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Characterization of Biochar Empty Fruit Bunches OPEFB at Various Temperatures and Burning Time

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ABSTRACT

Oil palm waste (OPW), comprising mainly of empty fruit bunch, mesocarp fiber, frond, trunk, and palm kernel shell generated from the palm oil industry, was collected, characterized, and then pyrolyzed to evaluate their potential to be converted into biochar. Oil Palm Empty Fruit Bunches (OPEFB) are a source of organic material with abundant nutrients and are highly potentially useful as biochar. This article provides experimental data for the production of biochar at a temperature range of 100 to 300 °C at time of 4 to 8 hours. The chemical components examined are pH, CEC, C-Organic, N-total, C/N, K dd, P, Ca, Mg, and Na, using Fourier Transform Infrared Spectroscopy (FTIR). The results showed that organic C, nitrogen, and pH were highest at 200–300 °C and had a burning time of 8 hours. Furthermore, the highest concentrations of P, Ca, and Mg were recorded at 200–300°C after 5 hours, Kdd at 100–200 °C after 5 hours, and Na and CEC at 200–300 °C after 4 hours. The transmittance intensity produced by the spectrum of hydroxyl (O-H) vibrations, carbonyl stretching (C=O), alkanes (-CH), and aromatics (C=C) decreased with increasing time, while stretching alcohol (C-O) vibrations increased with time. Our results demonstrate that OPEB is a biowaste that shows exceptional promise to be transformed into high-grade biochar rather than simply disposed of by landfilling or burning.

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1 Introduction

Oil palm is one of the growing plantation commodities and has an important role in the Indonesian current economy. According to the data BPS d (2018), in 2017 the total area of oil palm plantations in Indonesia has reached 12.30 million ha with an average productivity of 2.80 tons of CPO (crude palm oil) and 0.56 tons of PKO (palm kernel oil)/ ha/year. Currently, palm oil mill residues, especially empty oil palm fruit bunches, have been produced massively.

OPEFB is a source of organic matter rich in N, P, K, and Mg nutrients. The number of empty fruit bunches is estimated at 23% of the processed fresh fruit bunches. Oil palm from one tonne of empty fruit bunches contains 1.5% N, 0.5% P, 7.3% K, and 0.9% Mg. Further, it contains 6.79% N-nutrients, 40.2-45.3% C-macronutrients, 2.4-2.7% K_2O, 0.8-1.2% N, 0.05-2.6% P_2O_5, 0.4-0.5% MgO, and C/N 45–70, microelements, such as 10 ppm B, 23 ppm Cu, and 51 ppm Zn (Razali and Kamarulzaman 2020). The chemical properties of biochar, such as nutrients, have an important role in soil fertility

Furthermore, EPEFB contains 45.9% cellulose, 46.5% hemicellulose, and 22.8% lignin (Qiao et al. 2019; Ferreira et al. 2020). Oil palm shells contain 29.4% lignin, 27.7% hemicellulose, 26.6% cellulose, 8.0% water, 0.6% ash, and 4.2% extractive components, all included in hydrocarbons (Gupta et al. 2018). The lignin and cellulose content in each raw material affects the biochar formation.

Biochar provides opportunities to store carbon (C) in soil over much longer periods compared to unpyrolyzed biomass Application of biochar affects several soil properties including electrical conductivity (EC), pH, cation exchange capacity (CEC), nutrient levels, porosity, bulk density, and microbial community structures (Shaaban et al. 2018)

Biochar is a product of pyrolysis of biomass in the absence of oxygen and has a high potential to sequester carbon into more stable soil organic carbon (OC). Despite a large number of studies on biochar and soil properties, few studies have investigated the effects of biochar in contrasting soils (Ghorbani et al. 2019)

OPEFB is the best material for biochar because of its high calorific value (Bi et al. 2021). Results of pyrolysis products dependent on temperature, time, pressure, and composition of raw materials produce differences in physical form and structure. These differences significantly affect biomass carbonization, producing biochar and nutrient content (Qiao et al. 2019). The pyrolysis process at 100 - 170 °C releases water content due to evaporation The effect of biomass addition on pyrolysis characteristics and gas emission of coal gangue by multi-component (Ferreira et al. 2020). The temperature range of 170 - 270 °C causes exothermic reactions and decreases the evaporation of CO and CO_2 gases. The carbon sequestration potential (CSP) of zeolite is arguably zero while rubberwood sawdust and rapeseed meal biochars were produced at 300 °C which is too low to carbonize cellulose and lignin (Dominguez et al. 2020). The results showed that the biochars produced at different pyrolysis temperatures had different physical characteristics which influenced their reactivity to the gasifying agent during gasification. It was found that when the pyrolysis temperature was increased from 300 to 700 °C, the HHV of the biochars increased from 23.06 to 26.77 MJ/kg, and the surface area of the biochars increased from 0.0218 to 0.2720 m2/g, the biochar yield decreased from 54.83 % to 28.37 % (Selvarajoo and Oochit 2020). FTIR revealed the presence of functional groups such as -OH, -C = O, C = C, and –CH. These play important roles in the specific surface area of the biomass (Ighalo et al. 2020).

Pyrolysis using high temperatures produces biochar with a high surface area and aromatic carbon content. The temperature and duration of combustion determine the quality of biochar. Therefore, it is necessary to know the optimal temperature and burning time. This study aimed to examine the physicochemical characteristics of OPEFB biochar. Also, it aimed to determine the optimum temperature and burning time for establishing the OPEFB biochar quality. This report is part of research on increasing the productivity of lowland rice using methanotroph bacteria and TKSS Biochar in the Morowali Regency.

2 Materials and Methods

2.1 Production of Oil Palm Empty Fruit Bunch (OPEFB) Biochar

The raw material used in this study is Oil Palm Empty Fruit Bunches (OPEFB), shown in Figure 1. It was obtained from community plantations in Witaponda District Ungkaya Village, Morowali Regency, Central Sulawesi Province. OPEFB is burned using tools and procedures easily adopted by farmers. It was dried in the sun for three days and burned at temperatures around 100–200 °C and 200–300 °C (Sukmawati et al. 2020) using a closed drum without oxygen (Figure 2). Therefore, it did not undergo a complete oxidation process that removed much carbon. After forming biochar, it was doused with water and dried, at which point the temperature was adjusted using a thermocouple. The temperatures were 100–200 °C (T1) and 200–300 °C (T2), and the burn times were 4 hours (W1), 5 hours (W2), 6 hours (W3), 7 hours (W4), and 8 hours (W5). Therefore, it obtained 10 treatment combinations of T1W1, T1W2, T1W3, T1W4, T1W5, T2W1, T2W2, T2W3, and T2W4.

Figure 1 Oil Palm Empty Fruit Bunches

Figure 2 Drum and Temperature Measuring Equipment for Making OPEFB Biochar

2.2 Chemical component analysis of OPEFB biochar

The analysis of biochar samples were carried out under the laboratory conditions and the recorded parameters were pH, H_2O, KCl (Glass Electrode), C-organic (Walkley-Balck), total N (Kjeldahl), total P, and K (25% HCl), available P (Bray or Olsen); exchangeable bases (Ca, Mg, Ca, and Na) (extraction of NH4OAc pH 7,0), exchangeable Al (extraction of 1 N KCl); saturation of Al (ratio Σ exchangeable Al/KPKNH4OAc), KPK extraction of NH4OAc-pH 7), KBNH4OAc (pH 7); Al, Fe, and Si free oxide amorphous and organic associations (extraction of Citrate-Bicarbonate- Dithionite, Ammonium Oxalic, Sodium Pyrophosphate) The digestion method referred to the method for soil samples (Pierzynski 2009)

2.3 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The chemical functional groups of biochar were examined using FTIR analysis. The presence of certain chemical functional groups helps understand the mechanism of the biochar adsorption process. Before the analysis, the sample was ground to a powder and oven-dried overnight to remove residual moisture. It was then mixed with potassium bromide (KBr) in a ratio of 1 part sample to 200 parts KBr. The sample mixtures were analyzed using an FTIR analyzer (Nicolet 5700, FTIR, Thermo Fisher Scientific, Waltham, MA, USA).

The interpretation of the FTIR spectrum refers to hydroxyl acids OH (3000–3690 cm^{-1}), CH alkanes (2927–2856 cm^{-1}, 1446–1370 cm^{-1}), aromatic rings C = C (1560–1600 cm^{-1}), carboxylic acid C = O (1560–1600 cm^{-1}), C-OH (1033 cm^{-1}) (Tippayawong et al. 2018). FTIR spectroscopy was performed on a Prestige-21 FT-IR (Shimadzu Corp) IR spectrometer, equipped with a bright ceramic light source, KBr beamsplitter, and doped L-alanine tri-glycine sulfate (DLATGS). The sample measurements were collected in the range of 4000-600 cm^{-1} and 16 additional scans. All FTIR spectra are in the transmittance unit. The magnetic study of the samples was conducted using a vibrating magnetometer (Oxford Instruments, VSM 1.2 H).

3 Results and Discussion

3.1 Analysis of the Nutrient Content of Biochar in Oil Palm Empty Fruit Bunches

The main biomass components, including hemicellulose, cellulose, and lignin, were degraded during combustion. Each biomass directly influences the yield and characteristics of the resulting

Figure 3 Oil Palm Empty Fruit Bunch Biochar

OPEFB product, containing 10.45% carbon black (Figure 3). The product was analyzed for biochar content and the results are in Tables 1 and 2.

The results of chemical components produced by each biochar are presented in Table 1 and revealed that the organic C content for all treatments ranged between 24.01%-28.12%. The highest organic C content (28.12%) was reported at 200-300 ^{o}C, with a burning time of 8 hours, while it was reported 24.01% at 100-200 ^{o}C for 5 hours. Further, the results of the C-organic content can be concluded very high because it exceeds five percent.

Further, the C/N ratio analysis for each treatment was carried out and it was classified as very high and it exceeds more than 20%. A temperature of 100-200 °C with a burning time of 7 hours gives a very high C/N ratio of 163.41%, while the lowest C/N ratio was produced at 200-300 °C for 8 hours (13.13%). These results are consistent with previous studies, where the C/N of oil palm shell biochar was 31.6%, while it was reported 57.37% for the empty fruit bunches (Sung 2016). The high C/N value is due to the loss of nitrogen during pyrolysis (Qiao et al. 2019). The P content analysis results for each treatment were generally classified as very low (less than 11%). The highest P element was produced at 200-300 °C for 5 hours (0.52%), while the lowest was produced at 100-200 °C for 7 hours (0.04%). The results obtained in this study revealed that in general biochar properties are related to the influence of low pyrolysis temperature (≤500 C) that can be a source of nutrients (Zhang et al. 2017).

The result presented in Table 2 revealed that the highest concentration of micronutrient Ca, Mg, and Na (32.25, 16.35, 41.32 cmol/kg respectively) in OPEFB biochar was reported at 200-300 °C with a burning time of 5 hours, while the lowest concentration (16.21, 10.23, 21.85 cmol/kg respectively) was produced at 100-200 °C for 5 hours of burning time. Further, the pH of each biochar sample was tested and it was reported in the

Table 1 Nutrient Analysis Results on Organic matters in Oil Palm Empty Fruit Bunch Biochar

No.	Treatment	Nutrient Analysis				
		C Organic (%)	N (%)	C/N (%)	P2O5 (%)	K (cmol / kg)
1	T1W1	24.72	1.08	22.79	0.25	18.25
2	T1W2	24.01	0.61	39.20	0.32	32.25
3	T1W3	26.82	0.70	38.32	0.22	14.75
4	T1W4	26.15	0.16	163.41	0.04	15.26
5	T1W5	26.92	0.19	139.66	0.23	10.24
6	T2W1	27.38	0.38	72.77	0.46	12.36
7	T2W2	24.42	1.01	24.15	0.52	14.25
8	T2W3	27.20	0.28	98.21	0.35	13.85
9	T2W4	26.79	0.54	49.36	0.41	16.21
10	T2W5	28.12	2.14	13.13	0.39	14.02

Table 2 Nutrient Analysis Results of Oil Palm Empty Fruit Bunch Biochar

No.	Treatment	Analysis				
		Ca (cmol / kg)	Mg (cmol / kg)	Na (cmol / kg)	pH	CEC (cmol / kg)
1	T1W1	18.35	10.22	28.25	7.30	29.39
2	T1W2	16.21	13.25	35.00	7.69	20.58
3	T1W3	28.62	14.21	21.85	8.53	18.19
4	T1W4	26.32	15.32	31.14	8.51	16.68
5	T1W5	24.36	16.32	25.34	7.71	10.20
6	T2W1	22.41	10.34	41.32	7.77	23.05
7	T2W2	32.25	16.35	32.25	7.61	12.28
8	T2W3	27.15	15.21	32.27	8.20	11.48
9	T2W4	23.36	12.36	28.63	7.96	10.82
10	T2W5	21.14	10.25	32.25	8.56	15.35

moderately alkaline category. The highest pH value of 8.56 was reported from the OPEFB biochar samples burned at 100-200 °C temperature for 6 hours, while the lowest pH value 7.30 was reported from the OPEFB biochar sample burned at 100-200 °C for 4 hours. The results are consistent with, where oil palm shells and corn cobs had a pH value of 7.3, while empty fruit bunches had 7.2 (Sukmawati et al. 2020).

The analysis of CEC values showed various results in the moderate category of 10–29 cmol/kg (Table 2). The highest CEC value (29.39 cmol/kg) was reported at 100–200 °C for 4 hours, while the lowest (10.20 cmol/kg) was reported for 8 hours at 100–200 °C. In contrast to this, Mukherjee & Zimmerman (2013) reported that the CEC of biochar from oil palm empty fruit bunches was reported 21.5 cmol/kg while the biochar produced by pyrolysis at 300–400 °C had a high CEC value of 50.52–56.88 cmol/kg. Further, CEC values for oil palm shells, empty fruit bunches, and corn cobs were reported 50.52 cmol/kg, 52.36 cmol/kg, and 56.84 cmol/kg respectively.

The highest organic C and nitrogen content (28.12%) was reported from the OPEFB biochar samples burned at 200–300 °C after 8 hours of burning, while the lowest was at 100–200 °C for 5 hours of burning. Pyrolysis enhances the thermochemical process, forming cellulose and lignin from long-to-short carbon chain structures. These results are contradictory to the findings of Sukmawati et al. (2020) who reported the lowest value of 59.85% at 300–400 °C burning time for 4 hours. Good biochar is produced at high temperatures without oxygen. High temperatures accelerate the decomposition process of organic matter biomass components into lignin, cellulose, and hemicellulose. Moreover, a higher rate of decomposition of organic matter increases the formation of nutrients. The high lignin, cellulose, and hemicellulose content in OPEFB increase the organic C content. Furthermore, the high organic C content increases the amount of organic matter in the soil, improving its physical, chemical, and biological properties. The amount of lignin and cellulose in each raw material affects the process of biochar formation, and high lignin content makes more carbon.

The high value of C/N at 100–200 °C with a burning time of 7 hours is caused by the reduced N in nitrogenous biomass. The low organic C content reduces the C/N ratio at 200–300 °C for 8 hours due to organic carbon compounds' decomposition and nitrogen compounds' changes during combustion. The C/N ratio is reduced through combustion by converting organic C into CO_2 and nitrogen loss as NH_3. A higher C/N ratio increases the time it takes to overhaul the organic matter.

The analysis regarding the highest P_2O_5 content showed that it was lowest at 200-300°C for 5 hours and at 100-200°C for 7 hours. This indicates that phosphate decomposes and stabilizes at high temperatures. Phosphorus is difficult to decompose and reacts slowly at low temperatures. Similarly, the analysis showed the highest K content at 100-200 °C with a burning time of 5 hours and the lowest at 100-200 °C and 200-300 °C with a burning time of 8 hours. A longer burning time causes more potassium content to be lost because its volatility is different from that of hard-to-decompose phosphorus. Table 2 shows that low organic C contributed to the high potassium content. This is in line with what showed that increasing pyrolysis time increases the loss of volatile components and fixed carbon value.

The analysis showed that the highest content of calcium, magnesium, and sodium was at 200–300 °C with a burning time of 4–5 hours, while the lowest was at 100–200 °C for 4, 5, and 6 hours. The results showed that higher temperatures increase the produced Ca, Mg, and Na content. This might be due to the increased pH and cations content of biochar at 200–300°C. The results indicate that the pH of each biochar was alkaline, with a moderate base category ranging from 7.30 to 8.50. The pyrolysis process decomposed three biomasses of lignin, cellulose, and hemicellulose content. Furthermore, it produces volatile substances that regulate the pH of biochar, forming a carboxyl functional group on the biochar surface. Biochar is generally alkaline with a pH of between 7.1 to 10.5 due to the presence of carboxyl groups, oxygen, and carbonate (Sukmawati et al. 2020). These components are produced by decomposing cellulose and hemicellulose into organic and phenolic acids during pyrolysis at 200-300 °C (Spokas et al. 2012)

The highest CEC analysis results were reported at 200-300 °C with a burning time of 1 hour and the lowest at 100-200 °C for 8 hours. The cation exchange capacity of biochar shows how well the nutrients bound to biochar are available for plant uptake and prevent leaching from ground and surface water. The higher the CEC represents the better quality of the biochar and this could increase soil fertility. This is because biochar holds and stores nutrients in the soil colloid, which stops them from being washed away by ground or surface water.

3.2 FTIR Analysis of Oil Palm Empty Fruit Bunch Biochar

Results presented in Table 3 show the FTIR spectrum of oil palm empty fruit bunches biochar produced by pyrolysis at 100–200 °C (T1) and 200–300 °C (T2). The burning time was 4 hours (W1), 5 hours (W2), 6 hours (W3), 7 hours (W4), and 8 hours (W5). The results of the study revealed that the ten analyses have similar FTIR spectra based on the resulting functional groups. Functional group analysis also has similarities with research that has been carried out by Sukmawati et al. (2020) and Rosli et al. (2016). Figure 4 shows the FTIR spectrum pattern of biochar absorption from oil palm empty fruit bunches. Treatments T1 and T2 show the absorption spectrum of stretching hydroxyl vibrations (OH) appearing on all biochar surfaces at wavenumbers between 3415.93 and 3446.79 cm^{-1}. The spectrum shows a shift in wavenumber with changes in time and temperature.

Table 3 FTIR Spectrum of Oil Palm Empty Fruit Bunch Biochar

Biochar	Hydroxyl	Alkanes		Carbonyl	Aromatic	Alcohol
	(O-H)	(-CH)	(C-H)	(C=O)	(C=C)	(C-O)
T1W1	3414.00	2922.16	1427.32	1693.50	1600.92	1028.06
T1W2	3415.93	2922.16	1433.11	1701.22	1591.27	1029.99
T1W3	3444.87	2922.16	1427.32	1693.50	1602.85	1026.13
T1W4	3415.93	2922.16	1433.11	1701.22	1595.13	1024.20
T1W5	3415.93	2922.16	1417.68	1693.50	1598.99	1028.06
T2W1	3417.86	2924.09	1446.61	1689.64	1606.70	1029.99
T2W2	3444.87	2922.16	1427.32	1697.36	1591.27	1018.41
T2W3	3417.86	2922.16	1419.61	1689.64	1598.99	1028.06
T2W4	3446.79	2922.16	1427.32	1693.50	1597.06	1026.13
T2W5	3421.72	2924.09	1425.40	1716.68	1610.56	1045.42

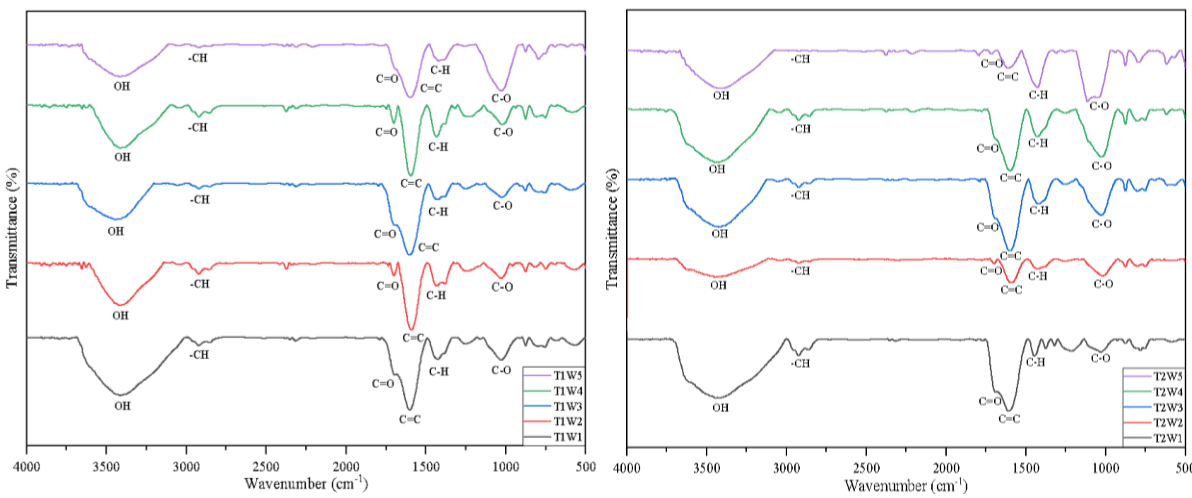

Figure 4 FTIR Spectrum of OPEFB Biochar at variations in temperature and burning time

Figures 4a and 4b show that the transmittance intensity produced by the hydroxyl (O-H) stretching vibration decreases with time. This indicates that the percentage of IR radiation transmitted decreases with combustion time. The stretching vibrational spectrum of alkanes (-CH) in the T1 and T2 treatments appears in the wavenumber of 2922.16–2924.09 cm^{-1}. The wavenumber is strengthened by bending the alkane (CH) vibration spectrum at 1417.68–1446.31 cm^{-1}. The stretching vibrational spectrum of alkanes (-CH) does not show a significant shift in wavenumber. The bending vibrational spectrum of alkanes (C-H) shows a shift in wavenumber with changes in temperature and time. Moreover, it shows that the transmittance intensity produced by the stretching vibrational spectrum of alkanes (-CH) decreases with time. In contrast, the transmittance intensity produced by the bending vibration spectrum of alkanes (C-H) increases with time. The spectrum of carbonyl (C=O) stretching vibrations in T1 and T2 treatments appeared at wavenumbers of 1689.64–1716.68 cm^{-1}, indicating a shift with changes in time and temperature.

Figures 4a and 4b also show that the transmittance intensity produced by the carbonyl (C=O) stretching vibration spectrum decreases with time. The spectrum of aromatic stretching vibrations (C = C) in T1 and T2 treatments appeared at wavenumbers of 1591.27–1610.56 cm^{-1}. This indicates a shift in wavenumbers with changes in time and temperature. Also, it showed that the transmittance intensity produced by the stretching aromatic vibration spectrum (C=C) decreased with time. The spectrum of alcohol (OH) stretching vibrations in T1 and T2 treatments appeared at a wavenumber of 1018.41–1045.42 cm^{-1}. This shows a shift in wavenumber with changes in time and temperature. The figures show that the transmittance intensity generated by the stretching alcohol (OH) vibration spectrum increases with time.

Conclusions

The choice of temperature and combustion time depends on the desired nutrients, each element required a specific temperature and time to produce the highest nutrients. The characterization results showed that organic C, nitrogen, and pH gave the highest values at a temperature of 200-300 ºC and a burning time of 8 hours. The highest content of P, Ca, and Mg is at a temperature of 200-300 ºC with a burning time of 5 hours, K dd content at a temperature of 100-200 ºC for 5 hours, and the content of Na and CEC at a temperature of 200-300 ºC with a burning time of 4 hours.

Conflicts of interest

There are no conflicts to declare.

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