EFFECT OF OPERATING TEMPERATURE ON PHYSICOCHEMICAL PROPERTIES OF EMPTY FRUIT BUNCH CELLULOSE-DERIVED BIOCHAR

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ABSTRACT

The oil palm lignocellulosic biomass is mass-produced which leads to management and disposal issue. Hence, converting it into carbonaceous material such as biochar is advantageous. One such by-products namely empty fruit bunch (EFB), comprises of 44.4 wt% cellulose, rendering it a prominent feedstock for biochar production. The study focuses on assessing the effect of pyrolysis temperatures on cellulose biochar properties and yields. The cellulose was extracted via standard method and carbonised using thermogravimetric analyser. The proximate and ultimate analyses and Fourier-transform infrared spectroscopy (FTIR) were performed to determine the biochar characteristics. Lower biochar yield, volatile matter (VM) and hydrogen contents were generated at higher temperature, whereas an opposite trend was observed for moisture, fixed carbon and ash contents. The FTIR spectra verified the presence of carboxyl, aromatic and hydroxyl groups at 250°C and 400°C; however, the bands diminished at 750°C. This work has identified that biochar produced at 250°C possesses excellent properties including higher biochar yield (32.51% ± 0.48), carbon content (57.98 wt%) and VM (38.68 wt%). High level of VM is beneficial for microbial rejuvenation, which is ideal for soil amendment. This study provides a key basis in establishing the suitable biochar properties and pyrolysis parameter for soil amendment as well as other applications.

Keywords: biochar, cellulose, empty fruit bunch, pyrolysis, thermal degradation.

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INTRODUCTION

Nowadays, heavy dependency on non-renewable fuels such as petroleum as main energy source has resulted in a severe energy calamity and degradation of the environment. As such, there has been a shift of interest in utilising biomass waste as a fossil fuel substitute, as it emits less greenhouse gas (GHG) when combusted due to its carbon-rich characteristics comprising of cellulose, hemicellulose and lignin and thus, greatly reduces reliance on fossil fuel.

With contribution of 47% to the world’s palm oil supply, Malaysia is the second largest country to produce and export palm oil (Kushairi et al., 2019). Processing of 1 t fresh fruit bunches (FFB) generates 20%-22% crude palm oil (CPO) along with other oil palm biomass by-products including 23%-25% empty fruit bunches (EFB), 13%-15% mesocarp fibre (MF) and 5%-6% palm kernel shell (PKS). In palm oil mills, EFB is normally utilised as fuel in boilers for steam and electricity generation or incinerated for fertiliser production. However, a vast amount is still available and unutilised, hence means of disposing them cost-effectively is important (Lahijani and Zainal, 2011). Disposal of oil palm biomass solid...
MATERIALS AND METHODS

Materials

Raw EFB fibres were sourced from a palm oil mill located at Kota Tinggi, Johor, Malaysia. All chemicals used were of analytical grade purchased from Merck, Germany. Commercial microcrystalline cellulose (Sigma Corporation, USA) was used as a control sample.

Method

Sample preparation. The EFB was washed with distilled water to eliminate contaminant prior to drying in an oven for 24 hr at temperature of 105°C. The dried EFB was then cut into small pieces and ground using fibre grinders (Retsch, model AS200, USA) and screened to a range of particle sizes between 180-250 μm.

Cellulose extraction. Extraction of cellulose from EFB was carried out according to ASTM standard method D1103-60. About 12 g of EFB was placed in a 1-litre beaker containing 480 ml distilled water, 1.5 ml acetic acid and 4.5 g sodium chlorite. The mixture was stirred on a hot plate with temperature set at 75°C. Acetic acid, 1.5 ml and 4.5 g sodium chlorite were added into the reaction mixture after 1 hr and the same amount repeated after 2 hr. The volume of water was maintained during the chlorination duration. After cooling the reaction
mixture to <10°C, the slurry was filtered using a 50-μm sieve filter for liquid and solid separation. The solid was then put in the petri dish and dried overnight to obtain holocellulose. Approximately 2 g of holocellulose was poured into a glass beaker together with 20 ml of cold sodium hydroxide (NaOH) (17.5% w/v) solution and stirred continuously for 5 min. Another 10 ml of NaOH (17.5% w/v) solution was added after 5 and 10 min repetitively and the mixture was allowed to stand for 30 min at 20°C. Then, 67 ml 20°C cold distilled water was put in the mixture and stirred thoroughly for 45 min with temperature maintained at 20°C before filtering. The α-cellulose extracted was washed with 50 ml of cold NaOH (8.3% w/v) twice and distilled water. Lastly, the α-cellulose was immersed in 25 ml acetic acid (10% v/v) twice and rinsed with distilled water prior to oven-drying at 105°C overnight to obtain a constant weight.

**Biochar production.** The experiment of biochar production was established by carbonising the EFB fibre via a thermogravimetric analyser (TGA 701 LECO, USA) at three different thermo-chemical conditions: 250°C, 400°C and 750°C. The EFB fibre was put in a ceramic cubicle and heated from 30°C-105°C at 10°C min⁻¹ heating rate under a nitrogen gas flow rate of 30 ml min⁻¹. The temperature was kept constant for 10 min at 105°C and then increased to the defined temperatures. The biochar produced at varied temperatures of 250°C, 400°C and 750°C were collected for further analysis.

**Characterisation.** The raw EFB fibre, commercial cellulose, EFBC and EFBC-derived biochars were characterised as follows:

i) **Fourier-transform Infrared Spectroscopy (FTIR)**

The chemical bond and functional groups of the samples were elucidated using FTIR (Perkin-Elmer Frontier, USA) at 4 cm⁻¹ resolution and eight scans. Potassium bromide was mixed with the oven-dried samples and then pressed using a mortar to form a disk. Spectra of the analysed samples were recorded over a wavelength from 4000 cm⁻¹ to 650 cm⁻¹.

ii) **Morphology Analysis**

The surface morphology and elemental analysis of the samples were conducted via scanning electron microscopy (SEM) (Hitachi Model S-3400N, Japan) at 30 kV accelerating voltage. The sample surfaces were placed on the aluminum stubs and sputter-coated thinly by palladium to prevent electrostatic charging during analysis.

iii) **Proximate and Ultimate Analyses**

The proximate analysis of the samples was employed to determine the fixed carbon, moisture, VM and ash content via a thermogravimetric analyser (Perkin Elmer TGA-Pyris 6, USA) in accordance with the standard method American Society for Testing and Materials (ASTM) D1103-60, USA (Mayoral et al., 2001). The ultimate analysis for chemical compositions of the samples was determined using a CHNS Determinator (LECO CHNS M628, USA) according to ASTM D5373.

**RESULTS AND DISCUSSION**

**FTIR Analysis**

The effects of different temperatures on raw EFB fibre, commercial cellulose, EFBC and EFBC-derived biochars functional groups were evaluated via FTIR spectroscopy. The FTIR spectra (Figure 1) showed two main regions i.e. fingerprint region (1450 to 400 cm⁻¹) and functional group region (4000-1450 cm⁻¹) (Lani et al., 2014). The absorption peaks at 3406 cm⁻¹ are assigned to polymeric hydroxide (-OH) intramolecular stretching, whereas those of 2850 cm⁻¹ are attributed to methylene (CH₂) asymmetric stretching vibration (Khalil et al., 2001; Lv et al., 2015). As depicted in Figure 1, the peaks at ~3406 cm⁻¹ found in all spectra correspond to the stretching of hydrogen-bonded hydroxyl groups, indicating the presence of phenols and alcohols (Cantrell et al., 2012). However, the absence of vibration peak between 1750 cm⁻¹ attributable to a waxy C=O acetyl group (hemicellulose) and ester carbonyl groups (p-coumaric lignin) in all the feedstocks except for raw EFB fibre was indicative of successful removal of hemicelluloses and lignin components during EFBC extraction. Although the extracted cellulose was quite pure, there was residual lignin remained as indicated by absorption peak at ~1630 cm⁻¹ (Table 1). These findings also complement those of SEM results, showing smoother and wrinkled surface of EFBC thus, confirming that lignin and hemicellulose components have been completely removed. Besides, absorption peaks at around 3300, 2890, 1638, 1426, 1365, 1310, 1210, 1028 and 895 cm⁻¹ were observed in both the commercial cellulose and EFBC, which indicated typical absorption characteristics for cellulose (Figures 1b and 1c) (Lv et al., 2015). The peaks at 1400 cm⁻¹, 760-800 cm⁻¹ and 710 cm⁻¹ (Figures 1e and 1f) appearing in the EFBC-derived biochars at higher temperature imply that some aromatic carbon structures are present due to condensation and aromatisation of biochar from decomposed cellulose.
Based on the findings (Table 1), the bonds of \( =\text{C-H} \) bending (1424 cm\(^{-1}\)) and \( \text{CH}_2 \) wagging in cellulose (1315 cm\(^{-1}\)) started to diminish when the temperature escalated from 250ºC-750ºC, suggesting depolymerisation and degradation of cellulose. Similar pattern was observed for peaks appearing around 1214 cm\(^{-1}\) attributable to phenolic and ester C-O (hemicellulose) stretching. Meanwhile, the band within the saturated region of 2893-2852 cm\(^{-1}\) visible earlier in the raw EFB fibre (Figure 1a), commercial cellulose (Figure 1b), EFBC (Figure 1c) and EFBC-derived biochar at 250ºC (Figure 1d) had slowly diminished (Figures 1e and 1f) with increasing pyrolysis temperature. High loss of mass throughout the thermal decomposition could be the reason. According to Claoston et al. (2014), decomposition in the biomass accelerates a decline in the polar functional groups when pyrolysis temperature increases. Overall, the FTIR spectra demonstrated that EFBC was successfully extracted from the raw EFB fibre, as confirmed by similar functional groups exhibited between EFBC and commercial cellulose (Figures 1b and 1c). Besides, the pyrolysis temperature employed was able to affect the existence of functional groups in the feedstock, where disappearance of functional groups became more distinct in the EFBC-derived biochars at higher pyrolysis temperature (Figures 1e and 1f).

Morphology Analysis

Figure 2 depicts the physical appearance of feedstocks (raw EFB fibre and EFBC) and EFBC-derived biochar produced at different temperatures. It showed that EFBC had been completely carbonised, turning from yellowish to black at temperatures of 250ºC, 400ºC and 750ºC (Figures 2d-f). The employed temperature ranges had successfully removed the surface and bound water resulted in great reduction of moisture content which then facilitated carbonisation of EFBC. These findings were consistent with a study previously reported by Uemura et al. (2011a). The morphological structures of raw EFB fibre, commercial cellulose, EFBC and EFBC-derived biochar were assessed via SEM at different magnification. These micrographs show gradual changes of the effect of different temperature on the surface textures. The SEM micrograph of raw EFB fibre in Figure 3a indicated a lignocellulosic composite with compact fibrillar packing and the external surface suggested an irregular heavy deposition of wax, hemicelluloses and lignin. On the other hand, the commercial cellulose and EFBC (Figures 3b and 3c) exhibited an isolated fibril which was separated from each other suggesting non-cellulosic components and impurities such as cuticle. The wax layer was removed from fibre surface during the extraction process which contributed to a more wrinkled and clearer surface (Draman et al., 2013; Nazir et al., 2013; Suriya et al., 2017). As illustrated in Figure 3d, the surface of biochar produced at 250ºC appeared to be smoother with rough texture unlike those biochars generated at higher temperature (400ºC and 750ºC), the external surface seemed to have been cracked with greater shrinkages. When temperature increased to 750ºC (Figure 3f), the outer surface of the biochar produced began to collapse and fracture. Claoston et al. (2014) also reported similar findings as evidenced by the inability of the produced biochar to endure high temperature as the thin wall structure was fragile and easily diminished.
TABLE 1. FUNCTIONAL GROUPS OBSERVED IN THE FTIR SPECTRA OF RAW EMPTY FRUIT BUNCH (EFB) FIBRE, COMMERCIAL CELLULOSE, EFB CELLULOSE (EFBC) AND EFBC-DERIVED BIOCHARS PRODUCED AT DIFFERENT TEMPERATURES

| Wavenumbers (cm⁻¹) | Functional groups and bonds | Raw EFB fibre | Commercial cellulose | EFBC 250°C | EFBC 400°C | EFBC 750°C | References |
|-------------------|-----------------------------|---------------|-----------------------|-------------|-------------|-------------|------------|
| 2 850             | S-H stretching, O=Cl, CH₃ asymmetric stretching | 2 852(w)      | 2 896(s)              | 2 893(s)    | 2 893(m)    | /           | Lv et al. (2015); Owi et al. (2016) |
| 1 750             | C=O ketone and aldehyde (hemicellulose) | 1 737(m)      | /                     | /           | /           | /           | Shanmugarajah et al. (2015); Nurul et al. (2017) |
| 1 640             | C≡C and C=O aromatic lignin ring | /             | 1 638(m)              | 1 634(m)    | 1 614(m)    | /           | Lv et al. (2015); Shanmugarajah et al. (2015) |
| 1 424             | μ=C-H bending, μ=C-H₂ scissoring | 1 412(w)      | 1 426(w)              | 1 424(w)    | /           | /           | Lv et al. (2015); Zaini et al. (2013) |
| 1 315             | CH₃ wagging in cellulose | 1 315(w)      | /                     | /           | /           | /           | Lv et al. (2015) |
| 1 214             | Phenolic and ester C-O stretching | 1 240(m)      | 1 202(w)              | 1 200(w)    | /           | /           | Kong et al. (2019); Nurul et al. (2017); Shanmugarajah et al. (2015) |
| 1 160             | Asymmetric vibration of C-O-C | 1 158(w)      | 1 160(m)              | 1 158(m)    | 1 156(w)    | /           | Huang et al. (2020) |
| 1 030             | Tertiary alcohol C-O stretch | 1 031(s)      | 1 030(s)              | 1 025(s)    | 1 025(s)    | 1 059(s)    | Lv et al. (2015); Shanmugarajah et al. (2015) |
| 875               | Aromatic C-H bending | /             | 897(m)                | 895(m)      | 876(s)      | 872(s)      | Kong et al. (2019) |
| 799               | Aromatic CH out-of-plane | /             | 710(w)                | 713(w)      | 713(m)      | 712(m)      | Lv et al. (2015); Keiluweit et al. (2010) |
| 670               | C-C stretching | 663 (m)       | 664(m)                | 667(m)      | 670(m)      | /           | Owi et al. (2016) |

Note: s-strong; m-moderate; w-weak; / means no visible peak.
FTIR - Fourier-transform infrared spectroscopy.

Figure 2. The physical image of (a) raw empty fruit bunch fibre in brown colour, (b) commercial cellulose as a fine white powder, (c) empty fruit bunch cellulose in whitish colour with rough texture; empty fruit bunch cellulose-derived biochar produced at (d) 250°C, (e) 400°C, and (f) 750°C - all of which had turned from whitish into blackish due to carbonisation process.
Proximate and Ultimate Analyses

The properties of raw EFB fibre, commercial cellulose, EFBC and EFBC-derived biochar are summarised in Table 2. It can be observed that raw EFB fibre contains large percentage of VM (72.46 wt%), followed by fixed carbon (16.71 wt%), moisture (8.03 wt%) and ash (2.80 wt%), respectively. Meanwhile, the extracted cellulose, i.e. EFBC comprises of slightly higher VM content than the feedstock by 1.35% but lower fixed carbon (15.42 wt%) and moisture content (7.89 wt%) and the properties of EFBC were comparable with the commercial cellulose. In this study, the moisture content of the biochar was reduced from the feedstock through losses of water as steam due to the elevated temperatures employed. However, the moisture levels in the generated biochars increased as the temperature increased, i.e. 7.14 wt% (250°C), 15.59 wt% (400°C) and 17.75 wt% (750°C), due to the hydroscopic effect exerted by their higher surface areas (Askeland et al., 2019). Meanwhile, the moisture levels in the generated biochars increased as the temperature increased, i.e. 7.14 wt% (250°C), 15.59 wt% (400°C) and 17.75 wt% (750°C), due to the hydroscopic effect exerted by their higher surface areas (Askeland et al., 2019). Meanwhile, opposite trend was observed for VM; the values decreased from 38.68 wt% (250°C) to 8.90 wt% (750°C) which were consistent with other studies (Tag et al., 2016; Zhao et al., 2017) due to losses through outgassing. Furthermore, high temperature might have resulted in the dehydration of hydroxyl groups and thermal degradation of cellulose. The EFBC-derived biochars have lower percentage of VM as compared to raw EFB fibre because VM was released during the pyrolysis process. This study revealed that the percentages of fixed carbons in biochars (50.85-63.69 wt%) were higher than those of the raw EFB fibre and EFBC. Fixed carbon was found to be higher in biochar produced at 750°C. Generally, higher values of fixed carbon would be indicative of a longer residence time of biochar in soil. These findings were in agreement with other studies by Shariff et al. (2014) and Domingues et al. (2017). Meanwhile, the carbon fractions increased at higher temperature: from 0.74 wt% to 5.40 wt% and this was expected because an increased devolatilisation during pyrolysis could reduce the oxygen content of biochar and thus, the ash content increased (Domingues et al., 2017). According to Tsai et al. (2012), increase in ash content in the biochar is due to the progressive concentration of minerals and destruction of lignocellulosic matters.

The ultimate analysis revealed that pyrolysis temperature affected the elemental compositions of the generated biochars (Table 2). The carbon content of EFBC (38.8 wt%) increased with increasing temperature and reached 65.26 wt% at temperature of 400°C but slightly decreased (42.27 wt%) with further increase of temperature until 750°C. The decline was attributed to the reduction rate of carbonisation. Meanwhile, hydrogen (H₂) content decreased at higher pyrolysis temperature as methane (CH₄) and hydrogen formed and released during the pyrolysis process. This phenomenon is indicative of disappearance of some functional groups such as hydroxyl (OH) and carbonyl (COOH) through volatilisation (Bridgeman et al., 2008; Uemura et al., 2011b). The carbon and nitrogen contents in the EFBC-derived biochar increased while the oxygen and hydrogen contents decreased with increasing temperature compared to the initial feedstock (empty fruit bunch cellulose). However, this trend was invalid at pyrolysis temperature of 750°C as the carbon and oxygen contents fluctuated. The carbon

\[ \text{Figure 3. The scanning electron microscope (SEM) micrograph of (a) compact fibrillar packing surface of raw empty fruit bunch fibre; (b) an isolated fibril fibre fragments of commercial cellulose (c) a rod-shaped and individualise fibrous structure of empty fruit bunch cellulose; empty fruit bunch cellulose-derived biochar produced at (d) 250°C with rough surface structure (e) 400°C with uneven surface, and (f) 750°C with crack outer surface owing to higher temperature intolerance.} \]
content of biochar is an important indicator to identify its potential carbon sequestration during soil application (Duku et al., 2011; Galinato et al., 2011). According to Imam and Capareda (2012), the fluctuation might be caused by the breaking of weak bonding in the biochar structure although high carbonaceous materials are produced with increasing temperature.

Figure 4 shows that the EFBC-derived biochar production decreased markedly from 32.51% ± 0.48 to 9.02% ± 0.13 as the pyrolysis temperature escalated from 250°C-400°C. At this point, most of the lignocellulosic materials were more likely to decompose as reported by Intani et al. (2016). Nevertheless, the production of EFBC-derived biochar merely decreased further by 5% at 750°C, which indicated extensive decomposition of most of the volatile fractions (Lua et al., 2004). At higher temperatures, organic compounds with higher molecular weight in the biomass could devolatilise into lower molecular weight compounds along with some light gases as well (Thangalazhy et al., 2010). Dehydration and elimination would proceed faster at higher temperatures, which leads to a decreased biochar yield. Demirbas (2004) reported that biochar production relies on the adverse reaction of cellulose and biochars polymerisation process. The biochar product yield is an important factor for realising economic benefit in biomass waste reutilisation. From the finding, higher pyrolysis temperatures caused greater losses of VM, oxygen, hydrogen as well as biochar yield due to depolymerisation of lignocelluloses and carbonisation to a more recalcitrant form through decarboxylation, dehydration, condensation and aromatisation reactions (Das and Sarmah, 2015; Heitkotter and Marschner, 2015). Future study will focus on pyrolytic mechanism and the associated kinetics to understand wholly the biomass-to-biochar conversion process.

**CONCLUSION**

The present work concludes that cellulose was successfully extracted from the raw EFB fibre and the extracted cellulose exhibited similar properties as the commercially available

| Parameter (wt.% d.b.) | C   | H   | N   | O   | Moisture content | Volatile matter | Fixed carbon | Ash content |
|-----------------------|-----|-----|-----|-----|------------------|----------------|--------------|-------------|
| Raw EFB fibre         | 44.08 | 7.54 | 0.15 | 48.21 | 8.03             | 72.46          | 16.71        | 2.80        |
| Commercial cellulose  | 41.02 | 7.37 | <0.01 | 51.58 | 6.40             | 83.49          | 10.17        | <0.01       |
| EFBC                 | 38.80 | 6.96 | 0.18 | 54.06 | 7.89             | 73.81          | 15.42        | <0.01       |
| EFBC-derived biochar (250°C) | 57.98 | 2.38 | <0.01 | 39.64 | 7.14             | 38.68          | 50.85        | 0.74        |
| EFBC-derived biochar (400°C) | 65.26 | <0.01 | 0.41 | 34.33 | 15.59            | 20.32          | 58.29        | 0.89        |
| EFBC-derived biochar (750°C) | 42.27 | <0.01 | <0.01 | 57.73 | 17.75            | 8.90           | 63.69        | 5.40        |

Note: *Estimated by difference: %O = 100% - [% carbon (C) + % hydrogen (H) + % nitrogen (N)].
cellulose. The findings highlighted that different pyrolysis temperature had greatly influenced the physicochemical properties of the EFBC-derived biochars. The morphology and structure of the produced biochars were distinctively different in shape and form with increasing temperature. The biochar yield and total elemental content (oxygen, hydrogen and nitrogen) reduced while the carbon and ash content increased with increasing temperature. The highest achievable biochar yield was 32.51% at 250°C. The FTIR spectra revealed some variations and similarities in functional groups between the feedstocks and the EFBC-derived biochars. The biochars produced at lower temperature (250°C) exhibited characteristics favourable as a soil enhancer in improving soil fertility and quality such as high VM (38.68 wt%) and carbon (57.98 wt%) contents. Furthermore, other potential biochar applications can be considered including as an energy source (biofuel) and pollution remediation. Findings from this study provide insights into relationships between pyrolysis conditions, biochar properties and biochar processing which are crucial in optimising biochar production for soil application.

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