Hydrothermal treatment, pelleting, and characterization of oil palm empty fruit bunches as solid fuel

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Abstract. Empty fruit bunch (EFB) is not yet utilized as a solid fuel for boiler due to their physical characteristics such as very bulky, high moisture and potassium contents. The hydrothermal treatment (HT) has been conducted in our research to overcome those problems. HT experiments were conducted in a 2 L digester, with 100 grams of EFB. The liquid to solid ratio was 5:1 mL/g. HT were conducted at temperature of 120, 150, 180, 200, and 220°C, and holding time of 60 minutes. The results indicated that the properties on solid products could be improved using HT, particularly its higher heating value (HHV) of 20.1 MJ/kg (obtained from HT at 180°C) compared to feedstock of 19.68 MJ/kg and commercial pellet of EFB of 13.03 MJ/kg. Pellets made of solid product from HT at 150°C and pelleted at 200 bar were considered to meet the characteristic of commercial pellets.

1. Introduction
Biomass from palm oil agro-industry is very prospective for new and renewable energy resource. In a crude palm oil mill, one ton of fresh fruit bunch produces approximately: 23%-mass empty fruit bunch (EFB), 6.5% palm kernel shell (PKS), and 13% fibre. PKS and fibre have been used internally in the CPO mill as fuel in boiler fuel. The use of EFB as a fuel for combustion and gasification is not maximum yet, due to some poor characteristics of EFB, such as high moisture content, high ash content, high alkali content in ash, low calorific value and low bulk density. Particularly, high ash and alkali contents in EFB may cause ash agglomeration, slagging and fouling in heat transfer tubes and other parts of boiler [1]. Therefore, EFB pre-treatment is needed to get better characteristic for solid fuel.

Hydrothermal Treatment (HT) is suitable for processing biomass with high moisture content without prior drying. Moreover, HT may reduce volatile matter in biomass and some alkali content such as K, Na, Mg, Ca, Mn and Fe [1-4]. HT process can be carried out for treating biomass waste order to reduce unutilized EFB waste and increased the added value of EFB. Many studies have been done to investigate HT process for various application.

The objective of this experimental study was improving the characteristic of EFB for its utilization as a solid fuel for gasification and combustion. The proposed improvement was the combination of HT and pelleting. During HT, potassium is expected to dissolve significantly into water, while pelleting is expected to increase the bulk density, and to make uniform particle size. Hopefully this HT and pelleting technique will make storage and transportation of EFB become more practical. In addition,
the hydrolysate (liquid product from HT) may content potassium which may be recovered as a source of fertilizer.

2. Experimental setup

2.1. Sample Preparation

EFB was obtained from a CPO mill in Lampung. Experiments consisted of preparation of sample, hydrothermal treatment process, making pellets and characterization of the pellet. EFB was firstly shredded into fibers having a length of about 4 cm. Then it was dried in the oven for five hours in an oven of 105°C. After drying the sample was kept the sample in a sealed plastic bag.

2.2. Hydrothermal Treatment

Hydrothermal treatment (HT) was conducted in a 2 L autoclave heated electrically. Temperatures of HT were set as an independent variable at 120, 150, 180, 200 and 220°C. The heating rate was about 4 °C/min, holding time at the desired temperature was 1 hour. The liquid and solid ratio were 5:1. One series of experiments were carried out with 500 mL water and 100 g EFB, and two series of experiments were carried out with 1000 mL water and 200 g EFB. After the HT process completed, the solid residue and the liquid separated using a filter. The solid residues were dried in an oven of 105°C for five hours, and this so-called biochar from HT was kept in sealed plastic bags for the proximate and ultimate analysis, and mineral content in ash. The liquid products of HT were kept in a sealed sample bottle for analysis of its potassium content.

2.3. Pelletization

Pellets were made manually in a mold with four holes. A pressure of 250 or 500 kg/cm² was applied using a hand hydraulic press. Each pellet had a diameter of 1 cm, the height of about 0.5 cm, and a weight of about 0.4 grams. This dimension of the pellet was similar to commercial EFB pellets obtained from Medan.

2.4. Pellet Characterization

Proximate analysis and ultimate analysis of solid residue were carried out in a certified laboratory, Research, and Development Center of a Mineral and Coal, the Ministry of Energy and Mineral Resources Indonesia (tekMIRA). HHV of products was also estimated using correlations based on the proximate and ultimate analysis. The correlations are presented in Table 1.

| No  | Name of author       | Correlation (HHV, MJ/kg)                                      |
|-----|----------------------|--------------------------------------------------------------|
| 1   | Jimenez et al, 1991  | HHV = −10.81408 + 0.3133 (VM+FC)                             |
| 2   | Demirbas et al, 1997 | HHV = 0.312*FC+0.1534*VM                                     |
| 3   | Cordero et al, 2001  | HHV = 0.3543*FC + 0.1708*VM                                  |
| 4   | Sheng et al, 2005    | HHV = −3.0368 + 0.2218VM + 0.2601*FC                         |
| 5   | Dulong               | HHV = 33.95 C + 144.2 (H – (O/8)) + 9.4 S                   |
| 6   | Boei, 1987           | HHV = 0.3516 C + 1.16225 H - 0.1109 O + 0.0628 N + 0.10465 S |
| 7   | Demirbars, 1997      | HHV = 0.335 C + 1.423 H – 0.154 O – 0.145 N                  |
| 8   | Channiwala, 2002     | HHV = 0.3491 C + 1.1783 H + 0.1005 S – 0.1034 O – 0.0151N – 0.0211 ash |
| 9   | Sheng, 2005          | HHV = −1.3675 + 0.3137 C + 0.7009 H + 0.0318 O               |

*Biomass composition, VM, FC, ash, C, H, O, N, S are in the weight percent on a dry basis
Minerals content in ash were measured using AAS and analyzed in Research and Development Center of Mineral and Coal, the Ministry of Energy and Mineral Resources Indonesia (tekMIRA). These minerals content in ash were used to estimate the potency of fouling and slagging of ash (formula for these indices are presented in Table 2).

| Index                      | Correlation, in weight % | Value and tendencies |
|----------------------------|--------------------------|----------------------|
| Base/acid ratio (B/A)      | (Fe₂O₃+CaO+MgO+Na₂O+K₂O) | <0.206 Low           |
|                            | (SiO₂+Al₂O₃+TiO₂)        | 0.206-0.4 Medium     |
| Fouling index (FI)         | (B/A) × (Na₂O+K₂O)       | >0.4 High            |
|                            | Sd = % of S from solid fuel |                      |
| Slagging Index (SI)        | SI = (B/A) × Sd          | <0.6 Low             |
|                            |                          | 0.6-2.0 Medium       |
|                            |                          | 2.0-2.6 High         |
|                            |                          | >2.6 Very High       |
|                            |                          | 0.17-0.34 Probable   |
|                            |                          | >0.34 High to Occurs |
| Alkali index (AI)          | AI = \( \frac{1000}{\text{HHV (MJ/kg-dry)}} \) × ash × (Na₂O+K₂O) |                      |

3. Result and Discussion
The HT temperature clearly influenced yields of bio-char (see Figure 1). Three series of experiments with different amount of feed gave similar trends in the decrease in yields of bio-char with increasing temperature. The decrease in yields of bio-char might be contributed by two factors, i.e. the dissolution of minerals in ash and thermal degradation of biomass. Thermal degradation of biomass could also be observed from significant changes in volatile matters and fixed carbon at HT temperatures of 200 and 220°C (Table 3). This range of temperature had been reported in many literatures as the initial temperature for thermal degradation of hemicelluloses. At the higher temperature of HT, cellulose and lignin in EFB might start to decompose to some extent. As a result, bio-char from HT at 200 and 220°C were more brittle and dark-black in color.
Figure 1. Biochar yield during hydrothermal treatment

In line with the proximate analysis, the ultimate analysis of bio-char also changed with the temperature of HT, although less significant (Table 3). Presumably, the elemental compositions of volatile matters were almost the same as those of raw EFB. An exception was observed again in bio-char obtained from HT at 220°C, i.e. a drastic decrease in carbon and oxygen contents.

Table 3. Proximate and ultimate analysis of HT solid products

| Parameters                  | Previous Work | Present work | Hydrothermal Treatment |
|-----------------------------|---------------|--------------|------------------------|
|                            | EFB [1]       | EFB [10]     | 120°C                  |
|                             |               |              | HT-120                 |
| Volatile Matter             | 78.70         | 78.34        | 77.22                  |
| Fixed Carbon                | 15.30         | 18.46        | 18.31                  |
| Ash                         | 5.90          | 3.20         | 4.47                   |
|                             |               |              | 150°C                  |
| Volatile Matter             | 78.34         | 79.42        | 74.22                  |
| Fixed Carbon                | 18.46         | 19.63        | 20.97                  |
| Ash                         | 3.20          | 2.85         | 4.82                   |
|                             |               |              | 180°C                  |
| Volatile Matter             | 79.42         | 20.97        | 24.13                  |
| Fixed Carbon                | 19.63         | 4.82         | 29.23                  |
| Ash                         | 2.85          | 4.82         | 4.02                   |
|                             |               |              | 200°C                  |
| Volatile Matter             | 74.22         | 4.82         | 24.13                  |
| Fixed Carbon                | 20.97         | 4.82         | 29.23                  |
| Ash                         | 4.82          | 4.02         | 3.74                   |
|                             |               |              | 220°C                  |
| Volatile Matter             | 71.85         | 4.02         | 24.13                  |
| Fixed Carbon                | 24.13         | 3.74         | 29.23                  |
| Ash                         | 4.02          | 3.74         | 3.74                   |

| Parameters                  | Hydrothermal Treatment |
|-----------------------------|------------------------|
| Carbon                      | 45.87                  |
| Hydrogen                    | 6.40                   |
| Oxygen                      | 46.94                  |
| Nitrogen                    | 0.78                   |
| HHV, MJ/kg                  | 19.68                  |
| HHV, MJ/kg *)               | 19.76                  |
| Potassium content in hydrolysate, mg/L | 1.534 |

*) calculated using Cordero formula (see Table 1)

Correlation proposed by Cordero, 2001 [5] was considered as the most optimum one since this formula used the low-cost proximate analysis and gave least errors [10]. Since the HHV of bio-chars (Table 3) were calculated based on both proximate and ultimate analysis, their values had the same tendency with the changes of this analysis as the above discussion.

Table 4. Ash content and its mineral composition of EFB and HT products
would change the physical properties of EFB, from originally tough in raw EFB to becoming brittle after HT. The higher temperature, HT will produce more brittle and darker color biochar. Comparison of bio-char pellets from our experiment and some commercial pellets are presented in Table 5. Commercial pellets were probably made of char from nearly complete pyrolysis, so it contained a low volatile matter. While our pellets still contained a large amount of volatile matter. In this respect, our pellets would probably have a lower ignition temperature.

The most interesting result in HT process was the significant change in the ash content in solid residue from HT (Table 4). Potassium (presented as K$_2$O) content in ash decreased, especially in HT with temperature of 220 °C. On the other hand, the concentration of SiO$_2$ in ash increased. This was probably due to the effects of large dissolution of potassium and sodium from EFB into water, while there was only slight dissolution of SiO$_2$.

Liquid products of HT contained a considerable amount of potassium (lower part of Table 3). Potassium is one of an important nutrient in oil palm plantation, so recovery of potassium from palm biomass will be very valuable. A research on recovery potassium from this dilute solution is being done.

Following the change in the minerals content in ash, ash melting properties change as well (see Table 2). It was surprising, that the tendencies of fouling and slagging of raw EFB used in these experiments were already low. These tendencies were similar to our previous work using different EFB from Riau [10], but they were different from data reported by [1] probably because of different palm species. Again, change in ash properties were observed significantly in HT with temperatures of 200 and 220 °C.

As expected, HT could change the physical properties of EFB, from originally tough in raw EFB becoming brittle after HT. The higher temperature, HT will produce more brittle and darker color biochar. Comparison of bio-char pellets from our experiment and some commercial pellets are presented in Table 5. Commercial pellets were probably made of char from nearly complete pyrolysis, so it contained a low volatile matter. While our pellets still contained a large amount of volatile matter. In this respect, our pellets would probably have a lower ignition temperature.

### Table 5. Combustion characteristic of pellets

| Pellet Parameters | Wood Pellets | Charcoal Briquette | Present Work |
|-------------------|--------------|--------------------|--------------|
|                   | EFB          | HT-120             | HT-150       |
|                   | HT-180       | HT-200             | HT-220       |
| SNI 8021-2014     | SNI 01-6235-2000 | Commercial EFB pellet |
| DIN 51731         |              |                    |              |

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4. Conclusions

HT and pelletization could improve the characteristic of EFB as a solid fuel by increasing fixed carbon, heating value, and bulk density. The decrease in minerals content in ash content could be expected to reduce the potencies of fouling and slagging during the use of EFB pellets in gasification or combustion process. EFB pellets made in this present work were better than commercial EFB pellets even without using adhesive substances. Our Hydrotreated EFB and EFB pellets also met standards of other commercial biomass pellets.

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