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RESEARCH ARTICLE

PRODUCTION AND CHARACTERIZATION OF BIODIESEL FROM THE SEED OF *DACRYODES EDULIS* (AFRICAN PEAR)

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ABSTRACT

In this study, Oil from the seed of *Dacryodes edulis* was extracted using cold maceration method. The oil obtained had high acid value of 4.40mgKOH/g. Therefore the production of biodiesel was processed via two step acid – base transesterification process. The first step reduced the acid level to 2.60mgKOH/g while the second step involved direct conversion to fatty acid methyl ester using 1wt% NaOH as catalyst, 1:6 of oil to methanol ratio, 333 Kelvin and 1 hour reaction time. The yields of the oil and its methyl ester were 29% and 67% respectively. The biodiesel produced was analyzed for its fuel properties and yielded the following result; viscosity at 40^oC ($5.17mm^2/s$), density (0.87g/ml), cloud point (8^oC), pour point (7.50^0C), flash point (152^0C), fire point (160^0C) and acid value (1.80mgKOH/g). The results obtained showed that most of the important properties were within the American standard test method (ASTM) of D 6751 of Biodiesel.

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INTRODUCTION

Biodiesel can be referred to as an animal or vegetable oil based diesel fuel that burns without the emission of much soot, carbon (iv) oxide and particulate matter. It is also a biofuel consisting of long chain mono-alkyl esters and is produced by transesterifying renewable lipid feedstock such as vegetable oil or animal fat (Demshmino *et al*., 2013). Biodiesel is like petro-diesel that is made of hydrocarbon chains that do not contain sulphur, or aromatic compounds in its composition. ''Bio''mean it is renewable and has biological source in contrast to petroleum-based diesel fuel; "diesel" refers to its use in diesel engines (Idusuyi *et al*., 2012). Biodiesel is an alternative approach to fossil fuel. It is ecofriendly, biodegradable, non-toxic and essentially carbon dioxide neutral. When used as fuel in diesel engines and heating systems, biodiesel has many advantages such as high energy density, more favourable combustion emission profile, improved lubricating properties, high cetane number, low volatility and low sulphur. The chemical process by which biodiesel is produced is known as the transesterification reaction.

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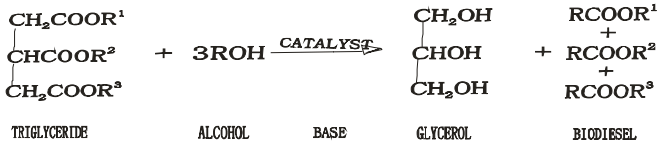
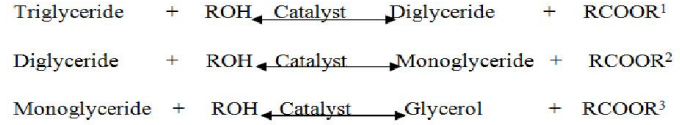
Transesterification means transforming one type of ester into a different type of ester. During transesterification a basic catalyst breaks the fatty acids from the glycerin one by one. If an alcohol typically methanol contacts a fatty acid they will bond and form biodiesel (Vendkata *et al*., 2012). The hydroxyl group from the catalyst stabilizes the glycerine as shown below:



Where R^1, R^2, R^3 are long chain hydrocarbons.

Triglyceride Alcohol Base Glycerol Biodiesel Where R^1, R^2, R^3 are long chain hydrocarbons.



Dacryodes edulis (African pear) which belongs to the family of *Burseraceae* is known as Safou (French), ube (Igbo), elemi

(Yoruba), eben (Efik) and orumu (Benin). They grow in a wide variety of climate, soil type and are widely distributed in Africa. They are found in Cabinda, Cameroon, Congo (Brazzaville and Kinshasa), Gabon, Ghana, Equatorial Guinea, Nigeria and Sao Tome. In south-east Nigeria, the trees are grown around homesteads and flowering takes place from January to April. The major fruiting season is between May and October. In both rural and urban areas of Nigeria, the fruits are boiled or roasted and then eaten with maize (Onuegbu *et al*., 2011). Fruits are ellipsoidal and their size varies approximately from 4 to 9cm long and from 2 to 5cm wide. As a percentage of dry matter, the pulp contains 31.9% oil, 25.9% proteins and 17.9% fiber. They could be an important source of pulp oil, seed oil and even whole fruit oil. The seeds are not economically useful and are often discarded as a waste into the environment. This research is aimed at extracting oil from the seed of *Dacryodes edulis* seed, analyzing the physiocchemical properties of the methyl ester biodiesel produced to ascertain its fuel properties.

MATERIALS AND METHODS

Sample collection and Preparation

The seed of *Dacryodes edulis* was obtained from Igboukwu in Aguata Local Government of Anambra State. 4kg of seed of *Dacryodes edulis* was sundried for 7 days after which they were pulverized to fine texture using an industrial blender.

Extraction of the oil

3kg (3000g) of African pear (Dacryodes edulis) seed was measured using an analytical weighing balance into a container and soaked with 3.5 litres of n-hexane for 2 days. The container were covered and made air tight to avoid evaporation of n-hexane. Decantation was carried out followed by sieving, then filtration. Distillation of the filtrate to recover the n-hexane was done at a temperature of 65^0C (AOAC, 1990). The percentage yield of the oil was calculated as thus:

$$\% \text{ yield} = \frac{weight\ of\ oil\ extracted}{weight\ of\ the\ sample\ used} \times 100 \quad \text{.....(1)}$$

Characterization of the oil obtained from DE (feedstock) and methyl ester biodiesel produced

Free Fatty Acid (FFA)/Acid value

0.5g of the sample was weighed into a dry beaker and 20ml of ethanol added to it. 3 drops of phenolphthalein indicator was added and shook. The solution was titrated with 0.1N sodium hydroxide until a pink colouration was observed. This procedure was equally repeated with biodiesel sample after production.

$$\%FFA = \frac{titre\ value\ \times\ 0.0282\ \times\ 10}{weight\ of\ the\ sample\ used} \quad \text{..... (2)}$$

$$Acid\ value = \frac{Titre\ value \times normality\ of\ the\ base \times 56.1}{wieght\ of\ the\ sample\ used} \quad \text{.....(3)}$$

Refractive Index

Refractometer was used in the determination. Few drops of the sample were transferred into the glass slide of the Refractometer. Water at 30^0C was circulated round the glass slide to keep its temperature uniform. Through the eye piece of the Refractometer the dark portion viewed was adjusted to be in line with the intersection of the cross. At no parallax error, the pointer on the scale pointed to the refractive index. This was repeated and the mean value noted and recorded as the refractive index.

Iodine Value

0.5g of the sample was weighed into a conical flask. 15ml of chloroform was added after which 25ml of Wiji solution (mixture of iodine, acetic acid and chloroform) was added and covered slightly using a foil and masking tape. The resulting solution was placed in the dark for 30 minutes. 20ml of potassium iodide was added followed by 150ml of distilled water. The solution turned red. 5ml of 1% starch indicator was added which turned the solution blue black. The whole solution was titrated with 0.1N sodium thiosulphate till the end point is achieved (V_1). A blank (V_2) was carried out too starting with 15ml of chloroform. Solution turned to blue black precipitate and then to colourless. Iodine value was calculated using equation 4

$$Iodine\ value = \frac{12.69 \times V_2 - V_1 \times Normality\ of\ the\ titrant}{Weight\ of\ sample} \quad \text{.....(4)}$$

Saponification Value

0.5g of the sample was weighed into a conical flask. 50mls of 0.5N ethanolic solution of potassium hydroxide was added and the solution was refluxed to ensure perfect dissolution. The solution was allowed to cool. 3 drops of phenolphthalein was added. The solution was titrated with 0.5N HCl (V_1). A blank (V_2) was carried out as well. Thus saponification value was calculated using this equation 5

$$saponification\ value = \frac{56.1 \times V_2 - V_1 \times 0.5}{Weight\ of\ the\ sample} \quad \text{.....(5)}$$

Peroxide Value

0.5g of the sample was weighed into a conical flask. Using measuring cylinder, 25mls of solvent mixture was added, that is, 2 volume of glacial acetic acid and 1 volume of chloroform. 1ml of 10% potassium iodide was added and shaken vigorously. The solution was covered and kept in the dark for 1 minute. 35mls of starch indicator was added to the solution (V_1) and titrated with 0.02N sodium thiosulphate until end point is attained. A blank was carried out as well. Colour changed from pale yellow to white. Peroxide value was calculated as shown below in Equation 6

$$Peroxide\ value = \frac{1000(V_1 - V_2) \times Normality\ of\ the\ titrant}{Weight\ of\ the\ sample} \quad \text{..... (6)}$$

Two-Step-Acid-Base Catalyzed Transesterification

Acid Pre-Treatment (Acid Catalyzed Esterification)

70ml of oil sample was heated on a heating mantle at 110^0C for 10 minutes for any available moisture to go off. The sample was cooled to 60^0C in a water bath. 46.54ml of methanol (60%

w/w of oil) was mixed with 2.3ml (7% w/w of oil) of concentrated sulphuric acid. The mixture was poured into the oil sample and the resulting mixture was then stirred on a Euro Sonic magnetic hot plate for 1 hour at 50^0C. The mixture was allowed to stand overnight in a separating funnel for thorough separation of the esterified oil from methanol and impurities. The three layers were then separated by tapping them out one after the other. Hot distilled water was poured into the oil, shaken and allowed to stand. This was to wash the esterified oil. After a little time, 2 layers were observed, water (below) and oil (above), the water is tapped out from the separating funnel. Any methanol present leaves from the top of the separating funnel because the water is hot and impurities which are water soluble goes off with the water. This procedure was repeated 3-4 times to get pure esterified oil. The oil sample was then heated at 110^0C to dry it and then cooled in a water bath.

Base Catalyzed Transesterification

50.87g of the pretreated oil was measured into a 250ml conical flask. A solution of 0.51g of sodium hydroxide (1% w/w of sample) in 15ml of methanol was dissolved at room temperature and the resulting mixture (sodium methoxide) was added to the pretreated oil. The mixture was poured into a conical flask, covered with a foil and masked with a tape. A thermometer was inserted with the tip touching the mixture to help in the temperature reading. The transesterification was done on a magnetic stirrer at a temperature of 60^0C for 1 hour keeping the agitation rate constant at 400rpm for oil to alcohol ratio of 1:6. The resulting mixture was poured into a separating funnel and allowed to stand overnight for separation of biodiesel after which two layers was observed. The lower glycerol layer was tapped off and the biodiesel layer washed with hot water 3-4 times. The biodiesel was then heated on a heating mantle at 110^0C to dry it.

Base stripping

0.5N sulphuric acid standard solution (3.5ml) was added to the biodiesel to eliminate the base from it. This was done while washing the biodiesel. The sulphuric acid standard solution was added until the water layer tapped off stopped changing to purple colour when a drop of phenolphthalein was added.

$$\% \text{ Conversion} = \frac{volume\ of\ biodiesel}{volume\ of\ the\ oil\ used} \qquad \text{……...(7)}$$

The biodiesel produced was subjected to characterization to determine its fuel properties.

Characterization of Biodiesel from DE

Flash Point

A quantity of the biodiesel sample was poured into a Petri dish on a 78 HW heating mantle. A thermometer was inserted into it by clamping it to a retort stand. A test flame applicator was used to pass flame across it with continuous smooth motion and the temperature at which the sample ignited was recorded as the flash point with the help of the thermometer.

Fire point

The test flame applicator was continuously moved across the centre of the biodiesel sample on the heating mantle. The oil ignited and continued to burn. After 5 seconds of burning, the thermometer reading was noted as the fire point.

Cloud point

The biodiesel in a test jar with a thermometer clamped to it was cooled inside a constant temperature cooling bath. It was monitored to know the temperature of first appearance of wax crystal. As the biodiesel in the cooling bath started forming wax crystals, the temperature was recorded as the cloud point.

Pour Point

The biodiesel in a test jar with a thermometer clamped to it was cooled inside a constant temperature cooling bath as its cools it forms a wax crystal. The test jar was removed at every 30^oC and tilted to check the surface movement. When the surface did not flow for 5 seconds, the temperature was recorded.

Moisture Content

The test jar was washed and dried on a heating mantle and then left to cool. This is to ensure that there is no water in the test jar. The test jar was weighed using SETRA BL-4105 electrical weighing balance. The weight was recorded as W_1. 1g of the sample was weighed into the test jar and the weight recorded as W_2. The test jar containing the sample was placed in a Universal hot air oven (NESCO) at a temperature of 105^0C for 1 hour after which it was cooled and weighed. The test jar was then placed back into the oven for another 1 hour, cooled again and reweighed. The drying, cooling and weighing process was continued until a constant weight was obtained showing that the moisture loss was complete. This constant weight was recorded as W_3. Moisture content was calculated using equation 8

$$\% moisture = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \qquad \text{…… (8)}$$

W_1 = Weight of empty test jar.
W_2 = Weight of empty test jar and sample before drying.
W_3 = Weight of empty test jar and sample after drying.

Specific Gravity

Empty specific gravity bottle was weighed and the weight was noted as W_1. The bottle was filled with distilled water and reweighed and the weight noted as W_2. The bottle was rinsed with acetone and then filled with the biodiesel sample and reweighed and the weight noted as W_3.

$$Specific\ gravity = \frac{W_3 - W_1}{W_2 - W_1} = \frac{Weight\ of\ biodiesel}{Weight\ of\ distilled\ water} \qquad \text{…….(9)}$$

W_1 = Weight of empty specific gravity bottle
W_2 = Weight of empty specific gravity bottle + distilled water
W_3 = Weight of empty specific gravity bottle + biodiesel

Kinematic Viscosity

A quantity of biodiesel sample was poured into a Townson + Mercer viscometer up to its upper timing mark. It was placed

into a holder and inserted into the viscometer water bath. A micro pipette was used to apply a suction force to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time by timing the flow of the sample as it flows freely from the upper to the lower timing mark was recorded. The kinematic viscosity was calculated using equation 10

$$Kinematic\ viscosity = \frac{Dynamics}{Density} \quad \text{.....(10)}$$

RESULTS AND DISCUSSION

The percentage of oil extracted from the seed of the DE using equation 1 is 29%, this oil was found to contained high acid value of 4.40mgKOH/g and free fatty acid, FFA 2%, which is above the permissible limit of 3.0 mgKOH/g. Therefore, it cannot be used directly for reaction with the NaOH and hence has to be esterified.

This is because higher amount of FFA can lead to emulsification and presents a great difficulty during separation of the biodiesel from glycerol and its washing. After acid catalysed esterification the acid value obtained was 2.60mgKOH/g which is now within the acceptable range and transesterification can take place. The % yield of biodiesel produced from DE oil was calculated as 67%. The transesterification reaction was carried out under the reaction conditions of 1:6 methanol/oil ratio, 60^{o}C reaction temperature, 1hour reaction time, stirring speed of 400rpm and 0.51g of NaOH (1% w/w of sample). The product of the reaction was a light yellow liquid. The reaction conditions above were used to produce biodiesel from Jatropha curcas seed, the yield was 87%, also with refined soya oil and the yield was 97.89% with the above reaction temperature and 1% w/w NaOH but in 3 hours (Elizabeth *et al*., 2012; Elizabeth *et al*., 2010). Another researcher used the above conditions except on catalyst concentration of 0.3% to produce biodiesel from waste groundnut oil using supported heteropolyacid and they achieved 90.75% yield (Anitha & Dawn, 2010).

Table 1 showed the result of the physiochemical parameters analysed. It is important to remove water prior to biodiesel production process because even a low level of water (1%) could increase soap production and measurably affect the completeness of the transesterification reaction (Deepak *et al*., 2013). From the Table 1 it could be infer that the moisture content of the DE oil is 0.08% while that of the biodiesel is 0.02% and this result conforms to the ASTM D6751 standard of 0.05% of moisture content value of Biodiesel. The FFA of the biosiedel from DE oil is slightly above the permissible limit of ASTM D6751 standard as shown in the same Table. The Presence of high free fatty acids can also lead to corrosion during storage or transportation and may be a sign of water in the fuel (Yunus *et al*., 2013). The acid value is a direct measure of the content of free fatty acids, thus the corrosiveness of the fuel of filter clogging and the presence of water in the biodiesel (Ezekwe and Ajiwe, 2014). In the same Table it was observed that the acid value specification limit of 0.8max mgKOH/g was exceeded by the biodiesel produced from DE oil and the seed oil of DE.

Table 1. Result of Characterization of *Dacryodes edulis* Seed Oil and Biodiesel from DE

S.No.	Parameters	Unit	ASTM D6751 Standard	Seed Oil of DE	Methyl ester Biodiesel from DE
1	% FFA	%	0.5 max	2	0.9
2	Acid value	mgKOH/g	0.8max	4.40	1.80
3	% moisture	%	0.05 max	0.80	0.02
4	Saponification value	mgKOH/g	<500	84.15	84.15
5	Iodine value	g/iodine/100g	<115	24.65	39.85
6	Peroxide value	meq/kg	-	0.4	2.0
7	Refractive index	-	-	1.37	1.39

Table 2. Result of the characterization of the methyl ester biodiesel from DE oil and seed oil of DE

S.No.	Parameters	Unit	ASTM D6751 Standard	Seed Oil of DE	Biodiesel From DE Oil
1	Flash point	^{o}C	130mins	-	152
2	Fire point	^{o}C	150min	-	160
3	Kinematic viscosity	mm^2/s	1.9-6.0	19.67	5.17
4	Cloud point	^{o}C	-3-12	17.00	8.00
5	Pour point	^{o}C	-5-10	14.0	7.50

Specific gravity has been described as one of the most important parameters of fuel since certain performance indicator like heating value, cetane number, storage time are correlated with it (Ejikeme *et al*., 2011). The specific gravity/relative density of biodiesel produced fell within the standard limit of ASTM D6751.This shows that it possesses a long storage time, high cetane number and excellent heating value. The lower saponification value of 84.15 mgKOH/g of the produced biodiesel & oil as shown in Table 1 above, suggests that the mean molecular weight of fatty acids is lower than that of other vegetable oil & the number of ester bonds is less when compared to that of other vegetable oil. The lower value indicates that there will be less soluble soap produced from the oil. The iodine value of the biodiesel produced as shown in Table 1 above is within the biodiesel standards and this suggests that the oil produced is saturated (Pandurangan *et al*., 2014). Biodiesel has high degree of saturation and therefore will not be converted into peroxide cross linking. Saturated fats produce a biodiesel fuel with superior oxidative stability, a higher cetane number, but poor low temperature properties. Freshly refined oil should have nil peroxide value (Pandurangan *et al*., 2014). But DE seed oil and its biodiesel have 0.40meq/kg and 2.00meq/kg value as shown in Table 1

above. Nevertheless, this low value indicates that there is low level of oxidative rancidity and presence of antioxidant in the oil and biodiesel.

The flash point & fire point of biodiesel produced were 152^{o}C and 160^{o}C respectively as shown in Table 2 above which are within the standard range for biodiesel (130min). The values were higher than that of petro diesel of value 52-96 and this makes them more stable to fire. Flash point has nothing to do with engine performance but rather fuel handling and storage, the result obtained allowed the produced biodiesel to fall under the non-hazardous category under National Fire Protection Association Codes (Ibeto *et al*., 2011). This flash point suggests that the biodiesel produced is not highly flammable, but would require safety precautions like any fuel during usage, storage and transportation (Yunus *et al*., 2013). The value of Kinematic viscosity obtained for the biodiesel was 5.17mm^2/s as shown in Table 2, It can be observed that it is within the standard range of the biodiesel but not petrodiesel of 1.9-4.1mm^2/s. Higher viscosity turns atomized fuel into larger droplets with high momentum and has a tendency to collide with the cylinder wall relatively. This leads to an increase in deposits and fuel emissions. Low-viscosity fuel produces a very subtle spray and cannot get into the combustion cylinder thus forming the fuel rich zone which led to the formation of soot (Endah *et al*., 2012; Ezekwe and Ajiwe, 2014). From the result it can be inferred that DE seed biodiesel will have inferior injection and atomization performance, but offer lubrication and protection for the moving parts of an engine superior to those of the diesel (Deepak *et al*., 2013). The Cloud and Pour point of biodiesel produced are within the standard limit of biodiesel according to ASTM D6751 standard as shown in Table 2. The Cloud and Pour points of 8^{o}C and 7.5^{o}C respectively might give rise to low running problems in cold season. This problem could be overcome by the addition of suitable cloud and pour point depressants or by blending with diesel oil (Prafulia *et al*., 2012).

Conclusion

This study has shown that oil from *Dacryodes edulis* (African pear) seed can be extracted and used to produce biodiesel by acid – base transesterification method. All the fuel properties tested for the transesterified product (biodiesel) compared well with biodiesel standard, that is, American Standard of Testing and Material (ASTM) except FFA, and acid value. It therefore calls for more methods which will remove this high acid value from the oil so that damage will not be done to the engine while using this biodiesel produced from DE seed.

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