

Production, Characterisation and Fatty Acid Composition of *Jatropha curcas* Biodiesel as a Viable Alternative to Conventional Diesel Fuel in Nigeria

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Abstract

Ethyl ester biodiesel has been produced from a non-edible *Jatropha curcas* oil. Oil was extracted from the plant seed using n-hexane at 60°C and pre-treated by alkaline refining process to reduce the free fatty acid level to less than 1%. Base-catalysed transesterification reaction with absolute ethanol using potassium hydroxide catalyst was adopted for the conversion. Various physicochemical properties of the refined *Jatropha curcas* oil were investigated. The ethyl ester biodiesel produced was characterised for its fuel properties such as specific gravity at 15°C, flash point, pour point, kinematic viscosity, cetane number, iodine value and higher heating value using American Society for Testing and Materials Standard Methods. The crude and refined *Jatropha curcas* oil yields were 58.16% and 52.5%. The physicochemical analysis revealed FFA, saponification value and peroxide value of refined *Jatropha curcas* oil to be 0.58 mg KOH/g, 159.9 and 1.92 m E/kg respectively. The fatty acid composition obtained from gas chromatography (GC) revealed that the oil contained 44.85% oleic acid as the dominant fatty acid, while Margaric 0.01% and Behenic 0.02% the least. The biodiesel yield was 57.6%, and its measured fuel properties conformed with ASTM 6751 and EN 14214 standards.

Keywords

Biodiesel, Ethyl Ester, Alternative Fuel, Refined *Jatropha curcas* Oil, Transesterification

1. Introduction

The most desired properties of materials, products and industrial processes are

sustainability, eco-efficiency and industrial ecology. Achieving a sustainable, resource-efficient and low-carbon economy is one of the key fundamental challenges in the 21st century [1]. For many years, fossil fuel has been the major source of energy worldwide [2]. Large utilisation of fossil fuels due to increasing human activities has contributed to national energy security concern, environmental pollution and depleting oil reserves. This has, in turn, led to an increase in demand for viable and sustainable alternatives. Recently, many countries have explored renewable sources such as solar, wind, geothermal and agricultural feedstocks as alternative fuel sources. However, the agricultural feedstock can also help in generating an ecofriendly and efficient fuel that can compete effectively with fossil fuel and even capture market currently dominated by the latter. Fuel from plant resources such as vegetable oil is known as a cheaper renewable source of energy [3].

Vegetable oils have long been in use as a substitute for diesel fuel as far back as 1930 [4]. Many vegetable oils from palm, groundnut, rapeseed, and soybean with good yields have been investigated. Non-edible oil such as jojoba and karanja oils have also been studied. Due to the problems associated with their use directly in conventional engines, vegetable oils are modified into biodiesel. Biodiesel is a promising alternative fuel. In the recent time, it is attracting the attention of many researchers because of its potential to address the growing concern of depletion of fossil fuel reserves and environmental problems [5]. Several conversion processes such as hydrotreatment and isomerisation, Fischer-Tropsch [6] and transesterification [3] have been reported. The most effective of these methods is transesterification, a reaction of oil with an alcohol to form ester and glycerol in the presence of an acid or base catalyst, due to its simplicity. It has been in commercial use not only in developed nations (e.g. Germany, France, Canada, Italy, New Zealand, etc.) but also in developing nations (India, Brazil, etc.). In some major oil exporting countries, biodiesel generates high foreign exchange savings. It has however been more attractive recently because of its environmental benefits, renewability, biodegradability, non-toxic nature and cost-effectiveness. Interestingly also, it has been promoted as a technical option to respond to climate change.

Jatropha curcas is a hardy plant that has the potential to grow under both arid and semi-arid conditions [7]. It belongs to the family of *Euphorbiaceae* [8] [9]. It is cultivated both in tropical and subtropical regions of the world and is high yielding. *Jatropha* is grown mainly to demarcate plots of land in Nigeria. Its oil has not been exploited for fuel purpose in the country [3]. Ideally, prime conditions for the choice of plant resources for the production of renewable fuel are abundance and low-cost. In sub-Saharan Africa, *Jatropha curcas* is readily available due to its ease of cultivation. It has high oil yield and maintains no competition with food. However, for a monocultured economy, such as Nigeria, in need of diversification, the use of *Jatropha* can be a viable alternative by harnessing its potential for biodiesel production on a large scale. Biodiesel ethyl esters of some vegetable oils using absolute ethanol have been reported [10] and often the

choice of vegetable oils for biodiesel production is a function of its abundance in the region of testing [11]. Based on the aforementioned excellent features, *Jatropha curcas* is thought to be an appropriate and sustainable alternative feedstock for the production of biodiesel. Consequently, the potential of using *Jatropha curcas* oil as feedstock for biodiesel production is the focus of this study.

This work provides technical data that can assist Nigeria in reaching informed decisions on industrial exploitation of local *Jatropha* seed for biodiesel production. It aimed at studying the physicochemical properties of *Jatropha curcas* seed oil sourced locally in Nigeria, to synthesise its ethyl ester biodiesel via base-catalysed transesterification process and to investigate the regulated and unregulated properties of the biodiesel thereof using standard techniques. This research provides a sustainable strategy, a viable alternative and renewable source for energy generation against over-dependence on fossil fuel that contributes to national energy security concern and environmental degradation.

2. Materials and Methods

2.1. Materials

All reagents (n-hexane, absolute ethanol and sodium sulphate) used were of analytical grade. All analyses were conducted according to standard test methods. The flash point was determined using a Pensky-Martens Closed Flash Tester (K-16270) by Kehler Instrument Company. A 6200 Automatic Isoperibol Oxygen Bomb Calorimeter was used to determine the higher heating value of the biodiesel. ASTM D613 was used to determine cetane number [12], for iodine value, ASTM D445 for kinetic viscosity and the pour point determination was carried out according to ASTM D97.

2.2. Oil Extraction and Pretreatment

Jatropha curcas seeds were collected from Ado-Ekiti and Igbara Odo, both towns in Ekiti State, Nigeria. The seeds were identified according to the flora of West Africa in the Department of Plant Science and Biotechnology, Ekiti State University, Ado-Ekiti, Ekiti State. The seed shells were cracked to remove the seed (endosperms), air dried and later oven dried at 60°C to constant weight. The dried seeds were crushed into cake with mortar and pestle before extracting the oil by Soxhlet extraction using n-hexane at 60°C. The extracted oil was refined by alkaline refining process as follows: 0.16 g of 18 M NaOH was added to 30 g of oil and agitated for 30 min. The mixture was heated to 75°C and centrifuged. The physicochemical analyses conducted on the oil include acid value, % FFA as oleic acid, peroxide value, saponification value and specific gravity at 15°C. These analyses were conducted according to the methods described by the Association of Official Analytical Chemists [13].

2.3. Synthesis of Biodiesel Ethyl Ester of Refined *Jatropha curcas* Seed Oil

Base-catalysed transesterification was adopted for this process. Ethanol was

dried over anhydrous sodium sulphate prior to use. Potassium ethoxide was prepared by dissolving 0.37 g KOH (catalyst) in 16.6 ml absolute ethanol in a 250 ml beaker. The potassium ethoxide was gently introduced into 200 ml refined oil in a 1 L round bottom flask. The mixture was stirred and refluxed at 70 °C for 30 min. This was later transferred into a separating funnel and allowed to stand for 1 h in order to separate the mixture into layers. The lower layer was run off while the upper layer ran into a round bottom flask and refluxed again at 70 °C at 30 min with another potassium ethoxide catalyst. The ethyl ester was washed with 20 ml distilled water five times, drained into a beaker and dried over anhydrous sodium sulphate. The fatty acid ethyl ester was analysed for its fuel properties (specific gravity, flash point, pour point, kinetic viscosity, cetane number, iodine value and higher heating value) and tested according to India, ASTM 6751 and EN 41214 Biodiesel Standards.

2.4. Gas Chromatograph Analysis

Agilent 6890 Gas chromatograph System equipped with HP INNOWAX (30 m × 0.25 mm × 0.25 μm) and Agilent ChemStation software was used for the analysis using the following conditions: split injection (split ratio: 20:1), carrier gas: nitrogen, inlet temperature: 250 °C; oven programme: initial temperature at 60 °C, ramped at 12 °C/min for 20 min and then maintained for 2 min, ramped again at 15 °C/min for 3 min and then maintained for 8 min, detector: FID, detector temperature: 320 °C, hydrogen pressure: 22 psi, compressed air: 35 psi. The fatty acid was determined as ethyl ester. The ethyl ester was prepared following a standard procedure for GC analysis as follows: 20 mg of the oil was mixed with 2 cm³ of toluene. Then 2 cm³ of 1.5% sulphuric acid in ethanol was added. The mixture was stirred and incubated at 55 °C overnight. 4 cm³ saturated solution of sodium chloride was added and vortexed. 2 cm³ HPLC grade hexane was added followed by addition of 3 cm³ of 2% NaHCO₃. The mixture was also vortexed, and 180 μl of the upper phase was taken for GC analysis.

3. Results and Discussion

3.1. Physicochemical Analysis

Results of physicochemical analyses carried out on the oil as shown in **Table 1** include acid value, peroxide value, saponification value and specific gravity. The

Table 1. Physicochemical properties of refined *Jatropha curcas* seed oil.

Properties	Acid Value (mg KOH/g)	Saponification Value (mg KOH/g)	Peroxide Value (mE/kg)	% FFA as Oleic Acid	% Refined Oil Yield	Specific gravity
Jatropha oil ^a	1.16	159.9	1.92	0.58	52.5	0.882
Jatropha oil ^b	-	193.55	1.93	2.23	-	0.903
Jatropha oil ^c	35.8	193	-	-	-	0.895

^aResult of this study, Crude Oil Yield = 58.16%; ^b[16]; ^c[17].

yield of the oil is an important parameter that can determine its cost for biodiesel production. The oil content of *Jatropha curcas* seed (58.16%) was found to be higher than 47.25% and 49.1% reported by Akintayo and Martin *et al.* respectively [14] [15] but lower than that of Malaysian *Jatropha curcas* crude oil (63.16%) as reported by Akbar *et al.* [16]. After alkaline pretreatment, the yield was found to be 52.5%. However, the high oil content of *Jatropha curcas* indicates its suitability as a non-edible vegetable oil feedstock for biodiesel production.

Acid value is a measure of the acidic component in an oil sample. It is determined as the mass in mg of KOH required to neutralise the free fatty acid components in one gram of sample and is used to monitor oil degradation during storage. This was carried out by titration according to ASTM D664. The experimental result revealed a very high acid value of the extracted oil of 4.56 mg KOH/g. This high value explains the reason why the oil was subjected to alkaline refining prior to the production of biodiesel. Due to the chosen strength of potassium hydroxide used for refining (*i.e.* to neutralise some of the free fatty acids), the acid value of *Jatropha curcas* oil was reduced to 1.16 mg KOH/g and the % FFA (as oleic acid) reported as 0.58, values appropriate to complete the reaction with base catalysts. About 74.6% reduction was achieved for 1 h after refining. *Jatropha curcas* oil with FFA level of <1% has been reported to give a high yield of biodiesel [9]. This value was far lower than 35.8 mg KOH/g and 9.78 mg KOH/g obtained by Aransiola and Lau *et al.* [17] [18] respectively, for crude *Jatropha* oil.

Saponification value is a measure of the mean molecular weight of the fatty acid present in an oil sample. Result obtained for oil under investigation revealed a saponification value of 159.9, which was lower than 193 obtained by [16] for Malaysian *Jatropha curcas* oil. A lower saponification value indicates a lower tendency of soap formation.

Peroxide value is an indicator of the deterioration of oil due to oxidation at double bond of an unsaturated fatty acid; rancidity. The peroxide value for refined *Jatropha curcas* oil was 1.92 mE/kg which is 0.05% lesser than 1.93 mE/kg obtained by [16] for Malaysian *Jatropha curcas* oil. The specific gravity of the refined *Jatropha curcas* oil was found to be 0.882.

3.2. Fuel Properties of *Jatropha curcas* (Ethyl Ester) Biodiesel

The result of measured fuel analyses for biodiesel produced is shown in **Table 2**.

A yield of 57.6% was obtained, and its specific gravity (15°C) was determined to be 0.882.

Flash point is the lowest temperature at which fuel begins to evolve vapour in sufficient quantity to form an explosive or inflammable mixture with air. It measures the response of fuels to heat and assesses their flammability hazard levels and safety conditions. It has been reported that residual alcohol can reduce flash point of biodiesel [19]. This implies that purity of biodiesel can be measured

Table 2. Results of fuel properties of ethyl ester of *Jatropha curcas* seed oil.

PROPERTIES	METHOD	JATROPHA ^d BIODIESEL	EN 14214	ASTM D6751	IBS ^e
Pour point (°C)	ASTM D97	-6	-15 - -16	-	-
Flash point (°C)	ASTM D93	215	101 (min)	130 (min)	120 (min)
Kinematic viscosity @40°C (mm²/sec)	ASTM D445	5.01	3.5 - 5.0	1.9 - 6.0	2.5 - 6.0
Heating value (MJ/kg)	ASTM D240	42.77	-	-	-
Cetane number	ASTM D613	49.77	51 (min)	47 (min)	51 (min)
Iodine value (g I₂/100 g)	AOCS, 1997b	99.20	120 (max)	-	-
Specific gravity @15°C	-	0.86	0.88 - 0.9	-	-

^dResult of this study; ^eIBS: India Biodiesel Standard.

using flash point. High flash point makes biodiesel safer for storage, handling and transportation [20]. The flash point for *Jatropha curcas* (ethyl ester) biodiesel was measured to be at 215°C. This value is within ASTM D6751, EN 14214 and India Biodiesel specifications.

The pour point is the lowest temperature at which fuel start to flow. It is used to measure the cold flow operability of fuel. The cold property is one of the most critical obstacles against the widespread of fuel usage. Pour point is mostly related to saturation of biodiesel [19]. *Jatropha curcas* (ethyl ester) biodiesel has a high amount of unsaturated fatty acids and low amount of saturated fatty acids. Pour point was therefore observed at -6°C which is lower than the temperature attainable in the region of testing. This value met the recommended specification.

Viscosity is a significant internal property of a fluid which indicates the degree of resistance to flow [2]. The result of the analysis reveals kinematic viscosity at 40°C to be 5.01 mm²/s. This falls within the threshold values of ASTM 6751 (1.9 - 6.0 mm²/s), EN 14214 (3.5 - 5.0 mm²/s) and Indian Biodiesel specifications (IBS) (2.5 - 6.0 mm²/s). IBS is used mainly in this discussion because of similarities in the climate and the level of development of Nigeria and India.

Cetane number is an indicator for measuring the ignition performance of fuel in a combustion engine. The cetane number of *Jatropha curcas* (ethyl ester) biodiesel was 49.77. This met ASTM D6751 standard of 47 but lower than the minimum of 51 recommended by EN 14214 and India Biodiesel specification (IBS).

Iodine value is a measure of the unsaturation of oils. High iodine value indicates high unsaturation of oils [21]. The experimental result obtained (99.20 g/100 g) using Wij's reagent showed a value higher than that of conventional di-

esel fuel. High unsaturation oil polymerises on heating. This problem could be worse with an increase in the number of double bonds in the fatty acid chains. However, a report proposed a limit for unsaturated fatty acid particularly oleic and linoleic acids in biodiesel to ensure the quality of biodiesel. The experimental result of iodine value met EN 14214 specification, indicating the suitability of this biodiesel for fuel purpose.

Heating value is an important parameter that measures the capacity of fuel to generate heat. It can be used to adjudge the suitability of biodiesel as an alternative to diesel fuel. The heating values of biodiesel are usually lower than that of diesel fuel—5 MJ/kg [2] [11] and this has been attributed to their higher oxygen content [22] [23]. The result obtained in this work revealed the heating value of *Jatropha curcas* (ethyl ester) biodiesel to be 42.77 MJ/kg. This result met the recommended specifications according to ASTM D6751, EN 14214 and India Biodiesel standards.

3.3. Fatty Acid Compositions

Results of the fatty acid composition of the ethyl ester of *Jatropha curcas* oil is shown in **Table 3**, containing mainly fatty acids with more than 16 carbon

Table 3. Fatty acid composition as ethyl ester of *Jatropha curcas* seed oil.

FATTY ACID	FORMULA	STRUCTURE ^f	SYSTEMATIC NAME	Wt (%)
Myristic	C ₁₄ H ₂₈ O ₂	14:0	Tetradecanoic	0.051
Palmitic	C ₁₆ H ₃₂ O ₂	16:0	Hexadecanoic	13.07
Palmitoleic	C ₁₆ H ₃₀ O ₂	16:1	cis-9-Hexadecenoic	0.003
Margaric	C ₁₇ H ₃₄ O ₂	17:0	Heptadecanoic	0.01
Stearic	C ₁₈ H ₃₆ O ₂	18:0	Octadecanoic	6.30
Oleic	C ₁₈ H ₃₄ O ₂	18:1	cis-9-octadecenoic	44.85
Linoleic	C ₁₈ H ₃₂ O ₂	18:2	cis-9-,cis-12-octadecadienoic	34.87
Linolenic	C ₁₈ H ₃₀ O ₂	18:3	cis-6-,cis-9-,cis-12-octadecatrienoic	0.50
Arachidic	C ₂₀ H ₄₀ O ₂	20:0	Eicosanoic	0.03
Arachidonic	C ₂₀ H ₃₂ O ₂	20:4	Cis-6-,cis-9-,cis-15-,Docosatetraenoic	0.20
Behenic	C ₂₂ H ₄₄ O ₂	22:0	Docosanoic	0.02
Erucic	C ₂₂ H ₄₂ O ₂	22:1	cis-9-Docosenoic	0.028
Lignoceric	C ₂₄ H ₄₈ O ₂	24:0	Tetracosanoic	0.05
Capric	C ₁₀ H ₂₀ O ₂	10:0	Decanoic	-
Lauric	C ₁₂ H ₂₄ O ₂	12:0	Dodecanoic	-
Caprylic	C ₈ H ₁₆ O ₂	8:0	Octanoic	-

^fxx: y indicates xx carbons in the fatty acids with y double bonds. Total saturated fatty acids = 19.53%. Total unsaturated fatty acids = 80.45%.

atoms. The total unsaturated fatty acids consisting of oleic, palmitoleic, linoleic, linolenic, arachidonic and erucic acids made up 80.45%, which is relatively higher than 78.5% obtained by Lau [18]. The fatty acid composition of *Jatropha curcas* (ethyl ester) biodiesel has oleic acid (44.85%) as the dominant fatty acid and this matched with the results reported by Aransiola *et al.* and Ong *et al.* [17] [24]. The presence of a large fraction of these high molecular fatty acids renders biodiesel less volatile. Fatty acid has been reported to influence regulated properties of biodiesel [3] [11] such as viscosity, flash point, cetane number and density.

4. Conclusion

The utilisation of non-edible *Jatropha curcas* seed oil for biodiesel production is sustainable. Biodiesel from refined jatropha oil can serve as a smart alternative to diesel fuel, which is non-renewable. The measured fuel properties of *Jatropha curcas* biodiesel compared favourably with those worked upon by other researchers and met both ASTM D6751 and EN 14214 standards as well as the India biodiesel specification (IBS). The pre-treatment of the extracted oil via alkaline refining process helped to reduce the free fatty acid content, which in turn enhanced the quality of the biodiesel produced thereof. The use of simple transesterification process using potassium hydroxide (base-catalyst) has proven to be an effective conversion route to obtain biodiesel from *Jatropha curcas* oil. The presence of high molecular fatty acids in the biodiesel can significantly affect fuel properties of the fuel. Although the biodiesel has comparatively compatible fuel properties with conventional diesel fuel, it was found to be less volatile than diesel fuel. But better still, the biodiesel produced from refined *Jatropha curcas* seed oil can be used as fuel blend in diesel engine. However, there is a need to evaluate the suitability of biodiesel as a blend in different diesel fuels and the blend proportion with optimum efficiency.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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