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SCHOLARENA

Journal of Environmental Pollution and Management

RESEARCH ARTICLE

ISSN: 2639-7269

Physico-Chemical Analysis of Pyrolyzed Bio-Oil from *Lophira alata* (Ironwood) Wood

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Citation: JA Oyebanji, OSI Fayomi, OI Oyeniyi, PG Akor, ST Ajayi (2022) Physico-chemical Analysis of Pyrolyzed Bio-Oil from Lophira alata (Ironwood) Wood. J Environ Pollut Manag 4: 101

Abstract

Lophira Alata (LA) bio-oil extract in a fixed bed reactor for 400 to 700 temperature range was study. Biomass material was characterized using analytical method to determine chemical composition and fuel properties. Carbon, hydrogen, nitrogen, oxygen, sulfur, MC, VM, FC and ash content in *Lophira alata* Sawdust are presented as 25.09, 2.98, 0.87, 70.11, 0.95, 7.27, 72.50, 18.48 and 1.75 wt.%, respectively while carbon, hydrogen, nitrogen, oxygen, sulfur content in the bio-oils were 65.67, 21.35, 0.43, 13.50 and 0.05 respectively. The flash point, pour point, pH, density, viscosity, API gravity, lower heating value (LHV) and Higher heating value (HHV) of bio-oil were found to be 86, -7, 4.90, 0.99 g/ml, 4.56 mm^2/s, 19.18 wt%, 29.96 MJ/kg and 34.62 MJ/kg respectively which is higher than the HHV (21.20 Mj/kg) and LHV (18.08 Mj/kg) of raw sample of *Lophira alata* Sawdust. GCMS analysis identifies the presence of *trans*-2-furanmethanol (4%), methyl-1-cyclohexenyl ketone (7.0%), palmitic acid (5.6%), %), cis-10-pentadecen- 1-ol (11.0%), 9-octadecenal (12.0%), trans-2-octadecadecen- 1-ol (13.0%), oleic acid (23.0%), and other compounds (23.8%). This study on bio-oil fuel properties has established *Lophira alata* wood has a potential fossil fuel substitute for production of bio-fuel for the generation of energy and industrial applications.

Keywords: Pyrolysis; Biomass; *Lophira alata;* Bio-Oil; Fuel Properties; Characterization

Introduction

Sustainability targets and overexploitation of fossil fuel leading to their depletion has been the major concerns in energy world, consumption increase rate, population growth and environmental consequences of fossil fuels have driven interest in renewable biofuels from agro residue (biomass). Biomass waste is one of the key potential energy resources to meet the ever-increasing energy needs without portending danger to the environment and food based crops. Biomass pyrolysis has received more consideration due to global energy crisis, high energy consumption and rapid population growth as an alternative for fossil fuels [1-3].

Biomass materials are majorly agro residues converted for energy generation, wood residues in terms of sawdust, wood chips and shavings are generated in large percentage yearly. This huge amount of solid agro residues, especially sawdust, though a possible source of energy often poses as nuisance to the environment [2-4]. The world is currently facing a high demand of fuel and on the other hand, the crisis bedeviling it at the same time is another disturbing challenge. The fossil fuel that is available on earth will no more be able to support this increasing demanded amount of energy in the nearest future [4]. Pollution is yet another disturbing matter of concern in the world for which the production and use of fossil fuel is solely responsible. Therefore, researchers are now looking for and trying to produce an alternate fuel such that it can fulfill the growing demand of energy and can keep the globe clean for the next generation [4-5]. Fossil fuels constituent are the major convectional energy source used in the world, Africa and specifically in Nigeria with 170 million and forecasted to grow to 310 million by 2035 [5-7]. Nevertheless, conversion of biomass into bio-oil, biochar, syn-gas, etc. involves completely thermochemical conversion procedures, such as pyrolysis, liquefaction, gasification, torrefaction, and carbonization [6-8]. Scientific research and development in the thermochemical conversion of biomass hold a vital part in solving practical and sustainable energy as a result of the current energy [9-13]. Pyrolytic products may be deployed as fuels with or without initial upgrading or could be used as feedstock for material or chemical plants. Also, the products have a significant economic value as well as foster a cleaner environment. An empirical study on the decomposition of sawdust via the fast pyrolysis process was conducted by Okekunle *et al.* (2021) [12]. The relationship between the yields of the products was investigated by altering the temperature range from 450-540°C in the reactor while other parameters such as resident time, system pressure, the number of feeds, and bed height were kept constant. Results showed that temperature had a great influence on the type and size of the products. In the work of Barik *et al.* (2019) [13], a temperature range of 300-700°C for fast pyrolysis microalgae, corn cobs, and rice husk using a fixed bed reactor was carried out. It was deduced that maximum biochar yield, bio-oil yield, and non-condensable gases yield were between 300-350°C, 350-450°C, and 450-650°C respectively. Chowdhury et al. (2017) [14] reported that pyrolysis yields are mainly affected by the percentage of their physicochemical properties such as proximate (MC, VM, FC, and ash), ultimate contents (C, H, N, O, S), and heating values of the biomass. Abundant wood residues are all over wood processing plant in Africa (Nigeria) that can undergo conversion process to bio-gas, bio-char, bio-oil and chemical feedstock for chemical industries [15-16]. This study is aimed at determining the elemental composition and fuel Properties of bio-oil extract from *Lophira alata* Pyrolysis.

Materials and Methods

Materials

The biomass samples for this study were taken from the trunk of *Lophira alata* from sawmills in Ota/Ado-odo and its environs, South Western Nigeria. The wood sample was rinsed with distilled water to remove impurities and sun-dried for three days. The sun dried biomass was milled to sawdust and sun-dried for seven days before the experiments [17]. Dried particle size of 0.5 mm (500 μm) were used, weighed and stored at room temperature in polythene bags for proximate and elemental analysis (according to ASTM C566 and ASTM D1102), and correlation equations were used in the determination of HHV.

Pyrolysis Experimental Setup

The pyrolysis set-up comprises N_2 gas cylinder, valve, pressure gauge, reactor, electric cell heater, condenser, ice bath and gas collector couple with PID temperature controller to control 4 kW electric heater. The pyrolysis experiments were carried out under nitrogen atmosphere in a fixed-bed reactor at varying temperature. Figure 1 shows the Pyrolysis experimental set-up for the pyrolysis process.

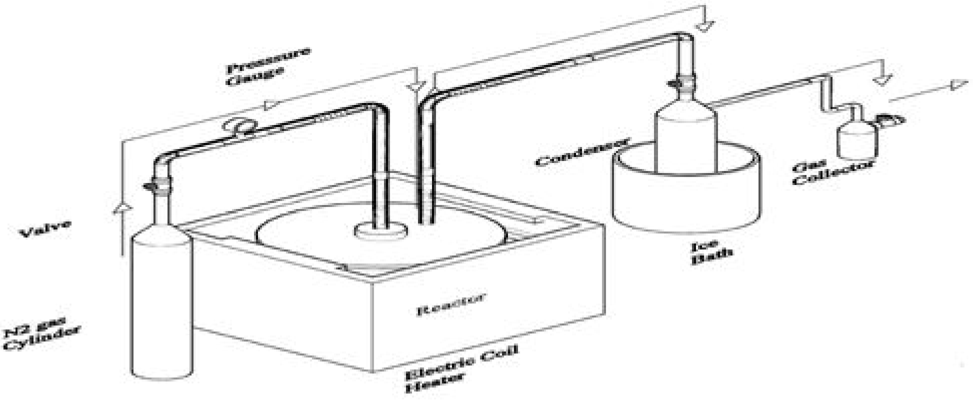

Figure 1: Pyrolysis Experimental setup [18]

Experimental Procedure

The sample was fed into the reactor, covered and fastened with bolts and nuts in preparation for a run at a pre-set furnace temperature range of 400, 500, 600 and 700 °C for a residence time of 25 min at 3 run each for each pyrolysis process. The weight of product yield were determined (bio-oil and bio-char) and obtained by mass balance (bio-gas).

Elemental composition of bio-oil

Raw sawdust samples were charterized using Eltra elemental analyzer according to ASTM D3176 - 09 and ASTM D7582. [19]. The moisture content, volatile matter, fixed carbon and ash percentage composition of the samples were obtained from proximate analysis while the percentage of carbon, hydrogen, Nitrogen and organics were determined from ultimate analysis to determine the fuel properties.

Heating values of bio-oil

The HHVs were estimated from the developed equations of Correlation for calculating HHV using ultimate and proximate analysis according to equations 1 and 2 [16,20].

$$HHV_{DRY}\left({}^{Mj}/_{kg}\right) = -1.3675 + 0.3137C + 0.7009H + 0.0318O \quad (1)$$

$$HHV_{dry} = 0.6042FCC + 0.4083VM + 0.2442\,Ash + 0.4107MC - 25.20 \quad (2)$$

Where: Moisture Content (MC), Volatile Content (VC), Fixed Carbon Content (FCC), Ash Content (AC) and Higher Heating Value (HHV)

The LHVs were estimated from the developed equations of Correlation for calculating LHV [18]:

$$LHV_{dry} = HHV_{dry} - [2.442. X\ 8.936\ (^{H}/_{100})] \tag{3}$$

GC/MS analysis

Model QP2010 Shimadzu gas chromatograph analyzer under a helium atmosphere of flow rate (1.5 ml min^{-1}) with mass scanning range between 40 and 700 m/z was used to determine GC/MS analysis [21].

Results and Discussion

Physio-chemical properties

Result of proximate and ultimate analysis of feedstock is presented in Table 1. The percentage of Moisture Content (MC), Volatile Content (VC), Fixed Carbon Content (FCC), Ash Content (AC) as proximate analysis results are 6.70, 78.47, 13.48, 1.35% respectively while the percentage of Carbon (C), Hydrogen (H), Nitrogen (N), Oxygen (O) and sulfur (S) as the ultimate analysis results are 54.60, 5.98, 0.27, 39.14 and 0.01% respectively. High volatile matter (78.47%) and low ash content (1.35%) indicate higher yield of bio-oil during pyrolysis process at 500 ℃. High percentage of fixed carbon (13.48%) was noticed in the wood samples compared to fixed carbon of wood residues (11 – 15%), hence indicate high energy content and higher heating value of 21.20 MJ/kg [22-23]. The low ash content (1.08 – 1.93%) and moisture content (8.70%) obtained from the results are reasonable for wood fuels as reported in the literature [24].

Ultimate Analysis		**Proximate Analysis**	
Component	Content (%)	Component	Content (%)
C	54.60	Moisture Content	6.70
H	5.98	Volatile matter	78.47
N	0.27	Fixed Carbon	13.48
O	39.14	Ash	1.35
S	0.01		
HHV (MJ/kg)	21.20	LHV (MJ/kg)	18.08

Table 1: Propertise of *Lophira Alata* (LA) residue

Fuel properties and chemical composition of bio-oil produced in this study are presented in table 2 contains Carbon (51.67%), Hydrogen (6.35%), Nitrogen (0.43%), Oxygen (41.50) and sulfur (<0.05%). Low sulfur content (<0.05%) indicate low pollutant effect, HHV and LHV are 29.60 MJ/kg and 27.98 MJ/kg respectively which are the same range of values as reported by Oyebanji et al. (2017) [21] and in agreement with what is reported by Chukwuneke et al. (2019) [25] and Okekunle et al. (2021) [3]. The flash (86) and pour points (-7) bio-oil are compare with flash (75) and pour points (-2) of diesel which very close to the range of flammability, lower flash point indicate good flammability and volatility of the oil while pour point indicate temperature at which a bio-oil ceases to flow due to the formation of wax crystals that increase its viscosity, The pH, density, Viscosity and API gravity are in agreement with the report of some researchers on bio-oils from biomass [26-29]. The HHV (29.60 Mj/kg) and LHV (27.98 Mj/kg) are also determines, the heavier the fuel and the harder it burns at low density [30]. Bio-oil pH values obtained (4.9) falls below of diesel (5.5-8.0) while densities at 40°C (0.99 g/cm^3) has slight variation with those of fuel oil (0.9101 g/cm^3), furnace oil (0.92 g/cm^3), and heavy fuel oil (0.9888 g/cm^3). gasoline (44-46 MJ/kg) and diesel (42-46 MJ/kg) HHVs are higher than the HHVs of the bio-oil (27.98- 29.60 MJ/kg) [3].

Properties	**Diesel**	**Heavy fuel oil**	**Bio-oil**
Flash point	75	-	86
Pour point	-2	-	-7
pH			4.90
Density (g/ml) @ 40 ℃	0.78	0.94	0.99
Viscosity (cst (mm^2/s)@ 40℃	1.8 -4.1	180	4.56
API gravity	45.0 -46.0	40	19.18
Elemental analysis (db. wt%) of bio-oil			
C	-	-	51.67
H	-	-	6.35
N	-	-	0.43
O	-	-	41.50
S	-	-	0.05
LHV (MJ/kg)	45.0 -46.0	40	27.98
HHV (MJ/kg)			29.60

Table 2: Fuel properties of bio-oil yield

Temperature effect on Product yield

Figure 2 present the influence of temperature on Ironwood [*Lophira Alata (LA)*] product yield between the range of 400 700 ℃. Increase in temperature is directly proportional to increase in bio-oil yield and gas. As the pyrolysis progressed, bio-oil yield increases from 45.6wt.% at 400 ℃. to 54.5wt.% at 500 ℃. And later decreases to 52.4wt.% and 49.9 wt.% at 600 and 700 ℃. respectively. As the temperature increases the char yield decreased from about 39wt.% to 18.8wt.%. Gas yield increased from 15.4wt.% at 400 ℃. to 31.3wt.% at 700 ℃. as pyrolysis temperature increased. The maximum bio-oil yield was realized at 500 ℃. In accordance with literature [30-32].

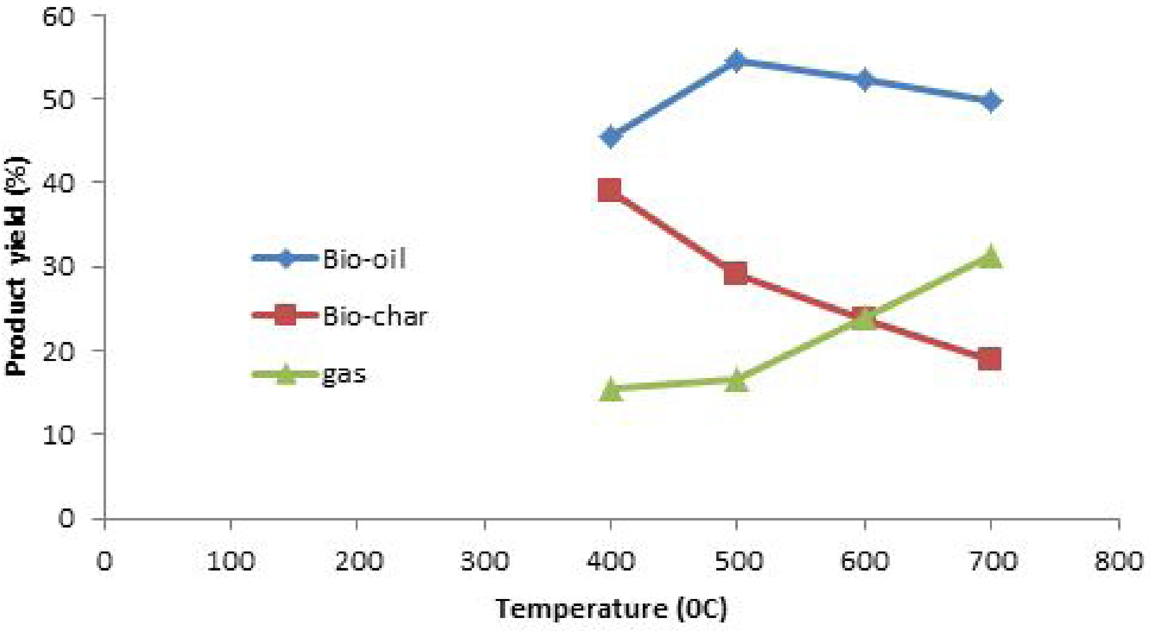

Figure 2: Product yield distribution

Chemical analysis of the bio-oil

GC-MS spectrometry analysis

GC-MS analysis identifies presence of Thirty-four compounds (99.5% of the volatile extract) in bio-oil at 500 °C as *trans*-2-furanmethanol (4%), methyl-1-cyclohexenyl ketone (7.0%), palmitic acid (5.6%), %), cis-10-pentadecen- 1-ol (11.0%), 9-octadecenal (12.0%), trans-2-octadecadecen- 1-ol (13.0%), oleic acid (23.0%), and other compounds (23.8%) as presented in table 3. Indication of phenolic, ketone, fatty acids, esters and alcohols compounds confirms the fuel and chemical composition that can be as feedstock for Pharmaceutical and dyes industries, and use as biodiesel for energy and power production in internal combustion engines. The chemical composition is in agreement with the studies of some researchers on woody biomass pyrolysis [3, 25, 33-34].

Compounds	**Retention Index**	**Percentage Composition**	**MF**	**MW**
Tetramethylmethane	434	0.4	C_5H_{12}	72
2-methylfuran	642	0.9	C_5H_6O	82
trans-2-octadecadecen-1-ol	2061	13.00	$C_{18}H_{36}O$	268
oleic acid	2175	23.00	$C_{18}H_{34}O_2$	282
3-methylfuran	643	0.8	C_5H_6O	82
Trimethylnitromethane	716	0.5	$C_4H_9NO_2$	103
2-pentynal	724	1.2	C_5H_6O	82
palmitic acid	1968	5.6	$C_{16}H_{32}O_2$	256
Dimethylvinylmethanol	600	0.5	$C_5H_{10}O$	86
4-ethyl-1,3-dioxolane	738	0.5	$C_5H_{10}O_2$	102
vinyl butyrate	775	0.5	$C_6H_{10}O_2$	114
1-hydroxy-2-butanone	798	1.0	$C_4H_8O_2$	88
cis-3-octene	823	1.0	C_8H_{16}	112
trans-3-octene	824	1.5	C_8H_{16}	112
2-methoxymethyltetrahydrofuran	825	0.5	$C_6H_{12}O$	116
2,4-pentadienoic acid	873	1.0	$C_5H_6O_2$	98
trans-2-furanmethanol	885	4.0	$C_5H_6O_2$	98
5-methyl-3-methylene-5-hexen-2-one	887	2.0	$C_8H_{12}O$	124
tetrahydro-2-furanmethanol	892	0.5	$C_5H_{10}O_2$	102
Corylone	972	1.0	$C_6H_8O_2$	112
Butylglyoxylate	973	0.5	$C_6H_{10}O_3$	130
3-hydroxy-6-methylpyridazine	975	1.0	$C_5H_6N_2O$	110
3-methyl-1,2-cyclopentanedione	1003	0.5	C_6H_8O	112
methyl-1-cyclohexenyl ketone	1027	7.0	$C_8H_{12}O$	124
methyl 2-butyl-2-cyclopropene-1-carboxylate	1057	1.0	$C_9H_{14}O_2$	154
o-guaiacol	1090	2.0	$C_7H_8O_2$	124
Syringol	1279	1.0	$C_5H_8N_2$	96
2,4-dimethoxyphenol	1279	1.0	$C_8H_{10}O_3$	154
3-nonynoic acid	1290	0.9	$C_9H_{14}O_2$	154
1-methyl-3-nitro-2(1H)-pyridinone	1376	1.0	$C_6H_6N_2O_3$	154
pelargic acid	1272	1.0	$C_9H_{18}O_2$	158
cis-10-pentadecen-1-ol	1763	11.0	$C_{15}H_{30}O$	226
n-pentadecanoic acid	1869	2.0	$C_{15}H_{30}O_2$	242
9-octadecenal	2007	12.0	$C_{18}H_{34}O$	266
Percentage Total		**99.4**		

Table 3: Chemical composition of *Lophira Alata* (LA) at 500 °C

Conclusion

Waste-to-energy potential has been study through production and characterization of bio-oil from *Lophira alata* sawdust within temperature range from 400 to 700 °C. Proximate and ultimate analyses, *GC-MS* analysis and fuel properties results were obtained. Bio-oil physio-chemical properties obtained from *Lophira alata* wood are comparable with those obtained from woody biomass materials liquid products, The fuel properties and heating values were obtained in which HHV (18.08 - 21.20 Mj/kg) *Lophira alata* raw sample are lower than HHV of bio-oil (27.98-29.60 Mj/kg) and GCMS result shows presence of aromatic hydrocarbons, phenolic compounds nitrogen-containing compounds and oleic acid. The presence of fuel properties in *Lophira alata* will aid its use as raw material for bio-diesel and chemical additive in chemical industries, and improvement of fuel quality will be of good advantage renewable energy applications.

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