Upgrading of Biochar through Co-torrefaction of Acid-Pretreated Oil Palm Empty Fruit Bunch and Used Cooking Oil

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Received: 20.11.2023 Accepted:23.12.2023

Abstract- Oil palm empty fruit bunches (OPEFB) can be used as solid fuel in boiler units. However, this biomass contains high potassium which can cause fouling and slagging on the tubes and boiler walls. The pretreatment method that can reduce the potassium in OPEFB is washing with acidic solutions. Peat water is acidic water and has the potential as a cleaning water for OPEFB. Furthermore, the calorific value of OPEFB can be increased through the co-torrefaction of the OPEFB with other materials having high calorific values. Used cooking oil has a higher calorific value than OPEFB. Current research aimed to examine the influence of the pretreatment of OPEFB with peat water, the ratio of pretreated OPEFB to used cooking oil in the co-torrefaction, and the temperature of the co-torrefaction on the quantity and quality of biochar products. The results showed that the best conditions were the pretreatment with peat water at 50°C for 30 minutes, the ratio of OPEFB:used cooking oil of 2:1 in the co-torrefaction, and the co-torrefaction temperature of 250°C, resulting in biochar with a mass yield of 52.78 wt.%, a calorific value of 34.36 kJ/g, and an energy yield of 91.16%.

Keywords Biochar; co-torrefaction, oil palm empty fruit bunches, peat water, used cooking oil.

1. Introduction

Current energy needs are predominantly met by fossil energy resources, such as petroleum, natural gas, and coal [1]. The increase in the use of fossil energy causes an increase in greenhouse gas emissions, causing an unstable climate and increasing the temperature of the earth and the surface level of the sea [2]. Therefore, renewable energy is urgently developed to reduce the consumption of fossil energy, thereby reducing the bad environmental impact due to fossil energy consumption [3].

The dependence on fossil energy can be reduced by developing renewable (non-fossil) energy based on renewable biomass materials [4]. Oil palm empty fruit bunches (OPEFB) are one of the most common biomass in Indonesia [5]. The area of oil palm plantations in Indonesia is around 14.32 million hectares, where every 1 hectare of oil palm plantations produces around 1.5 tons of OPEFB [6]. However, the utilization of waste biomass as an energy source faces obstacles because the biomass in its raw condition (without treatment) generally has a lower energy density, a higher moisture content, a higher volatile matter, and a higher ash content compared to fossil fuels such as coal [7].

Torrefaction is one of the methods for the initial processing of biomass to improve biomass quality. This process can reduce unwanted volatile materials, such as nitrogen oxides and sulfur oxides [8]. Furthermore, one of the pretreatment methods to improve the biomass quality is the acid pretreatment process using acid solutions which can remove large amounts of metal minerals [9]. Peat water contains humic acid which can dissolve minerals such as potassium [10].

Co-torrefaction technique is the latest technique that can be employed to improve the quality of biochar. In the co-torrefaction process, biomass is heated together with other materials that have a higher calorific value or carbon content at a temperature of 200-300ºC. Used cooking oil has a higher carbon content and calorific value than OPEFB [11].

Research on biomass torrefaction has been carried out by several researchers. Rasid and Thaim [12] performed torrefaction of OPEFB at 320ºC for 90 minutes. The results showed that volatile matter decreased by 69%, the value of fixed carbon (FC) increased by around 69.71%, and the heating value of biochar increased by 27.31% after torrefaction. Meanwhile, Faizal et al [13] carried out torrefaction of mesocarp fiber briquettes at 300ºC for 40 minutes and obtained a biochar mass yield of 50 wt.% with a calorific value of 23.38 MJ/kg. Kitrungloadjanaporna et al [14] performed torrefaction of betel nutshells at 300ºC for 60 minutes and produced a biochar mass yield of 27.50 wt.% with a calorific value of 25.20 MJ/kg.

Furthermore, research on co-torrefaction was conducted by Yek et al. [11] who performed co-torrefaction of unpretreated OPEFB and used cooking oil. The research showed that there was a decrease in ash content of around 5–6 wt.%, an increase in fixed carbon (FC) content of around 33–63 wt.%, and an increase in carbon content of around 47.3–60.5 wt.%. Furthermore, the calorific value of the biochar product became 24.4 MJ/kg. However, that calorific value is still lower than the calorific value of bituminous coal.

In this study, OPEFB was pretreated with peat water before the co-torrefaction process. Soaking OPEFB in peat water is expected to reduce the ash content, especially potassium. Co-torrefaction of pretreated OPEFB and used cooking oil is expected can result in biochar with a high calorific value which is equivalent to high-grade coal. The aims of this study were to examine the influence of the pretreatment of OPEFB with peat water, the ratio of pretreated OPEFB to used cooking oil in the co-torrefaction, and the co-torrefaction temperature on the quantity and quality of biochar products.

2. Materials and Methods

2.1. Materials

The raw materials used in this research were the oil palm empty fruit bunch (OPEFB), used cooking oil, peat water, and nitrogen gas. Physico-chemical characteristics of OPEFB and used cooking oil can be seen in Table 1.

2.2. Pretreatment of POEFB and Used Cooking Oil

Oil Palm Empty Fruit Bunches (OPEFB) were cut into pieces with a diameter of about 10 mm and a length of about 20 mm. Then, the OPEFB was mixed with peat water in a 1000 mL beaker glass and heated at 30-50ºC for 30, 60 and 90 minutes using a heater equipped with a temperature control. After that, the OPEFB was separated from the peat water and dried with an oven at 105ºC for 12 hours. The pretreatment of the used cooking oil was carried out by a filtration process using 5 mm filter paper. After that, the used cooking oil was put in the oven at 100ºC for 4 hours.

Table 1. Physico-chemical characteristics of OPEFB and used cooking oil

Sample		OPEFB	Used Cooking Oil
Proximate analysis	Volatile matter (wt.%)	82.2	98.7
	Fixed carbon (wt.%)	13.5	0.6
	Ash (wt.%)	4.3	0.7
Ultimate analysis	Carbon (wt.%)	47.54	72.14
	Hydrogen (wt.%)	7.11	11.4
	Nitrogen (wt.%)	0.62	0.07
	Sulfur (wt.%)	-	0.008
	Oxygen (wt.%)	39.82	16.38

2.3 Torrefaction and Co-torrefaction Processes

Treated OPEFB was mixed with the used cooking oil with OPEFB/used cooking oil weight ratios of 1:0, 1:1, 2:1 and 3:1. The raw materials of 50 grams were fed into the co-torrefaction reactor and heated at 200-300ºC with nitrogen gas flowing for 60 minutes. The detailed experimental equipment set-up is shown in Fig.1.

2.4 Analyses of Products Co-Pyrolysis

Biochar products were weighed to determine their mass yield. The calorific values of biochar were measured using a bomb calorimeter. Pottasium content in OPEFB ash were determined using X-ray fluorescence (XRF). The analyzes of proximate and ultimate were carried out with TGA Q500IR and a LECO-932 elemental analyzer.

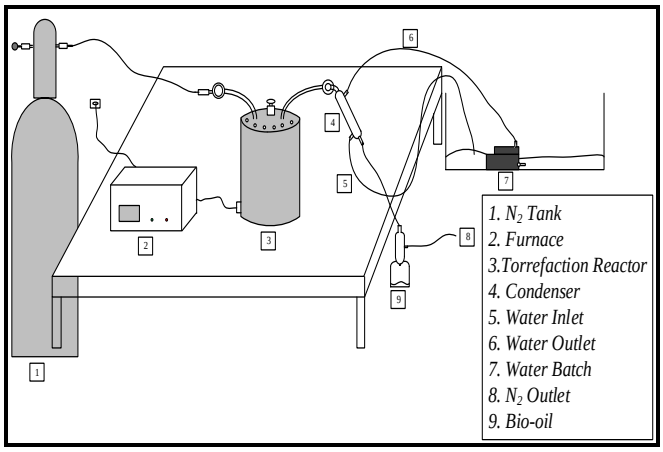


Fig.1. Co-torrefaction reactor used for upgrading of biochar.

3. Results and discussion

3.1. Effect of Pretreatment of Oil Palm Empty Fruit Bunches (OPEFB) on Ash and Potassium Content

The The effect of the pretreatment of OPEFB with peat water is presented in Fig.2. Based on Fig.2, the pretreatment successfully decreased the ash content in OPEFB. Furthermore, an increase in temperature in the pretreatment process decreased the ash content in the OPEFB. This finding was in line with the finding of research conducted by Di Blasi et al. [15] reported that pretreatment of straw with hot water for 10 minutes resulted in pretreated straw with 3.5 wt.% ash content.

Differences in soaking time during pretreatment with peat water also affected the ash content in OPEFB, as shown in Fig.2. The higher the soaking time was set, the lower the ash content would be. This was because the longer the OPEFB was soaked in the peat water, the more alkaline earth metals, especially potassium could be dissolved. The results of this research were in line with those of research conducted by Abdullah and Sulaiman [16].

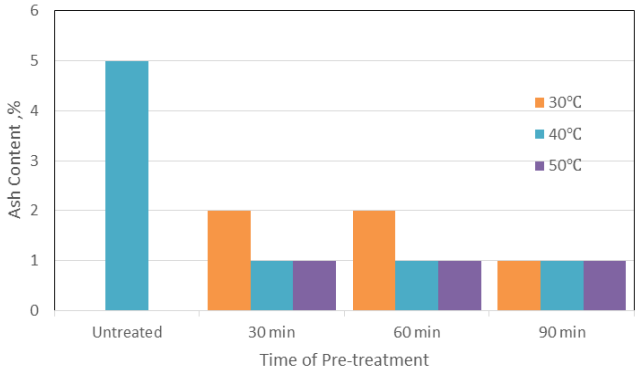


Fig. 2. Effect of Pretreatment on Ash Content in POEFB.

After the ash content analysis, several samples were analyzed using XRF analysis to examine the potassium and potassium oxide contents in OPEFB. The potassium and potassium oxide can cause fouling and slagging in the boiler when the OPEFB is used as fuel. The results of the potassium and potassium oxide analyses are presented in Fig.3. Based on Fig.3, the pretreatment of OPEFB with peat water can lower the amounts of potassium (K) and potassium Oxide (K_2O) contained in OPEFB. Samples without pretreatment contained potassium (K) of 50.46 wt.% and K_2O of 44.61 wt.%. When the OPEFB was immersed in peat water at a temperature of 50°C for 30 minutes, the potassium (K) and K_2O contents decreased to 12.75 wt.% and 11.07 wt.%, respectively. Meanwhile, through pretreatment with peat water at a temperature of 30°C for 90 minutes, the potassium (K) and K_2O contents decreased to 32.54 wt.% and 26 wt.%, respectively. The decrease in potassium and potassium oxide content after pretreatment was because the ash containing potassium and other minerals was dissolved in peat water during the soaking process. Peat water contains humic acid and fulvic acid and has mineral adsorption properties. Therefore, potassium can be dissolved more easily and completely in peat water than in ordinary water [10]. The finding of this study was similar to the finding of a previous study conducted by Abdullah and Sulaiman [14] who carried out the treatment by washing OPEFB with running water with varying flow rates. The optimum flow rate of 5 liters per minute (lpm) can decrease the potassium content in OPEFB from 55 wt.% to around 42 wt.%.

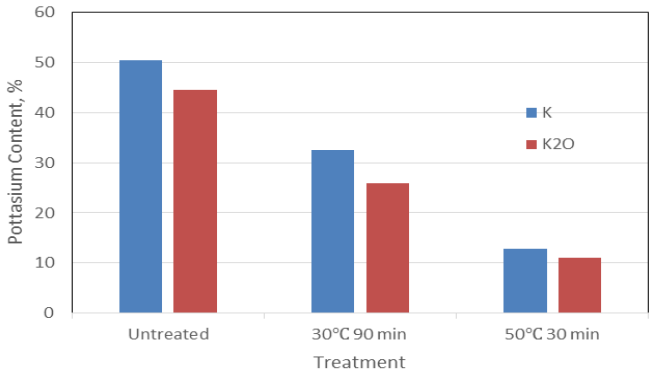


Fig. 3. The Effect of Pretreatment on Potassium Content in OPEFB.

3.2. The Effects of Ratios of Pretreated OPEFB to Used Cooking Oil in Co-Torrefaction on Quantity and Quality of Biochar

The co-torrefaction process was carried out by varying the ratio of pretreated OPEFB and used cooking oil of 1:1, 2:1, and 3:1 (wt/wt). The pretreated OPEFB resulting from the pretreatment with peat water at 50°C for 30 minutes was used in this stage. The pretreated OPEFB and used cooking oil were weighed according to each ratio variation with a total weight of 50 grams. Then, the materials were mixed and stirred evenly before being put into the torrefaction reactor. The co-torrefaction process was operated at 250°C for 1 hour. Furthermore, the resulting biochar was analyzed to examine

the mass yield, energy yield, and heating value. The results of the analyses are shown in Fig.4.

Figure 4 shows the influence of the ratio of pretreated OPEFB to used cooking oil on biochar products in the co-torrefaction process. The ratios of 1:0, 1:1, 2:1, and 3:1 resulted in biochar with mass yield of 36.43 wt.%, 56.06 wt.%, 52.78 wt.%, and 43.08 wt.%, respectively. When the used cooking oil was added, the mass yield of biochar increased. This was because the volatile compounds from pretreated OPEFB were blocked by the layer of used cooking oil during the co-torrefaction and reacted again to form biochar. This was commensurate with Rago et.al [17] who carried out co-torrefaction of biomass and LDPE plastic.

The addition of used cooking oil also affected the calorific value of the resulting biochar. Biochar resulting from the torrefaction of pretreated OPEFB without the addition of used cooking oil (ratio of pretreated OPEFB:used cooking oil = 1:0) had a calorific value of 25.27 kJ/g (Fig. 4). Then, the co-torrefaction was carried out with a ratio of pretreated OPEFB : used cooking oil of 1:1, 2:1, and 3:1, which increased the calorific value of biochar to 26.51, 34.36, and 32.29 kJ/g, respectively. This was because the addition of used cooking oil as a co-feedstock increased the carbon and hydrogen content in biochar, which consequently increased the calorific value of the resulting biochar. Lam et al. [18] explained that the addition of used cooking oil which has a long hydrocarbon chain plays a role in increasing the calorific value.

Figure 4 also shows that the addition of used cooking oil can increase the energy yield of the resulting biochar. Based on Fig. 4, the energy yield at a ratio of 1:0 was 46.27%. Then, at ratios of 1:1 and 3:1, the energy yields were 74.7% and 69.93%, respectively. Furthermore, a ratio of 2:1 resulted in the highest energy yield value which was 91.16%. The energy yield value was influenced by the mass yield and calorific value of the biochar. Lin and Zheng [19] reported that during the co-torrefaction process, a thermochemical reaction occurs to degrade the raw material but at the same time increase the calorific value (HHV) and energy yield of the products. Thus, the co-torrefaction produces biochar with better quality than the torrefaction.

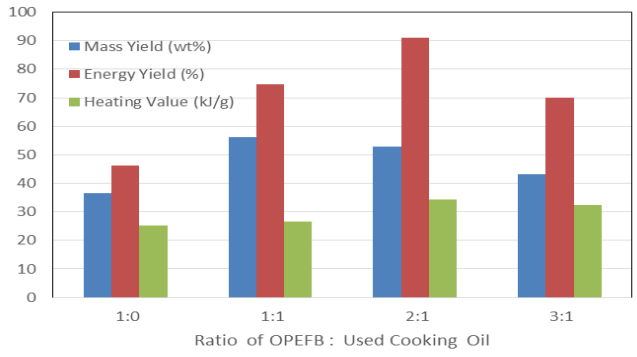


Fig. 4. The effect of the ratio of pretreated OPEFB : used cooking oil on biochar products.

3.3. The Effects of Co-torrefaction Temperature on Quantity and Quality of Biochar

The co-torrefaction process was conducted at various temperatures of 200, 250, and 300°C. In this process, the ratio of 2:1 was chosen because it was the optimum ratio based on subsection 3.2. The mass yield, energy yield, and heating value of the biochar resulting from the co-torrefaction process at various temperatures are shown in Fig.5.

Figure 5 shows that the mass yield of biochar decreased with increasing the operating temperature, namely 63.22 wt.% at 200°C, 52.78 wt.% at 250°C, and 36.94 wt.% at 300°C. The breakdown of hemicellulose and cellulose during the co-torrefaction process released volatile materials, which resulted in a decrease in mass yield. This research was in line with research conducted by Yek et al [11]. However, an increase in operating temperature increased the calorific value of the resulting biochar. The biochar at temperatures of 200, 250, and 300°C had heating values of 28.16 kJ/g, 34.36 kJ/g, and 37.049 kJ/g, respectively. An increase in operating temperature can increase the carbon content in the biochar, followed by a decrease in water content and volatile matter (VM) content [13]. Based on this research, the highest heating value was obtained at an operating temperature of 300°C with a biochar heating value of 37.05 kJ/g. This value was much higher than that in research conducted by Yek et al [11] with a heating value of 24 kJ/g. Li et.al [20] torrefied corncob under nitrogen atmospheres at 220–300°C and obtained biochar products with heating values of 16.58–24.77 kJ/g. Apart from that, Manatura et. al [21] carried out co-torrefaction of straw and medium-density fiberboard (MDF) and obtained biochar with a heating value of 22.13 kJ/g.

Figure 5 also shows that the energy yield of biochar resulting from the co-torrefaction at operating temperatures of 200, 250, and 300°C was 89.49%, 91.16%, and 68.79%, respectively. This occurred due to the decrease in biochar weight during the co-torrefaction process. In general, the addition of used cooking oil increased energy yield, whereas hydrocarbons in the used cooking oil tended to contribute to increasing the energy content of biomass resulting from the co-torrefaction. This is because used cooking oil has a higher heating value than OPEFB.

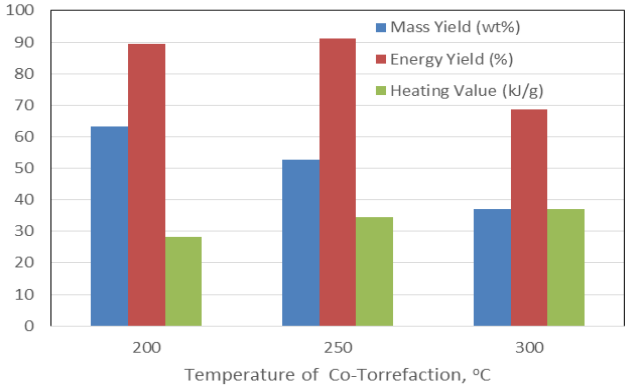


Fig. 5. The effect of temperature on biochar products.

The quality of biochar can be seen from its contents through proximate and ultimate analyses. The best biochar

resulting from the co-torrefaction process at the optimum operating temperature condition of 250°C (see subsection 3.3) was analyzed through proximate and ultimate analyses. In addition, the biochar resulting from the torrefaction (without the addition of the used cooking oil, a ratio of 1:0) was analyzed through proximate and ultimate analyses. Comparison of analysis results between the two biochar products is shown in Table 2.

Table 2 shows that the moisture content of the biochar resulting from the torrefaction and co-torrefaction processes was 4.46 wt.% and 2.89 wt.%, respectively. Meanwhile, coal (bituminous) had a moisture content value of 6.90 wt.%. This means that the addition of the used cooking oil in the co-torrefaction process successfully decreased the moisture content from 4.46 wt.% to 2.89 wt.%. Then, the decrease in moisture content value increased the heating value of the biochar from 25.268 kJ/g to 34.36 kJ/g (Table 2). This was commensurate with Mueanmas et. al [22] who stated that torrefied oil palm mesocarp fiber with a high water content had a low calorific value. Biochar which has a high moisture content causes mold to grow during the storage process [23].

Based on Table 2, the biochar from torrefaction had an ash content of 6 wt.%. Meanwhile, the biochar from co-torrefaction had a lower ash content which was 5 wt.%. In addition, the biochar from co-torrefaction had a lower fixed carbon (FC) content than that from torrefaction, namely 58.07 wt.% and 59.31 wt.%, respectively. This means that the addition of the used cooking oil in the co-torrefaction process decreased ash and FC contents. Meanwhile, the coal (bituminous) contained FC of 55.99 wt.% and ash of 5.14 wt.%. It shows that the FC level in both biochar products was higher than that in coal. These results indicate that the co-torrefaction process resulted in biochar with a higher quality compared to the torrefaction. For volatile matter (VM) levels, the co-torrefaction (with the used cooking oil addition) increased the VM contents in biochar from 29.44 wt.% to 33.02 wt.% (Table 2). This increase in VM was due to an incomplete carbonization process. The VM content in biochar produced by this study was lower than the VM content in biochar resulting from the torrefaction of cotton stalk, which was 68.53 wt.% [24]. Furthermore, Helwani et. al [25] carried out torrefaction of OPEFB and produced biochar containing VM of 47.73-63.20 wt.%. With an increase in torrefaction temperature, the volatile matter content in biochar decreases [26].

Based on Table 2, the carbon content was the highest, followed by oxygen, hydrogen, nitrogen, and sulfur contents for both biochar products. Between the two biochar products, the biochar from co-torrefaction had a higher carbon content than that from torrefaction, namely 75.41 wt.% and 69.79 wt.%, respectively. Apart from the carbon value, the biochar from co-torrefaction had a higher hydrogen content than that from torrefaction, namely 5.56 wt.% and 4.68 wt.%, respectively. However, the biochar from co-torrefaction had a lower oxygen content than that from torrefaction, namely 11.58 wt.% and 17.33 wt.%, respectively. This was due to the decarboxylation and devolatilization processes occurring during the torrefaction or co-torrefaction process [27]. The increase in carbon and hydrogen contents and the decrease in oxygen content increased the heating value of the biochar resulting from the co-torrefaction. Compared to coal, the biochar resulting from the co-torrefaction had higher carbon and hydrogen contents and lower sulfur content. This shows that biochar from co-torrefaction has good quality as a solid fuel substitute for coal.

Table 2. Results of Proximate and Ultimate Analyses of Biochar and Coal

Analyses	Biochar of torrefaction	Biochar of co-torrefaction	Coal (*bituminous*)*
Proximate (wt.%)			
Moisture Content	4.46	2.89	6.90
Volatile Matter	29.44	33.02	31.97
Ash Content	6.00	5.00	5.14
Fixed Carbon	59.31	58.07	55.99
Heating Value (kJ/g)	25.27	34.36	34.16
Ultimate (wt.%)			
Carbon (C)	69.79	75.41	71.48
Hydrogen(H)	4.68	5.56	4.57
Nitrogen (N)	1.4	1.2	1.0
Sulfur (S)	0.1	0.083	0.42
Oxygen (O)	17.33	11.58	10.49

Ref : *Huang et. al [7]

4. Conclusions

Pretreatment of oil palm empty palm oil bunches (OPEFB) with peat water can reduce the ash, potassium, and potassium oxide contents in OPEFB. By pretreatment with peat water at 50°C for 30 minutes, the ash content decreased by 80% (from 5 to 1 wt.%), potassium content decreased by 74.73% (from 50.46 to 12.75 wt.%), and potassium oxide content decreased by 75.18% (from 44.61 to 11.07 wt.%). The addition of used cooking oil as a co-feedstock in the torrefaction of pretreated OPEFB with a ratio of pretreated OPEFB:used cooking oil of 2:1 at 250°C increased in mass yield, calorific value, and energy yield of the biochar to become 52.78 wt.%, 34.36 kJ/g and 91.16%, respectively. The biochar resulting from the co-torrefaction process has good quality as a solid fuel that can replace bituminous coal.

Acknowledgements

The authors express their gratitude to Riau University for providing financial support via the research grant of research and community service institutions.

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