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Influence of Carbonization Temperature on Physicochemical Properties of Biochar derived from Slow Pyrolysis of Durian Wood (*Durio zibethinus*) Sawdust

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The objective of this study was to explore the influence of pyrolysis temperature on the physicochemical properties of biochar synthesized from durian wood (*Durio zibethinus*) sawdust. Surface morphological features, including the porosity and BET surface area of biochars, provide appropriate dimensions for growing clusters of microorganisms with excellent water retention capacity in soil. Oxygen-containing surface functional groups play a vital role in improving soil fertility by increasing its cation and anion exchange capacities with reduced leaching of nutrients from the soil surface. Biochar was produced *via* slow pyrolysis of woody biomass (WS) using a fixed bed reactor under an oxygen-free atmosphere at different pyrolysis temperatures (350, 450, and 550 °C). The biochars obtained were characterized using ultimate and proximate analyses, Brunauer-Emmett-Teller (BET) surface area, field-emission scanning electron microscopy (FE-SEM), Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD). The yield of biochar decreased from 66.46 to 24.56%, whereas the BET surface area increased sharply from 2.567 to 220.989 m^2/g, when the pyrolysis temperature was increased from 350 to 550 °C. The results highlighted the effect of pyrolysis temperature on the structure of the biochar, which could be advantageous for agricultural industries.

Keywords: Slow pyrolysis; Biomass, Biochar; Fixed bed; Carbonization; Sawdust

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INTRODUCTION

Thermochemical transformation of lignocellulosic residues using combustion, fast pyrolysis, and slow pyrolysis in conjunction with gasification techniques has received increasing research attention. These techniques are simple and flexible in converting biomass wastes into value-added chemicals and fuels (Yanik *et al.* 2007). Among these processes, slow pyrolysis has been used extensively to obtain syngas, liquids, and solid fractions of biochar due to its moderate operating conditions (Chowdhury *et al.* 2013; Xie *et al.* 2015). Biochar is a stable, recalcitrant, organic carbonaceous compound that can be procured from a variety of feedstocks such as lignocellulosic biomass, manure, and municipal solid waste. Despite the relatively recent introduction of the term “biochar”, versatile applications of charred materials have been identified due to their unique physiochemical properties (*e.g.*, high surface area, porosities, surface functional groups, and sorption capacities). It contains polycyclic aromatic hydrocarbon with an array of different functional groups. The surface of the biochar can exhibit hydrophilic, hydrophobic, acidic, and basic properties. It was observed that the pyrolysis temperature can greatly affect the extent of humic and fulvic acid in biochar derived from *Eucalyptus* wood (Trompowsky *et al.* 2005).

The physiochemical properties and compositions of biochar vary sharply based on the feedstock and the experimental conditions (Arash Arami-Niya *et al.* 2012, Bridgwater *et al.* 1999; Bahng *et al.* 2009; Brewer *et al.* 2009). Different types of lignocellulosic residues, including woody biomass (Abdullah and Wu 2010), wheat (Chun *et al.* 2004), rice straw (Peng *et al.* 2011), corn cob (Feng *et al.* 2012), and sugar beet tailings (Yao *et al.* 2011), have been used to synthesize biochar. Due to variation in carbon and lignin contents of different types of lignocellulosic materials, the yield of biochar produced from a given quantity of biomass varies greatly; the highest yield was obtained from woody biomass (Xie *et al.* 2015). Careful selection of the reaction conditions for thermochemical processes can transform compact, heterogeneous forms of lignocellulosic biomass into advantageous forms tailored to the user’s requirements (Abdul Hamid *et al.* 2014). Design of the pyrolysis reactor, biomass composition, reaction parameters (including temperature, time, heating rate, catalyst type, and pressure), and the physical characteristics of biomass residues in terms of particle size and porosity have robust influences on the yield and physiochemical characteristics of biochar (Bridgwater *et al.* 1999). The mechanism of biochar formation from biomass is complicated. However, the following three steps were suggested to describe the reaction mechanism of char formation by previous researchers (Demirbas 2004),

$$\text{1st step: Biomass} \longrightarrow \text{Water + Unreacted residue} \qquad (1)$$

$$\text{2nd step: Unreacted residue} \longrightarrow (\text{Volatile + Gases})_1 + (\text{Char})_1 \qquad (2)$$

$$\text{3rd step: } (\text{Char})_1 \longrightarrow (\text{Volatile + Gases})_2 + (\text{Char})_2 \qquad (3)$$

The biochar sample produced in the second stage undergoes further decomposition to yield carbonaceous residual solids through chemical changes in the biochar. Because of its wide range of applications, it has received significant research attention recently. Biochar can potentially be used as a carbon sequestration agent as well as a natural fertilizer to amend soil properties (Brewer *et al*. 2009). Biochar contains calcium, potassium, phosphorus, magnesium, and other elements that can act as liming agents for acidic soil. It improves nutrient holding capacity, water retention, the pH of the soil, and crop yield (Chan *et al*. 2008; Zweiten *et al.* 2010). Furthermore, it decreases the bioaccumulation of metallic cations and organic contaminants in the soil and adsorbent for waste water treatment (Zwieten *et al*. 2010, Masulili *et al*. 2010; Jones *et al.* 2011a,b, 2012). It can improve the quality of the soil by changing the microbial community (Jones *et al.* 2011a,b). Application of biochar is considered an effective means to sequester atmospheric carbon while concurrently producing carbon-negative energy and improving soil properties (Brewer *et al.* 2009). Therefore, detailed information concerning the properties of biochar is valuable.

Durian (*Durio zibethinus*) is a seasonal fruit that grows abundantly in Southeast Asian countries. It is a tropical fruit and belongs to the Bombaceae family and *Durio* genus (Jun *et al.* 2010). It is often referred to as the “King of Fruits”. *Durio zibethinus* is commercially grown in Malaysia. Statistics show that the total production of durian in Malaysia was approximately 376,273 metric tons in 2008 (Department of Agriculture Malaysia 2009). Prior to this study, no research has been conducted regarding the synthesis of biochar from durian wood sawdust using a slow pyrolysis technique. The purpose of this study was to investigate the properties of biochar produced under various pyrolysis temperatures using a fixed bed reactor. The present experimental work focused on important biochar properties including its elemental composition, crystallinity, structural changes, and morphology. The future perspective of this work is to apply the prepared biochar as a soil amending agent and an adsorbent material in single and multi-lab-scale and industrial-scale solute systems.

EXPERIMENTAL

Preparation of Biochar

The starting lignocellulosic material, durian wood sawdust, was collected from a local sawmill in Malacca, Malaysia. The wood chips were ground and sieved. The material was passed through a 50-mesh screen to remove large lumps. The average particle size of the wood sample was kept at approximately 0.7 to 0.8 mm. The ground wood was dried at 110 °C for 24 h and stored in a sealed container before initial characterization.

The sample was pyrolyzed from room temperature to the desired temperature using a heating rate of 10 °C/min. The pyrolysis process was carried out using a tube furnace with continuous nitrogen gas purging at a flow rate of 30 mL/min. The design of the fixed-bed reactor used in this research was described in previous works (Chowdhury *et al*. 2012a, b). Biochar samples were prepared at 350, 450, and 550 °C with a residence time of 3 h. The biochars obtained were labeled, according to the heating temperatures, as B-350, B-450, and B-550.

Analytical Methods

The raw samples and prepared biochar samples were characterized with a CHN elemental analyzer (PerkinElmer-2400, Tokyo, Japan). Thermogravimetric analysis coupled with a differential thermal analyzer (DTA) (Mettler Toledo Star SW901) was carried out to determine the thermal stability of the samples under 10 mL/min nitrogen flow. In the thermogravimetric analysis (TGA), 5 mg of each sample was heated to 1100 °C under a heating rate of 10 °C/min. Ultimate analysis was carried out using the Elemental Analyzer (PerkinElmer, Series II 2400) to evaluate the percentage of carbon, hydrogen, and nitrogen in WS, B-350, B-450, and B-550.

The pH of the biochar was determined according to Novak *et al.* (2009). To begin, 2 g of biochar was shaken with 40 mL deionized water for 30 min. The sample was allowed to settle for 15 min before recording the pH using pH electrode 827 pH Lab (Metrohm, USA). Biochar was shaken with deionized water for 24 h, during which the solid solution ratio was kept at 1:10. The electrical conductivity was measured using a conductimeter (Jenway 4510, UK). The cation exchange capacity (CEC) of biochar was measured by the ammonium acetate (NH_4OAC) extraction methods (Song and Guo 2012; Melo *et al.* 2013). Then 40 mL of 1 M NH_4OAC was agitated with 0.5 g of biochar in a 50-mL glass centrifuge tube at room temperature for 20 min. The resultant mixture was transferred onto a 0.45 µm glass fiber filter under vacuum. The biochar remaining on the filter was further washed with two 20-mL portions of 1 M NH_4OAC, followed by three 30-mL portions of isopropanol. The biochar was then rinsed with four 50-mL portions of 1 M KCl. The rinsate was collected in the vacuum flask and the volume was made up to 250 mL using deionized water. NH_4^+ ions concentration was measured by the Kjeldahl method (Melo *et al.* 2013). The CEC ($mmol_c$ kg^{-1}) of biochar was calculated by normalizing the amount of NH_4^+ in the rinsate with the biochar mass (0.5 g).

X-ray diffraction (XRD) patterns of the raw samples and biochars were examined using a D5005 apparatus (Bruker, Germany) at 40 kV and 40 mA with a Cu-Ka radiation source. A continuous 2θ scan mode from 5° to 60° was applied for high degree scanning at step size of 0.02 and step time of 2 s. A scanning electron microscope (SEM, Model Leo Supra 50VP Field Emission, UK) was used to evaluate the morphological features of the biochars prepared from raw wood samples. The Brunauer-Emmett-Teller (BET) surface area, pore volume, and pore size distribution of the prepared char samples were analyzed with an Autosorb 1 Quantachrome Autosorb automated gas sorption system. The char samples were outgassed under vacuum at 400 °C for 6 h to eliminate residual moisture before nitrogen gas adsorption. The BET equation was used to calculate the surface area of the biochar. The surface functional groups of the biochar and raw wood sawdust were determined by FT-IR, in which the test samples were prepared by mixing the biochar with KBr at a fixed ratio to fabricate a translucent disc. The FT-IR spectra were recorded in the range of 400 to 4000 cm^{-1}.

RESULTS AND DISCUSSION

Effect of Temperature on Yield Percentages

The yield of biochar decreased as the pyrolysis temperature was increased from 350 to 550 °C, as illustrated by Table 1. The biochar yield decreased from 66.56 to 24.46%. Usually, at lower temperatures, the primary thermal degradation of lignocellulosic biomass occurs. After that, subsequent increases in the temperature further crack the volatile materials into low-molecular weight organic compounds and gases rather than biochar (Thangalazhy-Gopakumar *et al.* 2010). Increasing the temperature would enhance the dehydration and elimination reactions, decreasing char yield (Bacaoui *et al.* 2001; Lua and Yang 2004; Adinata *et al.* 2007). The observed decrease in biochar yield with increasing temperature is consistent with other literature regarding the pyrolysis of woody biomasses (Wang *et al.* 2009, Garcia-Perez *et al.* 2008; DeSisto *et al.* 2010; Keiluweit *et al.* 2010).

Effect of Temperature on Physiochemical Properties of Biochar

Proximate and ultimate analysis of raw wood and biochars

The elemental composition of the raw biomass and biochar samples investigated are presented in Table 1. The carbon content in the samples increased from 3.38 to

70.89% with increasing pyrolysis temperature. However, the hydrogen and oxygen contents decreased successively with increasing temperature. This implies that the degree of carbonization was enhanced by increasing the pyrolysis temperature (Chun *et al*. 2004; Chen *et al*. 2012). The reduction of the hydrogen and oxygen contents at higher temperatures was due to the cleavage of weak bonds inside the biochar matrix (Demirbas 2004).

Table 1. Proximate and Ultimate Analyses of Wood Sawdust (WS) and Biochar

	Yield	Proximate Analysis (wt. %)				Ultimate Analysis (wt. %)							pH	EC	CEC
Sample	**(%)**	**Water Content**	**Volatile Matter**	**Fixed Carbon**	**Ash**	**C**	**H**	**N**	**O**	**H/C**	**O/C**	**C/N**		**dS/m**	**$mmol_c kg^{-1}$**
WS	-	8.07	78.88	3.38	9.67	33.77	11.40	6.06	48.77	0.23	1.44	-	-	-	-
B-350	66.56	4.58	45.88	36.88	12.66	40.88	8.35	5.12	45.65	0.18	1.11	7.98	6.1	0.76	27
B-450	41.22	3.05	22.77	55.88	18.30	52.99	6.01	2.01	38.99	0.15	0.74	27.8	6.4	0.89	25
B-550	24.46	1.57	6.77	70.89	20.77	75.99	1.02	0.55	22.44	0.04	0.30	128.9	6.8	0.98	22

Figure 1 is a Van Krevelen plot showing the H/C and O/C atomic ratios in terms of pyrolysis temperature. The H/C and O/C ratios are calculated to evaluate the degree of aromaticity and carbonization inside the biochar samples (Krull *et al.* 2009). Significant decreases in the H/C and O/C atomic ratios were observed compared to the raw saw dust sample with increased pyrolysis temperature.

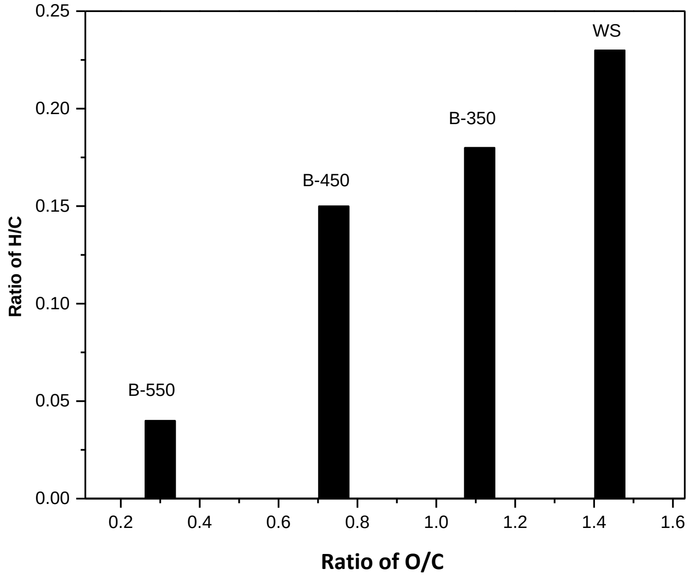

Fig. 1. Comparison of Van Krevelen plots of wood sawdust (WS) and biochars produced at various temperatures (B-350, B-450 and B-550)

Lower O/C ratios in biochar produced at higher temperature increases its stability in the environment (Melo *et al.* 2013). The raw wood sawdust had relatively high H/C atomic ratio, reflecting low aromaticity compared to that of the biochar samples. Woody biomass pyrolyzed at lower temperature contained some carbohydrates. Thus, the H/C and O/C ratios were higher for B-350 than B-450 and B-550. This is due to increased rate of dehydration, decarboxylation, and decarbonylation of the sample with increased temperature (Tang and Bacon 1964; Kim *et al.* 2011). At higher temperature, plant based biomass undergoes dehydration and depolymerization reactions to form volatile lignin and cellulose which condensate subsequently to yield graphitic structure inside the biochar matrix (Melo *et al.* 2013). These results indicated that at elevated temperature, more recalcitrant carbon structure was formed inside the biochar matrix. A similar observation was reported earlier for preparation of graphitic black carbon, switch grass, and pine wood biochar (Schmidt and Noack 2000; Kim *et al.* 2011). The C/N ratio was also increasing with increasing temperature. The C/N ratio obtained for B-450 and B-550 were greater than 20. This indicated that the wood derived biochar had good potential for net N immobilization in the soil (Stevenson and Cole 1999; Fungai *et al.* 2013).

Table 1 shows the proximate analyses of raw biomass and the biochar samples. Results demonstrated that the fixed carbon contents of the biomass increased following pyrolysis. The carbon content increased with increasing pyrolysis temperature. The ash content reflects the extent of non-volatile and non-combustible matters in biochar (Angin 2013). The ash contents of the biochar samples were also higher than in the raw biomass. Compared to B-350, the B-450 and B-550 contained more ash and fixed carbon, whereas their volatile component content decreased with increasing temperature. A similar trend was followed during carbonization of safflower seed press cake based biochar (Angin 2013).

Thermogravimetric analyses (TGA) of the raw biomass and biochars were performed to better understand the thermal stability of the samples. Figures 2(a) and 2(b) show the TG curves and differential calculations of the weight loss (DTG) of the biochars. The weight loss in the TG/DTG curves observed for the raw biomass indicated water loss up to 130 °C. After that, degradation of hemicelluloses and cellulose took place at 200 to 300 °C and 300 to 400 °C, respectively.

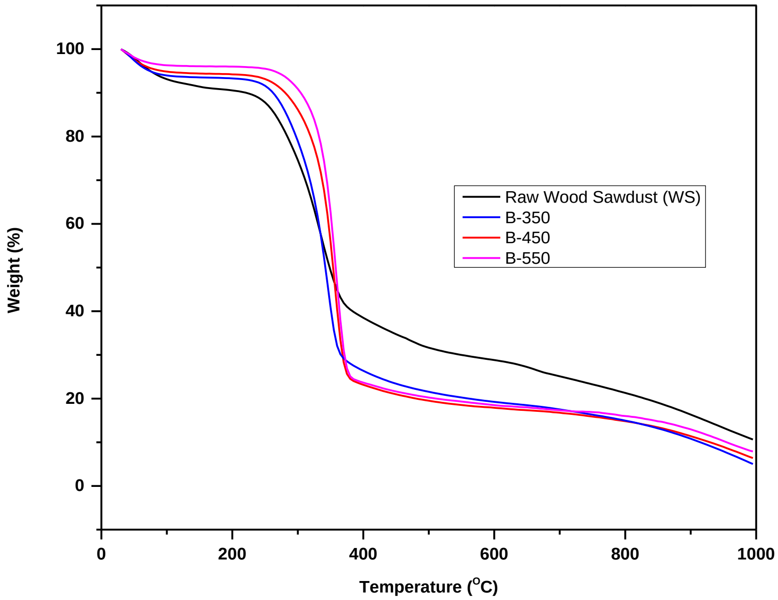

Fig. 2. Thermogravimetric curves for wood sawdust (WS) and biochars produced at various temperatures (B-350, B-450, and B-550)

As shown by previous research, the degradation of hemicelluloses partially overlaps with cellulose degradation in the biomass, and lignin degradation occurs within the temperature range of 200 to 700 °C (Gronli *et al.* 2002; Putun *et al.* 2007). The maximum weight loss of the biomass occurred at 359 °C, due to the thermal degradation of cellulose. The degradation features of the prepared biochars were notably different from one another. Relative to B-350, the B-450 and B-550 exhibited higher thermal stability. As the temperature increased, the biochar contained more stable forms of carbon that were more heat resistant. The mass loss for biochar prepared at different temperature was less than the starting biomass itself. Mass losses of B-350, B-450, and B-550 at were around 25.35%, 21.56%, and 19.79%, respectively. This trend was expected and was observed previously for saw dust based biochar (Azargohar *et al.* 2014). The mass loss at higher temperature is related to dehydrogenation and aromatization of char with subsequent decomposition of inorganic element (Oja *et al*. 2006).

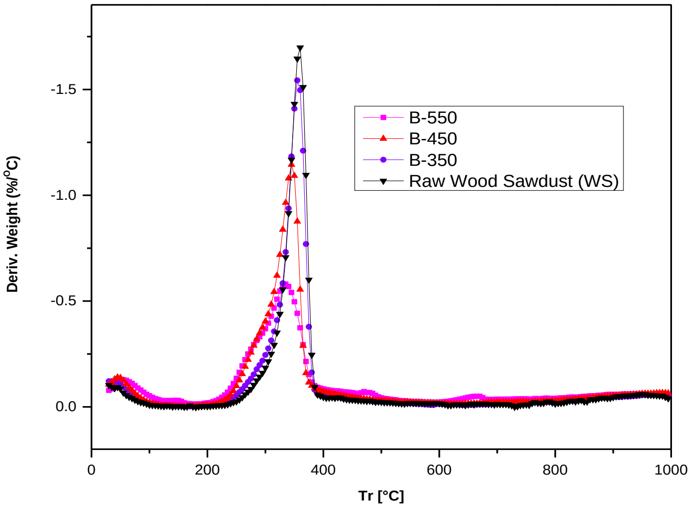

Fig. 3. Differential thermal gravimetric curves of wood sawdust (WS) and biochars produced at various temperatures (B-350, B-450, and B-550)

The pH and electrical conductivity (EC) of the prepared biochar increased slightly with increasing temperature. This is due to the presence of greater ash content in biochar prepared at higher pyrolysis temperature (Melo *et al.* 2013). The EC values give measure of the total water soluble ions (salinity). It can affect the plant growth negatively as it tends to decrease water uptake by plant roots resulting reduced nutrient intake (Chan *et al.* 2008). The biochar prepared here from wood sawdust (WS) had comparatively low EC values.

It was reported earlier that EC values around 4 ds/m in soil water extract cause salinity of the soil which is harmful for plant growth (Carrier *et al.* 2012). Thus the biochar prepared here should not have significant negative impact on salinity of the soil. The cation exchange properties (CEC) decreased with successive increase of temperature. With increasing temperature O/C and H/C ratios were decreasing. The reduction of CEC values may be due to reduction of oxygen containing functional groups (Singh *et al.* 2010; Song and Guo 2012; Mukherjee *et al.* 2011).

SEM image analysis

Pyrolysis degrades cellulose, hemicellulose, and lignin within the biomass sample. SEM images of the raw biomass and biochar samples obtained at different temperatures are shown in Fig. 4. The surface morphology of the biomass changed evidently following pyrolysis. The surface initially was comparatively smooth and without pores, cracks, or crevices before pyrolysis. However, after pyrolysis at 350 °C, the surface became uneven. Few pores were observed on the surface of the biochars derived at lower temperatures (Fig. 4b). The biochar samples produced at lower temperatures were blocked by volatile matter and ash (Fig. 4b), decreasing the porous volume. However, the porosity of the biochar increased with increasing temperature. After carbonization at 450 °C, the number of pores increased. Some large pores were observed over the surface of B-550, containing some micropores inside them. The large pores might be originated from the vascular bundles of the starting wood biomass (WS). This phenomenon was earlier reported for producing biochar from bamboo at 500 °C (Hernandez-Mena *et al*. 2014). The porous texture is beneficial for improving the soil quality by providing habitats for symbiotic microorganisms (Thies and Rilling 2009). This shows that thermal carbonization at 550 °C was effective in creating virtually well-developed pores on the surfaces of the biochar. The results were in agreement with the preparation of palm shell based biochar (Arami-Niya *et al.* 20012). This observation is consistent with the data reported in Table 2, in which the pore volume was almost 1.7 times higher for B-550 than B-450.

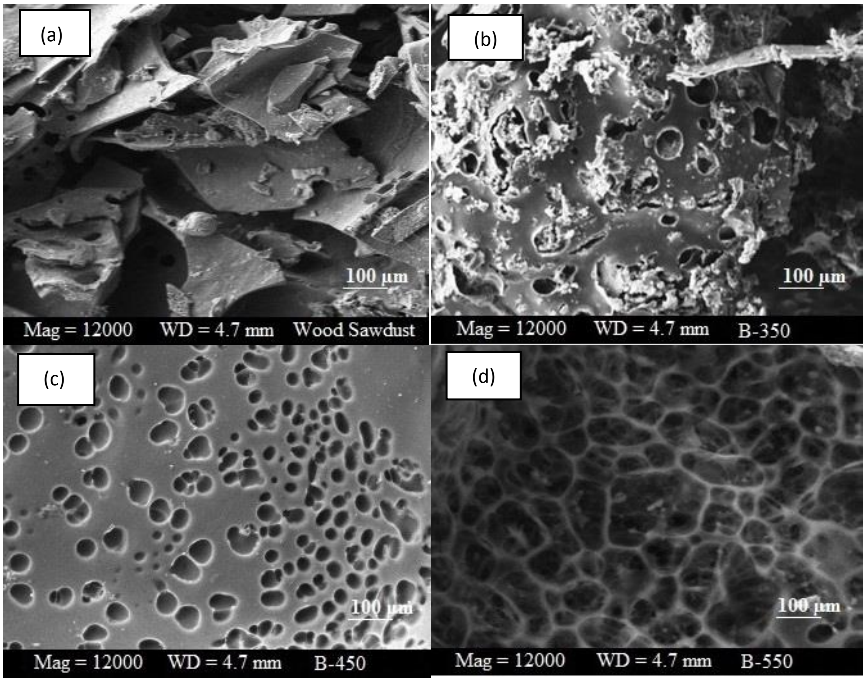

Fig. 4. SEM imagines of biomass and biochar: (a) wood sawdust; (b) B-350; (c) B-450; and (d) B-550 (Magnification 12000 x, 20.0 kV, Low Vacuum)

BET surface area analysis

Table 2 summarizes the BET surface areas and total pore volumes of the biochar samples B-350, B-450, and B-550.

Table 2. BET Surface Area Analysis of Wood Sawdust and Biochar

Sample	BET Surface Area (m^2/g)	External Surface Area (m^2/g)	Total Pore Volume x 10^{-3} (cc/g)	Average Pore Diameter (nm)
WS	1.384	1.006	0.789	1.787
B-350	2.567	2.009	4.543	6.098
B-450	45.78	42.89	5.786	8.098
B-550	221.0	209.6	8.587	12.78

It was observed that increasing the pyrolysis temperature from 350 to 450 °C substantially increased the BET surface area. Furthermore, at a high temperature of 550 °C, the BET surface area increased considerably, to 221.0 m^2/g. This was due to incomplete carbonization at lower temperatures. This phenomenon was observed earlier in the SEM images in which pores were blocked by deposition of tar-like substances and ash residues. Further increases in the temperature, to 550 °C, facilitated the release of volatile materials and created more pores, resulting in larger surface area. Greater surface area is desirable because it aids in improving soil properties and increases water retention capacity to a greater extent (Shaaban *et al*. 2013). It was reported that the biochars derived from rice husk and empty fruit bunch by slow pyrolysis at 500 °C for 2 h had BET surface areas of 230.91 and 15.42 m^2/g, respectively (Claoston *et al.* 2014). However, other literature reported that the BET surface areas of rice husk biochar and elm biochar produced by slow pyrolysis at 500 °C for 4 h were 12.2 and 84.3 m^2/g, respectively (Wang *et al*. 2013). The surface areas of crop residue-based biochar samples were increased from 116 to 438 m^2/g with increases in the temperature from 400 to 600 °C (Chun *et al.* 2004), but decreased at 700 °C. The specific surface area of the biochar is closely associated with the pyrolysis reactor type, temperature, time, flow rate, and other parameters. A sudden increase in surface area was also observed for wood char obtained from *Birch pendula* at a certain heating temperature previously (James *et al.* 2005). It was observed that the surface area was just 5.6 m^2/g when carbonized at 600 °C, but it increased drastically to 430 m^2/g when heated at 700 °C (James *et al.* 2005). Likewise, *Pinus sylvestris* based wood char had surface area of 3.6 m^2/g at 450 °C which was increased up to 320 m^2/g at 500 °C (James *et al.* 2005).

Surface functional group (FTIR) analysis

Table 3 lists the functional groups present in the raw wood sawdust and biochar samples obtained at different pyrolysis temperatures. The spectrum for the biochar obtained at the lower temperature of 350 °C was somewhat similar to that of the raw material. At higher temperatures, some peaks disappeared and the spectra became simpler. This was attributed to the rupture of various functional groups with increasing temperature. The peaks in the range of 474.02 to 579.54 cm were assigned to C-H out-of-plane bending and the O-H stretching vibrations of the C-O-H band. The peak around 1400 to 1550 cm^{-1} was due to the C=O stretching vibration of carboxylate groups. It was reported previously that different types of oxygen-containing functional groups, which existed in the raw pistachio nut shell, disappeared after thermal activation, initiating aromatization inside the carbon structure (Yang and Lua 2003). A similar phenomenon was observed for raw date pits, which after activation lost aliphatic -C-H groups and developed some aromatic C=C and oxygen groups (El-Hendawy 2005). A wide-ranging band around 3550 to 3420 cm^{-1} was ascribed to the hydrogen bonded -OH stretching of phenol (Ahmad *et al.* 2007). However, the intensities decreased significantly at higher temperatures (550 °C). A similar observation was reported earlier as a result of the dehydration of woody biomass and the cleavage of phenolic groups at temperatures above 500 °C (Wang *et al.* 2013).

Table 3. Functional Groups of Wood Sawdust (WS) and Biochar Samples Determined by the FTIR Analysis

Peak Number	Frequency				Assignment
	WS	B-350	B-450	B-550	
1	474.02	-	436.45	446.25	C-H out-of-plane bending of benzene derivatives
2	538.40	549.26	569.66	579.54	C-H bending
3	913.89	-	-	-	O-H bending
4	-	-	1079.67	1088.64	C-O-C stretching of esters, ethyl, or phenyl group
5	-	1380.98	1390.99	1345.94	CH_3 deformation
6	1590.58	1595.98	1599.87	1509.70	C=C ring stretching of benzene derivatives
7	1668.75	-	-	-	C=O stretching
8	-	1788.87	-	-	C=O stretching
9	2335.25	-	-	-	C=C stretching vibration of ketones, aldehydes, or carboxylic groups
10	2370.38	-	-	-	C=C stretching vibration of ketones, aldehydes, or carboxylic groups
11	2870.67	-	-	-	C-H stretching
12	2975.59	-	-	-	C-H stretching
13	3390.77	3453.89			O-H stretching vibration of hydroxyl functional groups
14	-	-	3514.67	3520.33	C-H out-of-plane bending of benzene derivatives

The peak observed in the raw sample at around 2800 to 2900 cm^{-1} was disappeared following carbonization. The disappearance of the peak representing aliphatic C-H stretching vibration (2975 to 2870 cm^{-1}) suggests the loss of aliphatic compounds during the pyrolysis process with development of additional recalcitrant, aromatic constituents (Wang *et al.* 2013). The loss of -OH and C-H groups indicate the formation of pores inside the biochar matrix with simultaneous development of fused ring structures (Bagreev *et al.* 2001).The acidic group of C=O for lactone, at around 1788.87 cm^{-1} in B-350, disappeared for B-450 and B-550.

X-Ray diffraction (XRD) analysis

The X-ray diffraction patterns of the raw wood biomass and biochar samples are illustrated in Fig. 5. Two narrow, sharp peaks at 2θ values of around 16 and 22° for the raw biomass were observed due to presence of crystalline cellulose in the wood sample (Yang *et al.* 2007).

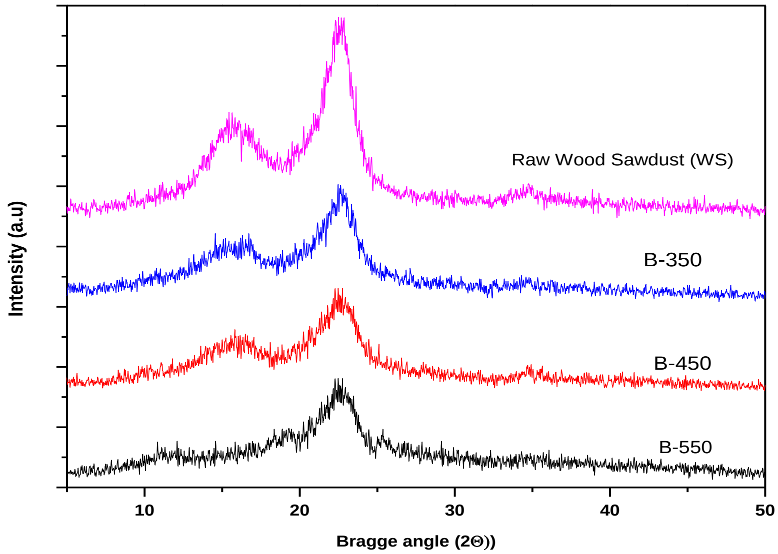

Fig. 5. X-ray diffractogram of raw wood sawdust (WS), B- 350, B-450, and B-550

For B-350, the intensities of these two peaks were decreased, demonstrating partial cellulose degradation (Keiluweit *et al.* 2010). However, the crystalline cellulose was totally destroyed at higher temperature. These two peaks were barely visible in the spectra for B-450 and B-550. Higher pyrolysis temperatures yielded a broader peak at 2θ values of around 22 to 24°. This indicated the development of atomic order in the carbonized sample at higher temperatures (Keiluweit *et al.* 2010). The absence of sharp peaks demonstrated the amorphous texture of the biochar sample (Tongpoothorn *et al.* 2011). It was reported previously that the formation of aromatic structures began after the complete disintegration of the nanocomposite structure during the charring process (Paris *et al.* 2005). The occurrence of a broad peak around 22 to 24° in the biochar prepared at higher temperatures indicated the formation of a crystalline structure with improved layer alignment (Yang and Lua 2006). Similar results have been reported for the carbonization of *Jatropha curcas* and nut shell (Pechyen *et al.* 2007; Tongpoothorn *et al.* 2011).

CONCLUSIONS

1. Biochar yield decreased significantly, from 66.46 to 24.56%, when the pyrolysis temperature was increased from 350 to 550 °C.
2. The physicochemical characteristics of the prepared biochar were functions of the pyrolysis temperature. The degree of carbonization of the B-350, B-450, and B-550

samples were enhanced with successive increases in the pyrolysis temperature from 350 to 550 °C.

3. Increasing the pyrolysis temperature significantly increased the carbon content and reduced the oxygen and hydrogen contents of the biochar sample.
4. The BET surface area and the porosity of the biochar sample increased from 2.567 to 220.989 m^2/g with increasing pyrolysis temperature.
5. With successive increase in temperature for char formation, the cation exchange capacity (CEC) reduced slightly; whereas the electrical conductivity (EC) and pH values increased with elevated temperature.
6. Acidic functional groups decreased and were largely replaced with basic groups such as quinone on the surface of the biochar following to the carbonization process. Increased basic functional groups can increase the pH of the soil. The future perspective of this research is to apply the prepared biochar to acidic soil to enhance its fertility and improve crop yield.

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