The Production And Investigation Of The Physico – Chemical Properties Of Biodiesel Produced From Neem (Azadiracta Indica) Seeds As Alternative Fuel In Compression Ignition Engines

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***Abstract*—This study considered the production of biodiesel from Neem oil and investigating its physico – chemical properties as a potential alternative to the conventional diesel fuel in compression ignition (CI) engines. The biodiesel obtained from Neem oil methyl ester, mono alkyl esters produced using 'Transesterification' process with methanol as alcohol and potassium hydroxide (KOH) as reaction catalyst. Oil content of the Neem seeds was extracted with solvent extraction method using Hexane as extraction solvent and its yield was determined to be 33.33%. The biodiesel yield was determined to be 71%. The physical and chemical properties of the Neem biodiesel were determined using the ASTM standard procedures; the results obtained were in agreement with the ASTM standard values for biodiesel. The viscosity which as a major physical property was determined to be 5.654mm^2/s and the free fatty acid (FFA); a major chemical property was determined to be 1.299gKOH/g. The values of the biodiesel properties are close to those of fossil diesel and therefore they can be blended in order to be used in compression ignition engines. Biodiesel can make a major contribution in the future if it meets the few percent of petroleum and it can provide improved fuel properties lower emission of unburned hydrocarbons, carbon monoxide but higher level of oxides of nitrogen.**

Keywords—Alternative fuel, Biodiesel, Neem oil, Physico – chemical, Transesterification

1.0 INTRODUCTION

During the last decades, a substantial effort to develop alternative fuel sources, most notably biofuels, has been in progress worldwide, motivated by both economic and environmental issues. Diminishing petroleum reserves and increasing prices, as well as continuously rising concern over energy security, environmental degradation and global warming have been identified as the most influential environmental ones.

Neem (*Azadiracta Indica)* is a tree of the mahogany family, melicae which is abundantly grown in varied parts of Asia and Africa. Neem grows on almost all types of soil. It is typically grown in tropical and semitropical regions. It is a fast growing plant with long productive life span of 150 to 200 years, its ability to survive on drought and poor soils at a very hot temperature of 44°C and a low temperature of up to 4°C has been reported [1]. The tree is well known for its medicinal features. Most of the parts such as leaves, bark, flower, fruit, seed and root have applications in the field of medicine [2]. Neem seed, which has higher concentration of oil (39.7 to 60%) is the major source of Neem oil, which is already in commercial use as an insecticide, lubricant and in medicine to treat various kinds of diseases [3]. However, despite its medicinal value and other uses, this will not deterred researchers from carrying investigations on the suitability of production of biodiesel from the oil. Neem oil varies in colour, ranging from yellowish brown to dark brown, it has a strong odour that is said to combine the odours of peanut and garlic, it is bitter in taste due to presence of triglyceride and triterpenoid as its constituets. Neem oil also contains steroids (Campestrol, Beta-sisterol, stigma sterol) and a plethora of triterpenoids of which azadirachtin is most widely studied. The percentage of the oil in the seed makes it a good resource and has high potential for production of biodiesel. The biodiesel is a mono alkyl ester (Methyl Ester) of long chain fatty acid derived from renewable lipids of neem oil [4].

Neem oil also contains steroids (campesterol, beta-sitosterol, stigma sterol) and a plethora of triterpenoids of which Azadirachtin is the most widely studied [5]. Neem oil will become a potential supplier of Biodiesel in future. Biodiesel is a mono alkyl ester (methyl or ethyl ester) of long chain fatty acids derived from renewable lipid of neem oil. Biodiesel thus obtained can be used in any compression ignition (diesel) engines without the need of modification and is therefore a good substitute for diesel fuel. The first documented commercial production of rapeseed oil methyl esters is reported to be in 1988 [6]. It possesses several distinct advantages over petro-diesel in following safety, biodegradability and environmental aspects [4].

Fossil fuels which are non-renewable in nature have been serving the world to meet its energy demand. These fuels are depleting and the world population is increasing every day, also there is a tremendous increase in the production of automotive and other related products resulting in greater demand for such energy sources, these lead to challenges in the price and supply of the fossil fuels [7]. Apart from the economic issues, the extensive use of fossil fuels is responsible for a long-term environmental threat in the form of climatic changes and the slow (but continuous) increase in the average global temperature. The main contributor to the warming of the climate system is the carbon dioxide (CO_2) emitted from various combustion sources. According to the US EPA Inventory of US Greenhouse Gas (GHG) Emissions and Sinks 1990-2009, the transportation sector collectively (including marine and air-transport) accounted for 27.4% of the total US GHG emissions from end-use fossil fuel combustion in 2009 [8]. Then, it appears that biofuels, possessing the critical merit of being renewable and thus showing an inherent benefit in mitigating CO_2 emissions; seem particularly suitable as viable alternatives to the current situation of the (almost) exclusive use of fossil fuels in automotive and truck applications [8].

Biodiesel can be produced from renewable resources such as vegetable oils and animal fats. Vegetable oils have become more attractive owing to its environmental benefits and the fact that it is produced from renewable sources, it is biodegradable and non-toxic. Another advantage using vegetable oils for biodiesel production is its high energy content compared to conventional diesel. Vegetable oils have 90% energy content of diesel [9]. Vegetable oils are extracted from edible and non-edible, renewable oilseeds such as groundnut, neem seeds, soybean, jatropha seeds, rapeseeds, cotton seeds e.t.c.

There are basically three methods of extraction from the Neem seeds: Mechanical pressing; supercritical fluid extraction and solvent extraction. Mechanical extraction is the most widely used method. However, the oil produced with this method usually has a low price, since it is turbid and contains a significant amount of water and metals contents. Extraction using supercritical fluid; the oil produced has very high purity; however the operating and investment cost is high. Extraction using solvent has several advantages; it gives higher yield and less turbid oil than mechanical extraction, and relatively low operating cost compared with supercritical fluid extraction [10].

The biodiesel production consists of reaction of an ester that reacts with alcohol to form another ester and another alcohol. Ester here is the vegetable oil (Neem oil) which consist triglyceride. There are four ways to use neat vegetable oils in diesel engine [10]:

i. Direct use or blending in diesel fuel
ii. Micro emulsions in diesel fuel
iii. Thermal cracking of vegetable oils
iv. Transesterification.

Out of the four methods, transesterification is the most popular and best way to use neat vegetable oils [11]. It was conducted as early as 1853 by scientists E. Duffy and J. Patrick, many years before the first diesel engine became functional [11].

Transesterification being the most widely used method of biodiesel production, a simple method was developed for biodiesel production from nonedible Jatropha oil using a bi-functional acid base catalyst CaO-La2O3 with a high biodiesel yield of 98.76% at transesterification conditions of 160°C, 3 h reaction time, 25 methanol/oil molar ratio and 3 wt% catalyst loading by [12]. [13] Developed a novel method to produce biodiesel with a microwave assisted heating system apart from the conventional heating system using a solid base catalyst $NaNH_2$ from Jatropha oil. Also, it was concluded that the total energy consumption for microwave assisted heating was 10 times less than that required for conventional heating system. [14] Conducted a Comparison study of biodiesel production from crude Jatropha oil and Krating oil by supercritical methanol transesterification. Using noncatalytic supercritical methanol transesterification, high methyl ester yield (85-90%) can be obtained in a very short time (5-10 min) [14].

The physico-chemical properties of fuel are the fuel specifications that define and set the quality standards. The physico-chemical properties that were studied include: cloud point, pour point, flash point, density, viscosity cetane number, iodine value, free fatty acid value and saponification value.

2.0 MATERIALS AND METHODS

2.1 Materials and Equipment

The following are the materials/equipment used in the study:

Neem seeds, Methanol, n – Hexane, Reagent water, Acetic Acid, Chloroform, Iodine Solution, Phenolphthalein Indicator, Sodium hydroxide (NaOH), Potassium iodide solution, Sodium thiosulphate solution, Metal hammer, Digital balance, Soxhlet extractor and accessories, Reflux condenser, Thermometer, Heating mantle, Stirrer, Reagent bottle, Stop watch, Hydrometer, Separating funnel, Conical flask, Volumetric flask, Desiccators, Beaker, Oven, Mantle and pestle.

2.2 Methods

The following methods were used in carrying out this research: Collection and Preparation of seeds, Oil Extraction, Biodiesel Production and Biodiesel Characterization.

2.2.1 Collection and preparation of Seeds

About five (5) kg of dried Neem seeds were collected from various available sources within Abubakar Tafawa Balewa University Bauchi campus, and seven (7) kg of neem fruits was obtained from Azare Local Government Area of the same Bauchi State due to its abundant availability in the area. The fruit is a smooth, olive-like drupe which varies in shape from elongate oval to nearly roundish, and when ripe is 1.4–2.8 centimetres by 1.0–1.5 centimetres. The fruit skin (exocarp) is thin and the bitter-sweet pulp (mesocarp) is yellowish-white and very fibrous. The mesocarp is 0.3–0.5 centimeters thick. The white, hard inner shell (endocarp) of the fruit encloses one, rarely two, or three, elongated seeds (kernels) having a brown seed coat.

The cleaned seeds were sun dried in the open at atmospheric pressure to reduce moisture content until the casing splits and sheds the seeds. The seeds were taken to chemistry laboratory in the department of Chemistry, Abubakar Tafawa Balewa University Bauchi for oil extraction.

2.2.2 Oil Extraction

250ml clean boiling flask was dried in an oven at 105-110^{o}C for about 30 minutes. This was then transferred into a dessicator and allowed to cool; a weighed amount of the sample (grounded seeds) was carefully poured into a labeled thimble. The boiling flask was filled with about 300ml of n- Hexane. The extraction thimble was plugged lightly with cotton wool. The soxhlet apparatus was assembled and refluxing was carried out for about 6 hours. The thimble was carefully removed and the n- hexane in the top container of the set up was drained into another container for re - use. When the flask was almost free of n-hexane, it was removed and dried at 105°C - 110°C for an hour. It was transferred from the oven into a dessicator and allowed to cool then weighed. The oil obtained, was therefore stored in hermetically closed bottle and kept in the refrigerator at 4°C.

2.2.3 Biodiesel production

After the oil extraction, biodiesel was produced through chemical reactions of transesterification and esterification processes**.**

The following are the steps taken in the production of the biodiesel from Neem seeds:

i. Vegetable oil or feedstock Pre-treatment: The extracted Neem oil is further pre-treated to remove microscopic impurities that could alter the reaction process during esterification and transesterification. Water is also removed, as its presence during base-catalyzed transesterification causes the triglycerides to hydrolyze, giving salts of the fatty acids (soaps) instead of producing biodiesel.

ii. Acid Esterification: A sample of the cleaned Neem oil is titrated with a standardized base solution in order to determine the concentration of free fatty acids (carboxylic acids) present in the vegetable oil sample. These acids are then esterified into biodiesel. Esterification increases the yield of biodiesel.

iii. The Transesterification Process: Base-catalyzed transesterification reacts the lipids of the neem oil with methanol, which is the alcohol used for the reactions to produce biodiesel and an impure co-product, glycerol. The catalyst used potassium hydroxide (KOH) is dissolved in methanol and then mixed with the pre-treated oil. The co-products of this reaction are biodiesel and glycerine.

iv. Methanol Recovery: The Methanol used for the reaction is usually removed after the biodiesel and glycerine have been separated into two layers, preventing reaction reversal. The methanol is then cleaned and recycled back to the beginning of the process.

v. Biodiesel Purification: Products of the reaction include not only biodiesel, but also by-products, soap, glycerol, excess alcohol, and trace amounts of water. All of these by-products must be removed to meet the standards.

2.2.4 Characterization

The physical and chemical properties of the extracted oil and the biodiesel produced were carried out in the chemistry laboratory of the Abubakar Tafawa Balewa University, Bauchi.

Physical Properties

The physical properties determined are the kinematic viscosity, density, Flash point, pour point and cloud point using ASTM: D445, D1298, D93, D97 and D2500 standard test procedure respectively.

Kinematic viscosity (V): The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. Viscosity is then equal to the product of this time and a calibration constant for the tube.

Density: A clean dry empty 50ml density bottle was weighed and the mass recorded as M, it was then filled up with distilled water and subsequently with the sample. The mass of the bottle and water was taken and recorded as M1 and that of biodiesel as M2 respectively. The specific gravity was then evaluated.

Flash point: The flash point was determined by heating the sample of the fuel in a stirred container and passing a flame over the surface of the liquid until the vapour ignites. The temperature at which the vapour ignites was noted and recorded as the flashpoint.

Pour point: 25ml of the oil sample was measured into a test tube. A thermometer was placed in the oil sample and test tube was inserted in a beaker filled with ice. The temperature for which the oil begins clogging was observed and recorded.

Cloud point: 25ml of the sample was measured into a test tube. A thermometer was placed inside the oil sample. The sample was cooled at a specified rate and examined at 3°C intervals for flow. The temperature corresponding to the first formation of a cloud was observed and recorded.

Chemical properties

The chemical properties were determined using OAOC 1995 standard test procedure as follows:

Acid value (AV): Potassium hydroxide (KOH) solution was prepared (1g of KOH Pellet was measured using a weighing balance into a volumetric flask. Water was added to the potassium hydroxide and shaken until the KOH dissolved completely).

1g of the extracted oil was weighed into 250ml beaker. Then 25ml of 95% of ethanol was added followed by 1ml of phenolphthalein indicator the solution was then titrated with the potassium solution prepared until a colour change was observed. The acid value was then evaluated using the following relationship.

$$A.V = \frac{(5.61 \times T)}{W}(mg\,KOH/g)$$

Where;

T = Volume of potassium hydroxide required for titration in ml.

W = Weight of sample taken in grams.

Free Fatty Acid (FFA) value: The FFA was evaluated using the following the relationship.

$$FFA = \frac{AV}{2}(mg\,KOH/g)$$

Saponification Value (SV): The alcoholic KOH was freshly prepared by dissolving KOH pellet in ethanol. 1g of oil was measured and poured into a conical flask. 25ml of the alcoholic KOH was added to it and refluxed for 30 minutes with occasional shaking. 1ml of phenolphthalein was added to the mixture and titrated against 0.5M HCl solution. A blank was also carried out. The colour changed from pink to colourless. The saponification value was then determined following the relationship below:

$$S.V=\frac{(V_b-V_a)\times M\times MM(T_2-T_1)}{W}\ (mg\,KOH/g\ of$$

Where;

Va = Volume of HCL used (test titre value), dm^3

Vb = Volume HCL used (black titre value), dm^3

M = Molarity HCL, mol/dm^3

MM = Molar mass of KOH = 56.1 g/mol

W = Weight in g of the sample taken.

Iodine Value: 1g of the oil sample was weighed into a dry 250ml conical flask followed by 10ml of chloroform and the flask was shaken until the oil was dissolved. A 20ml hanus solution was pipette into the flask. The flask was covered and kept in the dark at room temperature for 30 minutes. A blank containing all the reagents except the sample was also prepared. 20ml of 20% potassium hydroxide was pipette and made up to 100ml into the solution. The iodine liberated from the solution was back titrated with 0.1M sodium thiosulphate solution using 1ml of 2% starch indicator. The blue black coloration disappeared to colorless. The blank titration was also carried out. The iodine value was determined following the relation below:

$$I.V=\frac{(V_a-V_b)\times M\times 126.9}{W}(gI/100g)$$

Where;

VA = titre value (test titration), dm^3

Vb = black titre value, dm^3

W = Weight of oil sample, g

M = Molarity of sodium thiosulphate solution, mol/dm^3.

Cetane number (CN)

The calculated saponification value (SV) and iodine value (IV) were used to calculate the cetane number (CN) which is the ability of fatty acid methyl esters as a fuel to ignite quickly after being injected. Empirical formula was proposed by [15] and was used in the work. The higher its value, the better is its ignition quality. This is one of the most important parameter which is considered during the selection of fatty acid methyl esters for use as a biodiesel.

$$\text{Cetane } Number(CN)=46.3+\frac{5458}{[SV-0.225\times IV]}$$

3.0 RESULTS AND DISCUSSION

The Physico - chemical properties of Neem biodiesel from different sources were studied and presented in table 1. The percentage yield of the Neem oil was determined to be 33.33%, which fall within the range of the percentage oil content (30 - 60%) as specified by the ASTM standard. This is shown in plate 1. 500ml of Neem biodiesel was produced from 700ml oil sample as shown in plate 2. The percentage yield of the biodiesel was determined to be 71%; this is low when compared with 98% as reported in literature when a base catalyzed mechanism is used. Perhaps it could be due to the formation of soap which was so prominent during the conversion process.

Table 1: Results for the physico-chemical properties of neem biodiesel produced

Property	Test procedure	First test	Second test	Third test	Average value	Calculated value
Density	ASTMD 1298	-	-	-	-	0.9758 g/cm3
Kinematic Viscosity	ASTMD 445	-	-	-	-	5.645 mm^2/s
Flash point	ASTMD 93	146	148	144	146	146 ^oC
Cloud point	ASTMD 2500	12	12	12	12	12 ^oC
Pour point	ASTM97	6	6	6	6	6 ^oC
acid value	OAOAC 1995	3.8	5.6	4.5	4.63	2.598 gKOH/g
FFA value	OAOAC 1995	-	-	-	-	1.299 gKOH/g
Iodine value	OAOAC 1995	4.3	3.7	3.8	3.9	35.532gI/100g
Saponification value	OAOAC 1995	30.5	33.1	32.1	31.9	91.443mgKOH/g

Plate 1: Sample of extracted Neem oil

Plate 2: sample of biodiesel produced

Physical Properties

From table 1 above, the density obtained for the Neem biodiesel (0.9758 g/cm^3) was in agreement with the specified ASTM standard, which ranges from 0.860 to 0.90 for biodiesel. The density of the biodiesel was found to be slightly higher compared to that of conventional diesel which is 0.920g/cm^3. This is in agreement with what is obtained from literature as reported by [10] that; Biodiesel fuels are characterized by higher density than conventional petroleum diesel, which means that volumetrically-operating fuel pumps will inject greater mass of biodiesel than conventional diesel fuel.

The kinematic viscosity of Neem biodiesel as seen from table 1 was obtained to be 5.645mm^2/s which is in agreement with the specified ASTM standard of biodiesel. The viscosity of biodiesel is higher compared to that of conventional diesel which is 3.6mm^2/s. The implication is that biodiesel will have lubricating effect in engines which will be an added advantage to the users, since it will reduce wear and tear in the engine. 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. From the result it can be concluded that the Neem 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 conventional diesel.

The flash point of the neem biodiesel was determined to be 146^{o}C as seen from table 1, which is in agreement with the specified ASTM standard of > 120°C. The higher the flash point the safer the fuel and vice versa. The flash point of Biodiesel is higher than that of conventional diesel. The result obtained indicates that the produced biodiesel falls under the non-hazardous category of National Fire Protection Association Codes. 8279

The Cloud and Pour point of Neem biodiesel produced are within the standard limit of biodiesel according to ASTM D6751 standard. The Cloud and Pour points of 10^{o}C and 6^{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 the conventional diesel fuel.

Chemical Properties

The Iodine value was determined to be 35.532gI/100g as seen from table 1. The iodine is introduced in Neem biodiesel, reacts with the double bonds within the fatty acid structure. The result of iodine value of Neem biodiesel revealed a higher value compared with that of the conventional diesel fuel. Iodine value is used to measure the chemical stability property of substance against oxidation and the higher the iodine value the higher the number of double bond and hence lesser stability. This shows that pure diesel is more stable compared to the biodiesel. However, the double bonds in biodiesel helps attract oxygen to the compound, and aid proper burning of biodiesel over pure diesel fuel.

Acid value is defined as the number of milligrams of potassium hydroxide required to neutralize the free fatty acid (FFA) presents in 1 gram of the sample (Neem biodiesel). The acid value was determined to be 2.595mgKOH/g. The test was done in typical titration method with a potassium hydroxide (KOH) solvent with known concentration. As the concentration of biodiesel increases in the fuel samples, the acid value increases. Temperature also has an effect on the acid value. If fuel is exposed to high temperature, oxidation occurs due to higher rate of reaction of fuel molecules with oxygen in the air, resulting in increase of acid value.

The Saponification value is the number of milligrams of potassium hydroxide required to neutralize the fatty acids resulting from the complete hydrolysis of 1g of fat. It was determined to be 91.443mgKOH/g. It gives information concerning the character of the fatty acids of the fat- the longer the carbon chain; the less acid is liberated per gram of fat hydrolysed. It is also considered as a measure of the average molecular weight of all the fatty acids present. The oil extracted is characterised by long chain fatty acids, hence low saponification value because they have a relatively fewer number of carboxylic functional groups per unit mass of the fat and therefore high molecular weight.

Free Fatty Acid (FFA) was determined to be 1.299mgKOH/g as seen from table 1. FFA has a significant effect on the transesterfication of glycerides with alcohol using catalyst. The free fatty acid value is a measure of the amount of carboxylic acid groups present per gram of the oil and the higher value significantly affects the efficiency of transesterification in the sense that higher amount of FFA leads to emulsification and presents a great difficulty during separation of the biodiesel from glycerol.

The cetane number of the biodiesel was found to be 56.56. The standard values of cetane number of biodiesel as specified by the ASTM are 48-69. It was observed that, with increase in ethanol blends, there was a corresponding increase in cetane number. Therefore, the knocking tendency of the engine decreases with an increase in the percentage of ethanol in the blend.

4.0 Conclusion

Biodiesel as a domestic fuel produced from Neem seeds can serve as a very good alternative to the conventional diesel fuel in diesel engines, due to it availability, biodegradability and environmental friendliness. Neem oil has potential as an alternative energy source. But it is not possible for oil alone to solve dependency on foreign oil within any particular time frame. Significant commitment of resources would require increasing production of Neem oil. Based on the results obtained from all the experiments, it can be concluded that filtered Neem oil (biodiesel) can be a feasible substitute for the conventional diesel fuels. The present study also confirmed that location where the seeds are obtained affect oil yield. The biodiesel yield was determined to be 71%, which could be due to the high free fatty acid content of the oil (1.299mgKOH/g). The physical and chemical properties were determined and they conform to the specified ASTM standards.

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