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LABORATORY-SCALE PYROLYSIS OF OIL PALM PRESSED FRUIT FIBRES

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ABSTRACT

The slow pyrolysis of oil palm pressed fruit fibres (PFF) was investigated in a laboratory-scale pyrolyser at terminal temperatures of 450°C to 800°C and a heating rate of 10°C min^{-1}. The PFF was first pyrolysed in a thermogravimetric analyser (TGA) to investigate their thermal characteristics and to determine their kinetic parameters. Results from the laboratory-scale pyrolysis showed that as the terminal temperature increased, the yields of solid char and total condensates decreased, but the non-condensable gases increased. The quality of the char produced, which was found to be dependent on the terminal pyrolysis temperature, was of medium grade, as its average ash content of 16.60% was high. The best PFF char with 69.91% fixed carbon and an energy content of 27.07 MJ kg^{-1} was obtainable at a terminal pyrolysis temperature of 600°C. The chemical characterization of the liquid products, which separate into two fractions, showed that they may be a potential source of valuable fuel and chemical feedstocks.

Keywords: pressed fruit fibres (PFF), slow pyrolysis, PFF char, PFF oil.

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INTRODUCTION

With the depletion of fossil fuels and concerns over carbon dioxide emissions, renewable biomass is now being considered as an important energy resource all over the world including Malaysia (Lim, 2000). Indeed, there are a number of biomass sources being considered as potential sources of fuels and chemical feedstocks. The interest in using biomass and its products as a fuel arose during the 1970s due to the increase in conventional fossil fuel prices. Due to this fact, pyrolysis has attracted considerable attention all over the world, and bio-oil is of particular interest also. Among the processes of energy production from biomass, pyrolysis is the most promising thermal conversion and even incurs low cost. The direct products are char, liquid and gas fuels, which can be obtained simultaneously at moderate pyrolysis temperatures. Moreover, the solid and liquid products are easier to handle, store and transport, and can be upgraded into better quality fuels. However, the technology has still not been used for the disposal of biomass from palm oil mills.

In Malaysia, the oil palm is one of the most important commercial crops, supplying palm oil for household and commercial consumption since 1917. There are presently 4.30 million hectares of oil palm plantations, which in turn produce over 100 million tonnes of biomass annually in the form of trunks (OPT) and fronds (OPF) from the fields; and also empty fruit bunches (EFB), shells (OPS) and pressed fruit fibres (PFF) from the mills (MPOB, 2007). For every tonne of fresh fruit bunches processed, 220 kg of EFB, 670 kg of mesocarp fibre, 70 kg of OPS, and 30 kg of palm kernel cake are produced. It has also been estimated that from 1 ha of land, about 1.63 t of dry PFF and 1.10 t of dry OPS are generated (Chan, 1999). Thus, large quantities of biomass, of the order of hundreds of tonnes of dry matter per annum, are produced. Almost 80% of this biomass is used directly as fuel for the boilers to generate heat and power in the mills.

Currently, some of the PFF are used for the production of boards, roof tiles, pulp and paper, animal feed, and activated carbon (Chan, 1999; Wan *et al*., 2007). Even so, large quantities of PPF

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are still available from the mills. Information on the use of PFF as biofuel is still lacking, although a preliminary study on the briquetting of PFF with OPS had been done by Husain *et al*. (2002). Hence, projects were initiated at the Bioenergy Laboratory, Universiti Sains Malaysia, to explore the feasibility of converting not only the PFF, OPS (Khor *et al*., 2008c), and EFB (Khor and Lim, 2008b), but also the OPF (Khor and Lim, 2006) and OPT (Khor and Lim, 2008a) from the fields into more versatile forms of biofuel and chemicals. This article reports on the study on PFF. The thermochemical conversion method adopted for the study was slow pyrolysis, and the characteristics and properties of the products were investigated.

EXPERIMENTAL

Raw Materials

Astimar *et al*. (2002) reported that PFF, a lignocellulosic waste, consist of 32.4% cellulose, 38.2% hemicellulose and 20.5% lignin. Each of these components will contribute towards the formation of volatiles, gases and char when the lignocellulose is pyrolysed.

Figure 1 shows a picture of the fresh PFF which were procured from United Oil Palm Industry Sdn Bhd in Nibong Tebal. The PFF are a by-product from the mesocarp of the oil palm fruits after the oil has been extracted. They are fibrous, bulky and have a high moisture content of about 42%. Prior to use, the PFF were dried under the hot sun for five days, after which time their weight remained constant so that only an inherent moisture content of less than 10% was left. Proximate analysis, elemental composition and calorific value determination according to ASTM procedures (ASTM International, 2008) were done to investigate the characteristics of PFF as a feedstock for the pyrolysis process.

Thermal and Kinetic Studies

A pre-study of the thermal behaviour of PFF and the kinetics of their pyrolysis would serve as a useful guide for determining suitable pyrolysis parameters for subsequent use. As such, particulated PFF samples weighing about 10 mg were heated in a dynamic nitrogen gas flow of 30 $cm^3 min^{-1}$ over a temperature range from ambient to 900°C. A thermogravimetric analyser (TGA/SDTA851) as per ASTM D 3850 was used to plot thermogravimetric (TG) and derivative thermogravimetric (DTG) curves which indicate the fractional weight loss of matter in the raw sample in relation to temperature and time.

Pyrolysis Reactor

Slow pyrolysis of the PFF was carried out using a laboratory-scale pyrolyser. The pyrolyser was basically a cylindrical stainless-steel container. This sample holder had 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 110 g of dry (moisture contents < 10%) PFF packed inside this pyrolyser. The emissions of the pyrolysis process were recovered using a series of condensers. The schematic diagram of the slow pyrolysis test rig is shown in *Figure 2*.

Figure 1. Bundles of fresh pressed fruit fibres (PFF).

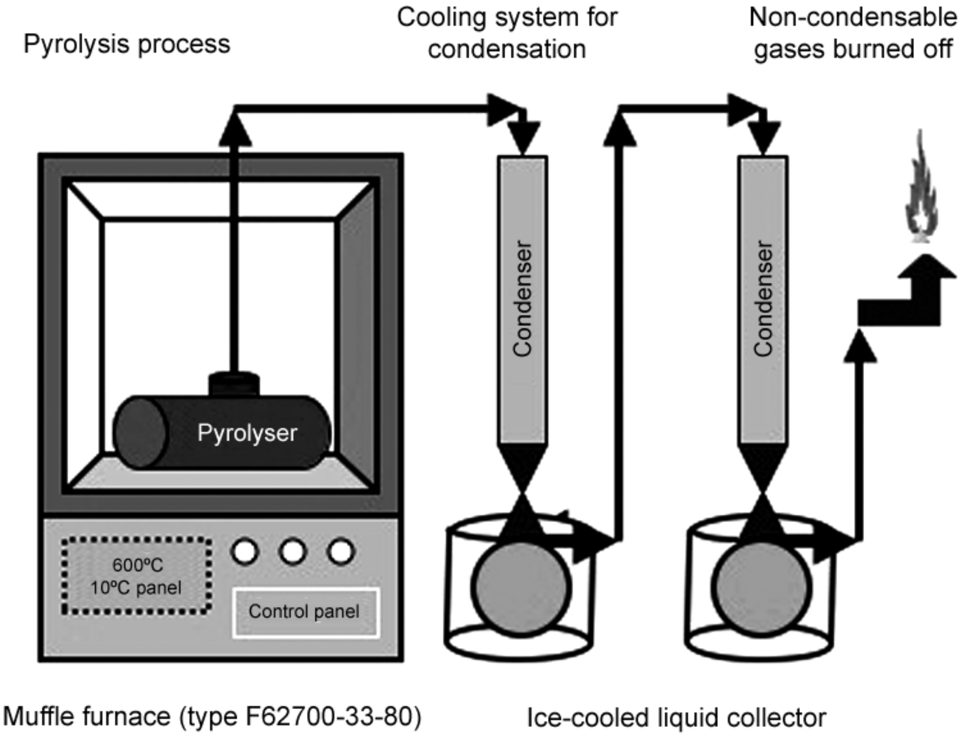

Figure 2. Schematic diagram of the slow pyrolysis test rig.

Pyrolysis Experiments

The experiments were divided into two parts. The first part was carried out to determine the effect of pyrolysis temperature on pyrolysis yields. The terminal pyrolysis temperature once attained was maintained for 1 hr, and the terminal temperature range investigated was from 450°C to 800°C, increasing in steps of 50°C, while the rate of heating used was 10°C min^{-1}. During pyrolysis, the emissions were led through an outlet port to two water-cooled condensers and the condensate was collected in two ice-cooled spherical flasks, following which there was also an outlet for the gaseous products that could not be condensed. Such gases were allowed to escape to the outside of the laboratory at the early stage, but eventually they were burned off to prevent air pollution.

The pyrolysis process resulted in a solid product of char, a liquid product and some non-condensable gases. The quantities of char produced were determined by weighing after the pyrolyser had been allowed to cool for at least 24 hr. 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 the raw material weight and the yields of char and condensate. The small quantity of condensate trapped in the connecting pipe was not determined. For each terminal temperature value, three pyrolysis runs were carried out.

The second part of the experiments involved various analyses of the products. Proximate analyses were done to determine the quality of the solid product and its elemental composition. Proximate analyses for moisture, ash and volatile contents were done as per ASTM D 3173, D 3174 and D 3175, respectively, and were further confirmed by thermal gravimetric analysis. Thermal analysis of the char was performed using a TG analyser (Perkin Elmer/TGA7) coupled with a TG controller (Perkin Elmer/TAC7/DX). Elemental analysis of the PPF char was done using a CHNS analyser (Perkin Elmer 2400 Series II). The gross calorific value of the PFF char was determined using an adiabatic oxygen bomb calorimeter (Parr Model 1261). Standard procedures were adopted for all of the analyses which were performed on the products of each experimental run.

The condensable emissions, or 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 maximum yield of tarry fraction was selected for further analysis, and it was labelled as PFF oil. The PFF oil obtained was analysed for its elemental composition and calorific value according to ASTM D 3176 and D 5865, respectively. The functional chemical groups in the tarry and aqueous fractions were identified by their Fourier Transform Infra-red spectra obtained by using a FT-IR spectrometer (Perkin Elmer FT-IR 2000). Standard procedures for the operation of the above spectrometer were followed.

RESULTS AND DISCUSSION

Table 1 indicates that oven-dried raw PFF had only inherent moisture left, had a rather high volatile content and a low amount of fixed carbon. This higher percentage of volatiles usually resulted in more liquid pyrolysis products. The ash content, however, appeared to be of medium level. As expected, the gross calorific value of the raw PFF was lower than that of coal (~24 MJ kg^{-1}). Results of the elemental analysis indicate that PFF is rather environment friendly, with small amounts of nitrogen and sulphur.

Although the chemistry of biomass is quite complex, it is generally assumed that biomass has three major constituents – hemicellulose, cellulose and lignin – according to the mass loss curve from thermogravimetric analysis. The TG and DTG profiles for PFF recorded for the temperature range of 30°C to 900°C, with a heating rate of 10°C min^{-1}, are shown in *Figure 3*. According to the TG curve, the main pyrolysis reactions included depolymerization, decarboxylation and cracking, which took place over a temperature range of 200°C -700°C.

The TG plot presented in *Figure 3* shows that heat propagated into the raw PFF and drove off the inherent moisture, which was about 9.65% of the

TABLE 1. COMPOSITION OF THE RAW PRESSED FRUIT FIBRES (PFF)
(values shown are the averages of three runs)

Proximate analysis (wt. %) (sun-dried)			Elemental analysis (wt. %) (sun-dried)	
Moisture	ASTM D 3173	9.02	Carbon	45.18
Volatiles	ASTM D 3176	71.34	Hydrogen	5.52
Ash	ASTM D 3175	7.13	Nitrogen	1.10
Fixed carbon	By difference	12.51	Sulphur Oxygen (by difference)	0.35 40.72
Higher calorific value	ASTM D 5865	18.76 MJ kg^{-1}	H/C molar ratio O/C molar ratio	1.466 0.676
Empirical formula	$CH_{1.466}O_{0.676}N_{0.021}S_{0.003}$		N/C molar ratio S/C molar ratio	0.021 0.003

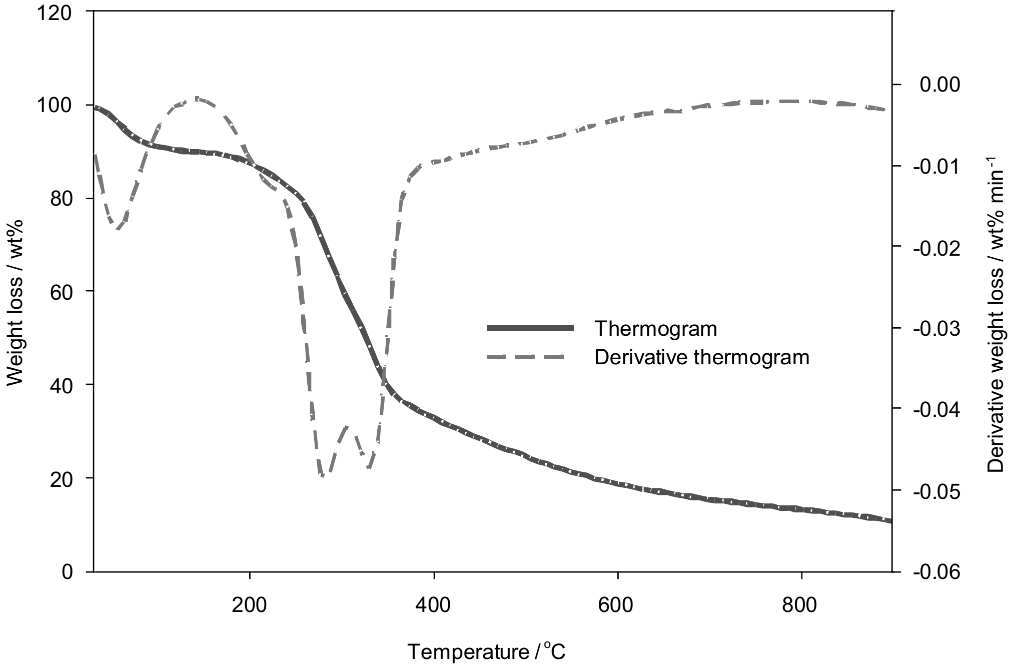

Figure 3. Thermogravimetric (TG) and derivative thermogravimetric (DTG) plots for pyrolysis of pressed fruit fibres (PFF) at a heating rate of 10°C min^{-1}.

sample weight, at about 110°C. This is comparable to the data in *Table 1*. At a heating rate of 10°C min^{-1}, thermal degradation of raw PPF was initiated at approximately 200°C, and the rate was maximum between 250°C and 300°C. At a temperature of about 600°C, the devolatization process of the sample was almost complete. The residue as char was left about 20% of the sample weight. From these data, it can be concluded that the operation of a pyrolysis system at a terminal temperature of not much more than 700°C should result in a reasonably high yield of char.

The TGA results for PFF showed two main regimes of weight loss: the lower temperature regime (200°C-300°C) could be correlated with the decomposition of hemicellulose and the initial stages of cellulose decomposition, while the upper temperature regime (300°C-400°C) correlated mainly with the later stages of cellulose decomposition. Lignin thermal decomposition occurred throughout the temperature range of pyrolysis (200°C-900°C). From the DTG curve in *Figure 3*, two distinct peaks at temperatures of around 280°C (at a devolatilization rate of 0.0482 weight loss min^{-1}) and 330°C (at a devolatilization rate of 0.0473 weight loss min^{-1}) corresponded to the thermal decomposition of hemicellulose and cellulose, respectively.

The kinetic parameters of thermal decomposition were calculated by the Arrhenius equation, using a three-step consecutive reaction model (Guo and Lua, 2001):

$$\frac{d\alpha}{dt} = k(1\text{-}\alpha)^n \tag{1}$$

where α is defined as $(w_0 - w)/(w_0 - w_f)$, while w_0 is the initial mass of the sample, w is the actual mass at time t, w_f is the mass of residue at the end of the reaction, t is the reaction time, n is the reaction order, and k is the rate constant of reaction which is defined as:

$$k = Ae^{-E/RT} \tag{2}$$

In equation 2, A is the pre-exponential factor, E is the apparent activation energy of the decomposition, R is the ideal gas constant, and T is the temperature. Both parameter values, A and E with their n-order of reactions, and also the thermal degradations at different temperature regimes are listed in *Table 2*.

Based on the above data, the actual pyrolysis experiments using the laboratory-scale pyrolyser were then carried out under the conditions indicated above in the section of Pyrolysis Experiments. For these experiments, the yield of char was defined as:

$$\frac{\text{weight of PFF char produced}}{\text{weight of dry raw PFF used}}$$

and the yield of condensates is defined as:

$$\frac{\text{weight of PFF condensates collected (whether tarry or aqueous fraction)}}{\text{weight of dry raw PFF used}}$$

The data in *Table 3* indicates that an increase in the pyrolysis temperature led to a decrease in char and total condensate (at 450°C-600°C) yields but an increase in the yield of non-condensable gases. The decrease in the char yield with increasing temperature could either be due to a greater primary decomposition of the PFF at higher temperatures or through the secondary decomposition of the char. The secondary decomposition of the char at higher temperatures might also have produced non-condensable gaseous products, thus contributing to the increase in the non-condensable gas yield as the pyrolysis temperature increased (Klass, 1998). The total condensate yield for all runs was nearly 40%, with a higher yield of 42.03% at 450°C, but it was almost constant at higher temperatures, with the tarry fraction being about 40% of the total condensates collected.

Figure 4 shows the results of the proximate analysis done on the PFF char. Not much dependence on terminal temperature was observed for the moisture and ash contents, although the latter showed a slight increase with increasing terminal temperature. Average values of 5.27% moisture content and 16.60% ash were found. As the products were allowed two days to equilibrate with the laboratory environment, the moisture content of the char is perhaps of little significance. The ash content increased by more than two times when compared to the value for the raw PFF. The fixed carbon content increased as the terminal temperature

TABLE 2. KINETIC PARAMETERS OF THE THERMAL DECOMPOSITION OF PRESSED FRUIT FIBRES (PFF)

Kinetic parameter	Temperature zone (°C)			
	Entire reaction zone	**200-300**	**300-400**	**400-700**
A (s^{-1})	2.96E+5	2.29E+6	3.13E+12	4.94E+11
E (kJ mol^{-1})	87	90	17	203
n	2.5	1.0	2.0	3.5
R^2	0.9284	0.9837	0.9850	0.8100
Total degradation (%)	84.55	27.29	27.51	16.71

TABLE 3. A COMPARISON OF PYROLYSIS PRODUCT YIELDS FOR VARIOUS PYROLYSIS TEMPERATURES (values shown are the averages of three runs)

Terminal temperature of pyrolysis (°C)	Char yield (wt. %)	Condensate			Non-condensible gases (wt. %)
		Tarry fraction	Aqueous fraction	Total	
450	34.82	15.99	26.04	42.03	23.15
500	33.10	16.61	22.27	38.88	28.02
550	31.10	14.08	23.45	37.53	31.37
600	30.64	14.39	21.83	36.22	33.14
650	29.15	15.33	20.85	36.18	34.77
700	28.85	14.61	21.45	36.06	35.09
750	28.13	17.25	18.77	36.02	35.85
800	25.89	15.03	22.22	37.25	36.86

increased, because more volatiles were driven off at elevated temperatures. However, the increase was not significant once a terminal temperature of about 600°C was reached. As such, it may not be necessary to carbonize the PFF beyond 600°C. This is in agreement with the conclusion drawn from the TGA studies.

Table 4 shows the elemental composition and calorific value of the char at varying terminal temperatures of pyrolysis. Although there was a slight drop in the carbon content beyond 600°C, it generally increased as the terminal temperature increased, while the hydrogen content decreased because of dehydrogenation reactions (Strezov *et al*., 2007). The percentage of the carbon content tallied well with the values found for the fixed carbon content. However, the nitrogen content did not exhibit any clear trend in relation to terminal temperature. In line with the carbon content, the calorific value increased when the terminal temperature increased, except for a slight drop at 650°C and beyond.

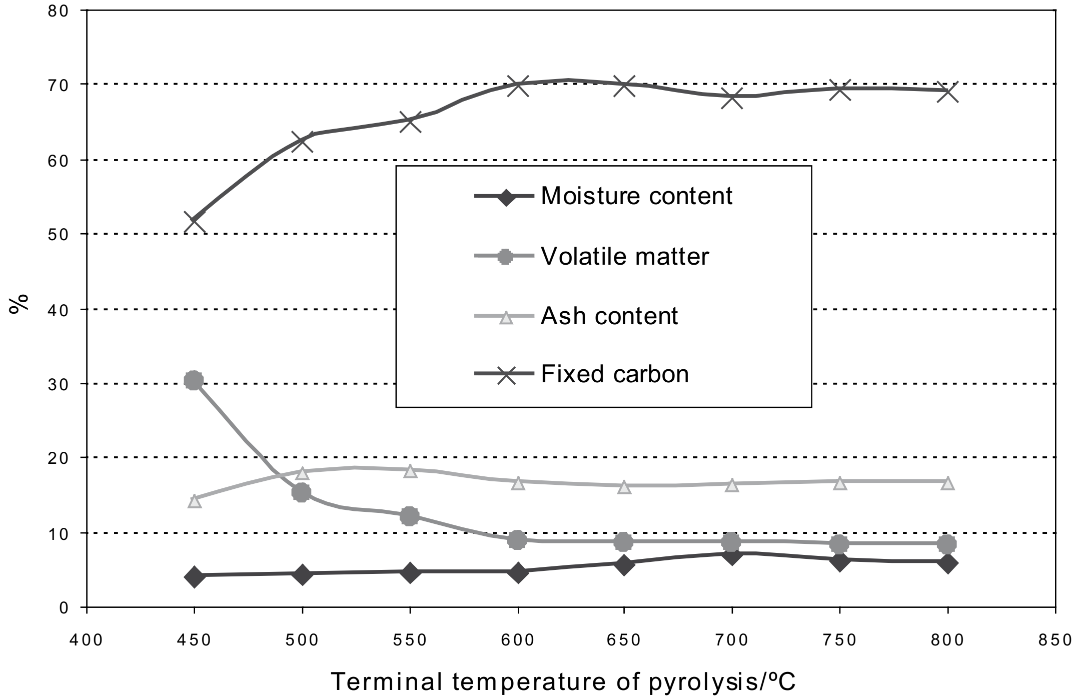

Figure 4. Proximate analysis of pressed fruit fibres (PFF) char at various pyrolysis temperature (points shown are averages from three runs).

TABLE 4. ELEMENTAL ANALYSIS OF PRESSED FRUIT FIBRES (PFF) CHARS AND THEIR CALORIFIC VALUES
(values shown are the averages from three runs)

Sample	Terminal temp. of pyrolysis (°C)	Elemental analysis (dry, ash and sulphur-free basic)						Higher calorific value (MJ kg^{-1})
		C (wt. %)	H (wt. %)	N (wt. %)	O (wt. %) by diff.	O/C molar ratio	H/C molar ratio	
1	450	65.22	2.59	1.49	30.70	0.3530	0.4765	23.54
2	500	66.63	2.08	1.74	29.55	0.3326	0.3746	24.97
3	550	68.80	1.53	1.34	28.33	0.3088	0.2669	25.53
4	600	72.43	1.30	1.44	24.83	0.2571	0.2154	27.07
5	650	71.37	1.04	1.36	26.23	0.2756	0.1749	26.89
6	700	70.04	0.98	1.42	27.56	0.2951	0.1679	26.59
7	750	69.54	0.76	1.43	28.27	0.3049	0.1311	26.67
8	800	69.26	0.64	1.46	28.64	0.3101	0.1109	26.37

The atomic ratios H/C and O/C are often used to characterize conventional fossil fuels. The ratios of H/C versus O/C for chars generated at different temperatures are presented in *Figure 5*. It is obvious that the H/C and O/C ratios of all the chars were much lower than that of the raw material, and char No. 4 had the lowest O/C, but the lowest H/C belonged to char No. 8. A comparison of the chars with the O/C and H/C values for coal showed that all the chars, especially those obtained at higher temperatures, corresponded to the regions occupied by anthracite, bituminous coal and lignite (McKendry, 2002).

The results relating to the quality of the PFF chars, as discussed above, indicate that the char produced at a terminal pyrolysis temperature of 600°C was of fairly good quality with a fixed carbon content of 69.91% (or 72.43% carbon content) and a calorific value of 27.07 MJ kg^{-1}, but the ash content of 16.67% was on the high side. Even so, depending on the application, the product was still judged as being suitable for use in barbecues after briquetting.

The condensates collected, even after 2 hr of standing, were found to be not homogeneous as two fractions – one fraction consisting of insoluble tar and the other of a less oily aqueous fraction – were observed. These fractions were separated by filtration, as most of the tarry substance could not get through the Whatman No. 1 filter paper. This observation is similar to that reported by Soltes and Elder (1981) when wood was pyrolysed.

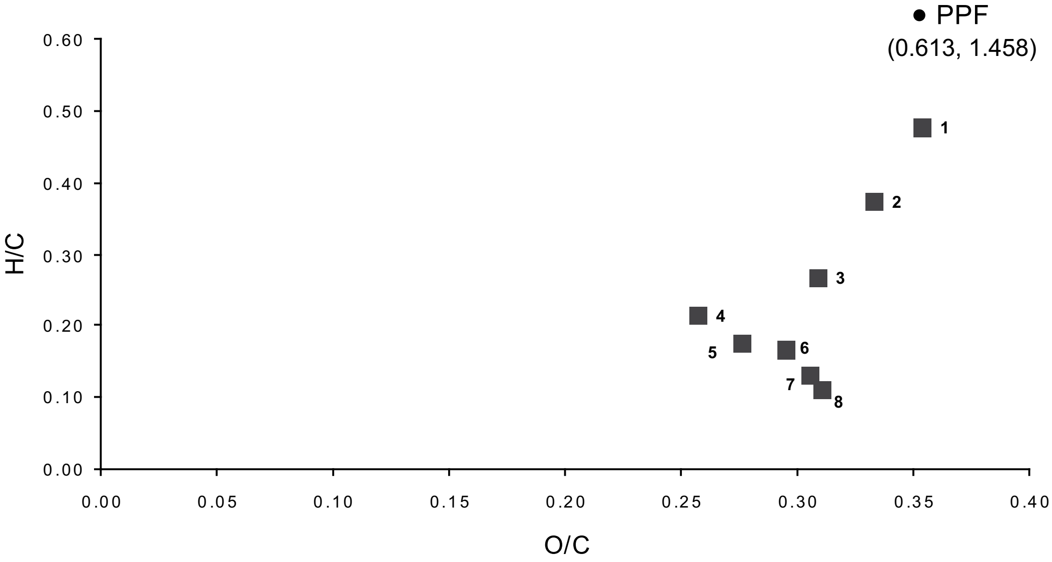

Figure 5. Van Krevelen diagram for raw pressed fruit fibres (PFF) and its chars obtained at different terminal temperatures (numbers 1 to 8 correspond to the sample numbers in Table 4*).*

The pH value of the aqueous fraction was about 3, indicating that it was quite acidic. It had an average density of 1015 kg m^{-3} and consisted of numerous chemical compounds that could be identified using a GC/MS. The main constituents may be similar to wood vinegar, which has many applications in agriculture such as fertilizer, for soil improvement, *etc*.

The PFF oil had a gross calorific value of 28.35 MJ kg^{-1}, and can be used as a liquid fuel. Therefore, the CHNS contents were also determined, and the data were compared to that of bio-oil obtained from hardwood (Solantausta *et al*., 1993). The comparison is tabulated in *Table 5*.

Table 5 shows that the carbon, hydrogen, nitrogen and sulphur contents of PFF oil were somewhat higher than hardwood bio-oil, while only the oxygen content was lower. Even though PPF oil appeared to be of slightly higher quality, these two types of oil were rather similar, which indicates that PFF could be used to produce a bio-oil comparable to that from hardwood. However, the high oxygen content of these bio-oils will affect their fuel quality (Zhang *et al*., 2007).

The FT-IR spectra of both the tarry and aqueous fractions of the condensates are shown in *Figures 6* and *7*, respectively. The spectral data show that alcohol, ketone, ester and carboxylic acid groups were the major oxygenated compounds present in both the tarry and aqueous fractions. Both also contained a moderate concentration of hydrocarbons, but their quantities in the tarry fraction were higher. Traces of aromatic ring compounds were found in the aqueous fraction. Even though both FT-IR spectra had rather similar functional groups, the water content in the aqueous fraction was definitely very high. The functional group compositions for both fractions as presented in *Table 6* indicate that the fractions may be fairly good sources of chemicals.

TABLE 5. A COMPARISON OF THE RESULTS OF ELEMENTAL ANALYSIS OF FRESSED FRUIT FIBRES (PFF) OIL WITH BIO-OIL FROM HARDWOOD
(each value for the PFF oil is the average of three runs)

Elemental analysis (dry, ash-free basis)	Method	PFF oil (wt. %)	Bio-oil from hardwood* (wt. %)
Carbon	ASTM D 5373	58.31	55.5
Hydrogen	ASTM D 5373	7.05	6.7
Oxygen	By difference	32.47	37.7
Nitrogen	ASTM D 5373	1.72	0.1
Sulphur	ASTM D 4239	0.45	0.00
H/C	By calculation	1.451	1.45
O/C	By calculation	0.418	0.51
N/C	By calculation	0.025	0.002
S/C	By calculation	0.003	0.00
Empirical formula	By calculation	$CH_{1.451}O_{0.418}N_{0.025}$	$CH_{1.45}O_{0.51}N_{0.002}$

Note: *Data from Solantausta *et al*. (1993).

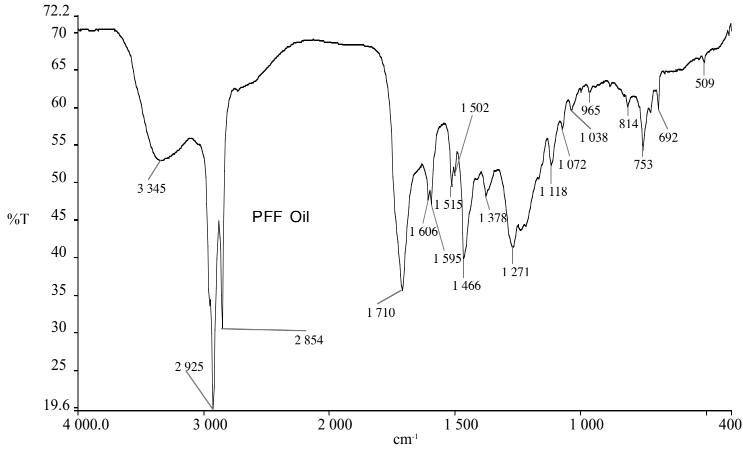

Figure 6. FT-IR spectra of the tarry fraction.

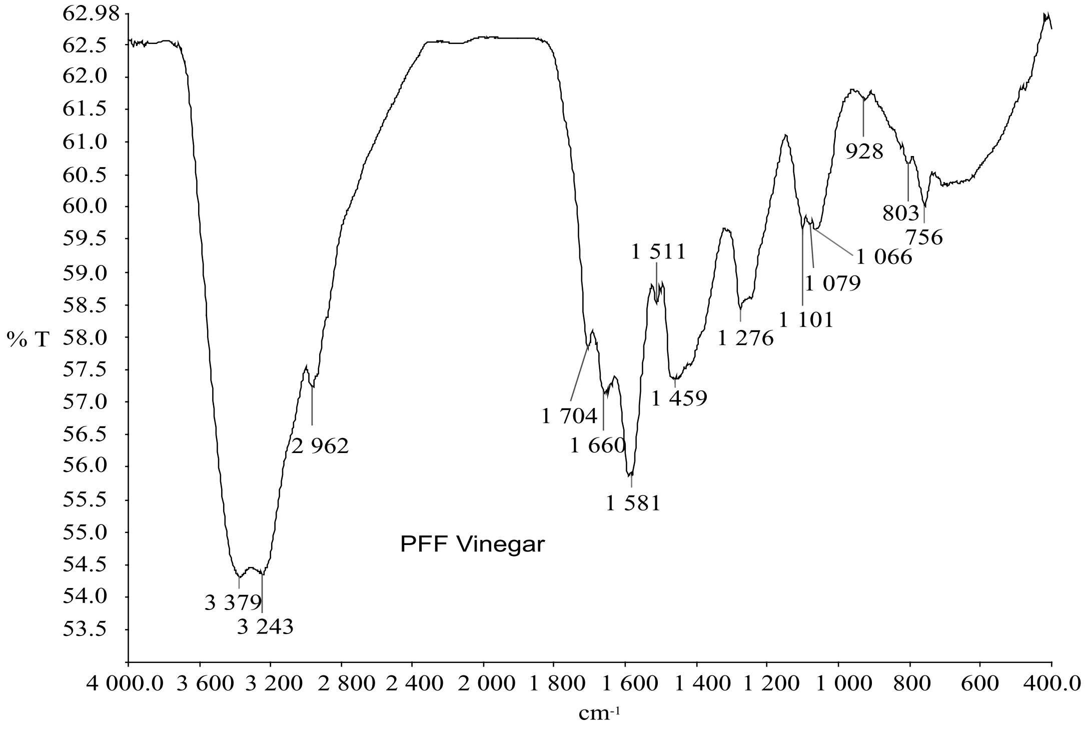

Figure 7. FT-IR spectra of the aqueous fraction.

TABLE 6. FT-IR FUNCTIONAL GROUP COMPOSITIONS OF THE TARRY AND AQUEOUS FRACTIONS FROM THE PYROLYSIS OF FRESSED FRUIT FIBRES (PFF)

Wave number range (cm^{-1})	Wave number (cm^{-1})		Group	Class of compound
	Tarry fraction	Aqueous fraction		
3 300 -3 600	3 345	3 379, 3 243	O-H stretching	Polymeric O-H, water impurities
3 050 -2 800	2 925, 2 854	2 962	C-H stretching	Alkanes
1 750 -1 650	1 710	1 704, 1 660	C=O stretching	Ketones, aldehydes, carboxylic acids
1 650 -1 580	1 606, 1 595	1 581	C=C stretching	Alkenes
1 550 -1 490	1 515, 1 502	1 511	NO_2 stretching	Nitrogenous compounds
1 470 -1 350	1 466, 1 378	1 459	C-H bending	Alkanes
1 300 -950	1 271, 1 118, 1 072, 1 038, 965	1 276, 1 101, 1 079, 1 066, 928	C-O stretching O-H bending	Primary, secondary and tertiary alcohols, phenols, esters, ethers
915 -650	814, 753, 692, 509	803, 756	C-H deformation	Aromatic compounds

CONCLUSION

1. Work on the slow pyrolysis of PFF was carried out in a laboratory-scale pyrolyser at eight different pyrolysis temperatures with a heating rate of 10°C min^{-1}. The pyrolysis products from PFF biomass consisted of solid char, condensable organic liquids, non-condensable gases and water. When the pyrolysis temperature was elevated, gas yield increased and the total condensate and char yields decreased.

2. The main product of PFF char with 69.91% fixed carbon and an energy content of 27.07 MJ kg^{-1} was obtainable at a terminal pyrolysis temperature of 600°C. The product obtained was of medium quality, as the ash content of 16.67% was quite high. However, it would be possible to use the product as a solid fuel where high ash content does not pose a problem. Moreover, the char could be ground easily, due to its friable nature, and then formed into briquettes, with a binder for densification.

3. The total condensate obtained at a terminal temperature of 750°C was separated into two fractions: a tarry and an aqueous fraction. Both fractions were analysed by FT-IR, and it was found that they can be sources of different ketones and acidic compounds, as well as phenolic compounds, if proper extraction processes can be adopted for the recovery of these chemicals. The PFF oil is a potential bio-oil with an energy content of 28.35 MJ kg^{-1}, and can be used as a liquid fuel. This complex mixture is highly-oxygenated, with a great amount of macro molecules, such as esters, ethers, aldehydes, ketones, phenols, carboxylic acids and alcohols.

4. The gaseous products that were non-condensable were not investigated. However, they are combustible and thus can be recycled and used as fuel in a commercial char retort.

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