Characterization of pyrolysis products of oil palm empty fruit bunch

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Abstract. Oil Palm Empty Fruit Bunch (EFB) is a waste product in the palm oil industry. Currently, EFB has not been optimally utilized because of its low calorific value. The purpose of this research is to analyze the potential of EFB conversion into solid (biochar), liquid (bio-oil), and synthesis gas (syngas). The experiment was carried out in the batch process at atmospheric pressure and temperature of 600 ºC (heating rate of 60 ºC/min). Biochar was cooled and analyzed its proximate, ultimate, and calorific value. The volatile matter of pyrolysis product was passed through a condenser so that bio-oil and water were condensed and separated based on density difference. Bio-oil was analyzed by fractionation based on its boiling point. The syngas composition was analyzed by using GC. The proximate analysis results of biochar, such as inherent moisture, ash, volatile matter, and fixed carbon, are 6.24%, 26.30%, 14.14%, and 53.32%, respectively. Meanwhile, the ultimate analysis of biochar showing the composition of C, H, O, S, and N is 55.76%, 2.92%, 12.5%, 0.34%, and 2.18%, respectively. The biochar calorific value is 4,966 kcal/kg adb, showing a similar characteristic to sub-bituminous coal, suggesting that biochar can be utilized as a coal substitution to reduce the CO₂ emission on Electric Steam Power Plant. Bio-oil fractionation showed that the initial boiling point (IBP) temperature started at 67.8 ºC, and the final boiling point (FBP) temperature at 666.8 ºC. The largest fraction was kerosene (36.2%) and diesel, indicating that bio-oil can be processed further into fuel oil. Syngas analysis results showed that the main gas compositions are CH₄ (13 – 17% vol), H₂ (28 – 33% vol), CO (17 – 26% vol), and CO₂ gases (16 – 31% vol) with a calorific value of 2,600 – 3,300 Kcal/Nm³. Some alternatives to syngas utilization are as a source of pyrolysis energy and for chemicals syntheses such as methanol and DME.

1. Introduction
Biomass availability in Indonesia is relatively abundant. However, it cannot be readily found in every region, making it difficult to be collected and utilized on a massive scale. Palm oil mills are a potential source of biomass waste [1]. There are 470 oil palm processing plants in Indonesia that produce solid waste of 15.2 million tons per year [1][2]. One kind of solid waste is the oil palm empty fruit bunch (EFB) that is usually used as fertilizer for palm crops. EFB is potential to be converted into solid, liquid, and gas products through the pyrolysis process. Solids, gases, and liquid products of the pyrolysis process have higher economic value either when used directly or when processed further into its derivative products. With the decreasing fossil fuel reserves, notably petroleum, it is necessary to search for renewable energy sources for fuel substitution. This research is a preliminary study to find out the alternative utilization of EFB as fuel or chemical that is in line with the business development
of PT Pertamina. Bio-oil pyrolysis product is the main product which is met the refinery feed supply. While gas products (syngas) and biochar offer some potential applications that need to be identified for further utilization. This research aims to characterize the potential of empty palm fruit bunch into solid, gas, and liquid pyrolysis products to find alternative sources for the feed of refineries.

2. Methodology

The oil palm empty fruit bunch (EFB) samples from PT Pertamina are in fine-size, approximately 5 mm in size. The pyrolysis reactor (figure 1) used in the experiment is a home-made stainless-steel tube with a diameter of 30 cm and a height of 50 cm. The reactor is electrically heated with a power of 500 Watt, and the maximum reactor temperature can reach 800 ºC. The reactor is equipped with a condenser connected to a water chiller to cool it down to 8 ºC.

![Figure 1. A set of the pyrolysis reactor.](image)

2.1. Experimental procedures

Steps in the EFB pyrolysis and characterization process can be seen in Figure 2. EFB was taken as feed and analyzed its water content, proximate, ultimate, and calorific value. Then, EFB feed was dried in an electric oven at a drying temperature of 120 ºC for 4 hours to eliminate the water content. Following the drying process, as-dried EFB is fed into the pyrolysis reactor. The flow diagram of the pyrolysis process can be seen in Figure 2. Pyrolysis test was carried out in a batch system at atmospheric pressure and temperature of 600 ºC (heating rate of about 60 ºC/min). During the pyrolysis process, the volatile matter will evaporate and flows towards a condenser series. In the condensers, the content of compounds with high dew points in the volatile matter will condense first, i.e., water components and long-chain hydrocarbon compounds. This liquid product is known by several terms, namely tar, biocrude, or bio-oil. The fluid is collected every 30 minutes, and measured the volume. Meanwhile, the volatile matter that does not condense (pyrolysis gas products) are passed impinger and gas filters using activated carbon media. The gas was subsequently burned before being discharged into the atmosphere. A portion of pyrolysis gaseous products is periodically sampled and analyzed using a portable gas analyzer. Before being streamed to the gas analyzer, gaseous pyrolysis products were mixed with the tracer N₂ (nitrogen) to calculate the mass and energy balance. After the pyrolysis test was completed, the electric heater on the pyrolysis reactor was turned off and let it cool down naturally. The bio-char product formed on the bottom of the reactor was taken and evaluated, including the water content, proximate, ultimate, and calorific value analysis. On the other hand, the
liquid product was analyzed following a specification standard of liquid pyrolysis as a biofuel product (ASTM D7544-12).

![Figure 2. Steps in the pyrolysis process of EFB and their characterizations.](image)

3. Results and discussion

3.1. Mass and energy balance calculation

The results of mass and energy balance calculation of the EFB pyrolysis process can be seen in Figure 3. The calculations were conducted using Chemcad software. The calculation results indicate that the 1,872 grams of EFB will be converted to 903 grams of charcoal (48% weight to feed), 280.5-gram liquid products (15% weight to feed), and 688.5 grams of gas products (37% weight to feed). The pyrolysis process is an endothermic reaction that requires a reaction heat of 0.72 MJ or 0.20 kWh. The heat of this reaction is supplied from electrical energy used for heating the reactor. However, we believe that further optimization of pyrolysis operating conditions needs to be done to increase oil yields [3].
3.2. Characterization of EFB and Biochar

EFB and biochar were analyzed its proximate, ultimate, and calorific value. The results are listed in Table 1. Moisture content (inherent moisture, IM) on EFB is 13.84% (adb), while bio-char is only 6.24% (adb). The moisture content explains the amount of adsorbed water on the solid surface, either physically or chemically bound in solids pore. Moisture content will affect the process of all thermal conversion, such as pyrolysis, gasification, and combustion. High water content requires high energy to vaporize, decreasing overall thermal efficiency [4][5].

| Parameters analysis                      | EFB      | Biochar  | Unit | Basis | Method       |
|-----------------------------------------|----------|----------|------|-------|--------------|
| **Proximate**                           |          |          |      |       |              |
| Moisture content (IM)                   | 13.84    | 6.24     | %    | adb   | ASTM D.3173  |
| Ash                                     | 9.32     | 26.3     | %    | adb   | ASTM D.3174  |
| Volatile Matter (VM)                    | 57.98    | 14.14    | %    | adb   | ASTM D.3175  |
| Fixed Carbon (FC)                       | 18.86    | 53.32    | %    | adb   | ASTM D.3172  |
| **Ultimate**                            |          |          |      |       |              |
| Total Sulfur (S)                        | 0.21     | 0.34     | %    | adb   | ASTM D.4239  |
| Carbon (C)                              | 40.05    | 55.76    | %    | adb   | ASTM D.5373  |
| Hydrogen (H)                            | 5.64     | 2.92     | %    | adb   | ASTM D.5373  |
| Nitrogen (N)                            | 1.67     | 2.18     | %    | adb   | ASTM D.5373  |
| Oxygen (O)                              | 43.11    | 12.5     | %    | adb   | ASTM D.3176  |
| **Gross Calorific Value (GCV)**         | 3.816    | 4,966    | cal/gr | adb | ASTM D.5865 |

All test data in Tables 1-4 are carried out in one single test. The composition of fixed carbon and ash of EFB is 18.86% and 9.32%, respectively. While the composition of fixed carbon and ash of biochar is 53.32% and 26.30%, respectively. Fixed carbon indicates solid carbon left in the material after the volatile material is removed. The amount of fixed carbon in the feed is barely evaporated and settled on solid products along with ash forming char material (bio-char). Volatile matter (VM) or a flying substance is a material that converts into liquid and gas products. It can be seen from a significant decrease in the volatile matter in a solid product [6]. Biochar has two advantages compared to EFB feed. First, biochar has a larger energy density (calorific value). Second, burning charcoal does not produce smoke that makes it more environmentally friendly. The calorific value of bio-char is 4,966 cal/gr.
cal/g, which is higher than that of EFB (3,816 cal/g). Such properties, suggesting that biochar is suitable for briquettes and used for household or industrial needs.

3.3. Bio-Oil Characterization

The number of liquid products obtained is 2,593.6 g, consisting of 1,972.8 g of water and 620.8 g of oil. The water content of liquid products is 76% of the mass because the C and H content of the ultimate analysis results in the EFB is relatively high, at 48.75% of mass [3]. Oil content in liquid products can be increased by minimizing condensable gases released during the condensation process of gaseous products. Analysis result of water content, specific gravity (SG 60/60 °F), and GC Simdis can be seen in table 2. The Simdis analysis results indicate that the initial boiling point (IBP) is obtained at a temperature of 67.8 °C, and the final boiling point (FBP) is obtained at a temperature of 666.8 °C. Gasoline fraction obtained from oil products is 5% of the mass, which is cut point distillation at IBP temperature – 100 °C.

| No | Testing          | Unit | Method       | Results |
|----|-----------------|------|--------------|---------|
| 1  | Water Content   | mass | ASTM D6304   | 0.10767 |
| 2  | SG 60/60 °F     |      | Pycnometers  | 1.0964  |
| 3  | GC Simdis       |      | ASTM D7169   |         |

Distillation: ~IBP °C 67.8
~ 5% of the mass °C 109.4
~ 10% of the mass °C 112.4
~ 20% of the mass °C 117.2
~ 30% of the mass °C 158.4
~ 40% of the mass °C 212.4
~ 50% of the mass °C 236.2
~ 60% of the mass °C 266.4
~ 70% of the mass °C 300.2
~ 80% of the mass °C 351.6
~ 90% of the mass °C 425.8
~ 95% of the mass °C 487.4
~ FBP °C 666.8

BP distribution – Cut point shown in table 3. The naphtha fraction obtained is 28.8% of the mass is the cut point distillation at a temperature range between 100 – 180 °C. Kerosene fraction and diesel acquired is 36.2% of the mass, which is cut point distillation between 180 – 300 °C. The residual fraction obtained at temperatures above 350 °C is 13.4% of the mass. This results in showing that kerosene fraction and a diesel fraction is the largest fraction of the oil product of EFB pyrolysis. These results suggest that oil products from the pyrolysis of EFB (biocrude) have the opportunity to be further processed into fuel [7][8].

| BP (°C) | Recovered (mass %) | Residual (mass %) | Fraction (mass %) |
|---------|---------------------|-------------------|-------------------|
| 180.0   | 33.8                | 66.2              | 33.8              |
| 270.0   | 60.8                | 39.2              | 27.0              |
| 300.0   | 70.0                | 30.0              | 9.2               |
| 370.0   | 83.4                | 16.6              | 13.4              |
| 30.0    | <IBP                | 100.0             | 0.0               |
3.4. Characterization of Gaseous Products

Volatile matter (VM) EFB was converted into liquid and gaseous products [9]. In general, the products of pyrolysis gases consist of hydrogen (H₂), carbon monoxide (CO), carbon monoxide (CO₂), and light chain hydrocarbons (CH₂, C₃H₈) with a calorific value of about 10 – 20 MJ/Nm³ or 2,000 – 4,700 kcal/Nm³. Results of product analysis of pyrolysis gases can be seen in table 4. Hydrogen gas composition (H₂) is about 28 – 33% mol, and carbon monoxide (CO) is between 17 – 25% mol. This suggests that the major component (>50 % mol) of gaseous products constitutes as synthesis gas (syngas). On the other hand, gaseous products are also containing methane gas (CH₄) that present about 13 – 17%mol. Methane can be reprocessed through steam reforming (using steam reactant) or dry reforming (using the reactant CO₂ already contained in pyrolysis gas products) to syngas. Syngas can be further utilized as the raw material of hydrogen generation, fuel synthesis, and petrochemical raw materials [8].

| Analysis No. | Time (min) | CO (%) | CO₂ (%) | O₂ (%) | H₂ (%) | CH₄ (%) | N₂ (%) | CaH₈ (%) | Calorific Value (HHV) (MJ/m³) |
|--------------|------------|--------|---------|--------|--------|---------|--------|----------|-------------------------------|
| 1            | 10.10      | 20.40  | 31.12   | 0.52   | 28.03  | 13.05   | 5.80   | 1.08     | 10.98                        |
| 2            | 10.40      | 17.47  | 29.38   | 0.15   | 33.28  | 13.47   | 5.09   | 1.16     | 11.38                        |
| 3            | 11.10      | 17.71  | 29.01   | 0.14   | 33.41  | 14.45   | 4.19   | 1.09     | 11.72                        |
| 4            | 11.40      | 21.65  | 24.54   | 0.13   | 31.54  | 16.95   | 5.19   | 0.00     | 12.22                        |
| 5            | 12.10      | 24.29  | 20.28   | 0.16   | 32.14  | 17.48   | 4.83   | 0.82     | 13.33                        |
| 6            | 12.40      | 25.58  | 16.24   | 0.09   | 31.38  | 16.24   | 9.63   | 0.84     | 13.83                        |

4. Conclusion

The pyrolysis of EFB can produce a value-added material that is charcoal, bio-oil, and syngas. Biochar can be used as biomass charcoal briquette or coal substitution for environmentally friendly fuel. Bio-oil has the potential to be processed further into fuel oil, while syngas can be converted to various kinds of fuel and chemicals.

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