Slow Pyrolysis of Oil Palm Empty Fruit Bunches for Biochar Production and Characterisation

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ABSTRACT: *The objective of this study was to investigate the influence of the ash content of oil palm empty fruit bunch (EFB) feedstock on the biochar yield percentage produced by slow pyrolysis experiments. The characteristics of the biochar produced were also investigated. Proximate analysis, elemental analysis and lignocellulosic analysis of EFB feedstock were performed. High heating values (HHVs) were determined using a bomb calorimeter. The ash content of the EFB feedstock was varied by washing. The properties of the washed and unwashed EFBs were analysed via thermogravimetric analysis (TGA) for thermal degradation behaviour. EFB feedstocks in the range of 1.60 to 5.29 mf wt% ash content were pyrolysed at 550°C with 1 h residence time. The heating rate was set at 5°C min^{-1}. The biochars produced by slow pyrolysis experiments were analysed by proximate, elemental, scanning electron microscopy (SEM) and Brunauer-Emmett-Teller (BET) surface area analyses. The EFB feedstock with the lowest ash content (1.60 mf wt%) produced the lowest biochar yield percentage (23.05 wt %) with the highest fixed carbon percentage (88.34 mf wt%). Reduction of the EFB feedstock ash content from 5.29 to 1.60 mf wt% produced significant changes in the morphological structure of the resulting biochar. The maximum BET surface area of the EFB biochar, obtained from the EFB feedstock with 2.21 mf wt% ash content, was found to be 11.1200 m^2 g^{-1}.*

Keywords: Biochar, empty fruit bunches, slow pyrolysis, washing method, ash content

1. INTRODUCTION

Biomass is one of the renewable energy resources. Generally, biomass refers to non-fossilised and biodegradable organic material originating from plants, animals or microorganisms.[1] Biomass resources include various natural and derived materials, such as woody and herbaceous species, wood wastes, bagasse, agricultural and industrial residues, waste paper, municipal solid waste, sawdust, biosolids, grass, waste from food processing, animal wastes, aquatic plants and algae.[2] The utilisation of biomass as an energy source offers a way to reduce carbon dioxide (CO_2) from the atmosphere and help to mitigate climate change by reducing greenhouse gases.

Oil palm wastes are high-potential biomass energy resources available in Malaysia because the country is the second largest producer and exporter of oil palm products in the world. In 2011, the oil palm planted area reached approximately 5 million hectares, and production mills generate a large amount of solid wastes such as extracted oil palm fibres, palm shells, palm stones and empty fruit bunches (EFB).[3,4] Most of these wastes, including EFBs, are dumped in the mill area due to their high production rate, together with their currently limited utilisation and application.[5] Therefore, the utilisation of oil palm EFBs as biomass feedstock is a good way to reduce waste management problems and decrease the emission of methane (CH_4) and nitrous oxide (N_2O) from the degradation of dumped EFBs.

Biochar is a product of thermal decomposition processes such as the slow pyrolysis process for organic materials, e.g., biomass, in the total absence or limited supply of oxygen (O_2) and at a relatively low temperature ($< 700°C$).[6,7] Biochar has been produced with the intent of being applied to soil. The application of biochar to soil improves soil productivity, reduces the emission of N_2O from soil, increases water holding capacity and has the potential to become a long-term carbon sink due to its high chemical stability, high carbon content and potential to reside in the soil for a long time.[7–9] The types and properties of biochar and soil determine the effectiveness of the use of biochar for soil treatment. The properties, composition and yield percentage of biochar depends on many factors. The initial state and type of biomass feedstock used (chemical composition, ash composition and size), pre-treatment process (drying, washing or crushing) and pyrolysis parameters (temperature, heating rate and residence hour) are among the major factors that influence the characteristics of the biochar produced.[10,11] Thus, the investigation and study of the characteristics of biochar is important so that it may be coordinated with the requirements of its application.

In this study, the influence of the ash content of EFB feedstock on biochar production from slow pyrolysis experiments was investigated. The characteristics of the raw EFBs used and the biochar produced were identified.

2.   EXPERIMENTAL

2.1   Feedstock Preparation

EFB samples used in this study were collected from Malpom Industry Sdn. Bhd., Nibong Tebal, Pulau Pinang, Malaysia. The EFBs were received in the wet condition and weighed 2.2 kg. Normally, the EFBs took more than 24 h of drying to reduce their moisture content until it was less than 10 mf wt%.[12] To avoid the growth of orange fungus and grey mould, the bunches were dried in a

conventional oven at 105°C for 48 h. The abbreviation "mf wt%" refers to the moisture free weight percentage, which is similar to the dry basis concept. Random portions of the dried bunch were selected and grounded for moisture determination according to ASTM 871 to ensure that their moisture was less than 10 mf wt%. The moisture content was calculated using the following equation:

$$\text{Moisture content (mf wt\%)} = \frac{\text{initial weight of sample - final weight of sample}}{\text{final weight of sample}} \times 100 \qquad (1)$$

where (initial weight of sample–final weight of sample) is equal to weight of water in the sample.

Then, the bunch was manually chopped into smaller sizes of approximately 5–10 cm and stored in an air-tight container prior to the pyrolysis process.

2.2   Feedstock Analysis

Preliminary analysis was conducted to investigate the characteristics of EFB samples. Proximate analysis, elemental analysis, calorific value determination, lignocellulosic percentage determination and thermogravimetric analysis (TGA) were performed. Proximate analysis was performed via ASTM E871 for moisture content,[13] ASTM E872 for volatile matter[14] and ASTM E1755-01 for ash content determination.[15] Fixed carbon was calculated by subtracting the sum of the ash content and volatile matter from 100%. This result was expressed in moisture-free weight percentage (mf wt%). Elemental analysis was performed using a Perkin Elmer 2400 analyser. The high heating value (HHV) was determined using a Nenken-type adiabatic bomb calorimeter. In addition, low heating values (LHVs) were determined using the following equation:

$$LHV = HHV \,(1 - M) - 2.447M \qquad (2)$$

where M is the wet basis moisture content of feedstock expressed as a decimal mass fraction.[16]

The lignocellulosic components of EFB feedstock (lignin, cellulose and hemicelluloses) were analysed according to the method of the Technical Association of the Pulp and Paper Industry (TAPPI). The determination of holocellulose, cellulose and lignin was performed following standard methods for TAPPI T6m-59 for extraction,[17] TAPPI T9m-54 for holocellulose and cellulose (Cross-Beran cellulose analysis)[18] and TAPPI T13m-54 for lignin (Klason lignin

analysis).[19] For hemicelluloses, different percentages of holocellulose and cellulose were determined.

TGA was performed using a Perkin Elmer Thermogravimetric Analyzer TGA7 with unwashed and washed EFB feedstock samples.

2.3   Washing Procedure

A washing treatment was performed to produce feedstock with different ash contents. Four different washing methods were applied to the EFB samples. Each method was distinguished by the amount of water used per 100 g sample, residence time of the washing treatment (in hours), washing method and water temperature. The EFB size was fixed at approximately 5–8 cm, and distilled water was used for all washing procedures. The washing procedure is summarised in Table 1. The washed EFB samples were dried in a conventional oven until the moisture content percentage was less than 10 mf wt%.

Table 1: Summary of washing procedure of EFB feedstock.

EFB sample	Washing method
1	The feedstock was not subjected to any washing treatment
2	Stir by manual 100 g of EFB feedstock in 5 l distilled water for 1 min at ambient temperature
3	Soak 100 g of EFB feedstock in 5 l distilled water for 10 min at ambient temperature
4	Soak 100 g of EFB feedstock in 5 l distilled water for 20 min at ambient temperature
5	Soak 100 g of EFB feedstock in 7 l distilled water for 120 min at 90°C

2.4   Experimental Setup

The slow pyrolysis experiment was performed using a lab-scale pyrolysis system (Figure 1). This system consists of a Thermolyne F600 Ashing Furnace, a cylindrical sample holder made of steel (pyrolyser), an L-shape iron pipe and a condensing system. The condensing system consists of two ice-cooled spherical flasks and condenser tubes.
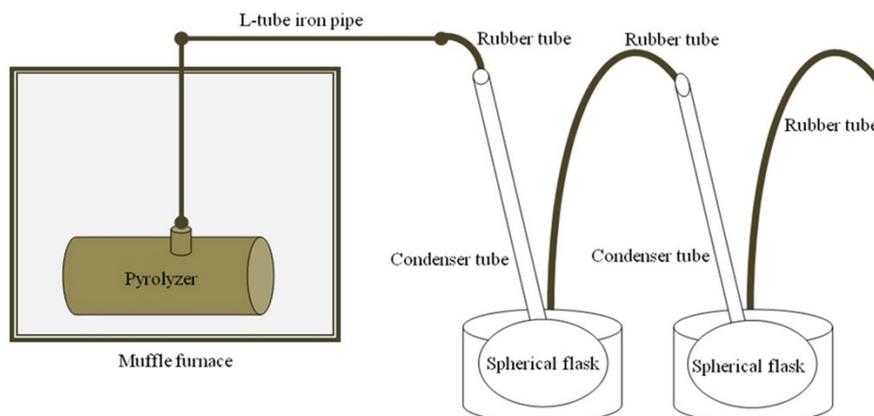
Figure 1: Lab-scale pyrolysis system.

2.5   Pyrolysis Experiment

For every slow pyrolysis experiment run, approximately 100 g of EFB samples were tightly packed by compression into the pyrolyser. Then, the pyrolyser was placed in a furnace and connected to a condensing system with an L-tube iron pipe. The terminal temperature, heating rate and residence time were set at 550°C, 5°C min^{-1} and 1 h, respectively.[20] The experiment was started with unwashed EFB feedstock (EFB Sample 1) followed by EFB Sample 2, EFB Sample 3, EFB Sample 4 and EFB Sample 5, which were obtained from various washing treatments as listed in Table 1. The experiment was repeated three times for each sample. During the pyrolysis experiment, white volatiles were emitted from the pyrolyser and passed through the L-tube iron pipe to the condensing system. The condensate was accumulated in spherical flasks. In addition non-condensable gases escaped into the fume cupboard.

When the slow pyrolysis experiment reached the fixed residence time, the pyrolyser was removed from the furnace and allowed to cool for approximately 2 h. The pyrolyser was weighed to determine the percentage of biochar yield using the following equation:

$$\text{Biochar Yield, wt \%} = [\text{Mass of char produced (g) / Mass of dry feedstock (g)}] \times 100 \quad (3)$$

The biochar obtained from the slow pyrolysis experiment was stored in desiccators to avoid moisture absorption.

2.6   Biochar Analysis

In addition to calculating the yield percentage of biochar products, analysis procedures were conducted on the EFB biochar itself. The EFB biochar was ground and stored in airtight bottles prior to these analyses. Proximate analysis was performed to determine the moisture content, volatile matter, ash content and fixed carbon content of EFB biochar. This analysis was conducted according to ASTM D1762 (Procedure for chemical analysis for wood charcoal)[21] with some modifications, as suggested by Hugh McLaughlin.[22] The temperature ranges and heating period for this study were modified with the intention to apply the biochar into soil rather than use it in the thermal energy furnace. Proximate analysis of EFB biochar first involved determining the moisture content. Biochar samples were dried in an oven until they reached 200°C. Then, the biochar samples were halved. Each half was used for the determination of ash content and volatile matter. One-half of the samples was subjected to 525°C for 6 h for the ashing process. The other half was subjected to 450°C for 30 min for volatile matter determination. In addition, elemental analysis of the EFB biochar was performed using a Perkin Elmer 2400 analyser. The percentages of carbon, hydrogen, nitrogen, sulphur and oxygen in biochar were determined by this analysis.

A surface morphology study was performed on the EFB biochar to observe the effects of various EFB feedstock ash contents on the physical characteristics of EFB biochar. Scanning electron microscopy (SEM) analysis was performed to observe structural changes in EFB biochar. High-resolution images of EFB biochar were obtained using a JEOL JSM 6400 LV model scanning electron microscope, which was operated at 15 kV. A Quantachrome Autosorb-1 Surface Analyzer was used to determine the BET surface area of the EFB biochar.

3.   RESULTS AND DISCUSSIONS

3.1   Characterisation of Raw Material

Table 2 shows the properties of the raw EFBs. From proximate analysis, it can be observed that EFB feedstock contains a large percentage of volatile matter, 77.46 mf wt%, a moderate percentage of ash content, 5.29 mf wt%, and a small percentage of fixed carbon content, 17.25 mf wt%. Thus, EFB is suitable feedstock for slow pyrolysis due to its high volatile content. The low percentage of nitrogen and sulphur in EFB obtained from elemental analysis results, which is less than 1%, indicate the environmentally friendly behaviour of this feedstock.

Table 2: Properties of EFB feedstock.

Proximate analysis (mf wt%)		Ultimate analysis (%)		Heating value (MJ kg⁻¹)	
Moisture content	1.33	Carbon	47.14	HHV	20.61
Volatile matter	77.46	Hydrogen	6.03	LHV	20.34
Ash content	5.29	Nitrogen	<0.1		
Fixed carbon[a]	17.25	Sulfur	0.84		
		Oxygen[a]	45.99		

[a]: calculated by difference

It was found that cellulose is a main component of EFB. Table 3 shows the percentages of lignin (22.8%), cellulose (57.8%) and hemicellulose (21.2%) in EFB feedstock. The percentages obtained by Hamzah et al.[23] and Kelly Yong et al.[24] are also presented in Table 3 for comparison.

Table 3: Lignocellulosic component of EFB.

Component	Measured values (%)	Literature values (%)	References
Cellulose	57.8	44.2, 38.3	23, 24
Hemicellulose	21.2	33.5, 35.3	23, 24
Lignin	22.8	20.4, 22.1	23, 24

The thermal degradation characteristics of washed and unwashed EFB are presented in the TG and derivative (DTG) curves shown in Figures 2 and 3, respectively. The TG curves in Figure 2 indicate the fractional weight loss of the various ash content samples as a function of temperature. In addition, the DTG curves in Figure 3 are plotted to show the rate of mass change, dM/dt, versus temperature.

In Figure 2, it can be observed that the thermal degradation for all feedstock samples began at approximately 200°C. Below this temperature, the weight loss for washed and unwashed EFB exhibited no significant change; this behaviour is due to moisture reduction in the samples during the analysis. When the temperature was greater than 200°C, the percent weight suddenly decreased. The weight loss for all feedstock samples was most prominent between 220°C–400°C, possibly due to the thermal degradation of polymer blocks within the biomass (such as hemicellulose, cellulose and lignin).[25] After the temperature reached 400°C, no obvious weight loss was observed. It is also apparent that when the temperature was greater than 370°C, the degradation rate for EFB with a higher ash content (5.29 mf wt%) was slower than that of washed EFB with a lower ash content (1.60 mf wt%).
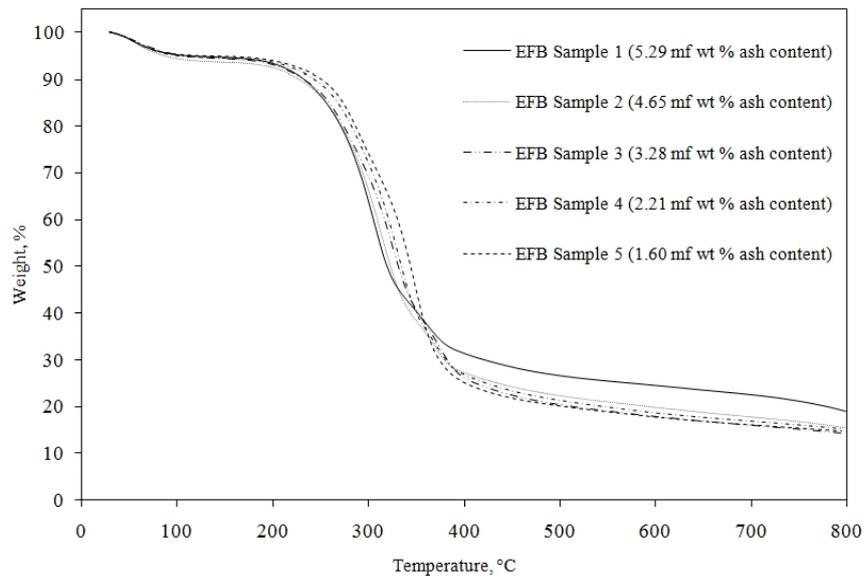
Figure 2: TG curves of EFB feedstock.
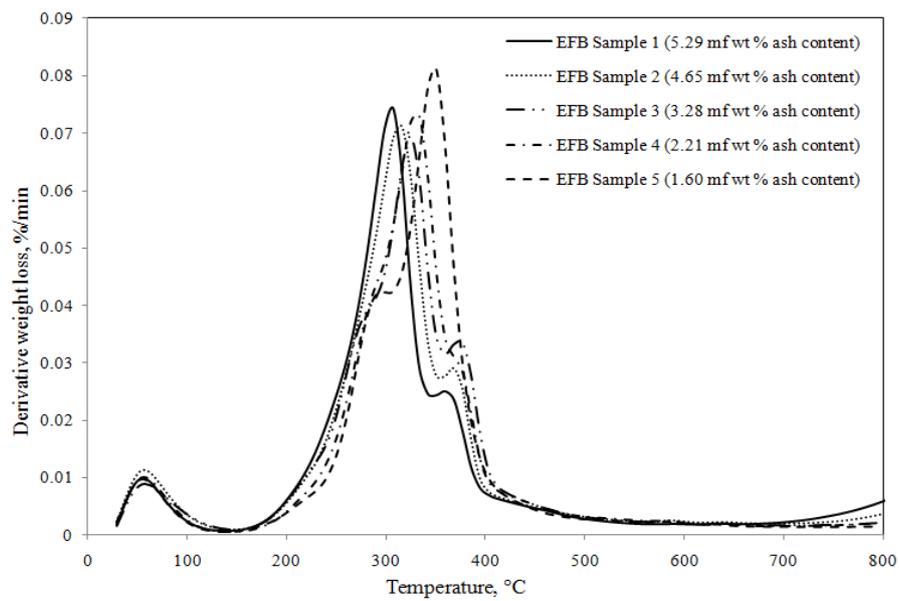
Figure 3: DTG curves of EFB feedstock.

The removal of ash content via washing had a significant influence on DTG curves. Generally, the peak of a DTG curve represents moisture reduction for temperatures < 220°C, hemicellulose degradation (220°C–300°C), cellulose degradation (300°C–340°C) and lignin degradation (> 340°C).[26]

In Figure 3, the lower peaks observed in the temperature range of 30°C to 120°C occur due to moisture reduction in the EFB feedstock. Thus, no peak shifts were observed within this temperature range. In addition, the lower peaks did not shift because the decomposition process of the EFB feedstock has not started.

In addition, at approximately 200°C–400°C, it can be observed that the peak value (mass loss rate) of the EFB samples shifted to a higher temperature within the range of 9°C–43°C after the ash was reduced from 5.29 to 1.60 mf wt% by washing treatment. This result shows that EFB samples with higher ash contents were degraded at lower temperatures. The peak shift for washed and unwashed EFBs occurs due to a reduction or loss of alkali metal content and inorganic materials during water washing. This phenomenon may be explained by the fact that the presence of ash in the EFB samples, which contain minerals, triggers a catalytic action during the thermal decomposition of the polymer blocks, thus facilitating hemicellulose and cellulose decomposition at lower temperatures.[25]

Figure 3 also shows that the peak values changed as ash was removed by washing. The peak value of EFB Sample 5, which has the lowest ash content (1.60 mf wt%), increased compared to the unwashed EFB sample with an ash content of 5.29 mf wt%. These data indicate that the highest volatile matter was released and resulted in an increase in liquid product,[27] which led to a decrease in biochar yield.

3.2   Washing Treatment of EFB Feedstock

EFB samples with different ash contents were obtained via washing, as listed in Table 1. The ash content percentages of the washed EFBs are listed in Table 4. The lowest percentage EFB ash content was 1.60 mf wt%. The highest percentage of EFB ash content was 5.29 mf wt%, which corresponded to a sample that was not subjected to washing.

Table 4: Ash content of EFB feedstock.

EFB sample	Washing method	Ash content (mf wt%)
1	The feedstock was not subjected to any washing treatment	5.29
2	Stir by manual 100 g of EFB feedstock in 5 l distilled water for 1 min at ambient temperature	4.65
3	Soak 100 g of EFB feedstock in 5 l distilled water for 10 min at ambient temperature	3.28
4	Soak 100 g of EFB feedstock in 5 l distilled water for 20 min at ambient temperature	2.21
5	Soak 100 g of EFB feedstock in 7 l distilled water for 120 min at 90°C	1.60

For the washing method applied, it can be observed that the EFB feedstock ash content decreased as the residence time was prolonged. The increase in temperature and water volume used for washing also contributed to the reduction in EFB feedstock ash content.

3.3 Product (Biochar Yield)

Biochar is the main product of the slow pyrolysis process. Using Equation 3, the biochar percentage yield was calculated and presented in Figure 4.
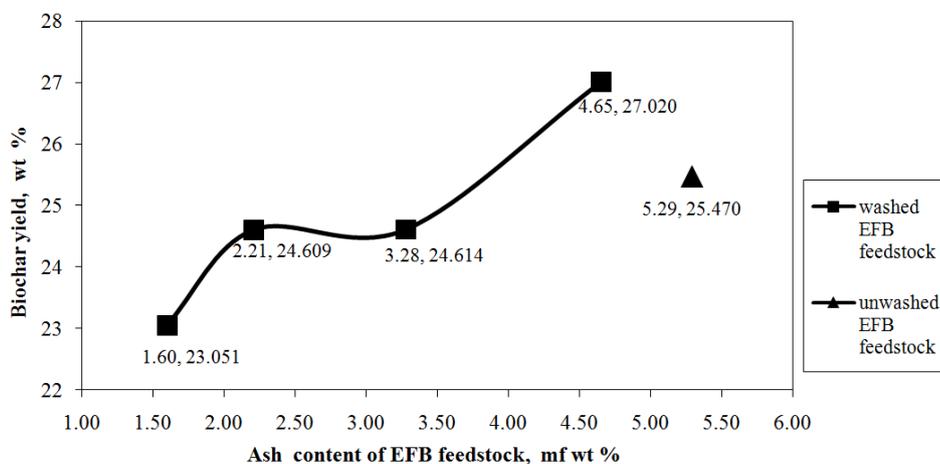
Figure 4: Effect of ash content on biochar yield.

Figure 4 shows that the biochar yield obtained from unwashed feedstock with an ash content of 5.29 mf wt% was 25.47 wt%. It also can be observed that

for the washed EFB feedstock, the biochar yield decreased as the feedstock ash content decreased. The highest biochar yield, 27.02 wt%, was obtained from the slow pyrolysis of EFB with an ash content of 4.65 mf wt%. In addition, the slow pyrolysis of EFB with the lowest ash content, 1.60 mf wt%, produced the lowest biochar percentage yield, 23.05 wt%. As expected, ash removal by washing caused a reduction in the biochar yield percentage. This result may attribute to secondary reactions among the primary pyrolysis products, which generate tars, char and gases and are slowed by the removal of the ash that generally promotes the reaction.[25]

3.4   Characterisation of Biochar

The proximate and elemental analysis results for biochar are provided in Table 5 and Table 6. The results of the proximate analysis of biochar are also presented in moisture-free weight percentage, mf wt%. According to the proximate analysis results for biochar shown in Table 5, it can be observed that as the ash content of EFB feedstock decreased from 5.29 to 1.60 mf wt%, the biochar moisture content decreased from 5.15 to 1.07 mf wt%. The volatile matter of biochar also decreased from 7.20 to 4.11 mf wt%. The ash content of the biochar produced also experienced a decrease from 19.86 to 7.55 mf wt%. In addition, the fixed carbons in the biochar increased from 72.94 to 88.34 mf wt%. The percentage of biochar carbons determined by elemental analysis also increased as the ash content of the EFB feedstock decreased. The fixed carbon obtained by proximate analysis differed from that obtained by elemental analysis. The fixed carbons in proximate analysis do not include the carbons in the volatile matter.[28] In addition, a fixed carbon is one of the important elements that determine the quality of biochar as a soil amendment. Good biochar should have a high fixed carbon content.

Table 5: Proximate analysis results of biochar.

Biochar	Ash content of EFB feedstock (mf wt%)	Proximate analysis (mf wt%)			
		Moisture content	Volatile matter	Ash content	Fixed carbon[a]
1	5.29	5.15	7.20	19.86	72.94
2	4.65	2.42	6.68	16.53	76.79
3	3.28	2.05	5.46	13.75	80.79
4	2.21	1.15	4.81	10.16	85.03
5	1.60	1.07	4.11	7.55	88.34

[a]: *Calculated by difference*

Table 6: Elemental analysis results of biochar.

Biochar	Ash content of EFB feedstock (mf wt%)	Elemental analysis (%)				
		C	H	N	S	O^a
1	5.29	67.09	2.02	6.83	0.16	23.90
2	4.65	67.26	1.59	1.12	0.29	29.74
3	3.28	72.23	1.50	6.53	<0.01	19.74
4	2.21	77.39	1.52	6.12	<0.01	14.97
5	1.60	79.04	1.82	1.34	0.26	17.54

^{a}: Calculated by difference

EFB biochars obtained from slow pyrolysis experiments have a lower percentage of volatile matter compared to EFB feedstock because volatile matter is released during the slow pyrolysis process. In addition, the percentages of fixed carbons and EFB biochar ash content are higher than that of the EFB feedstock. The increase in the ash content percentage may be due to the mineral matter that forms ash and remains in biochar after the slow pyrolysis process.[29]

SEM images of biochars produced by EFB feedstock with various ash contents are shown in Figure 5. All of the images were magnified 2,500 times. In Figure 5(a) it can be observed that the morphological structure of biochar produced from unwashed feedstock is more dense and lumpy compared with biochar produced from washed feedstock [Figure 5(b)]. In Figure 5(e), cell wall breakdown can be clearly observed for biochar produced from the lowest ash content (1.6 mf wt%).

The BET surface area of biochar was determined and shown in Table 7. The surface area of biochar is dependent on the ash content of the EFB feedstock. The results in Table 7 show that as the ash content of the EFB feedstock decreased from 5.29 to 2.21 mf wt%, the surface area increased from 0.1301 to 11.1200 m^2 g^{-1}. In general, the BET surface area of biochar increased as the ash content of raw EFB decreased. Table 7 also shows that the ash content of biochar produced from pyrolysis experiments decreases as the ash content of the EFB feedstock decreases. Thus, the low surface area of biochar produced from the EFB feedstock with higher ash content may be due to the plugging of pores by inorganic compounds in the ash present in biochar.[30,31] However, the BET surface area of biochar produced from the lowest ash content (1.6 mf wt%) was found to be reduced to 7.9890 m^2 g^{-1}, which may be due to the breakdown of cell walls, as can be observed in Figure 5(e).
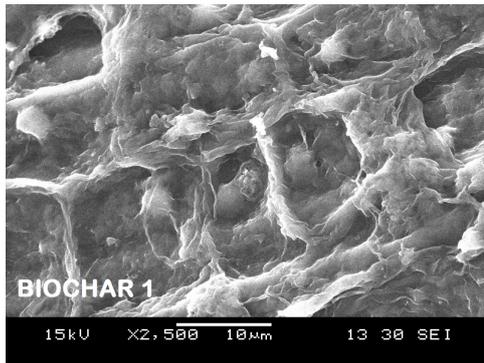
(a) Biochar 1 from EFB (unwashed feedstock) of 5.29 mf wt% ash content.
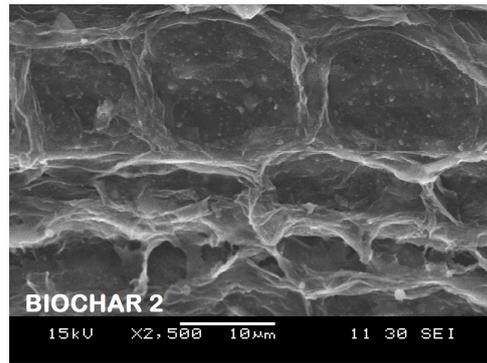
(b) Biochar 2 from EFB (washed feedstock) of 4.65 mf wt% ash content.
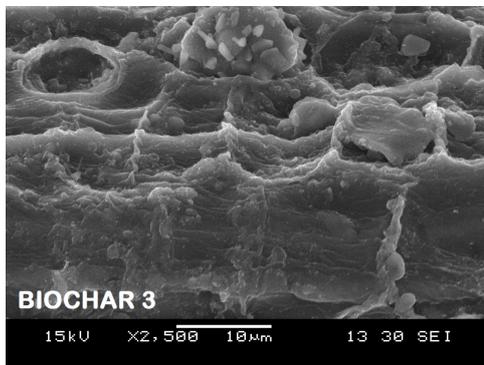
(c) Biochar 3 from EFB (washed feedstock) of 3.28 mf wt% ash content.
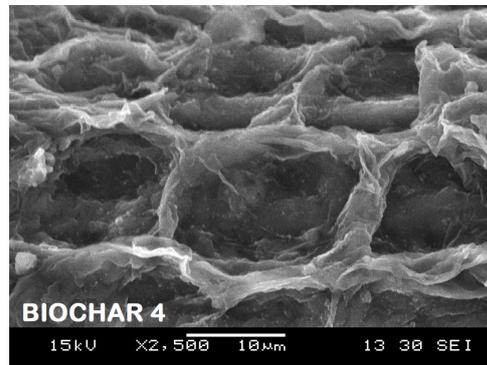
(d) Biochar 4 from EFB (washed feedstock) of 2.21 mf wt% ash content.
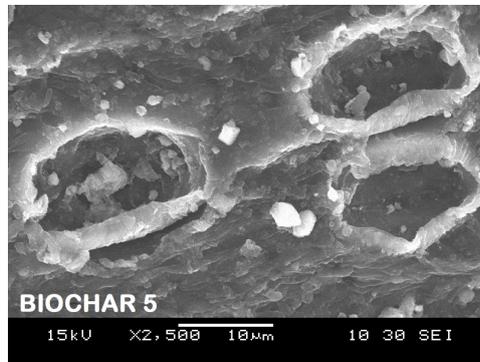
(e) Biochar 5 from EFB (washed feedstock) of 1.60 mf wt% ash content.

Figure 5: Biochars from different ash content of EFB feedstock.

Table 7: BET surface area of biochar from various ash content of EFB feedstock.

Biochar	Ash content of raw EFB (mf wt%)	Ash content of the biochar (mf wt%)	Biochar BET surface area ($m^2\,g^{-1}$)
1	5.29	19.86	0.1301
2	4.65	16.53	0.3843
3	3.28	13.75	9.2480
4	2.21	10.16	11.1200
5	1.60	7.55	7.9890

4. CONCLUSION

EFB is suitable for utilisation as a feedstock in the production of biochar via slow pyrolysis. Washing treatment of the EFB feedstock reduces its ash content. As the ash content of raw EFB feedstock decreases, the biochar yield percentage is reduced, and the fixed carbon percentage of biochar increases. Biochar with a high fixed carbon content of 88.34 mf wt% was produced from the pyrolysis of raw EFB with 1.60 mf wt% ash content. SEM images show significant changes in the morphological structure of biochar as the ash content of EFB was reduced. A maximum value of 11.1200 $m^2\,g^{-1}$ BET biochar surface area was obtained from raw EFB feedstock with a 2.21 mf wt% ash content.

5. ACKNOWLEDGEMENT

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