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Production of Ethyl Ester Biodiesel from *Jatropha curcas* Seed Oil

***Anaga, E. and Azeez, O.S.**
Department of Chemical Engineering, Federal University of Technology, PMB 65, Minna, Nigeria
*anaga.ekpe@st.futminna.edu.ng

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ABSTRACT

Production of biodiesel from Jatropha curcas oil using ethanol and dry purification with Jatropha husk ashes (JHA) was carried out with the sole objective of developing a farm-implementable processing technology for a developing country such as Nigeria. The Jatropha oil was extracted from Jatropha curcas seed (obtained from Bida town of Niger State) and its conversion to fatty acid ethyl ester (FAEE) was conducted via an acid catalysed ethanolysis at 65℃ leading to 83wt % biodiesel recovery. The physiochemical properties analysis gave an acid value of 5.61 mg KOH/g, iodine value of 7.487g I_2*/100g saponification value of 196.35mg KOH/g, viscosity of 4.82*mm^2/s*, specific gravity of 0.91, high moisture content of 9.81, flash point of 128℃, fire and cloud point of 136℃ and -2℃ respectively and cetane number of 42. Some of these values were within the ranges specified by ASTM values for biodiesel. Dry purification with Jatropha husk ashes (JHA) shows satisfactory result for removal of impurities from the produced biodiesel and can therefore be adopted as an alternative to the frequently used wet purification process. These results suggest the feasibility of biodiesel production by farmers even right there in their farms and this will greatly aid powering of farm machineries.*

1. INTRODUCTION

The use of vegetable oil to run engine was an idea brought up by Rudolf Diesel between the years 1895-1900 when he developed the first diesel engine to run on vegetable oil (peanut oil) (Parawira, 2010). He stated that "the diesel engine can be fed with vegetable oil and would help considerably in the development of agriculture in countries which uses it". Shortly after, it was observed that the use of vegetable oils for engine fuels may be insignificant with time. But such oils may become, in the course of time, as important as the petroleum and coal tar products of the present time (Zaltica, 2008). Unfortunately, Diesel did not live long enough to see his vision being fully realized but today, depleting fossil fuel reserves and environment hazards such as pollution caused by its usage rekindled interest in diversifying fuel resources, and thus biodiesel as fatty esters was developed as an alternative to petroleum diesel.

Biodiesel is an alternative fuel produced from renewable biological sources such as vegetable oils which may be edible or non-edible. It is a monoalkyl ester of fatty acids derived from vegetable oil or animal fat which is usually produced by the transesterification of the oil with methanol or ethanol in the presence of a suitable catalyst (Knothe, 2006). Oil from *Jatropha curcas* (readily available in Nigeria) is a non-edible vegetable oil which can be used as a feedstock for production of biodiesel because of its high oil yield of about 50-60%, drought resistant nature, and ability to grow on marginal soil (Bugaje and Mohammed, 2008).

Nigeria at present is heavily dependent on petroleum fuels for transportation and agricultural machinery. Her gigantic population of over 182 million, (Worldometer, 2017) people is putting a massive strain on her energy, environment, infrastructure, employment and natural resource. Recent discoveries being made has shown that there is a gradual depletion in the nation's oil and gas reserve and that in the near future, there will not be sufficient energy, from petroleum-based fuel to serve the nation. (Olasheu et al., 2015). Therefore, the sustainability of the energy industry will need continuous supply of renewable and sustainable energy. Although many options like fuel cell run by hydrogen, electric vehicles etc. are being explored, however the use of biodiesel as replacement of petro diesel and ethanol in place of petrol are amongst the best alternatives as they can be used without much modification in the existing engines, thus, avoiding significant extra cost (Anaga, 2017).

Amongst the various available vegetable oils which may serve as feedstock for production of biodiesel, *Jatropha curcas* oil seems to be a very good alternative because of its high oil content (30-60%), drought resistant nature thereby making it grow both in tropical and sub-tropical climatic regions of the world. Therefore, it can be successfully grown in all soil types even the problematic soils and arid regions (Olasheu et al., 2015). *Jatropha curcas* seed will serve as a source of revenue generation and provide employment opportunities nation-wide. This is because of its wide range of uses both for humans and industries. Glycerin, a by-product from *biodiesel production*, can be used to produce soap, leaves of *Jatropha curcas* plant can be used to treat malaria and the seed can be used as a cure for constipation and the sap was found to be effective in accelerating wound healing, (Barn and Sharma, 2005). Although a lot of research has been done on methyl ester production of biodiesel from *Jatropha curcas* oil but only a few researchers have actually worked on feedstock having high percentage free fatty acid (FFA) using alternative processes such as bioethanol with low cost homogenous catalyst which is actually an attractive alternative. Thus, the present work is aimed at ethyl ester biodiesel production from non-edible *Jatropha curcas* seed and dry purification using the husk of the *Jatropha* seeds as adsorbent, thus, ensuring optimized use of *Jatropha curcas* which would aid in developing an on-farm biodiesel processing technology which can be practiced even by farmers in rural communities to help in agricultural development and provides a source of energy to power their machineries (Raja et al., 2011).

2. MATERIALS AND METHODS

2.1. Materials

The *Jatropha curcas* oil (JCO) used for this research was extracted from the seed. *Jatropha curcas* seeds (locally known as "kasha") obtained from Bida town of Niger state, Nigeria were hand threshed. The damaged seeds were disposed while seeds in good condition were cleaned and sun-dried for three days to reduce initial moisture content of the seeds and further reduced by oven drying for 2 hours. The seeds were then grounded in the mechanical grinder to reduce its size and increase its surface area. 50g of the grounded seed was then used to extract oil in a soxhlet apparatus using a non-polar solvent, n-hexane (98.5% purity). The extracted oil was then filtered and evaporated to separate the oil from the solvent. Fatty acid composition of JCO is given in Table 1. Isopropyl alcohol of 98% purity, phenolphthalein of 1% and sodium hydroxide in pellets were used to determine the free fatty acids content of JCO. During the transesterification reaction, ethanol of 99.8% purity and sulphuric acid of 98% purity were used with potassium hydroxide (KOH) of 95% purity as catalyst.

Table 1: Chemical formulas and fatty acid composition in *Jatropha curcas* oil (Akbar, 2009)

Fatty acid	Chemical formula	Relative composition (%)
Lauric (12:0)	$CH_3(CH_2)_{10}$ COOH	-
Palmitic (16:0)	CH_3 $(CH_2)_{14}$ COOH	14.2
Stearic (18:0)	CH_3 $(CH_2)_{16}$ COOH	7.0
Oleic (18:1)	CH_3 $(CH_2)_7$ CH = CH $(CH_2)_7$ COOH	44.7
Linoleic (18:2)	$CH_3(CH_2)_4CH{=}CHCH_2CH{=}CH(CH_2)_7COOH$	32.8
Linolenic (18:3)	CH_3 CH_2 (CH = CH $CH_2)_3$ $(CH_2)_6$ COOH	0.2
Erucic (22:1)	CH_3 $(CH_2)_7$ CH = CH $(CH_2)_{11}$ COOH	-
Ricinoleic (18:1)	$CH_3(CH_2)_5CHOH$ $CH_2CH{=}CH(CH_2)_7COOH$	-

2.2. Equipment

The equipment used to conduct this experiment are basically processing equipment which includes oven, furnace, thermostat hotplate mechanical grinder, Tyler sieve, and magnetic stirrer. The analytic equipment included Pensky Martens Flash point tester, cloud and pour point bath, sulphur analyser, viscometer, pH meter). Glass wares included measuring cylinder, separating funnel, round bottom flask, flat bottom flask, beakers, thermometer, pycometer bottle and density bottle.

2.3. Methods

2.3.1. Pre-treatment of *Jatropha curcas* triglyceride

The pre-treatment was done basically to bring-down the FFA content of the *Jatropha curcas* oil (triglycerides) to below 1% (w/w). The processes were; *Jatropha curcas* oil was poured into a round-bottomed flask and was heated to the required temperature by using the electric thermostat hotplate. Sulphuric acid and ethanol (1.0% w/w) mixture was heated at 60 °C, and then decanted into the reactor containing the heated oil. The ratio of ethanol to *Jatropha curcas* oil was 6:1 and a pre-treatment residence time of 180 minutes was used after which the mixture was allowed to settle down for 2 hours and the ethanol-water constituent at the top layer was separated out. The feedstock with the smallest acid value was then used for further transesterification resulting in the production of biodiesel (Azhari et al., 2008).

2.3.2. Conversion of *Jatropha curcas* oil to biodiesel

In this work, the method suggested by Benjamin et al. (2007) was modified and adopted to convert JCO to biodiesel although dry-washing (using *Jatropha* Husk Ashes) was used here instead of wet-washing which he suggested. *Jatropha curcas* oil having the least FFA value was converted to biodiesel via transesterification reaction using KOH catalyst (due to its fast catalytic nature) (Ma et al., 1999; Agarwal, 2007; Marchetti and Errazu, 2008; Ranganathan et al., 2008). Extracted JCO was heated with constant vigorous stirring in a round bottom flask to lower its moisture content. Ethanol of 99.5% purity having density of 0.791 g/cm³ was used. 2.5gram of KOH catalyst was dissolved in ethanol in a mole ratio of 6:1 with continuous stirring. The solution was kept at atmospheric pressure and temperature of 60˚C for 2 hours. At the end of the transesterification process, the mixture was left in a separating funnel for 12 hours to settle out. The bottom layer of the funnel consisted of glycerin, excess ethanol, KOH catalyst, impurities and minute amount of unreacted oil. The top layer consists of biodiesel, alcohol and some soap.

2.3.3. Jatropha husk ashes (JHA) production

Jatropha husk obtained from the *Jatropha curcas* seed used was finely grounded, then collected in crucibles covered with aluminium foil**, a**nd carbonized in a furnace. The furnace operating temperature was 500°C and ashing time of 10h as recommended in literature (Svitlana et al., 2012) to obtain JHA optimal adsorbent properties. After carbonization was allowed to cool and stored at room temperature.

2.3.4. Biodiesel purification

Jatropha curcas ethyl ester was mixed and treated with JHA natural adsorbent to purify the biodiesel. Dry-purification by Manique et al. (2012) in batch mode i.e., one-stage treatment with 4% JHA in unpurified fatty acid ethyl esters (FAEE) sample, stirred for 20 minutes and heated at 65°C was the purification method for this work. Purification was conducted using two different processing temperatures (20°C and 65°C), and time (5min and 20min respectively). At the end of the stipulated duration, the purified ethyl ester product was filtered using Bűchner funnel with filter paper inside and then preserved for in a refrigerator away from sunlight for later analyses.

2.4. Biodiesel Characterization

2.4.1. Determination of viscosity

Biodiesel sample was measured into 200 ml beaker and heated to 40°C. A spindle of a viscometer was adjusted until it submerged into the sample. The viscometer was switched on after spinning and reading was taken from the scale. This analysis was carried with a viscometer at the National Institute of Leather and Science Technology (NILEST), Zaria, Nigeria.

2.4.2. Determination of density

The densities of the biodiesel samples produced were measured with relative density bottle at 15°C by first allowing the samples to cool to this temperature before taking the measurement in accordance with ISO 3675.

2.4.3. Determination of iodine value

The Iodine value analysis was performed in accordance to ASTM D1959-97 as presented by KEM Application Note (KEM, 2017). A mass of 0.3g sample of biodiesel was measured into in a 250ml conical flask with stopper, then 10ml cyclohexane was added, and dissolved then followed by 20ml Wijs (0.1mol/L iodine monochloride acetic acid solution) reagent and stirred. The flask was corked and kept away from sunlight for 30 minutes. Afterwards, 20ml of 100g/l potassium iodide solution was added and 100ml distilled water. The solution was titrated with 0.1mol/l sodium thiosulfate solution, to obtain iodine value. A similar but blank titration was also performed. This was done by titrating as above except without biodiesel sample. Iodine value was calculated using Equation 1.

$$\text{Iodine value} = \frac{(\text{BLI-EPI}) \text{ x TF x CI}}{\text{sample weight}} \tag{1}$$

Where, BLI is blank titration, EPI is the titration volume, TF is factor of titration, CI is concentration conversion coefficient (1.269).

2.4.4. Determination of saponification value

The saponification value analysis was carried out in accordance to ASTM D5558 as presented by KEM Application Note. A 2 g biodiesel sample was delivered into a 250ml conical flask, 25.0ml of 0.5mol/l potassium hydroxide and ethanol were added to the flask. The mixture was heated under reflux for 30 minutes to saponify the biodiesel sample. Two drops of phenolphthalein were added to the saponified sample and then titrated against 0.5N HCl. A blank test was performed by titrating 25ml of 0.5 KOH ethanol solution without biodiesel sample and also not heated under reflux against 0.5N HCl with two drops of phenolphthalein indicator.

$$\text{Saponification value}=\frac{\text{blank titre-sample titre}}{\text{mass of sample}}\times\text{normality}\times 56.1 \tag{2}$$

2.4.5. Determination of cetane number

The cetane numbers of the biodiesel samples were determined from the values of their saponification values and iodine numbers by calculation using Krisnagkura equation (FadhilA et al., 2012). The Krisnagkura equation is expressed in Equation 3.

$$\text{CI}= 46.3 + (5458 / \text{SV}) - (0.225 \times \text{IN}) \tag{3}$$

Where, CI is cetane index, SV is saponification value and IN is iodine number. The cetane number, CN is expressed as in Equation (4).

$$\text{CN} = \text{CI} \times 0.72 + 10 \tag{4}$$

2.4.6. Determination of acid value

The volumetric standard sample, potassium hydroxide, the titration solvent and indicator solution were prepared as outlined in ASTM D974. Using a syringe of 10ml, biodiesel samples were collected from the sample containers, transferred into 250ml conical flasks, and carefully weighed using digital weighing balance prior to analysis. Samples were then dissolved in 10 ml of the titration solvent (500 ml of toluene, 495 ml of isopropyl alcohol and 5 ml of water) (MacFarlane *et al.*, 2008) and then reacted with 0.02-N KOH solution in accordance with ASTM D664. At titration endpoint, a colour change from orange to green that remained so for at least 15s was observed in the titration mixture. The acid number was calculated as from Equation (5).

$$\text{Acid value}=\frac{\text{volume KOH x N KOH x 56.1}}{\text{sample weight}} \tag{5}$$

2.4.7. Determination of flash point

The flash points of the samples were determined in the temperature range 120-215°C by a digital Pensky-Martens closed-cup apparatus. The cup was filled with the sample fuel, inserted into the apparatus and closed in accordance with ASTM standard D93, (Barabas and Todorut, 2011). This analysis was performed on Multiple Flash Point Tester Pensky-Martens STANHOPE-SETA 34100-2 at the quality control laboratory of NILEST, Zaria.

2.4.8. Determination of pour point

The pour point was determined according to the ASTM official method (ASTM D-97). The biodiesel was poured into a test jar to the appropriate level. The test jar was then corked with a thermometer inserted into it. Heating of the oil without stirring at 48°C was carried out and then cooled to 35°C in a water bath. A ring jar was placed around the test jar 25mm from the bottom. After preliminary test, the sample was cooled at specific rate and examined at 3°C interval for flow characteristics. The pour point was the recorded at the least temperature biodiesel movement was noticed.

2.4.9. Determination of sulphur content

The sulphur content analyses of the samples were performed on Horiba (sulphur in oil analyzer SLFA-2800) in accordance with ASTM standard D5453 (Moser, 2009; Barabas and Todorut, 2011) at the quality control laboratory of NIlEST, Zaria.

3. RESULTS AND DISCUSSION

3.1. Properties of Extracted JCO

Extraction of oil from *Jatropha curcas* seed was done by soxhlet extraction method. The extracted *Jatropha curcas* oil (JCO) was then used as the feed stock for biodiesel production. The initial prime focus was obtaining the physiochemical properties of the extracted *Jatropha curcas* oil to see the suitability for biodiesel production. This will then be followed by obtaining the optimal parameters necessary to produce the highest yield and purity of biodiesel. Table 2 is a summary of the physiochemical characterization of JCO obtained in this research.

Table 2: Physiochemical properties of extracted Jatropha curcas oil

Properties	Extracted oil
Colour	Golden yellow
Viscosity at 40˚C	36.92
Viscosity Index	185
Specific Gravity	0.91
Refractive index at 40˚C	1.61
Free fatty acid (%)	2.805
Acid value (mg KOH/g)	5.61
Saponification value (mg KOH/g)	196.35
Iodine value (g I_2/100g)	7.487
Calorific value (MJ/kg)	36.96
Cetane number	40
Sulphur content (%w/w)	0.013
Density (kg/m^3)	0.912

As seen in Table 2, the percentage FFA in exctracted JCO was above the limit of about 1% recommended for oil as stated in the work of Azhari et al. (2008). The high percentage of FFA in JCO is attributed to poor handling and the storage (Azhari, et al.). It is also observed that some of the characteristics of JCO such as the viscosity at 40 ºC, specific gravity and flash point are not within the range specified for biodiesel for which the oil is meant to produce. Therefore, raw JCO needed to be neutralized and transesterification method can be adopted to produce biodiesel using the JCO. The physiochemical properties of raw Jatropha oil obtained from this research work were found to be nearly the same as those described in literature (Azhari et al., 2008).

3.2. Biodiesel Recovery from *Jatropha Curcas* Oil

Table 3 clearly shows that the mean biodiesel recovery was 47ml while that of glycerin was obtained to be 25ml. The biodiesel recovery from this work using the above-mentioned process was approximately 83%. The difference observed in the amount of glycerin and ethyl ester was as a result of the quality of JCO feedstock used (Tint and Mya, 2009).

Table 3: Biodiesel recovery in transesterification reaction

Particulars	Jatropha biodiesel (ml)	Glycerine (ml)
Run 1	40	29
Run 2	53	22
Run 3	48	24
Mean	47	25

3.3. Dry Purification of Recovered Biodiesel

3.3.1. Yield of incineration process

The average yield of *Jatropha* husk from the incineration process was 20wt%. Due to incomplete carbonation of J*atropha* husk, heterogeneous mixture of ashes of different colour (that is, light gray, dark gray and black ashes) with varying mass fractions of 32, 8, and 27wt % respectively were obtained. Furthermore, incomplete carbonation of Jatropha husk obtained clearly shows that the carbonation temperature was not sufficient and thus must be optimized.

3.3.2. Analysis of *Jatropha* husk ashes (JHA)

The JHA contains silicon (Si), potassium (K) and phosphorous (P) as the predominant constituent of the outer epidermis layer. It however contains a higher proportion of Si than other elements.

Table 4: Characterization of the inorganic composition of FAEE of Jatropha Oil before and after purification

Treatment procedure	Constituent elements present (mg/kg)						
	Si	K	S	Ca	Mg	Fe	P
Unpurified JCEE	8.94	2.4	7.7	<0.2	<0.2	<0.2	<0.2
JHA (20°C, 5min)	11.9	8.3	10.1	0.23	<0.2	<0.2	<0.2
JHA (65°C, 20min)	5.76	4.6	4.3	0.77	<0.2	<0.2	<0.2
Specification of EN 14214^{c}	d	5^{e}	10	5^{f}	5^{f}	d	10

3.4. Physiochemical Characterization of Ethyl Ester Biodiesel Produced

The physiochemical properties of the JCO ethyl ester produced was carried out and compared with the diesel fuel standard specifications of ASTM D 6751. The physiochemical properties determined included; viscosity, viscosity index, specific gravity, refractive index, moisture content, acid value, pour point, flash point, cloud point, fire point, and cetane number. The parameters measured were compared with the ASTM standard specifications and the results are shown in Table 5.

Table 5: Physiochemical properties of *Jatropha curcas* ethyl ester (biodiesel) produced after purification

Parameters	Biodiesel from Jatropha oil	ASTM D 6751	ASTM D 975
Viscosity at 40°C	4.82	1.9 - 6.0	1.9 – 4.1
Viscosity index	54	-	-
Specific Gravity	0.87	0.88	0.85
Refractive index (40°C)	1.46	-	-
Free fatty acid (%)	0.91	0.80	-
Moisture content	9.85	-	-
Flash Point (°C)	128	100 - 170	60 – 80
Pour Point (°C)	-2	-15 to 16	(- 35) to -15
Cloud Point (°C)	8	-3 to 12	-15 to 5
Fire Point (°C)	136	-	-
Cetane Number	42	48 - 60	40 – 55
Calorific Value (MJ/kg)	42.80	-	-

ASTM D6751 and ASTM D975 are from Lokman et al. (2014)

3.4.1. Acid value

The literature value for free fatty acid of *J. curcas* oil is approximately 14% or 28mg KOH/g (Mittelbach and Reshmidt, 2004). The fact that the oil has more than 1% FFA resulted in lower conversion to fatty acid ethyl ester (Tiwari et al., 2009). The acid content for this work was obtained as 0.91% which is comparable with the standard as specified by ASTM of FFA below 1%.

3.4.2. Viscosity

The viscosity of the biodiesel produced was 4.82mm^2/s which falls within ASTM acceptable standard for biodiesel. Therefore, it can be concluded that ethyl ester of JCO is a suitable fuel with regards to viscosity specifications.

3.4.3. Specific gravity

Specific gravity of the biodiesel ethyl ester of JCO after characterization at room temperature was 0.87 which is very close to the ASTM specified values for both biodiesel fuel (0.88) and diesel fuel (0.85) though slightly different from the value of 0.91 reported by (Pandey et al., 2012). This may be due to difference in region where the *Jatropha curcas* seed was obtained.

3.4.4 Moisture content

The presence of moisture could pose more negative effect than presence of high free fatty acid thus, the feedstock must be both water and moisture free (Kusdiana and Saka, 2004). The moisture of the produced biodiesel was obtained as 9.85. Although, there is no specified standard for moisture content but the obtained value was quite high thus contributed to the reduction in conversion of fatty acid ethyl ester.

3.4.5 Flash point

The flash point for the produced biodiesel was at 128°C. This value falls within the acceptable range as per ASTM standard. It also corresponds with the value reported by Raja et al. (2011). The obtained value of flash point shows high safety storage and less susceptibility to hazard during transportation

3.4.6 Fire point

Fire point is an important temperature indicating fuel safe handling, storage and transportation. The cloud point of the produced biodiesel was found to be 136°C. Though there is no specified standard for biodiesel but the obtained value is comparable to the literature value of 138°C reported by Raja et al. (2011) which shows that it is quite safe to handle.

3.4.7. Pour point

The value of the pour point for the produced biodiesel was (-2°C) due to the high amount of unsaturated fatty acid in the oil sample. The obtained value is within range as specified by ASTM D 6751 for biodiesel. This value shows that biodiesel from *Jatropha* oil is not only suitable for tropical region but also for moderate temperate region.

3.4.8. Cloud point

No limit is specified for cloud point for biodiesel production according to ASTM D 6751. The cloud point obtained for this research work was 8°C. This is quite different from the fire point reported in literature. This difference could be due to the variation in climatic conditions of the world which considerably affects the specification of biodiesel in different geographic regions of the world.

3.4.9. Cetane number

High cetane number implies that the diesel engine will be easier to start and vice-versa. The calculated cetane number for the produced biodiesel was 42 which of course is within range of standard as specified by ASTM D 975 for diesel fuel. Therefore, only a slight ignition delay is expected when the produced biodiesel is used to run a diesel engine.

4. CONCLUSION

Biodiesel production from JCO using ethanol and dry purification using *Jatropha* husk ashes (JHA) was carried out. The maximum yield of biodiesel was found to be 83% from FFA of 2.802% in unpurified JCO using optimum process parameters of molar ratio of ethanol to oil ratio 8:1, 1% KOH and reaction temperature of 65 °C. After dry purification JHA, FFA of 0.91% was obtained (within acceptable FFA range of below 1%). This successful reduction in FFA is an indication that whole biodiesel production from *Jatropha* seed production of biodiesel using ethanol and dry purification which reduces water need and effluent production is indeed a great alternative that could also be used to produce and purify biodiesel.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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