Basic Properties of Oven–Heat Treated Oil Palm Empty Fruit Bunch Stalk Fibers

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The fibers of oil palm empty fruit bunch (OPEFB) stalks were investigated to determine the changes in their basic properties after oven-heat treatment. The oven-heat treatment was conducted at 100 °C or 190 °C for 15 min. There were slightly noticeable morphological, chemical, and thermal alterations in oven-heat treated OPEFB stalk fibers for short duration. Scanning electron microscopy (SEM) revealed that the treated fibers had smooth surfaces and irregular heavy deposition of cementing agents. The highest cellulose content (41.68%) was present in OPEFB stalk fibers treated at 100 °C for 15 min, whereas the highest crystallinity index (48.74%) occurred in fibers treated at 190 °C for 15 min. The fibers comprised cellulose, lignin, and hemicellulose with a high percentage of C, O, K, and other elements. Based on differential scanning calorimetry (DSC) analysis, oven-heat treated and untreated OPEFB stalk fibers had similar thermal stability characteristics.

Keywords: Basic properties; OPEFB stalk; Fibers; Oven-heat treatment; Lignocellulose

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INTRODUCTION

In Indonesia, oil palm empty fruit bunch (OPEFB) is a renewable lignocellulose product with an annual yield of 1.7 million tons (Gurning *et al.* 2013). Malaysia annually produces 6.93 million dry tons (Nazir *et al.* 2013). OPEFB is tremendously abundant and renewable, and its biodegradable fibers can be converted into value-added products, such as composite board, nanofiller, bioetanol, organic fertilizer, pulp, and paper.

OPEFB residue contains lignocellulose material, composed of holocellulose, lignin, extractives, and inorganic materials. OPEFB has a similar chemical composition to wood materials, which contain holocellulose (67%), α-cellulose (48%), and lignin (24%) (Ferrer *et al.* 2011). OPEFB consists of three primary components: cellulose (20 to 40 wt%), hemicellulose (10 to 35 wt%), and lignin (23 to 52 wt%) (Bahrin *et al.* 2012a). Generally, the cellulose and hemicellulose of OPEFB fibers are reinforced in a lignin matrix similar to that of other natural fibers (Hassan *et al.* 2010). Fibers are the most abundant component of oil palm empty fresh fruit bunches, representing as much as 23% of OPEFB (Razali *et al.* 2012). The two major structures in OPEFB are stalks (20% to 25%) and spikelets (75% to 80%); approximately 54% of the fiber in these tissues can be extracted (Khalib *et al*. 2010). Because OPEFB stalk fibers are very abundant, it would be efficient to utilize them as reinforcing agents for composites.

Besides the aforementioned regard, heat treatment is the best way to modify the properties of wood, including dimensional stability, resistance to bio-corrosion (Gunduz

et al. 2008), strength and brittleness (Korkut *et al.* 2008), and chemical composition (Tuong and Li 2011). Heat treatment also affects the crystallinity index (CrI) of wood cellulose (Karinkanta *et al.* 2013). The treatment is usually applied in elevated temperatures, ranging from 160 to 280 °C, with a duration of 15 min to 24 h depending on the process, wood species, sample size, and moisture content of the sample, as well as the desired mechanical properties, microbial resistance, and dimensional stability of the final product (Korkut *et al.* 2008).

Heat treatments with various temperatures have been applied to OPEFB in a superheated steam dryer (Hasibuan and Daud 2009), high-pressure autoclave (Baharuddin *et al.* 2012), and superheated steam equipment (Bahrin *et al.* 2012b). To reduce OPEFB moisture content to less than 10 mf wt%, a conventional oven has been used for drying pre-treatments (Khalib *et al.* 2010). By using several heat treatments, especially conventional oven drying on OPEFB stalk fibers, it is a great challenge to find out the most suitable treatment to alter the fiber properties for certain uses. However, the basic property changes of OPEFB stalk fibers treated with a conventional oven have not yet been studied, especially for a short duration of oven drying. The basic properties studied in this research included physical, morphological, chemical, and thermal properties. Therefore, the main objective of this study was to investigate the effects of oven-heat treatment on the basic properties of OPEFB stalk fibers.

EXPERIMENTAL

Materials

Oil palm empty fruit bunch (OPEFB) stalk fibers were obtained from PT Perkebunan Kelapa Sawit Nusantara VIII Cikasunga, Bogor, West Java, Indonesia. Prior to use, the fibers were shredded to approximately 3.836 mm in length and 0.348 mm in width in the form of vascular bundles, and air-dried to prohibit fungal growth. The targeted moisture content obtained by air drying before oven-heat treatment was approximately 12%.

Treatment of OPEFB Stalk Fibers

Oil palm empty fruit bunch stalk fibers were treated with a conventional oven (Memmert, Germany). Fibers (30 g) were treated with two different temperatures, 100 °C or 190 °C, for 15 min. An untreated control sample control was prepared. After oven-heat treatment, fibers were wrapped in plastic to prevent water absorption.

Density and Vascular Bundle Dimension Measurement

Density measurements of solid or bulk samples of OPEFB stalk were carried out using Archimedes' method. Each OPEFB stalk immersed in a liquid was bouyed up by a force equal to the weight of the fluid which was displaced. The average OPEFB stalk bundle dimension was calculated from images captured by an electron microscope with the software Motic Image Plus 2.0 integrated with National DC 2-456 Digital Microscope (Motic China Group Co., Ltd., China).

Scanning Electron Microscopy (SEM)

Morphological studies of OPEFB stalk fibers were conducted with a scanning electron microscope (model JSM-6510LA, JEOL, Japan). The fibers were mounted on an

aluminum stub using double-sided adhesive tape with an applied potential of 20 kV. Each treated and untreated sample was measured three times with 400x magnification after pulverized with disc milling. Prior to 500x magnification, samples were disc-milled and filtered with a 200-mesh sieve analyzer (75 µm).

Chemical Composition Analysis (CHLE)

The OPEFB stalk chemical properties were determined with TAPPI standards, which were TAPPI T222 om-88 (1988) modified with Dence method for lignin, TAPPI T203 OS-61 (1961) for α-cellulose, TAPPI T9m-54 (1988) for holocellulose, and TAPPI T204 om-88 (1988) for ethanol-benzene extractives. Chemical analysis of mixed fibers (spikelet and stalk with a 1:1 ratio) was used for comparison.

X-Ray Diffraction (XRD)

The CrI of untreated and treated OPEFB stalk fibers was determined using an X-ray diffractometer (model EMMA 013B, GBC, Australia) with $Cu_{K\alpha 1}$ from 2θ = 10° to 80° with a step size of 0.02°. The crystallinity index was calculated using the following formula (Segal *et al.* 1959),

$$CrI = [(I_{002}\text{-}I_{am}/I_{002})] \times 100 \quad (1)$$

where I_{002} is the peak intensity corresponding to cellulose I and I_{am} is the peak intensity of the amorphous fraction.

Fourier Transform Infrared (FTIR) Analysis

Chemical changes in functional groups of the treated and untreated OPEFB stalk fibers were analyzed with a FTIR spectrometer (model MB3000, ABB, Canada). The analysis was conducted within a transmittance wavenumber mode, ranging from 4000 to 450 cm^{-1}. The ratio between KBr and the fibers was 1:1.

Energy Dispersive X-Ray Spectroscopy (EDS)

The elemental composition of untreated and treated OPEFB stalk fiber was characterized with an energy dispersive X-ray spectroscopy analysis station (XRD model JED-2300 in conjunction with SEM model JSM-6510LA, JEOL, Japan). The X-rays were detected by a Be drift detector with the following acquisition parameters: 20.0 kV accumulation voltage and 0 to 20 KeV energy range.

Differential Scanning Calorimetry (DSC)

Thermal analysis was characterized with a differential scanning calorimeter (model JADE, Perkin Elmer, USA). Dried OPEFB stalk fibers (approximately 6.4 mg) were analyzed in a temperature range from 30 to 450 °C under a nitrogen environment with a scanning rate of 10.00 °C/min.

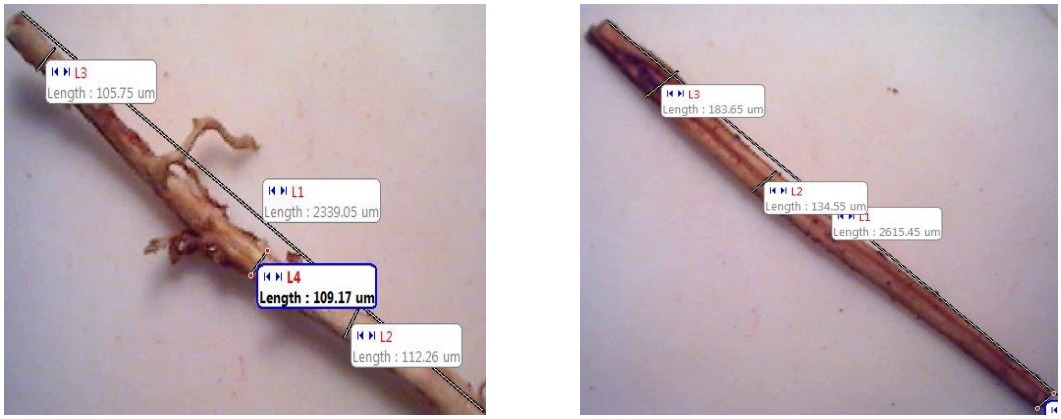
RESULTS AND DISCUSSION

Density and Vascular Bundle Dimension Measurement

The density of OPEFB stalks was 0.5 g/cm^3, whereas the density of spikelets was 0.71 to 1.53 g/cm^3. The density of spikelets was comparable with OPEFB fibers of previous reports, which were 1.15 g/cm^3 (Yusoff *et al.* 2010) and 0.70 to 1.55 g/cm^3

(Mahjoub *et al.* 2013). Because of their low density, stalk fibers are a real alternative to commonly used synthetic reinforcing fibers. Their light weight makes them easy to transport and thus energy-efficient.

Besides parenchyma, vascular bundles are common tissue of both OPEFB stalks and spikelets. In the samples examined in this study, stalk bundles were bright brown, while spikelet bundles were slightly dark brown. After treatment at 100 and 190 °C for 15 min, the color of stalk bundles changed from light brown to dark brown, and a burnt sugar odor was produced. These results were similar to those in a previous study (Bahrin *et al.* 2012b). The oven-heat treatment volatilizes certain extractives and promotes photochemical reactions between carbohydrate and lignin. The stalk bundle length ranged from 1944 to 5103 μm with a bundle width of 78 to 169 μm (Fig. 1). A previous study reported that each OPEFB fiber had an average length of 0.67 mm, width of 12.5 μm, lumen width of 7.9 μm, and surface area of 75.6 μm^2 (Rozman *et al*. 2005).



(a) **(b)**

Fig. 1. Fiber dimensions were determined with a digital microscope: (a) spikelet vascular bundle and (b) stalk vascular bundle

Scanning Electron Microscopy

The morphological structures of OPEFB stalk fibers with and without oven-heat treatment were observed at 400x and 500x magnification (Fig. 2). Both untreated and treated OPEFB stalk fibers had even, smooth, and irregular external surface deposited with cementing agents, including hemicellulose, lignin, extractive, and other inorganic substances (Fig. 2, panels a through c). These cementing agents were not removed by delignification, superheated steam-dried treatment, or steam explosion. In addition, short oven-heat treatment did not remove cementing agents. There were folds and scars in all stalk fiber samples as a result of disc milling, which damaged the fiber surfaces.

Many spherical silica particles were observed. The silica bodies were white in color, 11 to 16 μm in diameter, arranged in regular patterns, and embedded in extracellular cavities, as previously described (Hasibuan and Daud 2009; Omar *et al.* 2014). Silica bodies were not removed by oven-heat treatment at 100 °C or 190 °C for 15 min. According to Hasibuan and Daud (2009), their removal requires superheated steam drying.

Disc milling destroyed silica bodies, exposing cellulose and hemicellulose (Fig. 2c, d). Silica bodies function as physical barriers to fungal attack. The presence of silica

in the cell wall can act as a barrier in the enzymatic digestibility and SSF system (Akhtar *et al.* 2015). Hammering (milling) and washing removes only 88% of the silica. In addition, superheated steam drying conducted at 140 °C and a velocity of 0.40 m/s dislodges silica particles without damaging OPEFB fibers (Hasibuan and Daud 2009). According to Omar *et al.* (2014), the absence of silica bodies increases enzyme access to the OPEFB internal layer, resulting in good digestion and hydrolysis during saccharification. This result was supported by EDS data showing decreases in silica content. However, milling and grinding decreases cellulose crystallinity and polymerization; these processes also cause biomass shearing (Baharuddin *et al.* 2012).

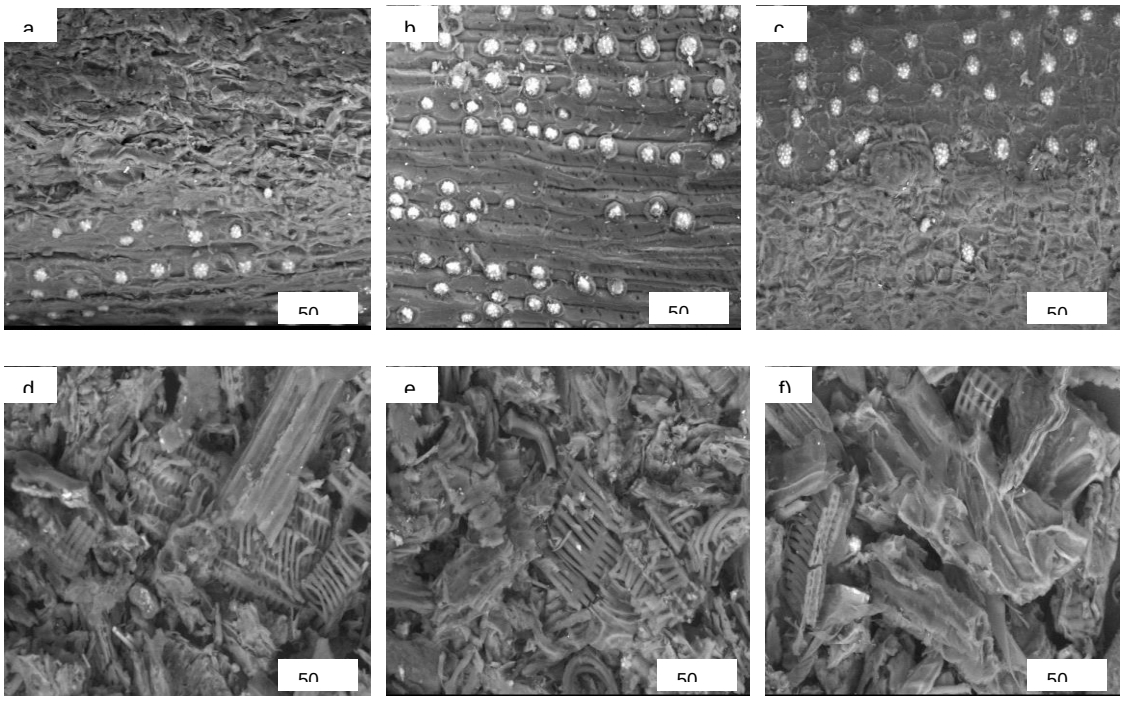


Fig. 2. Morphological microstructure analysis of OPEFB stalk fibers: (a) untreated stalk fibers, (b) stalk fibers treated at 100 °C for 15 min, (c) stalk fibers treated at 190 °C for 15 min, (d) 200-mesh untreated stalk fibers, (e) 200-mesh stalk fibers treated at 100 °C for 15 min, and (f) 200-mesh stalk fibers treated at 190 °C for 15 min

Chemical Composition Analysis

Table 1 shows the chemical composition of OPEFB stalk fibers and the spikelet/stalk mixture (1:1). Both stalk and mixture fiber samples showed increased cellulose after treatment at 100 °C for 15 min, but the cellulose content decreased after treatment at 190 °C for 15 min. The highest measured cellulose content of stalk and the mixture was 41.7% and 37.7%, respectively. In previous studies, the cellulose content was measured as 26.9 to 28.8% of OPEFB stalk fibers (Yunos *et al.* 2015), and the percentage of OPEFB fiber cellulose was in a range from 38.1% to 59.7% (Shahriarinour *et al.* 2011; Abdullah and Sulaiman 2013). Thus, the treatment at 100 °C for 15 min could increase the relative cellulose content.

The initial increase in the proportion of cellulose in the treated stalk material was brought about by the degradation of hemicellulose. The subsequent decrease in cellulose could be attributed to its lesser thermal stability in comparison to lignin. Crystalline regions of cellulose harbor the potential to be very resistant to oven heat relative to

amorphous regions. Hemicellulose is enriched with many polyose chains and branch structures, and it is susceptible to decomposition at low temperatures (Bahrin *et al.* 2012b). Furthermore, hemicellulose degradation is associated with the generation of organic acid by the cleavage of hemicellulose acetyl groups, which also generates cellulose (Baharuddin *et al*. 2012).

The other indispensable chemical components of OPEFB stalk fibers are lignin and extractives. Lignin and extractives in stalk fibers ranged from 15.5% to 16.4% and 2.7% to 3.8%, respectively, whereas these values in the mixture were 20.4% to 22.9% and 1.3% to 3.3%, respectively. According to Yunos *et al.* (2015), stalk fibers consist of 11.5% to 12.1% lignin and 28.0% to 28.4% extractives. Furthermore, raw OPEFB fibers consist of 16.2% lignin and 4.1% extractives (Wang *et al.* 2012). The decrease of lignin content after oven-heat treatment was due to the effects of a glass transition temperature of around 90 ºC and melting temperature around 170 ºC. The reason has been similiarly reported by Thomas *et al.* (2011).

Table 1. Chemical Composition Analysis of OPEFB Fibers

OPEFB	Chemical Composition (%)					
	Cellulose	Hemicellulose	Lignin	Extractives	Moisture Content	Holocellulose
Untreated stalk	35.63	32.10	16.38	3.07	18.54	67.73
Stalk treated 100 °C, 15 min	41.68	20.35	16.78	3.83	15.42	62.03
Stalk treated 190 °C, 15 min	30.11	22.01	15.51	2.72	12.07	52.12
Untreated mixture	32.39	30.25	21.64	1.29	14.34	62.64
Mixture treated 100 °C, 15 min	37.73	21.46	22.93	3.01	14.32	59.19
Mixture treated 190 °C, 15 min	32.39	20.70	20.43	3.34	11.35	53.09

X-Ray Diffraction

Raw stalk fibers are biopolymers containing amorphous (less ordered) and crystalline (ordered) regions. Amorphous regions are categorized as imperfect cellulose crystallites and less ordered structures, while crystalline regions are a perfect arrangement of ordered cellulose (Park *et al.* 2010). XRD analysis of untreated and treated OPEFB stalk fibers is shown in Fig. 3. In the diffractogram of OPEFB stalk fiber treated at 190 °C, there was a single high intensity peak at 22.5° (002), suggesting a crystalline nature (Karimi *et al.* 2014). Single high intensity peaks at 22.54° (002) and 22.28° (002) were observed for untreated fiber and fiber treated at 100 °C 15 min, respectively.

Cellulose crystalline peaks were more intense with increasing of oven-heat treatment because of the removal of hemicellulose. Similarly, Chan *et al*. (2013) reported that the removal of amorphous hemicellulose increases the CrI of kenaf core treated with

an alkaline treatment. In the XRD data from both treated and untreated OPEFB stalk fibers, an amorphous broad hump at 2θ value of 16º and the absence of the doublet located at 22.6° indicated that cellulose I, but not cellulose II, was present (Haafiz *et al*. 2013).

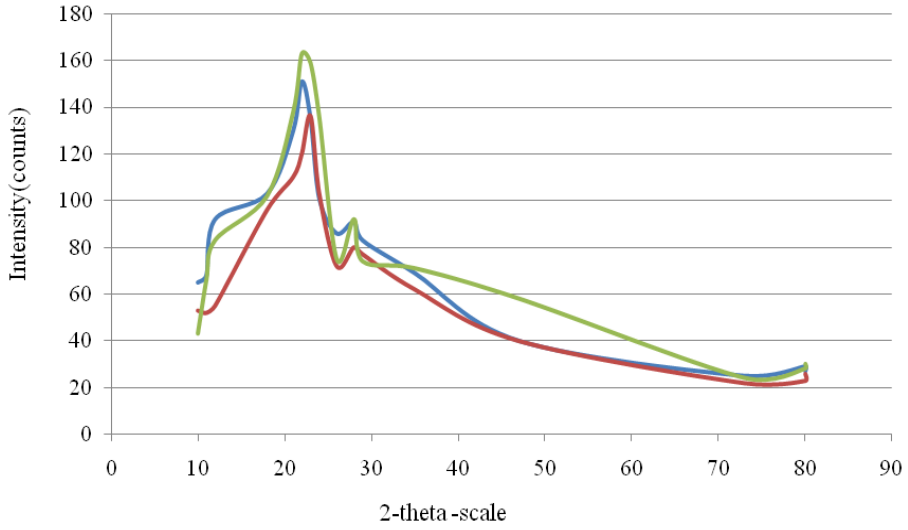


Fig. 3. X-ray diffractograms of (a) untreated OPEFB stalk fiber, (b) OPEFB stalk fiber treated at 100 °C for 15 min, and (c) OPEFB stalk fiber treated at 190 °C for 15 min

The crystallinity index (CrI) of untreated OPEFB stalk fibers was 41.4%, while the corresponding values for the CIs of stalk fibers treated at 100 °C and 190 °C for 15 min were 46.6% and 48.7%, respectively. The crystallinity index of raw OPEFB fibers can reach 43.9% (Nazir *et al.* 2013).

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of untreated and treated OPEFB stalk fibers are shown in Fig. 4. Most band characteristics were slightly changed after oven-heat treatment. The characteristic band located at 1034 cm^{-1} indicated the vibration of C-O-C or C-O in cellulose. Cellulose was also found in a peak at 1420 cm^{-1}, representing the bending vibrations of -CH_2. An initial band assigned to wood cellulose was located at 1425 cm^{-1} due to –CH_2 mode (Lionetto *et al.* 2012). The transmittance at 1250 cm^{-1} of untreated and treated OPEFB stalk fibers was associated with the C-O stretching of aryl alkyl ether in lignin. These results were similar to previous studies on kenaf bast fibers and OPEFB microcrystalline cellulose (Karimi *et al.* 2012; Nazir *et al*. 2013).

The transmittance peak at 1728 cm^{-1}, which appeared both in untreated and treated OPEFB stalk fibers, was attributed to a C=O stretching vibration of acetyl groups of hemicellulose or carbonyl ester of the *p*-coumaric monomeric lignin unit. This observation is in accordance with previous research (Chan *et al.* 2013; Nazir *et al.* 2013). The absorption peak of the –OH stretching and bending of cellulose was located at 3348 cm^{-1}. The transmittance peak at 2916 cm^{-1} corresponded to C-H group stretching vibrations and to the overlapping symmetric and asymmetric C-H stretching vibrations of aliphatic chains (Huq *et al.* 2012).

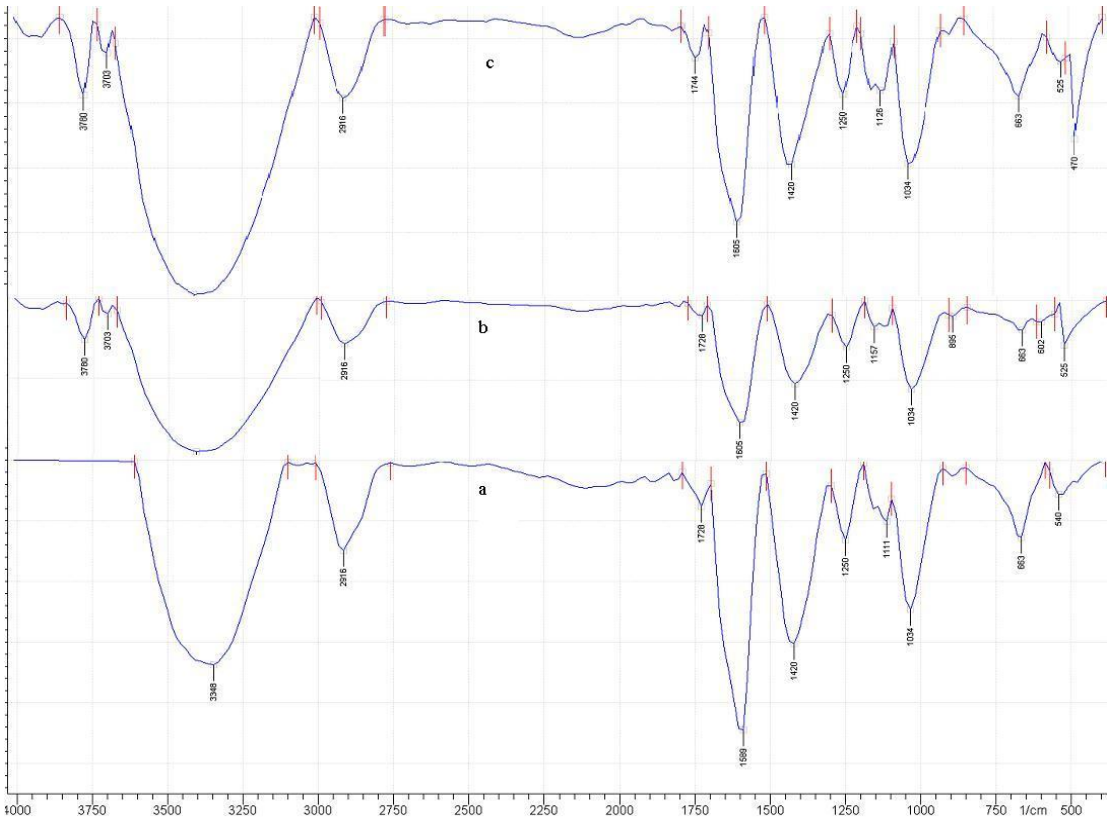


Fig. 4. FTIR spectra of OPEFB stalk fibers: (a) untreated, (b) treated at 100 °C for 15 min, (c) treated at 190 °C for 15 min

Energy Dispersive X-Ray Spectroscopy

The elemental composition of OPEFB stalk fibers is illustrated in Fig. 5. The fibers contained lignocellulose materials, including cellulose, hemicellulose, lignin, extractives, and ash (Sadikin *et al.* 2010). C and O were the major elemental components, whereas the predominant inorganic components of OPEFB stalk fibers were Si and K. After treatment with oven heat at 100 and 190 °C, there was slightly noticeable alteration of elemental components which was in accordance with FTIR analysis. Stalk fibers were composed of C (48.4% to 51.6%), O (33.8% to 44.6%), Si (1.6% to 2.1%), K (2.0% to 3.4%), P (0.09% to 0.2%), S (0.08% to 0.16%), Mg (0.13% to 0.40%), and Ca (0.08% to 0.77%). Generally, pressed-shredded OPEFB fibers consist of C, O, N, P, Ca, Mg, S, Ca, and other metal elements (Razali *et al*. 2012). In addition, OPEFB contains a large proportion of volatile contents (76.8 mf wt%) but very little nitrogen (2.2 mf wt%) or sulfur (0.9 mf wt%), so it can be regarded as an environmentally friendly biomass (Abdullah and Sulaiman 2013). After pulverizing stalk fibers by disc milling, there was a dramatic change in Si and K content. The percentage of silica content were reduced after disc milling the samples to 70 µm, while potassium was increased. It was predicted that milling would remove silica bodies and increase potassium. However, it is difficult to remove silica bodies from OPEFB fiber; they must be removed by high-pressure steam or chemical treatments (Omar *et al*. 2014).

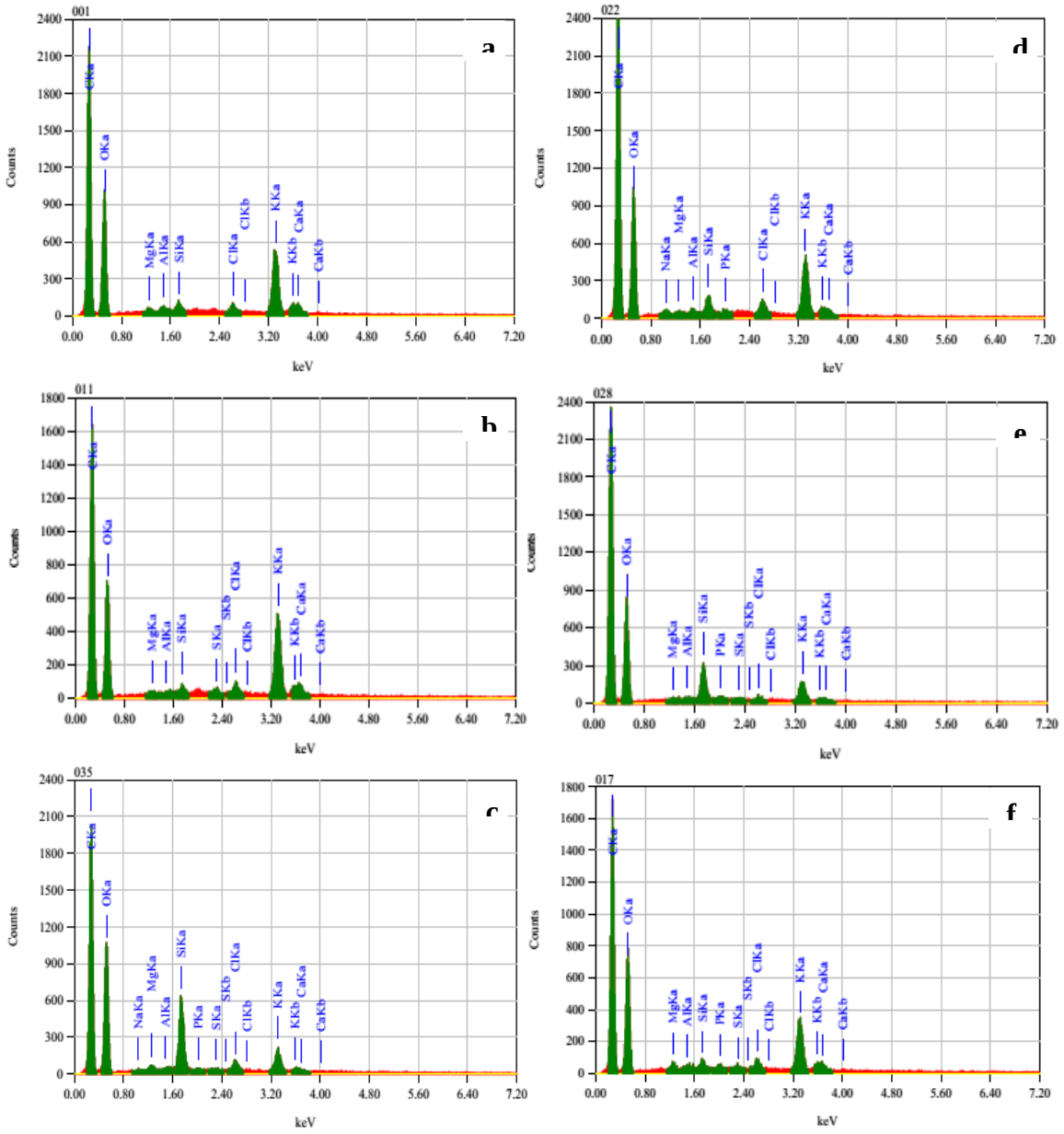


Fig. 5. Elemental composition analysis of OPEFB stalk fibers: (a) untreated stalk fibers, (b) stalk fibers treated at 100 °C for 15 min, (c) stalk fibers treated at 190 °C for 15 min, (d) 200-mesh untreated stalk fibers, (e) 200-mesh stalk fibers treated at 100 °C for 15 min, and (f) 200-mesh stalk fibers treated at 190 °C for 15 min

Differential Scanning Calorimetry

For typical lignocellulosic materials there are three stages of mass loss during thermal degradation, which are the removal of moisture (drying), release of organic matter (devolatilization), and oxidation of fixed carbon (slow combustion). Lignocellulosic materials degrade below 550 °C, with hemicelluloses degrading in the range 150 to 350 °C, cellulose degrading at 275 to 380 °C, and lignin degrading at 350 to 550 °C (Genieva *et al.* 2011). Thermal analysis of untreated and treated OPEFB stalk fibers showed that these samples have similar processes and peaks of thermal degradation

(Fig. 6). As predicted, oven-heat treatment of short duration resulted in changes in the thermal stability of OPEFB stalk fibers.

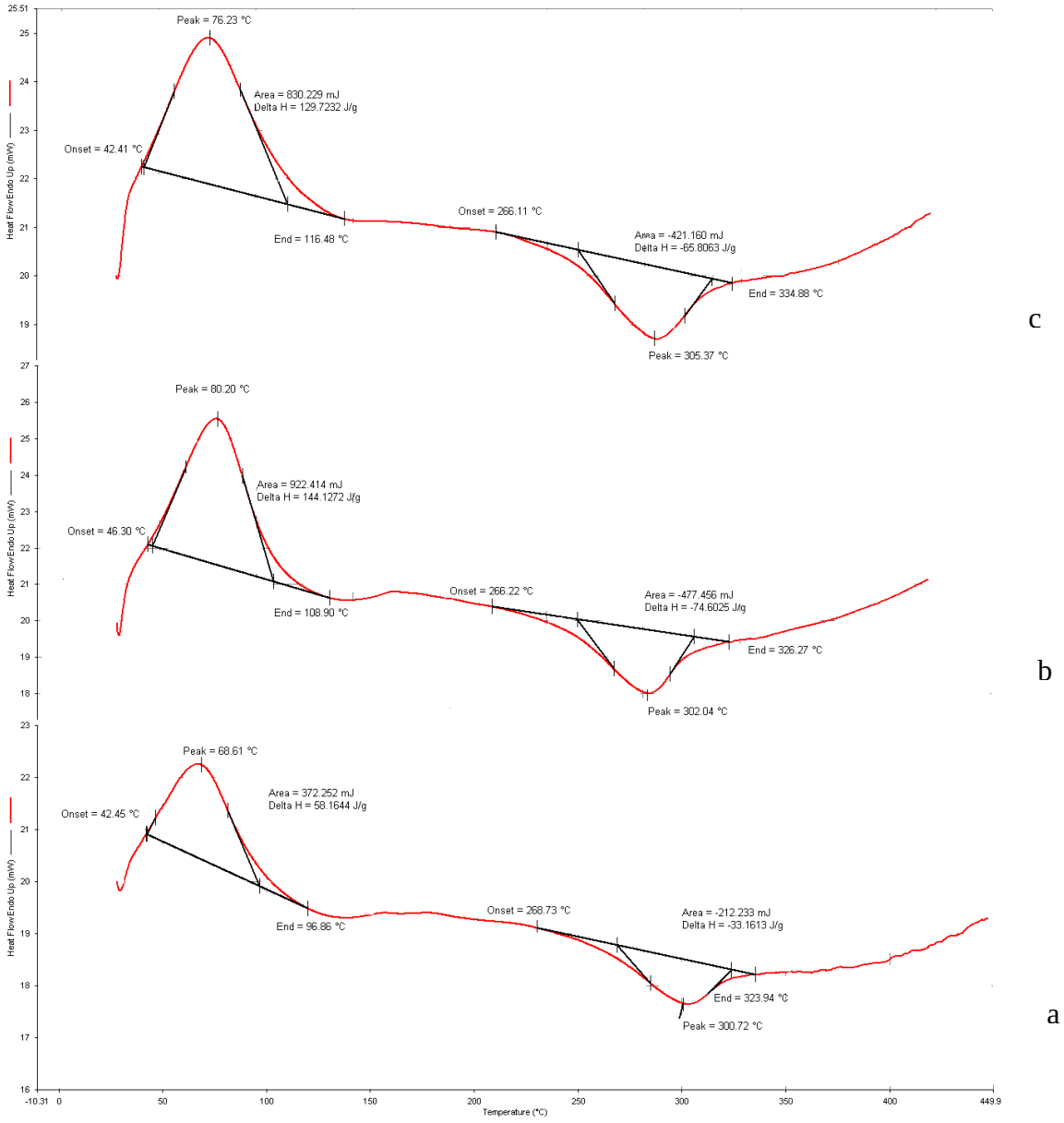


Fig. 6. DSC analysis comparision of (a) untreated stalk fibers, (b) stalk fibers treated at 100 °C for 15 min, (c) and stalk fibers treated at 190 °C for 15 min

A broad exothermic peak in the temperature range of 42 to 116 °C was observed. In the exothermic peak of 68 to 80 °C, a minor weight loss occurred as a result of water evaporation or water removal and low-temperature degradation of hemicelluloses, wax, and pectin. Water is lost at 110 °C during the low-temperature degradation of hemicelluloses, wax, and pectin, which ranges from 150 to 230 °C in raw OPEFB fibers (Nazir *et al.* 2013). The temperature range of 75 to 120 °C represents the removal of moisture in untreated, silane-treated, and alkali-treated natural fibers (Mathew *et al.*

2011). In the exothermic peak, the highest delta H was in the sample treated with 100 °C for 15 min (144 J/g). This sample absorbed heat for the removal of moisture.

At the second stage of degradation, an endothermic process for both untreated and treated OPEFB stalk fibers occurred between 266 and 334 °C, which represented the thermal depolymerisation of hemicellulose and cleavage of the glycosidic linkages of cellulose. At 230 °C, cellulose degradation occurs; it levels off at 350 °C indicating a slow decomposition of lignin (Nazir *et al.* 2013). In sisal fiber, the peak at 350 °C is due to the thermal depolymerisation of hemicellulose and the cleavage of the glycosidic linkages of cellulose (Saxena *et al.* 2011). In this study, the slow decomposition of lignin started at 326 °C of in the stalk fiber sample treated at 100 °C for 15 min. Furhermore, the highest delta enthalpy value (-477 J/g), and thus, release of heat, was observed in the sample treated at 100 °C for 15 min.

CONCLUSIONS

1. Oven-heat treatment was able to slightly alter the properties of OPEFB stalk fiber properties. The changes included morphological, chemical, and thermal properties.
2. There were color alterations in oven-heated OPEFB stalk fibers, and extractive substances may have been volatilized. Both untreated and treated OPEFB stalk fibers had even and smooth surfaces with irregular external surface of OPEFB stalk fibers deposited with cementing agents.
3. During oven-heat treatment, hemicellulose in stalk fibers was very sensitive to degradation, leading to increased cellulose content. Cellulose, hemicellulose, lignin, and extractives were present after oven-heat treatment.
4. Generally, treated and untreated OPEFB stalk fibers consisted of C, O, Si, and other elemental traces. In untreated and treated OPEFB stalk fibers, thermal degradation of cellulose, hemicellulose, and lignin occurred with almost similar temperatures.

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