Torrefaction of Empty Fruit Bunches: Evaluation of Fuel Characteristics Using Response Surface Methodology

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Abstract. Empty Fruit Bunches (EFB) can be used as an alternative energy source by torrefaction process. Torrefaction is a treatment process of biomass into solid fuel within temperature range of 200-300 °C in an inert condition. This research aims to study the effect of process variable interaction process and to study the characteristic of solid fuel resulted. Torrefaction of EFB was performed by using horizontal fixed bed reactor at three different temperatures, times and particle sizes, that are 225, 250 and 275 °C, for 15, 30 and 45 minutes, and particle size 2, 4 and 6 cm, respectively. Response surface methodology (RSM) was used for to optimization optimize of torrefaction conditions so that biofuel of with high energy density, maximum energy properties, and minimum weight loss could be manufactured. The analysis showed that increase in heating values was affected by treatment severity (cumulated effect of temperature, time and particle size). Our results clearly demonstrated an increased degradation of the material due to the combined effects of temperature and treatment time. While the reaction time had less impact on the energy density of torrefied biomass, the effect of reaction temperature was considerably stronger under the torrefaction conditions used in this study. The most significant factor affecting all responses was temperature of torrefaction, then followed by time and particle size. The optimized torrefaction conditions were verified empirically and the applicability of the model was confirmed. The torrefied biomass occurred more suitable than raw biomass in terms of calorific value, physical and chemical properties.

1. Introduction
As an agrarian country, Indonesia is abundant in plant biomass resources from plantation and agricultural wastes. This biomass has many benefits because it contains beneficial components such as cellulose, hemicellulose, lignin, monosaccharide, pectin, amino acids, aliphatic acids, ether, fats, oils,
waxes, resin, pigments and proteins[1–3]. The biomass can be utilized by extraction of beneficial components, conversion to other forms, or use as raw material for fuel production. Pectin can be isolated from coffee biomass [4] for membrane matrix material [5,6]. Biomass can be converted to char chemical feedstocks for soil repaired purpose [7]. Biomass is also a promising material for fuel production [8]. Especially for chemical feedstock and fuel production purpose, these utilizations are achieved by using conversion methods of polymer materials such as thermal conversion (pyrolysis, hydrothermal, gasification) [9,10], and also chemical, biochemical and electrochemical conversion [11–13].

As one of the largest producer and exporter of palm oil, Indonesia has abundant availability of oil palm biomass as a promising source of lignocellulosic biomass. This biomass is a primary source of renewable carbon that can be utilized as a feedstock for biofuels production in order to achieve energy independence. Additional quantities of lignocellulosic biomass can be yielded from energy crops. These crops are fast-growing plants which are harvested specifically for energy production. Innature, lignocellulosic biomass has high moisture content, low bulk energy density. It is also difficult to transport, to handle, to store and to feed into existing combustion systems without pretreatments. When lignocellulosic biomass is employed for bioenergy, pretreatments of lignocellulosic biomass are essential procedures for achieving high efficiencies of fuel production or consumption. For example, moisture content from the lignocellulosic biomass was driven off to enhance the combustion efficiency and prevent microbial degradation during storage [14].

While, in liquid biofuel production, acid pretreatments have been adopted widely to facilitate the conversion of hemicelluloses and cellulose into soluble sugars for lignocellulosic ethanol production [14,15]. In addition to the methods mentioned above, torrefaction of lignocellulosic biomass is another notable pretreatment method that enables energy densification of biomass and biomass homogenization [16–18]. This pretreatment has been recommended as an efficient way to enhance solid biofuel properties by water removal, to reduce the hygroscopic range, and to increase grindability. Torrefaction is a thermal pretreatment process by subjecting wood to temperature levels between 200 and 300°C in the absence of oxygen. During this process, the cell walls are degraded and the products produced are between wood and charcoal. The residual product is in solid form, which is referred to as torrefied biomass.

Torrefaction product quality is influenced by several factors like temperature, reaction time, biomass type, and particle size [18]. In the previous research, it showed that temperature and time give a significant effect on mass and energy yield, reducing and enhancing the energy density [19,20]. This is caused by the increase in the degradation rate of hemicellulose when temperature and time raised. Mass of lignocellulosic biomass mostly decreases in the range of 30–45 minute torrefaction [20]. Particle size is also one of the factors that influence the product. Different particle size in term length, gives different result for mass and energy yield [21].

One of lignocellulosic biomass from oil palm biomass is Empty Fruit Bunches (EFB). This biomass is mill waste that remains from oil pressing of fresh fruit bunches. Thus, the purpose of this study was to build a model of torrefaction procedure applicable in the production of solid biofuel from lignocellulosic biomass of EFB. Changes in the energy properties of the lignocellulosic biomass when subjected to torrefaction in the temperature range of 225–275 °C with different treatment time were investigated. Weight loss is an important parameter for optimizing the design and operation of a biomass torrefaction plant. So that understanding of the process variables for optimization is required. Torrefaction condition was optimized by using the technique of response surface methodology (RSM) that recently report enables to find the optimum torrefaction conditions for a high-density solid fuel.

2. Experimental

2.1 Material Preparation. The oil palm biomass used was EFB obtained from CPO-mill nearest in Riau province. EFB was cut with dimensions of length 2.4 and 6 cm. EFB was dried by sun for a few
days and remove the residual from EFB. The moisture content was measured in an oven at 105°C for 24 hours.

2.2 Torrefaction Process, 50 g dried wood fine was placed in a furnace and torrefied under the conditions as follows: temperature 225 °C, 250 °C and 275 °C for 15 min, 30 min, and 45 min. Torrefaction was performed in a horizontal fixed bed reactor of 6 cm diameter and 60 cm length, (Fig. 1) controlled by a continuous nitrogen flow rate at 100 ml/minutes. The carrier gas was continuously blown into the reactor to keep the system stay in inert condition and to remove the volatiles produced during the torrefaction.

![Torrefaction Reactor](Image)

**Figure 1. Torrefaction Reactor**

The reactor is also equipped with condenser to condense volatiles and bubble shop flow meter to control the flow rate of nitrogen. For each treatment, the mass yield and energy yield of the torrefied wood were calculated using the following expression (Eq (1) and (2)):

\[
MY = \frac{\text{Mass after torrefaction}}{\text{Mass before torrefaction}} \times 100\% \quad (1)
\]

\[
EY = \%MY \times \frac{\text{HHV product}}{\text{HHV biomass}} \quad (2)
\]

2.3 Determination of higher heating value (HHV), The calorific value was determined according to BSI standard EN 14918 by using bomb calorimeter, in which 0.50 g of oven-dried biomass was completely combusted under an oxygen pressurized (3000 kPa). Proximate Analysis: moisture content was measured by drying in an oven at 105°C according to ASTM D-3173. For ash content, a prescribed amount (1 g) of the sample was calcined in an electric furnace at 750°C for 2 hours, according to ASTM D-3174. The volatile matter was also measured using furnace which 1 g of sample was calcined at 950°C for 7 minutes according to ASTM D-3175.

2.4 Response Surface Analysis, Second-degree polynomials were calculated using Design Expert Version 7.0 software (Stat-Ease, USA) to estimate the response of the dependent variables. Temperature (\(X_1, 225-275°C\)); time (\(X_2, 15-45\) minutes); and particle size (\(X_3, 2-6\) cm) were chosen as the independent variables. HHV, mass yield, and energy yield of lignocellulosic biomass of EFB were used as the dependent output variables. Central Composite Design (CCD) was used as a design experiment.
3. Results and Discussion

The results of mass measurement, calorific value, and proximate analysis for the sample and torrefied samples are listed in Table 1. In this study, EFB color became darker with increasing of torrefaction temperature and time. A similar tendency was observed from previous research [19].

3.1 Calorific Value (HHV)

The HHV and weight loss depended on the torrefaction conditions listed in Table 1. Calorific value as energy density is one of the important parameter of the product of torrefaction. It gives the amount of energy released when unit mass of torrefied product is burnt [18]. Compared to fossil fuel, biomass contains less carbon and has lower calorific values on similar weight basis. HHV significantly increased with increasing of torrefaction temperature and time. The highest HHV obtained at 292 °C. This tendency is consistent with the previous study [14,18,20,22,23]. HHV increased mainly due to the increase of carbon content and decreasing of oxygen and hydrogen contents. The oxygen and hydrogen content decrease by decomposition of hemicellulose and organic compounds such as water, acetic acid, and phenol during torrefaction [20,24]. In term of increasing in length, the HHV decreased while particle size increased. The tendency was similar to the previous study, and it's mainly due to the surface area of biomass decrease along with increasing particle size. The smaller the surface area, the more difficult the volatiles formed during torrefaction to get out from inner core of biomass [21]. HHV represents the maximum amount of energy embedded per gram of material. It is important to mention that HHV increases with the extent of treatment. The energy value ranged from 18.08 to 28.94 MJ/kg depending on the torrefaction conditions. This implies that the HHV in the torrefied biomass increased by 6.5-30% compared to the untreated material.

| Table 1. Results of mass yield, energy yield, and proximate analysis of the product |
|----------------------------------|-----------------|------------------|
| HHV (kJ/kg) | Raw EFB | Torrefied EFB |
| Mass yield (%) | - | - |
| Energy yield (%) | - | - |
| Moisture (%) | 11,50 | 5,00-8,50 |
| Ash (%) | 1,45 | 1,75-3,41 |
| Volatile(%) | 69,45 | 47,73-63,20 |
| Fixed carbon(%) | 17,60 | 26,26-44,31 |

HHV was more affected by the temperature than the reaction time. The HHV significantly increased with a torrefaction temperature exceeding 250 °C, regardless of the reaction time. HHV slowly increased as the temperature raised until 250 °C, then drastically increased as the temperature further increased from 250 °C to 292 °C. It is clear that the characteristics of the thermal degradation of the biomass are different. Furthermore, from the viewpoint of practical applications of torrefaction, if the light torrefaction (225 °C) is applied for the pretreatment of biomass, its main function will be to remove moisture and light volatiles from the biomass (Pierre et al., 2011). Therefore, it is inferred that the heating value of the torrefied biomass is improved to a small extent. If biomass is pretreated using the severer torrefaction conditions (above 300 °C), the role of lignin (acid-insoluble fraction) in energy contribution might be more important than holocellulose resulting from a large portion of holocellulose being consumed in the severe torrefaction. High lignin content in torrefied biomass is associated with high energy density due to the ether and carbon linkages in lignin, which has higher energy than the C-O or C-H bonds (Lehtikangas, 2001). Therefore, high lignin content in the torrefied biomass results in a higher HHV.
3.2 Mass and Energy yield
Mass yield gives a measure of the solid yield of the torrefaction. It defines what fraction of the original mass of biomass would remain in the torrefied product. While energy yield gives the fraction of energy in biomass retained after torrefaction [18]. Solid fraction remained after torrefaction. Most of it consists of cellulose and lignin that was not degraded. Based on composition, carbon and oxygen are the main constituent of the solid product, followed by a small amount of nitrogen and hydrogen [24]. The measure of mass and energy yield based on equation 1 and 2.

Mass and energy yield decreased when torrefaction temperature and time increased. Its mainly due to more and more degraded hemicellulose in range of torrefaction temperature and time followed by a little bit of cellulose and lignin decreased gradually [19,20]. Energy yield obtained in this study also decreased when temperature and time increasing. This is due to the increase in the calorific value that was not able to offset the decrease in mass yield when temperature and time raise [14,19,20]. It can be also caused by the fibrous structure of EFB compared to the oil palm waste like shell, tree, and frond thus resulting in decreasing solid fraction significantly.

The particle size effect is less significant than the torrefaction temperature and time. Increase in particle size in length will increase mass and energy yield. It's mainly due to the smaller surface area of biomass, the higher the heat resistance, the more difficult the volatiles formed to get out through the biomass [21].

3.3 Proximate Analysis
Moisture content is an important property of biomass fuels because a high moisture level in a fuel leads to a high energy loss in the course of burning [25]. As a feedstock, it is desirable to lower the moisture content of biomass through drying process before thermochemical conversion. In addition to increasing energy in solid fuel, biomass which has a lower water content can be stored for a long time and minimize transportation costs. In this study, moisture content decreased when torrefaction of temperature and time increased. Its mainly due to dehydration during torrefaction [18].

Ash is the inorganic solid residue left after biomass is completely burned. Its primary ingredients are silica, aluminium, iron, and calcium with small amounts of magnesium, titanium, sodium, and potassium [18,25]. Effect of increasing of torrefaction temperature, time, and particle size gives the positive effect for increasing ash content. Maybe, the ash content increases because temperature does not reach the ashing temperature on biomass in range 550°C and for coal is 780°C. In addition, the mass reduction occurred during the process is not accompanied by degradation of the components forming ash.

Volatile matter is closely related to fixed carbon in biomass. Devolatilization was affected by torrefaction temperature and time. When temperature and time increased, volatile matter decreased but fixed carbon increased. This is due to the torrefaction temperature which is also in the temperature range of hemicellulose degradation [25]. Hydrogen and oxygen as part of major components in biomass are part of the most widely lost component during torrefaction. The reduced fraction of hydrogen and oxygen resulted in a decrease volatile matter and increase in the ratio of carbon to hydrogen and oxygen so that calorific value of the product also increased [18,25].

3.4 Response Surface Analysis for HHV, mass and energy yield
The primary goal of torrefaction is to refine raw biomass into upgrade solid fuel, improved handling qualities and enhanced combustible properties comparable to fossil coal, reading to reduce costs and to raise economic gains [26]. Regarding fuel efficiency, it's necessary to increase calorific value, mass yield, and energy yield. Those responses were fitted to the response surface model provided by the mathematic models in Table 2, in order to analyze the effect of the torrefaction factor on responses. \( Y_p \) is the predicted calorific value (kJ/kg), while \( Y_m \) and \( Y_e \) were predicted for mass yield and energy yield. Where \( X_1, X_2, \) and \( X_3 \) were temperature, time, and particle size.
Table 2 Summary of the results of various research responses to the heating value ($Y_1$); Mass yield ($Y_2$); Energy yield ($Y_3$)

| Run | Natural Variables | Coded Variables | Responses |
|-----|-------------------|----------------|-----------|
|     | $\xi_1$ (°C)     | $\xi_2$ (menit) | $\xi_3$ (cm) | $X_1$ | $X_2$ | $X_3$ | $Y_1$ (kJ/kg) | $Y_2$ (%) | $Y_3$ (%) |
| 1   | 225               | 15             | 2           | -1   | -1   | -1   | 18124,40     | 82,99     | 93,81     |
| 2   | 275               | 15             | 2           | 1    | -1   | -1   | 19038,35     | 70,02     | 87,86     |
| 3   | 225               | 45             | 2           | -1   | 1    | -1   | 19029,91     | 72,00     | 85,46     |
| 4   | 275               | 45             | 2           | 1    | 1    | -1   | 20068,94     | 66,82     | 87,62     |
| 5   | 225               | 15             | 6           | -1   | -1   | 1    | 17954,39     | 83,86     | 93,90     |
| 6   | 275               | 15             | 6           | 1    | -1   | 1    | 19659,74     | 70,38     | 86,29     |
| 7   | 225               | 45             | 6           | -1   | 1    | 1    | 18811,49     | 72,92     | 85,55     |
| 8   | 275               | 45             | 6           | 1    | 1    | 1    | 20078,40     | 57,65     | 72,20     |
| 9   | 208               | 30             | 4           | -1.682 | 0  | 0 | 18322,67 | 82,40 | 95,96 |
| 10  | 292               | 30             | 4           | 1.682 | 0  | 0 | 20461,13 | 66,31 | 85,57 |
| 11  | 250               | 5              | 4           | 0 | -1.682 | 0 | 18527,37 | 82,39 | 95,21 |
| 12  | 250               | 55             | 4           | 0 | 1.682 | 0 | 19543,13 | 53,19 | 64,83 |
| 13  | 250               | 30             | 0.7         | 0 | 0 | -1.682 | 18896,72 | 82,17 | 96,83 |
| 14  | 250               | 30             | 7.3         | 0 | 0 | 1.682 | 18056,56 | 75,92 | 85,52 |
| 15  | 250               | 30             | 4           | 0 | 0 | 0 | 19506,95 | 64,10 | 78,03 |
| 16  | 250               | 30             | 4           | 0 | 0 | 0 | 19186,38 | 66,60 | 79,69 |
| 17  | 250               | 30             | 4           | 0 | 0 | 0 | 18953,61 | 65,62 | 77,57 |
| 18  | 250               | 30             | 4           | 0 | 0 | 0 | 19229,87 | 66,52 | 79,78 |
| 19  | 250               | 30             | 4           | 0 | 0 | 0 | 19330,98 | 68,18 | 82,20 |
| 20  | 250               | 30             | 4           | 0 | 0 | 0 | 19340,74 | 66,14 | 79,79 |

To match the model to experimental data obtained, the regression analysis carried out on the second-degree polynomial using Analysis of Variance (ANOVA), which can be seen in Table 3. The mathematical model as shown in Table 2 correlates well that can be seen from $R^2$ that is high. The probability value (p-value) used for each model is $<$0.05 which states the model is significant at 95%. Based on results obtained, all variables; torrefaction temperature, time, and particle size have a significant influence over 95%.

The responses for calorific value, mass yield, and energy yield from torrefaction were plotted as three-dimensional surface plots of two factors; temperature vs time. Particle size was plotted on a fixed axis due to level of significantly smaller than the torrefaction temperature and time. Fig 2 shows the effect of torrefaction operating conditions of responses were analyzed.

Table 3. Analysis of Variance (ANOVA) quadratic model for calorific value, mass yield, and energy yield.

| Source | Sum of Squares | Degrees of Freedom | Mean Square | F-Value | p-Value (Prob>F) |
|--------|----------------|--------------------|-------------|---------|-----------------|
| Calorific Value | 1.744E+07 | 9 | 1.938E+06 | 39.67 | $< 0.0001$ |
| Model | A-Temp | 1.014E+07 | 1 | 1.014E+07 | 207.51 | $< 0.0001$ |
| B-Time | 3.545E+06 | 1 | 3.545E+06 | 72.57 | $< 0.0001$ |
| C-Size | 2.007E+05 | 1 | 2.007E+05 | 4.11 | 0.0579 |
| AB | 24548,62 | 1 | 2.455E+04 | 0.50 | 0.0285 |
Table 4. The mathematical models derived from the experimental results for calorific value, mass yield, and energy yield.

| Source | Sum of Squares | Degrees of Freedom | Mean Square | F-Value  | p-Value (Prob>F) |
|--------|----------------|--------------------|-------------|----------|-----------------|
| AC     | 2.597E+05      | 1                  | 2.597E+05   | 5.32     | 0.4841          |
| BC     | 1.090E+05      | 1                  | 1.090E+05   | 2.23     | 0.1460          |
| A²     | 7.276E+05      | 1                  | 7.276E+05   | 14.89    | 0.0006          |
| B²     | 1.402E+05      | 1                  | 1.402E+05   | 2.87     | 0.1010          |
| C²     | 2.059E+06      | 1                  | 2.059E+06   | 42.14    | < 0.0001        |
| Residual | 1.417E+06    | 29                 | 48851.66    |          |                 |
| Lack of Fit | 9.126E+05   | 19                 | 48033.93    | 1.03     | 0.5007          |
| Total  | 1.891E+07      | 38                 |             |          |                 |

R-Square: 0.925

| Source | Sum of Squares | Degrees of Freedom | Mean Square | F-Value  | p-Value (Prob>F) |
|--------|----------------|--------------------|-------------|----------|-----------------|
| Model  | 2574.87        | 9                  | 286.10      | 27.09    | < 0.0001        |
| A-Temp | 729.90         | 1                  | 729.90      | 69.10    | < 0.0001        |
| B-Time | 1107.65        | 1                  | 1107.65     | 104.87   | < 0.0001        |
| C-Size | 44.91          | 1                  | 44.91       | 4.25     | 0.0483          |
| AB     | 9.00           | 1                  | 9.00        | 0.85     | 0.3636          |
| AC     | 28.14          | 1                  | 28.14       | 2.66     | 0.1134          |
| BC     | 22.47          | 1                  | 22.47       | 2.13     | 0.1555          |
| A²     | 213.17         | 1                  | 213.17      | 20.18    | 0.0001          |
| B²     | 0.06           | 1                  | 0.06        | 5.29E-03 | 0.9425          |
| C²     | 466.75         | 1                  | 466.75      | 42.14    | < 0.0001        |
| Residual | 306.31        | 29                 | 10.56       |          |                 |
| Lack of Fit | 263.93        | 19                 | 13.89       | 3.28     | 0.293           |
| Total  | 2884.71        | 38                 |             |          |                 |

R-Square: 0.894

| Source | Sum of Squares | Degrees of Freedom | Mean Square | F-Value  | p-Value (Prob>F) |
|--------|----------------|--------------------|-------------|----------|-----------------|
| Model  | 2333.75        | 9                  | 259.31      | 17.18    | < 0.0001        |
| A-Temp | 261.05         | 1                  | 261.05      | 17.29    | 0.0003          |
| B-Time | 987.82         | 1                  | 987.82      | 65.44    | < 0.0001        |
| C-Size | 188.14         | 1                  | 188.14      | 12.46    | 0.0014          |
| AB     | 1.42           | 1                  | 1.42        | 0.094    | 0.7611          |
| AC     | 73.75          | 1                  | 73.75       | 4.89     | 0.0351          |
| BC     | 47.92          | 1                  | 47.92       | 3.17     | 0.0853          |
| A²     | 400.09         | 1                  | 400.09      | 26.5     | < 0.0001        |
| B²     | 0.16           | 1                  | 0.16        | 0.011    | 0.9182          |
| C²     | 431.63         | 1                  | 431.63      | 28.59    | < 0.0001        |
| Residual | 437.78        | 29                 | 15.1        |          |                 |
| Lack of Fit | 354.36        | 19                 | 18.65       | 2.24     | 0.0965          |
| Total  | 2771.72        | 38                 |             |          |                 |

R-Square: 0.842
4. Conclusion
Biomass especially EFB can be upgraded and used as a fuel by torrefaction process. The torrefied biomass is more suitable than raw biomass in terms of calorific value, energy yield, and proximate analysis. The reaction temperature had a strong impact on the responses analyzed followed with torrefaction time and particle size.

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