The Effect of Process Variables on the Transesterification of Refined Cottonseed Oil

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***Abstract*— This research work focuses on the production of biodiesel from refined cottonseed oil via alkaline-transesterification process using sodium hydroxide catalyst and methanol. Optimization was carried out using a 2^3 factorial design experiment, three variables namely mole ratio of methanol to oil, catalyst concentration and reaction temperature were studied at both high and low levels. The results obtained during the characterization of refined cottonseed oil include; Saponification Value (189.0 mgKOH/g), Acid Value (1.02 mgKOH/g), Free Fatty Acid (0.51%), Iodine Value (111.2 g/100g), Specific Gravity (0.920), Refractive Index (1.468). These results show the suitability of the oil for the production of biodiesel. Production yield and purity of biodiesel were used to verify the optimization. Results obtained showed that mole ratio of methanol to oil and catalyst concentrations were the most important variables affecting the yield of biodiesel. Optimum methyl ester yield of 96.23 % and a concentration of 99.54 % were gotten at a temperature of 40 °C, mole ratio of methanol to oil of (6:1) and catalyst concentration of 0.5 wt% at a constant reaction time of 90 minutes.**

***Index Terms*—Transesterification, Process variables, Energy source, Cottonseed oil**

I. INTRODUCTION

ENVIRONMENTAL concerns and depletion in petroleum resources have forced researchers to concentrate on finding renewable alternatives to conventional fossil fuels in general and in particular, diesel.

In today's world, petroleum is clearly the most important energy source, providing more than half the world's power, as well as being the basic raw material used in the manufacture of fertilizers, synthetic fibers, plastics, and synthetic rubber. As such, increasing environmental concerns and depletion in petroleum resources have forced researchers to concentrate finding renewable alternatives to conventional diesel fuels [1]. In Nigeria, concerns about dwindling supplies, its unstable and rising cost, and environmental problems have motivated researchers to extensively seek for an alternative, renewable energy sources. Among these alternative sources, vegetable oils have gained considerable attention since they are derived from renewable resources, can be domestically produced, and are not as harmful as petroleum to the environment [1]. However, the use of these oils is restricted to diesel engines, since they contain free fatty acids (FFAs), phospholipids, sterols, water, odorants, and other impurities. In addition, vegetable oil's high viscosity, about 11-17 times higher than diesel fuel, affects the flow properties of the fuel, such as spray atomization, consequent vaporization, and air-fuel mixing in the combustion chamber, leading to an adverse effect on the combustion process [2].

In Nigeria, it was reported by the Energy Commission of Nigeria in 2005 that Nigeria's fossil led economy is under severe pressure to diversify as the flaring of enormous amounts of natural gas has increased the level of CO_2 in the atmosphere, contributing to global warming [3].

Biodiesel is a variety of ester-based oxygenated fuels derived from natural, renewable biological sources such as vegetable oils. Its name indicates, use of this fuel in diesel engine alternate to diesel fuel. Biodiesel operates in compression ignition engines like petroleum diesel thereby requiring no essential engine modifications. Moreover it can maintain the payload capacity and range of conventional diesel [3-6]. It can be produced from renewable vegetable oils/animal fat and hence improves energy security and economic independence [6]. Various vegetable oil such as palm oil, soybean oil, sunflower oil, rape seed oil and canola oil have been used to produce biodiesel [7]. This study is concerned with the optimization and characterization of biodiesel from cotton seed.

II. MATERIALS AND METHOD

A. Materials

Refined cottonseed oil used in this experiment was purchased from Biodiesel Company and its physico-chemical properties are presented in Table 1. Chemicals such as methanol and sodium hydroxide (catalyst) used was manufactured by Aldrich Co. Ltd England with the methanol having a purity of 99.5 %.

B. Experimental Design

A 2^3 factorial experimental design was used to determine the optimum conditions, three variables were studied at both high and low levels with the response been methyl ester

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yield. The high level of alcohol/oil mole ratio was 1:8 and the low level was 1:6. The high level of temperature was chosen at 60 °C and the low level at 40 °C. The high level of catalyst concentration was chosen at 1.0 % and low level was 0.5 % sodium hydroxide catalyst by weight of cottonseed oil. A constant reaction time of 90 minutes and constant agitation were maintained throughout the experiment.

C. Method

Transesterification reactions were carried out in 500 ml flask. The reactor was filled with 100 g of refined cottonseed oil. Sodium hydroxide catalyst was dissolved in methanol and then added to the reactor. The mixture was heated to selected temperature. After the end of the reaction, the mixture was cooled to room temperature and transferred to a separating funnel. The two layers (biodiesel and glycerol) were separated by sedimentation. The methyl ester phase (biodiesel) was washed with hot distilled water and drying was done by heating the biodiesel to a temperature above 100 °C to remove water molecules.

TABLE I
SOME PROPERTIES OF REFINED COTTONSEED OIL

Properties	Refined cottonseed oil
Specific Gravity at 25 °C	0.921
Refractive Index at 40 °C	1.468
Iodine Value, g iodine/g oil	111.2
Saponification Value mg KOH/g oil	189
Acid Value, mg KOH/g oil	1.02
Free fatty acid value	0.51

Determined according to AOCS standards

TABLE II
REACTION CONDITIONS, PRODUCTION YIELD, METHYL ESTER CONCENTRATION AND METHYL ESTER YIELD.

Run	A	B	C	D	E	F
1	1:6	40	0.5	96.67	99.54	96.23
2	1:8	40	0.5	81.70	98.73	80.66
3	1:6	60	0.5	92.83	99.21	92.10
4	1:8	60	0.5	82.97	99.01	82.15
5	1:6	40	1.0	92.74	99.53	92.30
6	1:8	40	1.0	88.98	99.01	88.10
7	1:6	60	1.0	83.06	98.75	82.02
8	1:8	60	1.0	86.05	99.84	85.91

A= Mole ratio of oil to methanol, B= Reaction temperature (°C), C= Concentration of sodium hydroxide (wt %), D= Production yield (wt %), E= Methyl ester concentration (wt %), and F= Methyl ester yield (wt %)

III. RESULT AND DISCUSSION

The experimental results which were obtained from 8 runs according to the experimental design are shown in Table II. From the results obtained, catalyst concentration and molar ratio of oil to methanol were the most important factors affecting the methyl ester yield while temperature had little significant effect on methyl ester yield.

A. Effect of Oil to Methanol Molar Ratio

The methyl ester yield decreased with increasing molar ratio from 6:1 to 8:1. This can be explained on the basis of the reactant (oil) concentration in the reaction mixture. By increasing alcohol to oil molar ratio, the amount of alcohol was increased, therefore the cottonseed oil and catalyst concentrations were diminished, which reduced the rate of reaction. Boocock *et al.* [8] reported a similar trend. At higher reaction temperature, a similar trend of ester concentration (ester concentration decreased with an increase in alcohol to oil molar ratio) was observed. As alcohol to oil molar ratio was increased, the polarity of the system was also increased. As a result, the rate of the reaction as well as the % conversion was increased. Leung and Guo, [9], observed that further increase of alcohol content does not increase the yield of biodiesel but instead increases the cost of alcohol recovery.

B. Effect of Catalyst Concentration

Sodium hydroxide catalyst was used at 0.5 wt% and 1.0 wt% in this work. It can be observed that the methyl ester yield decreases with increase in catalyst concentration. This can be due to the formation of soap by the oil and excess sodium hydroxide used. Freedman *et al* [10] found that the sodium methoxide would be more effective because mixing of sodium hydroxide with methanol produce little amount of water which inhibit the formation of end product (Biodiesel) due to the hydrolysis reaction.

C. Effect of Reaction Temperature

In this work, the effects of reaction temperature on production and methyl ester yield decreases with increase in temperature. This can be as a result of higher solubility of reactants because higher temperature reduces the separation of methyl ester and glycerol phase. Also, a higher reaction temperature of 60 °C that was chosen is close to the boiling point of methanol which can lead to evaporation of some of the methanol during transesterification process. As a result, lower methyl ester yield was observed at 60 °C as compared to that obtained at 30 °C leaving all other variables constant under the same reaction conditions. Based on the result obtained, a temperature slightly above room temperature (40 °C) was considered to be the optimum temperature. Eevera *et al.*, [11], observed that longer reaction time leads to the reduction of end product (biodiesel) due to the reversible reaction of transesterification resulting in loss of esters as well as soap formation.

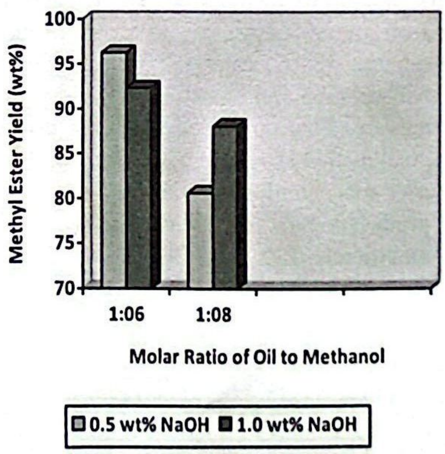

Fig 1. Effect of molar ratio of oil to methanol on the methyl ester yield at 90 minutes

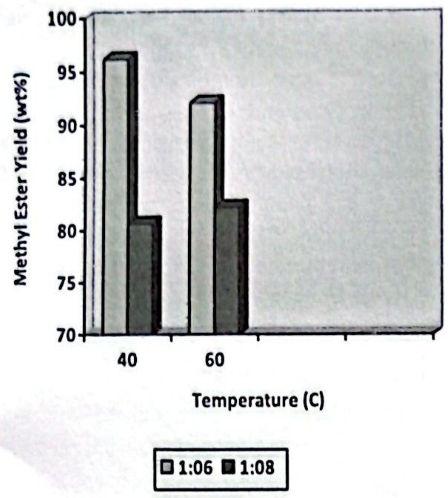

Fig 2. Effect of reaction temperature on methyl ester yield at 90 minute

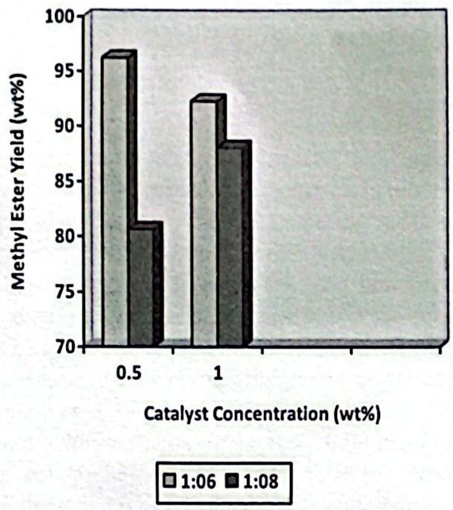

Fig 3. Effect of catalyst concentration on methyl ester yield at 90 minute

IV. Conclusion

Based on the experimental results, it was concluded that the optimal conditions for the transesterification of refined cottonseed oil are as follows: oil to methanol molar ratio, 1:6; temperature, 40 °C; catalyst concentration, 0.5 % at constant reaction time and agitation of 90 minutes and 300 rpm respectively. This optimized condition was validated with the actual methyl ester (biodiesel) yield of 96.23 % and a methyl ester concentration of 99.54 %. Characterization of biodiesel produced show that it can serve as a replacement for petroleum diesel.

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Production and Characterization of Biodiesel from Nigerian Mango Seed Oil

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***Abstract*—Feedstock costs accounts for the larger percentage of biodiesel production cost. The use of less expensive feedstock and optimization of the process variables that affect the yield presents the opportunity of significantly reduction of this cost. This paper investigates the effect of temperature and catalyst concentration on the transesterification of Mango (*Magnifera indica*) seed oil with methanol using potassium hydroxide as catalyst. The biodiesel produced was characterized to ascertain its suitability for use as fuel. Results obtained showed that increase in temperature result in corresponding increase in the biodiesel yield. A yield of 83% wt was obtained at an optimum temperature of 60 °C. A similar trend was observed on the effect of catalyst concentration, with the optimum being 80 %wt at 1% w/v. Analysis of the biodiesel produced showed consistency with the threshold standard values quoted by ASTM and EN for biodiesel and fossil diesel. This signifies that the biodiesel produced from mango seed is of good quality and can be used to consolidate the fossil based diesel.**

***Index Terms*—Biodiesel, transesterification, Mango seed oil, talyst concentration, Temperature**

I. Introduction

THE dependence on petroleum as the energy source comes at a high cost and the world petroleum production has perhaps reached its peak [1]. Experts suggested that existing oil and gas reserves would only last for a few additional years [2]. Besides emissions from the burning of these fuels such as CO_2 CO, NOx and sulfur containing residues are the principal causes of global warming [3]. This has stimulated the widespread search for a cheap and an eco- friendly alternative source [4].

Biofuels offer a partial solution to many of these problems [5]. The feed stocks for biofuel production are produced by domestic agriculture, which means that Biofuel production occurs domestically [5]. In order to meet the rising energy demand and diminishing petroleum reserves; fuels such as biodiesel and bioethanol, are in the forefront of alternative technologies under development. Accordingly, the viable alternative for the compression-ignition engines is biodiesel [2].

Biodiesel is a mono alkyl ester of fatty acids derived from vegetable oils and animal fats; it is a clean and renewable fuel [6-7]. It is a liquid which varies in colour between golden and dark brown depending on the production feedstock [8]. Biodiesel is one of the most attractive biofuel because of its biodegradability, higher flash point (150^{o}C), reduced exhaust emissions, miscibility in all ratios with petroleum diesel, compatibility with the existing fuel distribution infrastructure and inherent lubricity [9]. Other attractive features of biodiesel includes; non flammability, non toxic, reduced petroleum imports, low sulphur content, domestic production and oxygenating potentials [10-12]. Biodiesel production enjoys a positive social impact by enhancing rural revitalization [11]. Various vegetable oils (edible or non edible) and animal fats used for biodiesel production are rice bran oil, coconut oil, Jatropha curcas oil, castor oil, cotton seed oil, palm oil, olive oil, palm kernel oil, soybeans oil, sunflower oil, canola oil, tallow, waste grease, peanut oil, corn oil, madhuca indica, pongamia pinnala [6, 13-15] fish oil, safflower oil, and linseed oil [16].

This oil crops put together are not capable of providing enough vegetable oil for the commercial production of biodiesel that is required to put out of place all petroleum diesel as an alternative transport fuels. At present only small quantities of biodiesel are produced for blending with petroleum diesel at a level of a few percentages. There is therefore the need to search for other oil bearing seeds that has been under utilized for commercial production of biodiesel.

More so process design and operation parameters differ significantly depending on the properties of the raw materials used as feedstock oils and the quality of biodiesel desired.

Mango seed contain 9-13 % oil depending upon variety. The oil is high in unsaturated fatty acid such as oleic acid with 46.22 % [17], which compared favorably with 44.7 % oleic acid of Jatropha Curcas oil and higher than 15.4 % of Palm oil, 21.1 % for Sunflower oil and 23.4 % for Soybean

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oil [18]. Mango (*Magnifera indica*) belongs to the genus *Magnifera* of the family *Anacardiaceae*. The genus contains several species that bear edible fruits. They are tropical fruit bearing trees which thrive well in Asia and Africa [19-20]. Major producers of mango in Africa are Nigeria and Guinea [21]. The fruit is one of most economically important fruit which has been used as raw material for many canned fruit product, where the flesh is utilized resulting in a vast amount of mango seeds and peels being discarded as waste [22]. Aside from been used in canned fruits factories, there is need for further processing of the mango kernel (seed), following the facts that it seed contains a type of fat called mango seed Almond fat (MAF) that is high in stearic acid content [22]. To the best of the author knowledge there is no documented research on the production of methyl ester from Nigerian mango seed oil.

This paper provides insight into the extraction of oil from mango seed oil and its subsequent transesterification to biodiesel using potassium hydroxide. It also explores the relationships between the relevant reaction variables (i.e. catalyst amount and reaction temperature) and the product characteristics (i.e. ester content and yield). This is aimed at providing an alternative raw material for biodiesel production in Nigeria.

II. Materials and Methods

A. Oil Extraction

The extraction procedure described by Nzikou *et al.*, (2009) using petroleum ether at 60 ^{0}C in 5-1 soxhlet extractor for 8 hours was employed. The oil was then recovered by evaporating off the solvent using rotary evaporator model no. TT107R (Techmel and Techmel, USA) and residual solvent was removed by drying in an oven at 60 ^{0}C for 1 hour and flushed with 99.9 % Nitrogen. The extracted oil was then characterized and the result presented in Table I.

B. Transesterification Procedure

The oil extracted was dehydrated by heating the above 100 °C on a hot plate to dry away it water content. The dehydrated oil was esterified to reduce the percentage free fatty acid by agitating with a mixture of 300 ml methanol and 1 %w/v of H_2SO_4 acid, heated to 60 ° C for one hour. The mixture was allowed to settle in a separating funnel, the upper layer containing methanol and water while the lower layer contained the vegetable oil. The oil was separated and methanol was recovered from methanol – water mixture via distillation.

The biodiesel was synthesized in a 450 ml conical flask equipped with a thermometer and mounted on a magnetic stirrer hotplate. The agitation was kept constant. 240 ml of the oil were introduced in to the reactor and measured amount of methanol/KOH stock solution (60ml) was heated separately to the reaction temperature. It was added to the reactor and stirrer was started.

The temperature for the process was varied from 45 to 65 °C at a molar ratio of oil/alcohol of 6:1, catalyst concentration of 1% w/v KOH for one hour each. The reaction product mixture was allowed to stand for 16 hours to separate into the biodiesel phase (upper phase) and glycerol (lower phase). Acetic acid was added to the collected biodiesel, followed by warm water; washing to remove impurities. The washing was repeated until no trace of soap found and final product dried in an open beaker on a hot plate. The yield of each ester was measured.

The transesterification procedure was repeated for various catalyst concentrations. The catalyst was prepared in concentration range of 0.5 to 1.2 % w/v. To achieve the first concentration level 0.25 g of catalyst was dissolved in 40ml of methanol (BDH England) and the mixture stirred for 20 minutes, to form potassium methoxide. The methoxide was introduced gently into the heated oil and entire content was brought to a temperature of 60 ^{0}C and maintained at this temperature for 1 hr reaction time. The procedure described above was repeated for others catalyst concentration level. The yield of biodiesel produced, washed and dried were measured and recorded.

III. Result and Discussion

The oil extracted was characterized, and the result obtained are shown in Table I

TABLE I
PHYSIOCHEMICAL PROPERTIES OF MANGO SEED OIL

Characterization	Result
Oil Yield (%)	14
Saponification Value (mgKOH/g)	194.72
Peroxide Value (meq/kg)	0.60
Free Fatty Acid (mgKOH/g)	3.92
Specific Gravity	0.94
Iodine Value (g I/100g oil)	39.9
Acid value	7.84

A. Effect of Temperature

Temperature plays an important role during biodiesel production; this is because the rate of reaction is strongly influenced by the reaction temperature [15, 23]. Figure 1 shows the result of temperature variation from 45 ^{0}C to 65 ^{0}C at a catalyst concentration of 1 % w/v. As the temperature increase from 45 - 60 ^{0}C the conversion yields of biodiesel also increases considerably. Further increase in temperature results in decrease in the yield of biodiesel. Literatures have reported that alkaline transesterification are conducted close to the boiling point of the alcohol used and that temperature higher than this burns the alcohol resulting into lower yield. Patil and Deng [24], reported that alkaline transesterification at temperature above 60 ^{0}C cause excessive methanol loss due to evaporation and significantly reduce overall biodiesel yield. And that saponification of glycerides by alkali catalyst is much faster than the transesterification reaction above 60 ^{0}C. This may be another plausible reason for the observed low conversion at 65 °C. Results obtained in this study are also in agreement with the work by [13, 16, 25-26].

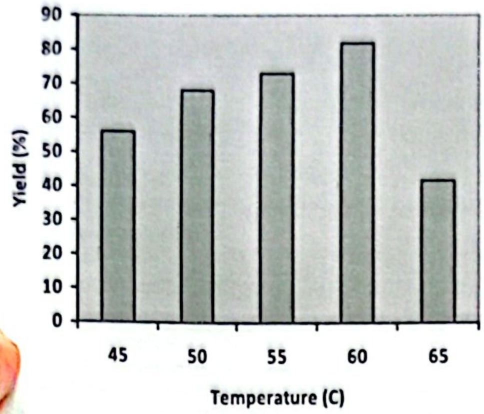

Fig 1. Yield of Biodiesel against Temperature

B. *Effect of Catalyst Concentration*

From the result obtained as shown in figure 2, the catalyst concentration increase from 0.33 to 1 %w/v, a progressive increase in percentage conversion in the reaction was achieved and thereafter experienced a decrease I yield above this concentration (1 % w/v of KOH). It was obvious that increase in catalyst concentration beyond 1 % w/v of KOH results in a decrease in biodiesel yield. Hence the yield at 1.17 % w/v was lower than the yield obtained at 0.33 % w/v. This can be clearly explained by the reversible nature of transesterification reaction. The findings from this studies is very consistent with opinion of [13], who reported that catalyst concentration greater than 1 % w/v may have favored the backward reaction, thereby shifting the equilibrium from the right to the left, hence the formation of glycerol. In comparing results of this findings with that of previous researchers such as [27] for Jatropha Curcas oil and [8] for Tiger nut oil as biodiesel resource, the result is in ccordance with their findings.

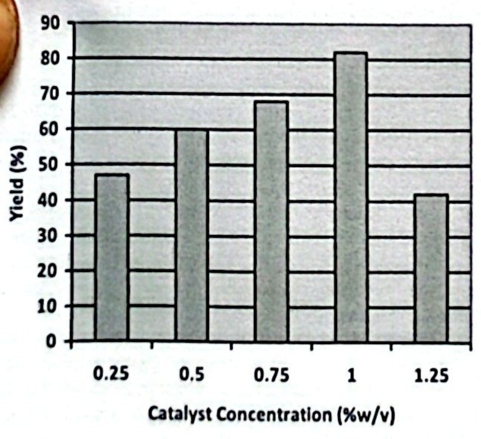

Fig 2. Yield of Biodiesel against Catalyst Concentration

C. *Characteristics of Transesterified Mango Seed Oil*

The biodiesel characterization shows similarities to that of fossil diesel. The physical and chemical properties of the biodiesel were determined. Other tests such as specific gravity, specific gravity, flash point, water content, viscosity, acid number and distillation profile were determined and the result are in accordance with the ASTM and EN standard. The result is as shown in Table II.

TABLE II
COMPARISON OF THE BIODIESEL PRODUCED AND CONVENTIONAL DIESEL

Fuel Property	Standards			This Work	[28]	[29]
	Diesel ASTM	Biodiesel ASTM	Biodiesel EN			
Kinematic Viscosity	1.3-4.1	4.0 – 6.0	3.5 – 5.0	5.82	4.42	4.82
Specific Gravity (kg/l)	0.85	0.88		0.881		0.84
Density (lb/gal)	7.1	7.3	860–900	7.02	877	
Carbon Residue	0.15 Max	0.05		0.30		
Flash Point (°C)	60 – 80	100 – 170	>101	127	171	128
Cloud Point (°C)	-35 – 5	-3 – 15		12	3	8
Pour Point (°C)	-35 – 15	-5 – 10		- 4	6	-2
Cetane Number	40 – 55	48 – 65	51 min	56	58	
Acid Number	0.50 Max		0.5 max	0.80	0.28	
Water and Sediment	0.05 Max	0.05		0.15	Trac e	

Source: [30]

Kinematic viscosity is a measure of resistance of fluid flow under the influence of gravity [30]. The result from this work shows a kinematic viscosity of 5.82 which is quite in agreement with the ASTM norms and the reported work. The result is however slightly higher than the European Norms (EN Standard) and ASTM standard for the kinematic viscosity of the fossil diesel. Viscosity of a fuel is related to the fuel lubricity. Low viscosity fuels are unlikely to provide satisfactory lubrication in fuel injection pumps; these often lead to seepage and increase in wear [29]. High viscosity in fuel are responsible for atomization of fuel, incomplete combustion and increased exhaust emissions, choking of the injections thereby forming larger droplets on injector, ring carbonization and accumulation of the fuel in the engine [31]. The result from this study shows that biodiesel from mango seed oil can be used for biodiesel production.

Specific gravity of the fuel is very important in diesel engine because fuel injection system operates on a volume metering basis. The values of specific gravity obtained for mango seed oil methyl ester was 0.881 with a corresponding density value of 881kg/m^3. This value lies within the notable standards and also in close proximity to the findings of other studies.

The carbon residue is an indicator of residual carbon after combustion. The carbon residue for this study is measured to be 0.030 % against the ASTM standard of 0.050 wt %.

Gerpen *et al.*, [30], reported that the major cause of surplus carbon residue in biodiesel is excessive total glycerin. The low value obtained in this study indicates that glycerin was minimal and in compliance with the minimum standard stipulated. This is as a result of complete separation and effective removal of glycerol after tranesterification.

Flash point is the minimum temperature at which a fuel must be heated for it to ignite air -vapor mixture. The U.S. Department of Transportation specified 90 ^{0}C as the flash point for non hazardous fuel [32]. The flash point for this work is 127 ^{0}C. This result shows appreciable consistency with both ASTM, EN standard for biodiesel and works of other researchers. The high value obtained in this study clearly signifies that the biodiesel produced is basically free from methanol; this is because even small quantity of methanol can reduce the flash point reasonably and also negatively affects diesel engine parts such as fuel pumps, seals and elastomers.

Cetane number is a measure of ignition quality of diesel fuel. The higher the cetane number, the easier the fuel will ignite when it is injected into the engine the better the fuel. Beside the reduction of viscosity resulting from transesterification of vegetable oil, one of the most evident changes that result from process is the significant increase in the cetane number of the fuel produced. This work indicates that the cetane number was 56. The value obtained is in agreement with both ASTM and EN standard. This implies the mango biodiesel produced to have high ignition quality.

Water content *shows* the cleanliness of the fuel produced. The water content of property that of 0.015 % volume was low when compared with the ASTM standard of 0.05 (% vol.). Hence the fuel does not have the tendency to support microbial growth in the storage tank [23]. This low water content is an indication of the efficiency of the drying method employed in this work.

The acid value obtained for this work is relatively high compare to the EN standard and reported work of [28]. Acid value measures directly the free fatty acids content of the methyl ester. It clearly helps to state the corrosive nature of the fuel, its filter clogging tendency and the amount of water that may be likely present in the biodiesel. This parameter can also be used to measure the freshness of the biodiesel. The higher the acid value the lower the quality of the fuel.

The cloud and pour point are criterion used for low temperature performance of a fuel. This work reporte a values 12 ^{0}C and - 4 ^{0}C for cloud and pour point respectively which are in agreement with the ASTM standard and the reported literatures. This properties help to show the behavior of the biodiesel under a specified climate setting. This shows the biodiesel produced from mango can be used in cold climate region.

IV. Conclusion

Mango seed oil is suitable for the production of methyl (biodiesel) which has similar properties to petro diesel. Although the oil content of the seed is about 14 % which is low compared to other oil seeds; there is certainly the possibility of breeding special genetically modified varieties to yield more oil. Optimum temperature of 60 ^{0}C and catalyst concentration of 1% w/v at reaction time of one hour and molar ratio of alcohol to oil of 6:1 gives the highest yield of 83 wt %. The result of biodiesel characterization shows that the fuel fulfills most of the ASTM and EN Standard so can be used as a possible candidate for replacement for petroleum diesel.

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