

Surface Modification of Biochar by using Steam Activation

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Abstract—Water pollution has been a major problem not only to the citizens but also the environment. In order to reduce water pollution and also other pollution, biochar had been chosen as an adsorbent. Surface modification by using steam activation had been performed in order to enhance the efficiency of removal of pollutants from water by increasing the surface area. The biochar undergoes pyrolysis process at a temperature 700°C before steam activation process. Steam activation at a temperature of 130°C for 30 minutes was found to be optimal in improving the surface of biochar as it shows greater surface area and high adsorption capacity. Although the temperature of 110°C produced higher fixed carbon and adsorption capacity, the differences were insignificant. In addition, the BET surface area increases at a higher temperature, thus also increases the pore volume of biochar. As a result, biochar at 130°C with specific area of 528.84 m² g⁻¹ and Methylene Blue adsorption capacity of 0.7296 mg g⁻¹ have a potential in providing useful information toward the direction of green technology.

Keywords: *biochar, pyrolysis, modification, steam activation, proximate analysis, ultimate analysis, adsorption capacity*

I. INTRODUCTION

Biochar has been widely used for decades as it is proven to be environmental friendly and is used for variety of purposes such as greenhouse gas reduction and to improve soil quality. The production of biochar depends on the raw materials of biomass used as the composition of biomass may affect the quality produced. Woody biomass is the major important source for charcoal making worldwide due to its varying amount of hemicellulose and other inorganic compounds [8]. However, the surface characteristics of the biochar depends on its raw materials properties and the temperature involve in the production of biochar [13]. Gaharu wood is described to be widely used in religious and cultural activities as the essential oil extract from the gaharu brings various benefit to human's health [21]. Woody feedstock provides a more carbon-rich biochar compared to other feedstock.

Due to its specific characteristics such as high adsorption capacity, ion exchange and high specific surface area, biochar can be used in many fields and applications. One of

the applications that biochar can be applied are adsorbent for water pollutants. Biochar act as a sorbent that helps to remove pollutants from water [19]. Water pollutants remains as one of the biggest challenges that humanity has to face even with the advance technology that existed in the world. As the number of micropores increases, the surface area of the biochar will also increase which in turn helps to provide more surface sites [26].

With the increasing number of surface sites, more pollutants can be adsorbed. These micropores or mesopores are one of the major distribution that contribute to the properties of biochar's large surface area. There are a lot of modifications done to biochar to improve its properties and one of it is by embedding different materials into the biochar structure pre- or post-pyrolysis [26]. The main objective of these modification is to enhance its adsorption efficiency by physically or chemically changing the biochar properties in term of surface area and surface functionality [27]. There are several characteristics that has a great influence on the properties of biochar such as pyrolytic temperature, residence time, feedstock and also thermochemical conversion technology [30].

Water is one of the most vital aspects for one's essential life. 95% of the water source in Malaysia are coming from inland river systems. Rapid growth and urbanization in the country has led to an increasing economic system in Malaysia as well as water pollution in some parts of the states. The demands for clean and fresh water are escalating and these gives the authority immense pressure to help preserve and keep the current water resources and quality in check as well to provide alternative ways on how to improve the water quality at affected areas [22].

As a developing country with approximately 30 million citizen living in Malaysia, water pollution level has been alarming not only to the citizens but also to the environment. It can greatly affect the whole ecosystems if not taken care of. Sewage and animal wastes together with manufacturing and agro-based industries becomes the biggest contributors to water pollution [22]. However, efforts are being made into reducing not only water pollution but also other pollutions and preserving the natural resources for the usage of upcoming future generations.

There are several efforts and research that has been done to remove various organics pollutants that cause water pollution with the applications of biochar. Biochar has been proved to be one of the most effective ways to enhance and refine the stability and performances by eliminating any potential toxic substances that are present in the aqueous phase especially in a various industrial processes [22].

Hardwood and softwood derived biochars were being used to treat wastewater from oil and mines and also chemical industries that contains furans (furfural and 5-hydroxymethyl furfural (HMF)). Biochar that uses steam activation proved to be more effective as it has over 99% removal efficiency for furfural and HMF [23].

Biochar was used as a precursor of activated carbon by using various types of activation method. One of the most used method to capture and store CO₂ was adsorption which also depends on various parameter. The effects and applications of biochar to soil and pollutions have yet to be determined due to numerous data that do not point to a definite conclusion. The benefits of biochar is said to reduce hazardous environmental impacts are still in the early towards greener technology. The current treatment to remove these contaminants are costly and involve the disposal of toxic waste [27]. Hence, with the new modification towards biochar it could provide environmental friendly and affordable cost to tackle the water pollution problems.

This research is carried out to improve the quality of biochar by using steam activation, highlighting the properties of the biochar by various method, as a way to better understand the essence of biochar application and technology. With a better understanding of the properties and behaviour of woody biochar derived from gaharu, it is hoped that the results may provide useful information toward the direction of green technology.

Two objectives are identified to counter the problem statement stated in the research background which are to improve the quality of biochar which involves the modification process and to modify the surface morphology of the biochar. For this purpose, different steam activation temperature were applied to reach high surface area. In other words, to identify the properties that results in the steam activation method.

The research is conducted to study on the quality or properties of the biochar, in which gaharu wood will be used as a feedstock. Moreover, there are many surface modification methods that are available but because of the limited time frame and it is a low-cost method, only one method is chosen for this research which is steam. As for properties of the modification biochar, six techniques are used to evaluate the composition that is present in the biochar which are by using BET, TGA, elemental, fourier transform infrared spectroscopy (FTIR), SEM and adsorption capacity.

II. METHODOLOGY

The process involved in this experiment are pyrolysis, drying, modification and data analysis. The equipment involved for drying and modification are furnace, microwave oven and autoclave. Figure 1 shows the process flowchart of the process.

A. Sample Preparation

Gaharu waste as a feedstock was obtained from a small wood oil industry in Kuala Nerang, Kedah. The extracted Gaharu residue was collected in the waste stream of the

industry. The residue was in wet condition and in small size chips. These resinous woods have many benefits in making of perfumes, incense and medicine, which this sources have pleasant odour and unique, similar with other nature, where these woods have high value and being treasured worldwide.

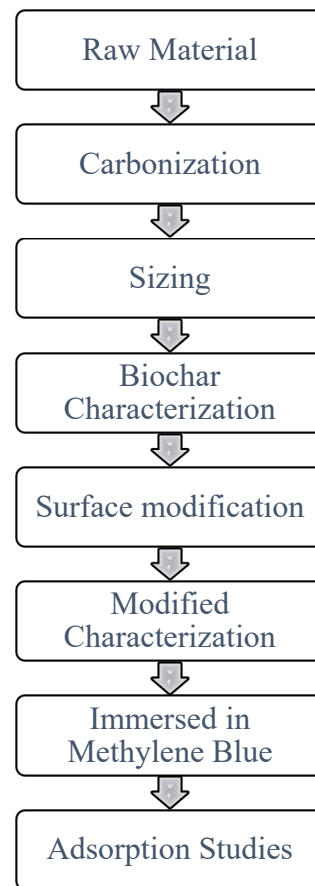


Figure 1: Process Flowchart of the Modification of Biochar by using steam activation

B. Sample Analysis

After the raw gaharu waste was collected, it needs to be prepared according to the requirements of the pyrolysis process. First, the samples need to undergo thermal adjustment which means it need to be dried to remove moisture content. The final moisture for the sample should be at a maximum of 5% to ensure the pyrolysis undergoes its optimum condition. Universal oven (Mettler Universal Oven UF750) at a temperature of 80°C was used to dried the sample. Then the sample were placed in an air-tight plastic at room temperature.

C. *Pyrolysis*

The sample were placed in an aluminum foil and was sealed tightly to prevent air or oxygen from the surrounding to enter and defect the result of pyrolysis. The samples undergo pyrolysis process at five different parameters which are at 400°C, 500°C, 600°C, 700°C and 800°C for 2 hours. Then, the samples were left cooled to a room temperature before it is taken out. Once the samples were left cooled to a room temperature, it is then taken out and were packed in a small packet and were labelled according to their corresponding pyrolysis temperature.

D. *Steam Activation*

The modification method was adopted from the previous researcher. For the modification process, 2g of samples from the pyrolysed biochar were weighed by using an electronic balance. The weighed samples were then placed in a beaker. Then, the samples were wrapped in a small thin cloth before being placed in the autoclave. Beaker were used to placed the samples in order to prevent it from getting wet. The autoclave was set to a different temperature which are 110°C, 120°C, 130°C for 30 minutes and 130°C for 45 minutes. The function of autoclave is the same as pressure cooker which uses pressure and steam to reach and maintain a temperature that is too high for any microorganisms or their spores to live. Once the process is completed, it is then left to cool at room temperature before being taken out. The samples were then placed in a small beaker then stored in the oven for drying process by using universal oven at a temperature of 80°C.

E. *Proximate Analysis and Ultimate Analysis*

Proximate analysis and ultimate analysis were carried out before and after the modification process in order to study the thermal behavior of the samples. Thermal Gravimetric Analyzer (Mettler Toledo TGA/SDTA 851^e) was used in this analysis to determine the moisture content, fixed carbon, ash and volatile matter. First, 20mg of samples were weighted by using electronic balance. Nitrogen gas was used as a carrier gas to ensure the inert condition inside the analyzer. Nitrogen gas was purged at flow rate of 20 mL/min. The sample was heated with a heating rate of 20 °C/min at room temperature to 1100 °C. The analysis was carried out three times to get the average results.

For ultimate analysis, ThermoFisher Scientific Flash EA 1112 Series CHNS-O Analyzer was used in this analysis to determine the elemental composition in the samples. C, H, N, S composition of the sample was directly determined by the analyzer. However, O content was determined by the difference of the C, H, and N. The analysis ran using $\sim 15 \pm 0.1$ mg of sample in triplicate as standard operating procedure (SOP) of the analyzer. The sample was crushed into smaller size prior to analysis due to the compatibility of the analyzer.

F. *Functional Group Analysis*

Fourier Transform Infrared Spectroscopy (FT-IR) is another method used to identify the functional groups present on the biochar surface. However, it is not easy to interpret the IR spectra because the peaks obtained are usually a sum of the interactions of different types of groups. The spectrum for spectrometer was set in the range of 500 to 4000 cm^{-1} . First, disinfect the equipment by using alcohol. Then, small amount of the sample was placed on the surface of the sample holder and force gauge was tighten in the range of 60 - 70 %. The result was analyzed and shown at the computer screen. Hence, the analysis was repeated two times to ensure the accuracy of the results obtained.

G. *BET Surface Area*

BET surface area was measured to explain the physical adsorption of gas molecules on a solid surface. Nitrogen gas sorption analysis at 77K (NOVA 4200e, Quantachrome Instruments, Boynton Beach, FL) was used in this analysis. The samples were weight 200 to 300mg and were purified by degassing at 300°C for 4-16 hours (conditions typical for carbons). Degassing time varied based on the time necessary to reach a stable surface area measurement. Particle density was measured by helium pycnometer (Pentapycnometer, Quantachrome Instruments) using degassed samples from BET analysis and long purge times (10 min) to prevent errors due to volatile content outgassing.

H. *SEM Pore Structure*

Char particle structure and surface topography were examined using a Hitachi S-2460N variable pressure scanning electron microscope (VP-SEM) for electron microscopy (SEM) scanning. Samples were mounted on carbon disks. Insulation samples are examined with very little sample preparation, where it allowed variable pressure mode. A residual atmosphere of 60 Pa (0.5 Torr) of helium was sufficient in order to remove charging from samples while enable reasonably high magnifications up to 1500x.

I. *Methylene Blue Adsorption Capacity*

The main purpose of this research is to learn the effect of changing temperature during pyrolysis toward properties of processed biochar. Thus, the biochar will be analyzed on how properties will affect the amount of methylene blue that will be adsorbed. In order to determine the methylene blue that adsorbs the amount of biochar, this analysis was completed by using DR 2800 UV-Vis Spectrophotometer (Hach, USA) to do the counting of absorbance of the methylene blue solution before and after the biochar was added.

$$q_e = \frac{(C_o - C_e)V}{W}$$

III. RESULTS AND DISCUSSION

a. Proximate analysis & Ultimate analysis

Table 1 Results of elemental analysis of biochar

At steam temperature of 110°C (30 min)					
	400°C	500°C	600°C	700°C	800°C
Moisture	7.74	6.12	6.44	9.63	10.85
Volatile Matter	27.21	19.76	14.73	10.29	16.66
Fixed Carbon	55.84	67.84	66.12	31.49	31.84
Ash	9.21	6.28	12.71	48.59	40.65
At steam temperature of 130°C (30 min)					
Moisture	3.37	2.69	8.01	11.87	7.33
Volatile Matter	19.88	9.71	30.30	18.34	8.33
Fixed Carbon	40.89	28.71	29.85	35.67	17.82
Ash	35.86	58.89	31.84	34.12	66.52

Thermogravimetric Analysis (TGA) was performed for the biomass sample, for a detailed analysis of the weight loss characteristics during activation. Table 1 shows the result of the proximate analysis of biochar acquired at different pyrolysis temperature followed by different steam activation temperature. Sample that undergoes pyrolysis at 700°C followed by steam activation at 130°C have the highest moisture content which is 11.87% while sample that undergo pyrolysis at 600°C followed by steam activation at 130°C have the highest volatile matter which is 30.30%. For the fixed carbon content, the highest is 67.84% from the sample 500°C which undergoes steam activation at 110°C while the highest ash content present is 66.52%.

The TGA curves of the samples shows two distinct peaks which indicates that the first peak corresponded to the decomposition of the hemicellulose while the second is cellulose [30]. Hemicellulose, cellulose and lignin are the three main polymer constituents of land biomass, but their chemical structures and corresponding thermal stability differ. Also, their proportions vary widely between biomass types and parts. Cellulose and hemicellulose consisting of simple sugar monomers decompose at temperatures lower than 450°C, mostly into light molecular weight compounds to be released as pyrolytic vapors. During pyrolysis, lignin slowly decomposes over a wide range of temperature and contributes more to the formation of biochar, leaving condensed aromatic carbons with reduced functional groups.

The TG curves of samples pyrolyse at temperature 700°C are presented in figure 1. The initial weight loss observed in the TG curves over the temperature range 50°C - 150°C, is related to the moisture content of samples. By increasing the temperature from 150°C to 950°C, the relatively intense peak shows the presence of tar and volatile components. As all volatile components of the initial biochar were anticipated to be burnt upon steam activation, the volatile peak observed with a very low intensity may only represent residual oxygenated functional groups in the

activated biochar [4]. Above 950°C, the change detected in both the sample was ascribed to the fixed carbon content. Steam activation temperature at 130°C shows the lowest ash content compared with the sample of temperature 110°C. Gonzalez et al (2009) states that a lower ash content of the activated biochar would be desirable since the char would have a better mechanical strength and higher adsorption capacity.

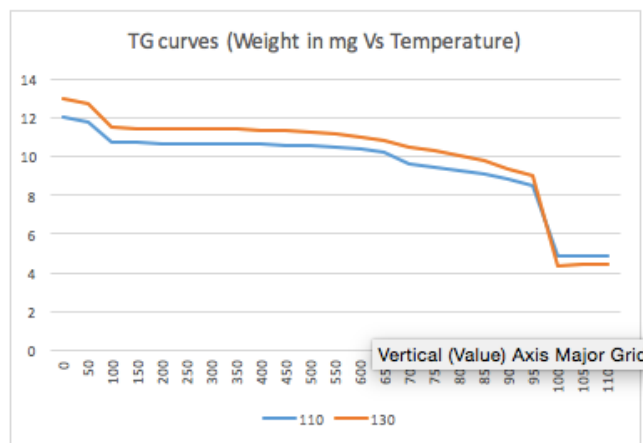


Figure 1 TG curves for sample at steam activation temperature of 110°C and 130°C

This study focuses mainly on the biochar produced from the gaharu waste. In order to retrieve data regarding the nutrient content of the branch biochar, an elemental analysis was carried out using the CHNS elemental analyser, VarioMicro cube. The compositions of biochar made from conversion of biomass is not only affected by the type of feedstock but the pyrolysis conditions [32].

Based on Table 2, the readings were obtained from CHNS elemental analyser to analyse the ultimate analysis content in the biochar. Highest C content which is 81.44% was observed when the pyrolysis temperature is at 800°C followed by the steam activation at temperature of 130°C while the lowest C content (79.55%) was found when the pyrolysis temperature is at 400°C followed by the steam activation at temperature of 110°C. When preparing the biochar from biomass, the pyrolysis temperature affects the properties of biochar the most [32]. With a raise in the steam activation temperature from 110 to 130°C, the carbon content of the chars increased however, the hydrogen and nitrogen content decreased for all heating rates. Losses in H and O content at high pyrolysis temperature are attributed to cleavage and cracking of weak bonds within in biochar structure [32]. As the mass fraction of carbon increased against temperature, the mass fraction of nitrogen however decreases gradually. The very low mass percentage of nitrogen in all samples indicates that all functional groups are mostly oxygenated. These compositions are clearly a result of different process conditions. As reported by Wang et al. (2014), yield and properties of biochar depend heavily

on pyrolysis reactor design, biomass type, reaction parameters (temperature, heating rate, residence time, catalyst and pressure) and feedstock characteristics (particle size, shape and structure).

Table 2 Ultimate analysis of biochar at different steam temperature

Pyrolysis Temperature (700°C)				
	110°C	120°C	130°C (30min)	130°C (45min)
N (%)	2.01	1.84	1.88	1.96
C (%)	79.55	80.09	81.15	81.44
H (%)	4.56	3.48	3.69	3.68
O (%)	13.88	14.59	13.28	12.92

b. BET Surface Area

The microscopic surface area of biochar was measured by using the N₂-BET method (Micrometrics, Tristar 3020). The N₂-BET surface area of biochar by small pores of 2–50 nm is listed in Table 3. The result shows that the surface area It had a wide variation between biochar samples.

Based on table 3, both sample at steam activation temperature of 110°C and 130°C shows high surface area among the others. In his research produced giant Miscanthus char from slow pyrolysis at 500°C and activated it with steam to increase its specific surface area from 181m²/g to 322m²/g [30]. It has been shown that physical or chemical activation increases the surface area and pore volume of carbonaceous materials [7,8]. Hence, the surface area and pore volume greatly increase as the temperature increase. However, it can be concluded that steam activation increased both the specific surface area and cation exchange capacity (CEC), resulting in a huge increase in the copper removal efficiency of the char.

Woody biochars have low ash contents compared to invasive plant- derived biochar and hence, steam application resulted in increased ash production [9]. High ash production tend to have low surface area. Moreover, the surface area determined by N₂ gas adsorption technique may primarily represent the surface of minerals in biochars with high ash content [30]. At a higher temperature, the BET surface area and pore volume are higher mostly due to the increase of the micropore surface area and micropore volume. Hence, steam activation temperature has a strong effect on biochar properties, and therefore biochar can be produced by changing steam activation temperature in order to meet their applications.

Pore size and pore volume constantly increasing as the temperature increased. Clearly, introduction of steam had shown a huge effect on the pore size of the activated biochar as it increased after steam application.

Table 3 BET surface area result of biochar

Pyrolysis Temperature (700°C)				
	110°C	120°C	130°C (30min)	130°C (45min)
BET surface area (m ² /g)	532.6616	489.6824	528.8395	483.1403
Pore volume (cm ³ /g)	0.110979	0.109940	0.113869	0.094967
Pore size (nm)	2.5834	2.6622	2.5201	2.6962

c. FTIR

Fourier-transform infrared spectroscopy (FTIR) is a technique used to identify the functional groups present on biochar and has been used as a qualitative technique for the evaluation of the chemical structure of carbon materials.

The FTIR spectra of the four biochars are shown in Figure 2. The chart clearly shown that there was slightly small change in the peak of the graph. There was a remarkable increased in the volume of peaks associated with O-H stretching (3000–3700 cm⁻¹) for every biochar and aliphatic C-H stretching (750–1400cm⁻¹) vibrations due to loss of labile aliphatic compounds in high temperature-derived biochars. The OH group tend to loss due to bond breaking when undergoes high temperature which is in the range of 700°C – 800°C. The broad and strong O-H in plane bend declined when temperature increased. The peaks between 750 and 885cm⁻¹ for biochar that undergoes steam activation can be attributed to out-of-plane deformations of aromatic C-H [35]. This confirms the highly aromatic nature of biochar that undergoes high steam temperature compared to low steam temperature. These changes in biochar properties based on production conditions are consistent with the detailed explanation given by [36], who highlighted changes in biochar properties according to changes in production parameters (i.e., peak temperature, heating rate, physical activation process using oxidizing agents such as steam and carbon dioxide). Overall, these characterization results confirm that production conditions affect biochar properties and may lead to different sorption capacities towards contaminants.

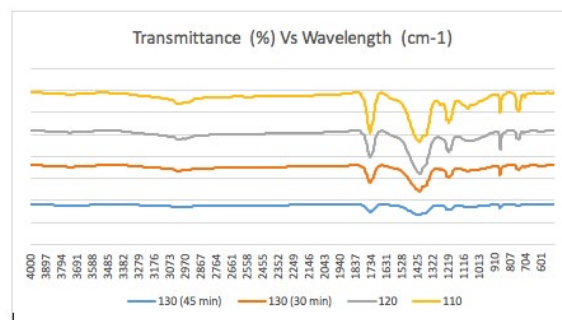


Figure 2 FTIR Spectra of Biochars Obtained at Different

Steam Activation Temperature

d. Methylene Blue Adsorption Capacity

Methylene blue showed enhanced sorption capacity under magnetic field [17]. The adsorption capacity of the biochars were analysed by methylene blue number. From the figure 3 and 4, it shows that the adsorption capacity fluctuates then increases as the temperature increases. Increasing in the pore structure of the biochar will result in increasing BET surface area and thus, increase the adsorption capacity. The capacity of the biochar at 800°C can be achieved up to 1.1054 mg/g which means that for every gram of biochar, 1.1054 mg of Methylene Blue can be adsorbed.

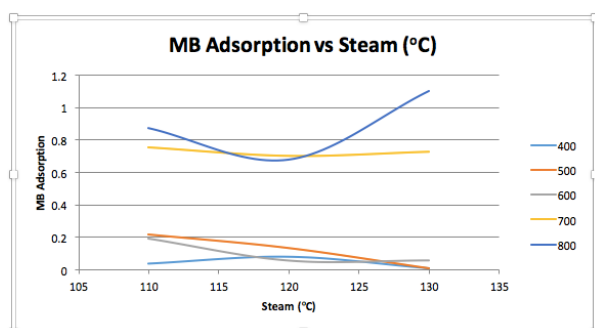


Figure 3 Effect of Steam Temperature on Methylene Blue Adsorption Capacity

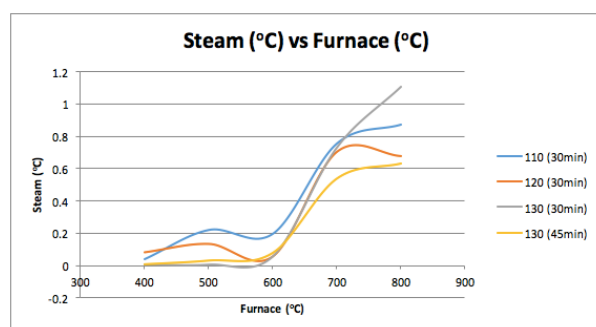


Figure 4 Effect of Pyrolysis Temperature on Steam Temperature

IV CONCLUSION

The surface of the gaharu waste has been modified by using steam activation. The characterization of the biochar includes qualitative and quantitative analysis. Hence, it also involves the determination of the pore and surface area of the biochar. The data presented in this work showed that both the pyrolysis and steam activation temperature strongly influence the physiochemical properties of the biochar. In particular, an increase in the temperature improved the adsorption properties such as surface area, pore area and porosity. Sample that is pyrolysed by temperature of 700°C followed by steam activation temperature at 130°C for 30 minutes was chosen as it fulfilled and is apt for such applications. Residence time also plays an important role in this method as it also

affects the physical and chemical properties of the biochar.

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