

Quality Parameters of Biodiesel Produced from Locally Sourced *Moringa oleifera* and *Citrullus colocynthis* L. Seeds Found in Kaduna, Nigeria

A. O. Aliyu^{1*}, J. M. Nwaedozi¹ and Ahmed Adams¹

¹*Department of Chemistry, Nigerian Defence Academy, PMB 2109, Kaduna, Nigeria.*

Authors' contributions

This work was carried out in collaboration between all authors. All authors read and approved the final manuscript.

Research Article

Received 5th April 2013
Accepted 30th July 2013
Published 14th August 2013

ABSTRACT

Oils were extracted from *Moringa oleifera* and *Citrullus colocynthis* L. (melon) seeds, using soxhlet extractor with n-hexane as solvent. The oil yield were; 51.00% and 50.87% for melon and moringa respectively. The oils physicochemical parameters such as moisture content, density at 50°C, specific gravity at 60°C, ash content, viscosity at 40°C, cetane index, flash point, fire point, cloud point, pour point, refractive index at 30°C, acid value and free fatty acid value were determined using standard methods. The conversion of the oil to biodiesel was achieved by trans-esterification process. The biodiesel yields of 66.81% and 87.20% were obtained for melon and moringa fatty acid methyl esters respectively. The biodiesel physicochemical parameters such as; density at 50°C, kinematic viscosity at 40°C, refractive index at 30°C, specific gravity at 60°C, cetane index, cloud point, flash point, fire point, pour point, acid value, carbon residue, sulphur content, mono-glyceride, di-glyceride, tri-glyceride, free and total glycerin content were also found using standard methods. The quality parameters of the biodiesel were found to be within international acceptable standards.

Keywords: *Transesterification; fatty acid methyl ester (FAME); biodiesel; biomass; Moringa oleifera and Citrullus colocynthis L.*

*Corresponding author: Email: aoaliyu@nda.edu.ng;

1. INTRODUCTION

For more than two centuries, the world has relied heavily on non-renewable crude oil, out of which about 90% is estimated for energy generation and transportation. These have resulted to importing petroleum products from other countries, which would affect the countries gross net income. Also gases such as; CO₂, CO, NO_x and SO_x, (x=1, 2 or 3) which are the principal causes of global warming, are emitted from these fossil fuel. Thus an increased environmental concern, tougher maintenance of clean air standards, necessitates the research for viable alternative fuels, which are environmentally friendly [1].

Biofuel is a generic term for any liquid fuel produced from sources other than mineral reserves such as oil, coal and gas. The most commonly used biofuel are biodiesel and bioethanol [2]. Biodiesel is a renewable alternative to petroleum based fuel, which can be blended at any level with petrol-diesel. It can be made from plant oil, animal fat or even used cooking oil [3]. Biomass are materials derived from living organism, which include plants, animals and their by-products, for example manure, garden waste and crop residues. Since plant produces oil from sunlight and air, and can do so all year round on cropland, these oils are renewable [4]. Trans-esterification is a process in which triglycerides (fat/oil) react with alcohol (methanol or ethanol), in the presence of a catalyst to produce fatty acid alkyl esters (or biodiesel) and glycerol. Animal fats and plant oil are typically made of triglycerides which are esters of free fatty acids with a trihydric alcohol. The alcohol is deprotonated with a base or acid to make it a stronger nucleophile; the acid or base is not consumed during the process thus they are not reactant but catalyst [5]. Once the glycerin is removed, the remaining product is similar to conventional diesel used in diesel engines. In general, methanol or ethanol is the alcohol used (methanol produces methyl esters, ethanol produces ethyl esters) and is base catalyzed by either potassium or sodium hydroxide. Potassium hydroxide has been found to be more suitable for the ethyl ester biodiesel while either base can be used for the methyl ester [6]. Biodiesel is generally produced from virgin vegetable oils requiring low temperature and pressure and produces over 98% conversion yield, provided the starting oil is low in moisture and free fatty acids, by trans-esterification [5].

1.1 Sources of the Oils

1.1.1 *Moringa oleifera* (*Moringa oleifera*)

Moringa is well known to the ancient world, but only recently has it been rediscovered as a multi-purpose tree with a tremendous variety of potential uses. Cultivation of moringa plant takes 3 years. One tree will produce 300 to 400 pods every year and a matured tree can produce up to 1,000 pods. Mature seeds of Moringa yield 38–40% edible oil called ben oil from its high concentration of behenic acid. The refined oil is clear and odorless, and resists rancidity. The seed cake remaining after oil extraction may be used as a fertilizer or as a flocculent to purify water. Moringa seed oil also has potential for use as a biofuel [6].

India is the largest producer of moringa, with an annual production of 1.1 to 1.3 million tones of tender fruits from an area of 380 km². Among the states, Andhra Pradesh leads in both area and production (156.65 km²) followed by Karnataka (102.8 km²) and Tamil Nadu (74.08 km²). In other states, it occupies an area of 46.13 km². Tamil Nadu is the pioneering state in so much as it has varied genotypes from diversified geographical areas and introductions from Sri Lanka [7].

In Nigeria, it is called different names such as: Bagaaruwar, Zogallagandi in Hausa; Ikwe-beke, Okweoyinbo in Igbo and Eweigbale, Eweile, Igiiyanu in Yoruba [8].

1.2 Melon Seed (*Citrullus colocynthis* L.)

Egusi melon belongs to the Cucurbitaceae plant family made up of over 750 genera and 750 species. It is a crawling crop that is widely grown in tropical Africa, for its seeds which are deshelled, dried and grinded into paste and can be added to vegetable soup to confer aroma, improves taste and to thicken the soup. It has a growth period of 8 months and an oil content of about 50% and protein of 30% [8]. The oil content is the main temptation for possible use as source of oil but it is also self sustaining as it is widely grown in Nigeria [9]. It is usually intercropped with standing crops like maize, where it helps to suppress weeds and reduce the cost production. It is called such names as: Shamu in Hausa; Ibara, Bara in Yoruba and Ogiri-isi in Igbo [10].

It has been estimated that in India, 30,000 tons of oil could be extracted from 4 million tons of the total annual harvest of 7 million tons. Some mango kernel oil has been commercially extracted in India with 150 tons being exported in 1976 rising to 850 tons in 1978. Oil is commercially extracted in India and finds use as a cocoa butter substitute. The cake remaining after oil extraction is used in animal feed [11].

Dennis [13], noted that biodiesel works very well as fuel for many diesel engines with only minor modifications. Diesel engines made before the early 1990s may need some modification since many have seals and other components made with natural rubber which degrades when in contact with biodiesel. Biodiesel has a low energy content (124,000 BTU/gallon) compared to petrol diesel (136,000 BTU/gallon). Biodiesel has a slightly higher cetane number than petrol diesel, resulting in better ignition performances properties [12]. Gross Calorific value is referred to as the heat released in calories per gram or BTU per pound at 25°C by the combustion of a unit mass of fuel in a constant volume bomb with substantially all of the water condensed to the liquid state. [12].

Senthil Kumar et al. [13] made a comparative study of the different methods to improve the engine performance using *Jatropha* oil as the primary fuel in a compression ignition engine. Raw *Jatropha* oil resulted in slightly reduced thermal efficiency, higher smoke emissions and increased hydrocarbon and carbon monoxide emissions. The major challenges of biodiesel are its cost and limited availability of fats and oil resources. The cost of raw materials accounts for 60 to 75% of the total cost of biodiesel fuel [14]. Moreover with the increase of human population, more land may be needed to produce food for human consumption rather than to produce oilseeds for biodiesel production.

The aim of this present work is to synthesize biodiesel from moringa and melon seed oils found in Nigeria and to compare the quality of the biodiesel produced with international standard.

2. EXPERIMENTALS

2.1 Materials and Methods

Moringa oleifera seeds and egusi melon seeds were obtained from Kawo market in Kaduna state, Nigeria. The seeds were dried for 32 hours, after which it was deshelled to recover the

fleshy kernels. The fleshy kernels which are soft with a thin whitish coloured skin were dried further to 3% moisture content at 70°C, before been crushed to a size of 1.0mm for extraction [15] [10]. Methanol (percentage purity 97%, specific gravity 0.7976), n-hexane, Isopropyl alcohol, Phenolphthalein, Potassium hydroxide (KOH), Propan-2-ol, Sodium hydroxide were purchased from Consat Chemical Company, Magajin Gari Kaduna, Kaduna State, Nigeria and were used without further purification.

2.2 Oil Extraction

Oil extraction was carried out using Soxhlet apparatus with n-hexane as solvent. 100g of the grinded sample was poured into the thimble. Two third volume of the round bottom flask was filled with the solvent. The heating mantle was adjusted to about (60-70)°C and heating commenced. As the solvent was heated continuously, it starts to evaporate and condenses back into the sample in the thimble. The oil extracted, containing some portion of the solvent was then recycled back to the round bottom flask as it refluxes and the total process of reflux continues until total oil extraction was observed. A rotary evaporator was used to separate the oil from the solvent, at the temperature of 65°C. The flask containing the extracted oil was dried in an oven at a temperature of 90°C - 100°C for 5 minutes and then cooled in a desiccator. This process was repeated for 5 more runs to obtain a reasonable oil quantity.

2.3 Biodiesel Production Procedures

2.3.1 Degumming

Degumming of the oil samples was accomplished by mixing 3 – 5% distilled water with 200 cm³ of oil. The water combines with the gums and precipitates out after allowing the mixture to settle for approximately one hour, and the water was drained off the bottom of the oil. Several washings were carried out to remove all gum traces. The oil was heated to a temperature above 100°C, and allowed to cool to a temperature of 60°C and then separated.

2.3.2 Titration procedure

The free fatty acid level was neutralized by carrying out titration in order to determine the amount of NaOH used for every litre of oil. 1g of NaOH was weighed into a 1000 cm³ standard volumetric flask and made up to the mark to produce a NaOH standard solution of 0.025 mol/dm³ concentration. 10cm³ of isopropyl alcohol was poured into a beaker and few drops of phenolphthalein indicator added. This was titrated against the NaOH standard solution which was in a burette, while stirring properly. As the NaOH solution was dropping into the alcohol, a flare of red was observed which disappeared as stirring continued. Stirring continued until the red colour persists. This occurs when the isopropyl alcohol has been neutralized. This is a blank titration. 1 cm³ of oil was added to the mixture of the blank titration using a calibrated pipette and stirred until the red colour disappeared again. Titration then continued, using the NaOH standard solution, while stirring until the red colour persist. This is when the free fatty acid has been completely neutralized. The amount of NaOH standard solution added since completion of the blank titration was recorded as x. The total amount of NaOH needed as catalyst for the experiment was determined using the relationship: 3.5 + x.

Where; x= End point of titration

3.5= amount in grams of NaOH needed as catalyst for fresh oil. This is as shown in Table 1 below.

Table 1. Mass of NaOH required for neutralizing the oils and as catalyst

Oil	Titre value (cm ³)	Mass of NaOH obtained from titration (g)	Mass of NaOH required to neutralize the oil (g)	Mass of NaOH required as catalyst (g)
Melon	23.860	0.0239	4.7725	3.5240
Moringa	22.850	0.0229	4.570	3.520

2.3.3 Neutralization

NaOH standard solution of 0.6 mol/dm³ concentration was used for the neutralization of 200 cm³ of the oil feedstock. The oil was heated while been stirred, the NaOH solution was then added while heating and stirring continuously for 15 mins, up to a temperature of 80°C. The neutralized oil was separated from soap using a centrifuge running at 4,500 rpm for 20 mins. Soap formed at the bottom of the centrifuge while the oil floated at the top. The oil was removed washed 3 times with warm water (50°C), dried and then analyzed, before biodiesel production.



Equation (1): neutralization reaction.

2.3.4 Preparation of methoxide

For every 1litre of oil, 200 cm³ of methanol would be needed. This was mixed with the amount of NaOH calculated from the titration. Thus, for 200 cm³ of oil, 40 cm³ of methanol was used. The methoxide solution was prepared by dissolving 3.524 and 3.520 grams of NaOH into 200 cm³ of methanol for melon and moringa seed oil respectively, in a stopper flask. The flask was swirled until the NaOH dissolved in the methanol. A mixture of sodium hydroxide and methanol was obtained after 25-30 minutes of shaking, producing sodium methoxide.

The methoxide solution was mixed with the oil sample (at 45-50°C) for 1.5 hours using a magnetic stirrer while heating lightly between 40°C and 50°C in other to avoid evaporation of methanol in the mixture. The mixture was then poured into separating funnels and allowed to stand for 24 hours. The byproduct mainly glycerin; a jellylike, dark brown, semi liquid residue was at the bottom, while the biodiesel, a light coloured liquid was at the top layer. After separation, the raw biodiesel was washed with warm distilled water to remove traces of soap and other contaminants and finally dried.



Equation(2): is the methoxide reaction

2.3.5 Washing

Water was added to the jar containing the biodiesel by carefully dribbling the water down the side of the glass. The biodiesel was observed flowing on top of the water. The mixture was

stirred gently but enough to allow the two layers to mixed properly together. The water become cloudy since it dissolves the impurities better than the biodiesel. Some sample of the biodiesel was carefully removed from the top of the jar using a ladle into another jar and the washing process repeated. After washing was completed, the sample was allowed to settle down before the water was drained off; the remaining little trace of water was gently evaporated on low heat at 50-55°C [16].

2.4 Quality Parameters of the Extracted Oil

Parameters of both the oil and biodiesel have been analyzed by specific method to verify whether the products conform to the American Standard of Biodiesel Testing Methods (ASTM D 6751). Viscosity was determined in mm^2/s at 40°C Using Cannon-Fenske 1972 – B50 Viscometer. A hand held refractometer model 3M range 58 – 90%, was used to determine the refractive index of the sample at 30°C. Total acid value was measured using titration method proposed by Okpuzor [17]. Cloud point was determined by ASTM D 2500; cetane index was determined by ASTM D 4737 standard method. Density at 50°C was determined by ASTM D 1298 method, Carbon residue was determined by ASTM D 4530, the sulphur content was determined by ASTM D5453, Pour point was determined by ASTM D 97 method. Flash point and fire point were determined by ASTM D 93 method. Finally, the total and free glycerine were determined by ASTM D 6584. Similar method was also used to determine the mono, di and tri – glyceride content. Both parameters were analyzed according to Van Gerpen [18].

3. RESULTS AND DISCUSSION

3.1 The results of quality parameters of the oil (Feedstock) are shown in the Table 2.

The percentage yield of the extracted oils was (50.87 and 51.00) % for moringa and melon oil respectively. This was lower than the values reported for *Jatropha* oil (52%) and Castor oil (56%) [19]. The color of Melon oil was dark brown while that of moringa oil was yellowish. The odors of the extracted oils were agreeably odorless for that of melon oil and a sweet nutty smell for moringa oil. Though natural fats and oil are oily, greasy or waxy, normally tasteless and colorless, but may sometimes be colored due to the presence of oil pigments [20].

The moisture content of the extracted oils were (2.6 and 3.4) % for moringa and melon respectively. These agree with (1.25 – 5.88) % ASTM D6751 standard specification.

The density of the extracted oils at 50°C were (0.903 and 0.906) g/cm^3 for moringa and melon oil respectively. These values were greater than the value specified in the ASTM D1298 standard and could among other things result from experimental procedures. Oils that are denser contain more energy [20]. Thus the oils can be said to be suitable biodiesel feedstock.

Table 2. Quality parameters of oil (feedstock)

Property	Mean ME oil value	Mean MO oil value	ASTM D6751.	Methods
Yield%	51.00± 0.12	50.87 ± 0.40		
Colour	Dark brown	Yellowish	White-yellow	Visual Method
Odour	Agreeably oily	Sweet nutty		Smelling
MC(%)	3.40± 0.15	2.60 ±0.16	1.25- 5.88	Tailang and Sharman,2008.
D (50 ⁰ C)g/Cm ³	0.91 ± 0.00	0.90 ± 0.01	0.86 -0.90	Weighing Method
SG (60 ⁰ C)	0.90 ± 0.00	0.90 ± 0.03	0.86- 1.00	Weighing Method
AC(%)	6.71 ± 0.07	6.50 ± 0.04	4.50 -10.00	Tailang and Sharman, 2008.
KV(40 ⁰ C) mm ² /s	31.10 ± 0.30	33.50 ± 0.20	6.0 max	Cannon Fenske 1972 –B50 viscometer
RI (30 ⁰ C)	1.47 ± 0.01	1.44 ± 0.01	1.245- 1.675	Refractometer 3M range 58-90%
CI	47.06 ± 0.30	52.00 ± 0.03	47.00 -51.00	Van Garpen et al. [19]
AV(mg KOH/g)	1.71 ± 0.01	1.08 ± 0.10	0.80 – 1.00	Titrimetric method
%FFA	0.24 ± 0.01	0.20 ± 0.03	3.00 -5.00 max	Titrimetric Method
FP (°C)	190.00± 0.06	186.00 ± 0.07	90.00 – 130.00	Pensky- Martins cup flash tester
FiP(°C)	193 .00 ± 0.09	190.00 ± 1.30		115V 50/60Hz Van Garpen et al. [19]
CP(°C)	15 .00 ± 0.10	5.00 ± 0.05	-15.00 -5.00	Van Garpen et al. [19]
PP(°C)	12.00 ± 0.07	4.00 ± 0.02	-15.00 -10.00	Van Garpen et al. [19]

KEY: Me = Melon; Mo = Moringa; MC = moisture content; D = density; SG = specific gravity; AC = ash content; KV = kinematic viscosity; RI = refractive index; CI = cetane index; AV = acid value; FFA = free fatty acid; FP = flash point; FiP = fire point; CP = cloud point; PP = pour point.

The specific gravity of the extracted oils at 60°C was (0.90 and 0.902)°C for moringa and melon oil respectively, these indicate a high relative density, which may be as a result of the presence of some impurities present in the oil. These values were also in agreement with the values in the ASTM D287 standard of (0.89 – 0.99).

The ash content of the extracted oils were (6.50 and 6.71) % for moringa and melon oil respectively, as specified by the ASTM standard. Ash content is important for the heating value, as heating value decreases with increasing ash content.

The kinematic viscosity of the extracted oils at 40°C were (31.10 and 33.50) mm²/s for melon and moringa oil respectively are higher than the ASTM specification standard, but are similar to that reported for Jatropha oil (31.5) mm²/s [19]. The high kinematic viscosity of the oils indicates that the oil cannot be used directly in diesel engines, thus there is need for transesterification.

Refractive index of the extracted oil at 30°C were (1.44 and 1.47) for moringa and melon oil respectively, which are in agreement with the (1.245 and 1.675) specified in the ASTM standard.

The cetane indexes of the extracted oils were (47.06 and 52.34) for melon and moringa oil respectively, these values agrees with the ASTM D613 standard of 47 – 51. The higher the cetane index, the shorter the ignitions delay. Fuel with lower cetane index will result in difficulty in starting, noise and exhaust smoke. In general, diesel engine will perform better on fuel with cetane index above 50 [26].

Acid value of the extracted oils were (1.08 and 1.71) mgKOH/g for moringa and melon oil respectively. These values were higher than the 0.5 – 0.8 mgKOH/g indicated in the ASTM D664 standard. The high acid value indicates that more quantity of the base will be required to neutralize the acidity of the oil to be transesterified. High acid value also indicates high free fatty acid content. The percentage free fatty acid content of the extracted oils were (0.204 and 0.240)% for moringa and melon oil respectively. These values were lower than the values reported for Jatropha oil and castor oil (0.27 and 0.26) % respectively and indicate that the oils are suitable since FFA content of not more than 3-5% is recommended for optimum biodiesel production, using base catalyzed reaction [19].

The flash point of the extracted oils were (186 and 190)°C for moringa and melon oil respectively, which were greater than that specified in the ASTM D93 standard. This makes the oils well suited for biodiesel production with respect to safety in handling, storage and transportation. Safety in handling the fuel is also exhibited in their high fire points values (190 and 193)°C for moringa and melon oil respectively. The cloud point and pour point of the extracted oil were (5 and 15)°C and (4 and 12)°C for moringa and melon oil respectively. The cloud point for the melon oil was (15)°C, and its higher than that specified in the ASTM D2500 standard, and also its pour point of 12°C was also higher than the ASTM standard specification, making it unsuitable for making biodiesel to be used in cold countries without the use of additives to improve their cold filter plugging point [21].

3.2 Result of the Physicochemical Parameters (Quality Test) for Biodiesel

Table 3 shows the result of the physicochemical parameters (quality test) of the biodiesel.

Table 3. The physicochemical parameters of biodiesel

Property	Mean ME methyl ester value	Mean MO methyl ester value	ASTM D6751.	Methods
Yield%	66.80 ± 1.06	87.00 ± 1.10		
Colour	Golden	Golden		VISUAL
D (50°C)g/cm ³	0.870 ± 0.07	0.861 ± 0.083	0.860 – 0.890 ASTM D1298	Weighing Method
SG (60°C)	0.897 ± 0.12	0.890 ± 0.10	0.860-0.90 ASTM D287	Weighing Method
SC(Wt%)	0.052 ± 0.03	0.037 ± 0.00	0.015 -0.05 ASTM D5453	Van Garpen et al. [19]
KV(40°C) mm ² /s	3.83 ± 0.03	3.68 ± 0.05	1.900 -6.00 ASTM D445	Cannon Fenske 1972 –B50 viscometer
RI (30°C)	1.33 ± 0.01	1.424 ± 0.02	1.245 -1.675ASTM D6751	Refractometer 3M range 58-90%
CI	50.06 ± 0.02	55.08 ± 0.03	47.00-51 00 Min ASTM D613	Van Garpen et al. [19]
AV(mg KOH/g)	1.03 ± 0.03	0.62 ± 0.02	0.50 -0.80 max ASTM D664	Titrimetric
CP (°C)	5.00 ± 0.08	-i.00 ± 0.00	-15.00 -5.00 ASTM D2500	Van Garpen et al. [19]
FP (°C)	136.00 ± 1.00	103.00 ± 0.02	90.00 -130.00max ASTM D93	Pensky- Martins cup flash tester 115V 50/60Hz
FiP(°C)	150.0 ± 1.30	115.00 ± 1.00		
PP(°C)	3.00 ± 0.01	-3.00 ± 0.00	-15 00 -10.00 ASTM D2500	Van Garpen et al. [19]
TG (wt%)	0.41 ± 0.04	0.20 ± 0.00	0.24 ASTM D6584	GC –MS
FG (wt%)	0.063 ± 0.001	0.06 ± 0.00	0.02 ASTM D6584	GC –MS
MG (wt%)	0.640 ± 0.002	0.61± 0.001	0.80 ASTM D6584	GC-MS
DG (wt%)	0.34 ± 0.001	0.320 ± 0.001	0.2 ASTM D6584	GC –MS
TriG (wt%)	0.30 ± 0.02	0.28 ± 0.01	0.2 ASTM D6584	GC-MS
Cal.V (MJ/L)	33.00 ± 0.01	040.50 ± 0.01	38.00 -43 .00 EN14213	
CR	0.090 ± 0.03	0.037± 0.00	0.015-0.05 ASTM D5453	Ramsbottom

KEY: D = density; SG = specific gravity; KV = kinematic viscosity;

RI= refractive index; refractive index; CR = carbon residue;

CI= cetane index; AV = acid value; SC = sulphur content; FP = flash point; FiP = fire point; CP = cloud point; PP = pour point; TG = total glycerine;

FG =free glycerine; MG= mono-glyceride; DG = di-glyceride;

TriG = tri glyceride

The biodiesel yield obtained after washing and drying was found to be higher in moringa (87.2)% than in melon (66.81)%. The biodiesel yield was generally low compared to 98% recorded by Stidham et al. for vegetable oil [5].

The densities at 50°C of the FAME under investigation were (0.861 and 0.870) g/cm³ and specific gravity at 60°C were (0.890 and 0.897) for moringa and melon respectively. These were in agreement with the ASTM D1298 and D287 standard respectively. The higher the mass of the biodiesel fuel, the higher the energy for work output per unit volume [20].

The viscosity value obtained for the biodiesels at 40°C were (3.68 and 3.83) mm²/s, for moringa and melon respectively, which falls within the ASTM D445 standard. The higher the viscosities, the poorer the atomization of the fuel. Thus, operation of the injection would be less accurate. Moreover, at decreased temperature viscosity of the biodiesel increases [22].

Refractive indexes of the biodiesels at 30°C were (1.33 and 1.424) for melon and moringa respectively and is also in agreement with the ASTM D6751 standard. The refractive index reduced tremendously after transesterification process as compared to that of their oils, this indicates that heavier molecules have been converted to lighter ones, enabling interface detection, enhancing lubricity and increasing functionality of the fuel oil [22].

Carbon residue values obtained for the biodiesels were (0.057 and 0.090) wt%, for moringa and melon respectively, which were in agreement with the (0.05 – 0.3) wt% specified in ASTM D4530, indicating low carbon formation in the biodiesel fuel. A high coke deposit would have been as a result of high total glycerine content, which would deposit hard carbonaceous substance, causing clogging of the fuel injectors. This might lead to more wear on the engine translating into more repairs, shorter engine life and increased pollution [22].

The sulphur content of the biodiesels were (0.037 and 0.052) wt%, for moringa and melon respectively, which are well within the (0.015 – 0.05) wt% value in ASTM D5453 standard. High sulphur content would have resulted from contamination of the protein materials and/or carryover catalyst materials or neutralization materials from the production process. Biodiesel feed stock typically has very little or no sulphur [3].

The cetane index (min) of the biodiesel methyl esters obtained from analysis was 50.06 and 55.08 for melon and moringa respectively. The cetane indexes of the methyl esters were higher than the ASTM D613 standard of (47 – 51 min). The cetane index of a fuel reflects its ignition delay, thus the higher the cetane index, the shorter the ignition delay. The cetane index obtained was low compared to that of soya bean oil methyl ester (75.8 and 56.9) [23]. Generally, higher cetane index gives a good ignition performance.

The acid value (mgKOH/g) of the methyl esters were (0.62 and 1.03) mgKOH/g for moringa and melon respectively. These were in agreement to the (0.5 – 0.8 max) mgKOH/g ASTM D664 standard, except for melon oil methyl ester which was slightly higher (1.03) mgKOH/g. This slight increase in the acid value of the melon oil methyl ester may be due to air and water contact with the biodiesel fuel. A high acid value will cause an increase in the FFA which will in turn lead to corrosion of engine parts as water is increased in the fuel [4].

The cloud point of the produced methyl esters were (-1 and 5)°C for moringa and melon respectively, and these were in agreement with the ASTM D2500 standard. This is an indication that the fuel will perform satisfactorily in both tropical and cold climatic conditions [21]. The flash point of the produced methyl esters were 103 and 136°C for moringa and melon respectively. These were well within the ASTM D93 standard (90 - 130)°C, except that of melon oil methyl ester was slightly higher. The flash point of biodiesel is generally higher than those of petrol diesel. It is higher than 90°C and thus safer than diesel in terms

of storage and transportation from the standpoint of fire hazard. Fuels with flash point above 66°C are considered safe [22]. The fire point of methyl esters were (115 and 150)°C for moringa and melon respectively. These values were lower than that reported for jatropha oil methyl ester having a fire point of up to 296°C [24]. The fire point is the lowest temperature at which a fuel will sustain burning for 5 seconds. The parameter helps to determine the temperature at which fuel will give off inflammable vapour (fire hazard). The pour point of the methyl esters were (-3 and 3)°C for moringa and melon respectively. This compares favorably with the ASTM D2500 standard. Pour point measures the lowest temperature at which the methyl ester is observed to flow. It is an important parameter as this determines the lowest temperature at which the fuel can still be moved before it gels. It also determines the suitability of the fuel in cold weather usage. The free glycerin of the methyl esters produced was slightly above that estimated by the ASTM D6584 specification of 0.02 wt%. They were (0.06 and 0.63) wt% for moringa and melon respectively. This may be as a result of imperfect separation and/or water washing during the production process. The total glycerin of the melon oil methyl ester was (0.41) wt% was higher than the ASTM D6584 standard, which may be as a result of their low mono, di and tri glyceride content. A fuel with excessive total and free glycerin will usually have problems with the fuel settling out in storage tanks, creating a very viscous mixture that clog fuel filter which cause combustion problems in engine.

The mono, glyceride content of the methyl esters were (0.61 and 0.64) wt% for moringa and melon respectively. These values are in agreement with that specified in the ASTM D6584. The di glyceride values were 0.32 and 0.34 wt% for moringa and melon respectively. These values were slightly higher than the 0.2 wt% specified in the ASTM D6584 standard. The tri glyceride values of the methyl esters also were 0.28 and 0.30 wt% for moringa and melon respectively. These values were also higher than the 0.2 wt% from ASTM specification. The increase in the di and tri glycerides may be due to improper washing and/or separation during production.

The calorific values for *Moringa oleifera* and Melon-egusi methyl esters are 34.25 and 33.0 MJ/L, respectively. Calorific value indicates the energy available in the fuel. The calific value of the methyl esters of both the *Moringa oleifera* and melon – egusi were low when compared to the diesel, i.e biodiesel has a calorific value which is about 12% lower than diesel which means biodiesel has lower energy content thus lead to higher consumption of biodiesel in order to achieve yield of diesel in the engine[26]. Generally, the biodiesel fuel oil parameters for melon and Moringa are within acceptable range as compared to the ASTM D6751 test method. They were of good quality and could be used as a fuel and/or blend with petro diesel.

4. CONCLUSION

The melon and moringa oil biodiesel provides a renewable less toxic fuel which can be used in diesel engine or as a blend with petrol diesel. The process employed was alkaline catalyzed transesterification, whose quality was observed to improve by neutralizing the oil feedstock. The oil and fuel yield from these agricultural materials were high. The results of the experimental analysis confirm that the biodiesel from these agricultural materials promotes a clean and healthy environmental friendly, non toxic, renewable fuel. It was observed that the biodiesel produced does not require very high operation temperature, which might have increased the cost of machineries needed. The properties of the biodiesel produced was tested and found to be in confinement with the ASTM D6751 specification for biodiesel fuels.

ACKNOWLEDGEMENTS

I wish to acknowledge the effort of my research supervisors Prof.JM Nwaedozie and Dr (Mrs) A.O. Aliyu for their untiring effort in ensuring that the research is successful. My special appreciation goes to Nigerian Defence Academy Kaduna, Nigeria for allowing me the use of the research laboratory.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

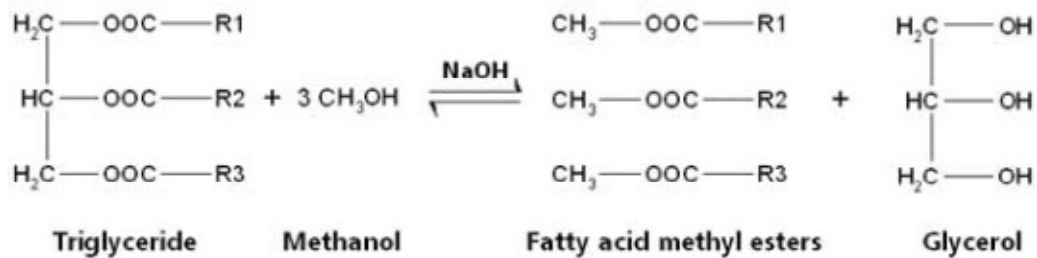
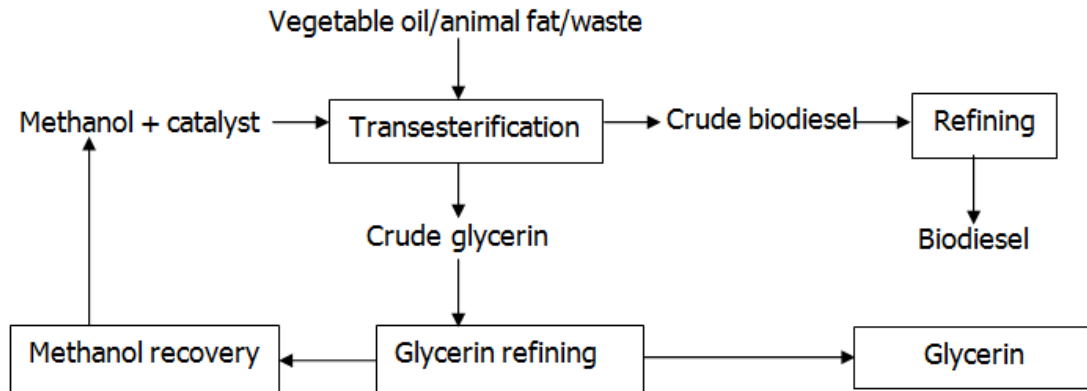
1. An-Fei HSU. Immobilization lipase catalyzed production of alkyl esters of restaurant grease as biodiesel. 2002; 77(4):181-186.
2. Available: <http://www.fleetbiodiesel.com/connect/Biodiesel>.
3. Kusdiana D, Saka S. Kinetics of Transesterification in Rapeseed oil to Biodiesel Fuel as Treated in Supercritical Methanol” Fuel. 2001s; 80: 693-698.
4. Knothe G, Steidley R, Kevin. Kinematic Viscosity of Biodiesel Fuel Component and Related Compounds, Influence of Compound Structure and Comparison to Petrodiesel Fuel Component. 2005; 4: 1059-1065.
5. Stidham WD, Saeman DW and Danzer MF. Method for preparing a lower alkyl fatty ester product from vegetable oil. US patent No. 6. 2007; 127:560.
6. Rashid U, Anwar F, Moser BR, Knothe G. Moringa oleifera oil: a possible source of biodiesel. Bioresour Technol. 2008;99(17):8175–9.
7. Abaye OJ, Onuoha EC, Ugah LA. Transesterified *Thevitia nerifolia* seed oil as a biodiesel fuel, Global Journal of Environmental research. 2007;1(3):124-127.
8. Oluwatomi Olatoye. Moringa Plant”. Nigeria Tribunal Newspaper. 2010; Tuesday 6 July, 14.
9. Achu MB, Fokou E, Tchiegang C, Fosto M, Tchouanguép FM. Nutritive value of some Curbitaceae Oil Seeds from Different Regions in Cameroun. Africa journal Biotechnology. 2000;(11):1329–1334.
10. Bello EI, Makanju A. Performance Evaluation of Egusi Melon (*Citrullus colocynthis* L.) Seed Oil Biodiesel. 2011; 2(5):741–745.
11. Giwa S, Abdullahi LC, Adam NM. Investigating Egusi (*Citrullus colocynthis* L.) seed oil as potential biodiesel feedstock. Energies. 2010;(3);607–618.
12. Rodjanakid K. Using Methyl Ester from Refined Palm Oil Stearin for Compression Ignition Engine. The 12th International Pacific Conference on Automotive Engineering; 2003.
13. Dennis. Biodiesel a renewable domestic energy resource. The Pennsylvania state University reversed edition. 2004; 1-5.
14. Senthil Kumar M, Ramesh A, Nagalingam B. A comparison of the different methods to improve engine performance while using jatropha oil as the primary fuel in a compression ignition engine, International conference on energy and environmental technologies for sustainable development. 2003; 8(10):267-274.
15. Krawczyk T. Biodiesel- Alternative fuel makes inroads but hurdle remain. INFORM, 1996;7 :800-815.
16. Sani A. The use of *Moringa oleifera* seed for surface water treatment”. Unpublished B.Eng (civil) thesis, Department of Civil Engineering Bayero University Kano. Kano state. 1990; 12-15.

17. National Biodiesel Board. Accessed Aug 1st Accessed Aug 1st Biodiesel Basics; 2007. Available: www.biodiesel.org/.
18. Okpuzor J, Okochi VI, Ogbunugafa HA, Ogbonno S, Fagbayi T, Obidiegwu C. Estimation of cholesterol level in different brands of vegetable oils. *Pak. J. Nutri* 2009;8: 57–62.
19. Van Gerpen JH, Shanks B, Pruszko R. Biodiesel Analytical Method". Iowa State University. National Renewable Energy Laboratory. 2004;42(5):3041-3393.
20. Nurugesan A, Neluzchezhain N. Production and analysis of biodiesel from non edible oils. A review and sustainable energy review. 2009; 13: 825–834.
21. Raheman H, Phadatare AG. Diesel engine emissions and performance from blends of Karanja methyl ester and diesel. *Biomass and Bio energy*. 2004; 27: 393-397.
22. Antony S. Raja, Robinson DS smart, Lindon C. Robert Lee. Biodiesel production from jatropha oil and its characterization *Res. J. Chem. Sci.* 2011; 1(1):81-87.
23. Sarma AK. Biodiesel Production from *Mesua ferrea* L (Nahar) and *Pongamia glabra* Vent (Koroch) Seed Oil, Ph.D. Thesis, Department of Energy, Tezpur University, India; 2006.
24. Ullman T, Mason R, Montalvo D. Study of cetane number and aromatic content effects on regulated Emission from a heavy-duty engine. Southwest Research Institute Report No. 08-2940, CRC contract VE1; 1990.
25. Chakraborty N, Sarka SC. Development of a biofuel lamp and its comparism with a kerosene lamp. *Journal of Energy in southern Africa*. 2008; 19(2):22-24.
26. Da-Silva MLCP. Relationship between cetane no and calorific value for biodiesel from viscera oil blends with mineral diesel. International conference on renewable energies and power quality (ICREPQ'13); 2013.

APPENDIX

Production of Biodiesel

The transesterification reaction equation is given thus:



Equation (ii) above; transesterification reaction.

© 2013 Aliyu et al.; This is an Open Access article distributed under the terms of the Creative Commons Attribution License (<http://creativecommons.org/licenses/by/3.0>), which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

Peer-review history:

The peer review history for this paper can be accessed here:
<http://www.sciencedomain.org/review-history.php?iid=245&id=7&aid=1853>