Effect of Torrefaction Conditions on Physicochemical Properties of Empty Fruit Bunches

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\textbf{Abstract.} Empty fruit bunches (EFB) in current bulky form are low in energy density and highly moist making them unsuitable as a solid fuel. Pre-treatment of EFB via thermochemical process i.e. torrefaction is required in order to improve its energy density via reduction of moisture and oxygen contents, which eventually will lead to an increased calorific value (CV). This study investigated the effects of temperature (225 °C, 250 °C and 300 °C) and retention time (20, 40 and 60 min) on the torrefaction products distribution (solid, liquid and gas) and physicochemical properties including proximate and ultimate (elemental) compositions and energy content of torrefied EFB. The results indicated that an increased temperature and retention time led to lower mass of torrefied EFB yield. The highest yield (90.44\%) was attainable after 20 min of torrefaction at the lowest deployed 225 °C. However, the CV of torrefied EFB increased with increasing temperature and retention time, reaching a maximum 25.73 MJ/kg at 300 °C for 60 min, i.e. up to 46% higher than its raw form. The resulting improved physicochemical properties indicated suitability of the torrefied EFB for bioenergy processes such as combustion, gasification, pyrolysis and pelletization.

1. Introduction

The energy consumption in Malaysia is mainly based on fossil fuels such as natural gas, coal and petroleum. Over dependence on fossil fuels for supply of primary energy source has led to global energy crisis, including an increased greenhouse gas emissions and a declined fossil fuels reserves [1]. Thus, renewable energy resources from biomass, solar, wind and hydro are potential substitutes for fossil fuels. Among these, biomass is one of the most potential resources due to its carbon neutrality and abundant availability [2]. As the second palm oil producer in the world, Malaysia produces huge amount of oil palm biomass every year mainly from oil palm plantations (i.e. oil palm frond and oil palm trunk) and milling activities i.e. empty fruit bunches (EFB), mesocarp fiber and palm kernel shell. Of these, EFB is the most abundantly generated in palm oil mills after palm fruits are separated from bunches in a rotating drum during palm oil processing [3]. Based on 17.16 t h\textsuperscript{-1} of fresh fruit bunches processed in 2018 [4], approximately 19.59 million t (wet weight) of EFB were generated. Based on these numbers and considering the undesirable environmental impact created, many researches have been devoted to investigate the possibilities of using EFB to produce high-end products such as biofuel, pulp, compost and fine chemicals. Recently, converting EFB into biofuel is among the popular research areas that has received a lot of attention [5].
However, there are some drawbacks from using raw EFB as a biofuel feedstock, such as high moisture and oxygen contents, low energy density, hygroscopic nature and low calorific (CV) value, which are responsible for the low combustion efficiency and also difficulty faced in using it as a direct fuel to achieve efficient utilization [6]. Thus, pretreatment is needed as means to improve fuel properties of EFB, and the best option is torrefaction which is currently widely accepted as a biomass pretreatment method. Torrefaction is a thermochemical process where biomass is subjected to thermal heating in the absence of air or oxygen, typically at temperatures in the range of 200 ºC to 300 ºC at atmospheric pressure [7]. This process is expected to improve the EFB fuel properties and reduce some of the problems as mentioned above. It will also increase the energy density of EFB by reducing moisture and oxygen contents, which will lead to an improved CV of the torrefied EFB [8]. After torrefaction process, oxygen and hydrogen contents of biomass will reduce, while carbon content will increase. It is anticipated that the produced final product will has better grindability, higher hydrophobicity and CV compared to the original raw biomass.

Torrefaction is influenced by many operating parameters, including temperature, residence time, particle size, type of biomass and moisture content [9]. Of these, temperature has the greatest influence on torrefaction process. The degree of thermal decomposition of biomass depends primarily on temperature. In torrefaction process, the mass yield decreases with increasing temperature [3]. Retention time is another important parameter that affects the torrefaction product. Nevertheless, time required was less significant than temperature in torrefaction process. Longer retention time provides lower mass yield, as well as energy yield.

The key objective of this study is to investigate the effects of temperature (225 ºC, 250 ºC and 300 ºC) and retention time (20, 40 and 60 min) on torrefaction products distribution (solid, liquid and gas) using a fixed bed reactor. In addition, the characteristics of torrefied EFB such as CV, proximate and ultimate compositions were determined. The energy yield of torrefied EFB was also calculated to provide some indication on suitability of these materials for further bioenergy processes such as combustion, gasification and pelletization.

2. Materials and methods

2.1. Sample preparation

EFB was collected from a palm oil mill located in Negeri Sembilan, Malaysia. It was milled and sieved using a test sieve shaker (Endecotts EFL 2000) and then separated into several different fractions consisting of particle sizes ranging from 106 – 250 µm. The resulting particles were dried at 103 ºC for 24 h until constant weight was achieved. Table 1 shows the properties of raw EFB used in this study.

| Table 1. Properties of raw empty fruit bunches. |    |
|-----------------------------------------------|----|
| **Characteristic**                           | **Value** |
| Proximate analysis (wt.%)                    |    |
| Volatile matter                             | 77.42 |
| Fixed carbon                                 | 13.84 |
| Ash                                          | 4.19 |
| Moisture                                     | 4.55 |
| Ultimate analysis (wt.%)                     |    |
| Carbon                                       | 42.82 |
| Hydrogen                                     | 6.07 |
| Nitrogen                                     | 0.54 |
| Oxygena                                      | 50.57 |
| Calorific value (MJ/kg)                      | 17.57 |

*a by difference*
The moisture content, ash content and fixed carbon of the raw EFB were 4.55 wt.%, 4.19 wt.% and 13.84 wt.% respectively. The oxygen content in the raw EFB was high (50.57 wt.%) while the carbon content was 42.82 wt.%. The CV of the raw EFB was determined to be 17.57 MJ/kg.

2.2. Torrefaction experiment
Torrefaction of EFB was carried out using a fixed bed reactor under a nitrogen atmosphere at three different temperatures (225 °C, 250 °C and 300 °C) and three different retention times (20, 40 and 60 min) (Figure 1). Approximately 10 g of EFB with particle size of 106 – 250 µm was used for each experiment. A reactor was placed in the furnace and was continuously purged with nitrogen at a rate of 1 L min⁻¹ to remove oxygen and maintain an inert atmosphere throughout the process. The EFB samples were heated up using an electric furnace at a heating rate of 10 °C min⁻¹ until desired temperature was achieved. The temperature inside the reactor was monitored using a thermocouple. A condenser was installed to condense the vapors released from the process. After the process was finished, the solid remaining inside the reactor was collected and then weighed, while the liquid product from the condensation unit was collected and weighed too to obtain the mass of both the torrefied yields. The final weight percentages of the torrefied solid and liquid yields were calculated using the following equation:

\[
\text{Mass yield (\%) = } \frac{\text{Mass of torrefied EFB/liquid product}}{\text{Mass of raw EFB}} \times 100\% \quad (1)
\]

The yield of the gas was calculated by subtracting the weight percentages of solid and liquid torrefied yields from the total of 100%.

![Figure 1. Schematic diagram of experimental setup for torrefaction of oil palm biomass. (1: Valve, 2: Flow meter, 3: Furnace, 4: Thermocouple, 5: Reactor, 6: Sample feeding, 7: Temperature controller, 8: Heating tape, 9: Condenser, 10: Gas exit)](image-url)

2.3. Characterisation of raw and torrefied empty fruit bunches
The physiochemical properties of the raw and torrefied EFB such as moisture content, volatile matter, fixed carbon and ash content were determined via proximate analysis using a thermogravimetric analyzer (TGA) (LECO TGA-701). The carbon, hydrogen and nitrogen contents of the raw and torrefied EFB were performed using a CHNS analyzer (LECO CHNS-628). The oxygen content was calculated from the difference between 100% and the total percentage of carbon, hydrogen and nitrogen. The CV of the raw and torrefied EFB were determined using an isoperobolic system bomb
calorimeter (LECO AC-600). Meanwhile, degradation of the raw EFB was investigated using TGA (Perkin Elmer-Pyris 6). The analysis was performed as follows: sample mass of approximately 10 mg, linear heating rate of 20 °C min⁻¹ within 30 °C to 900 °C and a flow of nitrogen at a rate of 0.1 L min⁻¹. The respective degradation was determined from the thermogravimetric curves. Based on the obtained mass yield and CV of torrefied product, energy yield (%) was calculated as follows:

\[
\text{Energy yield} = \frac{\text{Mass yield of torrefied EFB} \times \left( \frac{\text{Calorific value of torrefied EFB}}{\text{Calorific value of raw EFB}} \right)}{100}
\] (2)

3. Results and discussion
3.1. Thermogravimetric analysis of raw empty fruit bunches
The thermal degradation behavior of EFB during torrefaction process was represented as in thermograph (Figure 2). The curve shows that thermal degradation trend of EFB is similar with others lignocellulosic biomass such as rice straw [10] and olive tree pruning [11] when heated up to 900 °C. The initial decrease in the thermogravimetric curve was due to moisture release, after which thermal degradation occurred via two steps [12]. The main loss was observed during the second stage, at about 300 °C. At this stage, the cellulose and hemicellulose in the sample depolymerized [13]. Then the curve was characterized by a continuous slight devolatilization zone where lignin decomposition and char formation took place.

![Figure 2. Thermogravimetric analysis thermograph of empty fruit bunches.](image)

3.2. Products distribution
Figure 3 shows EFB’s torrefaction products distribution at different temperatures and retention times. As shown, the torrefaction of EFB produced three products; namely solid (torrefied product), liquid and gas. The yields of torrefied product decreased with increasing torrefaction temperature, ranged between 38.0 - 90.4%. The reduction in mass yield of torrefied product at 225 °C was found to be negligible. However, the reductions of mass yields were profound at temperature and residence time of 250 °C - 300 °C and 20 - 60 min, respectively. This was caused by decomposition of hemicellulose and partial decomposition of cellulose and lignin as well as release of the corresponding products [14]. The trend of mass reduction was consistent with other studies concerning torrefaction of biomass other than EFB [7]. Maximum reductions in mass yields of torrefied product were found at 300 °C, i.e.
38.0%. Meanwhile, the yields of liquid and gas products derived from torrefaction of EFB increased with torrefaction temperature. These trends were in accordance with a previous research done by Chen et al. (2018) [15]. When the temperature increased from 250 °C to 300 °C, the mass yields of liquid product increased from 11.5% to 17.3% (at retention time of 60 min). The maximum gas yield was obtained at 300 °C with retention time of 60 min i.e. 44.7%. On the other hand, the yield of torrefied EFB decreased with an increase in retention time, while liquid and gas yields increased. However, this effect was less significant than that of torrefaction temperature for all the experiments conducted.

![Figure 3. Torrefaction product distribution of empty fruit bunches at different temperatures and retention times.](image)

### 3.3. Characterisation of torrefied product

Figure 4 shows the proximate analysis of torrefied EFB, i.e. volatile matter, moisture content, fixed carbon and ash content. The results showed that the fixed carbon and ash contents of torrefied EFB increased with increasing temperature; for example, at 250 °C and 300 °C with retention time of 60 min, the fixed carbon increased from 21.1 wt.% to 40.9 wt.%, about 195% increment as compared to the raw EFB. Martin-Lara et al. (2017) [11] found similar results when they studied the torrefaction of olive tree pruning. This increment indicates that the torrefied product is good for combustion process as it can extend burning time during combustion. The ash content of the torrefied EFB ranged from 3.70 to 10.88 wt.%. The lowest ash content (3.70 wt.%) was obtained at the lowest temperature i.e. 200 °C. The changes in the ash content after torrefaction were mainly due to breakdown of carbon-hydrogen bonds, resulted in volatile loss and further concentration of the ash content in the torrefied product. Meanwhile, the volatile matter and moisture contents of torrefied EFB decreased when torrefaction temperature increased. At 300 °C, the volatile matters had significantly been reduced to 44.7 wt.% during a 60-min torrefaction period. Reduction of volatile matter was due to the catalytic effect of inorganic mineral matter in the biomass during torrefaction [16]. The moisture content of the torrefied EFB ranged from 1.18 to 5.7 wt.%. However, the results indicated that retention time was less impactful on the proximate analysis compared to temperature for all the experiments conducted.
Figure 4. Proximate analysis of torrefied empty fruit bunches.

As shown in Table 2, the elemental compositions of torrefied EFB changed as a function of torrefaction temperature and retention time. However, the changes were more dominant for temperature than retention time, especially on carbon and oxygen contents. As the torrefaction temperature increased from 225 to 300 ºC, the carbon content of torrefied EFB increased whereas the hydrogen and oxygen contents decreased. For example, the carbon content of the torrefied EFB increased from 45.7 wt.% to 62.2 wt.%, whereas the oxygen content decreased from 47.5 wt.% to 31.8 wt.% at retention time of 60 min. This range of variation is consistent with other torrefaction studies using different biomass feedstock [11, 13]. Reduction in hydrogen and oxygen contents is generally attributable to destroyed hydroxyl groups (-OH) in biomass during torrefaction, which has consequently led to solid product [17].

Table 2. Ultimate analysis of torrefied empty fruit bunches (EFB).

| Sample   | Ultimate analysis (wt. %) |   |
|----------|---------------------------|---|
|          | Carbon | Hydrogen | Nitrogen | Oxygena |
| Raw EFB  | 42.82  | 6.07     | 0.54     | 50.57    |
| 225-20   | 45.77  | 6.16     | 0.57     | 47.40    |
| 225-40   | 46.04  | 6.16     | 0.70     | 47.10    |
| 225-60   | 45.66  | 6.27     | 0.57     | 47.50    |
| 250-20   | 46.61  | 6.20     | 0.61     | 46.58    |
| 250-40   | 48.70  | 6.09     | 0.65     | 44.56    |
| 250-60   | 50.58  | 5.93     | 0.84     | 42.65    |
| 300-20   | 59.69  | 5.28     | 1.02     | 34.01    |
| 300-40   | 58.89  | 5.12     | 1.16     | 34.83    |
| 300-60   | 62.15  | 4.85     | 1.19     | 31.81    |

a by difference

Figure 5 shows the CV and energy yield of torrefied EFB as a function of final temperature and retention time. The CV increased when temperature and retention time were increased. It increased up to 25.73 MJ/kg at torrefaction temperature of 300 ºC and retention time of 60 min which was about 46% higher compared to CV of raw EFB. Similar trend of an increased CV for other torrefied biomasses has been reported previously [7]. On the other hand, energy yield of the torrefied EFB
decreased when temperature and retention time increased. The energy yield was ranged 55.71% to 95.33%. Torrefied product with the least energy yield (55.71%) was obtained at the most severe torrefaction conditions i.e. 300 °C and 60 min, though with higher CV. At this condition, large amount of energy was lost due to greater EFB mass loss thus lowering the energy yield of the torrefied product [7].

![Figure 5. Calorific value and energy yield of torrefied empty fruit bunches.](image)

4. Conclusion
Effect of temperature was more significant than retention time in affecting the torrefied EFB products distribution (solid, liquid and gas). The produced torrefied EFB underwent physicochemical changes, including mass reduction, rise in energy content and chemical compositions. These changes became more evident as torrefaction temperatures was increased. Both the CV and carbon content of torrefied product were improved up to 46% that of the raw EFB, while oxygen content decreased about 36%. Meanwhile, mass and energy yields decreased with increasing temperature and retention time of torrefaction especially at more severe conditions. As torrefaction can improve the physicochemical properties of EFB, its use as a solid fuel in thermal processes is therefore promising.

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