Pyrolysis and Characterization of Jatropha Curcas Shell and Seed Coat

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ABSTRACT: The utilization of dedicated energy crops and agricultural residues for producing biofuels and bio-oil in a range of energy conversion technology is attracting more research interests. Pyrolysis is one of such important thermochemical method for converting lignocellulosic biomass into biofuels. This work investigates the pyrolysis of residues from a dedicated energy crop, jatropha of Nigerian origin using intermediate pyrolysis. Pyrolysis of Jatropha biomass residues [Jatropha fruit shells (JFS) and Jatropha seed coat (JSC)] was carried out in a tubular fixed bed reactor at a temperature of 450^{o}C, using intermediate pyrolysis method. Bio-oils were obtained and subsequently characterised for their physico-chemical properties. The yields of the resulting bio-oil, biochar and gas were determined. The compositions of the bio-oils obtained were also determined by gas-chromatography mass spectrometry (GC-MS) and carbon, hydrogen, nitrogen, sulphur (CHNS) elemental analysis. The main constituents of the bio-oils obtained from JFS and JSC were acetic acid, guaiacol, 2,6-dimethoxyphenol and phenol. The empirical formula of the obtained JFS and JSC bio-oils were found to be $CH_{1.77}\ O_{0.28}\ N_{0.04}$ and $CH_{2.03}\ O_{0.47}\ N_{0.04}$ respectively. The bio-oil samples that were produced from JSC and JFS of Nigerian origin were found suitable for bio-oil production. Valuable compounds found in the bio-oils indicated potential industrial applications.

KEYWORDS: Jatropha shell, pyrolysis, biomass waste, biofuel, bio-products.

[Received June 29, 2018; Revised September 10, 2018; Accepted October 23, 2018] Print ISSN: 0189-9546 | Online ISSN: 2437-2110

I. INTRODUCTION

The utilization of biomass as alternative renewable energy source to meet growing energy needs and to reduce carbon emission, has attracted global attention in recent times due to its economic potential and environmental concerns (Titiloye et al., 2013; Odetoye *et al.*, 2014; Aysu *et al*., 2016; Thapa *et al*., 2018). Biomass is an important source of energy especially in the developing countries (Akinrinola *et al*, 2014).

Jatropha curcas is a widely cultivated plant for energy purpose as it has been regarded as one of the most suitable feedstocks for biodiesel production (Pambudi *et al*, 2017). Extensive studies have been done on the production of biodiesel from jatropha oil (Ajala *et al*., 2015; Thapa *et al*., 2018), therefore several researches have been extended towards the utilization of biomass residues generated from Jatropha fruits and seeds (Biradar *et al*., 2014; Adinurani, 2015; Kaewpengkrow, 2017; Patel, 2018).

In Nigeria, jatropha has been listed as one of the potential feedstocks considered for biofuel production and diversification of the monolithic petroleum-based economy by the Government (PPRA, 2010, Ohimain, 2013). More so, jatropha grows locally in Nigeria and the oil yields of up to 53% had been reported for jatropha of Nigerian origin (Aransiola *et al*., 2012; Somorin *et al*., 2017). Subsequently, large scale cultivation of jatropha has been recently embarked on by the Nigerian Government to encourage the industrial application of the seed oil for biodiesel production (Lateef *et al.,* 2014). Jatropha seed coat and the fruit shell are wastes generated during the processing of jatropha oil that can be converted to bio-oil as value added product (Patel *et al*., 2018).

One of the effective technologies for converting such biomass wastes to bio-oil is by pyrolysis, a method which involves thermal decomposition of the lignocelluosic biomass waste in the absence of air at high temperatures of about 400 ^{o}C (Aysu *et al*., 2016; Patel *et al*., 2018). Two main types of pyrolysis technology widely practiced had been the slow and fast pyrolysis (Yang *et al*., 2014; Abu Bakar and Titiloye, 2013). The classification was based on the variation in heating rate, residence time and the product distribution (char, bio-oil, gas). Fast pyrolysis involves rapid cooling of the short-residence-time-heated finely ground biomass to room temperature so as to obtain higher yield of the liquid products. Slow pyrolysis which is characterised by longer residence time, produces higher yield of char. Intermediate pyrolysis is a recently emerged pyrolysis technology which offers the opportunity of process integration as extended involvement of char in the pyrolysis process seems to

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doi: http://dx.doi.org/10.4314/njtd.v16i2.4

improve the bio-oil quality. This is a technology that has recently been patented by Aston University, the designer of a Pyroformer, an intermediate pyrolysis reactor (Yang *et al*., 2014).

Although several works have been done on pyrolysis of Jatropha of various origins (Sing *et al*., 2008; Manurung *et al*., 2009; Murata *et al*., 2011; Wever *et al.*, 2012), there is dearth of report about bio-oil production from Jatropha wastes of Nigerian origin. This study investigates the suitability of the biomass residues of Jatropha of Nigerian origin for the production of bio-oil via intermediate pyrolysis. The intermediate pyrolysis method on a fixed bed reactor was chosen over fast pyrolysis method because the method is relatively more affordable for growing economies.

II. MATERIALS AND METHOD

A. *Materials preparation*

The jatropha fruit shell (JFS) and jatropha seed coat (JSC) residues were obtained from the matured jatropha fruits collected at Ilorin, Kwara State, Nigeria. 5 kg of the residues were sundried for five days and ground using Fritsch blade heavy-duty cutting mill (USA) fitted with a 2 mm particle size screen and characterized according to standard methods as reported in another work (Odetoye et al., 2018).

B. *Pyrolysis*

The intermediate pyrolysis experimental set up was as shown in Figure 1. The experiments were carried out on a bench scale fixed – bed vertical tubular reactor at reaction temperature of 450 ^{o}C. The internal diameter and height of the quart glass reactor was about 8 cm and 35 cm, respectively. The reactor was filled with 90 g of JFS or JSC as required. A Carbolite vertical split furnace was used as a source of heating while the reactor temperature and pressure were monitored by AALBORG model DFM digital monitor. Nitrogen gas flow into the reactor was maintained at a flow rate of 50 cm^3/min. The heating rate of the reactor was 25 ^{o}C/min while the residence time was 25 minutes. The primary condenser was cooled with dry ice to enable the collection of condensable bio-oil in the oil pot while the non-condensable gases were scrubbed with isopropanol before sending a stream of the gases to the GC-MS HP Series 5890 for analysis. The remaining gases were vented through the fume cupboard.

The bio-oil yield was obtained considering the oil entrapped in the glass wares, condensers, by weighing each part of the glassware apparatus before and after each pyrolysis experiment since the pyrolysis procedures included setting up and running the experiment with a good consideration of mass balance. The mass of the bio-oil, biogas and biochar produced were accounted for by measuring the mass of each component of the apparatus (reactor tube, connecting tubes, and oil pots) before and after each pyrolysis experiment. The biogas was also accounted for by difference (Abu Bakar and Titiloye, 2013).

C. *Characterization of bio-oil*

The bio-oils obtained were characterized to determine the quality and composition. The following characteristics were determined: acid number, pH value, density and water content determinations. Acid number, pH value, density and water contents were carried out using apparatuses based on standard methods. The acid number of the oil was determined using Mettler Toledo acid number analyser G20 Compact Titrator, which was based on the standard method ASTM D664-04 (ASTM, 2011) for the determination of acid number in motor oil while the pH values of the bio-oils were determined using the Sartorius pH meter model PB-11.

Mettler Toledo PortableLab Densimeter was used to determine the density of the bio-oil. The water content of the bio-oil was determined with Karl –Fischer volumetric titration using a Metrohm 758 KFD Titrino water content analyser that was based on the standard ASTM method D1744 (ASTM,2011). The heating values of the bio-oil samples as well as the elemental analyses of the samples were carried out by MEDAC Ltd. Surrey, U.K. using standard method on biomass characterization with Carlo-Erba 1108 elemental analyser. Metals and inorganic components were also determined using a PerkinElmer Optima 7300DV Induced Coupled Plasma (ICP) Emission Spectrometer**.**

The composition of the bio-oils produced were determined using the Hewlett Packard 5890 Series II Plus Gas Chromatograph incorporated with a Hewlett Packard 5972 mass selective detector. Helium was used as the carrier gas with a DB 1706 non-polar capillary column. The initial oven temperature was 40 °C and ramped up to 290 °C at a rate of 3 °C/min. The injection temperature was held at 310 °C with a volume of 5µl. The dilution solvent used was ethanol and the dilution rate was 1:5. Identification of compounds in the spectral and chromatograph data was done with the aid of NIST mass spectra database.

1 = primary reactor, 2 = secondary reactor, 3 = dry ice condenser, 4 = oil pot 1, 5 = secondary condenser, 6 = scrubber

Figure 1: Experimental set up for intermediate pyrolysis experiment

III. RESULTS AND DISCUSSION

A. *Characterization of the bio-oils produced*

The relatively semi-homogenous bio-oil obtained clearly separates into aqueous and organic phases when stored. The properties of the bio-oils obtained from Jatropha seed coat and Jatropha fruit shells are as shown in Table 1.

Table1: Some physicochemical characteristics properties of the prepared Jatropha waste bio-oils.

Properties	Jatropha Seed Coat (This study)	Jatropha Fruit shell (This study)	Jatropha Seed Coat (Manurung et al., 2009)	Jatropha Seedshell cake Kim et al., 2013)
pH	5.2	5.8	3.3	
Density (g/cm^3)	1.006	1.008		
Water content (wt %)	18.47	21.76	23.3	
Elemental composition (wt %)				
C	52.6	62.4	65.6	65.8
H	8.89	9.19	9.5	8.92
N	2.29	2.73	0.9	5.62
O	33.04	23.14	nd	18.8
S	0.25	0.26	nd	0.19
Cl	2.93	2.31	nd	
HHV(MJ/kg)	25.24	29.84		39
Empirical Formula	$CH_{2.03}O_{0.47}N_{0.04}$	$CH_{1.77}O_{0.28}N_{0.04}$		
H/C molar ratio	2.0	1.8		1.63
O/C molar ratio	0.47	0.28		0.11

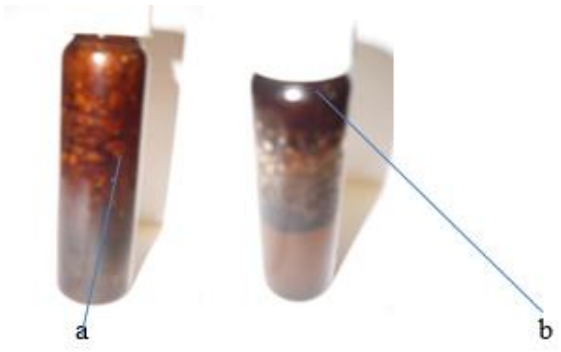

Figure 2: Jatropha residue bio-oils from (a) JSC and (b) JFS.

The bio oil samples obtained from the JFS and JSC biomass residues were dark brown in colour as shown in Figure 2.

The pH values of 5.3 and 5.8 of the Jatropha waste bio-oils obtained for JFS and JSC are relatively higher than the value of 3.3 obtained in literature for JSC (Manurung et al., 2009). Higher pH values (as the values approach 7, being neutral) are desirable for bio-oils since acidity can wear out the engine components when the bio-oil is used as fuel. Sulphur contents are desirably low in both JSC and JFS bio-oils indicating that the formation of sulphur dioxide is at lower risk (Tsai et al, 2018).

The higher heating value (HHV) of the Jatropha fruit shell bio-oil (29.84 MJ/kg) is relatively higher than that obtained for Jatropha seed coat (25.24 MJ/kg). However, the heating values of both bio-oil samples prepared were comparable to the value of 25.63 MJ/kg (Jourabchi et al., 2016), 30 MJ/kg (Das et al., 2015) available in literature for jatropha seed cake bio-oil.

Carbon contents were found to be desirably relatively higher in JFS (62.4%) compared to JSC bio-oil (52.6%) while the oxygen content was lower in JFS. The chlorine and the sulphur contents were desirably low in both bio-oil samples. Hence, there is a lower risk of sulphur dioxide formation which can lead to environmental pollution (Tsai et al, 2018).

B. Composition of the Bio-oil Produced

Analysis of the bio-oil samples indicated the presence of a vast number of complex mixtures of compounds which include alkenes, phenols, carboxylic acids and their derivatives. The GC-MS chromatograms for JFS and JSC pyrolysis oils were as shown in Figures 3 and 4. The most prominent peaks identified, corresponding chemical names, retention time, chemical formulae, molecular weight and % peak area measured are summarised in Tables 2 and 3.

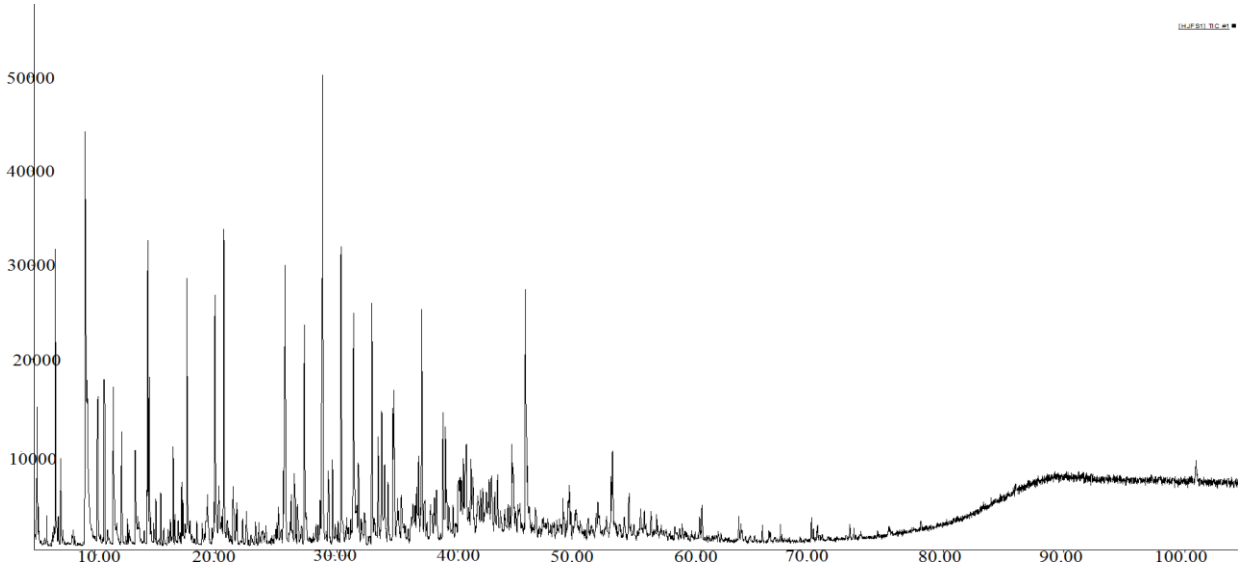

Figure 3: GC-MS chromatogram for Jatropha fruit shell.

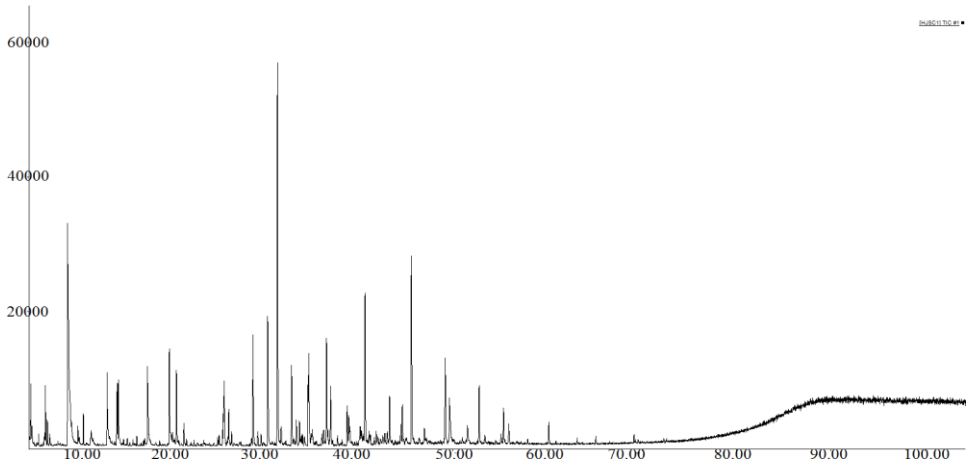

Figure 4: GC-MS chromatogram for Jatropha seed coat.

The main identified components of the Jatropha seed coat include dimethoxyphenol, guiacol and acetic acid while the main identified components of the fruit shells are acetic acid, 2-cyclopenten-1-one, phenol, guaiacol and 2,6-dimethoxyphenol as shown in Figures 5, 6 and 7. These compounds are characteristic components of bio-oil that are candidates for biorefinery. They are useful precursor for the synthesis of industrial products and can be used as fuels chemicals and food bioproducts after refining (Demiral *et al*, 2012). Guaiacol is useful for medicinal purposes, 2,6-dimethoxyphenol has been recognized as a volatile flavor component (Bridgwater et al., 2008) found in soy sauce, wine and smoke.

Lignin which is a phenolic bio-polymer (Das *et al*.,2015) was responsible for the phenolics and aromatics constituents in the bio-oils. The presence of acetic acid, an organic acid, undesirably contributes to low pH values in bio-oils. Such relatively high content of acetic acid indicates the need for upgrading procedure to be performed on the oil prior to applications as fuel in engines (Odetoye et al, 2014).

Table 2: Most prominent identified compounds of Jatropha Fruit Shell.

Peak ID	RT (min)	Compound Name	Area %
1	9.184	Acetic Acid	6.9
2	10.242	Hydroxyacetone	1.05
3	10.794	Toluene	1.12
4	11.518	Pyridine	1.79
5	15.059	Ethylbenzene	0.35
6	15.45	p-Xylene	0.44
7	16.462	Cyclohexanone	0.75
8	16.922	m-Xylene	0.23
9	20.659	2-Cyclopenten-1-one, 2-methyl-	2.89
10	21.452	2-Furyl Methyl Ketone	0.61
11	22.199	Mesitylene	0.24
12	25.545	3-Methyl-2-Cyclopentenone	0.66
13	25.717	3-Methyl-2-Cyclopentenone	3.04
14	26.718	3-Picoline	0.58
15	27.304	2,4-dimethyl-2-oxazoline-4-methanol Maple lactone /2-hydroxy-3-methyl-2	2.11
16	28.776	Cyclopenten-1-one	4.64
17	30.351	Phenol	3.67
18	31.374	Guaiacol	2.83
19	32.869	2-Methylphenol	2.44
20	33.375	3-Ethyl-2-hydroxy-2-cyclopenten-1-one	1.17
21	34.697	p-Cresol	1.95
22	36.996	2,4-Dimethylphenol	2.31
23	39.043	4-Ethylphenol	0.37
24	40.02	Decane	0.47
25	40.653	4-Ethylguaiacol	1.07
26	45.574	2,6-Dimethoxyphenol	2.63
27	60.141	4-Allyl-2,6-dimethoxyphenol	0.46

Table 3: Peak assignment for Jatropha seed coat bio-oil.

Peak ID	RT (min)	Compound Name	Area %
1	6.771	2-butanone	1.33
2	9.105	Acetic Acid	14.68
3	10.796	Toluene	0.69
4	11.612	Pyridine	0.52
5	13.302	Hydroxyacetone	2.43
6	14.383	Cyclopentanone	1.66
7	14.498	1-hydroxy-2-butanone	1.74
8	17.602	2-Cyclopenten-1-one	2.86
9	19.913	2-Furanmethanol	3.25
10	20.66	2-Cyclopenten-1-one, 2-methyl-	2.06
11	21.454	2-Furyl Methyl Ketone	0.73
12	25.719	3-Methyl-2-Cyclopentenone	2.5
13	26.214	Tetrahydro-2-furanmethanol	1.13
14	28.778	cyclopenten-1-one	3.6
15	30.341	Phenol	4.31
16	31.365	Guaiacol	12.14
17	32.871	2-Methylphenol	2.53
18	33.377	3-Ethyl-2-hydroxy-2-cyclopenten-1-one	0.76
19	34.687	m-Cresol	3.52
20	36.573	Isocreosol / 5-methylguaiacol	3.12
21	38.746	2,4-Dimethylphenol	1.17
22	38.918	4-Ethylphenol	0.83
23	40.137	Dianhydromannitol	0.91
24	40.655	4-Ethylguaiacol	5.06
25	45.576	2,6-Dimethoxyphenol	6.04
26	49.163	Isoeugenol	2.57
27	49.611	1,2,4-Trimethoxy-benzene	2.13
28	52.75	1,2,3-trimethoxy-5-methylbenzene	1.97
29	55.9	2,4-hexadienedioic acid, 3,4-diethyl-, dimethyl ester, (EZ)-	0.73
30	60.143	4-Allyl-2,6-dimethoxyphenol	0.72

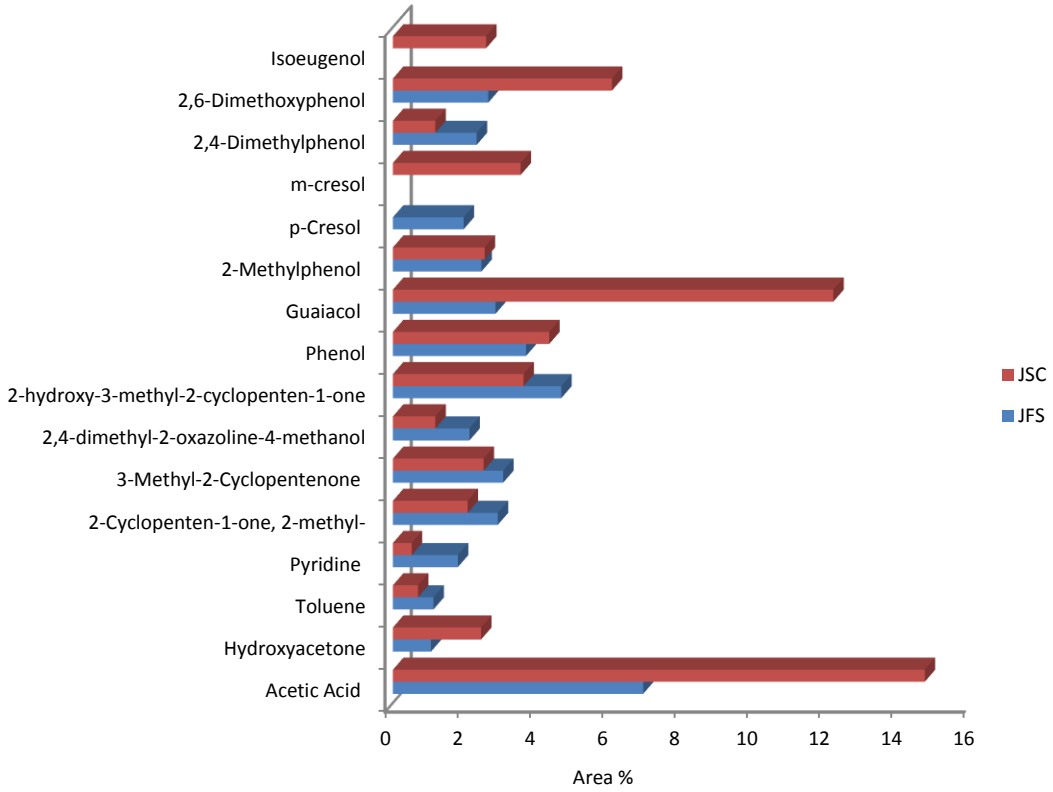

Figure 5: Main chemical constituents of Jatropha bio-oils.

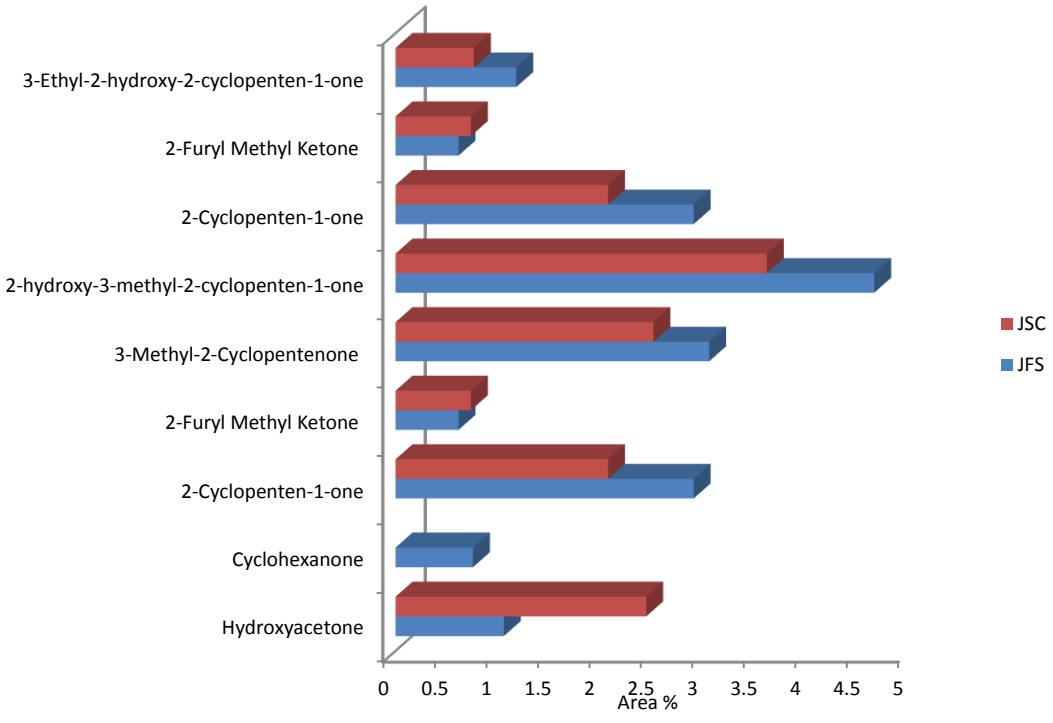

Figure 6: Main ketones constituents of Jatropha bio-oils.

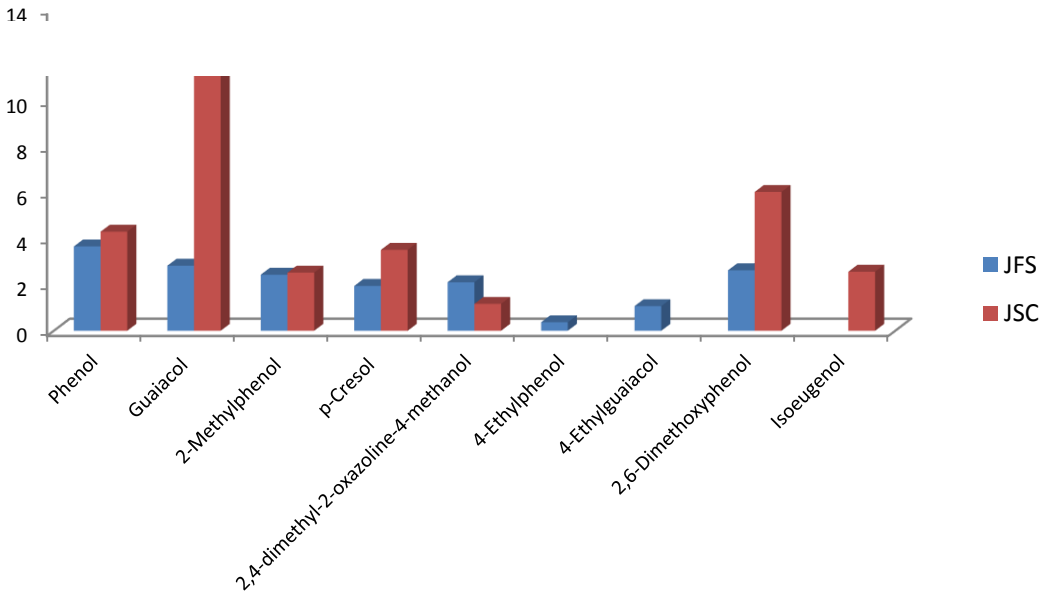

Figure 7: Phenols and its derivatives in Jatropha bio-oils

V. CONCLUSION

Jatropha wastes (seed coat and fruit shell) of Nigerian origin, have been found suitable for use as renewable energy feedstock with potential for bio-oil production. The characteristics of the bio-oils produced were found to be similar to those reported in literature. The presence of valuable compounds such as phenolic compounds suggests useful potential for industrial applications. Jatropha fruit shell (JFS) and Jatropha seed coat (JSC) bio-oils need to be upgraded before they can be utilized as a fuel substitute particularly in engines as they consist of various complex organic compounds with varying composition. This work has provided database on some properties of Jatropha residues bio-oils of Nigerian origin, that are applicable for bio-energy, bio-refinery, waste management and vegetable oil industries.

VI. ACKNOWLEDGEMENT

The authors are thankful for the financial support obtained from European Bioenergy Research Institute/School of Engineering and Applied Sciences, Aston University, Birmingham, UK.

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