Production by Pyrolysis and Analysis of Bio-oil from Mahogany Wood (*Swietenia macrophylla*)

Abstract- This work presents the production and the analysis of pyrolysis oil (bio-oil) obtained from a hard wood chips (Mahogany, *Swietenia macrophylla*). A pyrolysis batch type reactor was designed and constructed to carry out the production process of the pyrolysis oil. The yield of mahogany pyrolysis oil was optimized with reaction temperature to produce high quantity of gaseous outputs. Elemental analysis and Fourier transform infrared analysis were carried out. The FTIR analysis revealed the presence of aliphatic and aromatic compounds in the mahogany pyrolysis oil. The physio-chemical properties of mahogany pyrolysis oil were determined. During the production process, the temperatures at which the yields were obtained were recorded. This was done using a temperature regulator and a thermocouple mounted on the body of the furnace. The results showed that the flash point, pour point, gross and net calorific values are 125°C, 4, 21.96MJ/kg and 20.65MJ/kg respectively, at the end of pyrolysis. The maximum liquid yield is 60% at 450°C. The combustible organic compounds of pyrolysis oil obtained at these optimum process conditions are analyzed for some of their properties as an alternative fuel.

1. INTRODUCTION

The need for alternative fuels has recently come to the forefront of public interest, thanks to increased awareness in the global warming. The main contributor to global climate change has been carbon dioxide emissions created by burning fossil fuels. One of the proposed solutions to mitigating global climate change is fuel shifting; supplementing fossil fuels with renewable, carbon-neutral biomass derived fuels (biofuels). Different forms of liquid biofuels are being developed; ethanol, bio-diesel and bio-oil (Cooper and Alley, 2002)

Biomass is a vast renewable energy source that can be used to produce heat for home and industrial facilities, generate electricity and make transportation fuels. It is the earliest and now the third largest global source of energy, comprising up to 40% - 50% of energy usage in many developed countries (like USA) and other developing countries that have large agriculture and forest area. As a result, it has the highest potential among other alternative energy options in terms of contributing to modern society's near term energy needs (Bridgwater, 2003). The use of biomass also offers an opportunity to produce carbon neutral energy, helping to mitigate greenhouse gas emissions and the adverse effects of global climate change. Furthermore, localized biomass resources can allow nations to achieve foreign energy independence and strive toward the emissions targets (Bridgwater, 2003; Demirbas and Balat, 2006). Currently, it is estimated that 12% of the world's primary energy is supplied by biomass and with the continual consumption of viable fossil fuel reservoirs, policies such as the emissions trading scheme around Europe and the possible future implementation of carbon taxes, the utilization of biomass energy is likely to increase and play a significant role in near term energy markets (Demirbas and Balat, 2006). In order to take advantage of the current infrastructure and energy conversion technologies available for liquid fuels, raw biomass must be converted into a liquid. The thermo-chemical process for doing so is termed fast pyrolysis and other forms of conversion processes include biological and physical processes (Bridgwater, 2003).

The pyrolysis the biomass has attracted more interest in producing liquid fuel product because of its advantages in storage, transport and versatility in application such as combustion engines, boilers, turbines, among others. Pyrolysis is a thermal decomposition

49 process that occurs in the absence of air with short residence times at intermediate
50 temperatures (400 – 500°C) and with rapid quenching of vapours into a liquid 'bio-oil'
51 (Shaddix and Hardesty, 1999). It should be noted that the bio-oil feedstock (biomass) is the
52 main variable affecting the quality (Bridgwater, 2005). This technology has the capability to
53 produce bio-fuel with high fuel-to-feed ratios for competing and eventually replacing non-
54 renewable fossil fuels. However, the development of advanced technologies is the next
55 challenge for pyrolysis researchers to achieve this target. It is necessary to convert biomass
56 into liquid fuels for direct use in vehicles, trains, ships and aeroplanes to replace petrol and
57 diesel (Demirbas, 2002; Muradov and Veziroglu, 2008).

58 Bio-oil is a liquid dark brown, fuel, derived from plant material (biomass) via a thermal
59 decomposition process called fast pyrolysis. It constituted for a multicomponent mixture
60 composed of various molecules that are produced by the pyrolysis of cellulose, hemicellulose
61 and lignin. Bio-oil is most suitable as a "drop-in" replacement for heat and power
62 applications. Its production is done by treating the biomass in reactor. The biomass from
63 wood residues is fed into a reactor where it is rapidly converted into a vapour. The resulting
64 vapour passes into a cyclone where solid particles (char) are removed. The vapour then enters
65 a quench tower or condenser where it is quickly cooled turning into bio-oil. Non-condensable
66 gases are either returned to the reactor to maintain process heating or exported for other uses.
67 This process produces three products: bio-oil (60 - 75%), char (15 - 20%) and non-
68 condensable gases (10 - 20%) (Ozcimen and Karaosmanoglu, 2014). The presence of water
69 in bio-oil which lowers the heating value, viscosity, flame temperature, combustion rate
70 makes its application in power generation and engine use low demanding in many countries.
71 In general, bio-oil will separate into two distinct phases (aqueous and non-aqueous) if the
72 bio-oil contains more than 30% water (Oasmaa and Czernik, 1999). Research is currently
73 being performed to reduce water content of bio-oil by drying the feedstock prior to pyrolysis
74 (Wang *et al.*, 2008). Furthermore, availability of raw material through which the bio-oil
75 would be obtained is another major concern in the production of bio-oil when this raw
76 material (wood) is not readily available due to deforestation which has been a big challenge
77 threatening global warming (Samy and Boateng, 2010). Despite the name bio-oil, it is not an
78 oil like vegetable oil or petroleum oil because it contains up to 30% water. Bio-oil, unlike
79 ethanol and bio-diesel, is considered a second generation biofuel because the fuel can be
80 made from inedible lignocellulosic plant material using advanced thermochemical processes
81 (UN - Energy, 2007).

82 The reactor is the heart of the pyrolysis process and thermal apparatus or vessel use for
83 controlling the reaction process. This is where the reaction takes place, where biomass is
84 pyrolyzed to pyrolysis gas and char. In the reactor there are high temperatures, up to 1000 °C.
85 The temperature must not fluctuate more than 20% of the intended reactor temperature
86 because it can have effects on the quality of the char. One of the primary functions is to
87 ensure a low oxygen supply at 2%. (Schmidt et al., 2012). There are number of reactor
88 designs that can be employed and are capable of achieving the heat transfer requirements.
89 Fluidized beds; bubbling and circulating; Ablative (biomass particle moves across hot surface
90 like butter on a hot skillet); Vacuum; Transported beds without a carrier gas. Of these
91 designs, the fluidized and transported beds appear to have gained acceptance as the designs of
92 choice for being reliable thermal reaction devices capable of producing bio-oil in high yields
93 (Hariram and Mohammed, 2014). For example: a new approach in the heating system was
94 developed to recover the hydrocarbons from waste tire in a fixed bed reactor under inert
95 atmosphere. It was noticed that at 475°C, the pyrolysis reaction yielded optimum liquid/gas at
96 a vapor residence of 5 sec. The physio-chemical analysis of this bio-oil included elemental
97 analysis, gas chromatography/mass spectrometry, Fourier transform infrared analysis and

98 distillation showed the presence of hydrocarbon limonene, short and long chain alkyl,
99 aromatic hydrocarbons in various quantities (Suhanya et al, 2013).
100   Further development of pyrolysis technology is ongoing and many research articles have
101 been published on the theme in bio-oil applications in recent times. This work seek to
102 demonstrate how bio-oil is derived from plant material (biomass) via a thermal
103 decomposition process called fast pyrolysis, and can be extracted from wood; and the
104 chemical composition that makes it serve as a fuel. For the purpose of this work, pyrolysis
105 method was employed to extract bio-oil from wood (Mahogany). The resulting bio-oil was
106 characterized for some physical-chemical parameters using Elemental analysis and Fourier
107 transform infrared analysis.
108
109 **2. MATERIALS AND METHODS**
110 **2.1 Reactor Design and Operation**
111 The essential components of pyrolysis process are show in Fig. 1. The pyrolytic reactor (Fig.
112 2) was designed for waste materials of biomass, agricultural wastes, and scrap tyres, among
113 others. It consists of a batch reactor made of mild steel sealed at one end and constructed for
114 batch operation. The top of reactor can be open for feeding the raw material and solid residue
115 (char) can be removed at the end of the operation. The temperature inside the reactor is
116 measured by using thermocouple. The reactor is heated internally by an electric heater, with
117 the temperature being measured by a Cr-Al: K type thermocouple fixed inside the reactor,
118 and temperature is controlled by an external PID controller. During the reaction, the top is
119 kept closed by a cover plate tightly secured to the system, prevented ingression of
120 atmospheric air into the reactor, thereby achieving pyrolysis conditions. The pyrolyser it has
121 a coating with a clay mixed with bentonite insulation in between the thickness of the wall to
122 prevent the heat loss to the surrounding. An exit pipe at the side carries away the evolved
123 gases during pyrolysis. The load used was of 1kg of wood chips sample in each pyrolysis
124 reaction. The condensable liquid products were collected through the condenser and weighed.
125 After pyrolysis, the solid residue (left) inside the reactor was removed. Reactions were
126 carried out at different temperatures ranging from 350 – 460ºC.
127
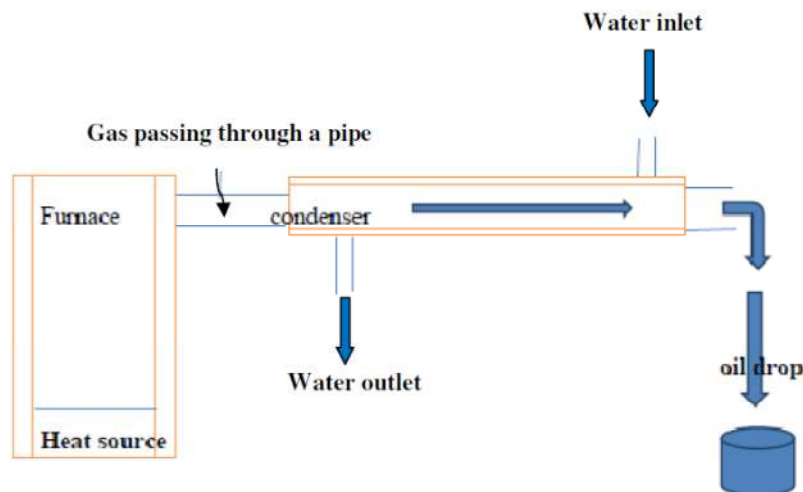
128
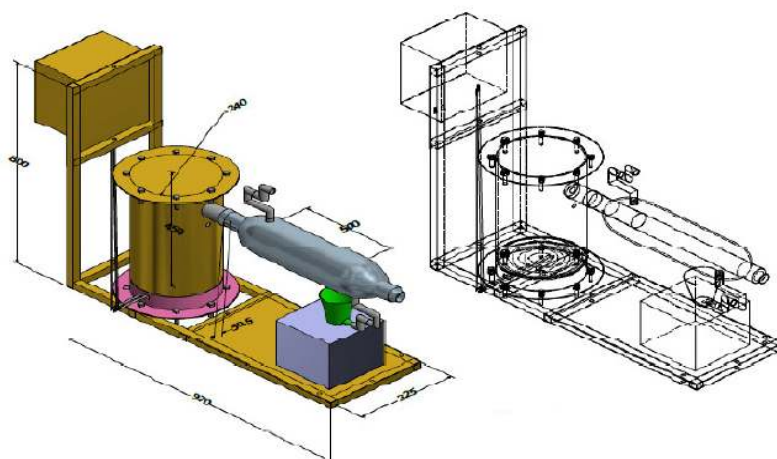129 **Fig. 1. Schematic diagram of pyrolysis reactor**

Fig. 2. Designed pyrolytic reactor setup (dimensions in "mm")

The experiments were conducted by maintaining the pyrolysis reactor under various conditions. Different temperature conditions and heating rate are required for different feed and types of pyrolysis. For slow pyrolysis temperature requirement is 250 to 500°C and heating rate is about 10 – 20 ^{o}C/min. Generally, the most important controlled process variables to be considered during start-up include: pressure and temperature. Start-up burner will be supplied to heat the reactor walls initially. The reactor vessel during start-up will not be pressurized. However, during the course of heat distribution the reactor walls via the start-up burner, gradual pressure will be added into the vacuum. The reactor process vessel will be inserted before supplying heat via the start-up burner. This will be necessary to provide conditions adequate and necessary for the pyrolysis reaction to occur efficiently without disturbances. The pyrolytic reactor design specifications are shown in Table 1.

Table 1. Design specifications of pyrolytic reactor

S/N	Components	Specifications
1.	Furnace outer dimensions	240mm x 400mm
2.	Furnace inner dimensions	230mm x 400mm
3.	Pipe size through which the hot gas passes	20mm x 250mm
4.	Condenser	20mm x 26mm
5.	Length of condenser exit to oil collector	50mm
6.	Height of the heater from furnace	9mm
7.	Length of condenser	500mm
8.	Electrical heater capacity	1.8kW
9.	Amount of feedstock by batch	1kg
10.	Required time for 1kg of sample	120minutes
11.	Operating temperature	350 – 460°C

2.2 Instrumentation

The instrumentation panel consists of a fuse unit, on-off switch, and thermometer. The temperature of the pyrolysis is measured by a thermocouple connected to a temperature indicator of 1°C accuracy. The time is measured by a digital timer of 0.01 second accuracy. The weight of input feedstock and residue after pyrolysis are measured by a weighing balance of 1g accuracy.

2.3 Design Calculations

The volume flow rate of the product gas from a desired net heating value is found by (Bridgwater, 2005):

$$V = Q \div LHV \qquad \text{Eq. 1}$$

For the reason that the volume of gases change with temperature or pressure, it is necessary to specify the temperature and pressure the flow rate was measured at. However, Eq. 1 assumes standard conditions of temperature and pressure (i.e. 1 atmosphere and $0 - 20^{o}C$) (Bridgwater, 2005).

Most pyrolytic reactions are predominantly endothermic. This Implies heat must be supplied to the reactor for these reactions to take place at the designed temperature. The amount of external heat supplied to the reactor depends on the heat requirements of the endothermic reactions as well as the pyrolysis temperature. The general energy balance equation is given by (Bridgwater, 2005).

$$Q = m \times C_p \times \Delta T \qquad \text{Eq. 2}$$

The first step of this mass balance involves resolving the heat energy content of wood supplied to the reactor. For the constant-pressure reaction, the enthalpy of the product is equal to the sum of the reactant enthalpies and any heat transfer to the system. That is (Bridgwater et al, 2001).

$$H_p = H_r + Q \qquad \text{Eq. 3}$$

This is basically the measure of time that wood particles will remain in the reactor until they are converted into products and ejected. Anticipated residence time for 1kg of feedstock is between 110-150minutes (Bridgwater, 2003).

2.4 Analyzes in the bio-oil

Physio-chemical analysis, Elemental analysis and Fourier transform infrared spectrometry analyses were carried out on the mahogany pyrolysis oil to understand its properties and characteristics for its usage in internal combustion engine. Fourier transform infrared spectrometry was used to study the FTIR pattern (Hariram and Mohammed, 2014).

2.4.1 <u>Experimental procedure for flash point</u>

The flash point of the pyrolysis oil indicates the lowest temperature at which the pyrolysis oil can form an ignitable mixture in air near the surface of the oil. To determine this, 10ml volume of the pyrolysis oil was poured into an evaporating dish and a thermometer was suspended at the centre of the dish ensuring that the bulb just dips inside the oil without touching the bottom of the dish. The temperature of the oil gradually rises using an electric stove at a temperature of $95^{0}C$. The temperature at which the oil sample gives off a bluish smoke continuously is noted as the smoke point and the temperature at which flame was observed is known as the flash point (Sipila et al, 1998).

2.4.2 <u>Experimental procedure for pour point</u>

Pour point is the temperature at which the pyrolysis oil becomes semi solid and loses its flow characteristics. To determine this 10ml of the oil sample was put in a refrigerator and allow for some time, when flow ability of the oil reduces, the temperature of the oil was taken (Sipila et al, 1998).

2.4.3 <u>Experimental procedure for calorific value</u>

Calorific value: the combustion heat of fuel is the amount of heat produced when the fuel is burned completely. The two heating (gross or high heating value and net or low heating value) are defined (Oasmaa and Kuoppala, 2003). The HHV of the sample was determined by means of the following Institute of Gas Technology (IGT) formula shown in Eq.4.

204 The amounts of elements (C, H, O, N and Ash) are expressed in mass percentages.

205 $$HHV \left(\frac{kJ}{kg}\right) = 354.68C + 1376.29H - 15.92Ash - 124.69(O + N) + 71.26 \qquad \text{Eq. 4}$$

206 The LHV is calculated from the HHV and hydrogen content of the pyrolysis oil by the
207 equation;

208 $$LHV \left(\frac{J}{g}\right) = HHV \left(\frac{J}{g}\right) - 218.13H \ (wt.\%) \qquad\qquad \text{Eq. 5}$$

209 No subtraction of free water has to be made because the water in bio-oil cannot be removed
210 by physical methods as is the case for heavy petroleum fuel oils (Oasmaa and Kuoppala,
211 2003). The bio-oil was collected with a beaker at different temperatures and placed on a
212 weighing balance. The weight of yield was taken and its percentage then was determined.
213
214 **2.4.4   Fourier Transform Infrared (FT-IR) Spectroscopy**
215 FT-IR analysis was carried out to characterize the organic functional groups in the bio-oil
216 samples. Bio-oil obtained from Mahogany wood were analyzed and each spectrum was an
217 average of 64 scans from 400-4000 cm^{-1} at 2 cm^{-1} spectral resolution. FT-IR data
218 interpretations for each spectrum were based on literature survey using extensive elucidation.
219
220 **3. RESULTS AND DISCUSSION**
221 **3.1 Results of physic-chemical parameters for pyrolitic bio-oil**
222 Table 2 shows thermal parameters and chemical composition of the pyrolytic bio-oil obtained
223 from mahogany wood.
224
225 **Table 2. Results of physic-chemical parameters for pyrolitic bio-oil**

Thermal parameters	Value	Elemental analysis (wt.%)	Value
Flash point	125 ^{0}C	Carbon	52
Pour point	4 ^{0}C	Hydrogen	6
Gross calorific value (HHV)	21.96 MJ/kg	Ash	0.03
Net calorific value (LHV)	20.65 MJ/kg	Oxygen	38
		Nitrogen	0.3

226 The elemental analysis was carried out on the mahogany pyrolysis oil to identify the presence
227 of various elements, organic and in-organic materials. It was noticed the mahogany pyrolysis
228 oil contained 52 wt.%, 6 wt.%, 0.3 wt.%, 38 wt.% and 0.03 wt.% by weight of Carbon,
229 Hydrogen, Nitrogen, Oxygen and in-organic ashes respectively as shown in Table 2.
230
231 **3.2 Results of yield for pyrolitic bio-oil**
232 Table 3 shows pyrolysis temperature and yield of the bio-oil obtained from mahogany wood.
233
234 **Table 3. Pyrolysis Temperature and Yield**

Pyrolysis temperature (^{0}C)	Yield (wt.%)
350	42
365	48
380	51
400	56
450	60
460	55

235
236 In Fig. 3, it can be observed that from the origin, at 0 ^{o}C that no bio-oil was produced, the
237 heat input to the wood was only used for the thermal transformation of the components of the
238 wood namely; cellulose, hemicellulose and lignin. As the temperature approaches 350 ^{0}C, the

239 first trace of bio-oil was observed. With further increase in the pyrolysis temperature, the
240 percentage of bio-oil yield increased rapidly with its maximum occurring at 450 ^{0}C. Above
241 450 ^{0}C, it was observed that despite the further increase in the pyrolysis temperature, that the
242 bio-oil yield did not improve as expected, rather it started diminishing. The explanation to
243 this is that above 450 ^{0}C, most of the wood particles must have pyrolysed. The system will
244 eventually get to a temperature at which no bio-oil production will be observed, showing that
245 the pyrolysis process has been completed.
246
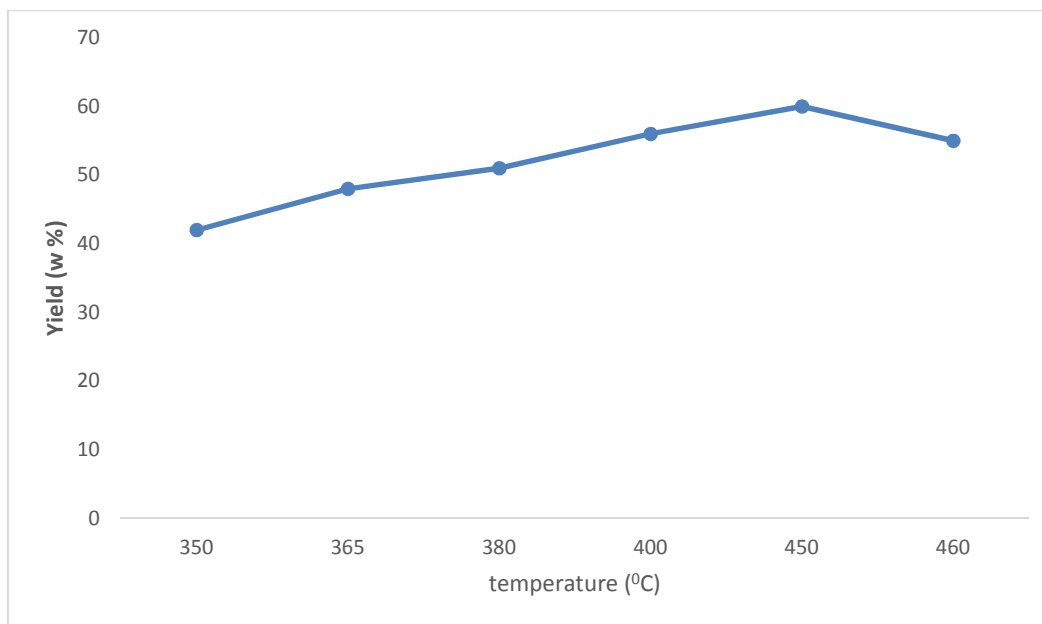
249 **Fig. 3. Variation of pyrolytic bio-oil yield with temperature**

251 **3.3 Results of analysis for infrared spectroscopic**
252 Fourier Transformed Infra-Red (FTIR) Spectroscopy is an excellent tool for functional group
253 analysis and identification of organic compounds (Silvestein et al, 2005). The spectrum
254 (FTIR) of bio-oil from Mahogany wood is shown in Fig. 4 and its interpretation in Table 4.
255 The spectrum shows the possibility of five hydrocarbons in chemical composition of bio-oil.
256 The medium band at 1613 cm^{-1}and 3294 cm^{-1} were thus assigned to N-H stretch of amine
257 compounds. The strong band around 3033 - 3937 cm^{-1} were found to correspond to O-H
258 stretching vibration of alcoholic compounds of methanol, ethanol and phenol. The spectra
259 wavelength at 2239 cm^{-1} were assigned to C=0 stretching vibration of carbonyl combustible
260 hydrocarbon, whereas the peak value at 2159 cm^{-1} and 2476 cm^{-1} were assigned to C=C and
261 C≡N anti-symmetric stretching vibration of ethene and nitrile compounds capable of
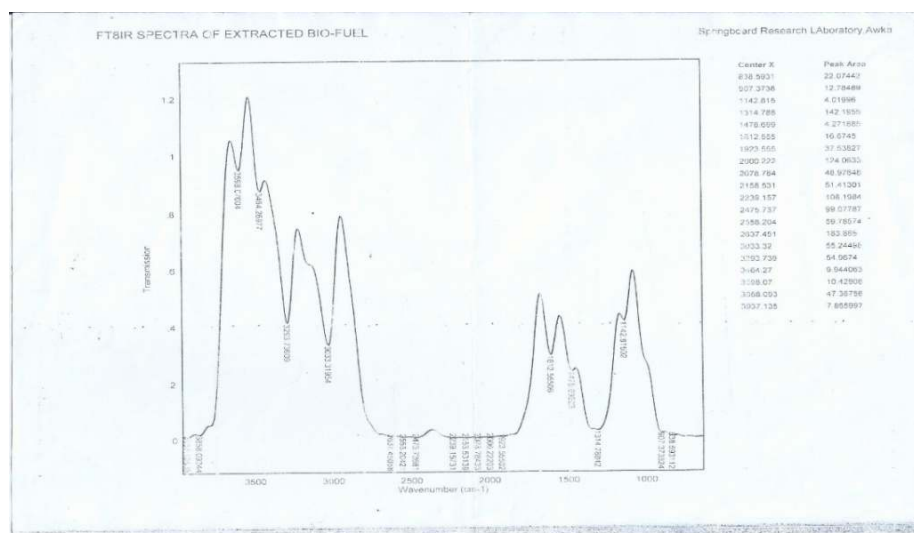262 burning.
263

Fig. 4. FTIR spectrum pyrolytic bio-oil of mahogany

Table 4. Interpretation of spectrum (FTIR) of pyrolysis oil from mahogany wood

S/N	Wavelength (cm^{-1})	Functional group	Compounds
1	836.5931	C-CI	Aliphatic chloro compound
2	907.3738	R-0-R	Ether C-0 stretch
3	1142.815	H$_2$C=CH	Vinylidene C=C vibration
4	2321.788	CH$_3$	Methyl CH stretch
5	1612.555	RNH$_3$	1^0 amine NH stretch
6	1923.555	R-S-C≡N	Thiocyanate SCN anti-symmetric Stretch
7	2000.222	RC00H	Carboxylic acid C0 stretch
8	2078.784	RC00H	Carboxylic acid C0 stretch
9	2158.531	H$_2$C=CH$_2$	Ethene CH antisymmetric stretch
10	2239.157	R$_2$C=0	Carbonyl compound C0 stretch
11	2475.737	R-C≡N	Nitriles CN antisymmetric stretch
12	2558.204	CH$_2$SH	Thiol SH stretch
13	2637.451	CH$_2$	Methylene CH stretch
14	3033.32	RCH0H	1^0 alcohol 0H stretch
15	3293.739	R$_2$NH	2^0 amine NH stretch
16	3464.27	R$_2$CH0H	2^0 alcohol OH stretch
17	3598.07	R$_2$CH0H	2^0 alcohol OH stretch
18	3858.07	R$_3$CH0H	3^0 alcohol OH stretch
19	3937.135	R$_3$CH0H	3^0 alcohol OH stretch

The FT-IR spectra of bio-oil fractions from pyrolysis of Mahogany wood are presented in Figure 4 (Table 4). Various peaks with strong, medium, broad and weak intensities were found indicating different bond types such as C–H, C–O, C–O, O–H, N–H and C–N in the bio-oil fractions. The C–O stretching vibrations between 2000 cm^{-1} and 2079 cm-1 was because of ketones and aldehydes, whereas the O–H stretching vibrations between 3033 cm^{-1} and 3937 cm-1 were from phenols and alcohols. Similarly, the bands between 1143 cm^{-1} and 2159 cm-1 represented C–C stretching vibrations due to the presence of alkenes and aromatics. The bands at 3294 cm-1 indicated the presence of hydrocarbon groups bound to aromatic rings. The C–H stretching vibrations between 2322 cm^{-1} and 2637 cm-1 and

deformation vibrations between 1350 cm^{-1} and 1475 cm-1 indicated the presence of alkanes. Presence of both O–H and C–O stretching vibrations indicated presence of carboxylic acids and their derivatives in the bio-oil. The main absorption bands corresponded to 3005 cm^{-1} assigned to =C–H stretching of the C–C double bonds. The 2963 cm^{-1} and 2852 cm-1 region is assigned as stretching of C–H saturated bonds, 1741 cm-1 as C=O stretching of the carbonyl functionalities, 1585 cm-1 as C=C stretching of the C–C double bonds, 1168 cm-1 as C–O–C stretching of the ester functionalities and 723 cm-1 as C–H plane stretching of saturated C–H bonds (Siengchum et al., 2013). The major peak at 2239 cm-1 arises from C=O stretching vibrations of ketones; the band at 1613 cm-1 arises from primary amide NH3 bend; and peaks at 907 cm^{-1} and 837 cm-1 may arise from out of plane ring bending vibration of aromatic rings. It was concluded that the presence of different functional group of compounds indicate the presence of hydrocarbons in the oil and also the O-H broad peak indicate the presence of water contents in bio-oil. The broad O-H peak was due to the interaction (hydrogen bonding) among the water contents. This oil sample consist of Alkane, Alkenes", ether, alcohol, aromatic, the presence of this functional group indicate the presence of hydrocarbons in the bio-oil.

4. CONCLUSION

From the experimental investigation and analysis, the following conclusions were derived: A pyrolysis batch reactor was successfully designed and constructed with the overall yield of mahogany pyrolysis oil was found to be 52 wt.%; The maximum yield of bio-oil (above 55 wt.%) was obtained under the conditions of pyrolysis at temperature of 450°C; the elemental analysis revealed the presence of Carbon, Hydrogen, Nitrogen and Oxygen, at 52 wt.%, 6 wt.%, 0.3 wt.% and 38 wt.% respectively; the FTIR analysis showed evidence of many aliphatic and aromatic compounds; the oil possessed favourable flash point; the heating value of the oil is moderate and has HHV of 21.96MJ/kg. The conclusion of this work is that pyrolysis is a good alternative source of fuel. Heat production from pyrolysis produce lower emissions including CO, NO$_X$ and smog particles

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