Preparation and Characteristics of Effective Biochar Derived from Sugarcane Bagasse as Adsorbent

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Abstract- Biochar is an efficient and economical adsorbent. Raw materials for biochar from biomass are abundantly available. This study aims to modify biochar from sugarcane bagasse using biochar HCl from the pyrolysis process with time variations from 300°C to 600°C. HCl mixture has a concentration of 3M. The research results show that pyrolysis operating temperature can affect biochar yield, structure, and energy distribution. The highest product of biochar was obtained at 300°C at 34.34%. The morphological structure of biochar at 300°C has holes with reasonably small sizes. However, at 400°C, pore size biochar increases from 9.90 nm to 18.42 nm at 600°C. The carbon content in biochar increases with the pyrolysis temperature. The highest carbon content was obtained at 89.91%. The structural properties of biochar have been discussed in this study in the form of pore volume, BET surface area, and pore radius. This research has shown that pyrolysis temperature can affect the condition of biochar.

Keywords Adsorbent, Bagasse, biochar, Pyrolysis, Temperature Effect.

1. Introduction

The Increasing population and industry grew in line with the development trends in the world. However, some people ignore that the increase in population and industry can be a factor in environmental pollution, such as industrial water, wastewater from the batik industry, cooking wastewater, used washing water, etc. In addition, the country's economic development has a relationship with energy consumption. Developed countries tend to reduce the use of fossil energy while developing countries still rely on fossil energy [1].

Environmental pollutants can threaten society and disrupt economic growth [2]. Currently, adsorption technology, membrane technology, oxidation technology, and biodegradation technology have been developed to protect the environment from water pollution [3]. Adsorption technology has advantages such as having a simple and environmentally friendly design, being more economical, not requiring high energy consumption and having abundant raw materials.

Currently, research on developing biochar from biomass through the pyrolysis process is of concern. Biochar produced from the pyrolysis process can absorb organic pollutants and commercial wastewater. Biomass raw materials are abundant and easy to find [4-5]. Pyrolysis is the thermochemical decomposition of organic materials through combustion without oxygen which can produce three products: bio-oil, biochar, and gas [6-7]. Biochar from the pyrolysis process has good adsorption properties [8,9] and a high surface area [10,11]. The modification and use of biochar in reducing environmental pollution have been described by [12] in Figure 1.

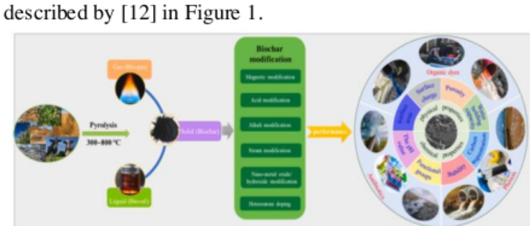


Fig 1. Schematic diagram preparation and modification of biochar [10]

Abundant types of biomass attract the attention of researchers. Abundant types of biomass attract the attention of researchers. A study investigated biochar from corn stalks [13], biochar from rice husk [14-16], biochar from coconut shell [17,18], biochar from algae [19], biochar from tea residues for supercapacitor electrode (20-22], biochar from sugarcane [23-24], etc. The carbon content in biochar is affected by operating temperature, which can cause thermal decomposition. Pyrolysis conditions also determine biochar properties such as porosity, surface area, adsorption quality, pore volume, and pore radius. To increase the adsorption ability of biochar, researchers have made various modifications using other materials, such as modification of biochar using KOH [25], Chitosan [26], $FeCl_3$ compared with HCl, NaOH [27], NH_3 [28], and HNO_3.

Therefore, this study focuses on activating biochar from sugarcane bagasse as an adsorbent. The activation used was 3M HCl. HCl can change biochar's structure. This research can provide an understanding of the effect of pyrolysis temperature on the form of biochar and the effect of activation using HCl.

2. Material and Methods

2.1. Preparation and Characterization of Sugarcane Bagasse

The raw materials in this study were obtained from PT Madukismo Yogyakarta, Indonesia. This research has three steps: production of biochar from the pyrolysis process, activation of biochar using HCl, and Characteristic of biochar. In this study, biochar was produced through a pyrolysis process with varying temperatures from 300°C to 600°C. The ultimate and proximate analysis on sugarcane bagasse can be seen in Table 1. The Ultimate analysis included the compound of Carbon (C), Hydrogen (H), Oxygen (O), Nitrogen (N), and Sulphur (S). For oxygen (O), it was calculated by the total compounds (100%) subtracted from the results of C, H, N, and S. This calculation is based on research from [29]. While, the proximate included the moisture, fixed carbon, volatile matter, ash, cellulose, hemicellulose, and lignin.

Table 1. Proximate and elemental analysis of sugarcane bagasse

Components	This study (wt.%)
Ultimate	
C	42.50
H	6.17
O*	51.00
N	0.23
S	0.10
Proximate	
Moisture	3.83
Fixed Carbon	21.95
Volatile matter	71.60
Ash	2.60
Cellulose	45.82
hemicellulose	20.20
Lignin	21.32

*O was calculated by difference

Based on Table 1, the sugarcane bagasse in this study contained cellulose of 45.82, hemicellulose of 20.20, and lignin of 21.32, three essential components of biomass for biomass heat generation. According to [30], biomass composition (cellulose and hemicellulose) affects the gas profile at CO and CO2 concentrations. Meanwhile, lignin acts as the concentration of CH4. Lignin is also a factor in thermochemical decomposition [31]. The results of the analysis of raw materials in research are relevant to previous research conducted by [32]. The type of biomass influences the content of cellulose, hemicellulose, and lignin can be seen in Table 2

Table 2. The comparison of cellulose, hemicellulose, and lignin

Biomass	Cellulose (%)	Hemi cellulose (%)	lignin (%)	References
Sugarcane leaves	44	22	17	[1]
Wood waste	44.7	18.0	33	[2]
Bagasse	45.82	20.20	21.32	This study

Water content, volatile matter, ash, and fixed carbon are biomass parameters that need to be considered. This parameter is applied to measure the quality of bagasse sugarcane. The proximate analysis was involved in this research using ASTM standards (E1535-93, D3172, E872-82, and NBR8112) and was carried out at the Research and Development Center for Tekmira Bandung. Measurement of moisture content seen from calculated by weighing the mass lost during the oven at 105°C for 6 hours. A range of 950°C determined it for 7 minutes for volatile content. Meanwhile, the ash content was obtained from the oven at 550°C for 3 hours. CHN analysis (Perkin Elmer 2400) based on ASTM D5291-96 was applied for Ultimate analysis. The calorific value (HHV) can be calculated using the Dulong and Petit as shown in Equation 1. Where C is carbon, O is oxygen, and H is hydrogen.

$$HHV = 33950\,C + 144200\left(C_2 - \frac{O_2}{8}\right) + 9400 \quad (1)$$

2.2. Biochar Production from Pyrolysis Process

Biochar is obtained from the pyrolysis process using a fixed bed reactor equipped with water cooling. The fixed reactor configuration consists of vertical cylinders with an inner diameter of 4.0 cm, an outer diameter of 4.4 cm, and a height of 60.0 cm. Three condenser units are applied with different size designs. The first condenser has a smaller size, namely a diameter of 0.6 cm and a length of 15.0 cm. The second and third condensers have the same size, namely a height of 40.0 cm. The temperature control used is a K-type thermocouple combined with a PID controller. The schematic design of pyrolysis can be seen in Figure 2.

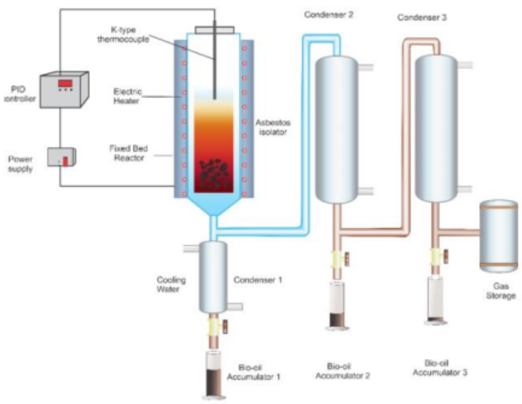


Fig 2. Schematic design of pyrolysis

This research is a development of a previous study [30] in which pyrolysis is operated at temperatures (300°C, 400°C, 500°C, 550°C, and 600°C) and a heating reaction of 10-12°C/min. The initial steps taken were to weigh about 15 grams of sugarcane bagasse as raw material, which was put into the reactor. The combustion in the reactor was carried out for 60 minutes. After the pyrolysis process is complete, the three pyrolysis products undergo a cooling process to produce bio-oil, biochar, and gas. In this study, biochar was applied as an absorbent. According to [12], Biochar produced during the pyrolysis of biomass has the advantage of being an efficient and inexpensive absorbent for dye removal, antibiotics, soil remediation, and removal of organic and inorganic contaminants from wastewater. That opinion was corroborated by research from [33] which biochar can be used for ammonia removal.

2.3. Activated Biochar

Activated biochar is used to improve the quality of biochar as an adsorbent with a low specific surface area, high C/O ratio, richer pore structure, and strong adsorption ability. According to [34], biochar is a solid material containing black carbon produced from low to high-temperature thermal cracking of waste materials. Biochar is derived from the thermochemical conversion of waste materials that can absorb well [35]. In this research, biochar was activated using 3M HCl. Biochar was collected and immersed in a 3M HCl solution. Then, the mixture of biochar and HCl was covered using Aluminum foil and ultrasonicated at 60°C for 1 hour. The final stage is the clarification process using distilled water until pH is neutral and dried in an oven at 120°C for 2 hours. According to previous research [36], activation using HCl can change the surface structure of biochar.

2.4. Biochar Analysis

In this study, the surface morphology of biochar was characterized using a Scanning Electron Microscope (SEM) during the biochar preparation process. Biochar surface area and pore volume were calculated using Brunauer-Emmett-Teller (BET). The Barret–Joyner–Halenda (BJH) method calculates pore distribution and average pore width. Energy mapping on biochar was calculated using energy dispersive spectroscopy (EDS).

3. Result and Discussion

3.1. Biochar Yield

Biochar produced from the pyrolysis process is affected by the operating temperature. Figure 3 shows biochar production at a temperature variation of 300°C to 600°C. The biochar produced at 300°C has the highest percentage, 34.34%. However, biochar production decreased as the pyrolysis operating temperature increased. At a temperature of 600°C, biochar production decreased by around 23.50%. However, biochar production is inversely proportional to operating temperature. According to [37,38], High temperatures in pyrolysis cause organic matter in biomass to decompose at high temperatures. Another effect occurs in the potential for high carbon absorption at high temperatures [39]. In addition, the increased production of biochar at low temperatures indicates that the material is only partially pyrolyzed. The temperature in pyrolysis causes the material to experience primary decomposition and secondary decomposition in the charcoal residue [40].

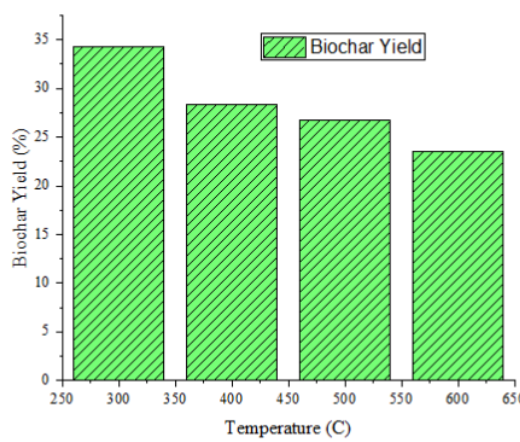
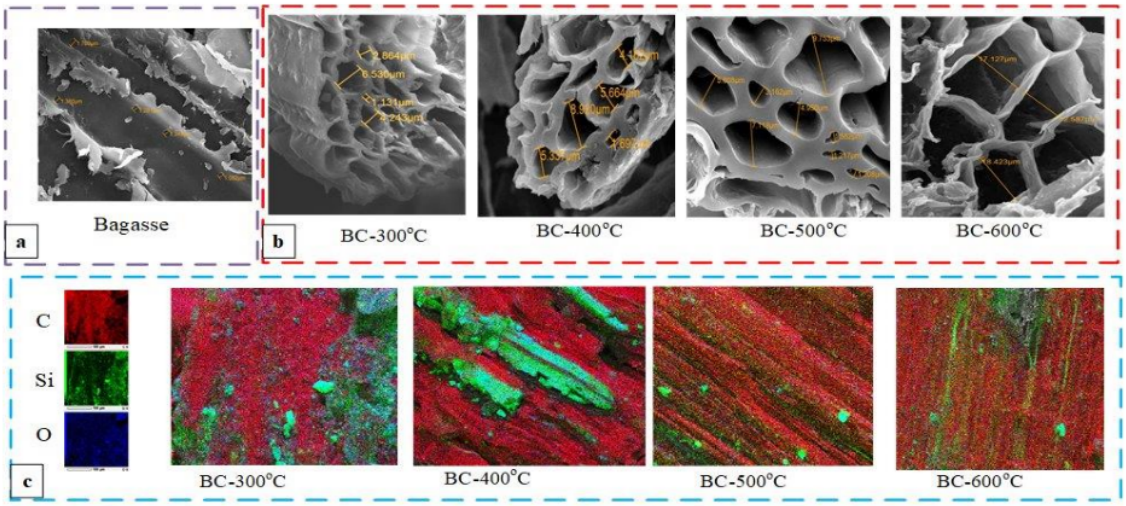


Fig 3. Biochar yield of pyrolysis

3.2. Characterization of Biochar

This stage aims to show the characteristics of biochar after going through the pyrolysis process. Fig. 4a shows this study's visualization of bagasse as a raw material. The bagasse component has relatively tiny pores and tends to be smooth. After the pyrolysis process, bagasse produces biochar with different structures and pores. The biochar structure changes as the temperature increases. Figure 4b. showed the morphology of biochar (BC) at various pyrolysis temperatures. At 300°C, the structure of biochar has holes or pores with a reasonably small size and relatively the same pore area. The morphology of biochar at 400°C showed a significant change in pore size and somewhat different dimensions. The largest hole size at 400°C is 9.90 nm.



Elements	Atom (%)			
	300°C	400°C	500°C	600°C
C	70.53	62.36	89.91	66.96
Si	1.97	3.00	0.56	1.30
O	25.49	18.09	9.03	10.47

Fig 4. Characteristics of biochar: a) morphology of bagasse, b) morphology of biochar, c) Mapping energy of biochar, dan d) element distribution.

As the pyrolysis temperature increases, the morphological structure of biochar is increasingly other. At 500°C and 600°C, the morphology of biochar showed large pores; the pores at 500°C reach about 9.75 nm. Meanwhile, the pores at 600°C reached 18.42 nm. These results are in line with research [41,42] that the pore size of biochar is affected by the increase in pyrolysis temperature. Element distribution in biochar can be seen in Fig. 4c. Through energy dispersive spectroscopy (EDS) mapping, this image classifies the distribution of C, Si, and O elements in biochar. The element content in the biochar shows the success of element transfer to the surface of the biochar. The percentage of C, Si, and O atoms can be seen in Figure 4d. The distribution of C atoms verifies the finding that the carbon content increases. Meanwhile, the atomic content of Si and O decreased. According to [43], The presence of Si particles is caused by dust particles in the biomass. The content of C atoms in biochar is relatively high, ranging from 60-80% [44].

3.3. Surface Area Analysis of Biochar

The pore structure parameter in biochar is essential in determining adsorption quality and consists of surface area, volume, and size. This section describes in detail the effect of temperature on the surface area, pore volume, and pore size. The BET method was applied to determine the pore structure. Table 3, Shows the results of the pore structure in biochar. The BET surface area and pore volume of biochar increased significantly as the pyrolysis temperature increased. The outer surface at 500°C and 600°C showed the highest increase in BET surface area of 193.802 m2/g and 180.196 m2/g. Pore volume also increased from 0.002 cc/g to 0.041 cc/g. However, the pore diameter decreased when the pyrolysis temperature increased. According to [43], pyrolysis temperature can create more gaps and pores on the biochar surface and greater porosity. The previous research from [46] and [47] also stated the effect of temperature on the surface area, pore volume, and micropores. The difference in value may be influenced by the source of the material, pyrolysis duration, and pyrolysis temperature [48, 49].

Table 3. The results of pore structure in biochar

Temperature (°C)	BET surface area (m^2/g)	Pore volume (cc/g)	Pore diameter (nm)
300	0.614	0.002	13.312
400	0.814	0.004	13.215
500	193.802	0.041	1.690
600	180.196	0.025	1.217

3.4. N_2 Adsorption-Desorption Isotherm

The process of material analysis can be seen through a comparison graph of the relative pressure P/Po to the adsorbent volume. The mesoporous material properties can be seen from the historicist loop results. Figure 5 shows the analysis of N2 adsorption-desorption on biochar against temperature variations.

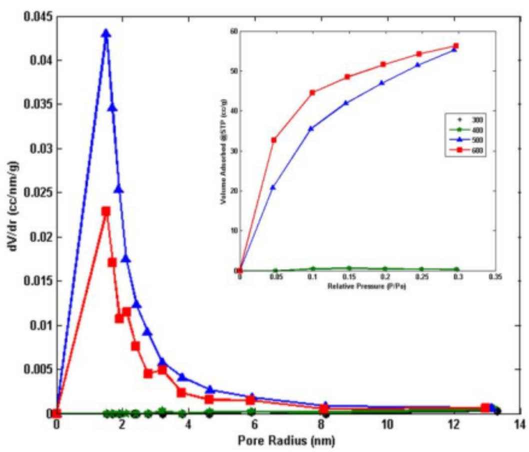


Fig 5. The correlation of pore radius, surface area, and relative pressure

This study's amount of N2 adsorption indicates that under relative pressure (P/Po) below 0.1 is a mesopore structure. The comparison between the pore radius and the surface area shows that the pore volume (DV/dr) decreases as the pore radius increases. The pore volume decreases as the pore radius increases. This condition illustrates that the biochar surface has increased electrochemical charge performance and increased porosity as a place for electrolyte ion transport. These results are relevant to research conducted by [43] that the surface area and pore volume of biochar has a significant relationship.

4. Conclusion

Biochar in this study was produced from the pyrolysis process of sugarcane bagasse through temperature variations of 300°C, 400°C, 500°C, and 600°C. Biochar production is affected by pyrolysis temperature. As the pyrolysis temperature increased, biochar yield decreased. The highest of biochar is 34.34% of 300°C. Pyrolysis temperature also affects the biochar structure. At 300°C, the form of biochar has holes or pores with a reasonably small size and relatively the same pore area. However, the biochar structure changed with the increase in temperature. Through EDS mapping, the distribution of elements in biochar can be known. In this study, the element content in the biochar shows the success of the element transfer to the surface of the biochar. The results of biochar surface analysis showed that the surface area of BET and pore volume of biochar increased significantly as the pyrolysis temperature increased. Comparing the pore radius and the surface area shows that the pore volume has decreased. Further research will focus on the adsorption mechanism of biochar from bagasse in the form of kinetic models, isotherms, and thermodynamic models.

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