A pilot-scale study of wet torrefaction treatment for upgrading palm oil empty fruit bunches as clean solid fuel

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Abstract. Less utilized empty fruit bunch (EFB) is seldom used as solid biofuel due to its high alkali content that potentially cause ash deposit called slagging and fouling. This phenomenon could harm biomass-fired power plant equipment. Some pre-treatment of EFB is needed to reduce EFB ash deposit potential. The effect of wet torrefaction pre-treatment in laboratory scale was successfully proven in decreasing slagging and fouling potential while increasing EFB calorific value that could fulfill clean solid fuel criteria. This research focuses on wet torrefaction process that conducted on a pilot scale with the capacity of 250 liters. It was found that wet torrefaction process can improve the product’s calorific value up to 9.41% while reduce its ash content down to 1.01% comparing to the raw EFB. The reduction of ash content also leads to the reduction of slagging and fouling tendency that presents in terms of alkali index. Alkali index is a quantitative method that can be calculated after obtaining metal oxides fraction on solid fuel. Metal oxides could be obtained by using energy dispersive x-ray spectroscopy.

1. Introduction

As one of the largest renewable energy resources, it is expected that biomass could substitute the utilization of fossil fuel. Biomass stockpile, obtained from agricultural plant by-product process, is abundant and produced continually, less utilized, and nearly free [1]. In the other hand, biomass as an energy resources cannot directly utilized due to its high moisture content, bulk volume, low heating value, and also high alkali content [2]. High moisture content and bulk volume will decrease the combustion performance inside the furnace. Subsequently, low heating value makes biomass no suitable enough to substitute fossil fuel. Whereas high alkali content can bring harm risks of ash deposit called slagging and fouling [3]. Therefore, biomass needs a pre-treatment process to fulfill clean solid fuel condition.

Indonesia plays important role in palm oil industry. In period 2015/2016, Indonesia alone contributes over 32 million tonnes of palm oil and accounting for over than 54 percent of world’s total palm oil production [4]. Referring to the process flow diagram, it was found that only 21-23 wt. % oil palm produced from process plant and the rest is left as biomass residue [5]. The largest biomass solid residue produced from palm oil mill process is palm oil empty fruit bunch with the amount of 23-25 wt. % from each unit of fresh fruit bunch. Palm oil empty fruit bunch (EFB) is termed as palm oil bunches waste residue generated that has loosen its fruit after sterilization and threshing process [2].
One of the most promising pre-treatment process to obtain clean solid fuel criteria is wet torrefaction. Wet torrefaction or so-called hydrothermal carbonization is one of thermal degradation conversion process in a subcritical water as its medium [6]. Wet torrefaction aims to decompose lignocellulosic component in biomass such as hemicellulose, cellulose and lignin through hydrolysis between 180 to 280 °C [7].

Similar to other biomass, empty fruit bunch (EFB) has high alkali content that potentially cause problem such as ash deposition on biomass-fired plant equipment. Potassium element in alkali content that present in EFB act as principal bonding agents in most deposits [3]. Previous research has proved that wet torrefaction process could effectively remove chlorine content [8] and potassium content [9]. By date, wet torrefaction experiments to produce clean solid fuel on a pilot scale reactor by using quantitative criteria has not been conducted.

In this research, the authors employed an experimental investigation on wet torrefied solid products that has been proceed by using pilot scale reactor (250 liters). Indonesian EFB was chosen as the raw material. This research focuses on finding process parameters that could increase high heating value while decrease ash deposits potential. Calorific value were obtained by using adiabatic oxygen bomb calorimeter while major ash metal oxides composition were obtained by using energy dispersive x-ray spectroscopy.

2. Materials and methods

2.1. Materials
Sterilized and pressed empty fruit bunches (EFB) were used in this research. The EFB were taken from PT Perkebunan Nusantara VIII, Kertajaya, Banten, Indonesia. It was found that the EFB had 39.08% of moisture content (as received) and no further treatment is employed to the EFB.

2.2. Wet torrefaction experimental process
Wet torrefaction experiments were conducted by using 250 L pilot-scale reactor, consist of a body covered by Rockwool insulation, an oil heater, steam input from boiler, and direct contact steam condenser. During pilot-scale experiments, 15 kg of feedstock were supplied to the reactor with biomass-to-water ratio (BWR) of 1 : 3 and 1 : 5. 150 and 175 °C were chosen as the holding temperature, with variation of holding period of 30 minutes and 90 minutes.
EFB feedstock was loaded and mixed with certain amount of water, resulting water-to-biomass ratio parameter. Then, reactor heated until reaching the holding temperature. Holding temperature will be held for certain holding period. After completion, treated products were taken out from the reactor and dried at 40 °C for subsequent experiments and analysis.

2.3. Analytical procedures

2.3.1. Drying process
The drying process was tested referring to ASTM D 3173. The samples were dried naturally until the mass difference reached 0.01%. The samples crushed until it could pass through a 60-mesh sieve.

2.3.2. High heating value analysis
The calorific value of products was tested referring to ASTM D 5865. The preparation process of the samples referring to section 2.3.1. Parr adiabatic oxygen bomb calorimeter apparatus is used to determine the HHV of untreated EFB and all solid products derived from wet torrefaction experiment.
2.3.3. **Physical composition analysis**
The physical composition analysis of product was tested referring to ASTM D 3172. The preparation process of the samples referring to section 2.3.1. Leco TGA - 601 apparatus is used to determine the ash content of untreated EFB and all solid products derived from wet torrefaction experiment.

2.3.4. **Ash composition analysis**
The untreated EFB and all products derived from wet torrefaction experiment were heated inside 815 °C furnace. Then, ash composition of all samples were evaluated. The ash composition analysis was determined by using Energy Dispersive X-Ray Spectrometry (EDX) – 720 Shimadzu referring to IK-paskal-05-01-5B/3 standard.

2.3.5. **Alkali index calculation**
The empirical calculation were applied by using percentage of potassium oxide and the amount of ash present in the untreated EFB and wet torrefied solid products. The alkali index is showing the alkali oxides contribution to the potential level of slagging and fouling. Equation 1 describe the amount of alkali resulted the energy produce by solid fuel utilization [10].

\[ Ai (\text{lb alkali} / \text{MM BTU}) = \frac{1 \times 10^6}{\text{BTU/lb}} \times \% ash \times \% alkali on ash \] (1)

Alkali index below 0.4 lb/MM Btu is considered a fairly low slagging risk. Values between 0.4 and 0.8 lb/MM Btu will probably slag with increasing certainty of slagging as 0.8 lb/MM Btu value is approached. Above 0.8 lb/MM Btu, the fuel is virtually certain to slag and foul [10].

3. **Results and Discussion**

3.1. **Calorific Value Analysis**
Table 1 shows that all wet torrefied solid products had an increasing of HHV compared to raw material. According to Figure 1, the increased process temperature and holding period affected to HHV. This phenomenon can most likely happened due to lignocellulosic decomposition during the process.

| Process Parameters | HHV (J/g) |
|--------------------|-----------|
| Raw material       | 17,252    |
| 150 °C – 30 minutes – BWR 1 : 5 | 17,311 |
| 150 °C – 90 minutes – BWR 1 : 5 | 17,531 |
| 175 °C – 30 minutes – BWR 1 : 5 | 17,853 |
| 175 °C – 30 minutes – BWR 1 : 3 | 18,560 |
| 175 °C – 90 minutes – BWR 1 : 5 | 18,874 |

Lignocellulosic substances decomposition process plays significant roles in the increased of HHV. Lignocellulosic is mainly composed of three polymers: (1) cellulose, (2) hemicellulose, and (3) lignin. Hydrothermal experiment has been conducted to observe hemicellulose decomposition temperature by using wheat bran, spent grain, corn cobs, and eucalyptus [11]. It was found that at 150 °C – 160 °C, the solid products shown similar structure to xylose. Xylose is a substance derived from xylan through...
hydrolysis process. It can be concluded that hemicellulose is poorly resistant to hydrolysis and starts to decompose in the temperature of 150 °C into its monomers. The increased of HHV has a significant correlation with lignin fraction. Lignin has the largest HHV among the other lignocellulosic substituent and lignin is not easy to decompose due to its higher degradation temperature. At 175 °C followed by maximum holding period, the increased of HHV was found at its largest. This effect can be occurred because at higher operating temperature, the time required to hydrolyze hemicellulose into its monomers is shorter, which mean at the same holding period, solid products yield from 175 °C shown plenteous decomposed substances compared to solid products yield from 150 °C.

**Figure 1.** Temperature and holding period effect on HHV (BWR 1 : 5)

The increased of HHV, not only can be reached with the increased of holding period and temperature. Figure 2 showing that lower water to biomass ratio could lead to higher HHV. Feedstock with less amount of water also has less EFB fraction that submerged. Less water used during the process may lead to partial dry torrefaction condition.

**Figure 2.** Water to Biomass ratio (WBR) effect on HHV
The feasibility process of wet torrefaction in larger scale should be investigated by comparing several criteria in both lab-scale and pilot-scale. This step of study allows investigation of a product and process on an intermediate scale before commercially applied in the industrial-scale production. Characteristics of treated products in pilot-scale reactor can be used for further apparatus design development as well as predicting wet torrefied solid products behavior.

Upscaling study were done by comparing several criteria such as HHV and ash composition. From the previous experiment that has been conducted by using 2.5 L reactor, the HHV of solid product obtained from laboratory scale experiment in the temperature of 175 ºC, BWR 1 : 3, and holding period 30 minutes reached 18,502 J/g, thus had slightly lower value than the pilot scale. This difference happened due to the time required to reach the desired holding temperature. On laboratory scale experiment, heat energy source provided by electric heater with rated power of 1.5 kW. The time needed to reach 175 ºC from 35 ºC is approximately 25 – 27 minutes. Meanwhile on pilot-scale experiment, the time needed to reach 175 ºC from 164.1 ºC is 130 minutes, which means pilot-scale reactor has lower heat energy input. This phenomenon leads to hemicellulose degradation through hydrolysis reaction that considerably occurred before holding temperature is reached.

### 3.2. Physical composition analysis

According to Table 2, the ash fraction in the solid products are showing fluctuated result. This could be happened due to two reasons. The first one is the unknown rate of ash dissolution into the water comparing to other substances. Water act as a solvent for biomass substances, organic and inorganic substances that dissolved in a high rate and high amount must be evaluated. The second one is the presence of impurities in the solvent in form of metal oxides and/or metal hydroxides.

**Table 2. Ash Fraction of raw material and wet torrefied solid products**

| Process Parameters          | Ash (%db) |
|-----------------------------|-----------|
| Raw Material                | 5.62      |
| 150 ºC – 30 minutes – BWR 1 : 5 | 6.62      |
| 150 ºC – 90 minutes – BWR 1 : 5 | 4.61      |
| 175 ºC – 30 minutes – BWR 1 : 5 | 10.36     |
| 175 ºC – 30 minutes – BWR 1 : 3 | 12.24     |
| 175 ºC – 90 minutes – BWR 1 : 5 | 14.32     |
Figure 4. Temperature and holding period effect on ash fraction (BWR 1 : 5)

According to Figure 4, the increased of operating temperature leads to the increase of ash fraction in the solid product. Meanwhile, correlation between holding period and ash fraction is yet to be concluded. According to Figure 5, the ash fraction in the solid product reduced along with the addition of water. The addition of water will soak most fraction of the EFB and enlarge the amount of impurities that could dissolved into the water.

Figure 5. Water to biomass ratio (WBR) effect on ash fraction
According to Figure 6, pilot scale at the temperature of 175 °C, holding period of 30 minutes, and BWR of 1:3 showing higher ash fraction comparing to the solid product derived from the laboratory scale. The ash fraction derived from laboratory scale solid product was found at 4.47% (dry basis). This happened due to the amount of water that soaked the biomass. At pilot scale experiment, BWR of 1:3 did not soaked the biomass at all. Meanwhile, at the laboratory scale, BWR of 1:3 has been soaked the biomass. On pilot scale, there are a lot of solid biomass fraction that did not exposed to water directly and continuously. So, the only way to eliminate ash from the biomass is through metal evaporation mechanism, which the mechanism needs higher operating temperature. So that the process will be similar to dry torrefaction process, which is not using water for its thermal decomposition and inorganic matter solvent.

3.3. Empirical calculation of slagging and fouling tendency

Table 3. Metal oxides composition of raw material and wet torrefied solid products

| Compound | Raw Material | 150 °C - BWR 1:5 | 175 °C - BWR 1:5 | 150 °C - BWR 1:3 |
|----------|--------------|------------------|------------------|------------------|
|          | 30 Minutes   | 90 Minutes       | 30 Minutes       | 90 Minutes       | 30 Minutes |
| SiO₂     | 39.842       | 31.463           | 27.919           | 29.122           | 33.39     | 25.268 |
| K₂O      | 18.204       | 7.852            | 6.813            | 10.803           | 6.810     | 4.703  |
| CaO      | 21.665       | 16.437           | 15.294           | 15.579           | 11.914    | 10.075 |
| Fe₂O₃    | 2.695        | 34.635           | 41.303           | 35.326           | 39.102    | 52.396 |
| SO₃      | 5.353        | 3.662            | 3.0246           | 3.373            | 3.378     | 2.918  |
| P₂O₅     | 10.719       | 4.321            | 4.275            | 4.527            | 4.064     | 3.330  |
| MnO      | 0.073        | 0.778            | 0.775            | 0.966            | 0.719     | 0.524  |
| TiO₂     | 0.103        | 0.852            | 0.596            | 0.304            | 0.623     | 0.785  |

The ash fraction and metal oxides composition in Table 3 were used to evaluate the alkali index of raw material and wet torrefied solid products based on semi-quantitative analysis. Based on the empirical formula calculation, it was found that raw material have a very high potential of slagging and fouling.
Figure 7. Operating temperature and holding period effect on alkali index of wet torrefied solid products

Based on Table 4, raw material showing a very high slagging and fouling tendencies. Meanwhile, there are three parameters that showing reduction level of slagging and fouling potential when compared with the raw material. The first one is at the operating temperature of 150 °C, holding period of 30 minutes, and biomass to water ratio of 1 : 5 that showing high level of slagging and fouling tendency. The second one is at the operating temperature of 150 °C, holding period of 90 minutes, and biomass to water ratio of 1 : 5 that showing low level of slagging and fouling tendency. The third one is at the operating temperature of 175 °C, holding period of 30 minutes, and biomass to water ratio of 1 : 3 that showing high level of slagging and fouling tendency. According to Figure 7, the alkali index is increasing along with the increasing of operating temperature. In every temperature, longer holding period resulting in lower alkali index. This probably happened because longer holding period leads to longer dissolution reaction.

From the previous research, wet torrefaction experiment on laboratory scale reactor has been conducted to obtain the alkali index of wet torrefied solid products. The comparison of the alkali index is shown throughout Table 5. Based on Table 5, it can be concluded that wet torrefied solid products derived from pilot scale reactor is showing smaller value of alkali index, which means that the products will have smaller tendencies of slagging and fouling potentials.

Table 4. Alkali index of raw material and wet torrefied solid products

| Process Parameters | Alkali Index (lb Alkali/MM Btu) | Level of Severity | Annotation |
|--------------------|---------------------------------|-------------------|------------|
| Raw Material       | 1.022                           | Very High         | Not Selected |
| 150 °C – 30 Minutes – 1 : 5 | 0.520                          | High              | Selected    |
| 175 °C – 30 Minutes – 1 : 5 | 1.120                          | Very High         | Not Selected |
| 175 °C – 30 Minutes – 1 : 3 | 0.511                          | High              | Selected    |
| 150 °C – 90 Minutes – 1 : 5 | 0.314                          | Low               | Selected    |
| 175 °C – 90 Minutes – 1 : 5 | 0.975                          | High              | Not Selected |
Table 5. Alkali index of raw material and wet torrefied solid products from pilot scale reactor and lab scale reactor

| Process Parameters | Alkali Index (lb Alkali/MM Btu) from Pilot-Scale Reactor | Alkali Index (lb Alkali/MM Btu) from Lab-Scale Reactor |
|--------------------|----------------------------------------------------------|--------------------------------------------------------|
| Raw Material       | 1.022                                                    | 1.022                                                  |
| 175 °C – 30 Minutes – 1 : 3 | 0.511                                                    | 0.642                                                  |

The HHV, ash fraction, and alkali index of pilot scale solid product are compared in Table 6. The solid product yield from temperature 150 °C, holding period of 30 minutes, and BWR of 1 : 5 and also temperature 175 °C, holding period of 30 minutes, and BWR of 1 : 3 are showing significant result because it shown an increased HHV and reduced level of slagging and fouling potential compared to the raw material. Solid product yield from temperature 150 °C, holding period of 90 minutes, and BWR of 1 : 5 is much more preferred because it shown an increased HHV, reduced of ash fraction, and decreased slagging and fouling potential down to the lowest level.

Table 6. Raw material and wet torrefied solid products overall comparison

| Process Parameters | HHV (J/g) | Ash (%db) | Alkali Index (lb Alkali/MM Btu) | Level of Severity | Annotation   |
|--------------------|-----------|-----------|----------------------------------|-------------------|-------------|
| Raw Material       | 17,252    | 5.62      | 1.022                            | Very High         | Not Selected|
| 150 °C – 30 Minutes – 1 : 5 | 17,311    | 6.62      | 0.520                            | High              | Selected    |
| 175 °C – 30 Minutes – 1 : 5 | 17,853    | 10.36     | 1.120                            | Very High         | Not Selected|
| 175 °C – 30 Minutes – 1 : 3 | 18,560    | 12.24     | 0.511                            | High              | Selected    |
| 150 °C – 90 Minutes – 1 : 5 | 17,531    | 4.61      | 0.314                            | Low               | Selected    |
| 175 °C – 90 Minutes – 1 : 5 | 18,874    | 14.32     | 0.975                            | High              | Not Selected|

4. Conclusion

The comparison of the trend and result for laboratory scale and pilot scale on wet torrefaction using empty fruit bunch (EFB) have been evaluated. This study has shown the relation of operating parameters (temperature, holding time, and biomass-to-water ratio) to the calorific value, also the slagging and fouling tendency. The relation of operating parameter that has been obtained will be useful to get preferable operating conditions choice on the industrial level. Also with the comparison, we can assure the result similarity between laboratory scale and pilot scale through diagrams and trend lines.

The result shown that the increasing of temperature and holding time with less used of water, affected on the increasing of calorific value. On the same operating parameter, pilot-scale reactor result a slight higher calorific value due to its reactor heat rate. For overall characteristics that has been identified, holding temperature of 150°C, holding period of 90 minutes and BWR of 1 : 5 is chosen as the best operating parameter to produce solid product with low level of ash severity and an increasing value of HHV comparing to the raw material. The ash severity level successfully drop from very high down to low level while the HHV increase from 17,252 J/g up to 17,531 J/g.

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