Torrefaction of oil palm frond: The effect of process condition to calorific value and proximate analysis

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Abstract. Oil palm frond can be used as alternative energy source by torrefaction process. Torrefaction is a treatment process of biomass into solid fuel by heating within temperature range of 200-300°C in an inert environment. This research aims to result solid fuel through torrefaction and to study the effect of process variable interaction. Torrefaction of oil palm frond was using fixed bed horizontal reactor with operation condition of temperature (225-275 °C), time (15-45 minutes) and nitrogen flow rate (50-150 ml/min). Responses resulted were calorific value and proximate (moisture, ash, volatile matter and fixed carbon). Analysis result was processed by using Design Expert v7.0.0. Result obtained for calorific value was 17.700-19.600 kJ/kg and for the proximate were moisture range of 3-4%; ash range of 1.5-4%; volatile matter of 45-55% and fixed carbon of 37-46%. The most affecting factor significantly towards the responses was temperature then followed by time and nitrogen flow rate.

1. Introduction
Biomass is a primary source of renewable carbon that can be utilized as a feedstock for solid fuel production in order to achieve energy independence. As the largest producer of palm oil in Indonesia, Riau has potential to produce more lignocellulosic biomass. Riau has the widest oil palm plantation which reached about 1.7 million hectares in 2011 [1]. This extens has made Riau as the biggest contributor of CPO for national production that reached 6.2 million tons. Beside producing abundant vegetable oil, this extens also produces so many oil palm frond. Oil palm tree that can be planted in one hectare of oil palm plantation is about 140 trees [2]. Harvesting of fresh fruit bunches, 1-2 pieces of palm frond are cut in order to facilitate the next pollination and harvest. There is 6,400-7,500 palm frond in one hectare per year, about 4.5 kg per palm frond [3]. So that the potential of palm fronds produced from oil palm plantations in Riau in 2014 reached 7.7 million tons of dry palm frond.

Oil palm frond (OPF) is still being an unutilized waste maximally, that is often to be left in plantation. OPF combustion will cause bad environment. In order to give economical value-added of oil palm frond, Oil palm frond can be used as alternative energy source. OPF utilization for energy-based industry has not been done maximally, while energy potential in OPF is about 1.3 TJ/year [4].

Torrefaction is recommended as an efficient way to enhance solid fuel properties by water removal, reduction of the hygroscopic range and increased grindability. Torrefaction is a thermal pretreatment process by subjecting biomass to temperature levels between 200-300 °C in inert environment. During this process, hemicellulose is degraded and volatiles will be released from biomass. Decreasing of hemicellulose and volatiles will increase calorific value and maximize mass yield and energy yield from solid product [5-8]
Uemura et al. [5] torrefied empty fruit bunch at 225-275 °C and stated that temperature played a significant role toward solid product. Chin et al. [7] investigated OPT at 200-300 °C for 15-45 minutes. They found that time also affected torrefaction but was not as significant as temperature. Lu et al. [6] torrefied oil palm fiber in nitrogen and air at 250-350 °C and found that torrefaction in air resulted in lower solid quality than in nitrogen. Chen et al. [8] investigated oil palm fiber at 250-350 °C and with varieties superficial velocities or carrier gas flow rate in 100-1000 ml/minutes. They stated that increasing carrier gas flow rate graphically did not give any effect for solid product.

All of the findings of the above-mentioned studies were reported relatively quick succession. However, the effect of interaction between variables has not been explored yet. If those variables like as temperature, time and carrier gas flow rate is investigated its effect to calorific value and proximate analysis, and processed statistically using Response Surface Methodology (RSM), then OPF utilization through torrefaction will be result the advantage of torrefaction as biomass pretreatment. RSM will give design and analysis of model so the affecting factor significantly will be known.

In this paper, torrefaction of OPF was carried out in a fixed bed horizontal reactor in inert condition. The effects of temperature, time and nitrogen flow rate on calorific value and proximate analysis of solid product were investigated using RSM.

2. Materials and methods

2.1 Material preparation

Oil palm frond was cut into blocks with the dimensions of 1x1 cm. Biomass samples were dried by sun for few hours and then also dried in an oven at 105 °C in order to remove residual water remaining in the biomass. The moisture content was measured for 24 hours.

2.2 Torrefaction process

The samples (50 g) was placed in a reactor and torrefied by heater under the conditions as follows: temperature 225, 250 and 275 °C for 15, 30 and 45 minutes. Torrefaction was performed in a fixed bed horizontal reactor of 6 cm diameter and 60 cm length, which is shown in Figure 1. They were torrified by flushing carrier gas with flow rate of 50-150 ml/minutes. The carrier gas was continuously blown into the reactor to keep the system in an inert environment and to remove volatiles produced in the reactor. After torrefaction, the heater was turned off and the reactor was left to cool down to the ambient temperature. Then the torrified sample was recovered, kept in desicator and weighed.

2.3 Result analysis

2.3.1 Calorific value (HHV)

The calorific value was determined according to ASTM standard D-5865-13 with a bomb calorimeter, in which 0.5 g of dried biomass was completely combusted under a pressurized oxygen atmosphere.

2.3.2 Proximate

Moisture content was measured by drying in an oven at 105 °C for an hour according to ASTM standard D-3173-13. For the ash content, a prescribed amount (1 g) of the sample was weighed in a crucible and calcined in an electric furnace at 750 °C for 2 hours, according to ASTM standard D-3174-13. Volatile matter was also measured using a furnace which 1 g of sample was calcined at 950 °C for 7 minutes according to ASTM standard D-3175-13.
3. Results and discussion

The results of calorific value (HHV) and proximate of OPF are listed in Table 1.

Table 1. Characteristic of torrefaction raw and product

| Characteristic    | Raw       | Product          |
|------------------|-----------|------------------|
| HHV (kJ/kg)      | 16,800    | 17,700-19,600    |
| Moisture (%)     | 6-8       | 3-4              |
| Ash (%)          | 0.95      | 1.5-4            |
| Volatile (%)     | 62        | 45-55            |
| Fixed Carbon (%) | 16.9      | 37-46            |

3.1 Appearance of torrefied sample

In Figure 2, pictures of all the samples in every operation condition are exhibited. The color of OPF becomes darker from brown as the torrefaction temperature increases. The darker the biomass, the more weight loss in biomass [5,10]. When the time increased, the color also changes from dark to darker, not as significant as temperature affecting the samples. Meanwhile, sample does not show a significant change of color when nitrogen flow rate is increased.

3.2 The influence of time (a. 5; b.30; c.55 minutes) in operation condition 250°C of temperature and 100 ml/minute nitrogen flow rate

Calorific Value (HHV): The HHV ranged from 16,800-19,600 kJ/kg depending on torrefaction conditions. This implies that the HHV in the torrefied biomass increased by 5.4-16.5% when compared to the untreated material. The highest HHV obtained at temperature of 275 °C for 45 minutes reaction times and with 100 ml/minutes of nitrogen flow rate. The HHV significantly increased with torrefaction temperature than reaction time. This tendency is consistent with the previous work [7, 9], and is mainly due to the carbon content increase meanwhile the oxygen and hydrogen decrease. This decrease in hydrogen and oxygen is due to dehydration and de-carbondioxide from the biomass during torrefaction [10].
3.3. Proximate
Moisture is one of important value in determination of solid fuel. High moisture will cause a lot of energy lost in combustion process. The higher the moisture, the more the calor needed to release water in biomass. Moisture decreased when temperature of torrefaction increased, this is due to dehydration reaction during torrefaction [11]. In contrast, ash content increased when temperature of torrefaction increased. Ash in inorganic component in biomass. The ash of biomass is not lost during torrefaction, though the overall mass of the biomass is reduced. The absolute amount of ash in the feed does not change while the overall mass reduces when temperature increased. That is, the ash percentage changes after torrefaction. Volatile matter is closely related with fixed carbon in biomass.
Devolatilization was affected by torrefaction temperature and torrefaction time. The result was volatile matter decreased when temperature and time increased. This due to torrefaction temperature which is also the range of hemicellulose degradation temperature, so that it will be maximally degraded and produce volatiles that characterized by higher proportion of solid products [8]. This decrease causes an increasing for fixed carbon content in biomass due to ratio O/C and H/C decrease so C will increase toward total yield.

3.4. Response surface analysis

HHV was fitted to the response surface model provided by the mathematic model in order to analyze the effect of torrefaction factor on HHV, that is shown in Equation 1:

\[ Y_1 = 18782.5 + 534.5X_1 + 378.2X_2 \]  \hspace{1cm} (1)

Y₁ is the predicted HHV (kJ/kg), where X₁ and X₂ denote temperature and time. To fit the response surface function and experimental data, regression analysis was performed and the model for HHV was evaluated by analysis of variance which is presented in Table 2. Before analyzing, confidence level (α) was determined which is 5%. This value represents the maximum probability of error that may be resulted during experiment. Data processing was started by using second order model to see the curvature.

![Figure 3](image-url)

**Figure 3.** Response surface graphic for (a).HHV; (b) mass yield and in nitrogen flow rate of 100 ml/minutes
Table 2. Analysis of variance (ANOVA) for HHV of biomass during torrefaction

| Source                  | DF | Adj SS     | Adj MS     | F-Value | P-Value |
|-------------------------|----|------------|------------|---------|---------|
| Model                   | 7  | 5487050    | 783864     | 7.31    | 0.000   |
| Linear                  | 3  | 220771     | 1740257    | 16.23   | 0.000   |
| X₁                      | 1  | 3368923    | 3368923    | 31.42   | 0.000   |
| X₂                      | 1  | 1527060    | 1527060    | 14.24   | 0.001   |
| X₃                      | 1  | 324789     | 324789     | 3.03    | 0.097   |
| 2-Way Interactions      | 3  | 258420     | 86140      | 0.80    | 0.507   |
| X₁X₂                    | 1  | 54666      | 54666      | 0.51    | 0.483   |
| X₁X₃                    | 1  | 195371     | 195371     | 1.82    | 0.192   |
| X₂X₃                    | 1  | 8384       | 8384       | 0.08    | 0.783   |
| 3-Way Interactions      | 1  | 7858       | 7858       | 0.07    | 0.789   |
| X₁X₂X₃                  | 1  | 7858       | 7858       | 0.07    | 0.789   |
| Error                   | 20 | 2144459    | 107223     |         |         |
| Lack-of-Fit             | 1  | 513051     | 513051     | 5.98    | 0.024   |
| Pure Error              | 19 | 1631408    | 85864      |         |         |
| Total                   | 27 | 7631508    |            |         |         |

R² = 71.90%  R² (adj)= 62.06%  R² (pred) 33.02%

The HHV and mass yield results obtained for all runs of the experimental design were represented by response surface in Figure 3. The responses for HHV from the torrefaction were depicted as three dimensional surface plots of three factors; temperature, reaction time and nitrogen flow rate with their corresponding contours plots. The strength of the effect of these three parameters on HHV is revealed more clearly by the surface plot. Temperature had a higher impact on the HHV of torrefied lignocellulosic biomass, while the effect of reaction time was considerable lower. Nitrogen flow rate insignificantly affected the HHV. Table 2 shows ANOVA of the linear model adjustment, where the total error was classified into lack of fit and pure error. The F-value estimated using the experimental data corresponded to the total residual and lack-of-fit values, respectively, and was lower than the tubular F value. This indicates that the model was significant in the region studied. The mathematic models (Equation 1) shows in good correlation with the actual data as justified by the relatively high R-squared value. For the model, the p-value was <0.0001, which shows that the models were strongly significant at the 99% confidence level.

4. Conclusion
Biomass can be upgraded and used as a solid fuel by torrefaction process. The torrefied biomass is more suitable than raw biomass in terms of HHV, physical and chemical properties. While the reaction temperature had a strong impact on the HHV yield of torrefied lignocellulosic biomass, the effect of reaction time and carrier gas flow rate were considerably lesser. As a whole, the torrefaction at 220 °C just degraded small amount of holocellulose and gave a mediocre effect on improving the energy properties. When biomass underwent the torrefaction at temperatures above 250 °C, large amount of hemicellulosas and cellulose were degraded which contributed to the increment of acid-insoluble material. The highest HHV obtained was 19,600 kJ/kg and it increased about 16.5% from the raw biomass.
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