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Biochar from Agricultural Waste for Soil Amendment Candidate under Different Pyrolysis Temperatures

Abdul Mutolib[1,2,3], Ali Rahmat[1,], Endra Triwisesa[1], H. Hidayat[1], Hari Hariadi[1], Kiki Kurniawan[1], Sutiharni[4], Sukamto[5]*

[1]Badan Riset dan Inovasi Nasional, Indonesia
[2]Lancaster Environment Centre, Lancaster University, United Kingdom
[3]Program Pascasarjana, Universitas Siliwangi, Indonesia
[4]Fakultas Pertanian, Universitas Papua, Indonesia
[5]Graduate school of Life Science, Hokkaido University, Japan
Correspondence: E-mail: alir001@brin.go.id

ABSTRACT

Indonesia as an agricultural country produces and consumes a huge amount of fruits. One of the popular fruits is durian (Durio zibethinus), which can create issues with the high amount of durian seeds. Durian seeds can be fermented quickly. Thus, if they are not processed immediately, it causes pollution to the environment, particularly an odor issue. One of the waste management is to convert durian seed waste into biochar, an ameliorant agent to improve soil quality. This study aims to analyze the physicochemical properties of durian seed-derived biochar under different pyrolysis temperatures. The results showed that the increases in the temperature allowed the creation of more porosity in the biochars, which is due to chemical structure and crystallinity arrangement. This is confirmed by the increases in surface area and total pore volume also decreases in particle size. The change in the chemical structure can be verified by the decreases in the biochar yield. The produced biochar from fruit seeds has excellent carbon content and elemental components such as potassium, magnesium, phosphor, and sulfur, informing prospective fruit seeds as a soil amendment fertilizer.

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1. INTRODUCTION

Indonesia is a developing country where the agriculture sector continues to be the primary income. The agricultural sector becomes of great interest to people, particularly those who live in rural areas. However, increasing population and industrialization resulting the conversion of productive agricultural land to residential or urban property. The negative impact of that conversion threatens national food security. One of the best ways to prepare for the loss of agricultural land would be to maximize the use of the acidic, dry, and marginal land, which totals more than 140 million ha. Dryland is a natural resource with enormous potential for plantations, horticulture, and agricultural expansion for food crops (Sumarniasih & Antara, 2021). However, the dryland has limitations such as shallow solum, low content of soil organic carbon, some areas with low pH conditions, and low fertility rates (Fajrina *et al.,* 2019). Dry land has been utilized for agriculture such as plantations, horticulture, and grazing despite its low soil fertility (Sukarman *et al*., 2021).

Farmers usually use organic materials such as composts, mulches, and manure to increase soil fertility. Indeed, Indonesia is a humic tropical country where the use of organic matter (compost, mulch, and manure) has frequently been shown to increase soil fertility; due to the rapid mineralization of organic matter, the benefits typically last only one or two growing seasons (Diels *et al.,* 2004). Because of that problem, the exploration of stable organic matter such as biochar is needed to increase soil fertility. Biochar, a carbon-rich material produced by heating organic biomass under oxygen-limited conditions, appears to be a more stable carbon source, remaining in the soil for an extended period (Lehmann *et al.,* 2006, Mašek *et al.,* 2013). The beneficial effects of biochar on improving soil properties have been reported:

(i) Soil organic content,
(ii) Electric Conductivity (EC),
(iii) Cation Exchange Capacity (CEC),
(iv) pH condition(Chintala *et al.,* 2014, Liu *et al.,* 2016),
(v) Soil physical properties (Chan *et al.,* 2008)
(vi) Other chemical properties (Yamato *et al.,* 2006),
(vii) Soil biological properties (Rondon *et al.,* 2007).

One of the potential sources for producing biochar is durian waste such as seeds of durian (*Durio zibethinus* L.). Durian is a type of tropical fruit plant known as The King of Fruit (Feng *et al.,* 2016). Durian is one of the most popular types of fruit in Indonesia, has a distinctive taste and aroma, and is loved by many people (Najira *et al.,* 2020).

There are about 31 types of durians worldwide, 19 of which are found in Kalimantan, and 7 other types of durians are scattered in Sumatra and most of them still grow wild in the forest (Navia & Chikmawati, 2015). In consuming durian fruit, people generally eat the flesh of the fruit (about 20-35%), while other parts such as the skin (60-75%) and seeds (5-15%) will become waste. Durian seeds are the part that ferments quickly. If they are not immediately processed, they will cause pollution to the environment, especially a foul smell. Thus, the maximum utilization of durian seed waste is needed and the conversion of this waste into some added-value products is the best choice, such as brake pads (Nandiyanto *et al*., 2021; Nandiyanto *et al*., 2022) and briquettes (Nandiyanto *et al*., 2020a). Other choices for solving the problems regarding organic waste have been well-documented, such as converting the organic waste into carbon that can be used as adsorbents. Several examples of carbon production from organic waste are available, including pineapple peel waste (Nandiyanto *et al*., 2020b), rice husk (Fiandini *et al*., 2020), pumpkin seeds (Nandiyanto, 2020), typa and grass (N'diaye *et al*., 2022), soursop peel

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(Nandiyanto *et al*., 2020c), and dragon fruit peel (Nandiyanto *et al*., 2020d). Different from other reports, here, this study converts durian seeds into biochar through the pyrolysis process. Pyrolysis is a thermochemical decomposition process of organic or synthetic materials to produce fuel (in the form of bio-oil) at high temperatures in oxygen-poor conditions. This process is one of the excellent processes and even it is learned in the school and vocational school as well as designed for supporting many production systems (Pebrianti & Salamah, 2021; Nayaggy & Putra, 2019; Subagyono *et al.*, 2021; Jamilatun *et al.*, 2023). This process is influenced by several factors, including the duration and temperature of the pyrolysis process. Good pyrolysis occurs at temperatures between 370-420°C (Thorat *et al.*, 2013). The excellent temperature for carbon preparation is more than 200°C (Ragadhita & Nandiyanto, 2023). The composition of biochar is determined by the type of biomass and the carbonization process (Antonangelo *et al*., 2019). Lignin and mineral content, particle size, pyrolysis temperature, heating rate, residence time, and pressure affect biochar properties (Alhinai *et al*. 2018). Reza *et al.,* (2020a) reported that increases in temperatures have increased the porosity of the biochars. Moreover, Claoston *et al*. (2014) explained that pyrolysis temperature influences the nutrient content of biochar. However, the research related to the physicochemical of durian seed biochar is still limited. Thus, this study aims to investigate the physicochemical properties of durian seed biochar under different temperatures of pyrolysis.

2. METHODS

2.1. Material Preparation

Samples of durian seed were obtained from the local market. Then, they were washed and cut into thin slices before drying in the air for 24 hours (**Figure 1a**). After air-drying for 24 hours (**Figure 1b**), the sample was placed in a 105°C oven for 24 hours to remove the moisture content. The sample is then sealed in plastic.

2.2. Pyrolysis

The pyrolysis of durian seed (*Durio zibethinus*) was accomplished by inserting dried durian seed samples into covered porcelain with a volume of 50 mL and a sample weight of 50-60 grams/porcelain. The porcelain was then tightly wrapped in aluminum foil to limit air interaction. The samples were then placed in a furnace at various pyrolysis temperatures (i.e. 300, 400, 500, and 600°C) for 4 hours. After the process, the remaining biochar in the reactor was collected, ground, and sieved with a 355-μm sieve. The sieve process results were used for further analyses.

(a) (b)

Figure 1. Raw material (a) and biochar after pyrolysis (b).

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2.3. Scanning Electron Microscope

The Field Emission Scanning Electron Microscopy (FESEM)-Energy Dispersive Spectroscopy (EDS) from the FESEM Thermo Scientific Quattro S completed with EDS Detector was used to capture the surface morphology of biochar. The same SEM-EDS engine was used to calculate the atomic and weight percentages of components, particularly carbon and oxygen.

2.4. Surface Analysis and Fourier Transform Infrared Analysis

Specific surface area and pore size distribution were measured using Brunauer, Emmett, and Teller (BET) methods. The biochar samples' functional groups were determined using Fourier transform infrared spectroscopy (FTIR-UATR Perkin Elmer Spectrum Two) (Nandiyanto *et al.,* 2019; Sukamto & Rahmat, 2022; Nandiyanto *et al*., 2023).

2.5. X-Ray Flourences Analysis

The chemical constituents of biochar samples were characterized using the X-ray fluorescence (XRF) spectroscopy technique. The Omnian ED- XRF PANalytical Epsilon 3 XLE X-ray fluorescence spectrometer was used, and the wavelength dispersion was measured.

3. RESULTS AND DISCUSSION

3.1. Biochar Yield, BET Surface and Scanning Electron Microscope (SEM)

Durian seed is a byproduct of agriculture that contains cellulose, hemicellulose, and lignin. The pyrolysis process is associated with the breakdown of cellulose, hemicellulose, and lignin. The pyrolysis response varies between species. The pyrolysis of durian seeds at temperatures of 300, 400, 500, and 600°C produced biochar yields of 37.88, 29.76, 26.04, and 23.11%, respectively. Biochar production decreased as pyrolysis temperature increased, as observed in the studies of Angin (2013) for safflower seed waste, Reza *et al*. (2020a) for *Pennisetum purpureum* grass, and Zhu *et al.,* (2019) for poplar wood sawdust. Because lignin has a complex structure, the decomposition pattern is slow and stable, which is essential for biochar production (Mccarthy & Islam, 2000). As a result, the yield loss in this study could be attributed primarily to the destruction of the lignin-cellulose structure and the combustion of organic materials at high pyrolysis temperatures (Al-Wabel *et al.,* 2013). The main factors influencing biochar properties are the pyrolysis temperature and the characteristics of the biomass (Antal, 2003).

Table 1 shows the BET surface area and total pore volume data of all biochar samples produced at different temperatures. All biochar samples generally show the same trend for BET surface area and total pore volume. BET surface area for all samples ranged from 1.037 to 26.62 m^2/g. At the pyrolysis temperature of 300 to 500°C, there was no significant increase in the BET surface area. However, when the pyrolysis temperature increased to 600°C, the BET surface area increased significantly to 26.62 m^2/g.

Table 1. Biochar yield and BET surface area analysis of durian seed biochar.

Sample	Yield (%)	BET Surface Area (m^2/g)	External Surface Area (m^2/g)	Total Pore Volume x 10^{-3} (cc/g)	Average Pore Diameter (nm)
BD-300	37.88	1.037	1.019	2.693	10.39
BD-400	29.76	2.319	2.289	5.133	8.855
BD-500	26.04	2.288	2.276	14.99	26.19
BD-600	23.11	26.62	26.25	18.14	2.726

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The evaporation of most volatile organic components with increasing pyrolysis temperature could cause it. It can be confirmed via the yield generated. At 300°C, the resulting biochar reached 37% in yield. The yield decreased with increasing pyrolysis temperature. The loss of volatile organic components in durian seeds causes the formation of pores and increases the BET surface area of the resulting biochar.

The higher the BET surface area produced, the higher the ability of biochar to absorb water and improve soil quality. If the surface area is small, then the material's ability to absorb water and nutrients in the soil will be limited, as in sandy soils, which have a relatively low surface area of 0.01 m^2/g. The total pore volume increases proportionally with increasing pyrolysis temperature. The highest total pore volume was achieved at a pyrolysis temperature of 600°C. It is possible due to the deformation of the boundary wall between the pores, which causes an increase in pore volume (Shaaban *et al.,* 2014).

According to **Figure 2**, the pyrolysis method using different temperatures can produce different morphology and pore structure in the obtained durian seed biochar. At 300°C, the morphological structure of the biochar shows the formation of pores that are larger when compared to other biochar. It can be promoted by the evaporation of volatile organic compounds contained in durian seeds (Shaaban *et al.,* 2013). At a low pyrolysis temperature (300°C), most organic compounds evaporate, providing more prominent pores. The higher the pyrolysis temperature, the lower the organic compound content and resulting smaller pores. This is consistent with the results of previous studies, where the higher temperature resulted in the creation of more pores and cracks, as shown in the biochar produced at 600°C (Hao *et al.,* 2013; Méndez *et al.,* 2013; Mohanty *et al.,* 2013).

The increase of pores can reduce the pore diameter on the surface of the biochar. Based on BET analysis in **Table 1**, the durian seed biochar produced at 300°C has a large pore diameter of 10.39 nm. At higher pyrolysis temperatures, the pore diameter decreases with an increasing number of pores. The pore diameters formed at pyrolysis temperatures 400, 500, and 600°C were 8.855, 26.19, and 2.726 nm, respectively. The existence of this pore structure can increase the potential of biochar as a soil amendment because it provides space for root movement and acts as a habitat for various microbes in the soil. Moreover, the decrease in pore size also correlated to the decrease of particle size by the increase in pyrolysis temperature as shown in **Figure 3.**

Figure 3 shows that the particle sizes of Durian seed biochar decreased with the increase in pyrolysis temperature based on SEM data using Image-J software. Detailed information on how to measure particle size is reported elsewhere (Yolanda & Nandiyanto, 2022). On the pyrolysis temperature of around 300-500°C, the reduction of biochar particle size does not occur significantly.

The average particle sizes of 300, 400, and 500°C are 29.29, 25.31, and 24.075, respectively. A significant particle size reduction is observed for pyrolysis temperature at 600oC, with the average particle size around 7.065 µm. At higher pyrolysis temperatures, the volatile organic compounds are decomposed into carbon elements by releasing gas and water that lead to the particle size reduction of biochar. It may be attributed to the surface area increase, as shown in **Table 1**.

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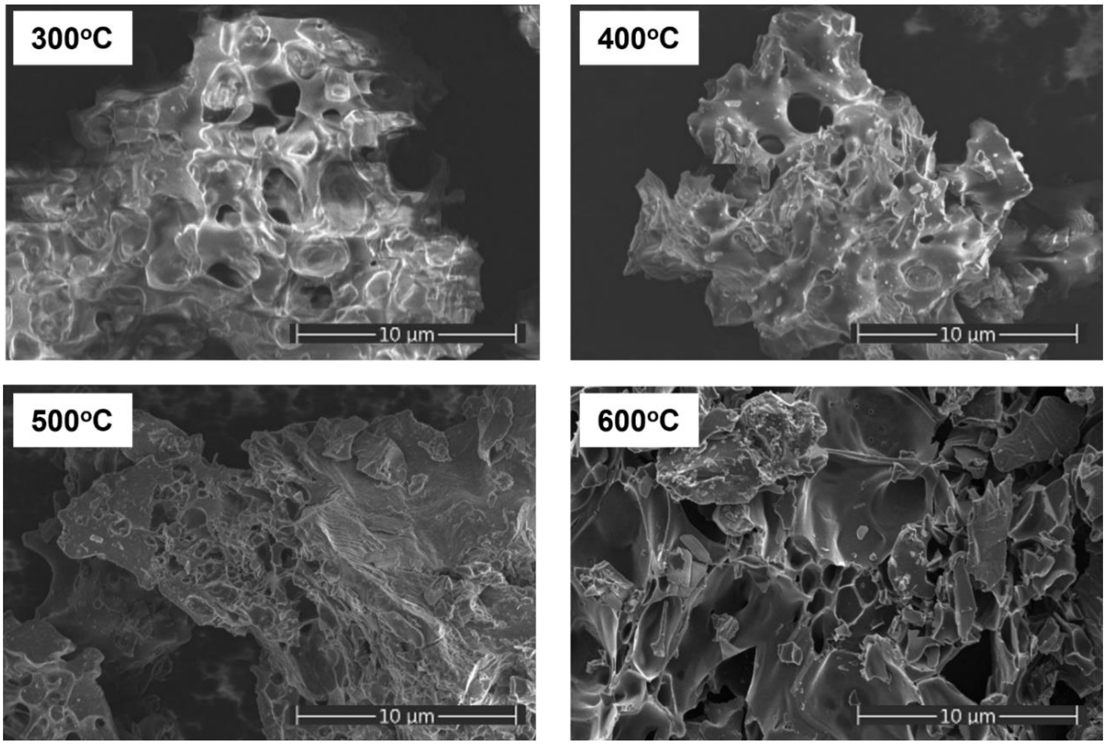

Figure 2. Effect temperature of pyrolysis on the surface morphology of durian seed biochar. Figures (a), (b), (c), and (d) are samples heated at 300, 400, 500, and 600°C.

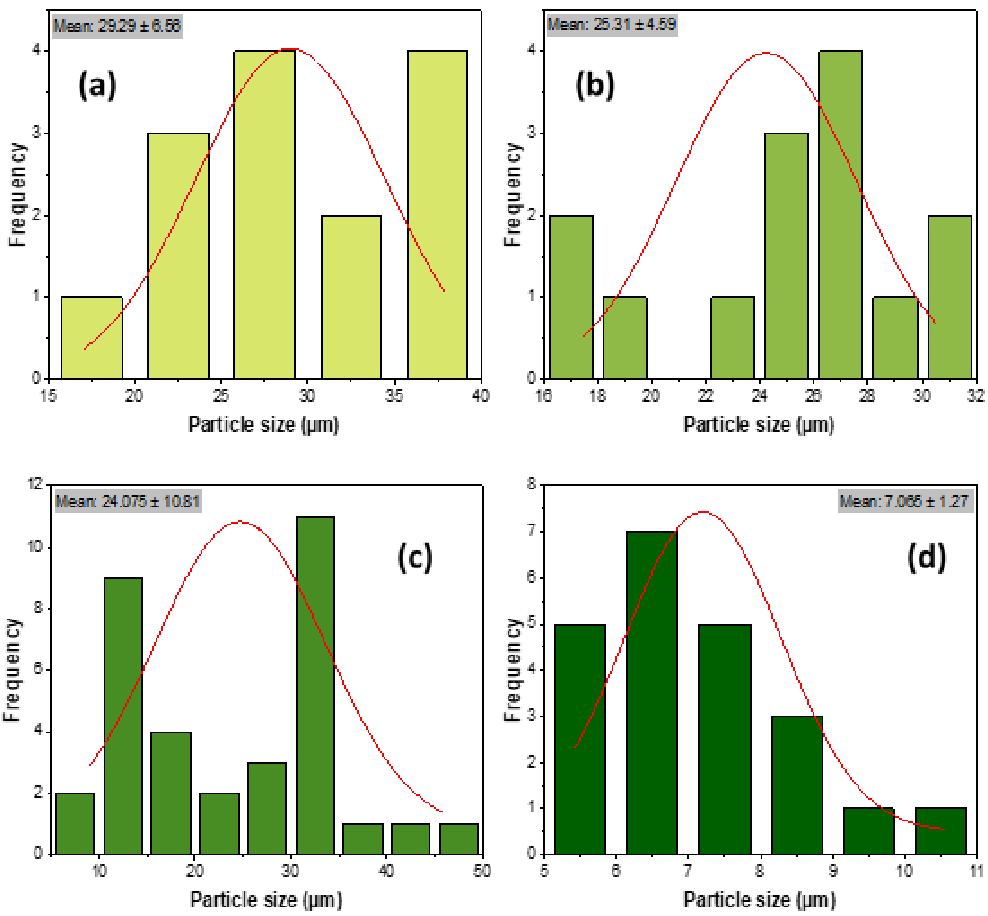

Figure 3. Particle size distribution at (a) 300oC, (b) 400oC, (c) 500oC, and (d) 600oC.

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3.2. Fourier Transform Infrared Spectroscopy

The FTIR measurements in **Figure 4** show the effect of pyrolysis temperature on the functional group's durian seed biochar. Detailed information for reading and interpreting FTIR analysis is shown in the literature. According to FTIR analysis, the biochar functional groups obtained at pyrolysis heating levels of 300, 400, 500, and 600°C are similar. However, the transmission percentage was higher for biochar at 300°C. It then decreased as the temperature rose to 500-600°C; The sample with 600°C was equivalent to that with 500°C (Özçimen & Ersoy-Meriçboyu, 2010).

The peak at 3250-3500 cm^{-1} decreased rapidly as the pyrolytic temperature increased, indicating that pyrolysis decomposes a large number of free hydroxyl bonds and associations, as well as hydroxyl structures (-COOH and -COH) associated with water, alcohols, and carboxylates (Keiluweit *et al.,* 2010). The wavenumber of 3284.37 cm^{-1} indicates O-H in this study was not detected due to the formation of a melting ring, especially at high pyrolysis temperatures, and consistently forms pore characteristics in durian samples.

The peaks detected were found in the range of wavenumber of 2960-2850 cm^{-1} for stretching of C-H bonds in aliphatic formation, generated by asymmetric and symmetric C-H stretching vibrations. This peak indicates the presence of cellulose, hemicellulose, and lignin in the durian seed raw material before increasing the pyrolysis temperature. The C-H peak disappears as the temperature rises to 500°C, indicating that aliphatic hydrocarbons are broken down into gases such as carbon dioxide and methyl hydride or converted into aromatics (Gao *et al.,* 2017). The peak wavelength was 2926 cm^{-1} for biochar at 300°C and decreased to 2915 cm^{-1} for biochar at 400°C (Antonangelo *et al.,* 2019; Reza *et al.,* 2020b) (**Table 2**). Peaks with a range of 2830–2670 cm^{-1} are C–H functional in aliphatic formation. At this stage, the wave number is 2828 cm^{-1} for durian raw material. The wavenumber for biochar 300 shifted at 2830 cm^{-1} and was no longer detected at pyrolysis temperatures of 400, 500, and 600°C (Özçimen & Ersoy-Meriçboyu, 2010; Antonangelo *et al.,* 2019).

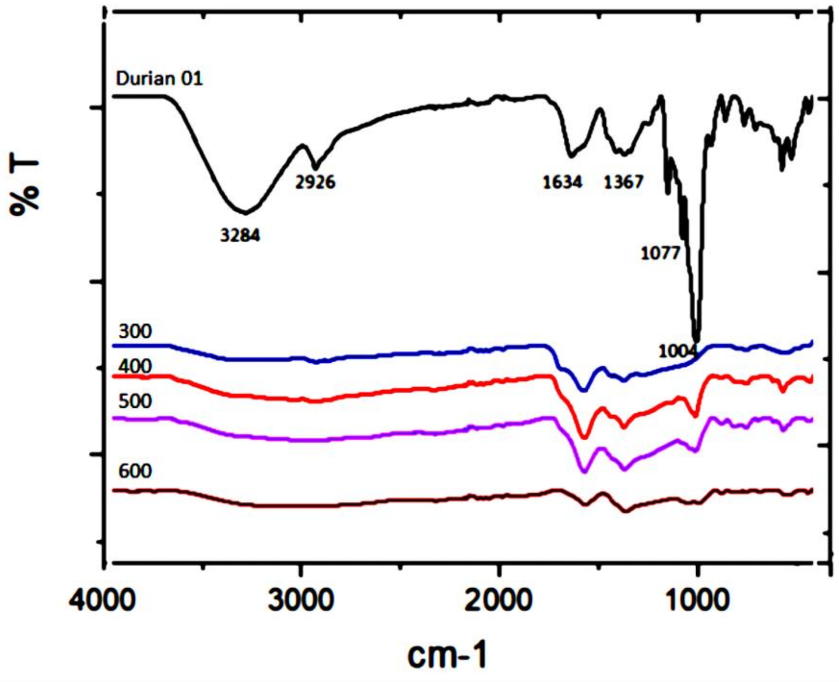

Figure 4. Raw material durian dan biochars FTIR spectrum.

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Table 2. Fourier Transform Infrared Spectroscopy.

Functional groups	Wavenumber (cm^{-1})						References
	In literature	Raw material	300 biochar	400 biochar	500 biochar	600 biochar	
O-H stretching	3250–3500	3284	-	-	-	-	Keiluweit *et al.,* 2010
C– H stretching in aliphatic formation	2960–2850	2926	2926	2915	-	-	Gao *et al.,* 2017
C–H Bending in aliphatic formation	2830–2670	2828	2830	-	-	-	Özçimen & Ersoy-Meriçboyu (2010), Antonangelo *et al.,* (2019)
C=O stretching in ketene groups	2360–1910	2324	2323	2323	2323	2323	El-Hendawy (2006), Reza et al. (2019)
C=C stretching of hemicelluloses	1610–1510	1634	1575	1575	1575	1575	Popescu et al. (2018)
C–H deformation in cellulose and hemicellulose	1480-1410	1416	1396	1378	1368	1376	Traoré et al. (2015)
C–O stretching vibration in cellulose and hemicelluloses	1120–1050	1077	-	1085	1079	1044	Popescu et al. (2018), Reza et al. (2019)
C–O stretching in cellulose	995–905	967	-	902	-	-	Traoré et al. (2015), Popescu et al. (2018)
C=C stretching alkene vinylidene	895–880	-	894	885	889	889	Özçimen and Ersoy-Meriçboyu (2010), Reza et al. (2019)
Aromatic rings	880–720	862	753	795	816	820	Antonangelo et al 2019

The peak for the area from 1910 to 2360 cm^{-1} represents C=O stretching in the ketene (El-Hendawy, 2006; Reza *et al.,* 2020b). Most of the carbonyl groups in the biomass component (i.e. lignin, cellulose, and hemicellulose) are in the hemicellulose chain elements. The group's concentration depends on the holocellulose content (the mixture of cellulose and hemicellulose in the cell walls of plants) ratio to the lignin portion in all biomass (Colom & Carrillo, 2005). In durian raw material, the wavenumber occurs at the peak of 2324 cm^{-1} and changes with an increase in temperature of 300°C. The wavenumber from 300-600°C tends to remain at 2323 cm^{-1}. All of this indicates that the soft carbon components are being removed, and the hard carbon components are still present (Chen & Chen, 2009).

The peak at a wavenumber of 1610 to 1590 cm^{-1} corresponds to C=C, indicating hemicellulose. The peak shifted down from 1575 cm^{-1} for biochar at 300°C to 1567 cm^{-1} and biochar at 600°C) (Popescu *et al.,* 2018).

The peak intensity decreased slightly as the temperature rose, indicating that temperature had little effect on the aromatics in the biochar. This could be due to carbohydrate ring de-hydrolysis and cyclization during pyrolysis, resulting in newly aromatized and carbonized substances (Chia *et al.,* 2012). The aromatic ring for C = C

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stretches and bends H–O–H for water (Akhtar *et al.,* 2016). Significant peaks for C-O stretching in the biochar samples occurred in the 1120-1050 cm^{-1} wavenumber range, indicating cellulose and hemicellulose. The highest wavenumber peaks discovered were 1085, 1079, and 1044 cm^{-1} for biochar 400, 500, and 600°C, respectively (Özçimen & Ersoy-Meriçboyu, 2010; Popescu *et al.,* 2018; Reza *et al.,* 2019). Because of the weaker aromatic C-O bonds, the C-O bond in the aryl ring decreases significantly with increasing pyrolysis temperature. This primary bond breaks when a larger aryl ring is formed (Abidi *et al.,* 2014). Meanwhile, in the pyrolysis process, various forms of oxygen in the sludge combine with activated carbon atoms and transfer to the aromatic ring to form new C-O bonds, making the aromatic C-O bonds more stable in pyrolysis (Jin *et al.,* 2016).

Peaks found between 995 and 905 cm^{-1} correspond to C-O spanning in cellulose. With higher-temperature biochar, the number of crest waves has decreased (Traoré *et al*., 2015; Popescu *et al*., 2018). The FTIR peaks for the C=C stretching of vinylidene alkenes range between 895 and 880 cm-1 as the number of peak waves increases. (Özçimen and Ersoy-Meriçboyu 2010; Reza *et al*., 2019). The wavenumber indicates a C=C bond has occurred in alkenes in the range 842 to 720 cm^{-1} for aromatic rings. For wavenumbers at 753, 795, 816, and 820 cm^{-1}, the peaks increase with increasing temperature (i.e. 300, 400, 500, and 600°C) (Chen *et al*., 2012; Antonangelo *et al*., 2019).

3.3. Element Content of Durian Seed Biochar

The results of the elemental analysis of biochar obtained at various pyrolysis temperatures are shown in **Table 3**. The pyrolysis temperatures affect the elemental composition of biochar. With an increase in pyrolysis temperature from 300 to 600°C, the carbon content of biochar increased from 80.20 to 84.30 wt%. Increasing temperature increases dehydration, elimination reactions, and carbon content. At lower temperatures, primary thermal degradation of lignocellulosic biomass occurs. Increasing temperature further breaks down volatile materials into organic compounds and gases with lower molecular weight than biochar (Chen et al. 2012).

The O/C ratio indicates polarity, and the abundance of polar oxygen-containing surface functional groups in biochar, the higher the ratio, the more polar functional groups there are. Also, these groups actively take part in the adsorption of heavy metals (Chatterjee *et al*., 2020). The highest biochar O/C ratio was at 400°C, while the lowest biochar O/C ratio was at 600°C. It can be concluded that the addition of pyrolysis temperature can reduce the value of the O/C ratio. Further increase in the pyrolysis temperature indicates a small change in elemental carbon. The increase in carbon content at higher temperatures reflects an increase in the degree of carbonization (Zhou *et al.,* 2013).

The content of volatile components decreased with increasing temperature. The same situation occurred during the carbonization of safflower seed-based biochar (Angin 2013). **Table 3** shows that the durian seed biochar contains various concentrations of the element such as K_2O, P_2O_5, MgO, and SO_3. K_2O is the most abundant element in durian seed biochar, accounting for 86.89-89.00% of the total. The highest K_2O and SO_3 concentrations were found in BD-600, at 89.00% and 2.41%, respectively. Meanwhile, BD-300 had the highest concentrations of P_2O_5 and MgO, with 9.49 and 2.25%, respectively.

Furthermore, durian seed biochar contains other elements such as MnO, Fe_2O_3, CuO, ZnO, Rb_2O, and SrO at concentrations well below 0.5%. The concentrations of the elements K_2O, P_2O_5, MgO, and SO_3 were influenced by increasing the pyrolysis temperature, but no clear pattern appeared.

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Table 3. Effect temperature of pyrolysis on chemical properties of Durian seed biochar.

Chemical Properties		Pyrolysis temperature			
Element	**Raw material**	**300°C**	**400°C**	**500°C**	**600°C**
C (%)	-	80.2	80.5	76.5	84.3
O (%)	-	15.0	16.4	9.2	8.6
O/C	-	0.187	0.204	0.120	0.102
Main Element					
K_2O (%)	81.231	86.898	87.412	86.704	89.003
P_2O_5 (%)	9.497	7.736	7.621	8.167	6.524
MgO (%)	1.992	2.248	2.266	2.654	0.558
SO_3 (%)	5.232	2.337	1.899	1.543	2.408
Other Element					
MnO (%)	0.123	0.097	0.077	0.0918	0.077
Fe_2O_3 (%)	0.569	0.274	0.319	0.347	0.804
CuO (%)	0.190	0.090	0.094	0.103	0.116
ZnO (%)	0.198	0.101	0.097	0.101	0.159
Rb_2O (%)	0.389	0.196	0.187	0.197	0.265
SrO (%)	0.039	0.022	0.020	0.192	0.006

The variability of element content in biochar with increasing temperature is due to their volatility as well as the effect of pyrolysis temperature on the composition and chemical structure of the biochar. Furthermore, the concentration of the element in biochar is affected by the process of partial defraction or devolatilization of these elements at high temperatures (Hossain *et al.,* 2011; Claoston *et al.,* 2014).

4. CONCLUSION

The experiment shows the properties of biochar made from durian seed waste at four different pyrolysis temperatures (300, 400, 500, and 600 C). Increased pyrolysis temperature reduces biochar yield while increasing BET surface, external surface area, and total pore volume. The BD-600 treatment had the highest BET surface, external surface area, and total pore volume values and the lowest particle size. Furthermore, the FTIR analysis revealed that the biochars' available functional groups were C-H, C=O, C=C, and C-O. The O/C atomic ratios for biochar at 300, 400, 500, and 600 °C were 0.187, 0.204, 0.120, and 0.102 based on energy dispersive X-ray analysis. The carbon content of durian seed biochar is high (76.5-84.3%), as is the K2O content (86.7-89.0%), followed by P2O4 (6.52-8.17%), MgO (0.56-2.65%), and SO3 (0.56-2.65%). (1.54-2.41). Biochar has the highest carbon and K2O content under 600 °C, with values of 84.3% and 89%, respectively. In this study, the elemental composition of biochar was affected by increasing pyrolysis temperature. Biochar from durian seed has a high potential for soil amendment based on its carbon and macronutrient content.

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6. AUTHORS' NOTE

The authors declare that there is no conflict of interest regarding the publication of this article. The authors confirmed that the paper was free of plagiarism.

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