JEE Journal of Ecological Engineering

Journal of Ecological Engineering 2023, 24(10), 242–251
https://doi.org/10.12911/22998993/170719
ISSN 2299–8993, License CC-BY 4.0

Received: 2023.07.21
Accepted: 2023.08.06
Published: 2023.08.22

The Influence of Pretreatment and Post Treatment with Alkaline Activators on the Adsorption Ability of Biochar from Palm Oil Empty Fruit

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ABSTRACT

Empty oil palm bunches (EFB) can be converted by hydrothermal carbonation process into biochar that can be used as low-cost adsorbent. This study aims to identify the effects of pretreatment and post-treatment using alkaline activators on the biochar characteristics produced from EFB. The activation process was carried out before and after pyrolysis by heating it using an autoclave at 121 °C for 90 minutes. Biochar was then soaked using NaOH or KOH with a concentration of 0%, 4%, 8% and 12% for 3h. The ability of biochar as an adsorbent was analyzed for its ability to absorb iodine and methylene blue. Iodine absorption analysis was carried out using the Titrimetric method, while the methylene blue absorption test was carried out using the Spectrophotometric method. Results of the analysis showed that the absorption capacity of the resulting biochar for iodine ranged from 208.86–616.32 mg/g, and the absorption capacity of biochar for methylene blue ranged from 62.53–81.11 mg/g.

Keywords: palm oil empty bunches, biochar activation, low-cost adsorbent, pretreatment, post-treatment.

INTRODUCTION

So far, conventional use of empty oil palm fruit bunches (EFB) has been limited to being used as mulch which functions as a surface covering medium to maintain soil moisture and temperature in oil palm plantations. After experiencing decomposition, this mulch also acts as a supplier of nutrients for soil and plants. The process of utilizing EFB as mulch is less profitable and less added value because the degradation process is very slow. Therefore, efforts need to be made to find alternative innovations in handling or utilizing this palm oil industrial waste. One alternative for the utilization of EFB which is considered prospective is to convert it into biochar.

The conversion of EFB into biochar can be carried out by a hydrothermal carbonation process to convert lignocellulose into solid carbon with a low oxygen to carbon ratio (Jamari dan Howse 2012). This process is known as pyrolysis and can increase the energy density contained in it compared to the energy density of the original biomass (Raju et al. 2016). Pyrolysis of EFB at temperatures of 200–700 °C produced biochar as much as 29.39–42.74% (Raju et al. 2016; Kresnawaty et al. 2018; Febriyanti et al. 2019; Tiara et al. 2019).

Raju et al. (2016), conducted pyrolysis research at various temperatures to determine the effect of temperature on the pyrolysis products to be produced. Increasing the pyrolysis temperature decreased the percentage of biochar produced due to continued devolatilization of the remaining volatile matter in the charcoal. Conversely, increasing the pyrolysis temperature increases the production of liquids and gases due to the addition of volatile matter devolatilization of biochar.

Previous studies have shown that biochar can be used as an adsorbent for methylene blue, heavy metals in the form of Cu, Pb, Zn, As, Cd and herbicide compounds (Foo dan Hameed 2011; Samsuri et al. 2014; Sari et al. 2014; Shariff et al. 2014; Yavari et al. 2017; Zamani et al. 2017). However, the adsorption capacity of the biochar is relatively low, due to its relatively low surface area. Pyrolysis of EFB at 300–700 °C without pre-treatment produces biochar with a low surface area of 3.34–5.76 m^2/g (Thoe et al. 2019). The pre-treatment and post-treatment stages in the biochar production process from EFB can increase the surface area of the resulting biochar, thereby increasing its adsorption capacity.

This study aims to identify the effect of the activation process with alkaline pre-treatment and post-treatment on the characteristics of activated biochar produced from EFB.

RESEARCH METHODS

Materials and equipment's

The main materials used in this study are EFB, NaOH, KOH, and equates, as well as chemicals for laboratory analyses. The equipment used is a pyrolysis reactor, furnace, Erlenmeyer, Mohr pipette, bulb, spectrophotometer, test jar, desiccator, porcelain dish, oven, pH meter, and other laboratory equipment for testing biochar characteristics. FTIR (Fourier Transform Infra-Red) was used for qualitative analysis of functional groups produced by a chemical compound contained in biochar.

Research procedure

Preparation of raw materials

Empty oil palm bunches (EFB) are dried to a moisture content of less than 10%. EFB is then reduced in size to a size of 1–3 cm.

Production of activated biochar

Pretreatment activation

Pretreatment was carried out by heating EFB using an autoclave at 121 °C for 90 minutes. The EFB was then soaked using NaOH or KOH with concentrations of 0%, 4%, 8%, and 12% for 3 h. The soaked EFB is then washed with clean water to a neutral pH and then dried at room temperature. The EFB produced from this process is then pyrolyzed at a temperature of 400–600 °C for 60 minutes.

Post-treatment activation

Biochar from empty oil palm bunches by pyrolysis at 400–600 °C for 60 minutes was activated using physicochemical methods. Biochar is heated using an autoclave at 121 °C for 90 minutes. The heated biochar was then soaked in NaOH or KOH solution with a concentration of 0%, 4%, 8%, and 12% for 3 h. The following process is the washing of the soaked biochar to neutral pH. Table 1 shows the combination of activator types and activator concentrations in pre-treatment and post-treatment.

Laboratory analysis

Laboratory analysis of the biochar samples was analyzed according to Indonesian Nasional Standard (SNI 06-3730-1995).

- Water content – a total of 1 gram of sample is placed in a dish of known mass, then dried in an oven at 105 °C until a constant mass is obtained. The sample is then cooled in a desiccator.
- Ash content – A total of 1 g of biochar is placed in a porcelain dish and heated in an oven at 105 °C until a constant mass is obtained. The sample in the dish is then put into the kiln and opened at 650 °C for 4 h, then cooled in a desiccator. The formed ash is then weighed.
- Volatile substances – sample of biochar is heated in a kiln at 900 °C for 15 minutes, cooled in a desiccator, and then weighed.

Table 1. Combination of activator types and activator concentrations in pretreatment and post treatment

Concentration	Heating + KOH	Heating + NaOH
0%	Heating + KOH/NaOH 0% (HKN0)	
4%	Heating + KOH 4% (HK4)	Heating + NaOH 4% (HN4)
8%	Heating + KOH 8% (HK8)	Heating + NaOH 8% (HN8)
12%	Heating + KOH 12% (HK12)	Heating + NaOH 12% (HN12)

- Carbon content – carbon content can be determined by calculating the absolute percentage difference with the sum of the percentage of moisture content, volatile substance content, and ash content of biochar.
- Iodine absorption – a total of 1 g biochar is put into an Erlenmeyer, then 0.125 N iodine solution is added as much as 25 mL. The suspension is stirred for 15 minutes, then the Erlenmeyer is covered and kept in the dark place for 2 h. The solution is filtered, then 10 mL of filtrate is taken, put into a clean Erlenmeyer, and titrated with $Na_2S_2O_3$ solution until the solution is light yellow. The amylum indicator is added as much as 1 mL to the filtrate; titration is carried out until the blue color disappears. The volume of $Na_2S_2O_3$ solution used was recorded, and the absorbency of biochar against iodine was calculated in mg/g.
- Absorption of methylene blue – a total of 1 g of biochar was put into a beaker glass and added 25 mL of 10,000 mg/L methylene blue solution was stirred with a magnetic stirrer for 30 minutes. The solution is filtered, then the absorbance of the filtrate is measured with a Shimadzu UV-1800 spectrophotometer at λ_{max} methylene blue, which is 664.4 nm. The determination of the residual methylene blue concentration in the filtrate is carried out by the calibration curve method.
- FTIR (Fourier Transform Infrared) – FTIR spectroscopy is an infrared spectroscopy equipped with a Fourier transform to detect and analyze the results of the spectrum. The infrared spectrum results from the transmission of light passing through the sample, measuring the intensity of light with a detector, and comparing it with the intensity without the sample as a function of wavelength. FTIR analysis is carried out to determine changes in biochar functional groups before and after activation.

RESULTS AND DISCUSSION

Mass balance of biochar manufacturing process

Biochar production begins with the process of reducing the size of EFB to about 1–5 cm in size. In the process of reducing the size, there is a loss of about 5% of raw materials. After reducing the size, EFB is dried to a moisture content of less than 10%. The pyrolysis process was carried out at a temperature of 400–600 °C for one h. The yield of biochar obtained from this pyrolysis process ranges from 46–65%. From 1000 g of EFB, 284–494 g of biochar is produced. The mass balance of biochar manufacturing is presented in Figure 1.

During the pyrolysis process, various processes occur subsequently and/or simultaneously including dehydration, depolymerization, breaking of polymeric hydrocarbon bonds in the material, and the formation of bonds or new compounds or fragments with a certain molecular weight. The low molecular weight fragments will break down into gas and liquid compounds (bio-oil), while the high molecular weight fragments will become biochar. The higher the carbonization temperature, the more volatile compounds will be decomposed and gasified, resulting in a reduction in the mass of the resulting carbon product.

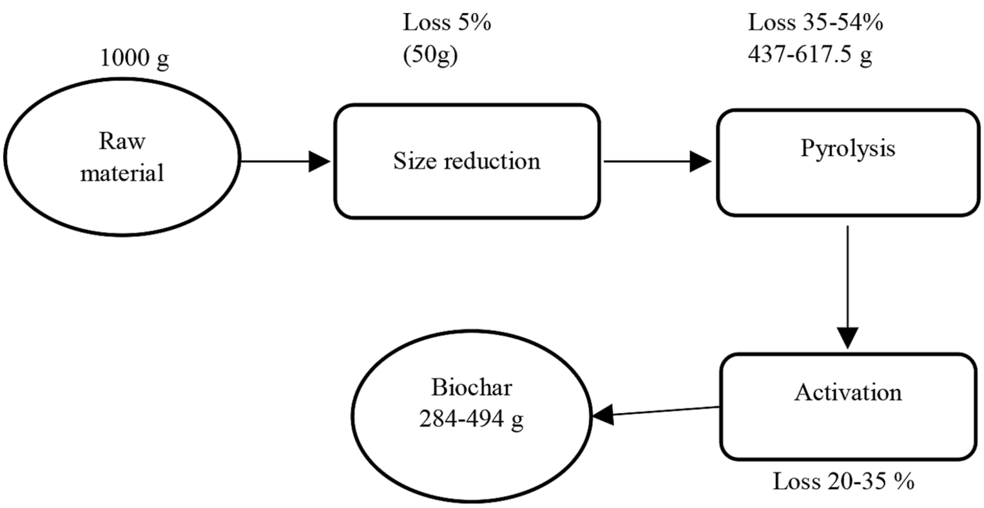

Figure 1. Mass balance of biochar production

Water content

The water content of the biochar from the EFB because of the research was quite low, ranging from 2.52–8.08% (Figure 2), lower than the Indonesian national standard (SNI 06-3730.1995). However, the water content of the resulting biochar adjusts to its hygroscopic properties. Biochar pores can absorb moisture from the air. The relatively low water content of biochar is caused by the surface of the biochar which contains fewer polar functional groups, so that the interaction between steam and biochar is low (Paramitadevi dan Rahmatullah 2017).

It can be seen from Figure 2 that the lowest water content was in the 12% KOH activated biochar, which was 2.52%, while the highest water content was in the heat-activated biochar, which was 8.08%.

Ash content

Ash is an inorganic mineral and silicate compound contained in the biochar structure and does not evaporate during the carbonization process. The ash content varies depending on the precursor used as a carbon source in the manufacture of biochar. Figure 4 shows the ash content of the biochar produced in various treatments. Activation with KOH and NaOH causes a decrease in the ash content of biochar. This is because the activation process can extract the silicate content in the biochar, which results in a decrease in the ash content after chemical activation. During the extraction process, the silicate on carbon reacts with KOH and NaOH to form potassium silicate and sodium silicate, with the reaction mechanism as follows:

$$SiO_{2(s)} + 2\ KOH_{(aq)} \rightarrow K_2SiO_{3(s)} + H_2O_{(aq)} \quad (1)$$

$$2\ NaOH_{(aq)} + SiO_{2(s)} \rightarrow Na_2SiO_{3(s)} + H_2O_{(aq)} \quad (2)$$

In the silica molecule (SiO_2), the high electronegativity of the O atom causes Si to be more electropositive and an unstable intermediate $[SiO_2OH]^-$ is formed, so that dehydrogenation will occur and hydroxyl ions originating from alkali will bond with hydrogen to form water molecules. Two K^+ ions will balance the negative charge formed and interact with SiO_3^{2-} ions to form potassium silicate. Apart from being caused by the silica extraction process, the decrease in ash content in activated biochar is also caused by several mineral compounds making up the ash content which can dissolve in KOH solution including Al_2O_3 and P_2O_5.

The ability of biochar to adsorb is affected by the ash content. The ash content of the biochar produced in this study was in the range of 4.19–9.01%. This value meets the Indonesian national standard for activated charcoal.

Laos et al. (2016), produced activated charcoal from candlenut shells with ash content is 8.5%. The high ash content is allegedly caused by contact with outside air during the pyrolysis process, so that more material is converted to ash. Various mineral elements in the ash can affect the formation of biochar pores, where these mineral elements can close the biochar pores in the biochar activation process. The number and size of the biochar pores affect its absorption of iodine.

Volatile material content

Volatile material is the substances that evaporate at a certain temperature and time. Volatile

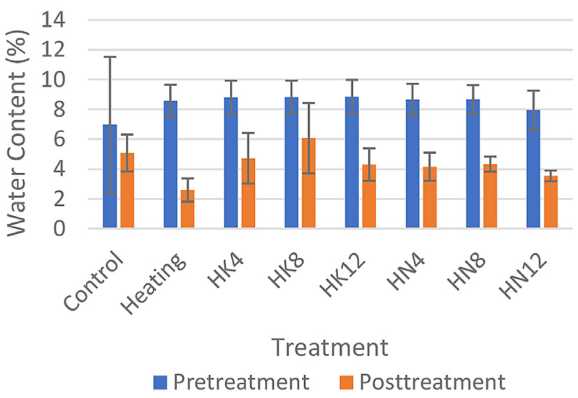

Figure 2. Water content of biochar activated by different treatments

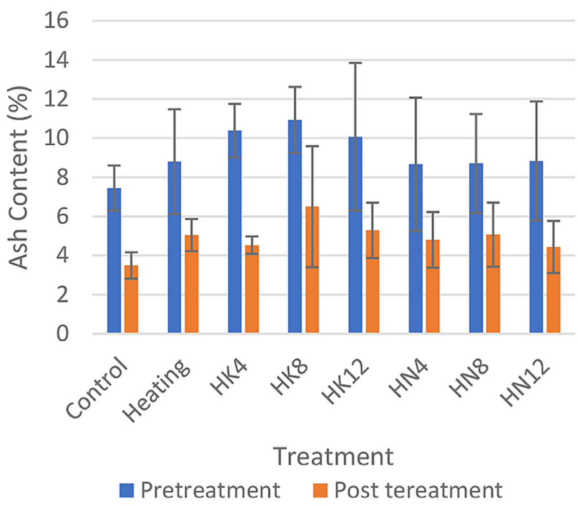

Figure 3. Ash content of biochar activated by different treatments

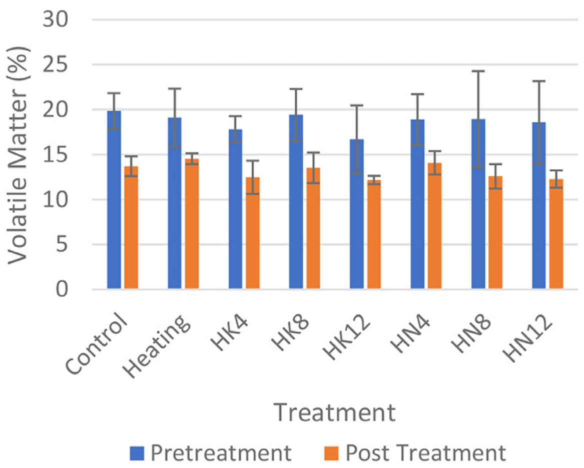

Figure 4. Volatile matter content of biochar activated by different treatments

substances consist mostly of flammable and non-combustible gases. Combustible gases include methane, hydrocarbons, hydrogen, carbon monoxide, while non-combustible gases include carbon dioxide and nitrogen (Iskandar dan Rofiatin 2017). Figure 4 shows that the levels of volatile matter (volatile meters) range from 10.85–17.55%. Lower volatile matter levels were observed in biochar with activation treatment using 4% KOH catalyst, while volatile matter with higher volatile matter values could be observed in biochar with activation treatment using 8% KOH catalyst. The results of this study indicate that the volatile matter content of biochar meets the Indonesian national standard (SNI 06-3730-1995) which is 15–25%.

Domingues et al. (2017), produced biochar from Coffee husk with a volatile content of 17.6%. Other researchers produced biochar from sugarcane bagasse, soybean stover, swine manure, and buckwheat husks with volatile content 7.7%, 14.7%, 35.5% and 11.7% (Jin et al. 2016; Domingues et al. 2017; Karunanithi et al. 2017; Zama et al. 2017).

Higher pyrolysis temperatures cause the release of volatile matter and form more biochar pores (Shaaban et al. 2014). The levels of volatile matter also affect the formation of pores in biochar (Tomczyk et al. 2020). An increase in temperature causes a decrease in the volatile matter content (Crombie et al. 2013; Tag et al. 2016). Zhao et al. (2017), reported that an increase in pyrolysis temperature reduced the volatile matter content from 60.8% to 14.9% in biochar from apple tree wood. On the other hand, high levels of volatile matter indicate that biochar may still contain some of the original organic plant residues such as cellulose (Tomczyk et al. 2020).

Carbon content

The bound carbon content in biochar which was activated by heating with the addition of an alkaline catalyst (KOH and NaOH) was in the range of 74.81–84.60% (Figure 5). A lower carbon percentage was observed in biochar with pretreatment activation using 8% KOH, while a higher carbon percentage was found in post-treatment activated biochar using 12% KOH. These results indicate that the carbon content of biochar meets the Indonesian national standard (SNI 06-3730-1995) for powdered activated carbon, which is a minimum of 65%. The carbon content can basically be increased by increasing the pyrolysis temperature (Domingues et al. 2017).

Zama et al. (2017), conducted investigations with buckwheat husk at a pyrolysis temperature of 350–700 °C to produce a fixed carbon content of 70.1–83.9%. Other researchers produced biochar from coffee husk, sugarcane bagasse, soybean, swine manure with carbon content 66%, 90.5%, 82%, 74.9% (Jin et al. 2016; Domingues et al. 2017; Karunanithi et al. 2017). The fixed carbon is directly proportional to the pyrolysis temperature (Iskandar dan Rofiatin 2017). According to Tomczyk et al. (2020), an increase in pyrolysis temperature can increase the fixed carbon due to a higher degree of polymerization, which causes the carbon structure in the biochar to condense. Tag et al. (2016), reported that an increase in pyrolysis temperature increased in the carbon content of pomace biochar. While increasing pyrolysis temperature causes carbon content in manure sources to decrease (Cantrell et al. 2012).

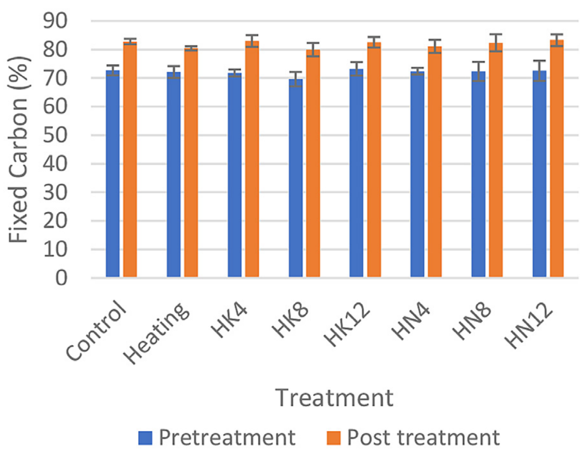

Figure 5. Carbon content of biochar activated by different treatments

Iodine absorbency

Biochar absorption of iodine was measured to determine the ability of biochar to absorb colored solutions with a molecular size of less than 10 Å (or 1 nm). The higher the results of the measurement of iodine absorption, the better the biochar produced will be for application as an adsorbent (Sidik et al. 2019). Absorption of iodine and methylene blue correlates with the surface area of biochar and is directly related to the ability of biochar to adsorb certain components (Tamrin dan Zahrim 2017). The results of measuring the absorption of iodine in this study were 208.86–616.32 mg/g (Figure 6). Biochar's ability to absorb iodine is lower than activated charcoal according to Indonesian national standards, which is at least 750 mg/g.

The absorption of iodine produced in this study is in line with the absorption of iodine of activated carbon from palm fruit bunches (Nurlia et al. 2020). The absorption capacity of iodine in dragon fruit peel reaches 195.2 mg/g (Jawad et al. 2021). The low absorption of iodine in this study is due to the temperature used in the activation process which, is ambient temperature. The high activation temperature affects the formation of pores in activated carbon.

Methylene blue absorption

Biochar absorption test for methylene blue solution was carried out to indirectly estimate the surface area and ability to absorb colored solutions (Nurlia et al. 2020). According to Desi et al. (2015), the ability of biochar to absorb methylene blue is due to the pore size of the biochar. The absorption capacity of biochar on methylene blue from this study was in the range of 62.53–81.11 mg/g with an absorption efficiency of 16.13%. (Figure 7).

The highest absorption value of biochar to methylene blue can be observed in the activation treatment using heating. From the absorption capacity of methylene blue, the resulting activated biochar meets the Indonesian national standard for activated charcoal, which is 60 mg/g. The addition of activator in the activation process has the potential to penetrate closed pores, so that the pores will open and the mesoporous-sized pores will widen (Guo et al. 2014; Kong et al. 2015).

The absorption capacity of methylene blue produced in this study was higher than that of activated carbon from palm fruit bunches (63–73.59 mg/g) (Nurlia et al. 2020). The absorption capacity of Methylene blue in dragon fruit peel biochar was 195.2 mg/g (Jawad et al. 2021), moringa leaves were 136.99 mg/g (Do et al. 2021). The activation temperature used greatly affects the formation of pores in activated carbon, thereby affecting the ability to adsorb Methylene blue. In addition, the absorption of methylene blue can increase with increasing pH of the methylene blue solution and the dose of adsorbent used (Hussein dan Ahmad 2007).

In closing, the characteristics of the biochar produced in this study are compared with the results of other studies (Table 2), including parameters of water content, ash content, volatile matter content, fixed carbon, iodine absorption and methylene blue absorption. From the table, it can be seen that the characteristics of biochar are influenced by raw material factors, the conditions of the pyrolysis process, and the method

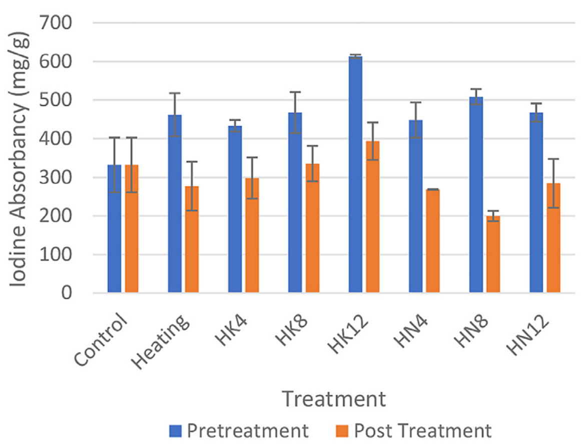

Figure 6. Iodine absorbency of biochar activated by different treatments

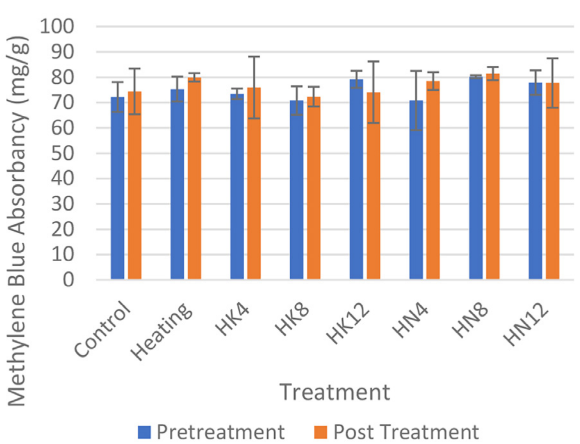

Figure 7. Methyl bule absorption of biochar activated by different treatments

Tabel 2. Characteristics of biochar from various biomasses

Biomass	Ash (%)	Volatile matter (%)	Fixed carbon (%)	Iodine absorbency (mg/g)	Methylene blue absorbency (mg/g)	Reference
EFB	9.01	17.55	74.81	616.32	81.11	This research
Coffee husk	19.6	17.6	66.0	-	-	Domingues et al. (2017)
Sugarcane bagasse	2.2	7.7	90.5	-	-	Domingues et al. (2017)
Soybean stover	17.2	14.7	82.0	-	-	Karunanithi et al. (2017)
Swine manure	49.8	35.5	74.9	-	-	Jin et al. (2016)
Buckwheat husks	33.1	11.7	83.9	-	-	Zama et al. (2017)
Palm bunches	-	-	-	325.8	73.59	Nurlia et al. (2020)
Dragon fruit peel	-	-	-	-	195.2	Jawad et al. (2021)
Moringa leaves	-	-	-	-	136.99	Do et al. (2021)
Candlenut shell	8.5			252.97		Laos et al. (2016)

of activation. The biochar from EFB has the potential to be further developed as a low-cost adsorbent that can be applied for various purposes, for example for advanced wastewater treatment. Furthermore, saturated adsorbent can be used as fertilizer or soil improver.

FTIR

FTIR analysis was performed to determine changes in biochar functional groups before and after activation. FTIR testing was carried out on four EFB biochar samples, namely biochar without

Table 3. IR uptake characteristics in each biochar sample

Absorption area	Functional	Pyrolysis	AK	AN	Del K
995–675	C-H (Alkena)	+	+	+	+
900–690	C-H (Aromatic Ring)	+	+	+	+
1300–1050	C-O (Alcohols / ethers / carboxylic acids / esters)	-	+	-	-
1360–1180	C-N (Amina / Amida)	-	+	-	-
1600–1500	C=C (Aromatic Ring)	+	+	+	+
1570–1500	Groups / component NO_2	+	+	+	+
2260–2100	C≡C (Alkuna)	+	+	+	+
3600–3200	O-H (hydrogen/phenol bonds)	-	+	-	-
3650–3590	O-H (monomer/phenol bond)	-	-	-	+
1238–1230	C-H component	-	+	-	-
2935–2915	Methylene C-H asym./sym. Stretch	+	-	-	-
2850–2815	Methoxy, methyl ether O-CH3, C-H stretch	-	-	+	-
3040–3010	Terminal (vinyl) C-H stretch	+	-	-	-
2100–1800	transition metal carbonyls	+	+	+	+
1380–1350	Nitrate ion	+	+	+	+
1100–900	Silicate ion	+	+	+	+
2323–2350	NH Component	-	+	+	+
1190–1130	Secondary amine, CN stretch	-	-	-	+

Note: Pyrolysis – pure biochar, AK – biochar post-treatment with the addition of KOH, AN – biochar post-treatment with the addition of NaOH, Del K – biochar pretreatment with the addition of KOH.

activation, biochar with pre-treatment, and biochar with KOH post-treatment, and biochar with NaOH post-treatment. The FTIR spectrum used to test the 4 samples is between 4000 and 650 cm^{-1}. The FTIR test results for the four samples can be seen in Figure 8. The biochar samples have similar aromatic groups and aliphatic functional groups. The complete IR absorption characteristics of each sample can be seen in Table 3.

In biochar samples, there are O-H functional groups, hydrogen bonds, C-H bonds derived from cellulose, and C=C bonds derived from lignin aromatic rings (Saelee et al. 2014). Biochar has functional groups O-H hydrogen bonds and C-H bonds originating from cellulose, as well as C=C bonds originating from lignin aromatic rings (Saelee et al. 2014). This C=O bond indicates the presence of carboxylic or carbonyl groups present in the aromatic ring (Prapagdee et al. 2014). In addition, biochar has a C-N functional group which can be an amine or an amide, and nitro compounds (NO_2) may come from the nitrogen content contained in EFB.

Heating at 350–650 °C causes chemical bonds in biomass to break and form new bonds, such as carboxyl, lactone, lactol, quinine, chromone, anhydride, phenol, ether, Pyron, pyridine, pyridine, and pyrrole (Mia et al. 2017). When biomass is pyrolyzed at temperatures higher than 400 °C, the peak intensities are mostly reduced or shifted. The phenol and amide groups are decomposed, so that the cellulose component turns into a biochar carbon structure (Ray et al. 2020). Peaks with a band of 4000 to 3500 cm^{-1} indicate the release of H_2O from the pyrolysis process (Ray et al. 2020). After the activation process, the methylene C-H bond at a wavelength of 2935–2915 and the vinyl C-H bond at a wavelength of 3040–3010 disappear. After the activation process is carried out there is a NH component in the absorption area 2323–2350.

CONCLUSION

Based on the results of this study, EFB pretreatment produced activated biochar with a larger surface characterized by a higher iodine absorption value. By the alkaline pretreatment, there was a change in a functional group which led to the formation of O-H (monomer/phenol bond), secondary amine, CN stretch, and NH Component. It is necessary to conduct experiments on applying biochar for the adsorption of wastewater pollutants, for example, biologically treated palm oil factory waste. Saturated biochar can then be used as fertilizer to improve soil structure. The saturated biochar can then be used as fertilizer to improve the soil's physical, chemical, and biological structure.

Acknowledgements

The author would like to thank the Ministry of Education, Culture, Research and Technology of the Republic of Indonesia for providing grants through the Doctoral Dissertation Research Scheme Year 2021–2022.

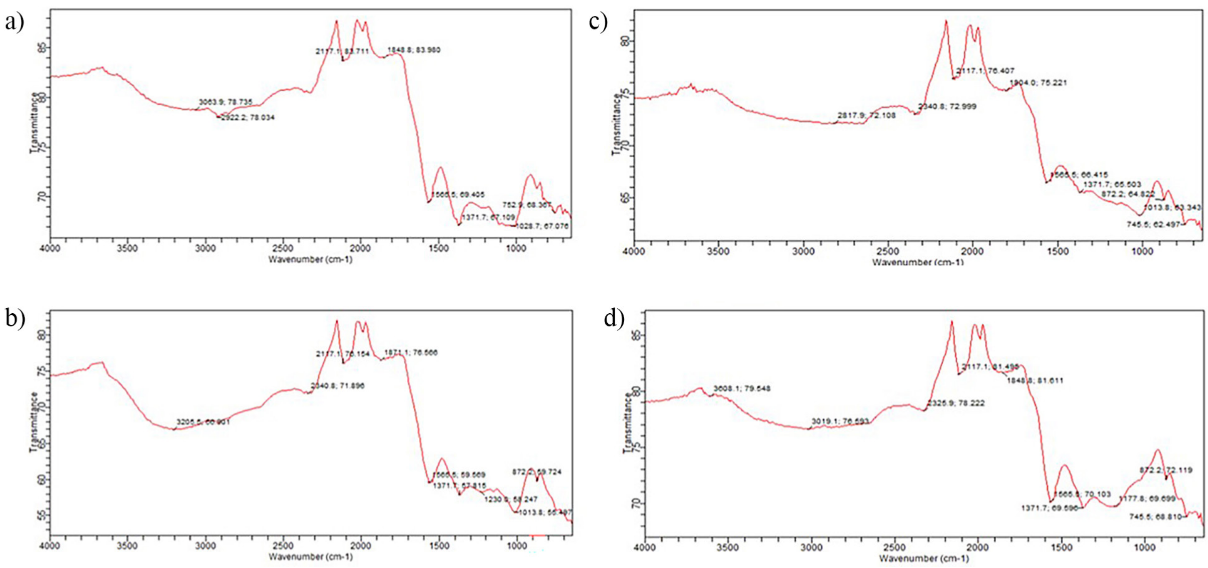

Figure 8. FTIR Biochar, a) Pyrolysis without activation; b) with NaOH post-treatment; c) with KOH post-treatment; d) with pretreatment

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