Slow Pyrolysis of Oil Palm Empty Fruit Bunches

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Abstract – *Biomass in the form of empty fruit bunches from oil palm trees (Elaeis guineensis) were pyrolysed in a laboratory-scale pyrolyser which was externally heated by a muffle furnace. The effects of pyrolysis temperature and heating rate on the yields and quality of the products were investigated. Pyrolysis runs were performed at terminal temperatures of 450 - 800 ^{o}C with heating rates of 5 and 10 ^{o}C min^{-1}. Product yields were found to be significantly influenced by the pyrolysing conditions. The products were mainly solid char and the condensates separated into a tarry fraction (EFB oil) and an aqueous fraction. Char with 74.8 % fixed carbon and a calorific value of 28.61 MJ kg^{-1} was obtained at 550 ^{o}C when the heating rate was 5 ^{o}C min^{-1}. The empirical formula of the EFB oil with a calorific value of 28.92 MJ kg^{-1} was established as $CH_{1.49}O_{0.42}N_{0.02}$.*

Keywords – Biomass, empty fruit bunch (EFB), EFB char, EFB oil, pyrolysis

1. INTRODUCTION

Lim [1] forecasted in 2000 that Malaysia's fossil fuel reserves together with hydropower potential can sustain the nation's energy needs up to about 2035. Therefore, alternatives especially renewable energy resources have to be found to replace the depletion of the nation's fossil fuels and the oil palm tree is one of the plausible renewable sources of energy [2].

In January 2008, Malaysia is still the largest palm oil producer in the world with over 4.305 million hectares of plantation planted with oil palms [3]. There are close to 430 palm oil mills that process the palm fruits and these mills generate an abundant amount of biowastes or biomass. The solid biomass generated consists of shells, fruit fibers and empty fruit bunches (EFBs). For every tonne of fresh fruit bunch (FFB) processed, about 55 kg, 135 kg and 220 kg of such biomass respectively are produced [4].

The fruit fibers and shells are used to fire the boilers in the mills to produce steam and generate power for the milling process but not the EFBs which can be a potential source of renewable energy. Malaysia has targeted 5 % of power generation from renewable sources by 2005 [5]. Previously, EFBs were incinerated yielding a 6.5% ash that was used as a good source of potash for young palms. With the introduction of new regulations by the Department of Environment (DOE), incineration is no longer permitted in new mills. The EFBs are now used by large plantations for mulching in the fields where they act as a fertilizer supplement, thereby reducing the cost of fertilizer in the oil palm plantations [6]. Even so, large quantities are still available; hence projects were initiated at Universiti Sains Malaysia to explore the feasibility of converting the rather wet EFBs into more versatile forms of biofuel.

The thermochemical conversion method adopted for this project was slow pyrolysis [7]. A preliminary study

on the carbonization of oil palm EFBs had previously been reported by [8]. In that study a single heating rate of 10 ^{o}C min^{-1} was used and the condensates from the pyrolysis process were not analysed. This paper reports on an extension of that study, in that, two rates of heating were used and the characteristics of the condensates were investigated.

2. MATERIALS AND METHODS

Raw Materials

Basiron and Husin [9] reported that EFBs, a lignocellulosic waste, consist of 44.2 % cellulose, 33.5 % hemicellulose and 20.4 % lignin. Each component will contribute towards the formation of volatiles, gases and char when the lignocellulose is pyrolysed.

Figure 1 shows the picture of a fresh EFB. The EFB is made up of a main fibrous stalk and numerous rather stiff spikelets with sharp tips. EFBs are the left over fruit bunches after the fruits for oil production are separated from the bunch stalk. EFBs are bulky and have a high moisture content of about 70 %.

Fig. 1. Picture of a fresh EFB from the oil palm mill.

Prior to use, the EFB was manually torn apart until all the spikelets separated from the stalk. Both spikelets and stalks were cut to about 10 cm of length. They were then left to dry in the open under the hot sun for two weeks. The EFBs for our experiments were obtained from the United Oil Palm Mill Sdn Bhd, Nibong Tebal, Penang.

Pyrolysis Reactor

Slow pyrolysis of the EFBs was carried out using a lab-scale pyrolyser. The pyrolyser is basically a cylindrical

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stainless-steel container. This sample holder has a length of 15.0 cm and an internal diameter of 7.0 cm. The pyrolyser was heated externally in a muffle furnace (Type F62700-33-80) and the temperature was controlled by a microprocessor. The pyrolysis experiments were performed with about 65 g of rather dry EFB packed inside this pyrolyser. The emissions of the pyrolysis process were recovered using a series of condensers.

Pyrolysis Experiments

The experiments were divided into two parts. The first was carried out to determine the effect of pyrolysis temperature and the effect of the heating rate on pyrolysis yields. The terminal pyrolysis temperature once attained was maintained for one hour and the terminal temperature range investigated was from 450 to 800 $^{\circ}$C increasing in steps of 50 $^{\circ}$C, while the heating rates used were 5 and 10 $^{\circ}$C min^{-1}. During pyrolysis, the emissions were led through an outlet port to two water-cooled condensers and the condensate was collected in ice-cooled spherical flasks which also had an outlet for gaseous products, that could not be condensed, to escape to the outside of the laboratory.

The pyrolysis process resulted in a solid product of char, a liquid product and non-condensable gases. The quantities of char produced were determined by weighing after the pyrolyser was allowed to cool for at least 24 hours. However the yield of condensable emissions was determined immediately after each pyrolysis run. The weight of non-condensable gases was estimated from the difference between raw material weight and the yields of char and condensate. The small quantity of condensate trapped in the connecting pipe was ignored. For each terminal temperature value and each heating rate, 3 pyrolysis runs were carried out.

The second part of the experiments involved the various analyses of the products. Proximate analysis for determining the quality of the solid product and its elemental composition were done. The proximate analysis for moisture, ash and volatile contents were done as per ASTM D 3173, D 3174 and D 3175 respectively [10] and was further confirmed with thermal gravimetric analysis. Thermal analysis of the char was performed using a thermogravimetric analyzer (Perkin Elmer/TGA7) coupled with a TG controller (Perkin Elmer / TAC 7/DX). Elemental analysis on the EFB charcoal produced was done using a CHNS analyser (Perkin Elmer 2400 Series II). The gross calorific value of EFB char was determined using an adiabatic oxygen bomb calorimeter (Parr Model 1261). Standard procedures were adopted for all of the above analyses which were performed on the products of each experimental run. Similar analyses were also done on the raw EFB.

The condensable emissions, the condensates, which were examined immediately after the pyrolysis process were found, after filtration, to separate into a more aqueous fraction and a rather viscous tarry fraction. The pH and density values of the aqueous fraction were determined. The tarry fraction selected for further analysis was those obtained from experiments that gave the maximum condensate yield and we will label this fraction as EFB oil. The EFB oil obtained was analyzed for its density, viscosity, flash point, pour point and calorific value as per ASTM D 1298, D 445, D 93, D 97 and D 5865 respectively [11]. The functional chemical groups in the tarry fraction and aqueous fraction were identified by their Fourier Transform Infra-red spectra obtained using a FTIR spectrometer (Perkin Elmer FTIR-2000). Further analysis was also performed to identify the major chemical compound of the aqueous fraction using a Gas Chromatography Mass Spectroscopy spectrometer (ThermoFinnigan GC-MS). Standard procedures for the operation of the above spectrometers were followed.

3.   RESULTS AND DISCUSSION

Raw Material Characteristics

A knowledge of the physical and chemical characteristics of raw materials is essential for optimizing the efficiency of any thermochemical conversion process. The proximate analysis, elemental composition and gross calorific value of the sun-dried raw EFB are given in Table 1.

Table 1. Composition of the sun-dried raw EFB. Each value is the average of three runs.

Proximate Analysis (wt. %)		Elemental Analysis (wt. %)	
Moisture	8.65	Carbon	43.21
Volatiles	75.90	Hydrogen	7.42
Ash	3.92	Nitrogen	0.86
Fixed carbon	11.53	Sulphur	0.75
Oxygen (by difference)			43.84
H/C molar ratio	2.06	Empirical formula of raw EFB : $CH_{2.06}O_{0.76}N_{0.02}S_{0.01}$	
O/C molar ratio	0.76		
N/C molar ratio	0.02		
S/C molar ratio	0.01		
Higher calorific value		18.66 MJ kg^{-1}	

The elemental analysis indicates that raw EFB is environment friendly, with trace amounts of nitrogen and sulphur, but the proximate analysis shows that raw EFB has a rather high volatile content, a medium level of ash content and a rather low fixed carbon content.

Product Yields

As the biomass is heated up, the initial stage involves the removal of moisture from the EFB. As the experiment proceeded, lots of white colour volatiles were observed to be released when the temperature inside the pyrolyser reached approximately 240°C. The release of white volatiles ended after about 11 minutes for the heating rate of 5 $^{\circ}$C min^{-1} but about 13 minutes for the heating rate of 10 $^{\circ}$C min^{-1}.The condensates collected in the ice-cooled flasks were found to consist of a rather viscous black tarry component floating in a fairly clear brownish solution.

Chars, gases, light and heavy organic liquids and water were formed in varying amounts on pyrolysis of biomass at varying conditions. Their yields depend not only on the raw material composition but on heating rate and terminal pyrolysis temperature as well [12]. In our study when EFBs were heated in the absence of air, they decomposed and were converted into char as a main product and a volatile fraction that can be partly condensed on cooling. Here, yield of char is defined as:

$$\frac{\text{Weight of EFB char produced}}{\text{Sun-dried weight of raw EFB used}} \quad (1)$$

and Yield of condensates is defined as:

$$\frac{\text{Weight of EFB condensates collected (tarry plus aqueous fractions)}}{\text{Sun-dried weight of raw EFB used}}$$

$$(2)$$

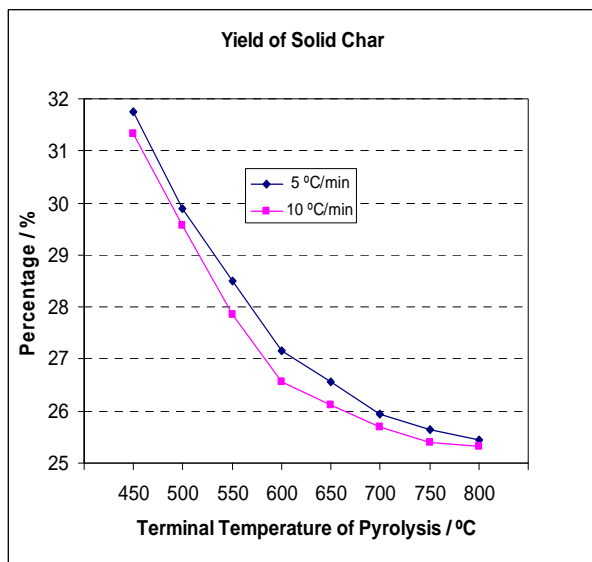The average yields of char, condensates and gases are plotted as graphs in Figures 2 to 6.

Yield of Solid Char

Fig. 2. A comparison of solid char yield for various pyrolysis temperatures and two heating rates.

Figure 2 indicates that EFB char yield decreases as the terminal pyrolysis temperature increases for both the heating rates. This observation is expected as at higher pyrolysis temperatures, more volatiles are driven off. Char yields for the 10 $^\circ$C min^{-1} heating rate are only slightly lower than yields at the lower heating rate of 5 $^\circ$C min^{-1}. This may be related to a faster depolymerization of the raw material at faster heating. After a pyrolysis temperature of about 650 $^\circ$C, most of the volatiles would have been released, hence the decrease of yields would be less significant.
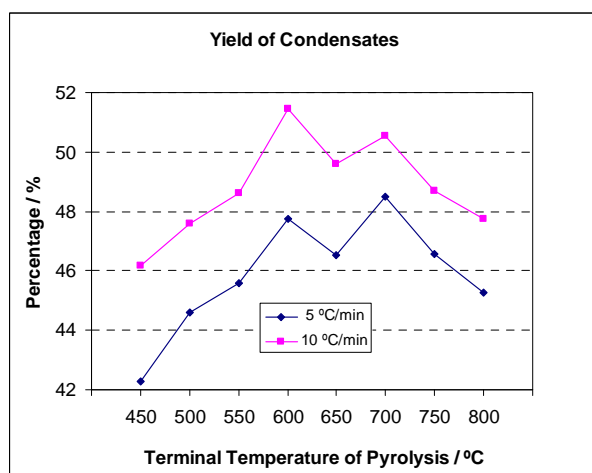
Yield of Condensates

Fig. 3. A comparison of condensate yields for various pyrolysis temperatures and two heating rates.

The condensates after 2 hours of standing at room temperature were still found to be not homogeneous as the separation into 2 distinct layers, one of viscous tar and the

other of a less oily brownish aqueous phase was still observed. The 2 layers were separated through filtration where most of the tarry substance did not get through the filter paper. As indicated earlier we labeled the black viscous tarry fraction as *EFB oil* while the brownish liquid is referred to as *aqueous fraction*. At both heating rates in Figure 3, condensate yields display a steady increase for terminal temperatures of 450°C-600 $^\circ$C and then a more gradual decrease was observed when the terminal temperature range was 700 $^\circ$C-800 $^\circ$C with a larger drop at 650 $^\circ$C.

The yields of the EFB oil and aqueous fraction were observed to be also dependent on pyrolysis temperature and heating rates as shown in Figures 4 and 5.
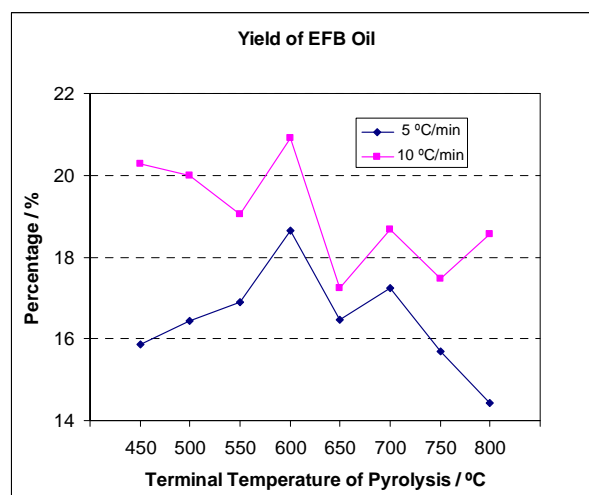
Yield of EFB Oil

Fig. 4. A comparison of EFB oil yield for various pyrolysis temperatures and two heating rates.
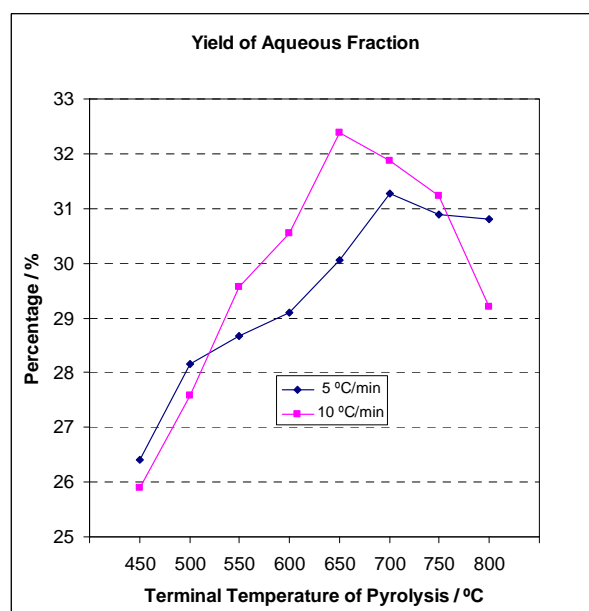
Yield of Aqueous Fraction

Fig. 5. A comparison of the less oily aqueous fraction yield for various pyrolysis temperatures and two heating rates.

A maximum oil yield was obtained at a pyrolysis temperature of about 600 $^\circ$C for both the heating rates. Oil yield is higher at 10 $^\circ$C min^{-1} and this is to be expected but generally both yields decrease after the peak. The data of Figure 5 show that for the heating rate of 5 $^\circ$C min^{-1} the yield of the aqueous fraction gradually increases with

pyrolysis temperature but after a temperature of 700 $^{\circ}$C, the change is not significant whereas for the heating rate of 10 $^{\circ}$C min^{-1}, a maximum was observed at 650 $^{\circ}$C.

The gaseous products, the non-condensable volatile emissions, which escape to the outside of the laboratory were found to be continuously combustible only after the pyrolysis temperature attains a value of about 500 $^{\circ}$C. Thus these gases may be used as fuel.
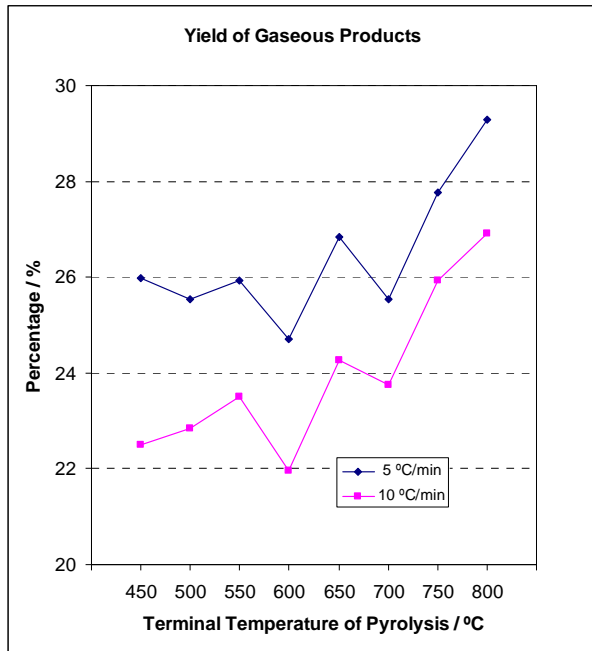

Fig. 6. A comparison of the gaseous products yield for various pyrolysis temperatures and two heating rates.

The data of Figure 6 indicate that the yield of the gaseous products is fairly independent of pyrolysis temperatures for the 450 - 600 $^{\circ}$C range but a gradual increase occurred when pyrolysis temperatures exceed 600 $^{\circ}$C. These trends were observed for both heating rates with a higher gas yield at the lower heating rate.

Quality Analysis of the Solid Char

The quality of the EFB char produced at different conditions is determined by proximate analysis, TGA runs and calorific value determinations. The proximate analysis and TGA runs gave similar information on quality parameters. Therefore, data from both analyses were taken and their averages are plotted in Figures 7 and 8.

The trends shown in Figures 7 and 8 appear rather the similar for both the heating rates. Both sets of data indicate that as terminal pyrolysis temperature increases the moisture content of the EFB char increases somewhat. This may due to the product becoming more porous at higher pyrolysis temperatures. Except for a peak at 550 $^{\circ}$C for the heating rate of 5 $^{\circ}$C min^{-1} and a peak at 600 $^{\circ}$C for the heating rate of 10 $^{\circ}$C min^{-1}, the fixed carbon content of the char is rather independent of the pyrolysis temperature. This is somewhat reflected in the calorific value data. A corresponding drop though slight in volatile content was observed at these temperature values. This observation, which may be characteristic to EFB, was rather unexpected as generally fixed carbon contents of carbonized biomass increase with pyrolysis temperature. Though there appears to be slight variations of the ash

content with pyrolysis temperature, the variations are not glaring. However, the ash content of the char is 2-3 times that of the raw material.
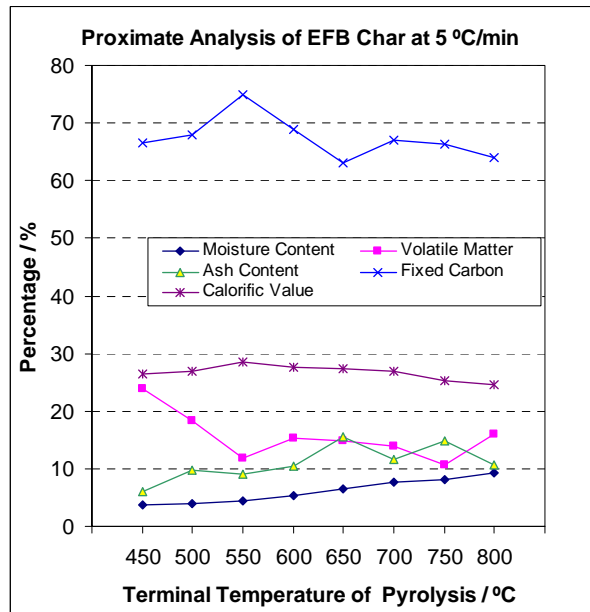

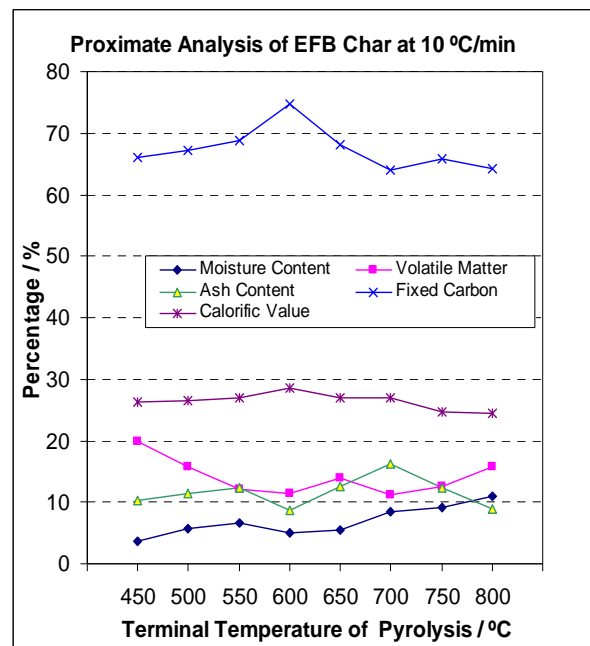**Fig. 7. Proximate analysis and calorific value of the EFB char produced. Heating rate: 5 $^{\circ}$C min^{-1}**

Fig. 8. Proximate analysis and calorific value of the EFB char produced. Heating rate: 10 $^{\circ}$C min^{-1}

The results indicate that under our pyrolysis conditions, the best quality char of fixed carbon 74.8 % and calorific value of 28.61 MJ kg^{-1} was produced at a terminal pyrolysis temperature of 550 $^{\circ}$C when the heating rate was 5 $^{\circ}$C min^{-1}. Elemental analysis done on one such sample is shown in Table 2.

Table 2. Elemental analysis of the selected EFB char with a fixed carbon content of 74.8%. Each value is the average of three runs.

Elemental Analysis	Method	EFB Char (wt. %)
Carbon	ASTM D 5373	76.17
Hydrogen	ASTM D 5373	2.49
Oxygen	By difference	11.41
Nitrogen	ASTM D 5373	0.65
Sulphur	ASTM D 4239	0.33
Ash Content	ASTM D 3174	8.95
H/C molar ratio	Calculation	0.39
O/C molar ratio	Calculation	0.11
N/C molar ratio	Calculation	0.01
S/C molar ratio	Calculation	0.002
Empirical formula	Calculation	$CH_{0.39}O_{0.11}N_{0.01}S_{0.002}$

When the qualities of char from oil palm trunks (OPT) [13] and sawdusts [14] are compared with the EFB char produced, it is found that EFB char has a higher fixed carbon content and a lower ash content than OPT char but it has a lower fixed carbon content than sawdust char. Even so, it is possible to use the EFB char in certain applications such as barbecue fuel when this more friable char is briquetted with a binder.

Characterization of the Condensed Products

The EFB oil selected for analysis in this study were those obtained at a pyrolysis temperature of 600 ^{o}C and a heating rate of 10 ^{o}C min^{-1}. Besides issues of production, storage, transport and upgrading, the bio-oil has a number of special features and characteristics, which require consideration in any application. Viscosity, density, flash point, pour point and heating value are known to be typical key properties for combustion applications in boilers, furnaces and engines [15].

The results of the oil properties and calorific values for the EFB oil are tabulated in Table 3 which also shows corresponding values of No. 6 fuel oil.

Table 3 shows that EFB oil has a flash point and a pour point those are higher than No. 6 fuel oil. This higher flash point suggests that the EFB oil can be safely stored at room temperature. On the other hand, the calorific value of EFB oil is only about 68 % that of No. 6 fuel oil though its viscosity is very much lower. EFB oil is acidic while No. 6 fuel oil is neutral. As such the data of Table 3 appear to suggest that EFB oil may not be a very good substitute for No. 6 fuel oil. The data on the elemental analysis of the EFB oil are also compared to those from hardwood in Table 4.

Table 3. Properties of EFB oil and No. 6 fuel oil. Each value is the average of three runs.

Properties	Method	EFB Oil	No. 6 Fuel Oil [12]
Colour	Visual	black	black
Acidity	pH meter	3.1	neutral
Water content (% wt)	Karl Fisher	4.9	nil
Density at 30 ^{o}C (kg m^{-3})	ASTM D 1298	0.85	0.98
Viscosity at 50 ^{o}C (cSt)	ASTM D 445	13.3	65*
Flash point (^{o}C)	ASTM D 93	> 95	> 65
Pour point (^{o}C)	ASTM D 97	24	15-30
Ash (%wt)	ASTM D 482	0.10	nil
Higher calorific value (MJ kg^{-1})	ASTM D 5865	28.92	42.30

*From reference [16].

Table 4. A comparison of the results of the elemental analysis of EFB oil with bio-oil from hardwood. Each value is the average of three runs.

Elemental Analysis (wt. %)	Method	EFB Oil	Bio-oil from Hardwood [17]
Carbon	ASTM D 5373	58.23	55.5
Hydrogen	ASTM D 5373	7.21	6.7
Oxygen	By difference	32.25	37.7
Nitrogen	ASTM D 5373	1.69	0.1
Sulphur	ASTM D 4239	0.62	0.00
H/C	Calculation	1.49	1.45
O/C	Calculation	0.42	0.51
N/C	Calculation	0.02	0.002
S/C	Calculation	0.004	0.00
Empirical formula	EFB Oil: $CH_{1.49}O_{0.42}N_{0.02}S_{0.004}$		
	Bio-oil from hardwood: $CH_{1.45}O_{0.51}N_{0.002}$		

Table 4 shows the elemental composition of the EFB oil and bio-oil from hardwood. It was found that while the carbon, hydrogen and oxygen contents do not vary much, the nitrogen and sulphur contents of EFB oil are somewhat higher. Even so, as a whole, these two types of oil are quite similar and indicate that EFB could produce a bio-oil comparable to that from hardwood. Anyway the high oxygen content and acidity of these bio-oils will affect their fuels quality [12].

From GCMS studies on the aqueous fraction of condensates, the fraction was found to consist of about 80 % water and some acetic acid, methanol, acetone, methyl acetate, phenol, acetic acid ethenyl ester, soluble tar and other trace components. The average density of this brownish fraction was found to be around 0.98 g cm^{-3} and it has a pH of 3 indicating that it is acidic and corrosive.
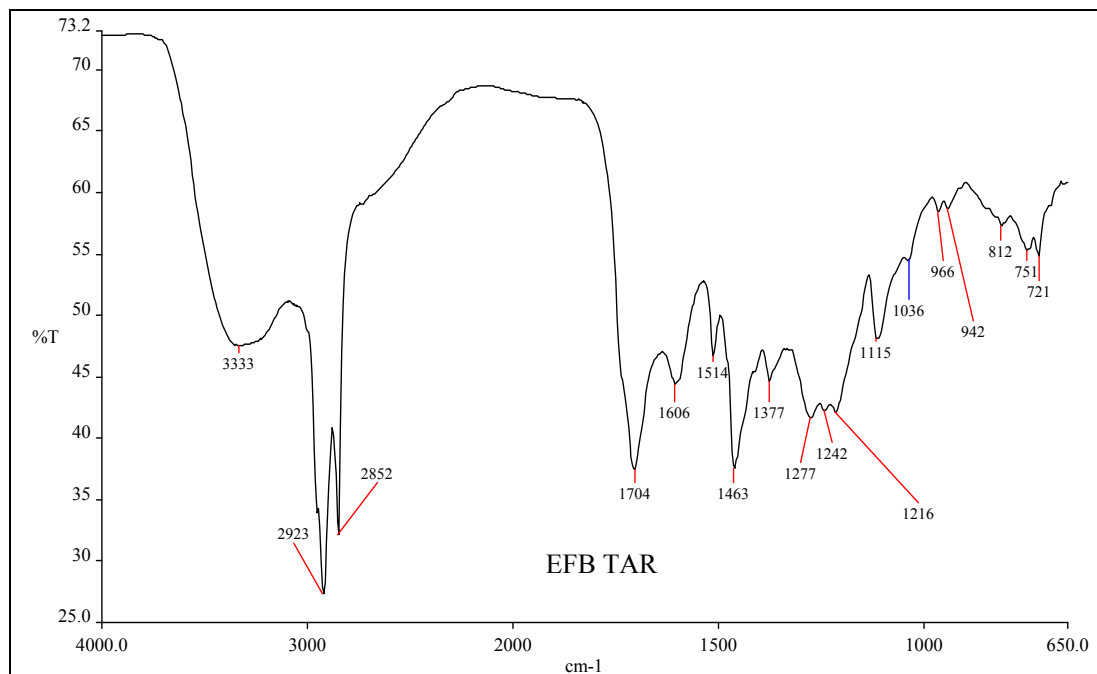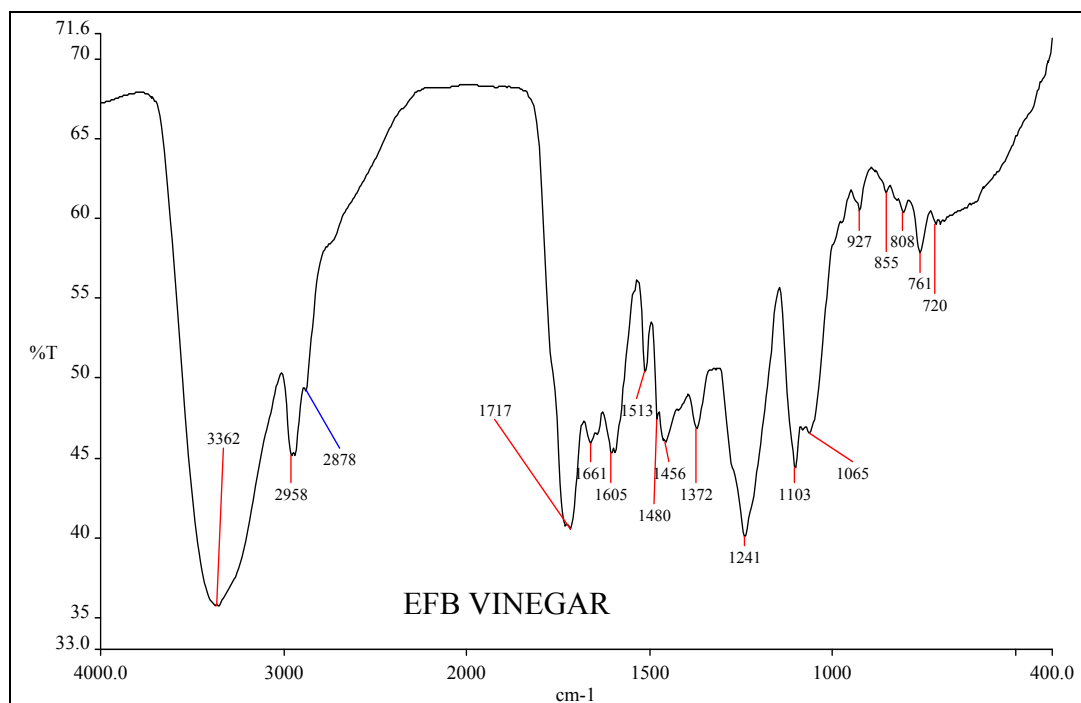

Fig. 9(a). FTIR spectra of the EFB oil fraction.

Fig. 9(b). FTIR spectra of the aqueous fraction.

Table 5. FTIR functional group compositions of the EFB oil and aqueous fraction

Wave number range (cm^{-1})	Wave number (cm^{-1}) EFB oil	Aqueous Fraction	Group	Class of compound
3300-3600	3333	3362	O-H stretching	Polymeric O-H and water impurities
3050-2800	2923, 2852	2958, 2878	C-H stretching	Alkanes
1750-1650	1704	1717, 1661	C=O stretching	Ketones, aldehydes and carboxylic acids
1650-1580	1606	1605	C=C stretching	Alkenes
1550-1490	1514	1513, 1480	NO$_2$ stretching	Nitrogenous compound
1470-1350	1377, 1463	1456, 1372	C-H bending	Alkanes
1300-950	1277, 1242, 1216, 1115, 1036, 966	1241, 1103, 1065	C-O stretching	Primary, secondary and tertiary alcohol
			O-H bending	Phenol, ester and ethers
915-650	812, 751, 721	855, 808, 761, 720		Aromatic compounds

Figures 9 (a) and (b) show the FTIR spectra of the EFB oil and aqueous fraction for wave numbers 4000 to 400 cm^{-1}. A thin uniform layer of the sample was placed between two plates of KRS-5 (Thallium Bromoiodide) and exposed to the IR beam. The transmission frequency spectra was recorded and plotted. Standard IR spectra of hydrocarbons were used to identify the functional group compositions in both fractions of condensates. The results are presented in Table 5.

From the FTIR spectra, it was found that the functional groups in the EFB oil are almost similar to those in the aqueous fraction but the latter has higher water and alcohol contents. Both sets of the spectral data of Figure 9 and the entries in Table 5 show that alcohol, ketone, ester and carboxylic acid groups are the major oxygenated compounds present in the aqueous fraction but the EFB oil contains more of phenol, ester and ether compounds. The EFB oil has a high content of hydrocarbons while the aqueous fraction contains only a moderate concentration of hydrocarbons. Both have traces of aromatic compounds. According to Soltes and Elder, phenol is the most valuable compound that is derived from the lignin component of wood [18]. EFB oil and the aqueous fraction may be good sources of phenol when compared to that from oil palm shell as reported by [19].

4. CONCLUSION

The pyrolysis of biomass is a promising technology as solid char, bio-oil, aqueous solution and combustible gases can be produced. In this study, slow pyrolysis of oil palm EFB were carried out in a laboratory-scale pyrolyser at 8 different terminal temperatures and two heating rates of 5 ^{o}C min^{-1} and 10 ^{o}C min^{-1}. The results indicate that EFB is a reasonable material for solid biofuel production and the condensates produced may be used as low quality liquid fuel and/or for the extraction of valuable chemicals.

The main product was EFB char which has a fixed carbon of 74.8 % and an energy content of 28.61 MJ kg^{-1} when the terminal pyrolysis temperature was 550 ^{o}C and the heating rate 5 ^{o}C min^{-1}. The yield of char was found to be slightly greater at 5 ^{o}C min^{-1} than at 10 ^{o}C min^{-1}. The char is of medium quality as the ash content is on the high side. Even so, it is possible to use the product as barbecue fuel when this rather friable charcoal is briquetted with a binder [8], [20].

A maximum EFB oil yield was obtained at a terminal pyrolysis temperature of 600 ^{o}C when the heating rate was 10 ^{o}C min^{-1}. The EFB oil has an energy content of 28.92 MJ kg^{-1}. The properties of this product were studied and characterized by elemental analysis and spectroscopic techniques. It was found that EFB oil may be a low quality fuel source as well as a source for chemicals and that the aqueous fraction can be a good source of ketones and acidic compounds as well as of phenols. In addition, the gaseous products are the non-condensable gases which can be used as a fuel supplement for the pyrolysis process.

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