Enhanced oil recovery and lignocellulosic quality from oil palm biomass using combined pretreatment with compressed water and steam

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A B S T R AC T

A large volume of oil palm empty fruit bunch (EFB) is generated as waste feedstock around the globe. This abundant waste containing 0.75% oil on average could be a promising feedstock for biodiesel production if oil recovery could be accomplished in an economically-viable and environmentally-friendly manner. To achieve that, a new method called High Pressure Water Spray (HPWS) system was introduced and performed by spraying pressurized water (500 psi) at 30 °C, 60 °C, and 90 °C and combination of water-steam at 120 °C and 150 °C onto the surface of the oil palm empty fruit bunches (EFB). The results obtained indicated that, the highest oil removal yield of 94.41 ± 0.02 wt% was obtained at 150 °C. Moreover, bioprospection of biodiesel properties based on fatty acid methyl ester (FAME) profile revealed that the biodiesels produced from the fresh crude palm oil and residual oil were comparable and were in accordance with international standards. In addition to that, the HPWS process led to an enhanced quality of the remaining lignocellulosic materials for conversion into other value added bio-products such as ligno-ethanol by decreasing lignin content and increasing cellulose content. In view of environmental impact assessment, the HPWS system showed favorable impacts on all the end-point damage categories especially in resources damage category. Moreover, economic assessment showed that the recovered CPO could be generated at a low price of USD 0.41 vs. USD 0.66 for CPO. Overall, this process could drastically increase the market value of an abundant type of waste in many parts of the world, i.e., EFB leading to the generation of additional wealth for the palm oil industry.

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1. Introduction

Approximately 21% of oil palm fresh fruit bunch (FFB) ends up as oil palm Empty Fruit Bunches (EFB) which is translated into 1.5 tons of hectare/year (Abd Majid et al., 2012). The EFB is generated after the stripping process, in which the sterilized fruits are
EFB is made up of 20\% (Baharuddin et al., 2009; Ibrahim et al., 2009; Kheong et al., 2010). Palm industry worldwide, EFB is currently used as a wood composite, separated from the bunch stalks. As the major waste of the oil extraction rate (OER) of palm oil mills (Sahad et al., 2014). A study and stripping (Abd Majid et al., 2012). The oil retained on the EFB prolonged sterilization and also long delays between sterilizing contact time between oil and oil could be transferred to EFB surface due to mechanical pressure well as in steam turbines for power-generation. During processing, oil could be transferred to EFB surface due to mechanical pressure imposed by the stripping and threshing processes, or by increased contact time between oil and fiber for instance as a result of prolonged sterilization and also long delays between sterilizing and stripping (Abd Majid et al., 2012). The oil retained on the EFB fibers, considered as oil loss, has a negative impact on the total oil extraction rate (OER) of palm oil mills (Sahad et al., 2014). A study conducted by Ngan (2005) showed that EFB does contain a significant amount of oil. The total oil content retained on EFB fibers (residual oil) reportedly range between 0.28 and 1.38\% with a mean value of 0.75\% (dry EFB). Currently, the technology used to recover residual oil from EFB includes pressing and shredding processes. More specifically the EFB are transported into a pressing machine, where they are screw-pressed, and are then shredded for easier handling. The product generated from this process is known as press-shredded EFB (Jorgensen, 1985). However, this technology is not efficiently removing the residual oil from the EFB.

Therefore, the aim of the present study was to develop a more efficient technique namely high pressure water spray (HPWS) system to remove the residual oil from EFB. In fact, instead of using harmful and flammable chemicals such as hexane with potential carcinogenic effect, a combination steam and water was used for rapid, environmentally-friendly recovery of residual oil from EFB. The process presented herein could be very advantageous owing to the minimal cost and availability of excess steam in palm oil mills and could be conducted in continuous mode for achieving the zero waste strategy. Moreover, the effects of the HPWS systems on oil recovery yield and the physicochemical characteristics of EFB fiber, residual oil and the resultant biodiesel were investigated. Finally, the environmental impact assessment as well as the economic assessment of the HPWS system was presented.

### List of abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>AAS</td>
<td>Atomic Absorption Spectrophotometer</td>
</tr>
<tr>
<td>ADF</td>
<td>Acid Detergent Fiber</td>
</tr>
<tr>
<td>ADL</td>
<td>Acid Detergent Lignin</td>
</tr>
<tr>
<td>APE</td>
<td>Allylic Position Equivalent</td>
</tr>
<tr>
<td>BAPE</td>
<td>Bis- Allylic Position Equivalent</td>
</tr>
<tr>
<td>BET</td>
<td>Brunauer, Emmett, And Teller</td>
</tr>
<tr>
<td>CFP</td>
<td>Cold Filter Plugging Point</td>
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<tr>
<td>CN</td>
<td>Cetane Number</td>
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<tr>
<td>CP</td>
<td>Cloud Point</td>
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<tr>
<td>CPO</td>
<td>Crude Palm Oil</td>
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<tr>
<td>DH</td>
<td>Dollimore Heal</td>
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<tr>
<td>DOBI</td>
<td>Deterioration Of Bleach Ability Index</td>
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<td>DU</td>
<td>Degree of Unsaturation</td>
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<tr>
<td>EFB</td>
<td>Empty Fruit Bunch</td>
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<tr>
<td>FA</td>
<td>Fatty Acid</td>
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<tr>
<td>FAME</td>
<td>Fatty Acid Methyl Ester</td>
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<tr>
<td>FFA</td>
<td>Free Fatty Acids</td>
</tr>
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<td>FFB</td>
<td>Fresh Fruit Bunch</td>
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<tr>
<td>FID</td>
<td>Flame Ionization Detector</td>
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<tr>
<td>FTIR</td>
<td>Fourier Transforms Infrared Spectroscopy</td>
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<tr>
<td>GC</td>
<td>Gas chromatography</td>
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<tr>
<td>HHV</td>
<td>Higher Heating Value</td>
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<tr>
<td>HPWS</td>
<td>High Pressure Water Spray</td>
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<td>IV</td>
<td>Iodine Value</td>
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<tr>
<td>LCA</td>
<td>Life Cycle Assessment</td>
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<tr>
<td>LCIA</td>
<td>Lifecycle Impact Assessment</td>
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<tr>
<td>LCSF</td>
<td>Long Chain Saturated Factor</td>
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<tr>
<td>MPOB</td>
<td>Malaysian Palm Oil Board</td>
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<tr>
<td>NDF</td>
<td>Neutral Detergent Fiber</td>
</tr>
<tr>
<td>OS</td>
<td>Oxidation Stability</td>
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<tr>
<td>PP</td>
<td>Pour Point</td>
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<tr>
<td>PV</td>
<td>Peroxide Value</td>
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<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>SV</td>
<td>Saponification Value</td>
</tr>
</tbody>
</table>

### 2. Materials and methods

#### 2.1. Materials and reagents

Oil palm empty fruit bunches (EFB), and raw crude palm oil (CPO) were obtained from Besout palm oil mill, Perak, Malaysia. Palm oil tester reagents for free fatty acids (FFAs), peroxide value (PV), and deterioration of bleach ability index (DOBI) were purchased from LTC Scientifik, Malaysia. Analytical grade n-hexane was purchased from Merck, (Germany).

#### 2.2. Combined pretreatment with compressed water and steam for oil recovery

A high pressure water spray (HPWS) system was developed to recover the residual oil entrapped on the surface of EFB. This system consisted of two components; a hot water high pressure cleaner (HDS 12/18-4S, Karcher) and a collector vessel (Fig. 1). In fact, combination of hot water and steam was used at high pressure condition to leach out the residual oil from EFB. A few spikelets were taken from a bunch to analyze the initial oil content. The EFB was then sprayed by pressurized hot water/steam for 5 min, sample were taken for oil analysis every 20 s. Table 1 presents the operating parameters used during the process. Subsequently, the residual oil floating on the surface of the collector vessel was skimmed and collected. Centrifugation was used to remove the remaining impurities and water from the skimmed oil. Samples were stored at – 5°C until analyzed.

#### 2.3. Residual oil content

Residual oil content was determined using soxhlet extraction according to the Malaysian Palm Oil Board (MPOB) Test Method, (MPOB, 2004). Briefly, the EFB samples were dried in an oven for 24 h. About 10 g of the samples were then extracted with 150 mL of hexane solvent for at least 6 h on an electrochemical extraction unit. Then, hexane was evaporated using a rotary evaporator (RV10 Digital V, IKA. The oil content was determined using the following equation (Eq (1)):

\[
\text{Oil content (\%)} = \left( \frac{\text{Weight of oil}}{\text{weight of sample}} \right) \times 100
\]
Table 1
Qualities parameters of the residual oil extracted from oil palm EFB using the HPWS system at different temperatures.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Processing media</th>
<th>FFA (%)</th>
<th>PV (meq/kg)</th>
<th>DOBI</th>
<th>Phosphorus (ppm)</th>
<th>Iron (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>Water</td>
<td>6.21 ± 0.35</td>
<td>4.89 ± 0.27</td>
<td>2.42 ± 0.23</td>
<td>6.87 ± 0.71</td>
<td>3.43 ± 0.35</td>
</tr>
<tr>
<td>60</td>
<td>Water</td>
<td>6.57 ± 0.30</td>
<td>5.20 ± 0.35</td>
<td>2.28 ± 0.20</td>
<td>7.00 ± 1.14</td>
<td>3.63 ± 0.15</td>
</tr>
<tr>
<td>90</td>
<td>Water</td>
<td>6.88 ± 0.30</td>
<td>5.42 ± 0.42</td>
<td>2.21 ± 0.30</td>
<td>7.37 ± 0.12</td>
<td>4.97 ± 0.15</td>
</tr>
<tr>
<td>120</td>
<td>Water-steam</td>
<td>7.41 ± 0.58</td>
<td>5.60 ± 0.52</td>
<td>1.90 ± 0.10</td>
<td>10.17 ± 0.06</td>
<td>5.37 ± 0.21</td>
</tr>
<tr>
<td>150</td>
<td>Water-steam</td>
<td>7.79 ± 0.77</td>
<td>5.85 ± 0.60</td>
<td>1.63 ± 0.38</td>
<td>11.70 ± 0.61</td>
<td>7.17 ± 0.15</td>
</tr>
</tbody>
</table>

2.4. Residual oil characterization

The oil samples were analyzed according to the MPOB Test Method, (MPOB, 2004) for deterioration bleachability index (DOBI), free fatty acid (FFA), peroxide value (PV), phosphorus content, iron content, total carotene content, and fatty acid compositions. A palm oil tester (Plus version-CDR, Italy) was used to determine the DOBI, FFA, PV, and carotene content. The phosphorus content was determined using a spectrophotometer and the iron content was measured using an atomic absorption spectrophotometer (AAS).

2.4.1. Fatty acid profiling

Fatty acid (FA) profiles were obtained using a gas chromatograph equipped with a flame ionization detector (FID) detector (GC-FID, Agilent Technologies, Palo Alto, CA, USA). Fatty acid separation was carried out with a capillary column BPX 70: 30 m L x 0.25 μm x 0.32 μm ID (equivalent column to stable wax-Crossbond Carbowax PEG, Agilent). The temperature program used was as follows; oven temperature of: 100 °C, initial temperature of: 100 °C, final temperature of: 230 °C, injector temperature of: 250 °C, and detector temperature of: 250 °C. The carrier gas used was helium and the split ratio was 1:20.

2.4.2. Fourier transforms infrared spectroscopy (FTIR)

A Fourier transform infrared spectrometer (FTIR-Perkin Elmer, USA) was used to identify and compare the variations in functional groups of the CPO and the residual oil after the treatment by the HPWS system.

2.4.3. Turbidity measurement

Turbidity analysis was conducted using a turbidity meter (Eutech, brand model TN 100) to determine the presence of impurities in the residual oil after the treatment by the HPWS system. All the measurements were carried out in triplicate.

2.5. Estimation of biodiesel properties based on FA profiles

To estimate biodiesel properties, the FA profiles obtained by gas chromatography (GC) were analyzed by using BiodieselAnalyzer ver. 2.2 software (available on http://www.brteam.ir/biodieselanalyzer) (Talebi et al., 2014). More specifically, the physiochemical characteristics of the prospective biodiesel such as, Saponification Value (SV), Iodine Value (IV), Cetane number (CN), Degree of Unsaturation (DU), Long Chain Saturated Factor (LCSF), Cold Filter Plugging Point (CFPP), Cloud Point (CP), Pour Point (PP), Allylic Position Equivalent (APE), Bis- Allylic Position Equivalent (BAPE), Oxidation Stability (OS), Higher Heating Value (HHV), kinematic viscosity (υ) and Density (ρ) were calculated based on the FAME profile recorded for oil sample extracted using pressurized hot water and steam at 150 °C.

2.6. EFB characterization

2.6.1. Lignocellulosic composition

EFB fiber sample was dried and ground into small particles of 1 mm in size prior to the analysis. Acid detergent fiber (ADF), neutral detergent fiber (NDF), and acid detergent lignin (ADL) were analyzed using a Fibertec™ 2010, Auto Fiber Analysis System (Foss Analytical, Denmark) (Goering and Van Soest, 1970). The percentage of cellulose, hemicellulose and lignin were then calculated using the following equations (Eqs (2)–(4)):

Cellulose (%) = ADF – ADL

Hemicellulose (%) = NDF – ADF

Lignin (%) = ADF

2.6.2. Scanning electron microscopy (SEM)

The EFB fibers were analyzed by a Scanning Electron Microscope (SEM-S-3400N, Hitachi, Japan) after being treated by the HPWS system. The samples were coated with gold before the morphological structures were analyzed by SEM at 300× and 1000× magnifications.

2.6.3. Nitrogen adsorption analysis (BET)

Surface area, pore size and volume of the EFB fibers were characterised after the HPWS treatment using an Autosorb-1 instrument (Quantomehre, USA). The surface area was calculated by the Brunauer, Emmett, and Teller (BET) equation using the nitrogen adsorption data while the porosity data (pore size and volume) were calculated based on the Dollimore Heal (DH) (Dollimore and
2.6.4. Residual oil localization

Lipophilic Sudan red dye was prepared according to Brundrett et al. (1991) to observe the oil attachment on the surface of the EFB fibers. Samples collected after the HPWS processes were immersed in a few drops of Sudan red dye solution. The samples were then dried and investigated under a fluorescent light. The presence of the residual oil was indicated by red colored spots.

2.7. Life cycle assessment (LCA)

Life Cycle Assessment (LCA) is a scientific approach behind modern environmental policies and business decision support with the aim of sustainable development. More specifically, LCA is a structured, comprehensive, and internationally standardized method which quantifies all relevant emissions and resources consumed in the life cycle of a given product, activity, or service (ILCD, 2010; Rajaieifar et al., 2016). Moreover, LCA addresses the related environmental and health impacts and resource depletion issues associated with the examined system (Guinée, 2001).

Every LCA study follows a standard procedure which includes four main steps i.e., 1) goal and scope definition, 2) inventory analysis, 3) impact assessment, and 4) interpretation (Rajaieifar et al., 2015a). The goal of the present LCA study was to evaluate the environmental impacts of applying the HPWS system (for recovery of residual oil from EFB) and to compare this system with the other possible EFB recycling alternatives, i.e., composting and solid biofuel production (fuel briquette). Fig. 2 shows the scope of the present study including the following scenarios:

Scenario A. Oil extraction from EFB using High Pressure Water Spray (HPWS) system.

Scenario B. Composting of EFB.

Scenario C. Briquette production from EFB (as a boiler fuel).

It is also worth quoting that the functional unit of the present study was the management of 1 ton of EFB (with the oil content of 3%). SimaPro software version 8.2.3 was used to perform the LCA study along with its associated database (professional).

In order to compile an inventory (the second step in a life cycle study), the background data sets for the production of input materials (e.g., chemicals, fossil fuels, and Malaysian electricity production) was adopted from the Ecoinvent version 3.3 database. The foreground data set for this assessment including using the HPWS system and biodiesel production were gathered and calculated through the course of the present study. Moreover, the foreground data set regarding bioethanol, compost, and briquette production was adopted from Chiew and Shimada (2013); Tan et al. (2010). It is also worth mentioning that the capital goods (including machinery and infrastructure) used in all technologies were not included in the scope of the study and only input materials flows, such as chemicals, fossil fuels, and electricity consumption were considered in the calculations.

In view of the lifecycle impact assessment (LCIA), IMPACT 2002+ was chosen as the LCIA method. This method has been frequently used in waste management studies (Rajaieifar et al., 2015b). Moreover, IMPACT 2002+ covers a wide range of impact categories (e.g., Carcinogens, Global warming, etc.) and damage categories (e.g., Human Health, Ecosystem Quality, Climate Change, and Resources) (Khoshnevisan et al., 2016). The areas of protection (impact and damage categories) and their units employed for this study are illustrated in Appendix A. (Jolliet et al., 2003).

2.8. Economic assessment

Aspen economic analyzer, linked to Aspen plus V7.2 (Aspen Technology Inc., Cambridge, Massachusetts, USA) was used for analyzing total operating and investment costs of the HPWS system. The average cost of feedstock (EFB) was considered at USD 1.5/ton. The plant was supposed to operate 8000 h/year. A presumptive industrial-scale feed rate of 30,000 kg EFB/h was considered. Therefore, based on the experimental data acquired during the course of this study the quantity of the steam and hot water required to complete the process was estimated at 32,000 kg/h. The main product of the considered plant based on the HPWS system was recovered CPO with the production rate of 900 kg recovered CPO/h. The estimation of the final cost of 1 kg of the recovered CPO was obtained according to total operating cost of the plant. Oil-free EFB fibers were considered as the by-product of the plant and were not included in the economic calculations.

3. Results and discussion

3.1. Bunch selection for residual oil removal using HPWS process

A total of 100 EFB were collected and were classified based on their size. Approximately 72.60% of the EFBs were medium in size, while about 17.33% and 10% bunches were large and small in size, respectively. It was also observed that old palm trees (i.e. > 10 years old) usually produced large EEFB. On the other hand, immature trees (i.e. < 3 years old) produce small bunches. This was in line with the report of Basiron (2005) in which they stated that the weight of the bunch increased with palm age. Large EFBs were selected for the residual oil recovery process using the HPWS system.

3.2. Residual oil recovery using the HPWS system

Fig. 3 shows the residual oil recovery expressed as the percentage of the oil extracted by subtracting the initial and final oil content of the spikelet sample obtained using the soxhlet oil extraction method. The results obtained showed that the removal efficiency was increased by increasing the contact time and temperature under the same pressure conditions, e.g., 500 psi. Based on the data presented in Fig. 3, the rate of oil removal from the EFB surface was highest during the first 20 s at all temperatures (i.e., 30, 60, 90, 120 and 150 °C). The highest (63.05 ± 0.02%) and lowest (14.24 ± 0.01%) oil removal rates achieved during the first 20 s were at 150 and 30 °C, respectively. The elevated temperature must have increased the oil solubility and diffusion while lowering the oil viscosity (Tunio et al., 2011). As suggested by Perez et al. (2011), this phenomenon could result in increments in oil yields.

Complete oil removal was achieved by the HPWS system (i.e., at 120 and 150 °C) during 120 s while a longer time period of 160 s was required for the hot water system, i.e., at 30, 60 and 90 °C temperature values. This could be explained by the high temperature of the steam coming into contact with the EFB surface which could easily liquefy the oil and grease and extract them effectively with the condensate remaining from the condensed steam.

The effect of the HPWS system treatment on residual oil removal from the EFB surface was also visualized by the sudan red dye analysis (Fig. 4). As shown, there exist obvious differences between the EFB surface before and after the treatment. As seen in Fig. 4e which represents the HPWS process at 150 °C approximately no red spot could be observed on the EFB surface suggesting that the residual oil was completely removed from the EFB surface.
Fig. 2. a: Process flow diagram High Pressure Water Spray (HPWS) system. b: process flow diagram for composting of EFB. c: Process flow diagram of EFB as fuel boiler.
3.3. Residual oil quality characterization

The characteristics of the residual oil removed from the EFB using HPWS system at different temperatures are tabulated in Table 2. The extracted oil from the EFB under different temperature conditions was a clear dark-orange in colour and was comparable with the CPO. The FFA content of the recovered oil (or in another word, oil degradation rate) increased by elevating the process temperature with the maximum FFA content of 7.79% observed at 150 °C. In fact, the rate of oil degradation reaction is greatly affected by temperature, moisture content, storage time, and initial free fatty acid concentration (Sambanthamurthi et al., 2000; Frank et al., 2011; Sampaio et al., 2011). Loh et al., (2006) and Huang and Chang (2010) reported that the FFA value of the extracted oil exceeded the standard value of 5% due to the high temperature used during the soxhlet extraction and supercritical carbon dioxide extraction method. It is worth quoting that high FFA contents leads to increased rate of crystallization of CPO and consequently reduce the quality of olein and stearin during refining (Ariffin, 2007).

The highest peroxide value (PV) of 5.85 meq/kg was also recorded for the oil recovered at 150 °C (Table 1). PV is used to evaluate the degree of oil oxidation or deterioration. Increasing PV value as a result of increasing treatment temperature has been reported previously as well (Chong, 1991; Abd Majid et al., 2012). The maximum recommended PV by the Food and Agriculture Organization of the United Nations or World Health Organization (FAO/WHO) for CPO is 10 meq/kg (Frank et al., 2011). CPOs possessing PV values above the standard are unsafe for human consumption as the free radicals generated are carcinogenic (Rosell, 1991). The PV values of all the residual oil recovered in the present study met the standard value.

Deterioration of bleachability index (DOBI) is another essential oil quality parameter. DOBI is the ratio of carotene (pro-Vitamin A) measured at 446 nm and secondary oxidation products (measured at 269 nm). When an oil deteriorates, carotenes breaks down to form secondary products leading to low DOBI value. DOBI is also regarded as an indicator showing the ease of bleaching and degumming process of CPO. The obtained DOBI values ranged from 1.63 to 2.42, with the maximum and minimum values recorded from the residual oil extracted at 150 and 30 °C, respectively (Table 1). In fact, the DOBI value decreased as the HPWS process temperature increased. The low DOBI value of 1.90 and 1.63 (which were lower than the minimum standard value) were observed at temperature of 120 °C and 150 °C indicate the occurrence of the oxidation reaction under these temperature. DOBI is of great importance when the quality of CPO is investigated. In line with that, Tan et al., (2009) proposed the CPO be classified accordingly (DOBI < 1) bad CPO, (DOBI: 1–2) poor CPO, (DOBI: 2–3) average CPO, and (DOBI >3) good CPO.

According to Gibon et al., (2007), the phosphorus contained in CPO is removed through the degumming and bleaching processes in refineries because it could lead to poor bleachability and increased susceptibility to oxidation. The phosphorus content of CPO is normally within the range of 10–20 ppm (Gibon et al., 2007). As presented in Table 1, the phosphorus content increased as the process temperature increased. This could be attributed to the fact that phospholipids are present in plant cells to maintain cell membrane integrity and play significant roles in membrane permeability, membrane fluidity, membrane interactions, and membrane deterioration (Bezrukov, 2000; White et al., 2001). In this study, as the temperature employed in the HPWS system increased, the amount of phospholipids released from the EFB cell and dissolved into oil also increased.

Similar trend was also observed for the iron content. According to the findings of Sambanthamurthi et al. (2000), phosphorus content in CPO is associated with iron and FFA contents causing oil instability and oxidative deterioration. The average standard iron content for CPO is 4.4 ppm (Sambanthamurthi et al., 2000), and iron in high quantities could act as pro-oxidant and induce the oxidation reaction of hydroperoxides into free radicals. During the processing by the HPWS system, iron impurities may be derived from wear and tear of steel container used and as a result of the high temperatures were applied. Increase of temperature could increase iron corrosion and could consequently increase the released iron intensity in the recovered CPO. These results were in agreement with those of Qi et al. (2014) who argued that increases in temperature led to increased corrosion rate and resulted in increased steel corrosion. Moreover, iron contamination in the CPO could also occur due to the mechanical wear and corrosion at the mill itself or during the storage and transportation stages (Gibon et al., 2007). Therefore, the use of stainless steel for certain mill machineries which are subjected to constant wear and tear is strongly recommended (Sambanthamurthi et al., 2000).

3.4. FTIR spectra analysis

FTIR (4000–600 cm⁻¹) was used to determine the probable variations in the physiochemical properties of the residual CPO after the HPWS process (Fig. 5). Table 3 presents the functional groups and their associated absorption frequencies for vegetable oil and can be used as a guideline to determine the specific functional groups contained in CPO as well. All the residual oil samples exhibited similar absorption peaks of comparable intensities at 2852 cm⁻¹ and 2922 cm⁻¹ caused by the symmetric and asymmetric stretching vibrations of aliphatic methylene (CH₂) group. The presence of these peaks indicated that the HPWS process did not change the amount of fatty acids aliphatic methylene groups in the residual oils. The peak observed for all oil samples at 1743 cm⁻¹ is attributed to the ester carbonyl functional group of triglycerides (Siddique et al., 2013). Moreover, the absorption peaks obtained at 1464 cm⁻¹ and 1377 cm⁻¹ are due to the bending vibrations of CH₂ and CH₃ aliphatic groups while the peak observed at 1159 cm⁻¹ could be attributed to the stretching vibration of the C–O ester groups. The last peak appearing at 721 cm⁻¹ is due to the overlapping of the CH₂ rocking vibration and the out of plane vibration of cis-disubstituted olefins. Based on the results obtained, it could be concluded that the residual oil recovered using the HPWS process at different temperature values was not chemically affected and retained its basic characteristics.

3.5. Turbidity measurement

The turbidity test was also performed to evaluate the cloudiness of the residual oil samples recovered through the HPWS process. In fact, the cloudy appearance in oil indicates the presence of impurities, e.g., saturated triglycerides, waxes, FFA, hydrocarbons, stearols, dirt or remaining particles dissolved in the oil (Fofana et al., 2008; Gammil and Proctor, 2009). Fresh CPO exhibited low turbidity value of 22.1 NTU while the lowest turbidity value among the residual oil samples was recorded at 31 NTU for the 30 °C treatment (Fig. 6). The results obtained revealed that the turbidity value increased as the process temperature increased. Nevertheless, since the standard turbidity value for palm oil ranges between 0.1 and 500 NTU (Bashi et al., 2006) therefore all the residual oil samples were in accordance with the standard.

3.6. Bioprospection of biodiesel properties based on FAME profile

The fatty acid methyl ester (FAME) profiles of the residual oil samples after the HPWS process were analyzed by gas
Oxygen and as the temperature increases, the rate of oxidation increases. According to Sambanthamurthi et al. (2000), carotenes are sensitive to light and as the process temperature increased. Moreover, the carotene concentration in the residual oil decreased. This could be explained by the poor presence of unsaturated fatty acids in the FAME products. These results were in agreement with those of Arora et al. (2006) where the major fatty acids were palmitic (43%), oleic (40%), and linoleic acid (12%) as well. Overall, the HPWS system at different temperature had no effects on the FAME profile of the residual oils. These results were in agreement with those of the FTIR analysis (Fig. 5).

Considering the low quality of the residual CPO as edible oil, as non-edible oil feedstock for biodiesel production seems promising. It is well documented that biodiesel properties are directly influenced by the FAME profile of the oil feedstock used (Talebi et al., 2013, 2014). Therefore, since the FAME profile of the residual oil recovered through the HPWS process was comparable with that of the fresh CPO, therefore, this waste oil feedstock could serve as a sustainable non-edible alternative of CPO in the biodiesel refineries. The results presented in Table 3 show the estimated properties of biodiesel obtained from the fresh and residual oil could meet the EN 14214 international biodiesel fuel standards. More specifically, the calculated cetane number (CN) for the residual oil obtained at 150 °C was at 60.55, which was comparable with that of CPO (i.e., 60.48). It is worth mentioning that CN is an important parameter attributed to the ignition delay time and combustion quality. Higher CN values are accompanied with better cold start, shorter ignition time, and less formation of white smoke (Talebi et al., 2014). Ramos et al. (2009) also reported a similar CN value of 61 for palm oil biodiesel. The oxidative stability of the studied residual oil biodiesel (i.e., 13.04 h) was found twice higher than the minimum standard value of 6 h. This could be explained by the poor presence of unsaturated fatty acids in the FAME profile (Arora et al., 2006). Moreover, the high oxidation stability (OS) of the residual oil could also be attributed to the carotene content of the residual oil (Table 2). Nevertheless, it is worth quoting that the carotene content of the residual oils was significantly lower than what normally found in fresh CPO (500–700 ppm) (Gee, 2007). Moreover, the carotene concentration in the residual oil decreased as the process temperature increased. According to Sambanthamurthi et al. (2000), carotenes are sensitive to light and oxygen and as the temperature increases, the rate of oxidation reaction and consequently the degradation rate of these compounds increases as well. On the other hand, the high content of saturated fatty acids in both fresh and residual oil (approx. 50%) could result in unfavorable cold flow properties. For instance, the estimated pour point (PP) of the residual oil and the fresh CPO stood at 10.38 and 11.55 °C, respectively, which are around 25 °C higher than that of the petroleum diesel. These results were aligned with those previously reported on palm biodiesel by (Ramos et al. (2009) and; Altun (2014). To improve the cold flow properties, blending different oil feedstock found could be taken into consideration (Altuna et al., 2010; Griffiths et al., 2012).

3.7. Variations in lignocellulosic composition and structure of EFB fiber after HPWS

3.7.1. Lignocellulosic composition

Lignocellulosic compositions of the EFB fibers before and after HPWS treatment under different temperatures are shown in Fig. 7. Accordingly, cellulose proportion was increased by 42% by using the HPWS process at 150 °C in comparison with its proportion in raw EFB fibers. The increases observed in the cellulose content throughout the process was ascribed to the high temperature and pressure applied. More specifically, the sever conditions caused the β-glycosidic bonds between D-glucosyl groups of cellulose to rupture, resulting in the breakage of cellulose. In better words, higher levels of solubilization were achieved as the pretreatment conditions were more drastic, so the cellulose in the treated biomass increased (Adaganti et al., 2013). On the other hand, Thomsen et al. (2006) argues that, increases in cellulose content have adverse effects on fibers’ strength. This statement was also confirmed by Kocaefe et al., (2008) who also reported that wood hardness was decreased when cellulose content increased by using high temperature conditions. Fig. 8 shows the morphological changes caused by the HPWS treatment.

The hemicellulose content decreased by increasing the process due to the degradation of hemicellulose (Fig. 7). Iroba et al., (2014) suggested that the degradation of hemicellulose by high temperature values is ascribed to its amorphous structure which can be easily damaged. The lignin content was also decreased by approximately 34% by using the HPWS system at 150 °C compared to the lignin content of the raw EFB. Lignin removal could exert a positive impact resulting in further exposure of the cellulose proportion of...
3.7.2. Lignocellulosic structure

3.7.2.1. Scanning electron microscopy (SEM) analysis. SEM was used to investigate the surface morphology changes of the EFB fibers before and after the HPWS process (Fig. 9). As shown, noticeable differences could be observed on the fibers surface after the HPWS treatment. More specifically, the raw EFB surface before the HPWS process had a rough, rigid and uneven surface (Fig. 9a). Shamsudin et al. (2012), suggested that the rough surface of raw EFB was due to the presence of a protective matrix layer like lignin or waxes coating the fiber surface to prevent water loss. In addition, the presence of the remaining oil coated on the surface of the EFB fibers could also be detected (Fig. 9aii). In fact, the rough surface of the EFB fibers is the reason behind retaining oil on the fibers.

As presented in Fig. 9, the application of the HPWS process led to significant changes of the fiber morphologies while the content of the oil attached to the fibers decreased as well. More specifically, by increasing the process temperature, the surface of the fibers slowly became increasingly more smooth and uniform compared to the raw EFB fibers. By using the HPWS process at 90 °C, the surface...
became completely clean and uniform indicating the removal of the protective layer coated on the surface (Fig. 9d). Norul Izani et al. (2013) reported that the elimination of the protective layer further increased the surface area of the fibers leading to more exposure to the applied treatments.

Table 2
Composition of the residual oil extracted through the HPWS process at different temperature treatments.

<table>
<thead>
<tr>
<th>Characteristics (%)</th>
<th>Temperature (°C)</th>
<th>30</th>
<th>60</th>
<th>90</th>
<th>120</th>
<th>150</th>
<th>CPO&lt;sup&gt;a&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fatty acids</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Saturated</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lauric (12:0)</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>0.00–1.00</td>
</tr>
<tr>
<td>Myristic (14:0)</td>
<td>0.91</td>
<td>0.91</td>
<td>0.92</td>
<td>0.91</td>
<td>0.92</td>
<td>0.92</td>
<td>0.90–1.50</td>
</tr>
<tr>
<td>Palmitic (16:0)</td>
<td>39.50</td>
<td>40.32</td>
<td>40.22</td>
<td>40.31</td>
<td>39.56</td>
<td>39.20</td>
<td>39.20–45.80</td>
</tr>
<tr>
<td>Stearic (18:0)</td>
<td>4.58</td>
<td>4.51</td>
<td>4.55</td>
<td>4.26</td>
<td>4.14</td>
<td>3.70</td>
<td>3.70–5.10</td>
</tr>
<tr>
<td>Arachidic (20:0)</td>
<td>0.44</td>
<td>0.43</td>
<td>0.43</td>
<td>0.44</td>
<td>0.43</td>
<td>0.43</td>
<td>0.00–0.40</td>
</tr>
<tr>
<td>Behenic (24:0)</td>
<td>4.05</td>
<td>4.04</td>
<td>4.04</td>
<td>4.06</td>
<td>4.05</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>Unsaturated</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oleic (18:1)</td>
<td>39.86</td>
<td>39.20</td>
<td>39.12</td>
<td>39.64</td>
<td>39.60</td>
<td>39.60</td>
<td>37.40–44.10</td>
</tr>
<tr>
<td>Linoleic (18:2)</td>
<td>10.35</td>
<td>10.27</td>
<td>10.30</td>
<td>10.08</td>
<td>10.97</td>
<td>10.97</td>
<td>8.70–12.50</td>
</tr>
<tr>
<td>Linolenic (18:3)</td>
<td>0.31</td>
<td>0.31</td>
<td>0.32</td>
<td>0.31</td>
<td>0.32</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>Carotene content (ppm)</td>
<td>439.10–561.80</td>
<td>408.60–421.90</td>
<td>351.070–408.20</td>
<td>312.40–324.90</td>
<td>295.70–379.40</td>
<td>NA</td>
<td></td>
</tr>
</tbody>
</table>

ND: not detected; NA: not available.
<sup>a</sup> Source: Sambanthamurthi et al. (2000).

Fig. 5. FTIR spectra of the fresh CPO and the residual oil (RO) recovered through the HPWS process at different temperatures.

3.7.2.2. Nitrogen adsorption analysis (BET). In this study, the effects of the HPWS process on the surface area, pore volume and average pore diameter of the EFB fibers were also determined by the Brunauer-Emmett-Teller (BET) analysis. Accordingly, it was found that the surface area of the EFB fibers was increased from 0.05 m²/g...
(raw EFB) to 1.91, 0.95, 1.08, 1.63, and 1.91 m²/g once the EFB was treated using the HPWS system at 30, 60, 90, 120, and 150 °C, respectively. The significant increases achieved in the surface area was possibly due to the elimination of oil and impurities attached on the surface the EFB fibers. This result was in agreement with the microscopic observation (SEM) where less oil droplets were observed and the EFB fibers surface was found more smooth and uniform when higher temperatures were applied. Ma et al., (2014) also reported an increased surface area of bamboo activated carbon fiber when the treatment temperature was increased from 650 °C to 850 °C. However, the surface area of raw EFB reported in a study conducted by Hidayu et al., (2013) was higher (2.01 m²/g) in comparison with that of the present study. Such differences could be ascribed to the different species of oil palm used that could affect the results obtained (Hazir et al., 2012). It is worth mentioning that increasing the surface area of EFB would be advantageous for bioconversion purposes as it leads to increase rate of chemical reactions (Sukiran et al., 2011).

The pore volume of the EFB fibers was also increased from 0.02 cm³/g (raw EFB), to 0.05 cm³/g (150 °C) (Table 5). Rong et al., (2003) also claimed that by increasing the treatment temperature from 449.85 °C to 949.85 °C, the surface area and total pore volume of the rayon-based activated carbon fibers increased. The increment in pore volume as higher temperature values were applied was ascribed to the impact of heat in promoting the generation of microspores and efficiently inhibiting the growth of graphic structures (Chiang and Chen, 2010). According to the findings of Han and Choi (2010), increases in pore volume led to reduce fiber strength.
which could consequently affect a number of fiber properties such as, increased swelling in water and fiber shrinkage as well as increased accessibility of fibers to chemical reactants like cellulase.

The average pore diameter of the EFB fibers was gradually decreased by increasing the HPWS process temperature from 30°C to 150°C. In better words, untreated or raw EFB fiber had larger pore diameters of 24,640.18Å in comparison with the treated EFB fibers i.e., 11,084.87Å (30°C), 1461.14Å (60°C), 1274.42Å (90°C), 902.89Å (120°C), and 496.44Å (150°C). This finding was in line with those of Zennen et al., (2014) who also reported that the average pore diameter of the fibers decreased when the temperature was increased up to 120°C.

3.8. Lifecycle assessment: results and interpretation

Based on the defined goal of the present study, the environmental impacts of using the HPWS system (for recovery of residual oil from EFB) were assessed and compared with the other possible EFB recycling alternatives (composting or Briquette production). For this purpose, the best treatment of the HPWS system (oil removal at 150°C and 500 PSI) was selected and the assessment was performed based on the management of 1 tone of EFB. The results in the level of mid-point impact categories were tabulated in Table 6.

Based on the mid-point level results, it should be highlighted...
that scenario A (HPWS system) and B (Composting) had favorable impacts in all the impact categories while the scenario C (Briquette production) had negative impacts on the environment in all the mid-point impact categories. In view of the carcinogens mid-point impact category, using the HPWS system led to a better environmental performance (the highest reduction potential) compared with composting and briquette production. In fact, the avoided product as a result of bio-ethanol production (when implementing scenario A) was the main reason behind this reduction potential. More specifically, production of 1 kg of bio-

![SEM micrograph](image_url)
ethanol could avoid using 0.67 kg gasoline (Chiew and Shimada, 2013) and consequently reduce the emissions during the production of this fossil fuel. The situation was somehow the same when analyzing the ionizing radiation and non-renewable energy mid-point impact categories in which the avoided product as a result of bio-ethanol production alongside the avoided product through biodiesel production led to a higher reduction potential for scenario A in these mid-point impact categories.

When analyzing the other impact categories, i.e., non-carcinogens, respiratory inorganics, ozone layer depletion, respiratory organics, aquatic ecotoxicity, terrestrial ecotoxicity, terrestrial acid/nutrient, land occupation, aquatic acidification, aquatic eutrophication, global warming, and mineral extraction mid-point impact categories, composting of EFB showed a better environmental performance (the highest reduction potential) among the scenarios investigated. This was due to the nutrients content of the EFB compost (i.e., 2.2% Nitrogen, 1.28% Phosphor, and 2.79% Potassium (K)) which could avoid the usage of Nitrogen, Phosphorus and Potassium fertilizers of fossil origins (Chiew and Shimada, 2013). In another word, each ton of EFB would results in about 385 kg of EFB compost and consequently would avoid the production and usage of about 18, 10, and 18 kg of Nitrogen, Phosphorus, and Potassium fertilizers of fossil origins, respectively (assuming that chemical nitrogen fertilizer contains 46% of N, phosphorus, and potassium fertilizers of fossil origins, respectively) (Chiew and Shimada, 2013). When analyzing the other mid-point impact categories among the examined scenarios (Fig 10), composting of EFB provided a significant benefit in the human health, ecosystem quality and climate change damage categories compared with the other scenarios. Similar to the interpretation used for mid-point impact categories, the avoided products of composting were the main reason behind these high performances. Nevertheless, the application of the HPWS system for recovery of the residual oil from EFB provided a significantly better environmental performance among the other scenarios when analyzing the resources damage categories. This was due to the fact that biodiesel and bioethanol (which are the main products of the HPWS system) have high potentials in substituting fossil-oriented diesel and gasoline, respectively, and consequently in reducing the negative environmental impacts caused by exploiting energy from fossil resources (Rajaefar et al., 2013).

Chiew and Shimada (2013) investigated the environmental impacts of utilizing EFB for fuel (only ethanol production or briquette or methane), fiber and fertilizer (EFB compost) production and reported that methane recovery and composting were more environmentally-friendly than the other studied technologies. However, they did not analyze all the mid-point impact categories included in the Impact 2002+ due to the different LCIA method used. More specifically, they did not include the carcinogens, respiratory organics, respiratory inorganics, ionizing radiation, mineral extraction, and non-renewable energy mid-point impact categories. On the contrary, the present study take into account all the mentioned mid-point impact categories. Moreover, EFB utilization for simultaneous production of both biodiesel and bio-ethanol using the HPWS system was investigated herein. This would offer a better index in carcinogens, ionizing radiation, and non-renewable energy mid-point impact categories as well as resources damage categories (as end-point impact category) in comparison with conventional scenarios, i.e., composting and briquette production. It is also worth quoting that, since there is lots of extra steam in palm oil extraction plants, the steam needed in the HPWS system could be supplied without posing any extra environmental and economic burdens. This further marked the HPWS system as an environmentally friendly approach for biofuel production from EFB. It is worth quoting that a number of previous studies argued that biofuel production from EFB was not environmentally-favorable (Chiew and Shimada, 2013; Lim and Lee, 2011). This could be attributed to the fact that oil extraction in those studies was not accomplished using the extra steam available at literally no cost at the plants.

As both using the HPWS system for recovery of residual oil from EFB (Scenario A) and composting of EFB (Scenario B) provided significant environmental benefits; therefore, both could be of interest from the environmental perspective. Nevertheless, if reduction in exploiting fossil resources is targeted, the Scenario A would be preferred. This could particularly be the case when the weight of resources damage category index in decision making or policy making is considered higher than those of the other damage categories.

### Table 5
The results of BET analysis on oil palm EFB fibers.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Surface area (m²/g)</th>
<th>Total pore volume (cm³/g)</th>
<th>Average pore diameter (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw OPEFB</td>
<td>0.05</td>
<td>0.02</td>
<td>24.640.18</td>
</tr>
<tr>
<td>30</td>
<td>0.19</td>
<td>0.03</td>
<td>11.084.87</td>
</tr>
<tr>
<td>60</td>
<td>0.95</td>
<td>0.03</td>
<td>1461.14</td>
</tr>
<tr>
<td>90</td>
<td>1.08</td>
<td>0.03</td>
<td>1274.42</td>
</tr>
<tr>
<td>120</td>
<td>1.63</td>
<td>0.04</td>
<td>902.89</td>
</tr>
<tr>
<td>150</td>
<td>1.91</td>
<td>0.05</td>
<td>496.44</td>
</tr>
</tbody>
</table>

### Table 6
The results of mid-point impact category analysis.

<table>
<thead>
<tr>
<th>Impact category</th>
<th>Unit</th>
<th>Scenarios</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>A</td>
</tr>
<tr>
<td>Carcinogens</td>
<td>kg C2H3Cl</td>
<td>-10.37</td>
</tr>
<tr>
<td>Non-carcinogens</td>
<td>kg C2H3Cl</td>
<td>-0.46</td>
</tr>
<tr>
<td>Respiratory inorganics</td>
<td>kg PM2.5</td>
<td>-0.02</td>
</tr>
<tr>
<td>Ionizing radiation</td>
<td>Bq C-14</td>
<td>-1720.52</td>
</tr>
<tr>
<td>Ozone layer depletion</td>
<td>kg CFC-11</td>
<td>-2.11E-05</td>
</tr>
<tr>
<td>Respiratory organics</td>
<td>kg C2H4</td>
<td>-0.16</td>
</tr>
<tr>
<td>Aquatic ecotoxicity</td>
<td>kg TEG</td>
<td>-4481.37</td>
</tr>
<tr>
<td>Terrestrial ecotoxicity</td>
<td>kg TEG</td>
<td>-544.08</td>
</tr>
<tr>
<td>Terrestrial acid/nutri</td>
<td>kg SO2</td>
<td>-1.22</td>
</tr>
<tr>
<td>Land occupation</td>
<td>m2org.arable</td>
<td>-0.13</td>
</tr>
<tr>
<td>Aquatic acidification</td>
<td>kg SO2</td>
<td>-0.31</td>
</tr>
<tr>
<td>Aquatic eutrophication</td>
<td>kg PO4</td>
<td>-2.19E-03</td>
</tr>
<tr>
<td>Global warming</td>
<td>kg CO2</td>
<td>-48.60</td>
</tr>
<tr>
<td>Non-renewable energy</td>
<td>MJ primary</td>
<td>-4693.44</td>
</tr>
<tr>
<td>Mineral extraction</td>
<td>MJ surplus</td>
<td>-0.07</td>
</tr>
</tbody>
</table>

3.9. Economic assessment of the HPWS system

The economic assessment results for the plant equipped with the HPWS system capable of processing 30 tons EFB/h or in another word producing 7200 tons of recovered CPO/year are summarized in Table 7. Taking into account an investment return period of 4 years, the estimated price of main product, i.e., the recovered CPO stood at USD 0.41. The reasonably low price estimation could be attributed to the inexpensive feedstock and the low cost of the utilities used.
Table 8 shows detailed information on the costs of the process equipment and engineering. Among the main equipment, the horizontal drum (used in the HPWS system) had the highest capital cost due to the longer residence time of the feedstock in the unit and consequently higher construction costs. Moreover, this equipment was designed and fabricated to withstand high pressure conditions and therefore, more materials were used. The second block was an oil-water separator for the separation of the recovered CPO from water, sludge, and fibers. The final component was a cartridge filter for removing sludge and water. The recycled water could then be reused. Stainless steel 316 was considered as the main material for constructing these three operating units. The summary of the engineering costs contributing approximately 20% of the total capital cost are also presented in Table 2. Other data on labor and maintenance costs are provided in Table 9. As presented, the maintenance cost was negligible compared with the labor and supervision costs. The total operating labor and maintenance cost presented in Table 7 is in fact the sum of labor, maintenance, and supervision costs.

### 4. Conclusions

The proposed method, i.e., the HPWS system was found promising for residual oil recovery from oil palm EFBs since it led an oil removal rate of 94.41 ± 0.02% at 150 °C. Moreover, the estimated
quality parameters of the resultant waste-oriented biodiesel were in accordance with the international standards. Hence, the recovered oil could serve as a sustainable alternative for edible CPO in biodiesel refineries. In addition to that, the proposed process significantly altered the composition and structure of the remaining lignocellulosic materials leading to higher cellulose content, less lignin content, and higher surface area. Therefore, the resultant lignocellulosic wastes obtained using the excess steam available in mills could be regarded more promising conversion into other value-added products such as ligno-ethanol, bio-sugar, bio-compost, and bio-char. In the view of environmental impact assessment, the HPWS system showed favorable impacts on all the end-point damage categories especially in resources damage category. Moreover, economic assessment showed that the recovered CPO could be generated at a low price of USD 0.41 vs. USD 0.66 for CPO reported in September 2016 (MPOB, 2016).

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jclepro.2016.10.078.

References


