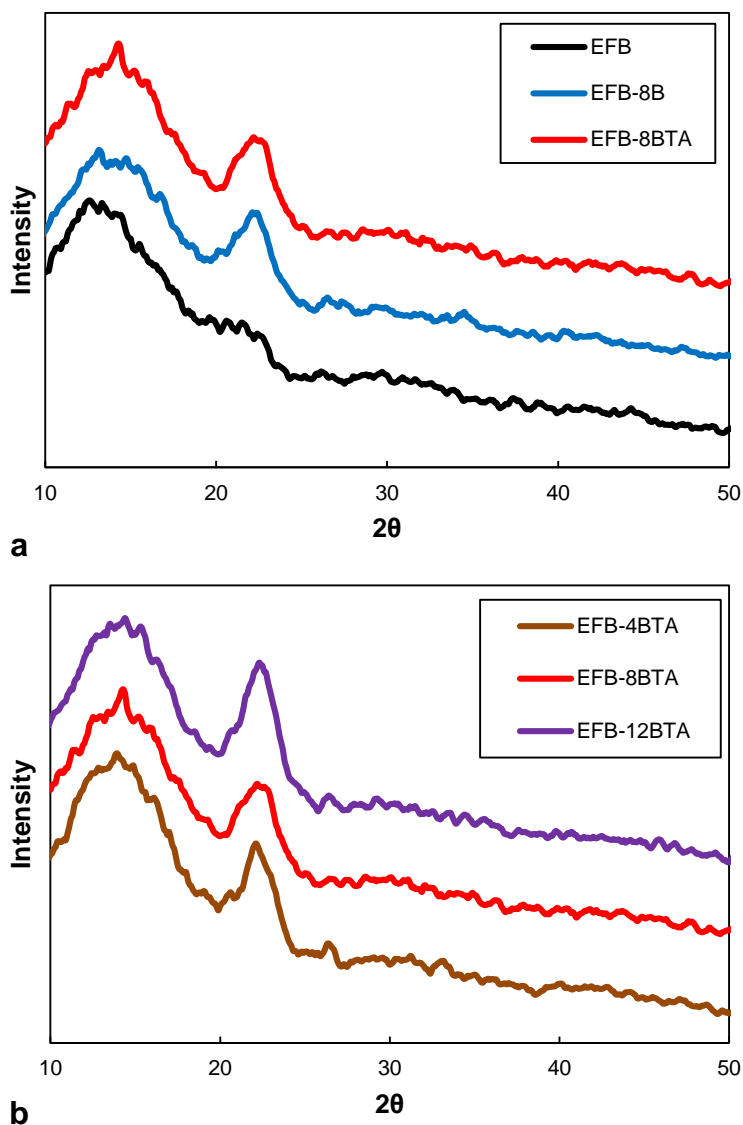
**Fig. 2.** The XRD spectra of (a) untreated OPEFB, treated OPEFB, and its derived extracted cellulose and (b) cellulose derived from OPEFB with different bleaching durations (4 h, 8 h, and 12 h)

Peak (110) at $2\theta$ = 13 to 14° with a high intensity is observed in Fig. 2. This phenomenon occurred due to the richness of the amorphous region of the OPEFB. The same result has been reported by previous researchers that the peak (110) is much higher than peak (200) (Voronova *et al.* 2012; Nazir *et al.* 2013). This shows that the raw OPEFB used in this study was highly amorphous. This result differs with the XRD results of other plants that were reported by previous researchers (Araki *et al.* 1998; Alemdar and Sain 2008; Li *et al.* 2009; Fahma *et al.* 2010; Rosa *et al.* 2010; Kargarzadeh *et al.* 2012; He *et al.* 2013; Santos *et al.* 2013; Zaini *et al.* 2013). The majority of the natural plants exhibit higher intensities at position (200) than at position (110). Voronova *et al.* (2012) reported cellulose particles possess high surface energy and easier forming aggregates. Therefore, cellulose did not appear as a powder, but rather an aerogel after drying (Voronova *et al.*

2012). However, the XRD pattern with a higher intensity at (110) shows that the cellulose was mainly in the amorphous phase. This amorphous-rich cellulose is suitable in the application of cellophane of electroactive polymers (Deshpande *et al.* 2005; Kim *et al.* 2005).

Figure 2a shows a peak at around 22°, which increases after the bleaching process (EFB-8B) compared to the raw OPEFB. This is due to the elimination of lignin and hemicellulose, with amorphous segments. Thus, crystalline segments of cellulose I were readily detected by XRD. The peak at (200) for sample after acid hydrolysis process (EFB-8BTA) was lower than the one before hydrolysis treatment (EFB-8B). The decrease of peak at (200) after acid hydrolysis has been explained as due to the fact that the acid hydrolysis process not only removed the amorphous region of the cellulose, but also tended to damage the crystalline portion of the treated sample (Rosa *et al.* 2010). The crystallinity of cellulose after sulfuric acid hydrolysis process was estimated at 58% which is comparable to the reported value of 59% (Fahma *et al*. 2010).

Figure 2b shows EFB-12BTA having the highest intensity at $2\theta = 22°$. Similar results were obtained by Rosa *et al.* (2010). As the bleaching duration increased, a higher fraction of the lignin being removed was obtained and observed in a sharper and narrower crystalline peak in XRD spectra (Rosa *et al.* 2010). In this study, increasing of bleaching durations from 4 h to 12 h slightly increased the crystallinity of the derived cellulose. Changing the duration of bleaching duration from 4 h (EFB-4BTA) to 12 h (EFB-12BTA) has resulted in very little difference in crystalline index, 57.8% and 59.2%, respectively.

## Optical Transparency Properties

The visible light transmission of EFB and its derived celluloses (EFB-4BTA, EFB-8BTA, and EFB-12BTA) are shown in Fig. 3. The optical transmission of derived celluloses increased when the bleaching duration increased from 4 h to 12 h. The EFB-12BTA clearly shows a higher optical transmission than EFB-4BTA and EFB-8BTA. The EFB-12BTA explores transmittance that increased by around 20%, compared to that of raw OPEFB (EFB) in the range of 400 to 700 nm (visible region).
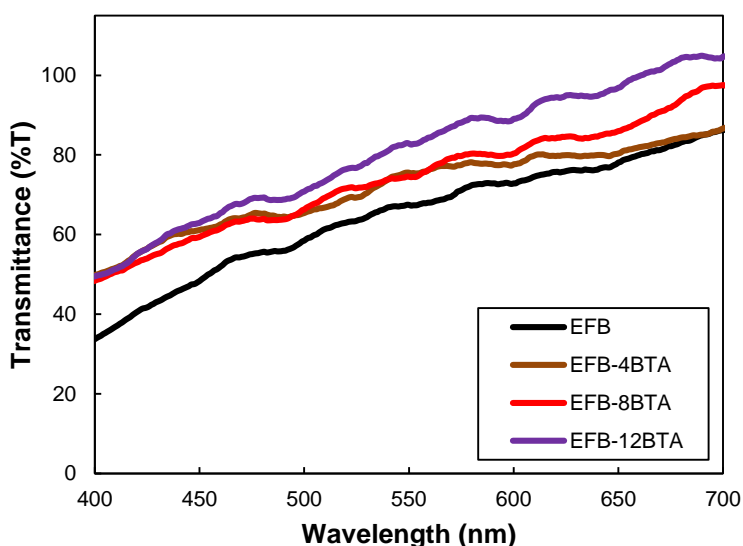


**Fig. 3.** The visible light transmittance spectra of raw OPEFB and cellulose derived from OPEFB fibers after various bleaching durations

The transmittance result generated by UV-vis spectroscopy is wavelength-dependent. When the wavelength reaches the diameter of the particles, more light is scattered (Saito *et al.* 2006). For example, transmittance of EFB-12BTA reached 100% at a wavelength of 700 nm indicating that the average size of EFB-12BTA is shorter than 700 nm. The suspension showed better light transmission as the bleaching process progressed. Lignin and hemicellulose disintegrated further as the bleaching period increased (Rosa *et al.* 2010). This indicated that the longer the bleaching duration, the smaller the particle size produced from the celluloses. The lowest visible light transmittance of EFB shows it had a larger particle size compared to its derived celluloses.

## Thermal Gravimetric Analysis (TGA)

Graphs of thermogravimetric weight loss of the OPEFB fiber before and after treatments are given in Fig. 4.
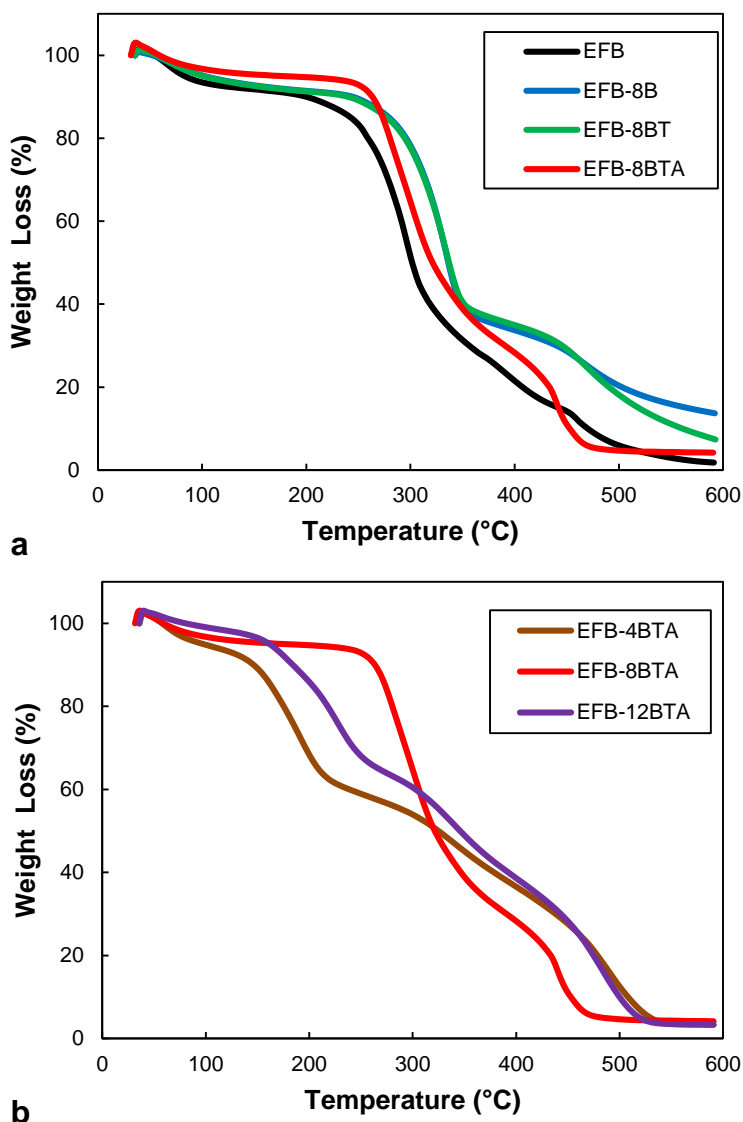


**Fig. 4.** Thermogravimetric curves of (a) untreated OPEFB, treated OPEFB, and its derived extracted cellulose and (b) cellulose derived from OPEFB with different bleaching durations (4 h, 8 h, and 12 h).=

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Weight loss changed slightly at lower temperatures (below 100 °C) due to the moisture evaporation or degradation of the low molecular weight compounds (Sheltami *et al.* 2012; Mohamad *et al.* 2013). Holocellulose destruction was observed above the temperature of 150 °C (Voronova *et al.* 2012).

Figure 4a shows the thermogravimetric curves of untreated OPEFB, treated OPEFB, and its extracted cellulose. The EFB had a greater weight loss from 30 to 450 °C compared to the chemical treated OPEFB fiber and its extracted cellulose. The OPEFB fiber after chlorite bleaching process (EFB-8B) and alkali treatment (EFB-8BT) showed a similar trend of thermal stability. Both of the treated fiber samples showed higher weight loss than the derived cellulose (EFB-8BTA). Jonoobi *et al.* (2011) has reported that the lignocellulosic materials start to degrade thermally with hemicelluloses, followed by pyrolysis of lignin, depolymerization of cellulose, and ending with char oxidation. Thus, EFB raw fiber with high amount of hemicellulose and lignin showed lower thermal stability (or higher weight loss) compared to EFB-8B, EFB-8BT, and EFB-8BTA samples that had been treated with chlorite bleaching, alkali treatment, and acid hydrolysis, respectively.

The decomposition temperature for the hemicelluloses and lignin components in OPEFB fiber and chemical-treated fibers have been reported as being below 270 °C (Sheltami *et al.* 2012). Thus, the hemicellulose and lignin are degraded at a lower temperature than that of cellulose (Jonoobi *et al.* 2011; Sheltami *et al.* 2012). The EFB-8BTA was the stage where the OPEFB was in the condition with a higher purity of cellulose due to the decomposition of the amorphous region (hemicelluloses and lignin region). Thus, the OPEFB cellulose (EFB-8BTA) experienced the smallest weight loss at temperatures below 270 °C. However, EFB-8BTA shows a higher thermal degradation after 270 ºC compared to both EFB-8B and EFB-8BT. This might be due to the high duration of acid hydrolysis, which had led to deterioration of thermal stability of the cellulose.

Negatively charged sulfate groups are introduced into the surface of the cellulose in the course of a hydrolysis process involving sulfuric acid (Fahma *et al.* 2010). The fact that such hydrolysis affected the thermal behavior indicates that a high period of acid hydrolysis not only dissolved the amorphous region of cellulose, but also partially removed the crystalline regions of the cellulose (Rosa *et al.* 2010). Thus, the highly sulfated amorphous region brought about by sulfuric acid during hydrolysis treatment had caused the reduction of thermal stability in the EFB-8BTA sample (Kargarzadeh *et al.* 2012). Figure 4a also illustrates that the OPEFB after bleaching (EFB-8B) and alkali treatment (EFB-8BT) had a significant residual mass at approximately 600 °C. A probable cause of the remaining mass could be the presence of char residue of the crystalline cellulose type I, which is more flame resistant (Mohamad *et al.* 2013).

Figure 4b shows the comparison of thermal stability between celluloses derived from OPEFB fiber with different bleaching durations. EFB-12BTA had a smaller weight loss compared to EFB-4BTA and EFB-8BTA at low temperature range from 50 to 150 °C. This indicates that EFB-12BTA is more thermally stable at lower temperatures. However, EFB-12BTA did show rapid degradation at 150 ºC which is less thermal stable than EFB-8BTA sample, which only showed rapid themal degradation at 270 °C. This phenomenon is similar to the results obtained by Rosa *et al.* (2010), who reported that longer bleaching time improved the thermal stability of the derived cellulose at low temperature.

## CONCLUSIONS

1. Based on the experiment data, about 58.5% of the cellulose was successfully extracted from OPEFB fiber after chemical treatment through chlorite bleaching, alkali treatment, and acid hydrolysis. The increase of chlorite bleaching duration from 4 h to 12 h do not help in the further removal of the amorphous region of lignin and hemicellulose from the OPEFB fibers.

2. Analysis by FTIR showed that the lignin and hemicellulose of OPEFB had mainly been removed after 4 h of the chlorite bleaching process. XRD analysis indicated the crystalline nature of the extracted cellulose through the (200) peak.

3. The light transmission of OPEFB cellulose was 20% higher than the raw kenaf fiber. The light transmission of the bleached kenaf fiber increases with the increase of bleaching duration.

4. The chemical treatment with bleaching, alkali, and hydrolysis successfully improved the low temperature thermal stability properties of OPEFB cellulose due to the removal of the lignin component, which is thermally less stable.

## ACKNOWLEDGMENTS

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