Optimization and Evaluation of Compression Ignition Engine Fuel Property of Neem Biodiesel

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Abstract-The universal increasing energy demand as a result of increase in population and high spate of industrialization coupled with the fast depletion of fossil fuel has resulted in the search of alternative energy sources to replace fossil fuel. This research work is therefore focused on optimization and evaluation of compression ignition engine fuel properties of neem seed oil fatty acid methyl ester (NSOFAME). The neem seed oil (NSO) was extracted using solvent extraction method. The oil was characterized based on American Society for Testing and Materials (ASTM) method. The fatty acid profile of the neem seed oil was determined using gas chromatography mass spectrometry while the functional group of the oil was analyzed using Fourier transform infrared spectroscopy. The effect of process parameter on the yield of NSOFAME was investigated using one factor at a time method. The NSO was pretreated to reduce the free fatty acid below 1% and then tranesterified using methanol in the presence of sodium hydroxide, (NaOH) catalyst. The fuel properties of the NSOFAME produced was determined based on ASTM standards. The physiochemical properties of NSO , density, saponification value, iodine value, peroxide value, kinematic viscosity, fire point, flash point cloud point, pour point, refractive index, specific gravity, moisture content, acid value, free fatty acid, and calorific value gave the values 984Kg/m^3, 21omgKOH/g, 64.88gI$_2$/100goil, 6meq/Kg , 64.06mm^2/s, ^{0}C, ^{0}C, ^{0}C, ^{0}C, 1.4662, 0.984, 0.25%, 4.40mgKOH/g 2.20% and 29.79MJ/Kg respectively. The fatty acid profile of NSO is evenly distribution with saturated and unsaturated acids suitable for biodiesel production. The process parameters , catalyst concentration, reaction temperature, ,methanol to oil molar ratio, reaction time and agitation speed greatly affected the biodiesel yield as their increase resulted in the increase of biodiesel yield until the optimum parameter was reached when the yield started decreasing. The experimentally determined properties of the NSOFAME; acid value, density, kinematic viscosity, fire point, flash point, cetane number, refractive index, specific gravity, calorific value, iodine value, cloud point and pour point gave the values, 0.420, 870Kg/m^3, 4.92mm^{2}s-1, 163^{0}C, 160^{0}C, 60.58, 1.4462, 0.870, 38.06MJ/Kg, 70.2I$_2$g/100g, 5^{0}C, 3^{0}C respectively. The optimum conditions suggested by the result analysis for maximum FAME yield of 86% within the ranges studied were: methanol to oil molar ratio 8:1, catalyst concentration 0.9%wt, reaction temperature of 65^{0}C, reaction time of 60 minutes and agitation speed 300 rpm. Actual experiment based on the optimum conditions produced 88% yield of FAME

Key words: Characterization, NSOFAME, optimization, synthesis, transesterification

1 INTRODUCTION

The enormous amount of energy used worldwide today derives from fossil fuel, namely coal, Petroleum and natural gas. These fossil fuels obtained from the earth crust are not inexhaustible as their fast depletion portends. Apart from being none-renewable, they are also none-biodegradable and environmentally not friendly. The growing concern due to fast depletion and environmental pollution caused by the conventional fossil fuels has led to the search for environmentally friendly and renewable fuels. Among the various alternatives investigated for diesel replacement, biodiesel has proved to be the foremost to reduce exhaust emission [1]. The additional advantages of biodiesel include high cetane number, being renewable, biodegradable and environmentally friendly [2]. Combustion of fossil fuel release into the atmosphere such gases as green house gases, sulphur oxides (SOx) , nitrogen oxides (NOx) , carbon monoxide(CO) and hydrocarbons (HC) [3].

Presently, the bulk of biodiesel produced all over the world has edible oil as its feedstock. This has therefore raised the fear of many researchers that the continuous use of edible oil for biodiesel production might stress the food uses, prize, production and availability of these oils (4). Consequently this has ignited research into the use of none-edible oil for biodiesel synthesis.

Although, biodiesel is gaining popularity, more than 95% of the renewable resources used for its production are edible oils (5), which will in a long term have serious implications on food availability and the cost of biodiesel as it may be more expensive than petro-diesel. Worldwide, biodiesel production is mainly from edible oils such as soybean, sunflower and canola oils etc. Utilization of edible oils as feedstock for biodiesel production poses a lot of concerns as this practice competes with food supply leading to high cost of edible vegetable oil, and consequently results in relative increase in biodiesel production cost. Therefore, concerted research efforts are geared towards identifying and evaluating non-edible seed oils as suitable feed stocks. There are vast numbers of non-edible oil plants in nature including, neem (*A. indica*), jatropha tree (*J. curcas*), karanja (*P.pinnata*), tobacco seed (*N. tabacum L.*), rice bran, mahua (*M. indica*), rubber plant (*H. brasiliensis*), castor, linseed, and microalgae (6). Jatropha curcas oil plants have been widely studied with respect to biodiesel production from non-edible oils [7]-[10] but the use of most of other non-edible oil plants such as neem, castor etc have not been intensely studied as Jatropha curcas. This research work is therefore geared towards optimization and evaluation of compression ignition engine fuel properties of neem oil biodiesel.

A mono-alkyle ester of long chain fatty acid, biodiesel is known to have characteristics similar to diesel with additional advantages of high lubricity, high cetane number, being biodegradable and environmentally friendly [11]. Burning of fossil fuel results in environmental pollution such as emission of green house gasses , including sulphur oxides (SOx), nitrogen oxides (NOx) and methane [3]. Biodiesel is produced by the reaction of oil or fat with an alcohol usually in the presence of catalyst, which could be a base, acid or an enzyme. If an alcohol typically monohydric alcohol like methanol contacts a fatty acid, it will bond to form biodiesel [12]. Various processes have been adduced for production of biodiesel from oils and fat, including, micro-emulsion with alcohol, catalytic cracking, pyrolysis and transesterification [6],[13],[9],[14]. Among these processes, transesterification has proved to be the most useful means of converting oil or fat into environmentally safe biodiesel [15],[16].

The neem oil plant 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 high temperature of 44°C and a low temperature of less than 4°C has been reported (17), and its high oil content of 39.7 to 60% [18],[19]. A mature neem tree produces 30 to 50 kg fruit every year [20]. It contains a high percentage of monounsaturated fatty acids, a low proportion of polyunsaturated acids and a controlled amount of saturated fatty acids [21]. The aforementioned characteristics of neem oil plants and the fatty acid composition of the oil makes it a useful renewable source for biodiesel production.

Optimization of neem oil was carried out using response surface methodology of central composite design (CCD) in order to determine the optimum reaction conditions for biodiesel production. The five processing factors, methanol to oil molar ratio, catalyst concentration reaction temperature, reaction time, and agitation speed are the independent variables while percentage biodiesel yield is the dependent variable or response. Design expert software version 12.0 was used for the design of experiment and optimization of the reaction conditions.

II. Materials and Methods

A Materials

Neem seeds, reagents, glass wares, equipments including gas chromatography mass spectrometer (GC-MS), Fourier transform infrared spectroscopy (FTIR), viscometer, magnetic hot plate, water bath, soxhlet extractor, design expert software version 12.0 etc.

B Material preparation

Neem seeds were purchased from Zaria in Northern Nigeria. The seeds were cleaned by removing the dirt and accompanying foreign bodies through hand picking. The cleaned seeds were then oven dried at 55^0C to obtain constant moisture content. Size reduction of the seeds was effected with mechanical grinder.

C Extraction of oil from neem seed

Solvent extraction was used for extraction of oil from the dried grind neem seeds. Soxhlet extractor was used to determine the oil content of the seeds . The oil used in this work was extracted thus: 5kg of dry, ground seed was introduced into a plastic container containing 4 liters of n-hexane. The content of the container were vigorously shaken after replacing the cover and the container made air tight to prevent evaporation of the n-hexane. The seed was left to mercerate for a day. Then the dissolved oil in hexane was decanted and the slurry filtered. The filtrate was then distilled to recover the n-hexane at 65^0C [22]. The percentage oil yield was calculated as:

$$\%\, oil\ yield\ = wejght\ of\ oil\ obtaind\ \div\ weight\ of\ seed\ sample\ x\ 100 \qquad (1)$$

D Characterization of neem oil

The physiochemical properties of the neem oil was characterized based on American Society for Testing Materials, ASTM 6751 (1973) method. Analytical equipments, GC- MS (QP2010 plus Shimadzu, Japan) and FTIR (M530 Bulk scientific(FTIR) were used to determine the fatty acid profile and the functional groups of the oil respectively.

E Effect of process parameters on biodiesel yield

The effects of process parameter on biodiesel yield from neem oil were investigated based on one factor at a time method, involving keeping a factor constant at a time and varying the others in turn. The five factors investigate are, molar ratio of methanol to oil, catalyst concentration, reaction time, reaction temperature and agitation speed.

F Pretreatment of the neem oil

A pre-treatment procedure was followed to reduce the excess free fatty acid of the neem oil below1%. The oil sample was first heated on a heating mantle at 110˚C for 10 minutes for any available moisture to be driven off. The sample was cooled to 60°C in a water bath, and then weighed into 500ml three necked round bottomed flask. Then methanol of 60%w/w of oil mixed with concentrated sulphuric acid of 7% w/w of oil was added. A reflux condenser was fitted into the middle arm of the flask and water circulated at the outer jacket of the condenser. A thermometer was inserted into the sample in the flask from one of the side arms. The whole setup was placed on a magnetic heating mantle and heated at 60^0C for 120 minutes at an agitation speed of 450rpm. The mixture was then transferred into 500ml separating funnel where it later separated into three layers comprising water at the bottom, pre-treated oil in the middle and methanol at the upper layer. The various components were carefully tapped off, water first, and then the oil and finally methanol. Hot distilled water was poured into the oil in a separating funnel, shaken and allowed to stand when it separated into water and oil layers below and above the funnel respectively. The water layer was tapped off from the separating funnel and the pre-treated oil was poured into 250ml beakers and dried carefully in an oven regulated at a temperature of 105^0C until the residual water evaporated completely. After this process, the pre-treated oil was made ready for transesterification [23].

G Production of biodiesel by transesterification

The oil was transesterified using methanol and sodium hydroxide catalyst. A 500ml three-necked round bottomed flask fitted with a condenser on the middle arm, a thermometer and sample outlet on the side arms respectively served as the reactor. The heating system consists of an electromagnetic hot plate which heats the reactor and rotates the metal knob in the reactor through an electromagnetic field. Specified quantity of the oil sample was introduced into the flask and the flask content heated to the temperature established for the reaction. Then methanol and the catalyst mixture (NaOH) was added in the amount established for the reaction, and the stirrer switched on at a specified speed, taking this moment as zero time of the reaction. The reaction mixture was vigorously stirred and refluxed for the required reaction time. At the end of methanolysis, the transesterfied product was made to stand for a day in a separating funnels where it separates into the upper biodiesel layer and the lower glycerol layer. The lower glycerol layer was tapped off first followed by the upper biodiesel layer.

H Biodiesel purification by wet washing

After transesterification, the upper ester layer may contain traces of methanol and glycerol. The remaining un-reacted methanol might corrode the engine components, and glycerin in the biodiesel will lessen the fuel lubricity and cause injector coking and deposits [24]. Such trace of methanol is soluble in water and is therefore removed by wet washing. The methyl ester or biodiesel layer was gently washed with hot distilled water in the ratio of 3:1 water to methyl ester. The methyl ester was gently washed to prevent its loss due to formation of emulsion that results in complete phase separation [25]. The washed biodiesel was dried by heating at 105^0C on a laboratory hot plate until all residual water was evaporated. This conforms with the findings of [26]. The percentage biodiesel yield is given by the expression of equation (1)

$$\%\ biodiesel\ yield = Volume\ of\ biodiesel\ produced \div volume\ of\ oil\ used\ x\ 100 \quad (1)$$

I Determination of the fuel properties of neem oil biodiesel

The properties of the biodiesel fuel were characterized based on ASTM standards. The properties include, density, viscosity, iodine value, saponification value, cetane number, acid value , free fatty acid , calorific value, flash point etc.

J Design of experiment for transesterification of neem oil catalyzed by NaOH

Design Expert software (version 12) was used in this study to design the experiment and to optimize the reaction conditions. The experimental design employed in this work was a two-level-five factor fractional factorial design, including 32 experiments. Reaction temperature, catalyst concentration, methanol to oil molar ratio, reaction time and agitation speed were selected as independent factors for the optimization study. The response chosen was the methyl ester yields obtained from transesterification of neem oil. 2^5 (2^n) factor fractional factorial experiments, 10(2n) star points and 6 center points were carried out in order to predict a good estimation of errors and experiments were performed in a randomized order. The actual and coded levels of each factor are shown in Table 1. Alpha is defined as a distance from the center point which can be either inside or outside the range, with the maximum value of $2^{n/4}$, where n is the number of factors [27]. It is noteworthy to point out that the software uses the concept of the coded values for the investigation of the significant terms, thus equation in coded values is used to study the effect of the variables on the response. The empirical equation is represented as shown in equation (2) below:

Table 1: Experimental range and levels of independent process variables for biodiesel production.

Independent Variables	Units	Range and Level				
		-α	-1	0	1	+α
Molar Ratio(X_1)	Mol/mol	2:1	4:1	6:1	8:1	10:1
Catalyst Concentration (X_2)	Wt %	0.25	0.50	0.75	1.00	1.25
Temperature (X_3)	°C	50	55	60	65	70
Reaction time (X_4)	Min.	30	45	60	75	90
Agitation speed (X_5)	Rpm	150	200	250	300	350

Table 2 Experimental design Matrix for transesterification studies of neem oil FAME

Run order	Methanol/Oil molar ratio X_1		Catalyst conc. (wt %) X_2		Temperature (^oC) X_3		Time (Mints) X_4		Agitation Speed (Rpm) X_5		Biodiesel Yield (%)
	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Coded	Real	
1	-1	4	-1	0.5	-1	55	-1	45	+1	300	
2	+1	8	-1	0.5	-1	55	-1	45	-1	200	
3	-1	4	+1	1	-1	55	-1	45	-1	200	
4	+1	8	+1	1	-1	55	-1	45	+1	300	
5	-1	4	-1	0.5	+1	65	-1	45	-1	200	
6	+1	8	-1	0.5	+1	65	-1	45	+1	300	
7	-1	4	+1	1	+1	65	-1	45	+1	300	
8	+1	8	+1	1	+1	65	-1	45	-1	200	
9	-1	4	-1	0.5	-1	55	+1	75	-1	200	
10	+1	8	-1	0.5	-1	55	+1	75	+1	300	
11	-1	4	+1	1	-1	55	+1	75	+1	300	
12	+1	8	+1	1	-1	55	+1	75	-1	200	
13	-1	4	-1	0.5	+1	65	+1	75	+1	300	
14	+1	8	-1	0.5	+1	65	+1	75	-1	200	
15	-1	4	+1	1	+1	65	+1	75	-1	200	
16	+1	8	+1	1	+1	65	+1	75	+1	300	
17	-2	2	0	0.75	0	60	0	60	0	250	
18	+2m	10	0	0.75	0	60	0	60	0	250	
19	0	6	-2	0.25	0	60	0	60	0	250	
20	0	6	+2	1.25	0	60	0	60	0	250	
21	0	6	0	0.75	-2	50	0	60	0	250	
22	0	6	0	0.75	+2	70	0	60	0	250	
23	0	6	0	0.75	0	60	-2	30	0	250	
24	0	6	0	0.75	0	60	+2	90	0	250	
25	0	6	0	0.75	0	60	0	60	-2	150	
26	0	6	0	0.75	0	60	0	60	+2	350	
27	0	6	0	0.75	0	60	0	60	0	250	
29	0	6	0	0.75	0	60	0	60	0	250	
30	0	6	0	0.75	0	60	0	60	0	250	
31	0	6	0	0.75	0	60	0	60	0	250	
32	0	6	0	0.75	0	60	0	60	0	250	

$$Y = \beta_0 + \sum_{i=1}^{5} \beta_i X_i + \sum_{i=1}^{5}\sum_{j=i+1}^{5} \beta_{ij} X_i X_j + \sum_{i=1}^{5} \beta_{ii} X^2_i \quad (2)$$

Where Y is the predicted yield of FAME (%), X_i and X_j represent the transesterification process variables, β_0 is the offset term, β_i is the coefficient of linear (single) effect, β_{ij} is the coefficient of interaction effect and β_{ii} is the coefficient of quadratic effect. Selection of levels for each factor was based on the experiments performed to study the effects of process variables on the yield of biodiesel by alkali catalyzed transesterification of neem oil. The lower level of temperature was 50^0C since below that the reaction rate is relatively slow and the upper level of temperature was 70^0C. The levels of methanol to oil molar ratio were selected between 2:1 and 10:1 and the catalyst concentration was limited between 0.25 wt% and 1.25wt%(based on the initial weight of the oil) with agitation speed range of 150 rpm to 350 rpm. The range and levels of the independent variables and the experimental design matrix for the transesterification are presented in Figures 1 and 2 respectively

III. RESULT AND DISCUSSION

A Oil Yield from Neem Seed

The percentage of oil extracted from the seed of neem using equation 1 is 38%. For neem, the oil yield of 38% approximates the 36.86% yield reported by [28].but was relatively higher than the yields reported for other non-edible seed oil like *Mangifera indica*, 30.7% [29], and almond seed oil 32% [30]. The relatively high oil content of neem seed is a pointer to its suitability for commercial biodiesel production.

B Characterization of the Neem Oil

The summary of characteristics of neem oil is as presented in table 3. From the table, it could be seen that the free fatty acid values of the neem oil is greater than 1%. The high acid value and free fatty acid value of 4.40mgKOH/g and 2.20% respectively for neem oil, is unacceptable for alkali transesterification reaction as this give rise to soap formation and inhibition of ester separation from biodiesel [7]. The kinematic viscosity measures the flow resistance of the fuel, while the density determines the quantity of the fuel metered as this is measured volumetrically. High density and viscosity of neem oil, 984kgm^{-3} and 64.06mm^{2}s^{-1} at 40^{0}C respectively will make its atomization in internal combustion engine difficult and has been associated with increased engine deposits hence it cannot be used directly as bio-fuel [31]. The saponification values of the neem oil feed stock is relatively low (210mgKOH/g), indicative of high concentration of triglycerides which is suitable for FAME production. The key flow properties for winter fuel specification are the cloud and pour point. The cloud point of 5^{0}C and pour point of 1^{0}C for neem are moderately low and suitable for use in warm and temperate climates but not suitable for cold climatic condition. Oils of high cloud and points can readily congeal and faces difficulty of handling during cold weather.

The moisture content of the neem oil, 0.25% is within the 0-1.0% suitable for alkali transesterification without giving rise to soap formation. Iodine value is useful for quantifying the amount of double bond present in the oil and therefore the degree of un-saturation of the oil and susceptibility of the oil to oxidation. The iodine value of 64.88gI_2/100g obtained for neem oil is less than the value 100gI_2/100g indicative of the oil being non-drying.and therefore suitable for biodiesel production. High iodine value of the oil indicates high degree of unsaturation of the fatty acid in the triglyceride, and if heated, such an oil is prone to thermal oxidation and polymerization of the triglyceride causing formation of deposits. Peroxide value, an index of rancidity obtained as 6meq/kg was high and indicative of poor resistance of the oil to peroxidation during storage and handling [32]. The flash point of neem oil, 231^{0}C is high making its handling and storage safe. The calorific value of the oil 29.79MJ/Kg is low compared to that of the biodiesel derived from it.

Table 3.1 Physiochemical properties of neem seed oil

Properties	Unit	NSO
Oil yield	%	38
Density	Kg/m3	984
Saponification value	mgKOH/g	210
Iodine value	(gI_2/100g oil)	64.88
Peroxide value	meq/kg	6
Kinematic viscosity	mm^2/s	64.06
Fire point	^{0}C	292
Flash point	^{0}C	231
Cloud point	^{0}C	5
Pour point	^{0}C	1
Refractive index		1.4662
Specific gravity		0.984
Moisture content	%	0.25
Acid value	mgKOH/g	4.40
Free fatty acid	%	2.20
Calorific vaue	MJ/Kg	29.79

C The Fatty Acid Profile of Neem Oil (GC –MS)

The fatty acid profile of neem oil was carried out with the aid of gas chromatography mass spectrometry (GC-MS). Figure1 shows the GC-MS spectra of neem oil The fatty acid composition of neem oil, is shown in table 2 below. Neem oil comprises 36.76% of saturated acids (stearic acid, palmitic acid, arachidic acid) and 63.24% unsaturated acids (oleic, linoleic and palmitholeic acid). The dominant monounsaturated fatty acid of the oil is oleic, which accounted for 40.59% of the total fatty acid content, hence, the oil belongs to oleic acid category [33]. The oleic acid content of neem oil is comparatively higher than 7-40% reported for coconut oil, palm oil and cottonseed oil [34],[35]. This shows that neem oil is highly unsaturated triglycerides (Triolein). However, the fatty acid components of the neem oil were found to be consistent with the fatty acids present in typical oils used for producing biodiesel.

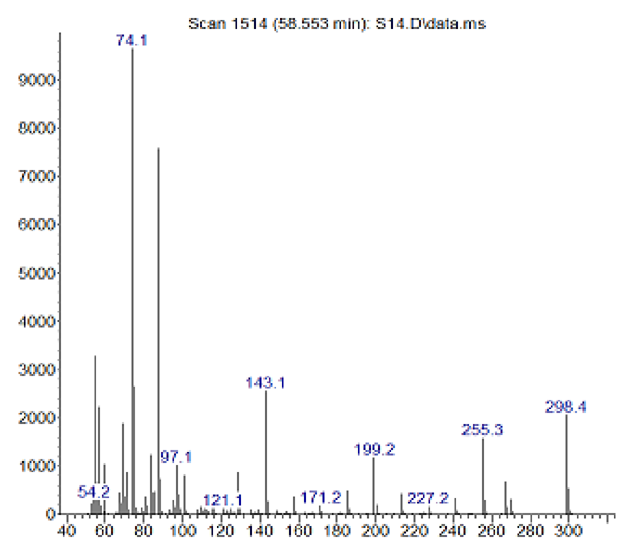

Figure I: GC-MS spectra of neem seed oil.

Table 4: Summary of fatty acid composition of neem oil.

Fatty acid	Structure	Composition (%)	Molecular weight (g/mol)
Linoleic acid	C18:2	22.42	294.48
Oleic acid	C18:1	40.59	282.465
Stearic acid	C18:0	17.56	284.48
Palmitic acid	C16:0	13.56	256.4
Arachidic acid	C20:0	2.69	304.470
Behenic acid	C22:0	1.80	340.58
Lignoceric acid	C24:0	1.21	368.63
Palmitholeic acid	C16:1	0.23	254.408

D Fourier Transform Infrared (FTIR) Spectra Analysis of Neem Oil

The FTIR spectra analysis of neem oil was carried out and shown in Figure 2. This was done to determine the different functional groups present in the feedstock. From the result, discernible peaks of note were recorded .The region 723.1 cm^{-1} (679.61 cm^{-1} – 886.65 cm^{-1}) indicate the presence of =C-H(alkenes) functional groups. They possess bending type of vibrations appering at low energy and frequency region in the spectrum and they are all double bounded. They are attributed to olefinic (alkenes) functional groups and are unsaturated. They will be part of fatty acid methyl esters with unsaturated bond in the biodiesel, such as methyl oleate and methyl linoleate [36], [37]. The characteristics peaks found in the region 1159 cm^{-1} (1050.15 – 1297.23 cm^{-1}) indicate stretching vibrations of C-O and C-O-C. They can also indicate the bending vibration of O-CH_3 in the spectrum [38], [39]. The band region of 1375cm-1 can be ascribed to the bending vibratio of C-H methyl group while the band at1461cm-^{1}(1400-1800 cm^{-1} is]ascribed to C=C bending vibration [40]. The peaks at 2855.75cm^{-1} and 2922.07 cm^{-1} indicate symmetric and asymmetric stretching vibrations of C-H alkane groups respectively. They could be methyl (CH_3) or methylene groups and they require high energy to cause stretching vibrations within their bond when compared to the ordinary C-H bending vibrations of alkene groups detected at low energy and frequency region [36],[37]. The peak at 3008cm^{-1} is attributed to the stretching vibration of =C-H alkene groups. They are detected above wave number 3000 cm^{-1} in the spectrum compared to corresponding alkane C-H stretching groups detected below 3000 cm^{-1}. The peak at 3473 cm^{-1} with stretching mode of vibration is ascribed to the presence of O-H groups. They are single bounded and at high energy region in the spectrum [41],[42].

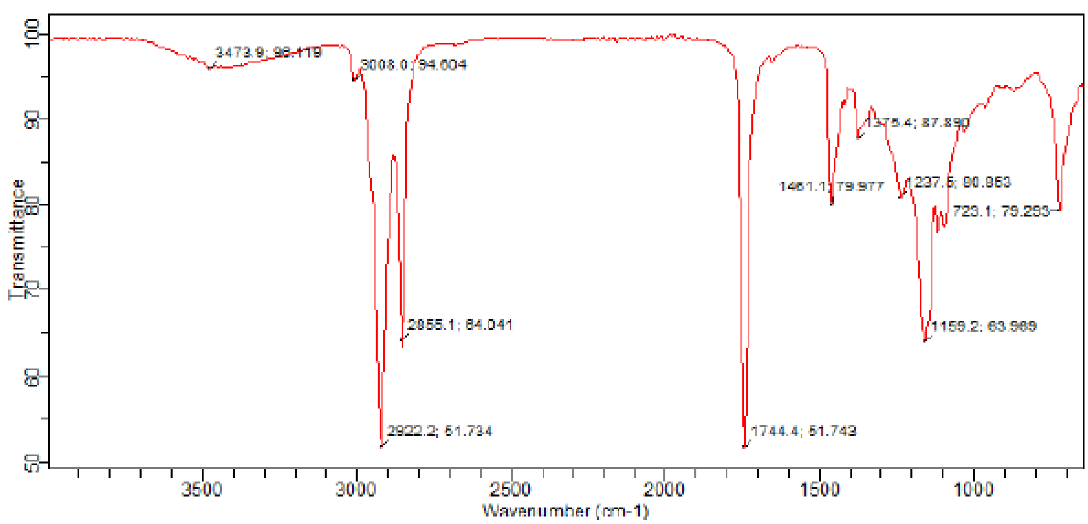

Figure 4.1: FT-IR spectra for neem oil.

E Effects of Process Parameter on Biodiesel Yield

Effects of methanol/oil molar ratio on biodiesel produced.

The alcohol to oil molar ratio is one of the most vital factors that can affect the yield of esters. The stoichiometry of the transesterification reaction requires 3:1 methanol to oil molar ratio to yield 3 moles of ester and 1 mole of glycerol, but most researchers have found that excess alcohol was required to drive the reaction close to completion. In this work, the effect of methanol to oil molar ratio of 2:1 to 12:1 was investigated, when other process parameters were fixed (catalyst concentration, temperature, time and agitation speed). The yield of methyl esters to the different molar ratio of methanol to oil is shown in Figure 3. The results indicated that methanol oil molar ratio has significant impact on FAME yield. The maximum ester yield was obtained at a methanol to oil molar ratio of 8:1 for neem oil. The yield reduced when the molar ratio was higher than 8:1 for neem oil. This trend can be explained by the fact that while the increase in methanol to oil molar ratio favors transesterification reaction, very high ratio of methanol to oil decreases the catalytic activity of the catalyst, resulting in the reduction of biodiesel produced. This is in agreement with the findings of [43] and [44]. It has also been reported by [45] that the use of excess alcohol for transesterification increases the polarity of the reaction mixture and this increases the solubility of the glycerol. Increase of solubility of glycerol retards separation of glycerol from biodiesel and thus reduce the yield of biodiesel.

Effects of catalyst concentration on biodiesel yield.

The alternative reaction pathways for breaking of bonds created by the use of catalysts most often involve lower activation energy. The yield of biodiesel increases with increase in catalyst concentration until an optimum yield was obtained at 1%wt. of catalyst when the yield starts declining. Decrease in biodiesel yield beyond the 1%wt. catalyst concentration can be explained by the fact that in the presence of excess catalyst above the optimum 1%wt., the excess catalyst react with the oil to form soap which increases the viscosity of the reaction mixture, hindering effective dispersion and mixing of the reactants and also separation of glycerol from biodiesel which gives rise to reduction of biodiesel production. This is in conformity with the findings of [46] and [47].

Effects of temperature on biodiesel yield.

The rate of reaction is known to increase with increase in temperature. In order to investigate the effects of temperature on the yield of neem oil biodiesel, the temperature was varied from 50^0C to 75^0C while the other parameters, catalyst concentration, methanol to oil to oil molar ratio, reaction time and agitation speed are kept constant as shown in figure 5. From the figure, it could be seen that biodiesel yield increased with increase in reaction temperature until a maximum yield was obtained at optimal temperature of 65^0C when the yield starts decreasing. The decrease in the biodiesel yield beyond 65^0C may be explained by the fact that the boiling point of methanol is approximately 65^0C, and therefore on exceeding this temperature, the backward reaction is favored as most of the methanol will be lost by evaporation, thus reducing the yield. This conforms with the findings of [9].

Effects of reaction time on biodiesel yield.

In this work, the effects of reaction duration from 15 to 90minutes) on the yield of biodiesel from neem oil was investigated. It was found that reaction time of 60 minutes was needed for a maximum yield of FAME for neem oil investigated and beyond it the yield decreased as shown in Figure 6. The decreased in the yield after 60 minutes may be due to reversible reaction of transesterification resulting in loss of esters [43]. Also longer reaction time most times allows the fatty acids present to react with alkali and this will result to soap formation. The presence of soap retards the formation of ester [24].

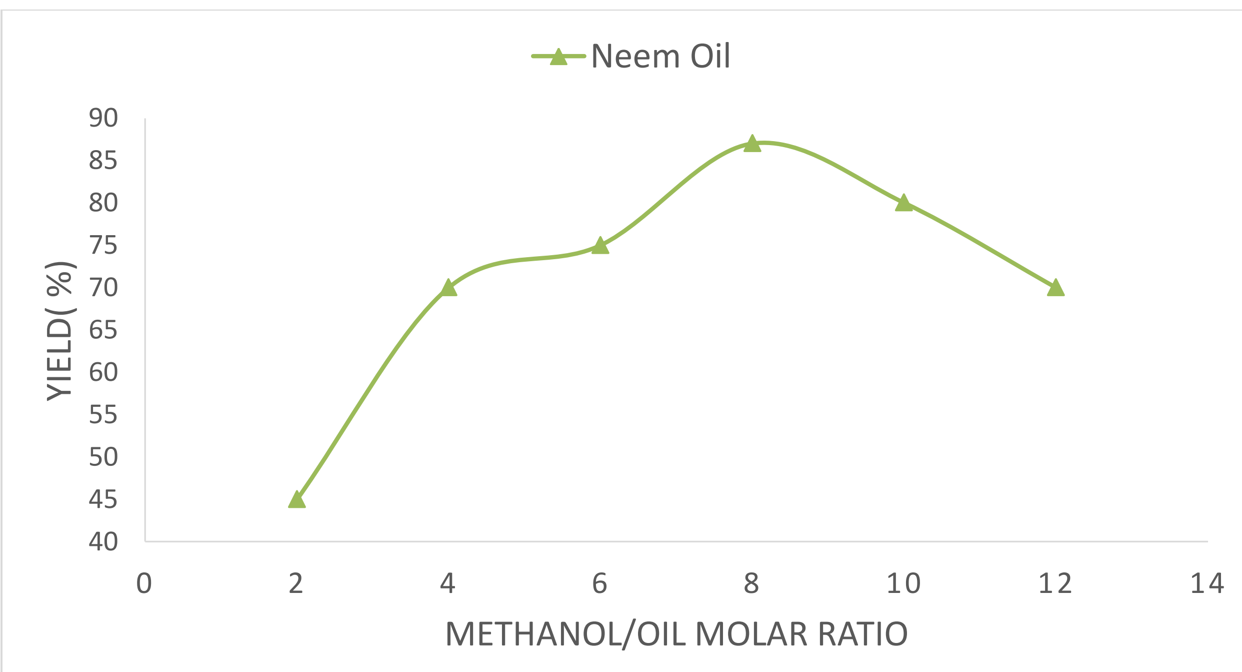

Figure3: Effect of methanol to oil molar ratio on biodiesel Yield.

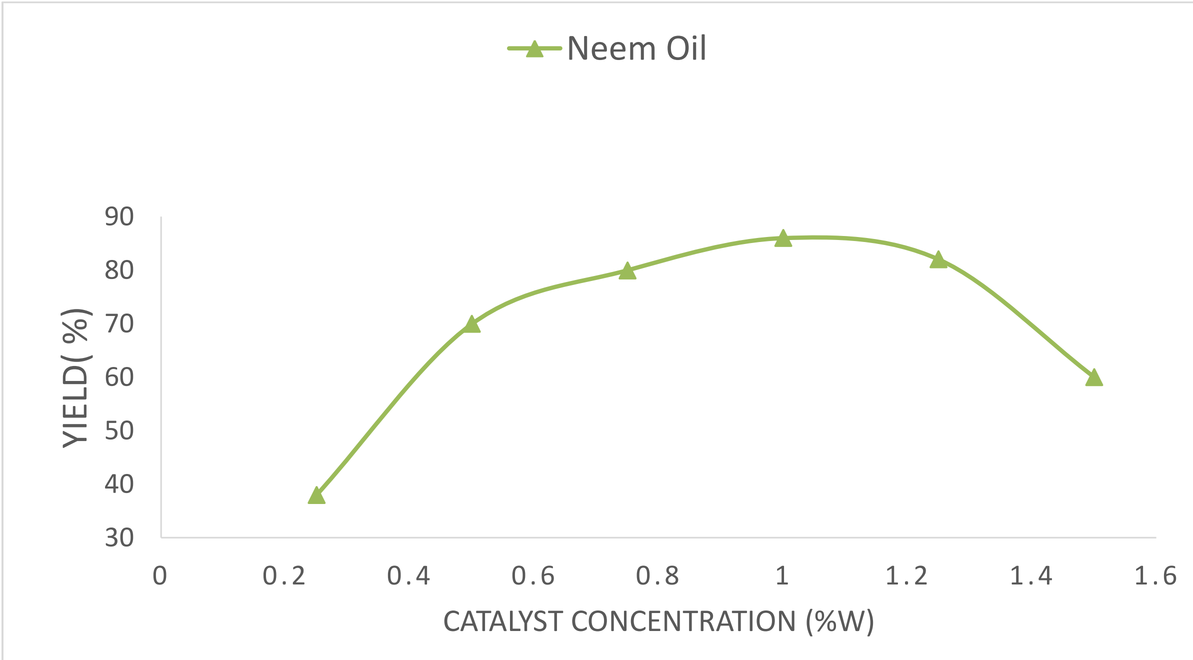

Figure 4: Effect of catalyst concentration on biodiesel yield

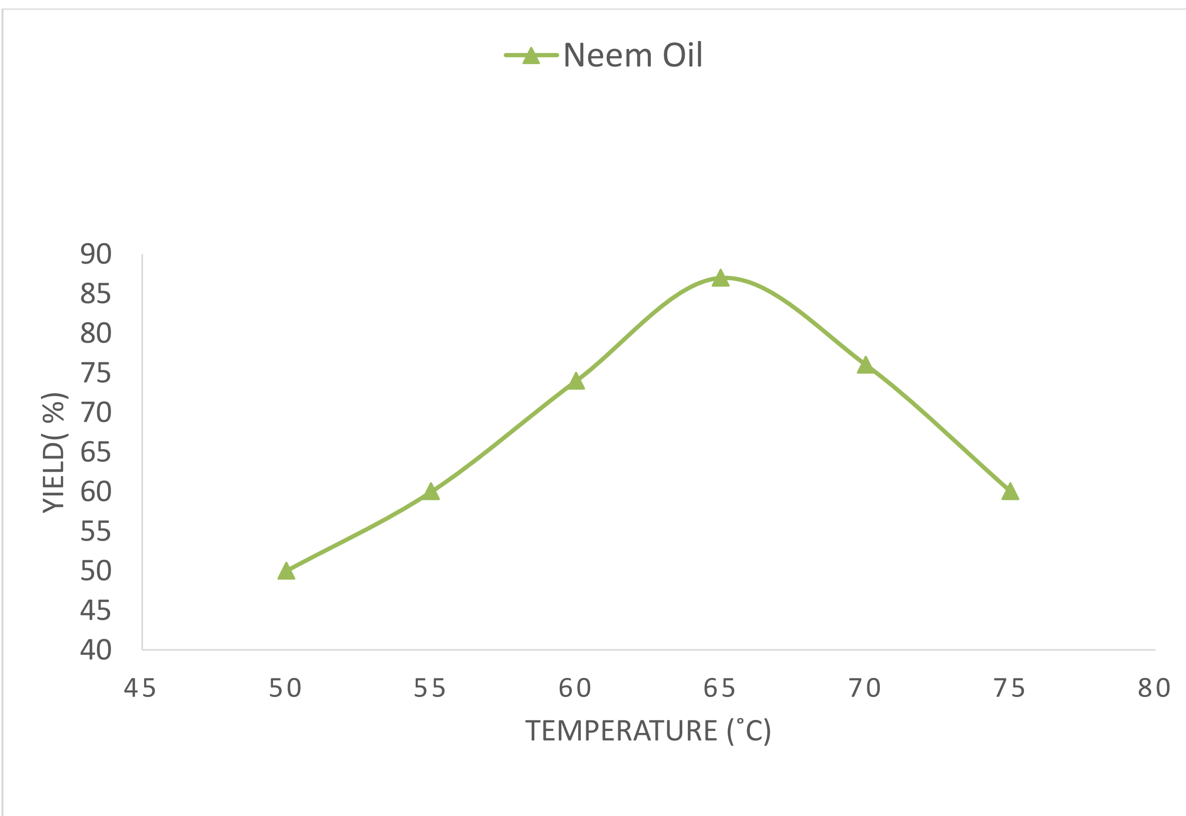

Figure5: Effect of temperature on biodiesel yield.

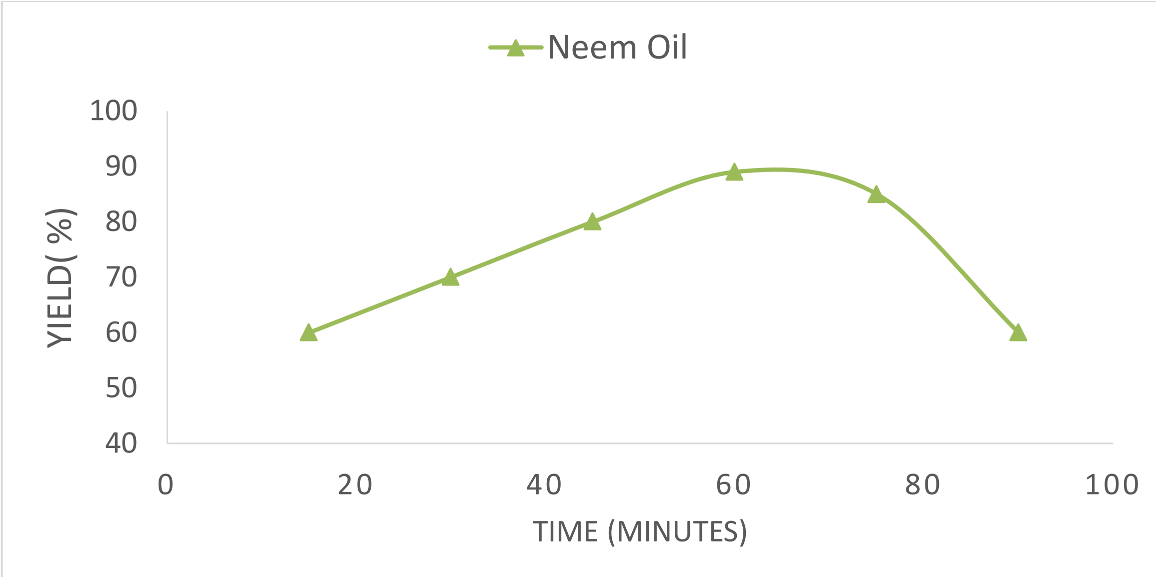

Figure 6: Effect of reaction time on biodiesel yield

Figures 12 shows the interaction effect between reaction temperature and catalyst concentration on FAME yield from neem oil. The figure indicated that the yield of FAME increases with reaction temperature and catalyst concentration. This may be as a result of the fact that more rapid reaction rate was obtained at high temperatures and high concentration of the catalyst which improved the yield. However, at higher catalyst concentration and reaction temperature above the boiling point of the alcohol , a decrease in the yield was observed due to evaporation of methanol at higher temperature and the fact that the quadratic terms of the two factors are more significant with a negative effect for yield of FAME from neem oil.

Effects of agitation speed on biodiesel yield.

In order to study the effect of agitation speed on the yield of neem biodiesel, agitation speed was varied from 150rpm to 400rpm while keeping the other parameters of methanol to oil molar ratio, catalyst concentration, reaction time and agitation speed were kept constant as shown in figure 7. Agitation is particularly important during transesterification in order to ensure homogeneity within the reaction mixture. From the figure it could be observed that neem biodiesel gradually increased with increase of agitation speed until an optimal value was attained at 250rpm when the yield starts decreasing. The decrease in yield on exceeding the optimal agitation speed of 250rpm may be explained by the fact that the backward reaction may have been favored when mixing intensity was went beyond the optimal value of 250rpm.thereby retarding the formation of biodiesel. These results are in conformity with observations made by Ogunsuyi [24], who studied the effect of agitation speed on the transesterification of non-edible oil and concluded that higher agitation promoted the homogenization of the reactants and thus led to higher yield.

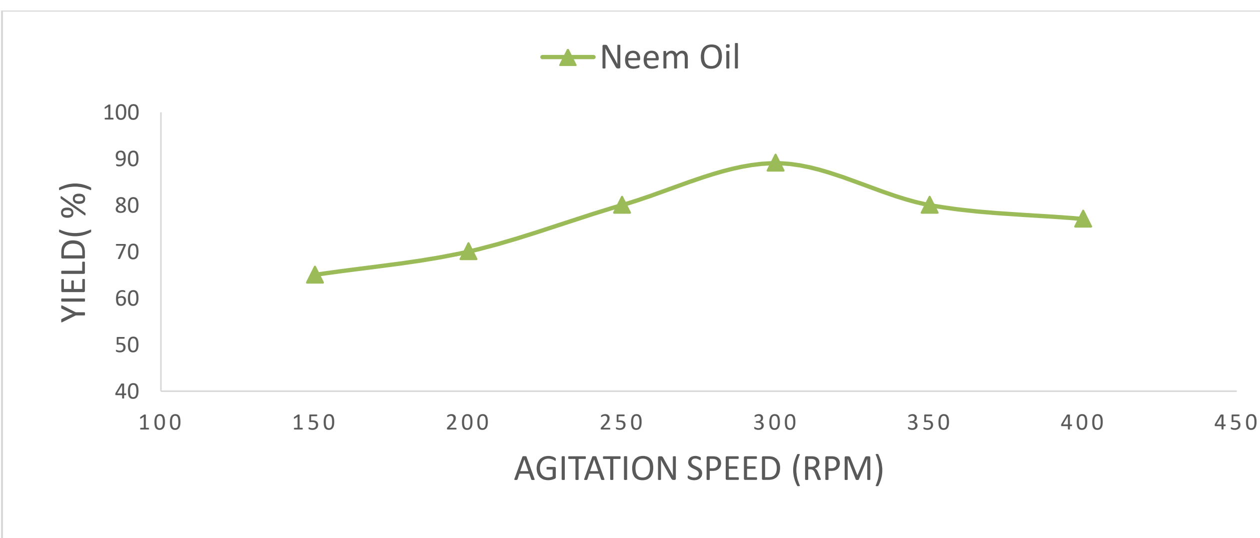

Figure 7: Effect of agitation speed on biodiesel yield.

F Fuel Properties of the FAME Produced.

Table 5 gives the summary of all the fuel properties studied in the course of this research work.

Biodiesel generally has a higher density than petro-diesel. This has a significant impact on fuel consumption as fuel introduced into the combustion chamber is determined volumetrically. The densities were of neem biodiesel was evaluated to be 870 kg/m^3, which is within the ASTM limits for biodiesel. The biodiesel has lower density compared to the density of neem (984 kg/m^3).The value of kinematic viscosity obtained for the biodiesel produced from neem oil is 4.97mm^2/s as shown in table 5, and it could be observed that it is within the standard range of ASTM limit. The increase in viscosity results in poor atomization, incomplete combustion which leads to coking of injector tips and engine power loss. Low-viscosity fuel produces a very subtle spray which cannot properly get into the combustion cylinder, thus forming the fuel-rich zone that give rise to formation of sooth [48],[49]. From the result it could be inferred that FAME from neem oil has a good injection and atomization performance. Furthermore it will offer superior lubrication to the moving parts of engine.

The flash point is a determinant for flammability classification of materials. The typical flash point of pure methyl ester is ≥130^{0}C, classifying them as "non-flammable". However, during production and purification of biodiesel, not all the methanol may be removed, making the fuel flammable and dangerous to handle and store if the flash point falls below 130^{0}C. The flash point of the neem ester is 160^{0}C. This falls within the ASTM standard as shown in table 5, indicative of its safety in handling and storage.

Cetane number serves as a measure of ignition quality of the fuel. This is the most pronounced change from vegetable oil to the transesterified product. Fuels with low cetane number show an increase in emission due to incomplete combustion. The lower limit for cetane number by ASTM and EN standards are 47 and 51 respectively. The value obtained for the neem oil biodiesel is 60.58. Thus the obtained results which are within the acceptable ASTM limits indicates that the produced biodiesel possess good ignition response. The acid value, saponification number and calorific value obtained for the neem seed oil is 0.420mgKOH/g, 200.72mgKOH/g and 38MJ/Kg respectively.

The cloud point which is the lowest temperature of first appearance of wax-like material on cooling the biodiesel was determined as 8^{0}C for neem oil biodiesel. The pour point which is the lowest temperature at which the fuel will still pour was determined as 4^{0}C for neem oil biodiesel. The properties of the biodiesel produced are within the ASTM limit for biodiesel, as shown in table 5. The cloud and pour points might give rise to cold flow problems in cold season. This problem however could be overcome by the addition of suitable cloud and pour point depressants or by blending with diesel oil [50].

Table 5: Fuel properties of NSOFAME

Properties	**Unit**	**NSOFAME**	**ASTM Standards**
Acid vlue	mgKOH/g	0.420	0.50
Density	Kg/m3	870	860-900
Kinematic viscosity	mm^{2}s^{-1} at 40^{0}C	4.92	1.9-6
FIre point	^{0}C	163	197
Flash point	^{0}C	160	100-170
Cloud point	^{0}C	8	-3-15
Cetane number		60.58	48-65
Refractive index		1.4462	1.38
Specific gravity	Kgm^{-3}	0.870	0.860-0.900
Calorific value	MJ/Kg	38	42.06
Pour point	^{0}C	4	0.5
Iodine value	gI$_2$/100g oil	70.2	42-46

Comparison of NSOFAME with diesel (CI engine) Fuel

Table 6 gave a comparison between the properties of the neem oil biodiesel produced and compression ignition (diesel) fuel. From the obtained property values in the table, it could be seen that the neem oil biodiesel properties approximate that of diesel or compression ignition engine fuel. A major difference lies with the cloud point and pour point of which the value for neem oil biodiesel are higher and therefore unsuitable for use in cold weather. However these values could be improved by the use of cloud point and pour point depressants

Table 6: Comparison between NSOFAME and CI engine (Diesel) Fuel

Property	Unit	NSOFAME	Diesel	Test Method
Density	Kgm^{-3}	870	850	D93
Kinematic Viscosity	mm^{2}s^{-1} at 40^{0}C	4.92	1.9-4.1	D445
Cetane number	^{0}C	60.58	40	D613
Flash point	^{0}C	160	100	D93
Cloud point	^{0}C	8	Varies	D2500

Water & sediments	%	0.60	0.5	D2209
Acid value	mgKOH/g	0.420		D664

F Statistical Analysis of Transesterification Using Central Composite Design (CCD)

To optimize transesterification of neem oil, central composite design (CCD), a response surface methodology (RSM) was used to determine the optimum values of the process variables. Fractional factorial design was used to obtain a quadratic model, consisting of factorial trials to estimate quadratic effects. To examine the combined effect of the five different factors; catalyst concentration, methanol to oil molar ratio, reaction temperature, reaction time and agitation speed, on biodiesel yield and derive a model, a two-level- five –factor (2^{5-1} + 2*5 + 6) central composite response design = 32 experiments were performed. The factor levels are shown in table 1. The matrix for the five variables was varied at two levels (-1 and +1). The lower level of variable was designated as "-1" and higher level as "+1". The experiments were performed in random order to avoid systematic error. Equations 2 and 3 represent the mathematical model relating the transesterification reaction of neem oil with the independent process variables obtained with the Design Expert 12. The design of experimental matrix for transesterification of neem oil and the experimental values of the biodiesel yield are presented in table 6. The response was expressed as % yield, calculated as $\{(V_b)/V_o\}$ x 100, where V_o is the initial volume of oil and V_b is the volume of biodiesel produced.

G Transesterification of neem oil.

The design plan as shown in table 2 was used to optimize the yield of biodiesel production from neem oil. The coded and un-coded values of the test variables were used to optimize the variables namely catalyst concentration, methanol to oil molar ratio, reaction temperature, reaction time and agitation speed. The experimental values of percentage yield were presented in table 7. The empirical relationship between yield (Y) and five variables in coded values obtained by using the statistical package design-expert 12 for determining the levels of factors which gives optimum percentage yield is given by equation (2), a quadratic regression equation that fitted the data:

$$Y = 84.95 + 6.67A + 6.92B + 2.17C + 5.58D + 1.67E - 0.6250AB + 0.625AC - 0.3750AD + 1.63AE + 1.87BC + 1.12BD + 0.6250BE + 0.6250CD + 0.1250CE + 0.3750DE - 16.08A^2 - 5.95B^2 - 1.58C^2 - 4.20D^2 - 2.08E^2 \quad (2)$$

Where $Y_{FAME(Neem)}$ is the response of the variables (percentage yield of FAME) and A-E are the coded values of the independent variables. The above equation represents the quantitative effect of the factors (A, B, C, D, and E) upon the response (Y). Equation (2) suggested that the yield of FAME has linear and quadratic effects on the five variables studied. Coefficients with one factor represent the single effect of that particular factor while the coefficients with more than one factor represent the interaction between those factors. Positive sign in front of the terms indicates synergistic effect while negative sign indicates antagonistic effect of the factors. The adequacy of the above proposed model was tested using the Design Expert sequential model sum of squares and the model test statistics. From the statistical analysis, the regression coefficient (R^2 = 0.99) is reasonable , and the predicted R^2 of 0.9985 is in a reasonable agreement with the adjusted R^2 of 0.9988. This test result is shown in table 7.

Table 7; Experimental Design Matrix for Factorial Design of FAME Produced from Neem oil

Run order	Methanol/Oil molar ratio X1		Catalyst conc. (wt %) X2		Temperature (oC) X3		Time (Mints) X4		Agitation Speed (Rpm) X5		FAME Yield From neem oil (%)
	Coded	Real	Coded	Real	Coded	Real	Coded	Real	Coded	Real	
1	-1	4	-1	0.5	-1	55	-1	45	+1	300	36
2	+1	8	-1	0.5	-1	55	-1	45	-1	200	49
3	-1	4	+1	1	-1	55	-1	45	-1	200	46
4	+1	8	+1	1	-1	55	-1	45	+1	300	61
5	-1	4	-1	0.5	+1	65	-1	45	-1	200	36
6	+1	8	-1	0.5	+1	65	-1	45	+1	300	54
7	-1	4	+1	1	+1	65	-1	45	+1	300	52
8	+1	8	+1	1	+1	65	-1	45	-1	200	62
9	-1	4	-1	0.5	-1	55	+1	75	-1	200	46
10	+1	8	-1	0.5	-1	55	+1	75	+1	300	61
11	-1	4	+1	1	-1	55	+1	75	+1	300	60
12	+1	8	+1	1	-1	55	+1	75	-1	200	65
13	-1	4	-1	0.5	+1	65	+1	75	+1	300	46

14	+1	8	-1	0.5	+1	65	+1	75	-1	200	58
15	-1	4	+1	1	+1	65	+1	75	-1	200	66
16	+1	8	+1	1	+1	65	+1	75	+1	300	84
17	-2	2	0	0.75	0	60	0	60	0	250	7
18	+2m	10	0	0.75	0	60	0	60	0	250	34
19	0	6	-2	0.25	0	60	0	60	0	250	47
20	0	6	+2	1.25	0	60	0	60	0	250	75
21	0	6	0	0.75	-2	50	0	60	0	250	74
22	0	6	0	0.75	+2	70	0	60	0	250	83
23	0	6	0	0.75	0	60	-2	30	0	250	57
24	0	6	0	0.75	0	60	+2	90	0	250	79
25	0	6	0	0.75	0	60	0	60	-2	150	73
26	0	6	0	0.75	0	60	0	60	+2	350	80
27	0	6	0	0.75	0	60	0	60	0	250	85
28	0	6	0	0.75	0	60	0	60	0	250	85
29	0	6	0	0.75	0	60	0	60	0	250	85
30	0	6	0	0.75	0	60	0	60	0	250	85
31	0	6	0	0.75	0	60	0	60	0	250	85
32	0	6	0	0.75	0	60	0	60	0	250	85

Table 8: Significance of regression coefficients of the yield of FAME produced from neem oil using the design-expert version 11

Source	Mean Square	Degree of freedom	Sum of square	F-value	P-value (Prob>F)
Model	584.76	20	11695.28	9330.52	< 0.0001
A	1066.67	1	1066.67	17019.78	< 0.0001
B	1148.17	1	1148.17	18320.20	< 0.0001
C	112.67	1	112.67	1797.71	< 0.0001
D	748.17	1	748.17	11937.78	< 0.0001
E	66.67	1	66.67	1063.74	< 0.0001
AB	6.25	1	6.25	99.73	< 0.0001
AC	6.25	1	6.25	99.73	< 0.0001
AD	2.25	1	2.25	35.90	< 0.0001
AE	42.25	1	42.25	674.14	< 0.0001
BC	56.25	1	56.25	897.53	< 0.0001
BD	20.25	1	20.25	323.11	< 0.0001
BE	6.25	1	6.25	99.73	< 0.0001
CD	6.25	1	6.25	99.73	< 0.0001
CE	0.2500	1	0.2500	3.99	0.0711
DE	2.25	1	2.25	35.90	< 0.0001
A^2	7584.19	1	7584.19	1.210E+05	< 0.0001
B^2	1040.06	1	1040.06	16595.25	< 0.0001
C^2	73.19	1	73.19	1167.75	< 0.0001
D^2	518.56	1	518.56	8274.18	< 0.0001
E^2	126.85	1	126.85	2024.06	< 0.0001

R^2=0.99, Predicted R^2=0.9985, Adjusted R^2=0.9988

H Analysis of variance (ANOVA) for yield of FAME from neem oil

The ANOVA results for the model terms are given in table 8. ANOVA was applied to estimate the significance of the model at 5% significance level as shown in the table. A model is considered significant if the p-value (significance probability value) is less than 0.05. From the p-values presented in tables 8, it can be stated that all the linear terms A, B, C, D and E , and interaction terms AB, AC,AD, AE, BC, BD, BE, CD, DE and quadratic terms A^2,B^2,C^2,$D^2$and $E^2$are significant model terms. Based on this, the insignificant term CE of the model was removed, and the adjusted model obtained for FAME produced from neem oil as a function of the more significant variables, as given in Eq.(3).

$$Y_{FAME(Neem)} = 84.95 + 6.67A + 6.92B + 2.17C + 5.58D + 1.67E - 0.6250AB + 0.625AC - 0.3750AD + 1.63AE + 1.87BC + 1.12BD + 0.6250BE + 0.6250CD + 0.3750DE - 16.08A^2 - 5.95B^2 - 1.58C^2 - 4.20D^2 - 2.08E^2 \quad (3)$$

Also, the adjusted model obtained for FAME produced from neem oil as a function of the more significant variables in real format, is given in Eq. (4).

$$Y_{FAME(Neem)} = 84.95 + 6.67 methanol\ molar\ ratio + 6.92 catalyst\ conc + 2.17 temperature + 5.58 time + 1.67 agitation\ speed - 0.6250 methanol\ molar\ ratio\ x\ catalyst\ conc + 0.625 methanol\ molar\ ratio\ x\ temperature - 0.3750 methanol\ molar\ ratio\ x\ time + 1.63 methanol\ molar\ ratio\ x\ agitation\ speed + 1.87 catalyst\ conc\ x\ temperature + 1.12 catalyst\ conc\ x\ time + 0.6250 catalyst\ conc\ x\ agitation\ speed + 0.6250 temperature\ x\ time + 0.3750 time\ x\ agitation\ speed - 16.08 methanol\ molar\ ratio^2 - 5.95 catalyst\ conc^2 - 1.58 temperature^2 - 4.20 time^2 - 2.08 agitation\ speed^2 \quad (4)$$

From Tables 7, it was clearly shown that among the five variables studied, catalyst concentration (B) has the largest effect on the yield of FAME from neem oil as it has the highest F-test value (18320) for single effect followed by the methano to oil molar ratio and reaction time A and D with F- test values of 17019 and 11937 respectively. Agitation speed (E) showed the most insignificant single effect as it has the lowest F-test values.

I Predicted and experimental or actual Values for yield of FAME from neem oil

Analysis was also carried out on the experimental data in table 7 to check the correlation between the experimental and predicted biodiesel yield from neem oil. The actual and predicted plot is shown in Figures 8. It could be seen from Figure 4.9 that the data points on the plot were linearly distributed, indicating a good relationship between the experimental and predicted values of the response, and that the underlying assumptions of the above analysis were appropriate. The result also suggests that the selected quadratic model was proper and adequate in predicting the response variables for the experimental data.

J Optimization of process parameters of FAME from neem oil

The optimization of process variables in this study was carried out using design expert version 12. The optimum conditions suggested by the result analysis for maximum FAME yield of 86% within the ranges studied were: methanol to oil molar ratio 8:1, catalyst concentration 0.9%wt, reaction temperature of 65^0C, reaction time of 60 minutes and agitation speed 300 rpm. Actual experiment based on the optimum conditions produced 88% yield of FAME with small percent errors of 2.3%. This percent errors of actual values compared to the predicted values indicate that the regression model developed in this study was accurate in representing the overall data and reliable in predicting the yield at any given conditions within the range studied for FAME produced from neem oil. Hence validation of result for the FAME yield is shown in table 9.

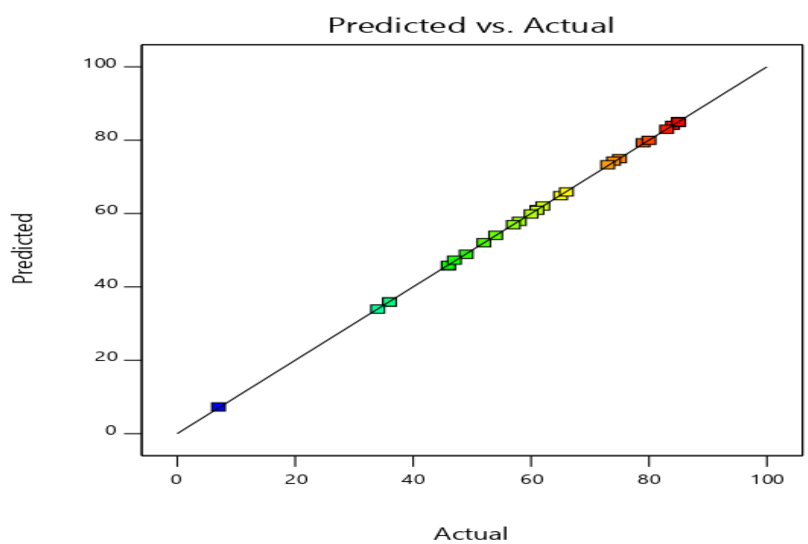

Figure 8: Plot of predicted value against the experimental values of FAME from neem oil.

Table 9: Validation of result for yield of FAME from neem oil.

Methanol/ 0il molar	Catalyst concentrat	Temperature (^{0}C)	Time (minutes)	Agitation speed	Experimen-tal FAME	Predicted FAME yield

ratio	ration (wt %)			(rpm)	yield (%)	(%)
8:1	0.9	65	60	300	88	86

K Three dimensional response surface plots for FAME yield from neem oil.

The 3D response surface plots of the different interaction effects were generated to estimate the effect of the combinations of the independent variables on the FAME yield. Figure 9 shows that the amount of methyl ester yield increases with increase in methanol to oil molar ratio and catalyst concentration. However, at higher catalyst concentrations a reduction in the yield was observed. This may be explained by the fact that high catalyst concentration has a negetive effect on the yield of FAME from neem oil as it is prone to soap formation which hinders dispersion and mixing efficiency of the reactants and reducet the amount of ester formation.

Figure10 depict the interaction effect between reaction temperature and methanol to oil molar ratio on FAME yield from neem oil. The figure indicates that the yield of FAME increases with increase in reaction temperature and methanol to oil molar ratio. This is as a result of a postive significant effect of methanol to oil molar ratio and reaction temperature. However, at higher reaction temperature above the boiling point of the alcohol used, a decrease in the yield was observed. This may be due to evaporation of methanol at higher temperature, and because the quadratic term of interaction between temperature and methanol to oil molar ratio is more significant with a negative effect .

Figures 11 shows the interaction effect between methanol/oil molar ratio and agitation speed on yield of FAME from neem oil. The figure shows that the FAME yield increases with methanol to oil molar ratio and agitation speed as a result of good homogenity , good reaction rate and presence of enough alcohol. However, at higher methanol to oil molar and agitation speed a reduction in the yield was observed . This may be due to poor contact between the reacting system, and the fact that the quadratic terms of the two factors are more significant with a negative effect on yield from neem oil.

Figures 12 shows the interaction effect between reaction temperature and catalyst concentration on FAME yield from neem oil. The figure indicated that the yield of FAME increases with reaction temperature and catalyst concentration. This may be as a result of the fact that more rapid reaction rate was obtained at high temperatures and high concentration of the catalyst which improved the yield. However, at higher catalyst concentration and reaction temperature above the boiling point of the alcohol , a decrease in the yield was observed due to evaporation of methanol at higher temperature and the fact that the quadratic terms of the two factors are more significant with a negative effect for yield of FAME from neem oil.

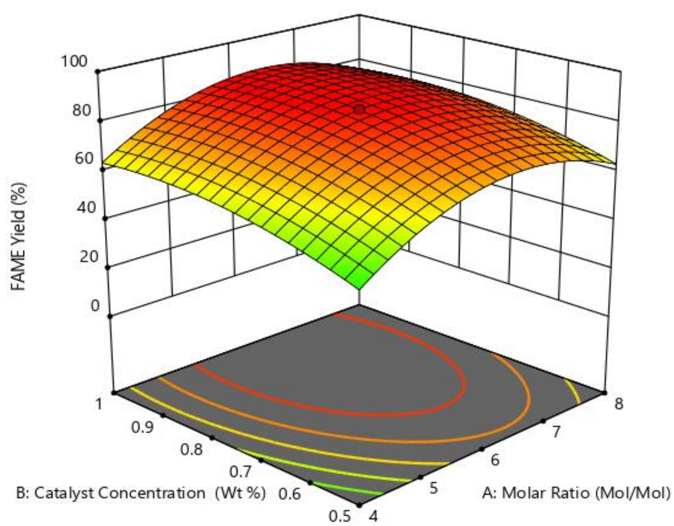

Figure 9: 3D response surface plot showing the effect of methanol/oil molar ratio and catalyst concentration on the yield of FAME from neem oil.

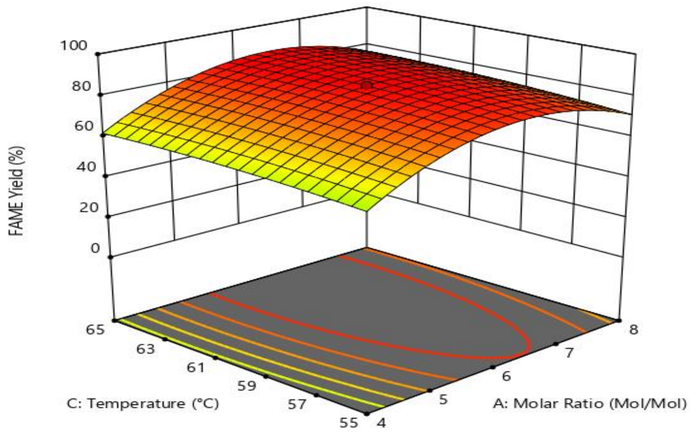

Figure 10: 3D response surface plot showing the effect of methanol/oil molar ratio and temperature on the yield of FAME from neem oil.

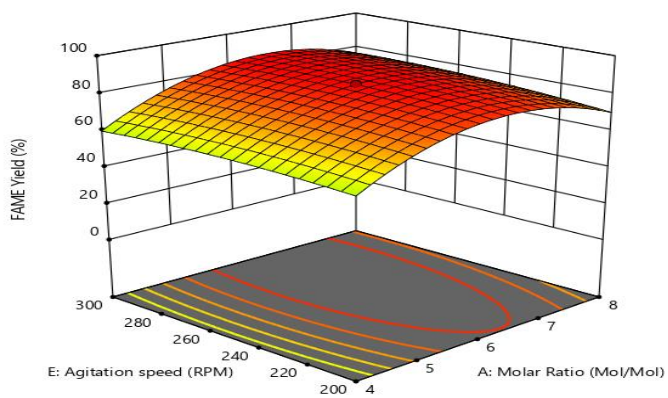

Figure 11: 3D response surface plot showing the effect of methanol/oil molar ratio and agitation speed on the yield of FAME from neem oil.

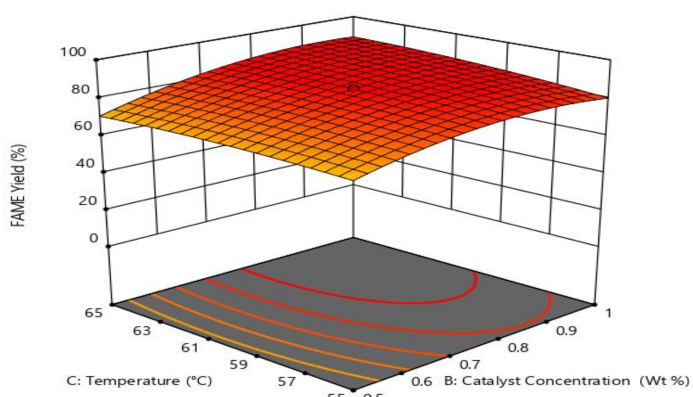

Figure 12: 3D response surface plot showing the effect of temperature and catalyst concentration on the yield of FAME from neem oil.

IV CNCLUSION

Neem seed oil biodiesel has properties that are within the ASTM limit for biodiesel. In addition the properties approximate that of the diesel and is therefore suitable for use as a compression ignition engine fuel. The additional advantages over petro-diese are that it is renewable, biodegradable, and of high cetane number and lubricity.the major set backs include, high cloud point and pour point. The transesterification parameters of methanol to oil molar ratio, catalyst concentration, reaction temperature, reaction time and agitation speedsignificantly affected the biodiesel yield as their increase resulted in increase of biodiesel yield until an optimal value is reached when the yield starts deceasing.

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