MJFAS MALAYSIAN JOURNAL OF FUNDAMENTAL AND APPLIED SCIENCES
PRINT ISSN: 2289-5981 | ONLINE ISSN: 2289-599X

RESEARCH ARTICLE

Characteristics of coconut frond as a potential feedstock for biochar via slow pyrolysis

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Article history
Submitted 2 February 2018
Revised 9 April 2018
Accepted 28 June 2018
Published Online 3 December 2018

Abstract

The aim of this study is to investigate the potential of coconut frond as a feedstock for biochar production via slow pyrolysis process. Proximate, elemental and thermogravimetric analysis were performed to evaluate the chemical and thermal properties of the coconut frond. The percentage of its lignocellulosic component and high heating value were determined. Surface morphology of coconut frond was examined using field emission scanning electron microscope (FESEM). Coconut frond (CF) contains 78.03±3.91 d.b. wt% of volatile matter, 4.96±0.07 d.b. wt% of ash content and 17.01±3.86 d.b. wt% of fixed carbon. Elemental analysis revealed a sulfur content of 0.94±0.12 %, while the percentage of nitrogen is 0.46±0.33%. The composition of carbon and hydrogen are 34.0±6.22 % and 7.71±0.34 % respectively. The high heating value of CF is 17.77±0.40 MJ/kg. CF consists of 43.91±1.80 % cellulose, 31.58±1.20 % hemicellulose, and 18.15±0.60 % lignin. From thermogravimetric (TG) analysis, it is apparent that the weight loss of CF occurred prominently in the temperature range 200°C - 400°C. The peaks of the DTG curve at 281.75±0.35 °C and 334.08±0.35°C indicate the weight loss of coconut frond sample due to the degradation of hemicellulose and cellulose, respectively. The FESEM images of CF show its fibrous strands are compact with a few large pores with diameters around 42.5 - 48.1 μm large pores in the center of the CF sample. The results of the analysis show that CF has a potential as a feedstock for biochar production via slow pyrolysis. CF also can be used in other application such as syngas and bio-oil production due to the low lignin percentage and high volatile percentage.

Keywords: Biomass, coconut frond, feedstock, properties

INTRODUCTION

Fossil fuel consumption has increased the concentration of carbon dioxide (CO_2) in the atmosphere. About 98% of carbon emissions result from fossil fuel combustion (Demirbas, 2005). The 12th annual Global Carbon Budget estimates the global carbon emissions from fossil fuels will rise significantly by 2% by the end of 2017 (Carrington, 2017). World Meteorological Organization reported that the global average concentration of CO_2 has reached 403 parts per million (ppm) in 2016, compared to 400 ppm in the previous year due to a strong El Nino event and the human activities such as deforestation and industrialisation (Watts, 2017; World Meteorological Organization, 2017). CO_2 is one of the greenhouse gases besides methane (CH_4) and nitrous oxide (N_2O), which cause global warming. The rise of sea level, an increase in the intensity of extreme weather and significant changes to the amount and pattern of precipitation are among the consequences of global warming. According to National Oceanic and Atmospheric Administration (NOAA), global sea level in 2014 was 2.6 inches above the highest annual average record since 1993 and it continues to rise at a rate of about one-eighth of an inch per year (NOAA, 2017). As the energy supplies are mainly based on the fossil fuel resources such as coal, oil and natural gas, the concerns on the depleting natural resources and the consequences of climate change and energy security should be fully addressed.

Renewable energy has been identified as the world's fastest-growing energy resource, increasing by an average 2.3% per year between 2015 and 2040 (US Department of Energy, 2017). Biomass is known as one of the major world renewable energy resources. Generally, biomass refers to forestry, purpose-grown agricultural crops, trees and plants, and organic, agricultural, agro-industrial and domestic wastes such as municipal and solid waste (Demirbas & Arin, 2002). Usually, biomass such as agricultural waste is not disposed of properly. In many countries, agricultural waste such as stalks, leaves, and husks are burned to reduce the residues from the agricultural activities (CEC, 2014).

The coconut tree, *Cocos nucifera* is a member of the palm family *Arecaceae*. It is grown for different purposes in various parts of the world. About 12 million ha of land are cultivated around the world with 60 million tonnes production reported for the year 2014 (FAO, 2017). Ninety percent of global coconut supply comes from Asia region (McAloon, 2017). Indonesia is the largest producer of coconut in the world with more than 18 million tonnes production, while Malaysia remains as one of the top 10 of producing countries in the world (Yon, 2016). The increment of coconut production and expansion of coconut plantation area will lead to the higher residues generation from the industry.

The residues generated from the coconut industry include husk, shell, frond, and residual coconut fiber, which could be obtained after the extraction of coconut milk. Meanwhile, coconut frond and trunk are the common residues generated at coconut plantations. Usually, coconut farmers dispose of the residues in the coconut plantation by burning or leaving it to rot in the fields. To some extent, burning can help to kill pest while in-situ composting can stimulate soil microbial activity and nutrient cycling. However, excessive burning activity is harmful to the environment. The burning process leads to air pollution and soil erosion while the accumulation of residues on the ground could cause a phytosanitary problem (Tomas, 2013).

According to a report by Raghavan (2010), the global total production of coconut biomass including the kernel but excluding the coconut water is about 106 million tonnes and 60.5% of them are unprocessed. In South East Asia and the Asia Pacific alone, the estimated annual waste is around 25 million tons (DP CleanTeach, 2017). Hence, the utilization of biomass especially coconut wastes as feedstock for chemicals and energy products could reduce the abundance of agricultural waste which usually lead to waste management problems.

The production and application of biochar have gained much attention due to its promising benefits and function in addressing the environmental problems. It is has been promoted as a tool for climate change mitigation because, in soil application, it is expected to sequester carbon for thousand years and help in reducing greenhouse gases emission from soils (Brassard et al., 2016).

Biochar is a solid material obtained from thermochemical conversion of biomass in an oxygen-limited environment. According to Lehmann & Joseph (2015), it can be used for a range of applications such as an agent for soil enhancement, improved resource use efficiency, remediation or protection against particular environmental pollution and as an avenue for greenhouse gases (GHG) mitigation. Works of literature reported that biochar has potentials to be applied as a soil amendment (Khalifa & Yousef, 2015) and activated carbon (Ahiduzzaman & Sadrul Islam, 2016). High carbon content and good adsorption capacity are the important properties in soil application and carbon sequestration. Meanwhile, high carbon content and high surface area are necessary for the utilization of biochar as activated carbon (Mahmood et al., 2015).

There have been few papers that studied the utilization of coconut frond as feedstock. Coconut fronds have been used to produce activated carbon for the adsorption of carbofuran insecticide (Njoku et al., 2014). However, the properties of CF feedstock was not reported. Sharif et al. (2016) studied the properties of coconut fronds (CF) and coconut husk (CH) feedstocks and the effect of slow pyrolysis temperature on biochar yield. It was reported that CF is a suitable feedstock for slow pyrolysis. They also found that the percentage yield of CF biochar was lower compared to biochar yield of CH, due to lower lignin composition.

The properties of biochar are greatly influenced by the characteristics of feedstock used. The data from the preliminary analysis of feedstock such as proximate, elemental and thermogravimetric analysis will provide useful information on feedstock behaviour during thermochemical conversion. The data also can be used in the prediction of biochar properties. For example, the feedstock with high carbon and lignin content will produce a high yield of biochar with high carbon content. Therefore, the objective of this paper is to study the properties of coconut fronds as a potential feedstock for biochar production. The understanding of the properties of feedstock will help in enhancing the quality of biochar produced

EXPERIMENTAL

Materials

Coconut frond samples were collected from a plantation in Butterworth, Pulau Pinang. The samples were collected from the fallen CF, which were found under the coconut trees in the plantation. Then, the fronds were cut into smaller pieces as shown in Figure 1. The CF was then dried in a conventional oven (Venticell 222-Standard) at 105°C until the moisture content was below 10 wt% to avoid the growth of fungi or mould. The CF was ground to pass a 600μm sieve and then stored in an airtight container prior to analysis.

Proximate analysis was carried out in accordance with ASTM E871 (ASTM, 2006a), ASTM E872 (ASTM, 2006b) and ASTM E1755-01 (ASTM, 2007) for moisture content (MC), volatile matter (VM) and ash content (AC) respectively. Fixed carbon was determined from the calculation as shown in Equation (1). The results of the proximate analysis were expressed on a dry basis (d.b. wt%).

$$FC\ (d.b.\ wt\ \%) = 100 - VM - AC \quad (1)$$

Elemental analysis was performed to determine the percentage of carbon (C), hydrogen (H), nitrogen (N) and sulfur (S) in CF using an elemental analyzer (Perkin Elmer 2400). The percentage of oxygen composition in CF is obtained from equation (2):

$$Oxygen\ (\%) = 100 - C - H - N - S - AC \quad (2)$$

Lignin and α-cellulose content of CF were determined according to ASTM D1106-96 (ASTM, 2013a) and ASTM D1103-60, (ASTM, 1977) respectively. The percentage of holocellulose was determined according to ASTM D1104-56 (ASTM, 1978). Then, the percentage of hemicellulose was determined from the difference between holocellulose and cellulose percentages (Li, 2004). Each analysis was performed in triplicate.

Fig. 1 Coconut frond sample.

The thermal study was done on the coconut frond sample by performing thermogravimetric (TG) analysis using a Mettler Toledo TG Analyzer. Five mg of ground CF sample was weighed and placed into the sample pan and heated from 30°C to 900°C at a heating rate of 10°C/min under a continuous nitrogen flow of 30 ml/min. This analysis was done in duplicate.

The higher heating value (HHV) of CF sample was determined using a bomb calorimeter (IKA C200). This analysis was carried out by placing 0.5 g of ground CF into a crucible and subsequently transferred inside a stainless steel container. The decomposition vessel was filled with 30 bar of oxygen and ignited through a cotton thread connected to a solid ignition wire inside the decomposition vessel and burned. This bomb calorimeter is validated in accordance with ASTM D5865-13 (ASTM, 2013b). This analysis was also done in duplicate.

The surface morphology of CF was examined using a field emission scanning electron microscope (FESEM) from FEI Nova NanoSEM 450. Approximately 7.5 mg of CF were attached to the stub and then placed on the stub holder inside the specimen chamber. The FESEM was operated at 10kV. The image of the sample was displayed on the connecting monitor.

RESULTS AND DISCUSSION

Table 1 shows the result of proximate analysis of the CF. It contains 7.08 d.b. wt% of moisture, 78.03 d.b. wt% of VM, an AC of 4.96 d.b. wt% and 17.01 d.b. wt% of FC. The high VM content in the CF is needed for autothermal pyrolytic conversion of CF to biochar (Rahman et al., 2004). The ash content of CF in this study is higher compared to the other coconut residues such as coconut shell and coconut husk that contain about 0.7 d.b. wt% and 0.33 d.b. wt% of AC respectively (Tsamba et al., 2006; Shariff et al., 2016). The high AC of CF might be due to the frond being on the ground before collection.

Table 1 Proximate analysis of CF and other biomass.

	Reference	MC	VM	AC	FC
CF (d.b. wt%)	This study	7.08± 0.35	78.03 ± 3.91	4.96 ±0.07	17.01 ±3.86
CF (d.b. wt%)	(Shariff et al., 2016)	0.37	89.96	4.67	5.37
OPF (d.b. wt%)	(Mahmood et al., 2015)	6.00	80.90	1.10	18.10

In Table 1, the results of the proximate analysis are also compared with existing literature of CF feedstock (Shariff et al., 2016) and oil palm frond (OPF) (Mahmood et al., 2015) to gain a better understanding on the feedstock properties. The properties of CF in this study were found to be similar to OPF in terms of the VM and FC compositions. Meanwhile, the AC of OPF was found to be lower than CF. This could be due to the condition of OPF and CF during the sample collection. The sample in this study was collected from the fallen CF on the ground. The AC of CF in this study is almost similar to the CF feedstock reported by Shariff et al. (2016). However, it could be observed that the percentage of VM in this study is lower than reported by Shariff et al. (2016). This could be due to the species variations and cultivation conditions of the samples.

From the proximate analysis of biomass feedstock, we could estimate the percentage of AC, FC, and VM of biochar. Previous studies (Noor et al., 2012; Shariff et al., 2014; Kabir et al., 2017) reported that FC and AC of derived biochar would be higher compared to the percentage in the feedstock. Meanwhile, the percentage of VM of biochar would be lower due to the release of volatiles from the breaking of weaker bridges and bonds in organic matrices (Pechyen et al., 2007). Therefore, it could be expected that the biochar derived from feedstock with higher FC and AC percentage will have a higher composition of AC and FC. However, the biochar yield percentage could not be estimated from the data of proximate analysis solely as it also depends on the percentage of lignocellulose composition.

The properties of the feedstock in Table 1 are plotted in Figure 2 presents the mean proximate composition of various biomass and solid fuels reported by Vassilev et al. (2010). The similarities among different groups of biomass feedstock could be observed clearly in this triangle. The properties of CF in this study have intermediate positions between woody biomass and herbaceous-agricultural biomass.

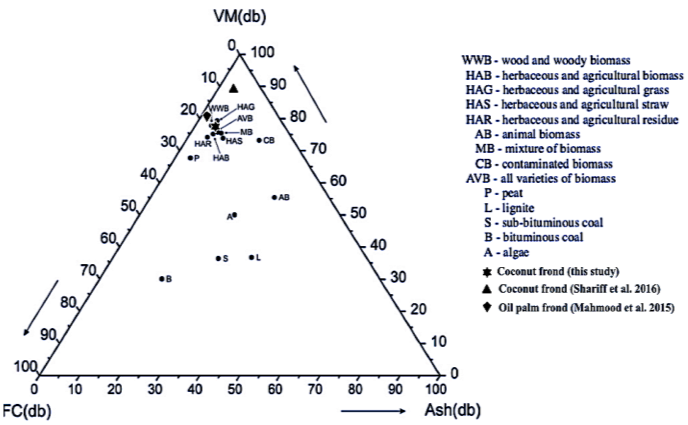

Fig. 2 Mean proximate composition of various biomass and solid fuels. Adapted from Vassilev (2010).

Elemental composition of CF is presented in Table 2 in dry ash-free basis. Coconut fronds contain 34.01% of carbon, 7.71% of hydrogen and 51.92% of oxygen. It can be observed that CF contains a very small amount of sulfur and nitrogen, 0.94% and 0.46% respectively. The low percentage of sulfur and nitrogen in CF will make it safe to be used as feedstock for slow pyrolysis process.

CF contains 31.58% of hemicellulose, 43.91% of cellulose and 18.15% of lignin. Cellulose is identified as the major component of CF. Cellulose degrades to a more stable anhydrocellulose, which produce higher biochar yield and is the dominant reaction at a temperature less than 302°C (575K) (Shafizadeh, 1985). Meanwhile, at a higher temperature, cellulose depolymerizes and produces volatiles (Demirbas, 2006). Lignin composition in the CF also contributes to the formation of char. In contrast, lignin slowly degrades over a wide range of temperature (Lee et al., 2013). Compared to other biomass such as coconut shell and OPF (Table 2), it can be observed that the percentage of lignin of CF is the lowest. Therefore, CF feedstock is also recommended for the production of syngas and bio-oil.

The lignocellulosic component plays significant roles in the determination of biomass behaviour during slow pyrolysis and the yield percentage of the products; biochar, bio-oil and syngas. According to Qu et al. (2011), the pyrolysis product distribution is possible to be predicted according to the lignocelllulosic component proportion in a biomass as these three components have very different thermal behaviours. For example, biomass feedstock with high lignin content usually will produce higher char yield since lignin is preferentially converted to char during pyrolysis (Sun et al., 2012).

However, the percentage of lignin composition is not the only influencing factor (Gómez et al., 2016). Previous studies (Eom et al., 2011; Qu et al., 2011; Shariff et al., 2014) reported that the percentage of inorganic matter such as ash also influences the percentage of biochar yield. Generally, feedstock with higher ash percentage will produce higher biochar yield. High ash in the feedstock acts as a catalyst to promotes secondary reactions of primary pyrolysis products, which further degrade to secondary tars, char and gases (Abdullah & Bridgwater, 2006; Ronsse, 2016).

Table 2 Properties of CF and other biomass.

Sample	Coconut Frond	Coconut Frond	Coconut Shell	Oil Palm Frond
Reference	This study	(Shariff et al., 2016)	(Cagnon et al., 2009)	(Kabir et al., 2017)
Elemental composition (daf %)				
Carbon	34.01±6.22	42.81	48.70	41.0
Hydrogen	7.71±0.34	7.23	5.80	6.74
Nitrogen	0.46±0.33	0.00	0.30	0.67
Sulfur	0.94±0.12	0.77	0.30	0.35
Oxygen	51.92±5.44	44.52	42.2	45.37
Empirical formula	$CH_{2.70}N_{0.01}O_{1.15}$	$CH_{2.01}N_{0.0.0}O_{0.86}$	$CH_{1.42}N_{0.01}O_{0.62}$	$CH_{1.96}N_{0.01}O_{0.83}$
Ligno-cellulosic composition (%)				
Hemicellulose	31.58±1.20	22.49	35.00	19.22
Cellulose	43.91±1.80	39.05	15.00	45.22
Lignin	18.15±0.60	21.46	50.00	31.24
HHV (MJ/kg)	17.77±0.40	n/a	n/a	16.0
HHV (MJ/kg)*	19.0	n/a	n/a	17.63

n/a : not available
*predicted using equation from Parikh et al. (2005)

The properties of the CF used in this study are also compared with the properties of CF, coconut shell (CS) and OPF reported in the literature as shown in Table 2. CF was found to have lower lignin content compared to coconut shell and OPF. As lignin contribution is higher than cellulose and hemicellulose contribution in the generation of char (Gupta et al., 2016), it could be expected that the char yield of CF will be lower than that of coconut shell and OPF under same pyrolysis conditions.

The result of higher heating value (HHV) of CF feedstock is also presented in Table 2. The higher heating value (HHV) of the CF is 17.77 MJ/kg. It is higher than the HHV of OPF reported by Kabir et al. (2017). The HHV value also could be predicted using equation

HHV = 0.3536FC + 0.1559VM – 0.0078AC as reported by Parikh et al. (2005). The predicted values of HHV are also presented in Table 2 and agree reasonably well with experimental data. The characterization of the heating value of biomass feedstock is necessary for energy analysis and numerical simulations of the thermochemical conversion system (Sheng & Azevedo, 2005; Nhuchhen & Abdul Salam, 2012).

It could be observed that the elemental properties of CF are almost similar to OPF compared to the other coconut wastes. In terms of ligno-cellulosic composition, it could be observed that cellulose is the main component for both CF and OPF. CF has lower carbon composition compared to coconut shell and OPF. It is also lower than the percentage of carbon reported by Shariff et al. (2016). This could be due to few factors such as the variations of species, geographic origin and fertilizer treatment (Jones et al., 2014). CF contains higher hydrogen and oxygen composition compared to other biomass in Table 2. A van Krevelen diagram (plot of H/C versus O/C atomic ratios in daf basis) of CF feedstock and other biomass is presented in Fig. 3. It was observed that the CF feedstock was not fallen within the cluster of herbaceous crops, which represented by mischantus gigantus and switchgrass. The ratios CF feedstock also did not fall near the OPF, CF and cellulose. The low percentage of C of CF feedstock found in this study could be one of the reasons of the deviation of H/C and O/C ratios of CF compared to the ratios reported from the works of literature.

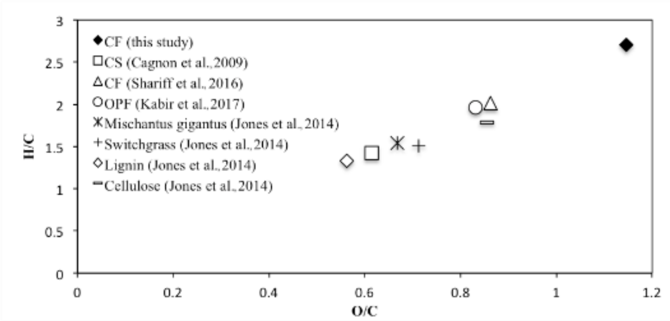

Fig. 3 Van Krevelen plot of CF, lignin, cellulose and other biomass.

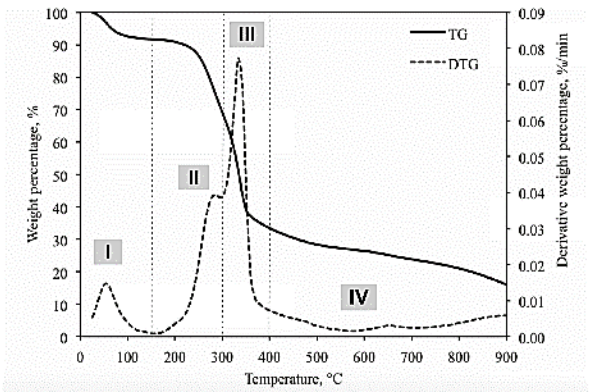

Fig. 4 TG and DTG curves of coconut frond sample.

The result of TG analysis of CF is shown in Figure 4. The TG curve indicates the fractional weight loss of feedstock as a function of temperature. The derivative TG (DTG) curve presented the rate of weight change versus temperature. The result from TG analysis could be used to detail the main steps of biomass conversion (Collard & Blin, 2014).

The weight loss of the feedstock sample at a temperature below 100°C was found to be 7.32 wt% and attributed to moisture loss. A similar MC was obtained using the conventional method (Table 1). However, the weight within 100°C to 200°C changed only by 1.6%. The weight loss of CF is prominent within the temperature range 200°C to 400°C amounting to about 60%. Weight loss of CF decreases gradually beyond 400°C. According to the TG plot in Figure 4, it is estimated that around 17 – 33 wt.% of char yield can be achieved by the pyrolysis of CF at a temperature beyond 400°C. Fan et al. (2017) achieved higher char yield from the coconut shell sample, around 20-40 wt% due to higher lignin content of coconut shell sample.

In Figure 4, it can be clearly observed that the peaks of the DTG curve exist at three different temperatures. To discuss the relationship between peaks on the DTG curve and the weight loss, the thermal degradation of CF has been divided into four regions, I-IV as shown in Figure 3. Region I is for the temperature up to 150°C accounting for the removal of moisture from CF. Region II is for the temperature range 150°C - 300°C. A distinct peak could be observed at 281°C indicating the degradation of hemicellulose and certain lignin components in the CF. According to Yang et al. (2004), the degradation of hemicellulose occurs within the temperature range 220°C - 300°C. Region III shows a large peak at 333°C. The formation of this peak could be explained by the degradation of cellulose and partial lignin. Cellulose component in biomass usually degrades within the temperature range 300°C-340°C, while the degradation of lignin starts at around 340°C (Yang et al., 2004). No DTG peak could be observed in region IV. Beyond 400°C, no high peak could be observed and both TG and DTG curves are gradual. This is due to the degradation of lignin, which is distributed along a wide range of temperature, and its peak is not commonly distinguishable (Li et al. 2008). In addition, weight loss in this region could be attributed to in-char structure rearrangement besides the lignin decomposition with an occasional transformation of ash (Fan et al., 2017).

The data from the TG analysis also can be used to determine the minimum temperature for the pyrolysis process of the feedstock. From Figure 4, it can be observed that the weight loss of CF gradually decreases after 400°C and no significant peaks could be observed beyond this temperature. Therefore, 400 °C can be determined as minimum pyrolysis temperature for CF feedstock to produce biochar.

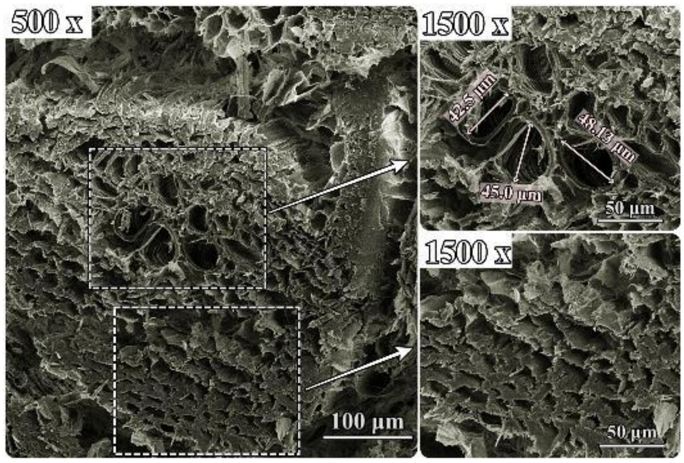

Fig. 5 FESEM image of coconut frond sample.

Figure 5 shows the FESEM image of CF. At 500x magnification, the cross section of CF shows that it consists of fibrous strands. It can be observed that most of the fibrous strands of CF are compact. As the image was magnified by 1500x, it can be clearly observed that the fibrous strands in the bottom part are more compact compared to the middle part of CF sample where a few pores could be observed. The diameters of the pores in the middle part of CF raw feedstock are around 42.5 – 48.1 μm. These pores are larger than the diameter of clay, root hair and fungi, which has a diameter around 2-5 μm, 2-10 μm and 15-17 μm respectively. The pores could be developed and further enhanced by the chemical activation during the course of pyrolysis as reported by Tsai et al. (2001). The pore size could be increased by several thousand folds depends on the pyrolysis temperature as well as the properties of feedstock (Thies et al., 2015).

In soil application, the porous structure of biochar could provide a habitat for microbes to colonize, grow and reproduce, particularly once biochar is aged in soil (Thies et al., 2015). However, the biochar also could provide a habitat for bacteria or toxins harmful to the plant, which will result in a negative outcome of biochar application (Howard, 2011). The overall effect of biochar as soil enhancer may be influenced by the type of microbes in the soil, type of soil or sand as well as the kind of plant used.

CONCLUSION

Previous studies have reported the production of biochar from different types of biomass such as empty fruit bunches (Shariff et al., 2014), oil palm frond (Kabir et al.,, 2017), rice husk (Gupta et al., 2016) and cassava wastes (Noor et al. 2012). The properties of CF are revealed in this study. It was shown that CF has a high potential as a feedstock for biochar production via a thermochemical process such as slow pyrolysis. CF contains 78.03 d.b. wt% volatile matter. The ash and fixed carbon contents of CF are 4.96 d.b. wt% and 17.01 d.b. wt% respectively. CF is an environmental friendly feedstock for slow pyrolysis due to its very low percentage of nitrogen and sulfur contents. CF contains 34.01% carbon, 7.71% hydrogen and 51.92% oxygen. The high heating value of CF is 17.77 MJ/kg. CF consists of 43.91% cellulose, 31.58% hemicellulose and 18.15% lignin. The weight loss of coconut frond sample was found to be prominent between 200°C and 400°C. The DTG peaks of TG analysis at 281°C and 333°C indicate the degradation of hemicellulose and cellulose respectively. FESEM images show that the fibrous strands are compact and a few pores can be seen in the center of the CF sample. It is evident from the properties of CF that CF is a suitable feedstock that can be used for producing biochar via slow pyrolysis.

ACKNOWLEDGEMENT

The authors wish to express their gratitude to the Universiti Sains Malaysia for the technical support and Ministry of Higher Education (MOHE) Malaysia for financing this research through the Fundamental Grant Research Scheme (FRGS: 203/PFIZIK/6711410).

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