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Slow Pyrolysis of Cassava Wastes for Biochar Production and Characterization

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Abstract: Production of biochar from slow pyrolysis of biomass is a promising carbon negative procedure since it removes the net carbon dioxide in the atmosphere and produce recalcitrant carbon suitable for sequestration in soil. Biochar production can vary significantly with the pyrolysis parameter. This study investigated the impact of temperature and heating rate on the yield and properties of biochar derived from cassava plantations residues which are cassava stem (CS) and cassava rhizome (CR). The pyrolysis temperatures ranged from 400°C to 600°C while the heating rate parameter was varied from 5°C/min to 25°C/min. The experiment was conducted using the lab scale slow pyrolysis system. The increment of temperature and heating rate of slow pyrolysis for both cassava wastes had raised the fixed carbon content of the biochar but decreased the biochar yield. More biochar was produced at lower temperature and lower heating rate. Temperature gave more influence on the biochar yield as compared to the heating rate parameter. The highest biochar yield of more than 35 mf wt. % can be obtained from both CS and CR at 400°C and heating rate of 5°C/min. From the proximate analysis, the results showed that cassava wastes contain high percentage of volatile matter which is more than 80 mf wt. %. Meanwhile, the biochar produced from cassava wastes contain high percentage of fixed carbon which is about 5−8 times higher than their raw samples. This suggested that, it is a good step to convert CS and CR into high carbon biochar via slow pyrolysis process that can substantially yield more biochar, up to 37 mf wt. % in this study. Since the fixed carbon content for both CS and CR biochar produced in any studied parameter were found to be more than 75 mf wt. %, it is suggested that biochar from cassava wastes is suitable for carbon sequestration.

Key words: Biochar; Biomass; Cassava rhizome; Cassava stem; Pyrolysis.

INTRODUCTION

Cassava (*Manihot esculenta*) is mainly grown for its starchy tuberous roots. It is the third largest source of carbohydrates for human consumption in the world with an estimated annual world production of 208 million tonnes [1]. Cassava harvest can take place most of the year and the soils used for the planting are usually low in fertility and there is a frequent need to apply fertilizers or organic manures [2]. These properties make the cassava tubers as the most suitable feedstock for the bio-ethanol production [1, 3]. Malaysia planted cassava mainly for starch extraction particularly for making monosodium glutamate that is using about 3,000 tonnes of starch per month [4]. The abundance of cassava wastes, such as the stem and rhizome parts, which are not edible for human are kept aside on the cassava field. The cassava wastes can be directly used for energy production via direct combustion process. However, there are about 50% of the carbon in the biomass agricultural wastes that can be lost upon burning [5]. The cassava wastes can be converted into biochar and applied to soil. This approach can be made to clear the cassava field and at the same time preserve the carbon content.

There were studies done on bio-oil production using the cassava wastes, but there was no report on the properties of biochar produced [6, 7]. In Malaysia, many researches were concentrating on the application of oil palm wastes towards the bioenergy production [8–10]. So, in order to maximize the biomass utilization in Malaysia especially on the agricultural wastes, in this work we are using the cassava wastes for biochar production and characterization.

Biochar is the carbon-rich product obtained when biomass is heated in a closed container with restricted oxygen. It is different from charcoal since biochar is applied to soil to improve soil properties, while charcoal is mainly used as fuel for heating process [11]. Biochar is high in surface area and has negative surface charge and charge density [12]. These properties increase the capacity of the biochar to hold nutrients and became more stable than most fertilizer or other organic matter in soil [11]. As a result, it makes the soil more fertile and causes the crops to grow faster.

Biochar can sequester carbon (C) in the soil for hundreds to thousands of years because pyrolysis process made C to become recalcitrant in the biomass itself.

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Besides, during the production process, biochar is able to scrub carbon dioxide, nitrous oxides and sulphur dioxide from the flue gas thus decrease those green house gases (GHG) emissions to the air [12]. Thus, the biochar production has received considerable interest as a potential tool to slow the global warming [13]. However, the properties of biochar are varied by the production parameter and choice of feedstock. Understanding of biochar properties would be beneficial to identify their appropriate applications and for upgrading them.

Pyrolysis offers a great opportunity from an environmental point of view. It allows the use of a wide variety of materials as the feedstock and produces low emission GHG, compared to the technologies that are used in the process of incinerator [14]. The condition of pyrolysis process can be optimized to maximize the production of the liquid, solid or gas product. Biochar solid product can be optimized using the slow pyrolysis conversion process [15–17]. Fast pyrolysis generates more liquid product which is bio-oil and its residence time is just in seconds compared to slow pyrolysis process that take hours [15].

The temperature and heating rates are two of the pyrolysis parameters that affect the yield and composition of the pyrolysis products [14, 18]. The impacts of these two parameters were studied on the biochar yield and its composition upon slow pyrolysis of cassava wastes. The temperature range of this study is from 400℃ to 600℃ with fixed heating rate at 5℃/min. Meanwhile, the range of heating rates for this study is from 5℃/min to 25℃/min at pyrolysis temperature of 400℃.

SAMPLES AND METHODS

Biomass samples: The agricultural wastes from the cassava plantation, i.e. cassava stem (CS) and cassava rhizome (CR) obtained from Sungai Bakap, Penang, Malaysia, were used as the feedstock in this study. CS and CR are the leftover on the plantation, after the edible part of the cassava trees mainly the starchy tuberous roots had been collected. For the sample pre-treatment, CS samples of 1.5−2 cm diameter were cut into pellets size about 3−4 cm length, while the CR samples were cut into 4 x 4 cm sizes and about 3 cm thick. Then, the entire samples undergo the pre-drying treatment in order to obtain moisture free samples. Wet feedstock will cause low efficiency of heating process during pyrolysis, which is related to the volatization of moisture [10]. So, pre-drying treatment will give off non-flammable component such as carbon dioxide and water. This pre-treatment was conducted in the conventional oven (Venticell 222-Standard) at a temperature of 105℃ and continued until the weight of sample remained constant.

Characterizations of biomass samples are important in order to identify their suitability to undergo the thermochemical conversion process. Biomass with high volatile matter content, low ash and sulphur content are some of the main criterion for pyrolysis feedstock [19]. The proximate analysis was done on the CS and CR sample for the determination of moisture, volatile and ash content, according to the ASTM International E1756-01, E872-82 and E1755-01 respectively. The average results from the proximate were presented in weight percentage on moisture free basis (mf wt. %). The fixed carbon content was calculated using the Eq. 1 as shown below:

$$FC \, (mf \, wt. \, \%) = 100 - [VM + AC] \, (mf \, wt. \, \%) \quad (1)$$

where FC is the fixed carbon content, VM is the volatile matter and AC is the ash content.

By using an elemental analyzer (Perkin Elmer 2400) the ultimate analysis was done to directly determine the mf wt. % of carbon (C), hydrogen (H), nitrogen (N) and sulphur (S) contents of the feedstock. Meanwhile the oxygen (O) content was calculated according to Eq. 2.

$$O \, (mf \, wt. \, \%) = 100 - [C + H + N + S] \, (mf \, wt. \, \%) \quad (2)$$

The calorific value of a raw feedstock induces its energy quality. A sample that contains high calorific value would produce more heat energy that facilitates the pyrolysis process [19]. The grounded CS and CR samples weighing about 0.5−0.7 g each was burned in a commercial Parr adiabatic bomb calorimeter to determine their calorific values. The procedure was carried out according to the ASTM D 2015 standard test method.

Pyrolysis Experiment: The slow pyrolysis experiments were carried out in the lab scale slow pyrolysis system as shown in Fig. 1(a) and Fig. 1(b). The stainless steel tube or pyrolyzer was externally heated in the electrical muffle furnace (Thermolyne F62700).

During the pyrolysis process, the emissions of product from the pyrolyzer were led out through an exit pipe to the first water-cooled condenser that was attached to the first ice-cooled spherical flask and further condensed in second water-cooled condenser, with second ice-cooled spherical flask attached. The incondensable gases were then allowed to escape out from the laboratory through the fume cupboard. Once the experiment reaches the terminal temperature, it was maintained for an hour until no further significant release of gas was observed.

The quantity of biochar produced was determined by weighing the pyrolyzer after each pyrolysis run. For each varied temperature and heating rates, the average biochar yields from three pyrolysis runs were presented. Biochar yield was calculated using Eq. 3 and expressed in weight percentage on moisture free basis (mf wt. %).

$$Biochar \, yield = \frac{Weight \, of \, biochar}{Weight \, of \, moisture \, free} x100 \quad (3)$$
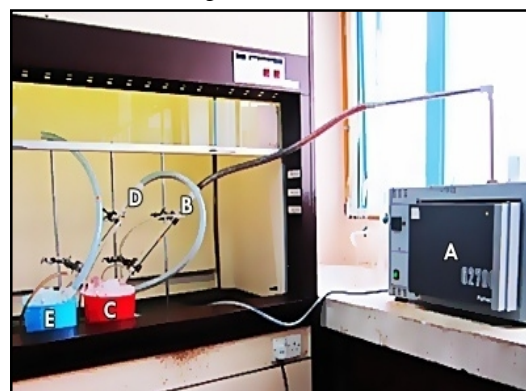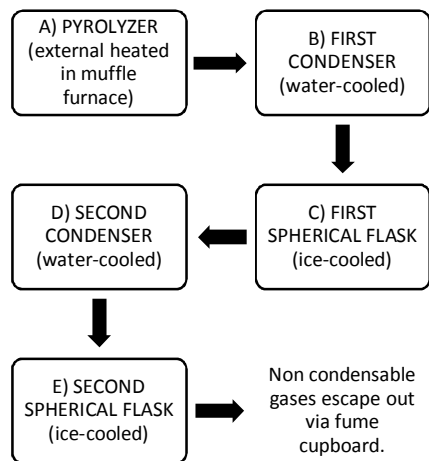

Fig. 1 a): Experimental set-up

Fig. 1 b): Flow chart of lab scale slow pyrolysis process

The biochar product were kept neat within the sealed plastic container and placed inside the dessicator. It is important to make sure that the biochar was always in dry environment to avoid them from absorbing the moisture from the surrounding, due to its highly porous property.

Biochar characterization: Prior to be used as asoil amendment, the biochar was characterized using the proximate analysis. Information from the proximate analysis of biochar especially on the amount of volatile matter and the fixed carbon content are appropriate to evaluate the general stability of biochar in the soil [20].

The analysis was done according to the ASTM D1762-84 Standard Method for Chemical Analysis of Wood Charcoal with some modification especially on the analysis temperature range, since biochar is not destined to be used as a fuel source. The oven temperature for moisture analysis was raised up to 200°C instead of typically overnight in a drying oven at 105°C because most biochars are hydroscopic and exhibit significant adsorption capacity for water vapour. So, to remove the adsorbed water, higher drying temperatures are appropriate. For determination of weight percentage of ash, the proximate analysis temperature of the muffle furnace was lowered to 500-550°C instead of 750°C. Meanwhile, to determine the percentage of volatile matter, the samples were heated in a muffle furnace at 450°C which is much lower than that stated in ASTM D1762-84 standard test method [21]. The weight percentage of fixed carbon content or the 'recalcitrant matter' of the biochar was calculated using Eq. 1.

RESULT AND DISCUSSION

Characterization of biomass samples: The chemical analysis results of the CS and CR are presented in Table 1. As can be seen from proximate analysis, both

of the cassava wastes contained about similar moisture content and volatile matter, but they have a big difference in ash and fixed carbon content. CR has a higher ash content which was contributed from the soil that was attached to it, since CR is the underground part of a cassava tree. The high volatile content and the relatively high calorific values in both CS and CR suggested that cassava wastes are good sources of feedstock for the thermochemical conversion process such as pyrolysis.

From the ultimate analysis, the nitrogen and sulphur content for both CS and CR are quite low which is less than 1 mf wt. %. This indicated that they are rather environmental friendly since if they were burnt as the feedstock for biochar production, it only will gives off low rates of nitrogen oxide and sulphur oxide.

Table 1: The properties of cassava wastes

	CS	CR
Proximate analysis (mf wt. %)		
Moisture	2.08	3.53
Volatiles	81.51	83.64
Ash	2.42	7.28
Fixed Carbon	16.07	9.08
Ultimate analysis (mf wt. %)		
Carbon	44.47	41.78
Hydrogen	5.82	5.97
Nitrogen	<0.01	0.26
Sulphur	0.83	0.92
Oxygen	48.88	51.07
Molecular formula	$CH_{1.56}O_{0.83}$	$CH_{1.70}O_{0.92}$
Calorific values (MJ/kg)	18.39	18.01

Effects of Temperature and Heating Rates on the Biochar Yield: Fig. 2 presents the biochar yield from CS and CR, as a function of pyrolysis temperature. As expected, the biochar yield from both cassava wastes decreased when the pyrolysis temperature increased. A similar trend was also observed in the open literature on the pyrolysis of various lignocellulosic biomass under similar conditions [10, 18, 22–24]. This could be due to greater primary decomposition of the biomass samples or through the further decomposition of the biochar itself as the pyrolysis temperature was high [25]. The weight percentage of biochar yield for CS was decreased by 27.13% (from 35.86 to 26.13 mf wt. %) as the temperature was raised from 400°C to 600°C. Meanwhile, biochar yield for CR decreased by 19.17% (from 36.98 to 29.89 mf wt. %) at the same conditions. Lower temperatures should be chosen to produce a high yield of biochar.

CR that has higher ash content (refer to Table 1) produced higher biochar yield compared to CS, for each studied temperature. The existence of alkali metal especially potassium and sodium from soil on the CR sample, known to be catalytically active and favors the secondary reactions, lead to a reduction in bio-oil yield and higher biochar yield [6]. The maximum biochar yield for both CS and CR was at 400°C, with 35.86 mf wt. % and 36.98 mf wt. % respectively.
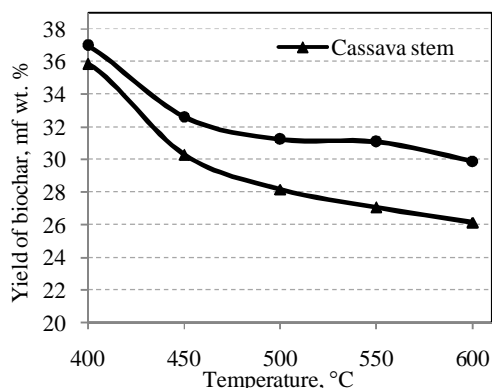
Fig. 2: Plots for effect of temperature on biochar yield from the cassava wastes (Heating rates: 5°C/min).
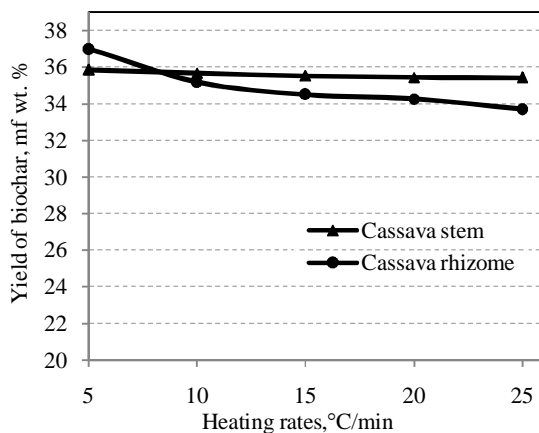

Fig. 3: Plots for effect of heating rates on biochar yield from the cassava wastes (Pyrolysis temperature: 400°C).

The plot for effect of heating rate on the yield of biochar from cassava wastes is shown in Fig. 3. At elevated heating rates, the amount of biochar yield from slow pyrolysis of both CS and CR were decreased. Compared to CR, the biochar yield from CS had only a slightly decrease and the graph is seen to be relatively maintained. For the pyrolysis of lignocellulosic biomass, their cellulose decomposition includes an exothermic pathway via anhydrocellulose that yields the biochar [26]. Anhydrocellulose is the term used for a more stable cellulose. During the rapid heating or high heating rate, the dehydration of the sample to become anhydrocellulose is limited and slow [27]. Consequently, smaller amounts of biochar are produced at a higher heating rate. The highest biochar yield was 35.86 mf wt. % and 36.98 mf wt. % for CS and CR respectively, both at the heating rate of 5°C/min.

Lower temperature and lower heating rates can favor the biochar formation. As we compare the graph pattern in Fig. 2 and Fig. 3, we can see that the pyrolysis temperature has more significant impact on

the biochar yield, compared to the heating rate parameter that turned out to give a more linear results.

Effects of Temperature and Heating Rates on the Biochar Composition: The effects of pyrolysis temperature on CS and CR biochar composition are presented in Table 2(a) and 2(b) respectively. Meanwhile, Table 3(a) and 3(b) show the results of CS and CR biochar composition produced at different heating rates. All results are the average results from triple experiment. Overall, we can see that the moisture content of both CS and CR biochar do not depend on the pyrolysis temperature and heating rate. More significant effect is seen for the volatility, ash and fixed carbon content. The ash content of CR biochar is always greater than the CS biochar in each of the studied temperatures and heating rates.

Table 2 (a): Composition of CS biochar in relation to the pyrolysis temperatures (Heating rate: 5°C/min).

Biochar	Cassava stem (CS)				
Temperature(°C)	400	450	500	550	600
Proximate analysis (mf wt. %)					
Moisture	1.90	1.49	1.87	1.57	1.26
Volatiles	14.91	10.03	5.72	5.50	5.75
Ash	6.70	6.54	8.89	8.54	7.25
Fixed carbon	78.39	83.43	85.39	85.96	87.00

Table 2 (b): Composition of CR biochar in relation to the pyrolysis temperatures (Heating rate: 5°C/min).

Biochar	Cassava rhizome (CR)				
Temperature(°C)	400	450	500	550	600
Proximate analysis (mf wt. %)					
Moisture	2.17	1.61	1.88	1.67	1.52
Volatiles	11.84	10.66	8.61	5.78	5.84
Ash	8.96	9.49	10.47	11.94	10.05
Fixed carbon	79.20	79.85	80.92	82.28	84.11

According to Table 2(a) and 2(b), the fixed carbon content for CS and CR biochar produced from the temperature of 400°C to 600°C increased by 10.98% and 6.20%, respectively, due to further decomposition of the raw sample [28].

Higher heating rate would enhance the release of volatiles since rapid heating leads to a fast depolymerization of the sample to primary volatiles [19]. The effect was shown in Table 3(a) and 3(b), where the elevated heating rates had reduced the remaining volatile matter in both CS and CR biochar. From the heating rate of 5°C/min to 25°C/min, the decrease of volatile matter for CS biochar was 13.95% (from 14.91 mf wt. % to 12.83 mf wt. %) while 11.40% (from 11.84 mf wt. % to 10.49 mf wt. %) for CR biochar. However, as the heating rate was raised, the fixed carbon content had slightly increased 2.74% (from 78.39 mf wt. % to 80.54 mf wt. %) for CS biochar and 3.12% (from 79.20 mf wt. % to 81.67 mf wt. %) for CR biochar.

Table 3 (a): Composition of CS biochar produced at different heating rates (Temperature: 400°C).

Biochar	Cassava stem (CS)				
Heating rate (°C/min)	5	10	15	20	25
Proximate analysis (mf wt. %) Moisture Volatiles Ash Fixed carbon	1.90 14.91 6.70 78.39	2.25 14.49 6.37 79.14	1.48 14.20 5.98 79.82	2.29 12.88 6.63 80.50	1.73 12.83 6.63 80.54

Table 3 (b): Composition of CR biochar produced at different heating rates (Temperature: 400°C).

Biochar	Cassava rhizome (CR)				
Heating rate (°C/min)	5	10	15	20	25
Proximate analysis (mf wt. %) Moisture Volatiles Ash Fixed carbon	2.17 11.84 8.96 79.20	2.50 10.97 8.89 80.14	1.98 10.80 8.87 80.33	1.42 10.72 7.93 81.35	1.61 10.49 7.84 81.67

As we compare the proximate analysis of raw cassava wastes in Table 1 with the composition of the derived biochar in Table 2(a), 2(b), 3(a) and 3(b), we can see that the volatile matter of the biochar has highly decreased compared to their raw sample. On the other hand, the fixed carbon content of the derived biochar had multiplied compared to the raw samples, with maximum increased of up to 5.41 times for CS and 9.26 times for CR, both produced at 600°C and heating rate of 5°C/min. According to Lehmann et al., the efficiency of carbon sequestration by a biochar was obtained when the carbon conversion into biochar leads to a sequestration of about 50% of the initial carbon content in the feedstock [29]. Since the fixed carbon content for both CS and CR biochar were all found to be more than 75 mf wt. % under any parameter, this study therefore suggests that cassava wastes biochar are suitable for carbon sequestration.

CONCLUSION

The pyrolysis temperature parameter had shown a more significant influence on the biochar yield as compared to the heating rate parameter. The lower temperature and heating rates produced more weight percentage of biochar yield from both cassava wastes. The highest biochar yield for both the CS and CR were obtained at a temperature of 400°C and a heating rate of 5°C/min. The biochar yield from CR was more affected by the heating rate parameter compared to CS that nearly had no substantial effect. The increased in temperature and heating rate raised the fixed carbon content in the biochar for both CS and CR biochar. The higher pyrolysis temperature increased more fixed carbon in the biochar as compared to the higher heating rate. The fixed carbon content in the derived biochar was much higher compared to their raw feedstock. The maximum fixed carbon content for both CS and CR biochar was obtained at the pyrolysis temperature of 600°C and at a heating rate of 5°C/min. Cassava wastes biochar are suitable for carbon sequestration.

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