JOURNAL OF NANO AND MATERIAL SCIENCE RESEARCH
Volume 1 (Maiden edition, 2022), pp. 1-8
Available online at journals.nanotechunn.com/jnmsr

ORIGINAL ARTICLE

Extraction and Characterization of Hura crepitans Seed Oil

Stella Ulakom[a], Veronica Ezekoye[a], Anthony Ofomatah[b], David Ezekoye[c] and Ada Agbogu[a]*

[a]*Department of Physics and Astronomy, University of Nigeria, Nsukka Enugu State, Nigeria.*
[b]*National Centre for Energy, Research and Development.*
[c]*Department of Civil Engineering, University of Nigeria, Nsukka, Nigeria.*

KEYWORDS

Hura crepitans
seed oil
biodiesel
physicochemical properties
transesterification

ARTICLE HISTORY

Received: May 30, 2022
Revised: June 15, 2022
Accepted: July 22, 2022

ABSTRACT

Hura crepitans (HC) seed oil was extracted using soxhlet extraction method with n-hexane as solvent at 66.0℃. The extracted oil was converted into biodiesel using alkali-catalytic-transesterification method at a constant temperature of 60℃ for 60 minutes. The oil yield was (42.00%) while biodiesel yield was (71.86%).The physiochemical properties of the analyzed biodiesel were specific gravity (0.902), density (903 kg/m^3) at 30℃, viscosity (2.4 mm^2/s) at 40℃, flash point (193.0℃), pour point (-10.0℃), cloud point (-2.0℃), acid value (Nil), iodine value (82.0 %), ash content (0.03%) moisture content (0.04%), calorific value (34,860 KJ/kg), Sulphur content (0.01%) Boiling point (202℃), calcium Ca (32.6667 mg/kg), Magnesium Mg (4.42 mg/kg), potassium K (2.32 mg/kg) and Sodium Na (2.15 mg/kg) and phosphorus (0.20, 0.10 mg/kg). Gas chromatography was used to determine biodiesel compositions. Infrared spectroscopy was used for the detection of functional groups and identification of organic compounds. The quality parameters of the biodiesel were found to be within international acceptable ASTM 6751 and EN14214 standards. The Hura crepitans (HC) is a good feedstock for biodiesel production.

1 Introduction

The need for energy is anticipated to increase due to rapid increase in population, expanding urbanization and to improve the living standards [1]. Fossil fuels are non-renewable energy resources. In spite of the fact that these fuels are assisting fully to the world energy supply, the production and usage have created environmental concerns and political argument. 98% of carbon emissions have been shown to come out of fossil fuel combustion [2]. The desire for energy is continuously increasing owing to the quick growth in the number of industries and vehicles and due to population explosion. Petroleum, natural gas, coal, hydrocarbon and nuclear energy are the sources of energy. The main drawbacks of using petroleum-based fuels are atmospheric pollution caused by the use of petroleum diesel. The emission of several greenhouse gases is created by the petroleum diesel combustion. Various fuels have been studied as well to replace diesel fuel completely. Vegetable oils are produced in rural areas which makes it a promising alternative to diesel. The oil gotten from seed can give self-employment opportunities [3]. Vegetable oil

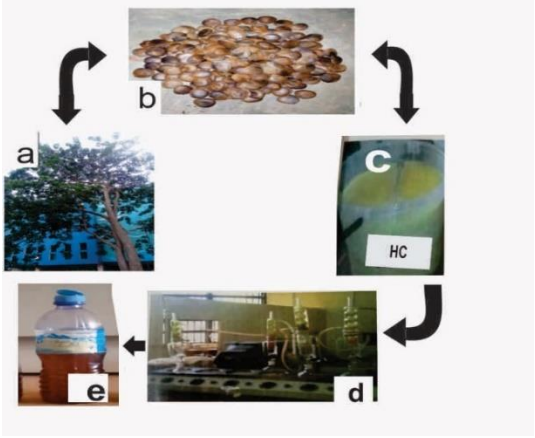

Figure 1: Soxhlets method for Hura crepitans (Hc) seed oil extraction

[*]**CORRESPONDING AUTHOR** | Agbogu Ada | ✉ada.agbogu@unn.edu.ng

(peanut oil) was also examined in diesel engine as a fuel at first by Rudolf in 1911. During the study on other fuels, it was observed that the vegetable oil can also be used in diesel engine with no modifications.

Different harmful matters are emitted by engine exhaust (smoke, unburnt hydrocarbons, carbon monoxide, particulate matter and nitrogen oxides), which are hazardous for human being and the environment. Nitrogen oxide and smoke are the most harmful pollutant [4, 5]. According to [6]. Carbon monoxide and other gases retained in the environment are as well the primary cause for climate condition change. The amount of CO_2 has been calculated to become larger by 80% approximately from 2007 to 2030 [7]. The decrease in fuel reserves and rising environmental issues has driven the attention of researchers towards the alternative fuels instead of conventional fuels [8, 9]. In current era different countries have stress and recommend the use of alternative fuel like biodiesel fuel by governmental and regulatory track by using both incentives and prescriptive volumetric certainty. Vegetable oil is a better source of energy as alternative fuels from economic and emission quality point of view. [10, 11].

Biodiesel is a mono alkyl ester that is produced from fatty acid ester of edible oil, non-edible oil and waste oils [12]. Biodiesel is used directly in engine in its clean form or blend with diesel in different proportions to give alternative solution of fuel in compression ignition (CI)engines. It is a renewable source of energy, oxygenated, Sulphur-free, sustainable and biodegradable. In diesel engine no modifications are required while using biodiesel as fuel [13, 14]. As compared to diesel fuel biodiesel shows less regulated and unregulated emissions [10]. Different reasons for biodiesel used as alternative focus on reducing greenhouse gas (GHG) emissions, little effect on global climate and renewable energy solution and to have more promising alternative fuel supply to meet the present energy demand. With the aid of biodiesel emissions of particulate matter, unburned hydrocarbons, carbon monoxide and carbon dioxide can be lessened [6], [8], [15]. The Biodiesel obtained from raw vegetable oil has its properties very close to diesel, in order that it can be used as an alternate fuel. The chief benefit of biodiesel as compared to diesel fuel are eco-friendliness, renewability, high flash point, biodegradability and non-toxicity [16]. Biodiesel is used in the transport sector as alternative solution to diesel fuel because it has same properties to petroleum diesel and decrease in emissions [17, 18]. With rise in biodiesel usage, it could less the pollutants and movable carcinogens [19].

[20] state various sources of feedstocks like vegetable oil can be use in producing biodiesel. The following techniques are employed in biodiesel production: Transesterification, pyrolysis and supercritical fluid method etc. Among these techniques, transesterification is the hot favourite, in which biodiesel and glycerol can be obtained as secondary product from the oil [21]. In the production of biodiesel, a vegetable oil or animal fat reacts with ethanol or methanol in the presence of a catalyst. From this, methyl or ethyl esters are obtained, which are the components of biodiesel. Along with the esters, glycerol is also produced [22]. That reaction is called transesterification. Stoichiometrically, when the reaction takes place, for every mole of triglycerides reacting, three moles of alcohol are used. However, a higher molar ratio of alcohol is usually used for maximum ester production [23]. Factors affecting transesterification reaction are: presence of moisture and free fatty acids (FFA), reaction time, reaction temperature, catalyst and molar ratio of alcohol and oil [24, 25]

Hura crepitans (Hc) seed usually called sandbox tree seed trees are found in the family Euphorbiaceae and the seeds are known to have great potential to yield oil [27]. Extraction of oil is one of the processes involved in biodiesel production [28]. The aim of the study was to extract Hura crepitans seed oil using soxhlet extraction method and convert the oil into biodiesel by transesterification process and characterize both the oil and biodiesel to obtain their physiochemical properties that determine their suitability for use as biodiesel.

2 Materials & Methods

2.1 Materials

The reagents in the study were used without further purification: potassium hydroxide (KOH), n-hexane and analytical methanol (CH_3OH). All the chemicals used were analytical reagent grade except industrial hexane (Sigma Aldrich). Potassium hydroxide pellet, Methanol were purchased from Sigma Aldrich. Other materials include: soxhlet extractor, oven, separating flask, electronic weighing balance, FTIR spectrometer (Shimadzu, Japan) and Gas Chromatography (GC), Buck 530, USA, HC seeds.

2.2 Methods

2.2.1 Extraction of oil from Hura crepitans seed

Soxhlet extraction method was used to extract the oil from Hura crepitans seeds. The study was carried out at the National Centre for Energy Research and Development (NCERD), University of Nigeria. Fresh and well ripe fruits of the Hura crepitans were harvested from the University community.

The seeds were removed from the fruits. 800g of the harvested Hura crepitans seeds were cleaned, de-shelled manually and air dried at ambient temperature (35 – 40°C) for one month to reduce the moisture content to 20%. The dried seeds were reduced to fine particle sizes with a mechanical grinding machine to enhance oil extraction, as described in a similar study [29, 30]. The sample was placed in a bucket, mixed 400 ml of n-hexane solvent and stirred continuously for one hour at room temperature. This process was repeated 3-4 times with the fine samples using 400 ml of fresh n-hexane at each time in order to extract most of the oil from the sample. The oil was collected from the mixtures by filtration method. The solvent in the extracted oil was removed by drying in the oven at 80°C and stored in a plastic container for characterizations as shown in figure 1.

2.2.2 Biodiesel production from Hura crepitans seed oil

90 ml of extracted Hura crepitans seed oil was measured into a 500 ml beaker mixed the catalyst; methoxide, consisting of 0.531 g, Potassium hydroxide (KOH) dissolved in 22.5 ml of methanol (CH_3OH), set to heat at a constant temperature of 600℃ and continuously stirred at a constant speed of 50 rpm to ensure high quality transesterification of the oil.

At the completion of transesterification reaction for 60 minutes, the mixtures were poured into separating flask and left to stand for 24 hours for proper separation into two layers of biodiesel (methyl esters) and glycerol. The upper layer which is the crude biodiesel was collected in a beaker and subjected to further purification in order to dry it and remove any trace of moisture whereas lower layer glycerol which is the bye product of the reaction was run off as illustrated in figure 2.

The general transesterification reaction equation is present in equation (1) [31]:

$$RCOOR^1 + R^{11}OH \overset{\text{catalyst}}{\Longleftrightarrow} R^1OH + RCOOR^{11} \quad (1)$$

Where $RCOOR^I$ is an ester, $R^{II}OH$ is an alcohol (methanol), R^IOH is another alcohol (glycerol), $RCOOR^{II}$ is an ester and a catalyst (KOH).

2.2.3 Purification of biodiesel

The synthesized biodiesel was purified to remove unwanted materials as oil, catalyst and methanol in order to obtain quality end product. The purified biodiesel obtained was washed (biodiesel mixed with water, the mixture was poured into a separating flask and two layers was formed, the upper one was the purified biodiesel) and dried in the oven at the temperature of 105℃ for 6 hours. Figure 2 is the schematics for the biodiesel production processes.

2.2.4 Material Characterizations

The oil content of the seeds was determined using equation (2):

$$\%\ oil\ yield = \frac{wieght\ of\ oil\ extracted\ in\ gram}{mass\ of\ sample\ dry\ taken} \times 100 \quad (2)$$

Moisture content: The moisture content was determined using equation (3):

$$Moisture\ content\ \% = \frac{mass\ of\ the\ sample - mass\ of\ dry\ matter}{mass\ of\ sample} \times 100 \quad (3)$$

The physicochemical properties were determined using standard procedures [31-33].

Ash content: The ash content was determined using equation (4) [31, 32]:

$$Ash\ content = \frac{mass\ of\ the\ ash\ in\ gram}{Volume\ of\ sample} \times 100 \quad (4)$$

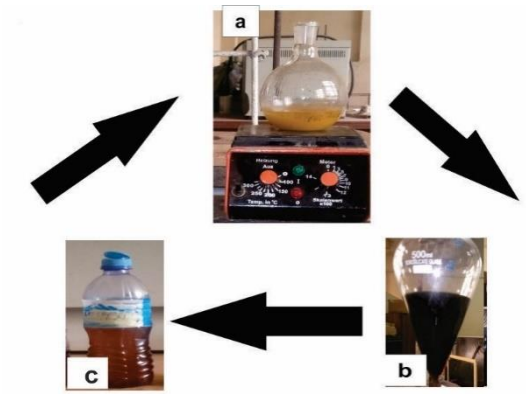

Figure 2: Transesterification process of biodiesel production from, Hura crepitans (a) Hc seed oil (b) Hc oil with catalyst (c) biodiesel

Density: The density was determined using equation (5):

$$Density = \frac{mass\ of\ the\ sample\ in\ gram}{Volume\ of\ sample} \quad (5)$$

Free fatty acid: The free fatty acid value was determined using equation (6):

$$Free\ fatty\ acid = \frac{T \times N \times NM}{W} \quad (6)$$

Where W is the weight of the sample, T is the titer value.

Acid value: The acid value was determined using equation (7):

$$Acid\ value = \frac{56.1 \times N \times T}{W} \quad (7)$$

Where T is the titer value, N is the normality Sodium thiosulphate and W is the weight of the sample.

Viscosity: The kinematic viscosity was determined using equation (8):

$$Kinematic\ viscosity\ \eta = \frac{\eta_w \rho_1 t_1}{\rho_w t_w} \quad (8)$$

Where t_1 time of flow is, t_w is taken for water to flow from top mark to bottom mark, ρ_1 is the density of the sample, η_w is the viscosity of the water and ρ_w is the density of water.

Sulphur content: The Sulphur content was determined using equation (9):

$$Sulphur\ content\ \% = \frac{0.1373b}{w} \times 100 \quad (9)$$

Where W is the weight of the sample and b is the weight of $BaSO_4$ ppt.

Iodine value: The Iodine value was determined using equation (10):

$$\text{Iodine value } (W_{ijrs}) = \frac{(B - T) \times N \times 12.69}{W} \quad (10)$$

Where T is the sample titre, B is blank titre, N is normality of Sodium thiosulphate, and W is the weight of the sample.

3 Results and Discussions

3.1 HCSO Extraction

The ground seeds of Hura crepitans sample was placed in a bucket and 400 ml of n-hexane solvent was poured into the bucket containing the ground seeds each to enable n-hexane solvent to react with the fine particles of the seeds, bringing out the oil from the seeds. The mixture was stirred continuously for one hour at room temperature.

This process was repeated 3-4 times with the fine particles of seeds using fresh n-hexane (400 ml) at each interval in order to extract most of the oil from the seeds. The liquid mixture was collected from the sample (seeds) and filtered into a bottle using a filter paper. At the end of extraction, the solvent (n-hexane) was recovered from the oil using soxhlet extractor set at 66℃ as illustrated in figure 1.

Figure 1 shows the Soxhlet extraction method for the synthesis of the Hura crepitans. The percentage yield of the HCSO was 42.0% at 66℃. furthermore, the oil obtained was dried in the oven at 80℃ to remove all traces of solvent (n-hexane) in it and stored in a plastic container for further use.

3.2 Fatty Acid Composition

The gas chromatographic (GC) analysis of the fatty-acid's composition Hura crepitans, is as presented in Table 1. The HCSO has 79.1% saturated fatty acid and 39.2% unsaturated, hence, it was dominated by the high amount of saturated fatty acids. Fatty acid composition includes long chain saturated fatty acids and unsaturated fatty acids, which are the important factors that determine the quality of a biodiesel before moving into the production of diesel [34 – 35].

The presence of one carbon-carbon double bond means the fatty acid is monounsaturated, while the presence of many carbon-carbon doubles bonds means polyunsaturated fatty acids. The fatty acid composition of *HCSO* obtained agreed with values obtained in previous studies [36, 37]. Hence acceptable as a feedstock for biodiesel production [38, 39].

Table 1: Fatty acid composition of HCSO

Fatty acid	Structure	HC
Lauric acid	$C_{12:0}$	8.8
Myristic acid	$C_{14:0}$	12.7
Oleic acid	$C_{18:1}$	29.1
Palmitic acid	$C_{16:0}$	18.3
Margaric acid	$C_{17:0}$	24.1
Linoleic acid	$C_{18:2}$	15.7
Linolenic acid	$C_{18:3}$	1.9
Arachidic acid	$C_{20:0}$	4.8
Eicosapentaenoic	$C_{20:5}$	39.2
Docosapentaenoic	$C_{22:5}$	31.4
Docosahexaenoic	$C_{22:6}$	32.3

Table 2: Gas chromatography (GC) composition analysis of biodiesel

Parameters	% Composition HC
Triglyceride	7 .55
Alcohol	8.61
Fatty acid methyl ester	71.86
Monoglyceride	5.93
Glycerol	2.69
Diglyceride	3.35

3.3 Gas chromatography (GC) composition analysis of Hura crepitans biodiesel

Quantitative results of GC analysis are presented in Table 2 and showed 71.859% biodiesel yield from Hura crepitans. The different stages of the transesterification reaction were observed by Gas Chromatography.

GC was used to observe the process of biodiesel conversion from triglyceride to diglyceride to monoglyceride. The monoglyceride was further broken down into fatty acids methyl ester (biodiesel) and glycerol as the by-product with methanol in the presence of a catalyst. (The oven temperature was set to 180℃ and the GC was allowed to warm up. While it was warming, the temperature condition of GC was set to 70℃ and 220℃ for initial temperature, 5 minutes and 2 minutes for the hold, 10 minutes, and 5 minutes for the ramp and 220℃ and 280℃ for the final temperature).

3.4 Physicochemical Properties of the oil

The results of the physicochemical properties of the Hura crepitans and is presented in Table 3 revealed that the oil was good for biodiesel production when compared with the work of [40 – 42].

3.5 Physicochemical properties of the biodiesel

Table 4 shows the result of the physicochemical properties of the biodiesel from Hura crepitans The properties obtained agrees with the international standards for biodiesel with exception of the flash point of the biodiesel which was higher than the given limit.

This is considered as an added advantage because it indicates high non-flammability of the biodiesel relative to petro-diesel [43, 44]. Iodine value obtained from the biodiesel showed little variation when compared with the EN and ASTM standard value.

Also, the results of the alkaline metals, alkaline earth metals and phosphorus found among the physicochemical properties (Table 3) showed that the obtained values when compared with ASTM6751 & EN14214 were within the acceptable standards and agrees with values obtained in previous studies of [45].

Table 3: Physicochemical parameters of the HC

Property	Hura crepitans seed oil
Density (kg/m3)	0.910
Specific Gravity	0.909
Viscosity (mm2/s)	3.1
Refractive index	1.467
Peroxide value, Meq/Kg	1.1
Acid value (mg KOH/g)	0.90
Iodine value	99.4
Saponification value m(gKOH/g)	160.4
Free fatty acid (%)	0.45

The high values of calcium in *HC* could be as a result of the type of fruit specie, the environmental factors and the type of soil where the fruits were planted. Table 4 shows the comparison of physicochemical properties of *HC* with biodiesel standard (ASTM 6751 (USA) and EN 14214 (Europe)).

3.6 Infrared spectroscopy (FTIR) analysis

Table 5 and 6 are the results of the Fourier transform infrared spectrometry (FTIR) analysis used to identify and evaluate the possible unknown compounds and functional groups (the type and the nature of functional groups) present in the oil and biodiesel produced.

The presence and the nature of functional groups provide information on the reactivity and the stability of the biodiesel fuel as well as the efficiency conversion of the oil to biodiesel. The generated spectrum was used to identify the functional groups in the fuel sample for qualitative analysis and associated type of vibrations. The samples of biodiesel and oil were scanned within mid-infrared region of 4000 cm^{-1} – 400 cm^{-1} with Shimadzu FTIR Spectrometer.

The FTIR spectrum for the biodiesel were interpreted with the aid of [46] and it revealed that the functional groups present in oil and biodiesel are with characteristics bands of esters (C=O), Alkene (C=C) and Alkanes (C-H).

The absorption bands in oils from HC seed indicate that the oil contains characteristics functional groups of C=O esters carbonyl group and C-O-C esters, revealing that the oil molecules contain esters functional groups. The functional groups of C=C (alkenes), C–H (alkanes) and the triple bond C≡C (alkynes) that must be present in every good oil was noticed in the FTIR analysis of the oil.

Table 4: Physicochemical properties of Hura crepitans biodiesel compare with Biodiesel standard ASTM D6751 and EN 14214

Properties	HC biodiesel	ASTM6751	EN14214
Specific gravity	0.902	-	-
Density at 30°C (kg/m^3)	903	-	860-900
Kinematics viscosity (mm^2/s) at 40°C	2.4	1.9-6.0	3.5-5.0
Flash point (°C)	193	≥ 130	≥ 120
Pour point (°C)	-10	0	-
Cloud point (°C)	-2	Report	-
Acid value (mg KOH/g)	Nil	≤ 0.80	≤ 0.50
Iodine value ($W_{ij's}$)	82.2	-	120
Ash content (%)	0.03	-	-
Moisture content (%)	0.04	-	≤ 500
Calorific value (KJ/kg)	36,120	-	≥ 35,000
Sulphur content (%)	0.01	≤ 0.05	≤ 0.001
Boiling point (°C)	202	-	-
Saponification value (mgKOH/g)	224.00	-	-
Ca (Calcium) Mg/kg	32.67	-	≤ 5.0
Mg (Magnesium) Mg/kg	4.42	-	≤ 5.0
K (Potassium) Mg/kg	2.32	-	≤ 5.0
Na (Sodium) Mg/kg	2.15	-	≤ 5.0
P (Phosphorus) Mg/kg	0.10	-	≤ 5.0

From Table 5, the absorption peaks at 3579.68 – 3176.40 cm^{-1} were assigned to the intermolecular hydrogen-bonded O-H of alcohols which strongly indicated the presence of alcohol functional groups in the esterified biodiesel.

Table 5: FTIR absorbance spectra of HC

S/N	WAVE NUMBER (cm^{-1})	TYPE OF VIBRATION	FUNCTIONAL GROUP
1.	3570.32 – 3208.33	Stretching	Intermolecular hydrogen bonded O-H (alcohol)
2.	3120.52 – 3021.25	Stretching	C – H (alkene)
3.	2834.95	Stretching	C – H (alkane)
4.	2233.14– 2110.34	Stretching	C ≡ C (alkyne)
5.	1795.93	Stretching	C=O (Ester)
6.	1667.44	Stretching	C=C (alkene)
7.	1499.16	Asymmetrical bending	C – H (alkane)
8.	1388.42 – 1176.25	Sym-and asym-stretching	C-O-C (Ester)
9.	997.12 – 680.53	Out of plane bending	C – H (alkene)

Table 6: FTIR absorbance spectra of HC biodiesel

S/N	Wave number (cm^{-1})	Type of vibration	Functional group
1.	3579.68 – 3176.40	Stretching	Intermolecular hydrogen bonded O-H(alcohol)
2	3006.88	Stretching	C – H (alkene)
3.	2800.51	Stretching	C – H (alkane)
4.	2281.22 – 2065.90	Stretching	C ≡ C (alkyne)
5.	1715.35	Stretching	C=O(Ester)
6.	1605.16	Stretching	C=C(alkene)
7.	1482.86 – 1386.35	Asymmetric bending	C – H (alkane)

The characteristic strong stretching vibration at 3006.88 cm^{-1} was assigned to the C-H stretching vibrations of the alkene moieties signifying the presence of C=C unsaturation present in the fatty acid moieties of the trans-esters (biodiesel). The C-H stretching of alkanes was responsible for the stretching vibrations at 2800.51cm^{-1}, and the absorption peaks at 2281.22 – 2065.90 cm^{-1} were assigned to the C≡C stretching vibration of a non-terminal alkyne since there was the absence of C-H stretching vibration around 3310 – 3300 cm^{-1} for terminal alkynes. The C=O stretching vibration for the ester functional group was duly identified at 1715.35 cm^{-1} and the C=C stretching vibration was assigned to the peak at 1605.16 cm^{-1}.

The absorption bands below 1500cm^{-1} are regarded as the fingerprint region because every compound has a unique absorption pattern within the region [44] and it is very useful in differentiating the biodiesel from the oil(triglyceride) or it is used to show that the transesterification process was successful [45]. The strong asymmetric bending vibration at 1386.35 indicates the presence of the $–CH_3$ group which confirms the formation of the methyl ester(biodiesel). The characteristic peaks at 1219.79 and 1017.86cm^{-1} indicate the presence of the C-O-C symmetric and asymmetric stretching vibration of an ester and the out of plane bending vibration is duly identified at 866.54 - 751.36 cm^{-1}.

4 Conclusion

Biodiesel was synthesized from HCSO using alkaline catalytic transesterification. The oil was extracted from AMS, HCS, TNS using Soxhlet extraction method. The extracted oil was converted to methyl ester (biodiesel) using alkaline catalyzed transesterification method at temperature of 60.0℃. The oil and biodiesel produced was characterized for their physicochemical properties.

The comparison of the biodiesel properties with standards showed close agreement with biodiesel standard (ASTM6751 and EN 14214). The results revealed that alkaline catalyzed transesterification method is good and safe method for producing biodiesel even at large scale. The functional group of the biodiesel study indicate high level of ester (biodiesel) functional group. The quantity is high enough yield and can be an alternative renewable source for diesel engines. High quality yield of 71.86% and low lead content of the produced biodiesel make Hura crepitans seed a good feedstock for biodiesel. Hence, *Hura crepitans* is a good feedstock for biodiesel.

Acknowledgements

The authors acknowledge the National Centre for Energy Research and Development, University of Nigeria for their assistance with the laboratory work. This research did not receive any specific grant from funding agencies in the public, commercial or not-for-profit sectors.

Declaration of Competing Interest

The authors declare no conflict of interest in the funding of this work.

References

[1] Y. G. Keneni and J. M. Marchetti, “Oil extraction from plant seeds for biodiesel production,” vol. 5, no. March, pp. 316–340, 2017, doi: 10.3934/energy.2017.2.316.

[2] A. Ef, E. Betiku, I. Dio, and O. Tv, “Production of biodiesel from crude neem oil feedstock and its emissions from internal combustion engines,” vol. 11, no. 22, pp. 6178–6186, 2012, doi: 10.5897/AJB11.2301.

[3] H. Mulimani, M. C. Navindgi, and T. P. Engineering, “Extraction of biodiesel from vegetable,” vol. 2, pp. 242–250, 2012.

[4] V. Senthilkumar, P. Gunasekaran. Bioethanol production from cellulosic substrate: Engineering bacteria and process integration vol. 64, pp.845-853, 2005.

[5] A. Ramadhas, S. Jayaraj, C. Muraleedharan. Theoretical modelling and experimental studies on biodiesel fueled engine, vol. 31, pp. 1813-1826

[6] B. Metz and C. I. W. G. Iii, “Mitigation of Climate Change Fourth Assessment Report,” 2007.

[7] Nabi, M.N., Rahman, M.M. and Akhter, M.S. (2009) Biodiesel from Cotton Seed Oil and Its Effect on Engine Performance and Exhaust Emissions. Applied Thermal Engineering, 29, 2265-2270. https://doi.org/10.1016/j.applthermaleng.2008.11.009

[8] P. K. Sharma et al., “rip t N ot Co py ed ite d Ac ce pt ed t N ot Co py ed d,” no. c, 2019, doi: 10.1115/1.4042717.

[9] No SY (2011) Inedible vegetable oils and their derivatives for alternative diesel fuels in CI engines: a review. Renew Sust Energ Rev 15: 131–149

[10] M. Gumus and S. Kasifoglu, "Performance and emission evaluation of a compression ignition engine using a biodiesel (apricot seed kernel oil methyl ester) and its blends with diesel fuel," Biomass and Bioenergy, vol. 34, no. 1, pp. 134–139, 2010, doi: 10.1016/j.biombioe.2009.10.010.

[11] A. K. Azad, M. G. Rasul, M. M. K. Khan, S. C. Sharma, and M. A. Hazrat, "Prospect of biofuels as an alternative transport fuel in Australia," Renew. Sustain. Energy Rev., vol. 43, pp. 331–351, 2015, doi: 10.1016/j.rser.2014.11.047.

[12] D. Singh, D. Sharma, S. L. Soni, S. Sharma, and P. K. Sharma, "Review article A review on feedstocks , production processes , and yield for di ff erent generations of biodiesel," Fuel, no. July, p. 116553, 2019, doi: 10.1016/j.fuel.2019.116553.

[13] M. Shahabuddin, A. M. Liaquat, H. H. Masjuki, M. A. Kalam, and M. Mofijur, "Ignition delay , combustion and emission characteristics of diesel engine fueled with biodiesel," Renew. Sustain. Energy Rev., vol. 21, pp. 623–632, 2013, doi: 10.1016/j.rser.2013.01.019.

[14] Z. Hu, P. Tan, X. Yan, and D. Lou, "Life cycle energy, environment and economic assessment of soybean-based biodiesel as an alternative automotive fuel in China," vol. 33, pp. 1654–1658, 2008, doi: 10.1016/j.energy.2008.06.004.

[15] M. J. Abedin, H. H. Masjuki, M. A. Kalam, A. Sanjid, S. M. A. Rahman, and I. M. R. Fattah, "Performance , emissions , and heat losses of palm and jatropha biodiesel blends in a diesel engine," Ind. Crop. Prod., vol. 59, pp. 96–104, 2014, doi: 10.1016/j.indcrop.2014.05.001.

[16] W. Shi et al., "Bioresource Technology Biodiesel production from waste chicken fat with low free fatty acids by an integrated catalytic process of composite membrane and sodium methoxide," Bioresour. Technol., vol. 139, pp. 316–322, 2013, doi: 10.1016/j.biortech.2013.04.040.

[17] T. Nguyen, L. Do, and D. A. Sabatini, "Biodiesel production via peanut oil extraction using diesel-based reverse-micellar microemulsions," Fuel, vol. 89, no. 9, pp. 2285–2291, 2010, doi: 10.1016/j.fuel.2010.03.021.

[18] G. Baskar and R. Aiswarya, "Trends in catalytic production of biodiesel from various feedstocks," Renew. Sustain. Energy Rev., vol. 57, pp. 496–504, 2016, doi: 10.1016/j.rser.2015.12.101.

[19] Y. Tang, X. Gu, and G. Chen, "99 % yield biodiesel production from rapeseed oil using benzyl bromide – CaO catalyst," pp. 203–208, 2013, doi: 10.1007/s10311-013-0403-9.

[20] M. Kaur and A. Ali, "Lithium ion impregnated calcium oxide as nano catalyst for the biodiesel production from karanja and jatropha oils," Renew. Energy, vol. 36, no. 11, pp. 2866–2871, 2011, doi: 10.1016. j.renene.2011.04.014.

[21] D. Rajagopal et al., "Linked references are available on JSTOR for this article : Recent Developments in Renewable Technologies : R & D Investment in Advanced Biofuels," vol. 1, no. 2009, pp. 621–644, 2019.

[22] Demirbaş, A. (2008). Environmental Effects Production of Biodiesel from Algae Oils Production of Biodiesel from Algae Oils. 7036. https://doi.org/10.1080/15567030701521775

[23] Singh, D., Sharma, D., Soni, S. L., Sharma, S., & Sharma, P. K. (2019). Review article A review on feedstocks , production processes , and yield for di ff erent generations of biodiesel. Fuel, July, 116553. https://doi.org/10.1016/j.fuel.2019.116553

[24] Parawira, W. (2010) Biodiesel production from Jatrophacurcas: A review. Scientific Research and Essays, 5(14), 1796-1808.

[25] Highina, B.K., Bugaje, I.M., Umar, B. (2012) Biodiesel Production from Jatropha Caucus Oil in a Batch Reactor Using Zinc Oxide as Catalyst, Journal of Applied Phytotechnology in Environmental Sanitation, 1(2), 61-66.

[26] M. A. Fowomola and A. A. Akindahunsi, Nutrients andAntinutrients of Hura crepitan seed. Journal of Medicinal Food, 10(1):159-164, 2007

[27] M. A. Fowomola and A. A. Akindahunsi, Nutrients andAntinutrients of Hura crepitan seed. Journal of Medicinal Food, 10(1):159-164, 2007

[28] Anthony Cemaluk C. Egbuonu, Olorunshola D. Omodamiro Christian E. Odo1 and Roberts I. Uroko1. Some Antinutritive and Antioxidative Properties of Pulverized Citrus sinensis (Sweet Orange) Peels and Seeds. Journal of Scientific Research Reports,10 (6);1-9

[29] Ibrahim I. A. Yusuf A. J. Extraction and physicochemical analysis of Citrus sinnesis seed oil (sweet orange). European Journal of Experimental Biology, 2015, 5(7): 77-81

[30] Narasimharao K, Lee A, Wilson K. Catalysts in production of biodiesel: a review. Journal of Biobased Materials and Bioenergy 2007; 1: 19–30

[31] Reuther W. The Citrus Industry: Crop protection, postharvest technology, and early history of citrus research in California 3326 UCANR Publications;1967

[32] Hoekman SK, Broch A, Robbins C, Ceniceros E, Natarajan M. Review of biodiesel composition, properties, and specifications. Renewable and Sustainable Energy Reviews 2012; 16: 143–169.

[33] Atabani AE, Silitonga AS, Badruddin IA, Mahlia TMI, Masjuki HH, Mekhilef SA comprehensive review on

biodiesel as an alternative energy resource and its characteristics. Renewable and Sustainable Energy Reviews 2012; 16: 2070–2093.

[34] Ezekoye Veronica , Adinde Rita , Ezekoye David , Offormata Anthony , syntheses and characterization of biodiesel from citrus sinensis seed oil, Scientific African (2019), doi: https://doi.org/10.1016/j.sciaf.2019.e00217

[35] Ashraful AM, Masjuki HH, Kalam MA, Fattah IR, Imtenan S, Shahir SA, Mobarak HM. Production and comparison of fuel properties, engine performance, and emission characteristics of biodiesel from various non-edible vegetable oils: a review. Energy Conversion and Management 2014;80: 202–228.

[36] J. O. Oyelade, D. O. Idowu, O. O. Oniya & O. Ogunkunle (2017):Optimization of biodiesel production from sandbox (Hura crepitans L.) seed oil using two different catalysts, Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, doi: 10.1080/15567036.2017.1320691

[37] M. Abdulkadir, I. Amoo, A. Adesina. Investigation of Solvents Efficiencies on Oil Extraction and Biodiesel Production from Hura Crepitans Seeds 2013; 3: 2319 – 7064

[38] Ahmed F, Giwa SO, Ibrahim M, Giwa A. Production of biodiesel from Jatropha curcas seed oil using base catalysed transesterification. International Journal of ChemTech Research 2016; 9: 322–332.

[39] Pramanik K. Properties and use of Jatropha curcas oil and diesel fuel blends in compression ignition engine. Renewable Energy 2003; 28 :239–248.

[40] Ved K, Padam K. Study of physical and chemical properties of biodiesel from sorghum oil. Research Journal of Chemical Sciences 2013; 3: 64–68.

[41] Barabas I, Todorut IA Biodiesel quality, standards and properties. In: Biodiesel-quality, emissions and by. Intech; 2011.

[42] AOAC (Association of Analytical Chemist): Official Method of Analysis 13th ed. (1990). In: William Horwitz. editor, Washington DC; Association of Official Analytical Chemists; 1990 (7) p. 56-132.

[43] Achten WM J, Verchot L, Franken Y, Mathijs E, Singh VP, Aerts R, Muys B. Jatropha bio-diesel production and use. Biomass and Bioenergy, 2008; 32:1063–1084.

[44] Yadav L DS. Organic spectroscopy. Springer Science & Business Media; 2015

[45] Samuel Kofi Tulashie & Francis Kotoka (2019): Kinetics and thermodynamic studies on Moringa oleifera oil extraction for biodiesel production via transesterification, Biofuels, DOI: 10.1080/17597269.2019.169704