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Carbonisation of Oil Palm Fronds (December 2006)

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Abstract - Among renewable energy sources, biomass has a great potential for commercial usage. Pyrolysis is one of the most promising thermal conversion processes for biomass. In this study, pruned oil palm fronds (OPF) from the plantation were carbonised at terminal temperatures of 450-800 °C with a holding time of 1 hour and at a constant heating rate of 10 °C min⁻¹. A lab-scale pyrolyser was used and the emissions were condensed to result in charcoal, liquid (tarry and aqueous fractions) and gaseous products. The quality of the charcoal produced, which was found to be somewhat dependent on the terminal pyrolysis temperature is of medium grade. The best OPF charcoal with 75.54 % of fixed carbon and an energy content of 28.92 MJ kg⁻¹ was obtained at a terminal pyrolysis temperature of 750 °C. The chemical characterization of the liquid products showed that both fractions may be a potential source of valuable fuel and chemical feedstocks.

Keywords - Biomass, Carbonisation, Oil palm fronds (OPF), Charcoal.

1. INTRODUCTION

In Jan 2006 Malaysia is still the largest palm oil producer in the world with over 4.05 million hectares of plantations planted with oil palms and nearly 450 mills to process the palm fruits [1]. Wastes generated from the oil palm industry are from these two sources, that is the palm oil mill and the plantation field. Oil palm empty fruit bunches form the largest quantity of lignocellulosic biomass produced in the mills. On the other hand the biomass from the field are mainly the oil palm trunks (OPT) and fronds.

Oil palm fronds (OPF) have to be cut during the harvesting of the ripe fruit bunches. This is called pruning. Pruning may also be done on a periodic basis and not necessarily at the time of fruit harvesting. Mohamad Husin et al. [2] reported that the pruning process generates roughly 11 dry tonnes of fronds annually from one ha of land.

The current practice is to leave the fronds to rot on the plantation grounds, while the old palm trees when felled during replanting are normally chipped on site or stacked across the contours of steep terrains and left to decompose. Thus both OPT and OPF act as mulches that help to reduce soil erosion by up to 33 % and are also effective in recycling the plant nutrients [3]. However recently, OPF have been identified as a potential feed for ruminants and other herbivores [4].

At Universiti Sains Malaysia (USM), various attempts have been made to further add value of these lignocellulosic

wastes. One such attempt investigated the feasibility of carbonising the OPT and EFB to produce charcoal and possibly bio-oil from the emissions of the carbonisation process [5, 6]. A similar study on the carbonisation of OPF was also initiated. This paper reports on the results of that study.

2. METHODS

Material

A fresh oil palm frond (OPF) was procured from an oil palm estate in Nibong Tebal, Penang. The sample was first cut into small pieces of about 6 cm in length, 3 cm in width and dried overnight in an oven at 105 °C. (see Fig. 1).



Fig. 1. Picture of a few cut pieces of fresh OPF

It has been reported that this lignocellulosic waste consists of 38.8 % cellulose, 36.4 % hemicellulose and 19.3% lignin [7]. Proximate analysis, elemental composition and calorific value determinations were first done on the raw OPF sample. The methods were similar to that done on the solid product as described in section "Solid and Liquid Product Analysis".

Pyrolysis Experiment

A muffle furnace (Thermolyne F62700-33-80) was used to carbonise the OPF which was placed inside a cylindrical stainless steel holder of about 15 cm in length, 7 cm in

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internal diameter and 7.5 cm in external diameter. This holder has a tight screw-on cap and an outlet port for the escape of the volatile emissions during the carbonisation process. This port exited the furnace via an opening at its top. The outlet port for the volatiles was connected to a series of water-cooled condensers where condensable gaseous emissions were condensed and collected in ice-cooled spherical flasks. Non-condensable gases were allowed to escape to the outside of the laboratory.

Heating was carried out at a constant rate of 10 °C min⁻¹ temperature rise to different terminal carbonisation temperatures. When the desired terminal temperature was attained, carbonisation was allowed to proceed at that temperature for one hour. Carbonisation experiments were done for terminal temperatures of 450 °C to 800 °C increasing in steps of 50 °C. For each terminal temperature value, 3 carbonisation runs were carried out.

After each experiment the sample holder was left inside the muffle furnace for at least 24 hours for it to cool before the char yield was determined. However the yield of condensable emissions was determined immediately after each carbonisation run. It was found that the cooled condensate separated into a tarry fraction and a less oily aqueous fraction. Mass balance analyses were done on the yields of the products where the non-condensable product yield was determined by the difference between raw material weight and the yields of the solid and liquid products.

Solid and Liquid Products Analysis

The charcoals produced were left exposed in the laboratory environment for a further 2 days before they were milled into granules for the analysis of quality. Proximate analysis was performed on 3 samples of each carbonised product using a thermogravimetric analyzer (TGA / SDTA851). Besides that, elemental analysis on the OPF charcoal produced was done using a CHN analyzer (Perkin Elmer 2400 Series II). The calorific value was determined using an adiabatic oxygen Bomb Calorimeter (Parr Model 1261). Standard procedures were adopted for all of the above analyses.

The liquid product selected for analysis was that obtained at the carbonisation temperature, which gave the maximum condensate yield. Fourier transform infrared spectroscopic analysis was performed using a Perkin Elmer FTIR 2000 spectrometer. A thin uniform layer of the liquid was placed between two salt cells and exposed to the IR beam. The functional chemical groups in both liquid fractions were identified by their FTIR absorption frequency spectra. Their pH and density were also determined.

3. RESULTS AND DISCUSSION

The weight of oven dried raw OPF that was packed into the sample holder for each carbonisation run was found to be about 130 to 150 g. Table 1 indicates that oven dried raw OPF has a rather high volatile content and a low amount of fixed carbon. The ash content however appears to be of medium level. As expected the gross calorific value of the

raw OPF is lower than that of coal. The elemental analysis results indicate that OPF is rather environment friendly, with trace amounts of nitrogen and sulfur.

Table 1. Composition of the oven dried raw OPF. The values shown are the average from 3 runs

Proximate analysis (wt. %)		Elemental analysis (wt. %)	
Moisture	5.07	Carbon	41.13
Volatiles	73.92	Hydrogen	4.56
Fixed Carbon	17.09	Nitrogen	0.03
Ash	3.92	Sulphur	0.49
		Oxygen (by difference)	49.87
Gross Calorific Value (MJ kg ⁻¹)	15.66	Ash	3.92
Empirical formula of raw OPF: CH_{1.33}O_{0.91}N_{0.0006}S_{0.0044}		H/C molar ratio	1.33
		O/C molar ratio	0.91
		N/C molar ratio	0.0006
		S/C molar ratio	0.0044

In this paper, yield of charcoal is defined as:

$$\frac{\text{Weight of OPF charcoal produced}}{\text{Weight of dry raw OPF used}}$$

and

yield of condensates is defined as:

$$\frac{\text{Weight of OPF condensates collected}}{\text{(whether tarry or aqueous fraction)}} \div \text{Weight of dry raw OPF used}$$

The data of the graph in Fig. 2 indicate that an increase of the carbonisation temperature leads to a decrease in charcoal yield and an increase in the yield of non-condensable gases. The decrease in the charcoal yield with increasing temperature could either be due to a greater primary decomposition of the OPF at higher temperatures or through secondary decompositions of the charcoal. The secondary decompositions of the charcoal at higher temperatures may also produce non-condensable gaseous products thus contributing to the increase in the non-condensable gas yield as carbonisation temperature increases [8]. The total condensate yield for all runs was more than 40 % with a maximum yield of 46.82 % at 750 °C and the tarry fraction was about 20 % of the total condensates collected.

Figure 3 shows the results of the proximate analysis done on OPF charcoal. There was not much dependence on terminal temperature observed for the moisture and ash contents. An average value of about 5 % moisture content and 12 % ash were found. Since the products were allowed 2 days to equilibrate with the laboratory environment, the moisture content of the charcoal is perhaps of little significance. The ash content increased by more than 3 folds compared to the value for the raw OPF. The fixed

carbon content increases as terminal temperature increases as at elevated temperatures more volatiles are driven off. However the increase is not significant once a terminal temperature of about 650°C is reached. As such it may not be necessary to carbonise the OPF beyond 650°C.

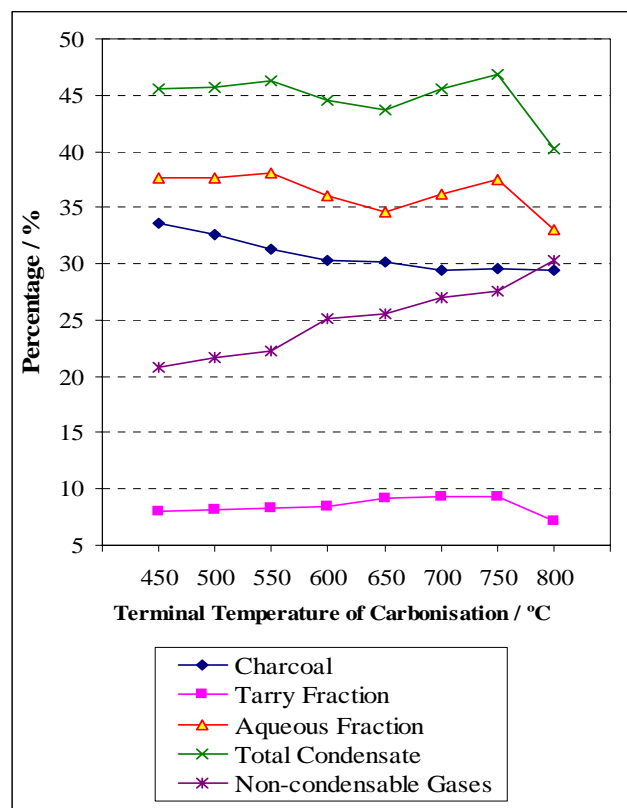


Fig. 2. Comparison of carbonisation product yields for various carbonisation temperatures.

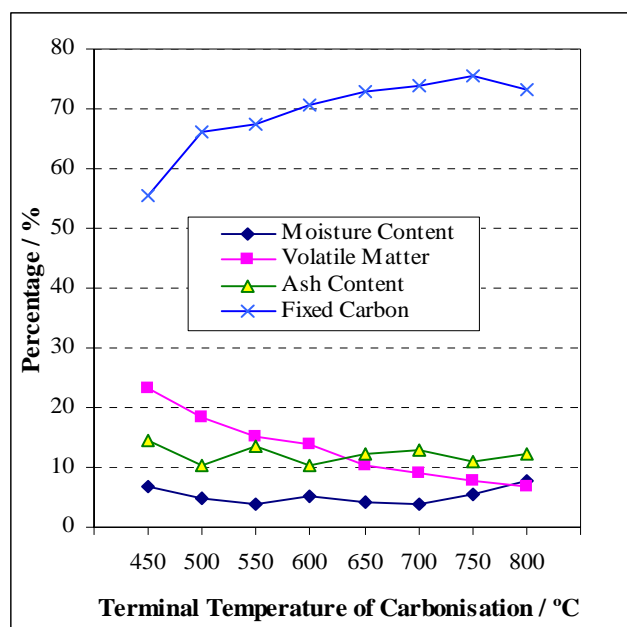


Fig. 3. Proximate analysis of OPF charcoal for various carbonisation temperatures.

Table 2 shows the elemental compositions and calorific values of the charcoal produced. Though there is a slight

drop of the carbon content at 800 °C, it generally increases as terminal temperature increases while the hydrogen content decreases. The percentage of the carbon content is comparable to the values found for the fixed carbon content. However the nitrogen content does not exhibit any clear trend with the terminal temperature. In line with the carbon content, the calorific value increases when terminal temperature increases except for a slight drop at 800°C.

Table 2. Elemental analysis of OPF charcoal and their calorific values.

Terminal Temperature of Carbonisation (°C)	Elemental Analysis			Calorific Value (MJ kg ⁻¹)
	Carbon (wt.%)	Hydrogen (wt.%)	Nitrogen (wt.%)	
450	55.70	3.20	0.54	25.22
500	65.57	2.62	0.60	27.98
550	68.68	2.02	0.74	28.36
600	70.59	1.63	0.61	28.75
650	71.11	1.29	0.51	28.86
700	72.25	1.08	0.41	28.88
750	73.48	1.01	0.62	28.92
800	69.50	0.79	0.48	25.84

Note: The values shown are the average from 3 runs.

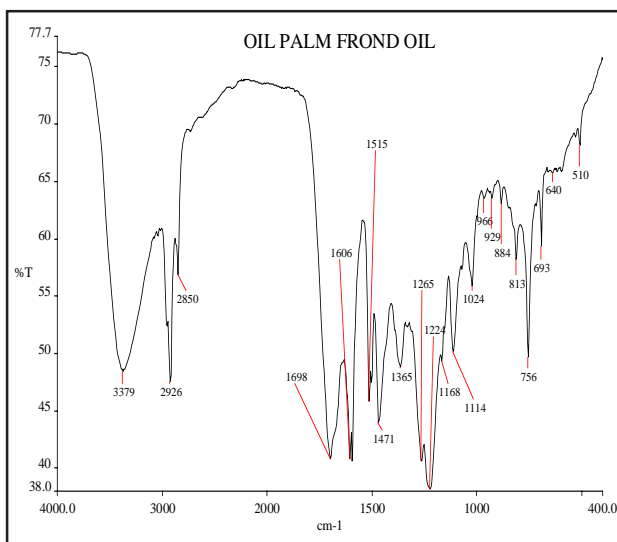
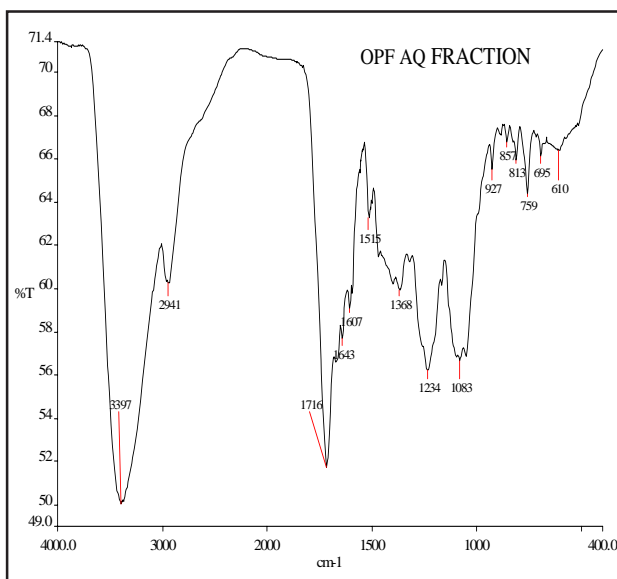
The results on the quality of the OPF charcoal as discussed above indicate that the charcoal produced is of fairly good quality as OPF charcoal with a fixed carbon content of 75.54 % (or 73.48 % carbon content) and a calorific value of nearly 29 MJ kg⁻¹ can be produced but the ash content is on the high side. Even so, depending on application, the product can still be used such as for barbecue.

The condensates collected, even after 2 hours of standing, were found to be not homogeneous as 2 fractions; one of insoluble tar and the other of a less oily aqueous fraction were observed. These fractions were separated through filtration as most of the tarry substance could not get through the filter paper. This observation is similar to that reported by Soltes and Elder [9] when wood was pyrolysed. They also reported that the tarry oil from wood pyrolysis can be upgraded to bio-oil while wood vinegar, the aqueous fraction consists of about 80 % water and some methanol, acetic acid, acetone, methyl acetate, soluble tar etc. In this study, the average density of the brownish colour aqueous fraction was found to be about 1.03 g cm⁻³ and the pH value was 2, indicating that it is very acidic .

The FTIR absorption frequency spectra of both tarry and aqueous fractions are shown in Fig. 4 and Fig. 5 respectively. The spectral data show that alcohol, ketone, ester and carboxylic acid groups are the major oxygenated compounds present in both the tarry and aqueous fractions. Both also contain a moderate concentration of hydrocarbons but their quantities in the tarry fraction are higher. Traces of aromatic compounds were found in both fractions. Even through both FTIR spectra have rather similar functional groups, the water content in the aqueous fraction is definitely very high. The functional group compositions for both fractions as presented in Table 3 indicate that the fractions may be a fairly good source of chemicals.

Table 3. FTIR functional group compositions of the tarry and aqueous fractions from the carbonisation of OPF

Wave number range (cm ⁻¹)	Wave number (cm ⁻¹)		Group	Class of compound
	Tarry Fraction	Aqueous Fraction		
3300-3600	3379	3397	O-H stretching	Polymeric O-H, water impurities
3050-2800	2926, 2850	2941	C-H stretching	Alkanes
1750-1650	1698	1716	C=O stretching	Ketones, aldehydes, Carboxylic acids
1650-1580	1606	1643, 1607	C=C stretching	Alkenes
1550-1490	1515	1515	NO ₂ stretching	Nitrogenous compound
1470-1350	1471, 1365	1368	C-H bending	Alkanes
1300-950	1265, 1224, 1168, 1114, 1024, 966	1234, 1083	C-O stretching O-H bending	Primary, secondary and tertiary alcohol, phenol, ester and ethers.
915-650	884, 813, 756, 693	813, 759, 695		Aromatic compounds

**Fig. 4. FTIR spectra of the tarry fraction****Fig 5. FTIR spectra of the aqueous fraction**

4. CONCLUSION

Work on the carbonisation of OPF was carried out in a laboratory-scale pyrolyser at 8 different carbonisation temperatures and at a heating rate of 10 °C min⁻¹. The carbonisation products from OPF biomass consist of solid charcoal, condensable oxygenated organic liquids, water and non-condensable gases.

The main product of OPF charcoal with 75.54 % fixed carbon and an energy content of 28.92 MJ kg⁻¹ was obtainable at a terminal carbonisation temperature of 750 °C. Even so the product is of medium quality as the ash content is on the high side. However, it is possible to use the product as fuel when a high ash content is not a problem.

The maximum total condensate yield was obtained at a terminal temperature of 750 °C. The condensate separated into two fractions: a tarry and an aqueous fraction. Both fractions were analysed by FTIR and they can be a source of different ketones and acidic compounds as well as of alkyl phenols if proper extraction processes can be adopted for the recovery of these chemicals. The tarry fraction can also be used as a liquid fuel.

The gaseous products which were non-condensable were not investigated. However they are combustible and thus can be used as a fuel also.

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