Production of Low-Potassium Solid Fuel from Empty Fruit Bunches (EFB) by Employing Hydrothermal Treatment and Water Washing Process

Anissa NURDIJAWATI※1, Srikandi NOVIANTI ※1, Ilman Nuran ZAINI ※1,
Hiroaki SUMIDA※2, Kunio YOSHIKAWA※1

(Received November 18, 2014)

Empty fruit bunch (EFB) can be used as a solid fuel for heat and power generation. However, high ash particularly potassium content can cause slagging, and fouling which negatively affect thermal conversion systems. A pretreatment process to remove potassium and achieve better solid fuel characteristics from EFB was proposed by employing the hydrothermal treatment (HT) followed by the washing process. EFB was treated via HT at the temperatures of 100, 150, 180 and 220 °C with the holding time of 30 min. A batch washing process was then conducted at different solid-product to liquid ratios. In this study, raw EFB and HT products were experimentally investigated in the laboratory to determine the effects of HT and the washing process on the potassium removal efficiency. Thermal behaviors of unwashed and washed products were also investigated. The results showed that the combination of HT-180 and washing can remove potassium up to 92 %, lowering the ash content down to 0.9 % and the chlorine content down to 0.19 % which led to a significant improvement in its thermal behavior with lowering the slagging tendencies. These results indicated that HT followed by the washing process is a viable way to obtain a low potassium content solid fuel.

Key Words
EFB, Fuel, Low-potassium, Hydrothermal treatment, Water washing

1. Introduction

Renewable energy and low CO₂ emission system are increasingly being considered as a response to the climate change and fossil fuel shortage concerns. Among the various sources of renewable energy, biomass is increasingly used as it is considered to be carbon neutral, more evenly distributed than finite fossil-fuel energy, and may be developed using more environmentally friendly technologies. However, some of the inherent problems with raw biomass materials, like low bulk density, high moisture content, hydrophilic nature, and low calorific value, limit its ease of use. In real power plants, combustion of biomass such as crop residue can cause some fouling of convection passes and severe deposits. Hence, fuel pretreatment is required to obtain better characteristics.

Empty fruit bunch (EFB), a byproduct of the palm oil industry, is being recognized as one of the most potential kinds of biomass for energy production in Southeast Asian and with Malaysia, Indonesia, and Thailand listed as top three. Indonesia, the world’s largest supplier of palm oil, produces about 20.25 million tons of palm oil per year while at the same time produces 22 million tons of EFB byproduct. Despite the abundant resource of EFB, as like other biomass, it also faces several limitations in its use. It has been reported that, in combusting EFB in boilers, some compounds evolving from abundant alkali metals in EFB into gas-phase condensate and deposit on low-temperature surfaces of heat exchange equipment, causing fouling and corrosion problems.

Depots prevention can be conducted by eliminating worst acting fuel components. Empty fruit bunches (EFB) has relatively high amount of potassium, 3-4 %-dry wt. Potassium is transformed during combustion and combines with other elements such as chlorine, sulfur and silica to form low melting point compounds. The potassium content, in particular, is important to indicate potential ash deposition...
through vaporization and condensation. Over 90% of the potassium in clean (non-solid) fuels occurs as either water soluble or ion exchangeable material. Therefore, potassium can be removed by pretreatment using water.

A number of authors have proposed a controlled washing or leaching process prior to conversion to remove troublesome elements from biomass. Removing elements after initial primary conversion (e.g. char wash) has been proposed as well.

The hydrothermal treatment (HT) is known for converting high moisture content solid wastes into dried, uniform, pulverized, coal-like solid fuel. Additionally, HT also can remove inorganics such as Ca, S, P, Mg, K, Fe, and Mn from biomass.

The combination of the two pre-treatments, HT, and washing, is worth to investigate to maximize the effectiveness in increasing physical characteristic, fuel properties as well as reduce ash content particularly potassium element of EFB.

2. Experimental

2.1 Sample Preparation

The EFB samples were collected from a palm oil mill in Malaysia. The samples were received in dried milled condition from Yonsei University, Korea. The dried milled samples were prepared into the particle size of less than 10 mm long and kept in sealed plastic bags at the room temperature until the use for pretreatment.

2.2 Hydrothermal treatment

HT was carried out in a lab scale facility, a 500 mL batch type autoclave reactor, at four temperatures of 100, 150, 180 and 220°C. The temperature of the reactor was controlled using a PID controller. The reactor pressure was not controlled but indicated by the pressure gauge. Fig. 1 shows the experimental apparatus. For each run, the reactor was loaded with 20 g of the sample and distilled water in 1:10 biomass-water ratio under the atmospheric pressure. Air in the reactor was purged with nitrogen gas to give an inert atmosphere inside the reactor. The reactor was then heated by an electrical heater to achieve the target temperature and kept for the holding time of 30 min. After completed, the product was discharged from the reactor. The solid part was separated from the liquid by using the vacuum filtration, then oven dried at 105°C for 24 h, and stored in a sealed bag before further experiment and analysis. The liquid part was filled in the bottle and kept in a refrigerator. The condensate was also collected and refrigerated prior to the analysis.

2.3 Water washing process

The washing process was studied applying several process parameters. Different hydrochars and solid-liquid ratios were applied. The washing processes were conducted by heating a certain ratio of distilled water to 60°C and adding 3 g of the sample. The sample and water were mixed with a magnetic stirrer rotating at 600 rpm. Five samples: raw EFB, hydrochar produced at 100°C (HT-100), HT-150, HT-180, HT-220 were used in the washing experiment. The biomass to distilled water ratio during the washing was 1:5, 1:8, 1:10, 1:20 and 1:50. The investigated leaching duration was 15 min. After the washing process, the mixture was separated using a vacuum filter. The solid was oven dried at 105°C for 24 h, and stored in a sealed bag. HTW denotes for the hydrothermally treated and washed sample.

2.4 Analyses

2.4.1 Atomic absorption spectrophotometry

A Hitachi Z-5010 Polarized Zeeman Atomic Absorption Spectrophotometer was used for inorganic analysis, particularly potassium element. Acid digestion was used to dissolve solid samples for Atomic Absorption Spectrophotometry. A volume of 3 mL HNO₃ 1.38 g/mL was added to 0.5 g of the dry solid sample or 2 mL of the liquid sample. A volume of 5 mL HClO₄ 60% was added to the solution to dissolve SiO₂.

2.4.2 Ultimate analysis and HHV

The determination of the CHNS and Cl of samples was performed using Vario Micro Cube Elemental Analyzer (Elementar, Germany).

The ultimate correlation by Channiwala and Parikh (2002) was used to estimate the higher heating value (HHV) of the samples. Kieseler’s study showed that this correlation gives the most accurate result compare to proximate correlation and Dulong-formula ultimate correlation for hydrothermal treatment chars. The correlation is given...
below:

\[ \text{HHV} = 0.3491\ C + 1.1783\ H + 0.1005\ S - 0.1034\ O - 0.0151\ N - 0.0211\ Ash \] (1)

2.4.3 Ash composition

The fuel elemental composition and the concentration of alkali, sulfur, chlorine and silica in the fuels appear to be the best indicators of the tendency of fuels to slag. The elemental and composition analysis were determined by using S2 Ranger energy dispersive X-ray fluorescence spectrometer (Bruker AXS, Germany).

2.5 Data Analysis

In order to analyze and compare the extent of element removal, the removal efficiency is defined as,

\[ \text{Efficiency} = \frac{m_i - m_f}{m_i} = 1 - \frac{x_i}{x_f} \] (2)

\( m_i \) = initial mass

\( m_f \) = final mass

\( x_i \) = initial concentration

\( x_f \) = final concentration

Several indices of ash and fouling were defined in Table 1.

3. Results and Discussion

3.1 Fuel properties of hydrothermal and washed products

Results of ultimate analyses for various treatments are listed in Table 2. The result showed that HT process can reduce ash content in raw EFB. Raw EFB has an ash content of 4.9 %, while after HT at 180 °C it has 2.2 % of ash. Inorganic material either as loose dirt from harvesting or loosely bonded in a crosslinked matrix is removed during this process leading to the decrease of the ash content. There is also some expectation that additional acidity produced in HT may solubilize and remove inorganics.

Table 2 Ultimate analysis

| Element   | Weight Percentage [wt%], dry | Raw Unwashed | Raw Washed | HT-100 Unwashed | HT-100 Washed | HT-150 Unwashed | HT-150 Washed | HT-180 Unwashed | HT-180 Washed | HT-220 Unwashed | HT-220 Washed |
|-----------|-----------------------------|--------------|------------|-----------------|---------------|-----------------|---------------|----------------|---------------|----------------|---------------|
| Ash       |                             | 4.9          | 2          | 4.6             | 1.1           | 3.4             | 3.4           | 2.7            | 0.9           | 4.1            | 27            |
| Carbon    |                             | 43.56        | 45.02      | 44.07           | 44.78         | 45.25           | 45.05         | 46.43          | 46.82         | 49.98          | 50.74         |
| Hydrogen  |                             | 5.34         | 5.79       | 5.24            | 5.42          | 5.51            | 5.52          | 5.57           | 5.88          | 5.38           | 5.46          |
| Nitrogen  |                             | 0.56         | 0.44       | 0.79            | 0.80          | 0.59            | 0.63          | 0.4            | 0.26          | 0.77           | 0.73          |
| Sulfur    |                             | 0.11         | 0.07       | 0.08            | 0.09          | 0.09            | 0.04          | 0.03           | 0.04          | 0.09           | 0.06          |
| Chlorine  |                             | 0.67         | 0.5        | 0.23            | 0.13          | 0.26            | 0.12          | 0.18           | 0.19          | 0.5            | 0.3           |
| Oxygen*   |                             | 44.86        | 46.18      | 44.99           | 45.68         | 44.94           | 45.94         | 45.19          | 45.23         | 39.18          | 40.01         |
| Total     |                             | 100          | 100        | 100             | 100           | 100             | 100           | 100            | 100           | 100            | 100           |
| HHV [MJ/kg-dry] |   | 16.69        | 17.67      | 17.78           | 17.21         | 17.55           | 17.41         | 18.03          | 18.49         | 19.60          | 19.92         |
| Energy Yield** |            | 79%          | 76%        | 81%             | 75%           | 82%             | 79%           | 66%            | 65%           |                |               |

*) by difference **) [HHV product x mass yield]/HHV raw
extractives have been reacted, and much of the cellulose has reacted as well. Porous structure might absorb some inorganics, which might explain the increase in the ash content from HT-180 to HT-220.

Additional washing process can remove more ash down to less than 1% for HT-180 hydrochar. Since the end-use of hydrochar is for power generation, the ash content of the feedstock was the main concern for the combustion process. For pellet production, according to the Pellet Fuels Institute, the most common pellets currently on the market must have an ash content of less than 1%, whereas “standard” pellets may have as much as 2% ash.

HHV increases with the increase of the HT temperature. Since the washing process can remove more ash, a slight increase of HHV in washed product was observed.

Higher reaction temperature produces relatively higher HHV especially at HT-220, but considering energy yield and ash content of the product, HT at 180°C seems more favorable for the large-scale production of solid fuel from EFB.

3.2 Effect of hydrothermal and washing pretreatment on potassium and chlorine removal

A high concentration of potassium in biomass fuel tends to result in the easy formation of compounds with low melting points. Thus, the potassium content, in particular, is important to indicate potential ash fusion or ash deposition. Alkali and alkaline earth metals (sodium and potassium) occur in organic structures. They are usually deposited as a water-soluble fraction, so they are easily removed by the water washing.

Fig. 2 shows the potassium concentration in the solid product and the removal efficiency of each treatment. Raw EFB contains around 32 g/kg or 3.2% of potassium. By washing with 1:10 biomass-water ratio, up to 54.9% of potassium in raw EFB can be removed. Applying HT can enhance the potassium removal process. By increasing the severity of the HT condition, more potassium can be released where about 20% is still remained in the solid. By combining HT and the washing, the removal efficiency can be enhanced to above 80% and even 92% in the case of HT 180°C.

Chlorine as like potassium and sodium is commonly found in the form of water-soluble salts and in low quantities as organic compounds. From Table 2, it can be seen that chlorine is also much reduced about 72% after employing HT and washing.

Fig. 3 shows the potassium concentration and the removal efficiency of HT-180 sample washed with various biomass/water ratios ranging from 1:5 to 1:50. It can be observed that using solid-liquid ratio less than eight resulted in insignificant difference of the potassium release. By using the water ratio up to 1:50 in the washing stage, 95% of potassium in EFB can be removed. The increase of the water volume will decrease the concentration of waste liquor, so, potassium will be easily diffused. However, the addition of water into the biomass should be optimized because of the higher energy input required to dry the biomass prior to combustion.

3.3 Mass balance model on potassium removal

Fig. 4 shows the potassium distribution after the combined pretreatment of HT and washing. It can be seen that considering the characteristic of potassium in biomass which is easily water-soluble, only less than 20% of potassium is retained in the solid product. Besides potassium, another nutrient such as phosphorus, sulfur, nitrogen might also move to the liquid side making it valuable for plant growth. Hence, the probability of using...
leachate as fertilizer should be further studied.

During the HT process, condensed water was obtained. This condensed water contains only slight potassium which can be re-used as make-up water in the washing process in order to minimize the water requirement. Application of HT where washing can be integrated would seem more feasible from a practical point of view.

3.4 Ash composition

Table 3 shows the ash elemental composition in oxide forms. Alkali levels are very high in the raw EFB (about 50%) which make this biomass prone to cause fouling and slagging.

Employing HT and washing can change the relative composition significantly where the alkali and alkali metal were much reduced. However, elements such as SiO2, and Al2O3 only have a slight change after both processes. Si is very stable which is covalently bonded within biomass' organic matrix.

Table 3 also shows the slagging and fouling tendencies of the raw EFB, HT-180, and HTW-180 based on indices defined in Table 1. Except for the slagging index (SI), all the indices were clearly shown that raw EFB has high tendency of deposition. The SI value for raw EFB was low due to low sulfur content in the biomass. Base component is more dominant than acid in all products, which results in high probability indices. However, from IA, IF, and CI indices, the effect of HT and washing is more pronounced in reducing the deposition probability. After HT-180, the IF and CI of the EFB can be reduced to a low probability regime showing the low tendency of deposition. Additional washing process shifts the initially high occurrence of slagging and fouling to a probable occurrence represented by IA value.

4. Conclusion

The hydrothermal treatment followed by the washing process is a viable process for upgrading the fuel characteristics of biomass as well as lowering slagging and fouling tendency in the end-use application. Combination of HT and washing can remove up to 92 % of potassium and lowering ash to 0.9 % and chlorine content to 0.19 % in EFB. Combination of HT-180 and washing showed effective removal of alkali content, particularly potassium in EFB, resulting in a low fouling index and alkali index.

References
1) Arias, B. R.; Pevida, C. G.; Fermoso, J. D.; Plaza, M. G.; Rubiera, F. G.; Pis Martinez, J. J., Fuel Process. Technol.
2) Miles, T. R.; Miles, TR jnr.; Baxter, L. L.; Bryer, R. W.; Jenkins, B. M.; Oden, L. L., Alkali Deposits Found in biomass power plant: A preliminary investigation of their extent and nature, Volume II, National Renewable Energy Laboratory, Golden, Co. US, 1996

3) Jensen, P. A. et al., Biomass Bioenergy, 20, 431-446 (2001)

4) Reza, M. T.; Lynam, J. G.; Helal Uddin, M.; Coronella, C. J., Biomass Bioenergy, 49, 86-94 (2013)

5) Nakhshiniev, B.; Gonzales, M. K.; Yoshikawa, K., Compost Sci. Util., 20(4), 245-253 (2012)

6) Kieseler, S.; Neubauer, Y.; Zobel, N., Energy Fuels, 27, 908-918(2013)

7) Lynam, J. G.; Coronella, C. J.; Yan, W.; Reza, M. T.; Vasquez, V. R., Bioresour. Technol., 102(10), 6192-9 (2011)

8) Pellet Fuels Institute, PFI Standard Specification for Residential/Commercial Densified Fuel, Arlington, Va.: Pellet Fuels Institute, (2008)

9) Masia, A. A. T.; Buhre, B. J. P.; Gupta, R. P.; Wall, T. F., Fuel Process. Inst., 88(11-12), 1071-81(2007)