Production and Characterization of Biochar from Gaharu Residue via Pyrolysis

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Abstract— A lab-scale high temperature furnace was utilized to study the effect of residence time (30 min - 150 min) on the yield, chemical and physical properties of biochars issued from the pyrolysis process at 800 °C. Afterward, an adsorption capacity analysis was carried out to investigate the amount of Methylene Blue dye adsorbed by the chars. Residence time of 120 minutes was found to be optimal in inducing biochar with improved properties, such as substantial fixed carbon content, enriched functional groups, greater specific surface area and high adsorption capacity. Although the residence time of 150 minutes produced biochar with higher fixed carbon, specific surface area and adsorption capacity, the differences were insignificant. In addition, it was noticed by SEM image that the porous structure of biochar at 150 minutes was blocked by ash content. The pore diameter was also bigger. As a result, biochar at 120 minutes with specific surface area of 152.75 $m^2\ g^{\text{-1}}$ and Methylene Blue adsorption capacity of 68.5 mg g⁻¹ has promising potential towards wide range of applications.

Keywords— Pyrolysis, Biochar, Gaharu residue, proximate analysis, ultimate analysis, Methylene Blue, Adsorption capacity.

I. INTRODUCTION

Environmental issues like pollutions, global warming, natural resources depletion and waste disposal have become a controversial topic that have been discussed among public officials and independent researches for both local and global levels. Environmental degradation is one of the largest threats to every country over the world that should be conserved due to its adverse impacts on human livelihood, international relation, biodiversity and economic [1]. Someone can't deny the fact that economic and technological changes, law and political globalization and social mobilization contribute to environmental issues. However, the impacts from those activities could be reduced by implementing or adapting new sustainable solutions.

Biochar, a black solid carbon material has a great solution for that scenario. Hence, interest in biochar for environmental application has become significantly increase day by day due to its unique characteristics to adsorb organic and inorganic materials [2]. 'Black is the New Green' is a relevant analogy to describe that biochar complies the environmental-friendly approaches from the beginning of the feedstock preparation to final use in various fields. However, there are certain criteria for the biochar to be branded as a green material which are type of feedstock used, type of methods implemented, and how its efficiency towards environmental purposes [3].

On the other hand, biochar also attracts researchers' attention due to its intriguing potential advantages in wide range of disciplines; engineering, biotechnology, agronomy and pedology. Therefore, many researches that related to the biochar have done whether for academic purposes, research and development (R&D) phases, or domestic and industrial applications. Using the varieties of feedstocks or methods in term of creating harmonic studies towards different outcomes can develop a reliable system for the biochar itself. For example, a comparison can be made between two studies of different feedstock used in term of economic, efficiency and complexity.

In recent years, researchers put their effort to produce biochar from different kinds of feedstock that contain high carbon content such as coconut husks [4], empty fruit bunches [5], goat manure [6] and sewage sludge [7]. They study the effectiveness of varying parameters like method, operating condition and reactor towards resultant biochar. So, there are many feedstocks that have been used in order to produce biochar. However, Gaharu, whether its roundwood, bark or residue used as a feedstock to produce biochar has not yet been reported. In fact, Malaysia which one of the countries that planted huge amount of Gaharu species [8] still lack of the related studies. Many studies on Gaharu species are focusing on its oil or resin rather than its residue. This is because the oil or resin is relatively high market value [9]. Therefore, this research is accomplished based on (1) need for environmental problems solution, (2) waste generation versus utilization and (3) lack of studies on potential Gaharu species. The objectives of this research are clear; to study the effect of pyrolysis operating condition (residence time) towards properties of biochar and to study the adsorption kinetics of biochar on the Methylene Blue.

Biochar is usually produced from biomass as a feedstock and slow pyrolysis as a production method in different types of reactor, furnace or any equipment that provides heating mechanism. Researches are basically conducted based on case-by-case approach which adjustment is made by optimizing the biomass, method and reactor types. Hence, this research is conducted to add a new, significant input or finding in the biochar's field of study as well as the consequences from the risen issues. Gaharu residue is used as a biomass feedstock, meanwhile high temperature furnace is used as a heating medium to produce the best quality of biochar. The furnace is made up of silicon carbide type brick layer and covered with inner coil that surrounds within bricks. By using slow pyrolysis method, some conditions or parameters are being manipulated to optimize the production. Besides, the resulted biochar is analyzed and tested to determine its quality and efficiency.

II. METHODOLOGY

A. Biomass Characterization

Raw Gaharu biomass was collected from a small wood oil industry in Kuala Nerang, Kedah. It was collected as small size solid state chips and in wet condition. The biomass was then dried in a Memmert Universal Oven UF750 (Memmert, Germany) in order to remove almost 95 % moisture. The oven was set at 80 °C for about 7 days of drying. The dried biomass was then collected and packed in sealed plastics to prevent air from entering.

A series of analysis was done for raw biomass in order to characterize the its physicochemical characteristics. Proximate analysis was performed using a Mettler Toledo TGA/SDTA 851e Analyzer (Mettler Toledo, Switzerland). 20 mg of sample was performed at heating of 20 °C/min using 20 mL/min nitrogen gas from room temperature to 1100 °C. Ultimate analysis was done in a Thermo Fisher Scientific Flash EA 1112 Series CHNS-O Analyzer (Thermo Fisher Scientific, USA). Functional groups of the sample were analyzed using Perkin Elmer Spectrum ONE FT-IR Spectrometer (Perkin Elmer, USA). The spectra were recorded at infrared region of 4000 to 500 cm⁻¹. Another important analysis that has been done is calorific value. It was carried out in C5000 IKA[®]-Werke Bomb Calorimeter (IKA[®]-Werke GmBH & Co, Germany).

B. Pyrolysis Experiment

The pyrolysis experiments were carried out using laboratory scale Carbolite RHF 16-8 Chamber Furnace (Carbolite, UK). The experiments were carried out under inert condition where the continuous nitrogen gas at 40 cm³/min was supplied to the furnace. The furnace temperature was increased steadily at 15 °C/min up to 800 °C. The sample was divided into five portions and packed in the aluminum foil which every portion was weighted as 100 g. Each sample was loaded in a furnace with different residence times (30, 60, 90, 120 and 150 min). In order to control environment, the released smoke was collected by ventilation system. The samples were then taken out and weighted. The yield of biochar was determined by using Eq 1 as described elsewhere [10]:

Yield (%) = (Weight of biochar)/(Weight of biomass) \times 100 (1)

C. Biochar Characterization

There were four analyses of resulted biochar that have been accomplished. It includes proximate, functional group, pore structure and pore size analyses. Proximate and functional groups analyses were consequences from the previous analyses on biomass. So, these analyses were used to compared the effects of pyrolysis on biomass before and after. Additional of two analyses; pore structure and BET surface area were carried out in Hitachi Tabletop Microscope TM3030Plus (Hitachi High Technologies, USA) and 3Flex Surface Characterization Analyzer (Micromeritics, USA), respectively. The BET surface area was measured from the amount of nitrogen adsorbed by biochar at the 77 K under atmospheric pressure.

D. Methylene Blue Adsorption Capacity

This analysis was carried out followed the procedure described elsewhere [11]. Standard curve of methylene blue solution was first prepared prior to adsorption analysis. The standard curve is ranging from concentration of 0 to 100 ppm. So, the stock of methylene blue was prepared at 100 ppm. A series of dilution was done from 100 ppm to 10 ppm, 1 ppm, 0.1 ppm and 0 ppm (pure distilled water). After that, the adsorption analysis was carried out by adding 0.05 g of biochar into 100 ml of 40 ppm solution from stock. It was then stirred for about 3 hours. The absorbance from the resulted solution was tested using DR 2800 UV-Vis Spectrophotometer (Hach, USA) at 665 nm. The absorbance was then compared with the standard curve to get the final concentration of the solution. Hence, the adsorption capacity at equilibrium was obtained using Eq 2:

$$q_e = ((C_o - C_e)V)/w \tag{2}$$

where,

 $q_e = MB$ uptake at equilibrium (mg/g)

 C_o = liquid phase concentration of MB at initial (mg/L)

 C_e = liquid phase concentration of MB at equilibrium (mg/L)

V = volume of the solution (L) w = mass of biochar used (g)

III. RESULTS AND DISCUSSION

A. Biomass Characterization

The process began with drying the biomass at 80 °C in order to achieve not more than 5 % of moisture content. It is important to remove some water in biomass because it will reduce the drying time during pyrolysis. Furthermore, there are possible interferences of water on the analysis that need to be neglected [12]. For this case, it took about 2 days to reduce the moisture content at below than 5 %. An additional of 5 days drying process was implemented to stabilize the reading as well as to ensure no further moisture reduction can occur. So, at last day of drying, it showed an ultimate reduction of moisture. Figure 1 shows the graph of moisture content for 6 days of drying starting from second day.



Figure 1: Moisture Content of Biomass from day 2 to day 7

The properties of biomass are given in Table 1. It includes proximate, ultimate and calorific value analysis. From the data, it shows that the biomass contains high amount of volatile matter. The amount of fixed carbon was found as 16.84 %. It is relative low than other biomass; straw (29.86 %), lignosulfonate (25.18 %) and cocopeat (25.3 %) [13][14]. Ultimate analysis tells that the C and O composition were within the range of expected. The N composition was very insignificant which only 1 % and there was almost nothing S in the biomass. Thus, it does not pollute the environment [15]. The amount of HHV in the biomass was found to be quite high which is 18.178 MJ/kg. From Figure 3, it shows that the biomass contains O-H group ($3000 - 3600 \text{ cm}^{-1}$), C=O and C=C group ($1600 - 1750 \text{ cm}^{-1}$) and C-O (1050 cm^{-1}).

Table 1: The properties of bion	mass
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Proximate Analysis (%)	Moisture	8.48
	Volatile Matter	62.68
	Fixed Carbon*	16.84
	Ash	8.48
Ultimate Analysis (%)	С	41.34
	Н	6.30
	0*	48.70
	Ν	1.06
	H/C	1.83
	O/C	0.88
Empirical Formula		CH1.83N0.022O0.88
High Heating Value (MJ/k	g)	18.178

High Heating Value (MJ/kg) *by difference



Figure 2: FTIR spectrum of biomass

B. Yields

Figure 3 shows a bar chart of biochar yield with respect to residence time. The effect of residence time on yield is shown in the figure, where yield decreased as the residence time increased. For instance, biochar yield decreased about 15 % from 30 min to 120 min. However, the yield of biochar at 150 min is about 29 % which 1 % higher than the biochar at 120 min. According to Onay et al., [16] the yield of biochar is highly dependent on the decomposition of biomass and release of volatile matter with respect to temperature and residence time. Onay et al. also mentioned that the decrease of biochar yield is related to biomass primary decomposition and solid residue secondary reaction. Increasing residence time will add more time for biomass to decompose further and release more volatile matter [17]. However, according to Park et al. [18], increasing residence time helps the re-polymerization of the solid residue to occur hence results in higher yield. Triphati et al. [19] concluded that at high pyrolysis temperature, biochar yield increased if the residence time increased. Thus, the increasing trend of yield at 150 min shows the possibility of re-polymerization of solid residue to occur. Similar findings have been observed by Sun et al. [20], where at high temperature (600 °C), there were no significant decreases of wheat straw biochar from residence time of 0.5 to 8 h. However, the yield slightly increased at residence time of 24 h.



Figure 3: Comparison of Biochar Yield at Different Residence Times

С. Proximate Analysis

The different amounts of volatile matter were highly correlated with the biochar yield. The proximate analysis results as shown in Table 2 describe that volatiles (hemicellulose, cellulose and lignin) are negatively correlated with increasing residence time. As mentioned before, the reduction mass of solid residue biochar from its original biomass is because of the volatile matter released. Chen et al. [21] observed the decline trend of volatile contents in the pine nut shells biochar from 300 °C to 700 °C was due to intense reactions of hemicellulose, cellulose and lignin. Hemicellulose, cellulose and lignin are usually decomposed at 200 - 300 °C, 250 -350 °C and 200 - 500 °C, respectively [22]. Chen et al. concluded that at temperature 300 °C, the lignin structure slightly decomposed because the pyrolysis temperature might not achieved its decomposition temperature. For this case, which at 800 °C, the decomposition of volatiles was solely dependent on residence time. At this temperature, hemicellulose, cellulose and lignin were all decomposed but their distributions differ on how long the decomposition holding time. The previous study also demonstrated that the volatiles (pine sawdust biochar produced at 600 °C) reduced from 42.88 % to 19.33 % while increasing residence time from 3 min to 7 min, respectively [23].

Fixed carbon of the resulted biochar also having a notable effect on residence time. In contrast to volatile matter, fixed carbon shows a constant relation with the residence time. Fixed carbon content of biochar produced at highest residence time; 150 min increased about 15 % from the biochar at lowest; 30 min. This increase was actually due to the increasing amounts of released volatiles. Jouhara et al. [24] confirmed that the amount of fixed carbon increases at a longer residence time. There are many studies that reported the increase in

fixed carbon as residence time increased. Yuan et al. [25] reported a 6 % increase of biochar fixed carbon when medicinal herb residue pyrolysis residence time increased from 10 min to 180 min (at 500 °C), while Zhao et al. [26] also reported similar findings. According to Ronsse et al. [27], it is favorable to have biochar with high amount of fixed carbon and low volatile matter because it is stable and resistance to further degradation (aerobic or anaerobic). Brewer et al [28] suggested that a longer residence time forms more carbon clusters and the biochar becomes more graphitic and stable. Apart from fixed carbon, another component; ash content experienced slightly reduction. The loss of the ash content might be the volatilization of ash species like phosphorus and magnesium during pyrolysis [29][30][31].

Table 2: Proximate	Analysis Data fo	r Biochar	Samples at	Different
Residence Times				

		Proximate Analysis*		
	Fixed Carbon	Volatile Matter	Ash	
Raw	18.40	68.49	13.11	
30 min	31.36	17.41	51.23	
60 min	36.27	13.98	49.75	
90 min	36.98	13.30	49.72	
120 min	38.84	11.59	49.57	
150 min	45.03	6.61	48.36	
*Moisture free ba	eie			

Moisture-free basis

D. Functional Group Analysis



Figure 4: FTIR Spectra of Biochar at Different Residence Times

The FTIR analysis demonstrated that the functional groups of biochars are quite similar to each other as shown in Figure 4. The spectra indicate that the biochars are predominantly containing organic compounds; aliphatic and aromatic groups. A broad and strong OH stretching bands was noticed between 3000 and 3600 cm-¹ for every biochars. The biochars were suspected to have phenols, alcohols and carboxylic acids. The nature of biomass also contains this group but the intensity seems to be higher than other biochars. When the biomass undergoes pyrolysis at 800 °C, the OH group might loss due to bond breaking and shifting. According to Sharma et al. [32], the broad and strong OH in-plane bend declined when temperature increased. There was an alkene (C=C) group and a carbonyl (C=O) group in biochar of 30 min residence time which is between 1600 - 1700 cm⁻¹. However, these aromatic carbon peaks were disappeared when the residence time increased. Lee et al. [33] claimed as the temperature increased, it modified functional groups and thus aromatic increases and aliphatic decreases. Pehlivan et al. [34] also claimed the similar findings. For this case, the aromatic only clear at biochar of 30 min residence time. Another aromatic bands; C-H bending at ~860 cm⁻¹ and ~1470 cm⁻¹ were found to be clear at all of the biochars but nothing at biomass. This bend was claimed to be the same finding with Lee et al. and Pehlivan et al. The intensity of the peak at ~1050 cm⁻¹ gradually reduced as the

residence time increased. At this region, the functional groups are normally primary alcohol with C-O stretching or may be silicate and phosphate vibrations. However, alcohol might be degraded at elevated temperature and the bands should be the phosphate residue from the biomass [35][36][37].

E. BET and SEM Analyses

According to Guerrero et al. [38], micro-, meso- and macroporous structured biochars were resulted from the pyrolysis process of the biomass. This porosity is one of the important physical properties that measures the quality of the biochar. Thus, this BET analysis provided a quantitative measurement of porosity including specific surface area, pore volume (porosity) and pore diameter as shown in Table 3. By considering the biomass has low surface area, the results shown in the table are basically multiple times larger. This great increase can be considered as the change in the chemical structure of the biomass during pyrolysis. Basically, specific surface area was highly dependent on temperature that had been reported by many researchers [39][40][41]. However, Klasson et al. [42] proved that residence time also affects the specific surface area. Klasson demonstrated an increase of specific area from 44 to 423 m^2/g when the almond shell was carried out the 800 °C pyrolysis at residence time from 120 to 240 min, respectively. In this study, the relation between the residence time and specific surface area was also noticeable because the differences were significant. For instance, biochar at 150 min experienced a great formation of pore which increased the surface area up to 21 % from biochar at 30 min. This increase indicates an increase in the extent of solid residue cracking and continuous formation of the pore structure. However, the increase was significant at high residence times; 120 and 150 min which suggest that the formed pores were become larger. It was evidenced that the pore diameter increased as the residence time increased. According to Rehrah et al. [43], another possibility could be due to blocking of pores by ash, tars and other amorphous decomposition products. Study conducted on kapok (Ceiba insignis) fibers by Wang et al. [44] demonstrated that the surface area of biochar at 800 °C and 120 min was 880 m²/g. In addition to structural modification of the biochar at high temperature, the high surface area was also explained by the higher lignin content.

Table 3: Porous Structure Properties of Biochars Obtained at Different Residence Times

	BET Surface Area Analysis		
	BET Surface Area (m ² /g)	Pore Volume (cm ³ /g)	Pore Diameter (nm)
30 min	121.34	0.13	2.56
60 min	145.56	0.21	4.78
90 min	146.73	0.22	5.18
120 min	152.75	0.35	6.56
150 min	153.11	0.41	7.22

Advancement of microscopic technology gives biochar surface morphology an advantage to investigate the pore structure through a SEM image. SEM promotes a clear image of the biochar pore structure effect from the given pyrolysis conditions. Figure 5 shows the comparison of biochar's surface affected from the pyrolysis at different residence times. Notably, pyrolysis process carried out at high temperature exhibited a porous structure formation. This formation is due to the forced opening of biomass structure to allow volatile matter released [21]. As the residence time increased, the formation of the pore also increased. This could be clear as the amount of pore structure biochar at 120 min is compared with 30 min. The increase in residence time also causes the pore size to be larger. Biochar at 150 min showed a larger pore size as compared to biochar at 120 min. The pores are broadened as the residence time permitted high temperature to release volatile matter. Biochar at 150 min also showed the pore structure was not clear at certain region. It showed that the structure was heterogeneous and amorphous. According to Chen et al. [45], blocked region of the pore structure might be caused by melting of ash content at high temperature. Thus, the pore structure might have sufficient time to be blocked.



Figure 5: Scanning Electron Micrographs a) 30 min b) 60 min c) 90 min d) 120 min e) 150 min

F. Methylene Blue Adsorption Capacity Analysis

The resulted biochars were also characterized by adsorption capacity or Methylene Blue number. From the Figure 6, it shows that the adsorption capacity increases after the residence time increases. The BET surface area associated with pore structure and size reflect the biochar adsorption capacity. The increase pore structure will increase the BET surface area and thus, increase the adsorption capacity. The capacity of the biochar at 800 °C can be achieved up to 73 mg/g which means for every gram of biochar, 73 mg of Methylene Blue can be adsorbed. However, Rajeshwar [11] studied that seed stones of Lapsi fruits biochar's adsorption capacity can achieve up to 180 mg/g when the pyrolysis conditions of 400 °C and 2 hours was applied. The adsorption capacity of the biochar is 2.5 greater than the biochar in this study although the temperature is lower. Rajeshwar also found as the temperature increased, the adsorption capacity also increased. This is due to the increase of mesopore structure in the biochar since adsorption capacity represents distribution of mesopore in biochar. The formation of mesopore at high temperature is due to the escaping of volatile matter and thus widening the micropores [46]. Hence, this contradiction was might be due to the formation of high ash concentration. Another contribution is initial Methylene Blue concentration which only 40 ppm.



Figure 6: Effect of Residence Time on Methylene Blue Adsorption Capacity

IV. CONCLUSION

Residence time has been found to be an essential factor in demonstrating the pyrolysis effect towards chemical and physical properties of biochar. This study has shown that increasing residence time enhances the biochar quality by evaluating its chemical characteristics and surface morphology. Residence time at 120 minutes offered an improved quality biochar which is high surface area, substantial functional groups, better pore structure development and great adsorption capacity. However, prolong residence time at high temperature pyrolysis persuades energy wasting. Hence, temperature has to take into account to bring about considerable production cost but keep the biochar at optimum quality.

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