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Manufacture of Industrial Scale Bagasse Biochar: Effect of Temperature And Residence Time and Biochar Characterization

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ABSTRACT. Bagasse is a waste that can still be utilized, one of which is processed into biochar. In this study, bagasse waste came from a sugar factory in Majalengka, Cirebon, West Java. The bagasse pyrolysis process was carried out at PT XXX biochar factory located in the area. Industrial-scale biochar production is done by Rotary Carbonization Furnace. The research objective was to characterize biochar with variable residence time and pyrolysis temperature. Biochar was characterized based on physicochemical properties and surface composition analysis for use as a soil improver and adsorbent. The test results show that residence time and pyrolysis temperature affect biochar products. Physical characterization showed that the pyrolysis residence time of 24.73 minutes with a temperature of 400^{o}C gave biochar results with pH (8.92), c-organic (24.15%), total N (0.2%), P_2O_5 (0.17%) and high C/N ratio (1,208.00). This biochar has good quality for application as a soil improver, especially in increasing carbon storage capacity and improving soil pH. The high C/N ratio and low nitrogen content require alloying with other sources to increase nitrogen and phosphorus and lower the C/N ratio. Chemical characterization by BET test showed that pyrolysis residence time of 24.73 min at 400^{o}C gave the best results in terms of increasing surface area (0.554 m²/g) and *pore volume* (0.00364 cc/g), making it the optimal temperature to produce biochar with high adsorption capacity. Surface characterization by SEM-EDX mapping analysis showed that the pyrolysis residence time of 24.73 min at 400^{o}C gave results with a relatively high composition of carbon (82.17%), oxygen (14.89%), silica (1.97%), potassium (0.42%), and made it more effective for soil conditioner applications.

1. INTRODUCTION

Sugarcane (Saccharum officinarum Linn) is a plant that is cultivated as a sugar producer [1]. Based on data from the Indonesian Sugar Plantation Research Center, milled sugar cane produces bagasse by as much as 32% [2]. Bagasse has a moisture content of about 46-52%, fiber content of 43-52%, and soluble solids of about 2-6%. [3]. Sugarcane bagasse (Saccharum officinarum Linn) contains organic compounds in the form of cellulose, hemicellulose, and lignin, which can be converted into carbon (biochar) through pyrolysis from 200^{o}C to 700^{o}C for approximately 1 to 4 hours [4]. Sugarcane bagasse biochar has carbon content from the results of carbonization in the form of cellulose 35.01%, hemicellulose 25.24%, lignin 6.4% and silicate 9.35%. Sugarcane bagasse biochar has a porous structure, so it can increase the availability of water and minerals in the soil [5].

Pyrolysis is a thermochemical conversion process that converts biomass and organic matter into biofuels, i.e., vegetable oil, biochar, and non-condensable. The process is defined as the thermal decomposition of biomass in a closed reactor in the absence of oxygen. The distribution of pyrolysis products depends on the process parameters as well as the biomass composition. The most important parameters are reactor type, pyrolysis temperature, heating rate, biomass particle size, residence time and sweep gas (N_2) flow rate. The product yield from pyrolysis depends on the operating parameters, the properties of the biomass, and the type of pyrolysis process. Controlling and optimizing these parameters is critical to maximize the yield of edible oil and optimal product distribution [6].

The *surface* characterization of biochar was carried out by Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX) spectrophotometer by scanning the electron beam on the surface of the sample to identify the characteristics of biochar, which aims to determine the presence of pores on its surface. In addition, SEM collects data on the pore shape from the electron morphology of the sample and the amount of minerals contained in the composition [1,7].

Biochar quality is influenced by the type of biomass and the pyrolysis process performed [8]. Biochar is produced using a pyrolysis temperature gradient ranging from 300°C to 700°C. Low-temperature pyrolysis produces slightly acidic biochar, while high-temperature pyrolysis produces slightly alkaline biochar; with increasing pyrolysis temperature, biochar yield and plant-available nitrogen content decrease, while the content of trace elements (Ca, Fe, Mg, S, Cu, and Zn) in biochar increases [9].

In this study, biochar was prepared from bagasse on an industrial scale with variable residence time and pyrolysis temperature. Biochar was characterized based on its physicochemical properties and surface use as a soil conditioner and adsorbent.

2. Experimental Parts

2.1 Preparation and Characterization of Sugarcane Bagasse

The raw materials in this study were obtained from a sugar factory in Majalengka Cirebon, West Java, Indonesia. This research is an industrial-scale biochar production, namely the pyrolysis process and biochar characterization. This study produced biochar through a pyrolysis process with a temperature variation of 350°C to 450°C and a residence time of 17 to 52 min. Sugarcane amps were analyzed for cellulose, hemicellulose and lignin.

2.2 Biochar Production and Pyrolysis Process

Pyrolysis equipment with a series of dryers, biochar equipment with auxiliary support in the form of feeder belt conveyor, screw conveyor, fan and combustion system for starting. The pyrolysis process can be seen in Figure 1.

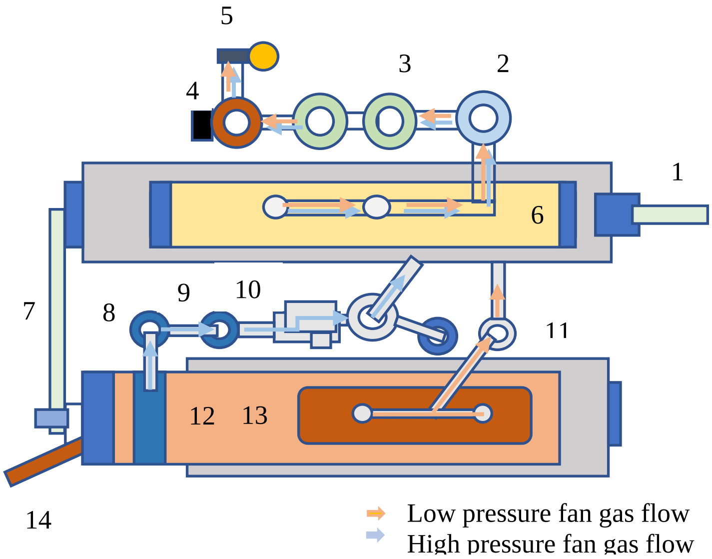

Figure 1. Pyrolysis process.

Description:

1.	Feeder conveyor	6.	Drum dryer	11.	Low pressure fan
2.	Dust removal	7.	Screw feeder conveyor	12.	Primary drum pyrolysis
3.	Cooling & purification	8.	Dust removal a	13.	Secondary drum pyrolysis
4.	Scrubber	9.	Dust removal b	14.	Biochar screw dicharge conveyor
5.	Exhaust fan	10.	High pressure fan		

2.3 Flow Chart of Biochar Manufacturing Process

The process of industrial-scale biochar production and organization can be seen in Figure 2.

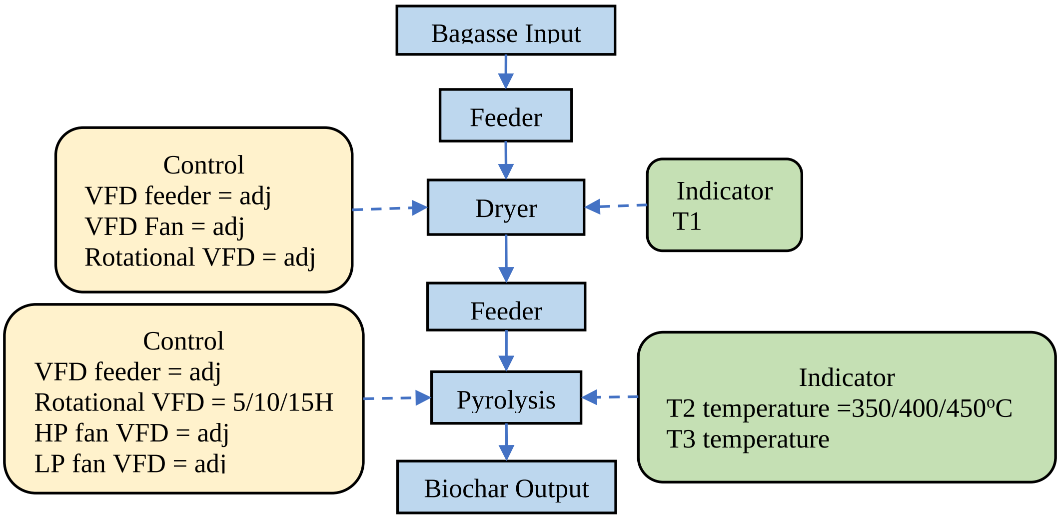

Figure 2. Process flow.

2.4 Testing the residence time of the biochar process

Pyrolysis residence time is observed by counting the time required from the start of the bagasse entering the pyrolysis rotary drum until the biochar results come out at the output. VFD indicator speed starts from 52.47 min (5 H), 24.73 min (10 H) and 17.23 min (15 H).

2.5 Biochar characterization

Biochar samples in the manufacturing process are made with 2 different processes, namely:

a. Processing with a constant residence time of 24.73 min (VFD 10 H) and pyrolysis temperatures varying at 350ºC, 400ºC and 450ºC.
b. Processing with a constant temperature of 400ºC with residence times starting at 52.47 min (VFD 5 H), 24.73 min (VFD 10 H) and 17.23 min (VFD 15 H).

Biochar samples will be characterized as follows [10]:

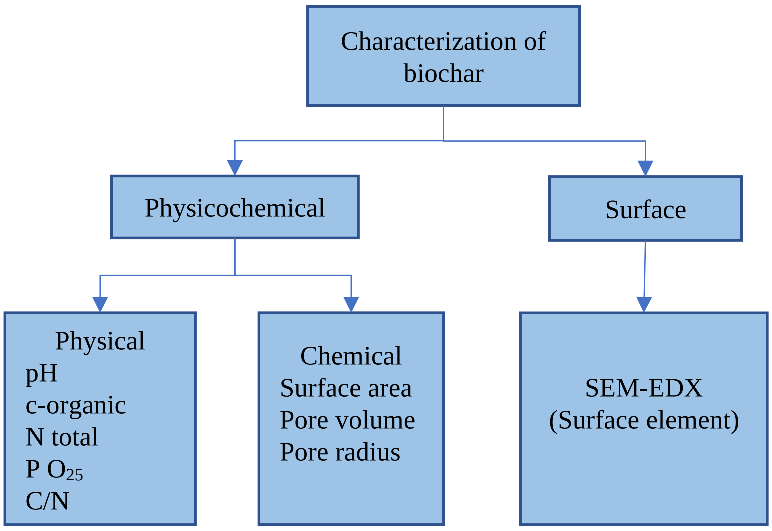

Figure 3. Characterization of biochar.

3. RESULTS AND DISCUSSION

3.1 Results of the pyrolysis residence time test

The results of the pyrolysis residence time test were calculated by counting the time required from the start of bagasse entering the rotary drum pyrolysis until the biochar results came out at the output. The observation results are presented in Table 1.

Table 1. Residence time.

Pyrolysis rotating speed VFD (H)	Residence time from entry to exit min.s	min
5 (52.47 min)	00.52.28	52,47
10 (24.73 min)	00.24.44	24,73
15 (17.23 min)	00.17.14	17,23

Furthermore, in the mention of pyrolysis residence time, the time in minutes will be used: 5H with 52.47 minutes, 10H with 24.73 min and 15H with 17.23 min.

3.2 Characterization of bagasse

In this study, three essential biomass components for biomass heat generation are cellulose 21.07%, hemicellulose 26.93%, and lignin 20.14%. Biomass composition (cellulose and hemicellulose) influences the gas profile at CO and CO_2 concentrations. Meanwhile, lignin plays a role in CH_4 concentration [11]. Lignin also plays a role in thermochemical decomposition [12]. At 325-375ºC, cellulose with the chemical formula $(C_6H_{10}O_5)_n$ will be deformed or decomposed into its components, and hemicellulose with the chemical formula $(C_5H_8O_4)_n$ will be deformed at 225-325ºC, and lignin with the chemical formula $[(C_9H_{10}O_3(CH_3O)]_n$ will be deformed at 300-500ºC [3]. The results of the raw materials analysis in this study align with the findings of previous studies by Chen and Jamilatun [13,14]. Table 2 shows the test results of bagasse for cellulose, hemicellulose, and lignin content.

Table 2. Bagasse with comparative composition of cellulose, hemicellulose and lignin**.**

No.	Cellulose, %	Hemicellulose, %	Lignin, %	Reference
1	35,01	25,24	6,4	[15]
2	34,21	9,11	22,13	[16]
3	58,00	10,00	11,00	[17]
4	45,82	20,20	21,32	[14]
5	21,07	26,93	20,14	This research

3.3 Characterization of bagasse biochar

Physical characteristics of bagasse biochar as a soil improver

Biochar made from sugarcane bagasse is an alternative environmentally friendly soil conditioner. The use of bagasse biochar in accordance with **SNI 7763-2018** refers to the basic principles of organic fertilizer, whose function is to improve soil quality without damaging the environment [18,19]. From the parameters in SNI 7763-2018 in this study, tests were carried out, including c-organic, C/N, pH and macronutrients (N, P_2O_5). Tests were carried out with 2 kinds of samples, namely: samples with a fixed temperature treatment of 400ºC with residence times varying 52.47 minutes, 24.73 minutes and 17.23 minutes and samples with a fixed residence time treatment of 24.73 minutes with pyrolysis temperatures varying 350ºC, 400ºC and 450ºC.

Based on the analysis of biochar produced in pyrolysis with a fixed residence time of 24.73 minutes at 350°C, 400°C, and 450°C, shown in Table 3. Test results show that the pH of biochar increases with the increase in pyrolysis temperature [9, 20]. The pH value of biochar is in the range of the SNI 7763-2018 standard (4-9), making it suitable for use as a soil amendment. The increase in biochar pH is due to the loss of acidic functional groups and the formation of oxide, hydroxide and carbonate mineral phases containing Ca-Mg-Na and K [21]. C-organic biochar is in the high range, indicating the potential of biochar to increase carbon content in soil and improve soil fertility. The total N of biochar decreased with increasing pyrolysis temperature, which may affect the availability of nitrogen in biochar. P_2O_5 biochar tended to be stable despite a slight decrease at 400°C pyrolysis temperature, but still met the standard phosphorus requirement for soil. The C/N ratio is very high at all three pyrolysis temperatures, far exceeding the recommended limit (max. 25) based on SNI 7763-2018. This can inhibit the availability of nitrogen in the soil when biochar is used as a soil improver.

Table 3. Physical Test Results of biochar with a constant residence time of 24.73 minutes and varied temperature

Parameters	Unit	350ºC	400ºC	450ºC	SNI 7763 -2018
pH	-	7,40	8,92	9,35	4-9
Temperature	ºC	24,90	24,87	25,00	
C-Organic	%b/b	23,66	24,16	22,71	Min. 15
N total	%b/b	0,04	0,02	0,02	
P_2O_5	%b/b	0,27	0,17	0,25	
C/N Ratio	-	591,50	1.208,00	1.135,50	Max. 25

The test results with a constant temperature of 400ºC with a residence time of 51.47 minutes, 24.73 minutes and 17.23 minutes are shown in Table 4. The pH test results of biochar produced at 400°C are within the limits by SNI 7763-2018 standards (4-9) and are classified as alkaline, which can increase the pH of acidic soil. The C-organic content of biochar is very good, exceeding the minimum standard set, and indicates that the biochar produced is rich in carbon that can improve soil quality. The total N content in biochar is very low, which indicates that biochar functions more as a carbon source and does not contribute significantly to soil nitrogen levels. The P_2O_5 content is relatively low but can still provide benefits to soils that need phosphorus. The very high C/N ratio indicates that biochar is richer in carbon than nitrogen, which may be less suitable for application as a nitrogen-rich fertilizer but is suitable as a soil amendment to increase the carbon storage capacity of the soil.

Table 4. Physical test results of biochar with a constant temperature 400ºC and varying residence time

Parameters	Unit	52.47 min	24.73 min	17.23 min	SNI 7763 -2018
pH		9,27	8,92	8,43	4-9
Temperature	ºC	24,80	24,87	24,87	
C-Organic	%b/b	19,66	24,16	23,57	Min. 15
N total	%b/b	0,05	0,02	0,02	
P_2O_5	%b/b	0,27	0,17	0,15	
C/N Ratio	-	393,20	1.208,00	1.178,50	Max. 25

Table 5. Test of biochar fertilizer with previous studies

C-organic, %	C/N	pH	P_2O_5 , %	Total N, %	Reference
17,94	19,71	8,13	-	0,91	[22]
55,66	-	5,19	-	-	[19]
62,5	-	9	-	-	[23]
24,16	1.208,00	8,92	0,17	0,02	This research is 24.73 min-400ºC

Developments in agriculture have identified biochar as a viable option due to its potential to improve soil. Biochar can significantly increase soil pH, organic carbon, and nutrients in the soil [24].

Chemical characteristics, surface area of biochar

Biochar surface area testing was carried out using the Brunauer-Emmet-Teller (BET) test method. Testing with a surface area analyzer brand St 2 on NOVA touch 4LX in the integrated laboratory of the Islamic University of Indonesia (UII). Tests were carried out with 2 kinds of samples, namely: samples with fixed temperature treatment of 400ºC with residence time varying from 52.47 min, 24.73 min and 17.23 min and samples with fixed residence time treatment of 24.73 min with pyrolysis temperatures varying from 350ºC, 400ºC and 450ºC. Biochar pore structure parameters are very important to determine the quality of absorption. These parameters consist of surface area, volume and pore size. [25,10,26]. Pore size is grouped into three categories: micropores with pore sizes less than 2.0 nm, mesopores with pore sizes larger than 2.0 nm but smaller than 50 nm and macropores with pores larger than 50 nm [27].

The biochar produced in pyrolysis with a fixed residence time of 24.73 min at 350ºC, 400ºC and 450ºC was analyzed for surface area, pore volume, pore radius and pore classification. The test results are shown in Table 6. At 400°C produced biochar with the highest surface area (0.554 m²/g) and pore volume (0.00364 cc/g), indicating that this temperature is most effective in opening pores and increasing adsorption capacity compared to other temperatures. At 350°C produced biochar with the lowest surface area (0.322 m²/g) and pore volume (0.00219 cc/g), indicating that the pyrolysis process at lower temperatures produces fewer and smaller pores. The 450°C temperature produced biochar with a slightly lower surface area (0.343 m²/g) and pore volume (0.00221 cc/g) than the 400 °C temperature, but still quite good, with a slight increase in pore radius (13.321 nm), indicating the presence of larger pores. All samples fall into the mesoporous category, indicating that all biochars have a pore structure suitable for soil amendment applications [28] and applications as adsorbents [5,10,29,30]. The increase in specific surface area is a function of the decrease in cell pore diameter, which coincides with the loss of tar, hydrogen oil and oxygen [31,32]. Previous research also provides results that pyrolysis temperature affects surface area and pore volume [33,34,21]. The difference in value is influenced by the length of the pyrolysis process and the pyrolysis temperature [35,36].

Tabel 6. Structure of biochar with residence time of 24.73 minutes and varying temperature

Temperature (°C)	BET surface area (m^2 /g)	Pore volume (cc/g)	Pore radius (nm)	Classification
350	0.322	0.00219	13.572	Mesopores
400	0.554	0.00364	13.154	Mesopores
450	0.343	0.00221	13.321	Mesopores

The biochar produced in pyrolysis with a constant temperature of 400°C and residence time of 51.47 min, 24.73 min, and 17.23 min were also analyzed for surface area, pore volume, pore radius, and pore classification. The test results are shown in Table 7. The 51.47 min residence time resulted in the highest surface area (0.574 m²/g), which indicates that longer residence time tends to increase the surface area of biochar. However, thereafter, the surface area decreased at shorter residence times (24.73 min and 17.23 min) due to a reduction in pore opening or the formation of more denser carbon. The highest pore volume was found at a residence time of 24.73 minutes (0.00364 cc/g), indicating that this residence time is most optimal for forming more and larger pore volumes. Pore volume decreased at longer (51.47 min) and shorter (17.23 min) residence times, indicating that they were not optimal in forming larger pores. The pore radius slightly increased at 17.23 min residence time (13.201 nm), indicating that at shorter residence time, larger pores were formed, although the number and pore volume were less. The residence time of 51.47 min resulted in the smallest pore radius (13.078 nm), indicating smaller pore formation despite more volume and surface area. All samples fall into the mesoporous category, indicating that all biochars have a pore structure suitable for soil amendment applications [28] and applications as adsorbent [5,10,29,30]. Previous research by Jamal (2023) also showed that pyrolysis residence time increased the surface [37]. The difference in value is influenced by the raw material, the length of the pyrolysis process and the pyrolysis temperature [35,36].

Table 7. BET test results with a constant temperature 400°C

Resident time H	BET surface area (m^2 /g)	Pore volume (cc/g)	Pore radius (nm)	Classification
52.47 min	0,574	0,00313	13,078	Mesopores
24.73 min	0,554	0,00364	13,154	Mesopores
17.23 min	0,406	0,00268	13,201	Mesopores

Surfacce characterization, biochar composition

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscoy (EDX) mapping tests are performed to look at surface morphology and composition on a micro or nanoscale [7]. Testing was carried out at the integrated laboratory of Gadjah Mada University (UGM). Tests were carried out with 2 kinds of samples, namely: samples with a fixed temperature treatment of 400°C with residence times varying from 52.47 min, 24.73 min and 17.23 min and samples with a fixed residence time treatment of 24.73 min with pyrolysis temperatures varying from 350ºC, 400ºC and 450ºC.

Based on the test results of making biochar from bagasse with temperature variations of 350°C, 400°C, and 450°C and a fixed residence time of 24.73 min, shown in Table 8. The results show that 450°C produces the highest carbon content (83.91%), indicating that higher temperatures support a more intense pyrolysis process, resulting in a higher carbon concentration of biochar. High carbon content is the main characteristic of good biochar. The increase in C content of biochar is due to the loss of other elements due to vaporization, especially hydrogen and oxygen [21]. The oxygen content decreases as the temperature increases, with the lowest oxygen content at 450 °C (13.48%). This indicates that high temperatures are more effective in removing oxygen-containing components, resulting in lower and more stable oxygen-content biochar. Silica content increases at higher temperatures, with the highest silica content found at 400 °C (1.97%) and 450 °C (1.75%). Silica in biochar can improve soil quality and the potential of biochar for application in agriculture. Potassium content also increased with temperature, with the highest potassium content at 450 °C (0.71%). Higher potassium in biochar increases its potential use as a fertilizer or soil improver that is beneficial to plants. Silica particles appear due to dust particles in the biomass [38].

Table 8. Biochar composition test results fixed residence time of 24.73 min and varied temperature.

Element	350ºC Spc 005 Atom %	400ºC Spc 010 Atom %	450ºC Spc 014 Atom %
C	83,12	82,17	83,91
O	15,43	14,89	13,48
Si	0,99	1,97	1,75
K	0,28	0,42	0,71

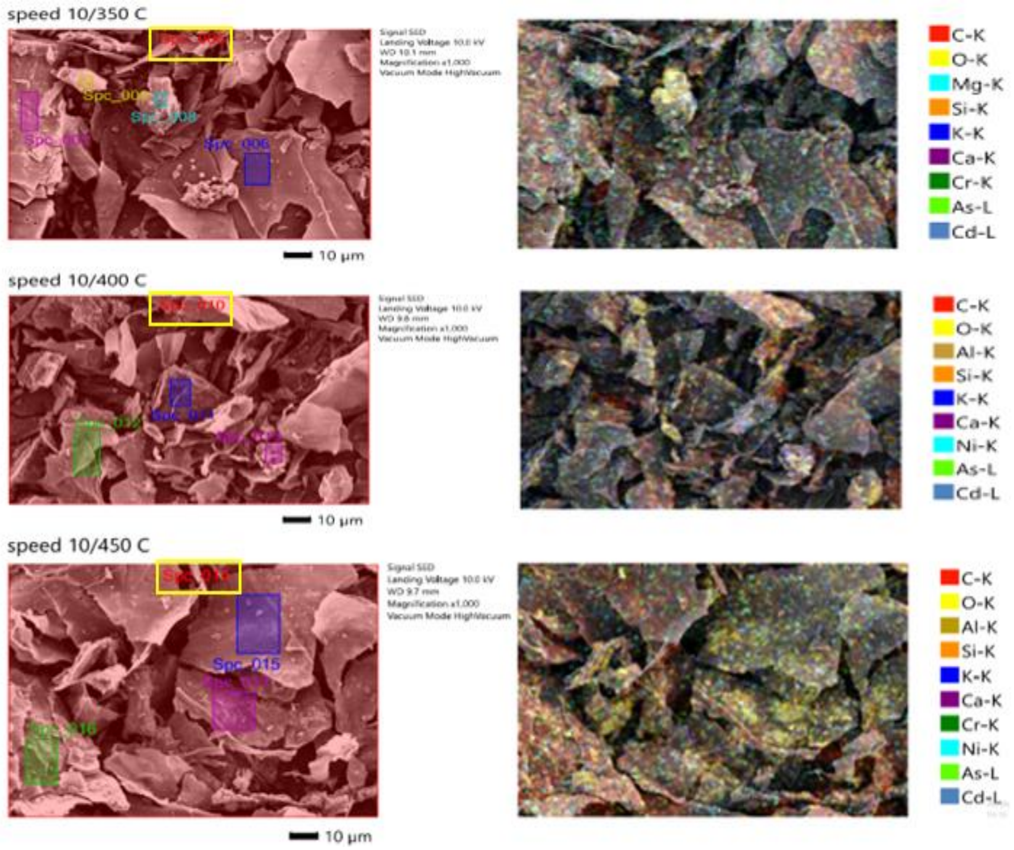

Figure 4. SEM EDX Mapping of biochar at a fixed residence time of 24.73 min and varied temperature.

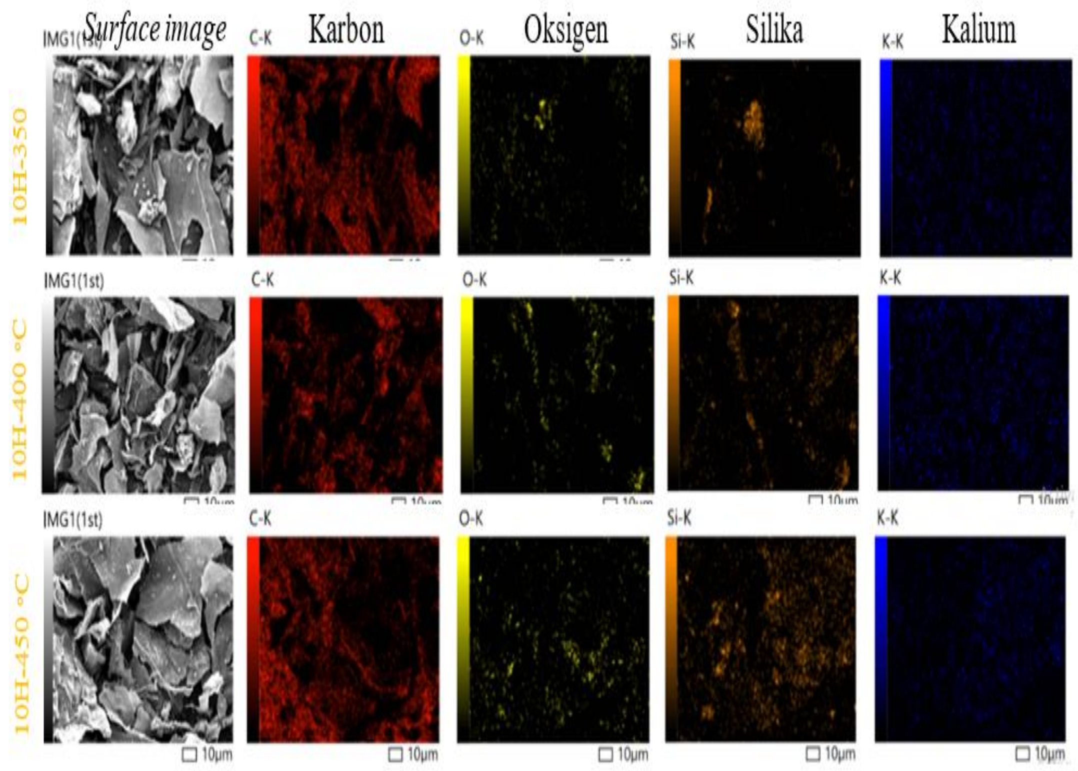

Figure 5. X-ray surface area-SEM elements: surface image, carbon, oxygen, silica, potassium. Biochar at a fixed residence time of 24.73 min and varied temperature.

Based on the test results of making biochar from bagasse with a fixed temperature of 400°C and variations in residence time (52.47 min, 24.73 min, and 17.23 min), shown in Table 9. The results provide information that the highest carbon content is found in the residence time of 52.47 min (86.55%), indicating that longer residence times tend to produce biochar with higher carbon content. This is due to the more complete pyrolysis process at longer residence times, which converts organic matter into carbon. The lowest oxygen content was found at a residence time of 52.47 min (12.15%), indicating that the longer the residence time, the more oxygen is released during pyrolysis. In contrast, a shorter residence time (24.73 min) resulted in a higher oxygen content. The highest silica content was found at a residence time of 24.73 min (1.97%), indicating that a shorter residence time increased the accumulation of silica in the biochar. Longer residence times may cause a slight reduction in silica as longer processes may cause physical changes to the silica. The highest potassium content was found at 24.73 min residence time (0.42%), indicating that potassium accumulated more in biochar at a shorter residence time. A longer residence time (52.47 minutes) may cause more potassium to be released. The highest content is carbon ranging from 82-86%, this result is in line with research by Huang, (2023) [39].

Table 9. Biochar composition test results at a fixed temperature of 400^{o}C and varying residence time.

Element	52.47 min Spc 001 Atom %	24.73 min Spc 010 Atom %	17.23 min Spc 018 Atom %
C	86,55	82,17	85,33
O	12,15	14,89	13,58
Si	0,99	1,97	0,67
K	0,10	0,42	0,23

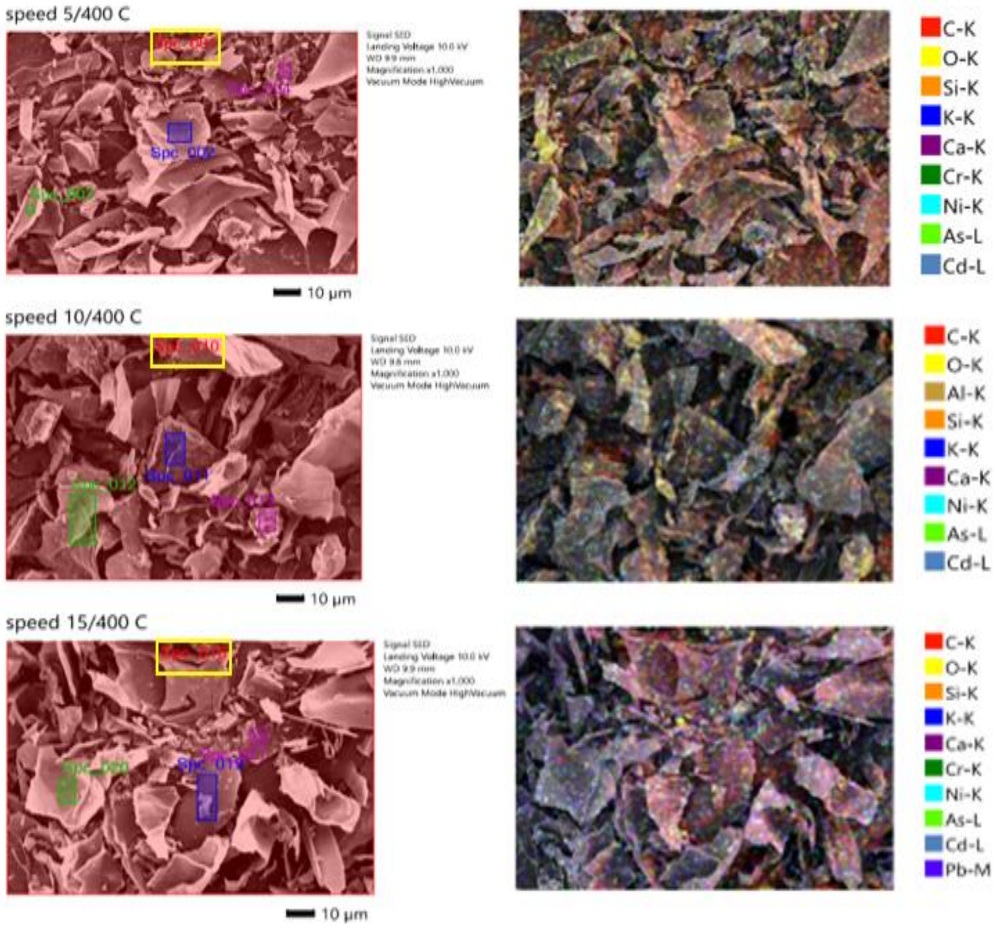

Figure 6. SEM EDX Mapping of biochar at a fixed temperature of 400ºC and varied residence time.

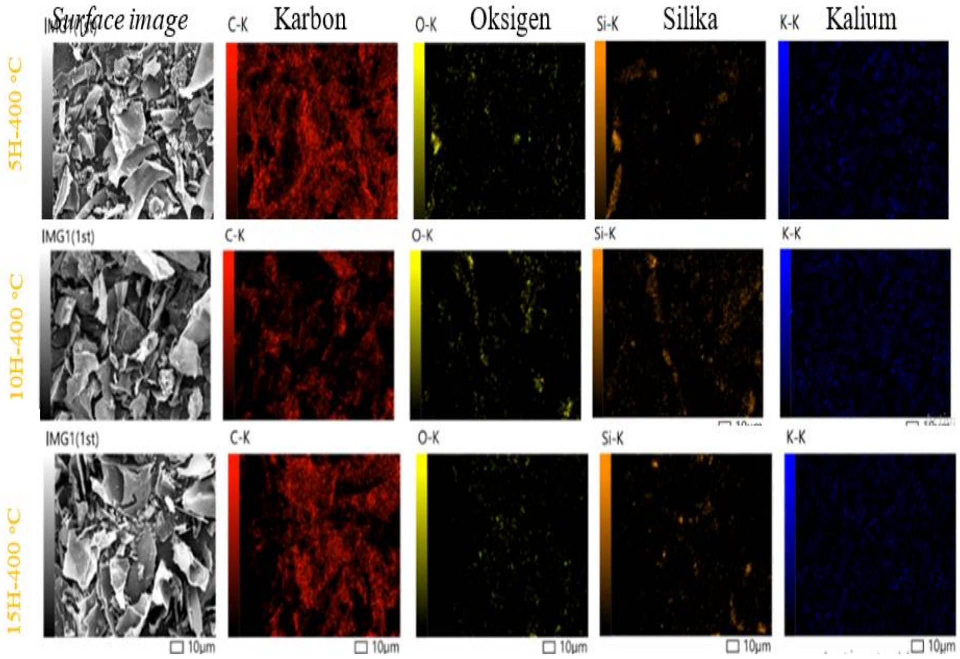

Figure 7. X ray surface area-SEM elements SEM: surface image, carbon, oxygen, silica, potassium. Biochar at a fixed temperature of 400ºC and varied residence time.

4. CONCLUSIONS AND SUGGESTIONS

4.1 Conclusion

a. The results of the lignocellulose component test of bagasse as raw material obtained cellulose at 21.07%, hemicellulose 26.93% and lignin 20.14%.
b. Characterization of physical properties of pyrolysis residence time and temperature affects pH, c-organic, total N, P_2O_5 and C/N ratio. The pyrolysis residence time of 24.73 min at 400ºC gave biochar with pH 8.92, c-organic 24.15%, total N 0.2%, P_2O_5 0.17% and high C/N ratio (1,208.00). This biochar is good for application as a soil improver, especially in increasing carbon storage capacity and improving soil pH. The high C/N ratio and low N content require alloying with other sources to increase N and phosphorus and decrease the C/N ratio.
c. Characterization of physical properties of residence time and pyrolysis temperature affects the Brunauer-Emmet-Teller (BET) test results, including surface area, pore volume and pore radius. Pyrolysis residence time of 24.73 min at 400ºC gave the best results in terms of increased surface area (0.554 m²/g), pore volume (0.00364 cc/g) and decreased pore radius (13.154 nm), making it the optimal temperature to produce biochar with higher adsorption capacity.
d. Characterization of the composition of biochar surface elements, residence time and pyrolysis temperature affect the composition of biochar surface elements. SEM EDX mapping results showed that biochar has 4 main compositions: carbon, oxygen, silica and potassium. The pyrolysis residence time of 24.73 minutes with a temperature of 400ºC gives results with a relatively high composition of carbon 62.17%, oxygen 14.89%, silica 1.97%, potassium 0.42%, and makes it more effective for soil conditioner applications.

4.2 Suggestion

a. Research on the same issue can be conducted with varying temperature conditions and residence times, incorporating heat rate test variables.
b. Industrial-scale pyrolysis results can be developed with bio-oil and syngas yield research.

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