Ijeas Ideal Journal of Engineering and Applied Sciences 2(3) 129-137 Ijeas
 (ISSN: 2067-7720)
http://ijeas.truescholar.org

EFFECT OF PYROLYSIS TEMPERATURE ON THE PROPERTIES OF COCONUT FROND BIOCHAR

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ABSTRACT

Biochar has attracted interest due to its benefits in agricultural sector and environmental sustainability. Biochar can act as a useful tool to sequester carbon and reduce carbon dioxide (CO_2) which cause global warming and climate change. Biochar is also able to improve soil fertility and mitigate climate change by sequestering carbon. The functions of biochar depend greatly on its physical and chemical properties. The type of feedstock and pyrolysis conditions such as temperature, heating rate and residence time are the most important factors influencing the biochar properties. The objective of this study is to investigate and evaluate the effect of pyrolysis temperature on the biochar yield as well as the properties of biochar produced from slow pyrolysis of coconut frond (CF). The preliminary analysis such as proximate and elemental analysis, lignocellulosic determination and thermogravimetric (TG) analysis were carried out to determine the properties of CF feedstock. Laboratory-scale slow pyrolysis experiments were performed at five different temperatures, 400°C, 450°C, 500°C, 550°C and 600°C. Heating rate, residence time and nitrogen flowrate were set at 5°C/min, 1 hour and 0.5 L/min respectively. CF biochars produced at different temperatures were investigated using proximate and elemental analysis, Field Emission Scanning Electron Microscope (FESEM) and ***Brunauer–Emmett–Teller*** (BET) surface area analysis. The preliminary analysis results show that the CF is suitable to be used as feedstock for pyrolysis process. It contains high volatile matter of 75.27 mf wt% and low percentage of sulfur, 0.77%. CF feedstock also comprises of 21.46% of cellulose, 39.05% of hemicelluloses and 22.49% of lignin. The biochar yield decreased from 35.71 wt% to 28.53 wt% as the temperature increased from 400°C to 600°C. The fixed carbon of the CF biochar increases from 76.40 mf wt% to 78.38 mf wt% as the temperature increased from 400°C to 600°C. The FESEM shows the existence of pores at the wall of the fibrous strands of the CF biochar. The increment of pyrolysis temperature also lead to the formation of biochar with higher BET surface area and micropore volume. BET analysis reported maximum surface area of 215.30 m^2/g and micropore volume of 0.07912 cm^3/g at pyrolysis temperature of 600°C. The findings of this study show that an increased of pyrolysis temperature decreased the percentage yield and increase the fixed carbon, BET surface area and micropore volume of CF biochar. CF biochar therefore has the potential to be applied as soil amendments.

KEYWORDS: Biochar, Slow Pyrolysis, Coconut Frond, Pyrolysis Temperature, Characterization

INTRODUCTION

Global warming is largely caused by the greenhouse gases especially carbon dioxide (CO_2) emissions from the human activities. The anthropogenic activities increase the atmospheric concentration of CO_2 to almost 400 ppm at present compared to 280 ppm in the pre-industrial era (Lal, 2011). Since 1880, the mean global temperature has risen by 0.85°C and the impact is now evident from the rise in global sea level, melting of snow, increase in frequency of extreme events such as hurricanes including Katrina, Sandy and Rita, drought in many region including India and recently severe heat wave that stroke Pakistan (IPCC, 2014). The effect of global warming will bring risk to human support systems, such as food, agriculture, water resources, ecosystems and human health (IPCC, 2014; PIK, 2012). Malaysia is likely to feel the force of climate events sooner, due to its climate and location. The effects of climate change can be seen in the form of coastal and inland flooding, rise in vector borne diseases and drops in agricultural yields due to continuous occurrence of droughts (DOSM, 2011).

Common renewable energy strategies can at best off set fuel emissions of CO_2, but not able to reverse the climate change. One promising approach of reducing the concentration CO_2 in the atmosphere is the conversion of biomass to biochar and its utilization as soil amendment. The International Biochar Initiative

(IBI) defines biochar as "the solid material obtained from the carbonization of biomass that may be added to soils with the intention to improve soil functions and to reduce emissions from biomass that would otherwise naturally degrade to green house gases (GHG)" (IBI, 2013). Biochar help 'cleaned the air' in two ways. Firstly, biochar is produced from biomass, which would otherwise left to decay and thereby release harmful GHG such as CO_2 and methane (CH_4) into the atmosphere. Secondly, biochar as the soil application allowed the plants to safely store CO_2 they pull out of the air during photosynthesis, which enable the atmospheric C sequestration and reduce the C in the atmosphere (Ansari, 2009; Sedjo & Sohngen, 2012). Biochar can sequester its C content in soil for many years because it had higher stability to against decay compared to its raw biomass (Lehmann, Gaunt, & Rondon, 2006). Interestingly, the stability of a biochar was similar whether the biochar was at the age of few hundred years old or several thousand years old (Liang et al., 2008). This is due to the pyrolysis process during the biochar production that made the C content becomes fixed into a more stable form (Hunt, DuPonte, Sato, & Kawabata, 2010) and being recalcitrant in the biomass itself (Kwapinski et al., 2010). Moreover, using biochar as soil amendment will improve the crop productivity. Positive responses as a result of biochar applications have been reported by (Hoshi, 2001; Rondon, Lehmann, Ramírez, & Hurtado, 2007; Van Zwieten et al., 2007).

The utilization of biomass as the feedstock for biochar production via thermochemical conversion process is also a way to reduce the waste management problems. The biomass residues which are improperly managed and unsuitable for certain application could be used as pyrolysis feedstock (Gómez et al., 2016). Besides, it is also a way to avoid negative impacts on human health and environment which is caused by the open burning activities.

Pyrolysis is a process in which thermal degradation of the chemical constituent of the biomass occurs (Tripathi et al., 2016). Generally, pyrolysis process is classified into three categories: slow pyrolysis fast pyrolysis and flash pyrolysis. These categories are distinguished by its temperature range, heating rate and also the percentage of product yields (Balat, Balat, Kırtay, & Balat, 2009; Jahirul, Rasul, Chowdhury, & Ashwath, 2012). The proportion of pyrolysis products such as biochar, bio-oil and gas vary according to the type of pyrolysis employed. For high char yield, low temperature and low heating rate are preffered (Yaman, 2004). Slow pyrolysis is the suitable process for biochar production.

Previous studies reported that the properties of biochar are influenced by the characteristics of the feedstock (Shariff, Aziz, & Abdullah, 2014) as well as the pyrolysis conditions such as terminal temperature (Noor, Shariff, & Abdullah, 2012; Rahman, Abdullah, & Sulaiman, 2014), heating rate (Shariff, Noor, & Abdullah, 2012), residence time (Y. Wang, Hu, Zhao, Wang, & Xing, 2013) and carrier gas flow rate (Crombie & Mašek, 2015). Terminal temperature has been reported as the main factor that influence properties of the biochar significantly (Budai et al., 2014; Downie, Crosky, & Munroe, 2009).

Coconut palm is grown in more than 93 countries around the world. Coconut is one of the crops which produced vegetables oil besides soybean, sunflower, cottonseed, rapeseed and olive. In Malaysia, coconut is one of the oldest agro-based industries and the fourth important industrial crop after oil palm, rubber and paddy. Coconut is known as a tree of life and many products could be derived from coconut tree such as coconut oil,virgin coconut oil (VCO), cocopeat, coconut milk and other food product. The coconut leaves also can be used to make "ketupat" case, while the stiff mid-ribs of coconut leaves has been used to make brooms in Malaysia (Anem, 2014). According to Ministry of Agriculture and Agro-Based Industry Malaysia, MOA (2015), the total plantation area of coconut in Malaysia decreased from year 2007-2009 and year 2011- 2013. This reduction is due to the conversion of land utilization to the industrial crop such as oil palm as well as other development such as housing and industry (MOA, 2011). However, the total production of coconut shows the opposite trends from year 2009 to 2013 as it increased from 379,251 tonnes to 653,489 tonnes, and it is expected to continue to increase in the following years (MOA, 2015). Along with the increment of coconut production, higher percentages of residues generated from the coconut industry can be expected. The total production of coconut biomass excluding the coconut water is about 106,100 kilo tonnes, where 60.5% are unprocessed (Raghavan, 2010).The residues generated from coconut industries include coconut husk, coir fibre, coconut pith, coconut shell and coconut flesh waste were obtained after the extraction of coconut milk or coconut oil. Meanwhile, coconut frond and trunk are the common wastes which could be obtained from the coconut plantation. Large quantities of fronds are produced by natural pruning and silvicultural practice every year, and usually remain as waste in the forest floor (Njoku, Islam, Asif, & Hameed, 2014).

Biochars are not created to be equal. Presently, there is lack of understanding of the parameters that will affect the quality of the biochar being produced and insufficient data on the physical and chemical characteristics of biochar produced from coconut wastes especially coconut frond. The findings of this study will provide more understanding on the chemical and physical properties of coconut frond (CF) biochar produced at various temperature. The study of biochar characterization is necessary to better understand the effect of temperature on the properties of biochar produced from CF feedstock. Biochar with large surface area and porosity will provide higher capability of water and nutrient retention. Good quality biochar has the potential for soil application and consequently benefit the environment.

The objective of this work is to investigate the effect of pyrolysis temperature on biochar produced from coconut frond. Slow pyrolysis experiments were conducted and the temperatures were varied between 400°C and 600°C. The percentage of biochar yield produced at various temperatures were determined. The properties of the biochar were analyzed via proximate and elemental analysis, Field Emission Scanning Electron Microscope (FESEM), and BET surface area analysis.

MATERIAL AND METHOD

Sample Collection and Preliminary Analysis

The coconut fronds originate from Butterworth, Penang, Malaysia. The samples were collected and dried in the oven for 24 hour at 105°C. The coconut fronds were cut into smaller size, around 3-5 cm and stored in dessicators.

The CF feedstock was analyzed via proximate and elemental analysis, lignocellulosic determination and thermogravimetric (TG) analysis. The proximate analysis was carried out to determine moisture, ash content and volatile matter. The result of proximate analysis is expressed in dry basis which is represented by moisture-free weight percentage (mf wt%). The moisture, ash content and volatile matter were determined according to ASTM E871 (ASTM, 2006b), ASTM E1755-01 (ASTM, 2007) and ASTM E872 (ASTM, 2006a) respectively. The fixed carbon was calculated from equation (1).

Fixed carbon (mf wt%) =
100 – (ash content + volatile matter) (1)

Perkin Elmer 2400 analyzer was used to conduct the elemental analysis to determine the percentages of carbon, hydrogen, nitrogen and sulfur in the feedstock. The percentage of oxygen was determined from equation (2)

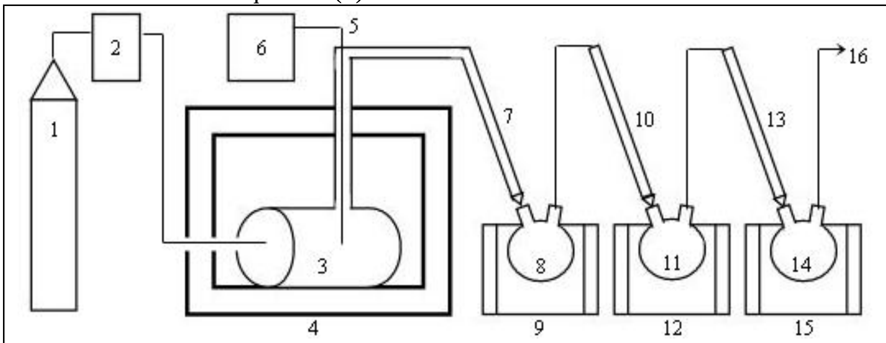
Oxygen (%) =
100 – (carbon + hydrogen + nitrogen + sulfur) (2)

The percentages of lignin, cellulose and hemicelluloses were also determined using ASTM methods (Li, 2004). Mettler Toledo TG analyzer was used to evaluate the thermal behavior of CF feedstock. The analysis was carried out at 10°C/min heating rate and under 100 ml/min nitrogen gas flow.

Pyrolysis Experiment

The pyrolysis experiment was carried out using laboratory-scale slow pyroysis system. This system consists of muffle furnace, sample holder (pyrolyzer), nitrogen gas system and condensing system. The diagram of the experimental setup is shown in Figure 1. The feedstock was tightly packed in the pyrolyzer and placed in the muffle furnace. The terminal temperature was varied between 400°C and 600°C while the heating rate, residence time and nitrogen flowrate were set at 5°C/min, 1 hour and 0.5L/min respectively. Biochar yield was calculated on weight basis according to Equation (3) below. The experiment was performed twice for each pyrolysis temperature and the average of biochar yield was determined.

Biochar Yield (wt %) =
(Mass of biochar (g) / Mass of feedstock (g)) x 100
(3)



Description:
1. Nitrogen tank
2. Flowmeter
3. Pyrolyzer
4. Muffle furnace
5. Thermocouple
6. Data logger
7, 10&13. Condenser
8, 11&14. Spherical flask
9, 12&15. Ice bath
16. Gas out

Figure 1 : Lab-scale slow pyrolysis system setup

Analysis of Biochar

The biochar derived from the slow pyrolysis of CF was analyzed via proximate and elemental analysis. The biochar was ground to powder form prior these analysis. Proximate analysis was carried out according to ASTM 1762 with some modification of temperature and heating period as suggested by McLaughlin (2010). The modification was done to provide much insight on how biochar actually partitions when applied as a soil amendment, hence the char is subjected to temperatures and conditions that are encountered in soils (McLaughlin, Anderson, Shields, & Reed, 2009).

Perkin Elmer 2400 analyzer was used to conduct the elemental analysis of biochar to determine the percentage of carbon, hydrogen, nitrogen and sulfur in the CF biochar. The percentage of oxygen was determined from Equation (2).

Field Emission Scnaning Electron Microscope (FESEM) FEI Nova NanoSEM 450 was used to examine the surface morphology of CF biochar. The voltage applied was set to 10 kV. The images of biochars were magnified at 500 times. The images of biochar also were compared with the FESEM image of the CF feedstock.

Micromeritics ASAP 2020 surface area and porosimetry analyzer was used to determine the BET surface area and pore volume of biochar. The BET surface area was calculated by the BET equation, while the micropore volume was obtained using the *t*-plot method.

RESULTS AND DISCUSSION

Preliminary Analysis

The result of the proximate, elemental and lignocellulosic analysis of the CF feedstock are listed in Table 1. From proximate analysis, it was found that the CF feedstock contains 75.27 mf wt% of volatile matter, 5.03 mf wt% of ash content and 19.70 mf wt% of fixed carbon. From elemental analysis, the percentages of carbon, hydrogen, sulfur and oxygen of the CF feedstock are 42.81%, 7.23%, 0.77% and 49.19% respectively. CF is suitable to be used as feedstock for slow pyrolysis experiments due to its high percentage of volatile matter and low percentage of sulfur. From the lignocellulosic analysis, it was found that CF feedstock comprised of 21.46% of cellulose, 39.05% of hemicelluloses and 22.49% of lignin. Clearly, hemicellulose is the main component in the CF structure.

Table 1: Properties of the CF feedstock

Analysis	Results
Proximate Analysis (mf wt%)	
Moisture content	6.76
Ash content	5.03
Volatile matter	75.27
Fixed carbon[a]	19.70
Elemental Analysis	
Carbon	42.81
Hydrogen	7.23
Nitrogen	BDL
Sulfur	0.77
Oxygen[a]	49.19
Lignocellulosic Analysis (%)	
Cellulose	21.46
Hemicellulose	39.05
Lignin	22.49

[a] : Calculated by difference
BDL: below instrumental detection limit

The thermal degradation behavior of the CF feedstock is shown by the thermogravimeteric (TG) and derivative thermogravimetric (DTG) curve in Figure 2. TG curve represents the fractional weight loss of the feedstock as a function of temperature, while the DTG curve is the plot of the rate of mass change, dM/dt versus temperature.

The TG curve in Figure 2 shows that the weight loss of the CF feedstock was prominent between 245°C and 350°C. The weight of CF sample decreased from 87% to 39% in that temperature range. Beyond 350°C, the weight of CF sample continue to reduce but the weight loss is insignificant.

For the DTG curve, a small peak was observed below 100°C. The formation of this small peak is due to the removal of water in the CF sample. Between 200°C and 380°C, a small hump and a high peak could be observed. The small hump appeared in the range of 200°C – 300°C, with the maximum weight loss rate 0.0392 wt%/min at 281°C. Meanwhile the high peak occur in the range of 300°C – 380°C., with the maximum weight loss rate 0.0772 wt%/min at 333°C. These hump and peak represent the degradation of lignocelluloses component. According to Yang et al. (2007), the decomposition of hemicelluloses occur between 220°C – 315°C while the cellulose decomposed between 315°C – 400°C. Therefore, it could be concluded that the formation of small hump is due to the degradation of hemicelluloses while the the appearance of the high peak represents the degradation of cellulose. The decomposition of lignin could not be observed clearly in the DTG curve as its degradation occurs over a wide range of temperature, from ambient temperature to 900°C (Yang et al., 2007).

The percentage of lignin in biomass will influence the percentage of char yield. Biomass with higher lignin content will produce higher char yield as lignin preferentially forms char during pyrolysis (Antal & Grønli, 2003). The percentage of CF biochar yield in this study is expected to be lower than those produced by other biomass feedstock containing higher lignin percentage such as olive husk, corn cob (Demirbas, 2004) and oil palm shells (Abnisa, Arami-Niya, Daud, & Sahu, 2013) as coconut frond used as the feedstock contained lower lignin percentage.

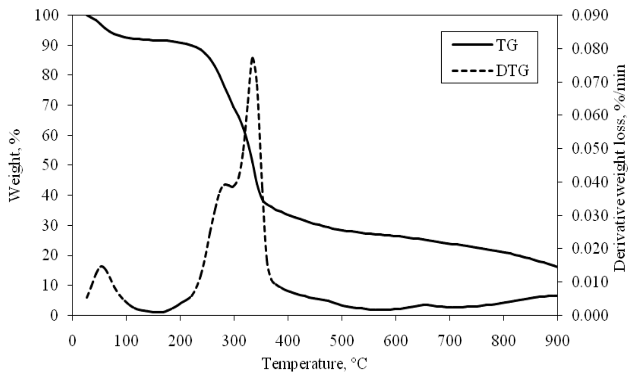


Figure 2: TG and DTG curves of CF feedstock

Biochar Yield From Pyrolysis Experiment
Biochar yield obtained from the slow pyrolysis experiments were calculated using Equation (3). The yield percentage of biochar produced at various temperature are presented in Figure 3.

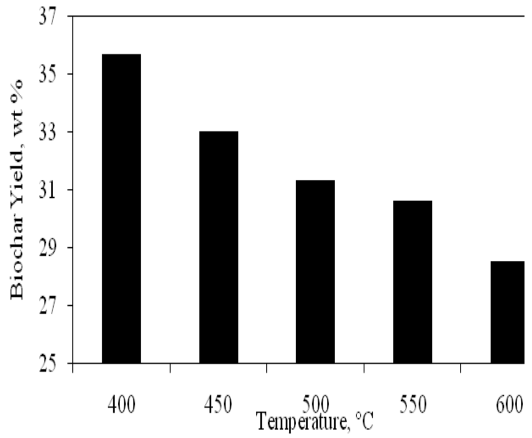


Figure 3 : CF Biochar Yield Produced at Various Temperature

From Figure 3, it could be observed that the yield percentage of the CF biochars decreased as the pyrolysis temperature increased. The highest biochar yield, 35.71 wt% was obtained from the 400°C pyrolysis experiment. The biochar yield decreased to 28.53 wt% as the pyrolysis temperature increased to 600°C. The decrement of biochar yield is associated with the reduction of volatile matter as shown in Table 2 and degradation of lignocellulosic component such as lignin in biochar as shown by the TG analysis results. The similar trend of decreasing biochar yield as the temperature was elevated also has been reported by Y. Lee et al. (2013), Al-Wabel, Al-Omran, El-Naggar, Nadeem, and Usman (2013) and Budai et al. (2014).

Analysis of Biochar
The result of proximate and elemental analysis of the CF biochar produced at different terminal temperature are shown in Table 2.

For the proximate analysis, the volatile matter of CF biochar decrease from 12.36 mf wt% to 4.90 mf wt%. The release of volatiles from breaking of weaker bridges and bonds in organic matrices caused the reduction of volatile matter in biochar as temperature increased (Pechyen, Atong, Aht-Ong, & Sricharoenchaikul, 2007). An increase in temperature also promoted the ash formation in biochar. Fixed carbon content of the CF biochar increased from 76.40 mf wt% to 78.38 mf wt%.

Table 2: Proximate and elemental analysis of CF biochar

Analysis	Biochar				
	400°C	450°C	500°C	550°C	600°C
Proximate Analysis (mf wt%)					
Moisture content	1.41	1.23	1.37	1.94	1.37
Volatile matter	12.36	10.78	8.57	6.53	4.90
Ash content	11.24	12.48	13.81	15.38	16.72
Fixed carbon[a]	76.40	76.74	77.62	78.09	78.38
Elemental Analysis (%)					
Carbon	31.87	36.24	45.32	48.36	58.34
Hydrogen	2.06	2.00	1.94	1.11	1.08
Nitrogen	9.72	8.27	6.89	10.29	9.10
Sulfur	BDL	BDL	BDL	BDL	BDL
Oxygen[a]	56.35	53.49	46.85	40.24	31.48
H/C	0.065	0.055	0.043	0.023	0.019
O/C	1.768	1.476	1.034	0.832	0.539

[a] : Calculated by difference
BDL: below instrumental detection limit

The elemental composition of biochar was also influenced by the pyrolysis temperature. The C content increase from 31.87% to 58.34% as the temperature increased from 400°C to 600°C. In contrast, H and O content of the CF biochar decreased with the increasing temperature. According to Novak et al. (2009), this happened because the feedstock loses surface functional –OH groups due to dehydaration, and structural core degradation which causes the loss of C-bound O and H atoms at the higher temperature. H/C and O/C ratios are also calculated and presented in Table 2. H/C and O/C ratios are useful indicators of the character of biochars such as aromacity (Mukome, Zhang, Silva, Six, & Parikh, 2013) and resistance to microbial degradation (Spokas et al., 2012). Result in Table 2 shows that H/C and O/C ratios are decreasing with increasing pyrolysis temperature. The reduction of H/C and O/C ratios are the result of dehydration and decarboxylation reactions respectively (Jindo, Mizumoto, Sawada, Sanchez-Monedero, & Sonoki, 2014).

The FESEM images of the CF feedstocks and biochar are shown in Figure 4. Significant differences could be observed between the images of the CF feedstock and CF biochars produced at 400°C, 500°C and 600°C. All the images were magnified at 500x. For the CF feedstock, the fibrous strands are compact. No pores could be observed at the wall of the strands. For the CF biochars, the pores appeared at the wall of the strands. The existence of pores on the biochar is important for microbial activity, retaining soil nutrients and improving the water holding capacity (Shaaban et al., 2014).

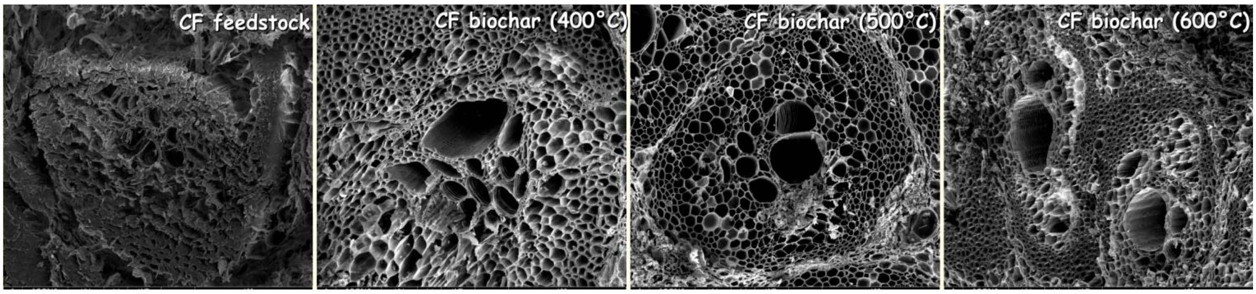


Figure 4: FESEM images of CF feedstock and biochars

Table 3: BET surface area and surface porosities of the CF biochar

Biochar	BET surface area (m^2/g)	Micropore Volume (cm^3/g)
CF Biochar 400°C	4.26	0.00477
CF Biochar 500°C	76.43	0.01590
CF Biochar 600°C	215.30	0.07912

In Table 3, result of BET surface area of CF biochar shows that increased from 4.26 m^2/g to 215.30 m^2/g as the pyrolysis temperature increased from 400°C to 600°C. Y. Lee et al. (2013) and X. Wang, Zhou, Liang, Song, and Zhang (2015) also observed a similar trend of BET surface area increment. A gradual increase of surface area is due to the formation of micropores on the biochar surface (Suliman et al., 2016) as a results of the removal of volatilized residual material that blocked micropores (J. W. Lee et al., 2010). This is exhibited by an increase of micropore volume as shown in Table 3. The micropore volume increased from 0.00477 cm^3/g to 0.07912 cm^3/g as the temperature elevated from 400°C to 600°C. Biochar with higher surface area is preferable for soil amendment application as it helps in improving the soil structure and increases the total water retention in the soils (Shaaban et al., 2014).

CONCLUSION

In this study, biochar was produced from coconut frond using a laboratory-scale slow pyrolysis setup. The pyrolysis temperature influenced the percentage yield of CF biochar and its properties. CF biochar yield decreased from 35.71wt% to 28.53 wt% while the fixed carbon content increased from 76.40 mf wt% to 78.38 mf wt% as the temperature increased from 400°C to 600°C. The BET surface area increased significantly from 4.26 m^2/g to 215.30 m^2/g due to enhancement of pore development which could be observed from FESEM images. The micropore volume also increased sixteen times from 0.00477 cm^3/g to 0.07912 cm^3/g. The biochar produced at 600°C has the lowest yield percentage of 28.53 wt%, but contained the highest fixed carbon and BET surface area which made it preferable for soil application.

Biochar is a potential tool to manage agricultural wastes, mitigate climate change and reduce food insecurity by improving soil fertility. Application of CF biochar with high surface area and fixed carbon content into soil has high potential to retain soil nutrients, improve soil quality, increase soil water retention and crop yield. The findings of this study could be one of the initiatives towards the sustainable development and climate change abatement. Further studies on biochar soil application versus crop yield may be conducted in the future.

ACKNOWLEDGEMENT

The authors wished to acknowledge the Ministry of Education Fundamanetal Research Grant Scheme (203/PFIZIK/6711410) and Universiti Sains Malaysia Short Term Grant (304/PFIZIK/6312102) for their financial support.

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