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Full Length Research Paper

Production of biodiesel from crude neem oil feedstock and its emissions from internal combustion engines

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This study investigates biodiesel production using crude neem oil having high acid value, as a feedstock. The effects of some operating variables were ascertained and its combustion performance was assessed in an internal combustion engine. Due to its high acid value, the neem oil was processed via two step acid – base transesterification process. The first step reduced the acid level to <2 mgKOH/g while the second step involved direct conversion to fatty acid methyl ester using 1% NaOH as catalyst. The lowest viscosity value was used as a proxy measure to determine the extent of the reaction. The results reveal the optimum conditions for biodiesel production to be ratio 1:6 of oil to methanol and 1.5 h reaction time. The viscosity at this condition was 5.53 cSt. The same procedure was repeated for $NaOCH_3$ catalyst concentrations of 0.5, 0.75, 1 and 1.25%. The lowest viscosity of 6.79 cSt was recorded at both 1 and 1.25% catalyst concentrations. The fuel properties of the biodiesel compared favorably with the recommendation by the American Standard Testing Method. The emissions of different blends showed that neem biodiesel has lower emissions of CO and NO than petrol diesel but higher NO_X. Thus, neem oil as non-edible oil can be a good renewable raw material for biodiesel production.

Key words: Neem, biodiesel, internal combustion, transesterification, free fatty acid.

INTRODUCTION

Fossil fuels are non-renewable energy resources. Although, these fuels are contributing largely to the world energy supply, their production and use have raised environmental concerns and political debates (Wang et al., 2011). It has been shown that 98% of carbon emissions result from fossil fuel combustion (Balat and Balat, 2010). According to Shay (1993), the continued and increasing use of petroleum diesel intensifies air pollution and magnifies the global warming problem caused by CO_2 and other particulate matters. The growing demand for fuel and the increasing concern for the environment due to the use of fossil fuel have led to the increasing popularity of biodiesel as a useful alternative and environmentally friendly energy resource (Ma and Hanna, 1999). It advantages over petroleum diesel viz: its safety, renewability and biodegradability have been reviewed widely (Demirbas, 2007; Fukuda et al., 2001; Knothe, 2006). Although, both have similar properties and performance parameters (Table 1), its non-sulfur content makes it a preferred alternative.

Biodiesel fuel is presently attracting increasing attention worldwide as a blending component or a direct replacement for diesel fuel in vehicle engines (Demirba, 2009). When blended with diesel fuel the designation BXX indicates the XX% amount of biodiesel in the blend,

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Table 1. Petroleum diesel versus biodiesel[a].

Fuel Property	Diesel	Biodiesel
Fuel standard	ASTM D975	ASTM D6751
Fuel composition	C10-C21 HC[b]	C12-C22 FAME[c]
Kinematic viscosity, mm^2/s (at 40 °C)	1.3 – 4.1	1.9 – 6.0
Specific gravity	0.85	0.88
Boiling point, °C	188–343	182 – 338
Flash point, °C	60 – 80	100 –170
Cloud point, °C	-15 to 5	-3 to 12
Pour point, °C	-35 to –15	-15 to 10
Cetane number (ignition quality)	40 – 55	48 – 65
Stoichiometric air/fuel ratio (AFR)	15	13.8

[a](Kiss et al., 2008); HC[b], Hydrocarbons; FAME[c], fatty acid methyl esters.

for example B30 is 30% biodiesel and 70% diesel. It has been reported that blends up to B20 can be used in nearly all diesel equipment and are compatible with most storage and distribution equipment (Balat, 2011). These low-level blends generally do not require any engine modifications. However, higher blends or B100 can be used in many engines built with little or no modification (Demirbas, 2007). The presence of oxygen in biodiesel (≈10%) improves combustion and reduces CO, soot, and hydrocarbon emissions while slightly increasing the NO_x emissions. Fukuda et al. (2001) showed that using B20 in trucks and buses would completely eliminate the black smoke released during acceleration.

Although, biodiesel is gaining popularity, more than 95% of the renewable resources used for its production are edible oils (Gui et al., 2008), which will in a long term have serious implications on food availability and the cost of biodiesel as it may be more expensive than petroleum diesel. Current studies on this subject have focused on the use of non-edible oils from plants with the view to addressing the aforementioned concerns. There are vast numbers of non-edible oil plants in nature; these include 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 (Demirbas, 2011). *Jatropha curcas oil* plants have been widely studied with respect to biodiesel production from non-edible oils (Berchmans and Hirata, 2008; Jain and Sharma, 2010; Lu et al., 2009; Raja et al., 2011) however, limited studies exist on the neem oil plant (Muthu et al., 2010; Ragit et al., 2011).

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 hot temperature of 44°C and a low temperature of up to 4°C has been reported (Karmakar et al., 2011), and its high oil content of 39.7 to 60% (Martín et al., 2010; Narwal et al., 1997). A mature neem tree produces 30 to 50 kg fruit every year (Karmakar et al. 2011). It contains a high percentage of monounsaturated fatty acids (C16:1, C18:1), a low proportion of polyunsaturated acids (C18:2, C18:3) and a controlled amount of saturated fatty acids (C16:0, C18:0) (Wang et al., 2011). The aforementioned characteristics of neem oil plants and its fatty acid composition of the oil (Table 2) make it to be a useful renewable source for biodiesel production.

The basic reaction involved in the production of biodiesel (also called fatty acid methyl esters, FAME) is not knew and has been reviewed in the literatures (Fukuda et al., 2001; Kusdiana and Saka, 2001; Ma and Hanna, 1999; Schuchardt et al., 1998)–reaction between triglycerides with methanol in the presence of a strong base as a catalyst yielding the desired FAME and a by-product, glycerol (Patil and Deng, 2009; Tsai et al., 2007; Keera et al., 2011). It has been reported that the triglycerides used in alkaline transesterification reactions should contain not more than 1% free fatty acids (FFA), which is equivalent to 2 mg KOH/g triglyceride, otherwise saponification reaction will hinder separation of the ester from glycerine thereby reducing the yield and formation rate of FAME. Mittelbach (1996), Zhang et al. (2003) and Berrios (2007). A two step acid – base catalyzed transesterification reaction has been shown to be appropriate for biodiesel production from neem oil due to its high FFA (32.538 mg KOH/g) (Berchmans and Hirata, 2008; Wang et al. 2011; Jain and Sharma, 2010; Liu et al., 2010), the pre-treatment of the oil is critical before the alkaline transesterification.

The aim of this study was to investigate the production of biodiesel from neem oil with a view to determine its performance in Internal Combustion engine (I.C. engine). The physicochemical properties of the biodiesel produced were measured with time and the emissions released from various blends in an internal combustion were

Table 2. Fatty acid composition of Neem Oil[a].

Fatty acid	Formula	Systemic name	Structure	Wt (%)
Palmitic	$C_{16}H_{32}O_2$	Hexadecanoic	16:0	18.1
Stearic	$C_{18}H_{36}O_2$	Octadecanoic	18:0	18.1
Oleic	$C_{18}H_{34}O_2$	cis-9-Octadecenoic	18:1	44.5
Linoleic	$C_{18}H_{32}O_2$	cis-9,cis-12-Octadecedianoic	18:2	18.3
Linolenic	$C_{18}H_{30}O_2$	cis-6,cis-9,cis-12-Octadecatrienoic	18:3	0.2
Arachidic	$C_{20}H_{40}O_2$	Eicosanoic	20:0	0.8
Total Saturated Fatty Acid	37%			
Total Mono – unsaturated Fatty Acid	44.5%			
Total Poly – unsaturated Fatty Acid	18.5			

[a]Adapted from Martín et al. (2010).

determined. The results of this study would form a basis for the development of a database for biodiesel production from this feedstock, especially in countries where this feedstock is in abundance.

MATERIALS AND METHODS

The neem oil used for this study was bought from National Research Institute of Chemical Technology, Zaria. The conventional diesel fuel was purchased from Obafemi Awolowo University's petrol station in Ile-Ife. All the chemicals like Methanol, Sodium methoxide, anhydrous Calcium chloride, sulphuric acid, sodium thiosulphate etc. were analytical grades.

Two – step acid – base catalyzed transesterification

Crude neem oil when transesterified using NaOH catalyst produced a significant amount of soaps from saponification side reaction. This was due to the high level of free fatty acids and small quantity of moisture in the crude neem oil. Therefore, a two step process acid catalyzed esterification followed by alkali catalyzed transesterification was employed according to the method of Berchmans and Hirata (2008).

Acid pretreatment (acid catalyzed esterification)

The crude oil was weighed, heated at 60°C for about 10 min and mixed with 55.8 ml of methanol (60%w/w of oil). To the mixture was added 1.2%w/w of concentrated H_2SO_4.

The resulting mixture was then stirred on magnetic hot plate for 1 h at 50°C, after which it was allowed to settle for 2 h. The pre-treated oil was separated from the methanol - water phase at the top.

Base catalyzed transesterification

These laboratory scale experiments were carried out batch wise in a 250 ml conical flask containing 40 g of pretreated neem oil. These were done on a magnetic stirrer at temperature of 60°C, keeping the agitation rate constant at 400 rpm for oil to alcohol ratio of 1:4, 1:5, 1:6 and 1:7. The pretreated oil was poured into the reaction flask and heated. A solution of NaOH in methanol (1%) was dissolved at room temperature and the pretreated oil was added. The reaction was allowed to run for various periods of 0.5, 1, 1.5 and 2 h. The reaction condition of 60°C and 1% NaOH catalyst was maintained as reported previously (Aransiola et al., 2010; Muthu et al., 2010).

The resulting mixture was poured into a separating funnel and allowed to settle under gravity for 24 h for separation of biodiesel. The lower glycerol layer was tapped off after which the biodiesel layer was washed with warm water three to four times and dried over anhydrous calcium chloride. The viscosity value was used a proxy measure of the extent of reaction with reference to ASTM standard which served as an indicator of completeness of the reaction. This approach has been used in the literatures (Aransiola et al., 2010; Belewu et al., 2010; Fukuda et al., 2001). This procedure was repeated using sodium methoxide with a view to explore the effect of other readily available catalyst on biodiesel yield. Different catalyst ratios of 0.5, 0.75, 1 and 1.25% were used, keeping rate of agitation, temperature and reaction time constant at 250 rpm, 60°C and 1.5 h, respectively. This variation of Sodium methoxide catalyst was carried out because it is not commonly used as Sodium hydroxide catalyst. The biodiesel produced were analyzed for the following parameters: pour point, flash points, cloud points, Specific gravity at 15°C, water content, colour, density, and kinematic viscosity at 40°C.

Analysis

Physicochemical analysis

The oil was analyzed for some physicochemical properties (iodine value, saponification value, acid value) by methods described by the association of Official Analytical Chemists (AOAC, 1984; Aransiola et al., 2010).

Table 3. The Physicochemical Properties of Neem Oil.

Property	Value	Reference value
Acid Value(mgKOH/g)	32.538	44[a]
Iodine Value	81.28	82 – 98[a]
Viscosity (cSt)	@30°C 43.75	@40°C 35.83[b]
Saponification	199.86	191 – 202[a]
Physical state at room temperature	Liquid (Golden yellow)	-
Cloud point	13	19[b]
Pour point	7.0	10[b]
Density	0.9182	0.92[b]

[a]Muthu et al. (2010); b Ragit et al.(2011).

Characterization of biodiesel

The characterization of the biodiesel was carried out according to the methods used by Aransiola et al. (2010). The density and the viscosity were measured at room temperature using the density bottle and the Brooke auto viscometer (DV-I PRIME, Brookfield, USA), respectively. The parameters are determined with the standard methods.

Investigation of biodiesel on internal combustion engine

The investigations on the combustion characteristics of the conventional diesel, neem methyl ester (B100) and its blends were conducted on a single cylinder one-stroke 165F jet diesel engine having a rated output of 3.23 kW at 2600 rpm. This was fueled with prepared test fuels. The emissions (CO, NO_x and NO) were measured through an automatic EGA4 palm top flue gas analyzer having Ni-MH rechargeable battery.

RESULTS AND DISCUSSION

The physical and chemical properties of the neem oil are presented in Table 3. These properties compared favourably with literature values (Muthu et al., 2010; Ragit et al., 2011). The relative degree of unsaturation of a fat or oil is determined by its iodine number. The results suggest that neem oil possess certain degree of unsaturation as reported by Martín (2010). An iodine number of 81.28 obtained in this study falls within the range of 30 (reported for butter which is known to be saturated) and 101 for corn oil – a polyunsaturated feedstock (Ramos et al., 2009). It should be noted that the higher the number of double bonds, the higher the iodine number. As discussed earlier, oils high in monounsaturation level are good sources of biodiesel production. The high saponification and high acid values found in neem oil are common to most non edible oils used for biodiesel production (Veljkovic et al., 2006; Zhang and Jiang, 2008). These results suggest neem oil as an ideal choice for biodiesel production. After acid pretreatment, the oil acid value reduced to less than 2 mgKOH/g and its color became lighter. This makes it fit for the alkali transesterification process (Berrios et al., 2007; Ghadge and Raheman, 2006; Veljkovic et al., 2006; Wang et al., 2011). The viscosity of the oil reduced after transesterifcation from 43.75 to 5.53 cSt likewise the density from 0.9182 to 0.8762 g/ml, which is acceptable as per ASTM recommendation for biodiesel.

Effect of reaction time and oil to alcohol ratio on the transesterification of neem oil

Base catalyzed transesterification is preferred over acid catalyzed transesterification reactions for the production of biodiesel at industrial level because it provides better conversion rates and efficiencies (Fukuda et al., 2001). The basic parameter reflecting the extent of the reaction could be the viscosity, since it is directly related with the fatty acid methyl ester (FAME) content of the product and is one of the specifications to comply with in biodiesel production (Shu et al., 2007). In this study therefore, the effectiveness and completeness of the ester conversion process is shown as the variation of the viscosity with reaction time at various oil to alcohol ratio investigated (Table 4). The viscosity of the neem methyl ester decreases as the oil to alcohol ratio and reaction time are increasing. Aransiola et al. (2010) investigated the transesterification of soybean oil and showed that viscosity decreases with increasing reaction time and temperature. Similar study on biodiesel production fromused vegetable oil also revealed that viscosity of the product obtained diminishes with reaction temperature and also decreases with increased space time in the reactor (Brito et al., 2007). The lowest viscosity obtained in this study was 5.51 cSt at oil-to-alcohol ratios of 1:6 and 1:7 and reaction times of 1.5 h and 2 h. From economic point of view, ratio 1:6 is preferred to 1:7 as it minimizes amount of methanol used in the process. On

Table 4. Effect of reaction time and oil to alcohol ratio on kinematic viscosity of the ransesterification of neem oil.

Time (h)	Viscosity (cSt)			
Oil:Methanol ratio	**1:4**	**1:5**	**1:6**	**1:7**
0.5	5.7	5.62	5.56	5.55
1	5.65	5.58	5.54	5.53
1.5	5.6	5.54	5.53	5.51
2	5.58	5.53	5.51	5.51

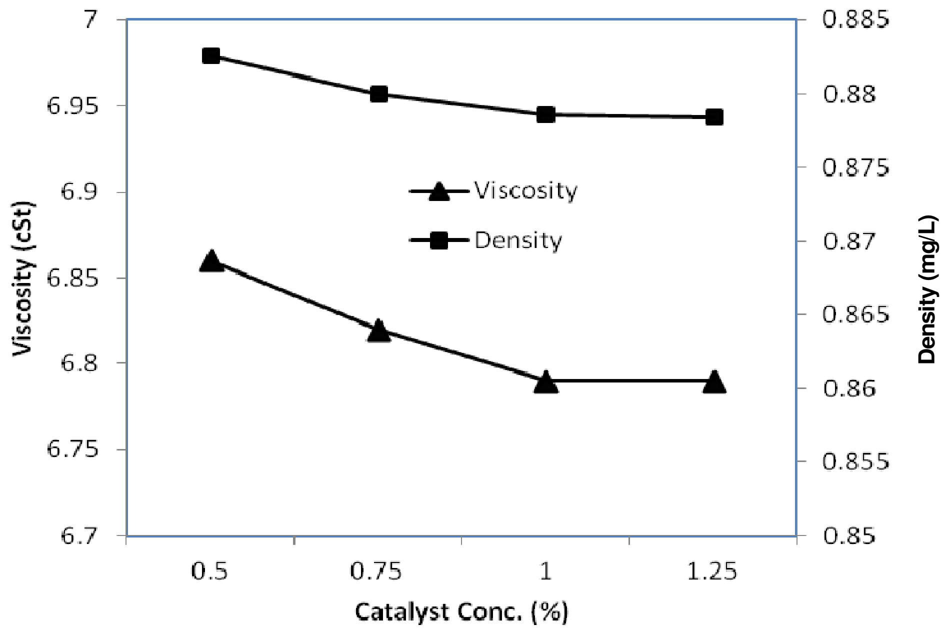

Figure 1. Variation of viscosity and density with sodium methoxide catalyst concentration.

the other hand, the reaction time of 1.5 h is preferred to 2 h since there is no significant difference in the biodiesel parameters (Figures 2 and 3) beyond 1.5 h. Although it may be argued that the reaction completed within 30 min due to comparatively similar viscosity values; it has been shown previously that the FAME yield increased significantly for reaction time greater than 1 h (Aransiola, 2010). For this study therefore, the optimum oil to alcohol ratio and reaction time are taken to be 1:6 and 1.5 h, respectively. This is in agreement with the literature (Balat and Balat, 2010; Fukuda et al., 2001; Lu et al., 2009; Ragit et al., 2011; Wang et al., 2011).

Effect of catalyst variation using sodium methoxide on the density and viscosity of the neem methyl ester

The results of the viscosity and density of neem methyl ester using sodium methoxide concentration are presented in Figure 1. This was conducted at the optimum conditions obtained previously. The results show that as the percentage concentration of the sodium methoxide catalyst increases, there is corresponding decrease in the values of both density and viscosity. The lowest viscosity of 6.79 cSt was recorded at both 1 and 1.25% catalyst concentrations while the lowest density of 878.4 g/l was recorded at 1.25% catalyst concentrations but the density value at 1% catalyst concentration was also very close (878.6 g/l).

The result shows that biodiesel produced using NaOH catalyst (5.53 cSt) is better than that obtained using sodium methoxide at the same operating conditions. A drop of the biodiesel from NaOH catalyst is smaller than that from methoxide catalyst as a smaller amount of the fuel is desired in engine combustion chamber for complete combustion to occur.

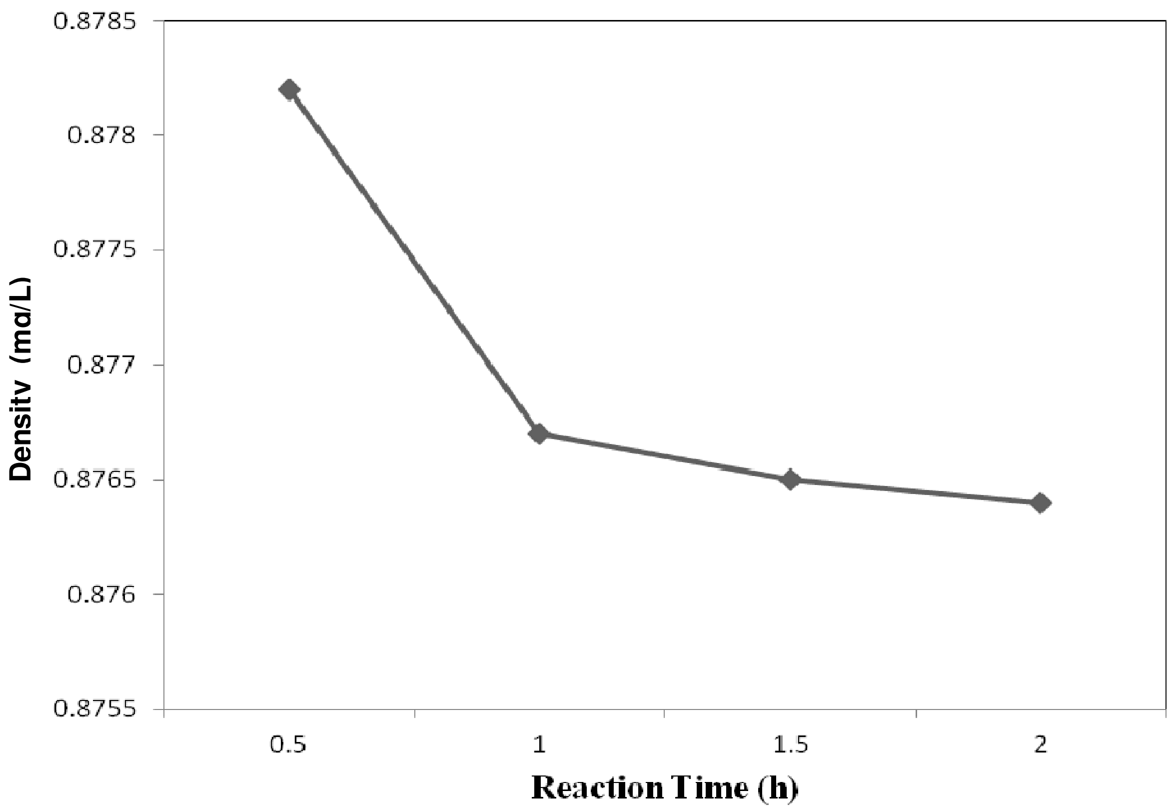

Figure 2. Variation of density using sodium hydroxide catalyst with reaction time.

Fuel properties of the neem methyl ester

The density, cloud point and flash point of the neem methyl ester using sodium hydroxide as the catalyst at the optimum conditions (Temperature of 60°C and oil to alcohol ratio of 1:6) were studied with reaction time. These are shown in Figures 2 and 3. Both flash and cloud points decreased significantly with increase in the reaction time, however, beyond the reaction time of 1.5 h these parameters appear to be constant. Therefore, it is reasonable to terminate the transesterification reaction at 1.5 h. Fuels above flash point of 66°C are considered safe fuel (Raja et al., 2011) and are suitable for all climatic conditions, while fuels with higher cloud points can affect engine performance and emission adversely under cold climatic conditions. The fuel properties obtained at the reaction condition is in agreement with the ASTM biodiesel standard. The density, cloud point and flash point at the optimum conditions at reaction time of 1.5 h are 0.8765 g/ml, 6.0 and 150°C, respectively. Other fuel properties like colour, water content and pour point were characterized at the optimum conditions to be 1.0, 0 and 3.0, respectively. These properties compared favourably with the acceptable biodiesel standards and are presented in Table 5.

Investigation of biodiesel on internal combustion engines

Experimental investigation was carried out for different blends of neem oil biodiesel and the emissions (CO, NO, NO_x) were evaluated and compared with conventional diesel. The results for the various emission data generated are presented in Figure 4.

The results show that biodiesel gave a less carbon monoxide (CO) compared to petroleum diesel. The emissions of CO and NO were found to be decreasing with biodiesel blends. The minimum and maximum reduction of CO was 8.32 and 41.32%, while that of NO was 14.51 and 37.74% respectively as compared to diesel. The emissions of NO_x increased with the biodiesel blends.

The percentage increment between B100 and diesel was 10.74%. Similar trends of observations on CO, NO and NOx production were also reported while running the diesel engines with karanja, neem, Honge, Jatropha and sesame oil methyl esters (Banapurmath and Tewari,

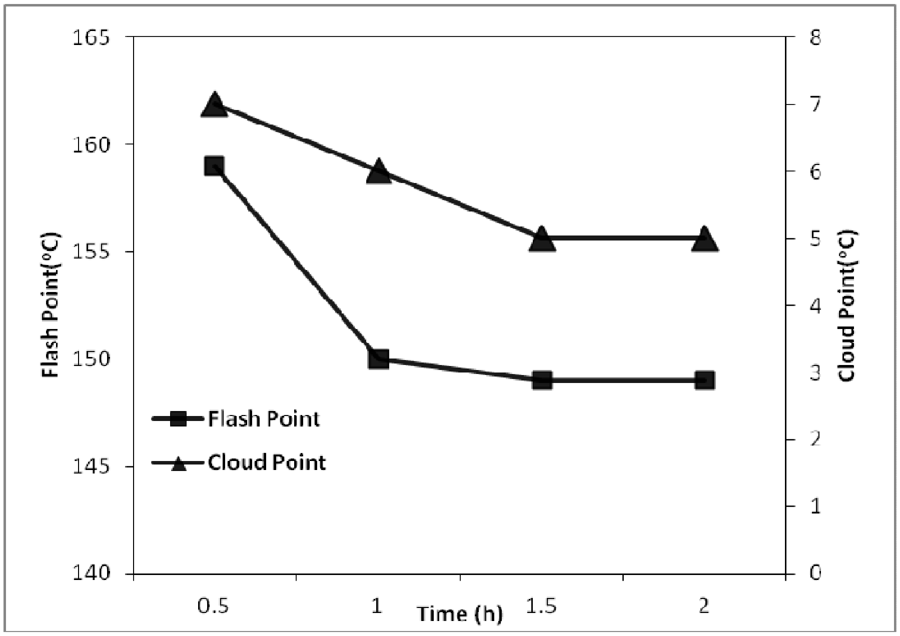

Figure 3. Variation of Flash Point and Cloud Point with Reaction Time using Sodium hydroxide Catalyst.

Table 5. Fuel properties of neem oil biodiesel.

Property	Neem oil biodiesel	Biodiesel standard	Test method
Flash point (°C)	150	130 (min)	ASTMD – 93
Moisture content	Nil	0.050 max	ASTMD – 2709
Kinematic viscosity	5.53	1.9 – 6.0	ASTMD – 445
Cloud Point (°C)	6	-	ASTMD – 2500
Specific gravity at 15/15°C	0.8762	0.860 – 0.900	-
Pour point (°C)	3	-	ASTMD – 97

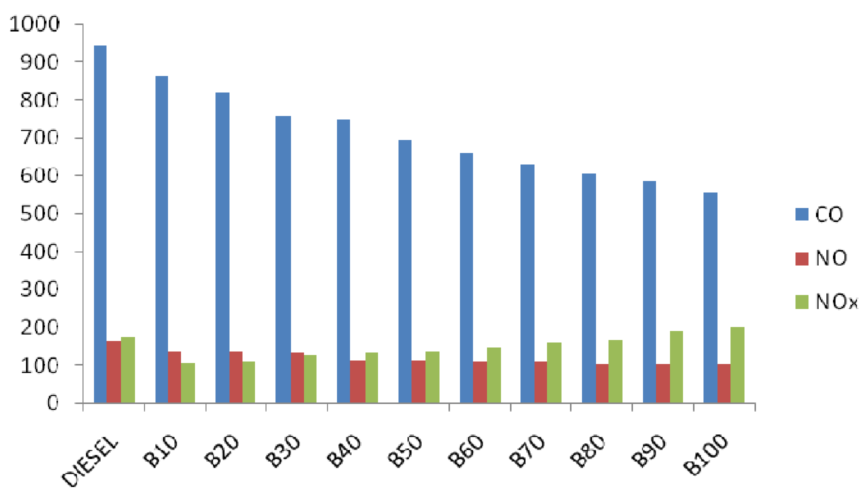

Figure 4. The plot of different blends of biodiesel and diesel against CO, NO and NO_x emissions.

2009; Nabi, 2006; Raheman and Phadatare, 2004).

Conclusion

In this study, production of biodiesel from crude neem oil with high acid value was investigated. The effect of some operating variables were investigated and it performance was assessed in an internal combustion engine. The fatty acid methyl ester from neem oil was produced by two-step acid – base transesterification process. The first step reduced the acid level to less than 2 mgKOH/g. The second step converted it to fatty acid methyl ester using 1% NaOH catalyst. Viscosity of 5.53 cSt was obtained at the optimum oil to alcohol ratio and reaction time of 1:6 and 1.5 h, respectively. When sodium methoxide catalyst was used, the lowest viscosity of 6.79 cSt was recorded at both 1 and 1.25% catalyst concentrations while the lowest density of 878.4 g/l was recorded at 1.25% catalyst concentrations. The emissions of CO and NO decreased with the biodiesel blends while that of NO_X increased. The properties of the biodiesel produced in this study compared favourably with the values prescribed by American Standard Testing Method. The results obtained in this study would be relevant in the development of biodiesel production from neem oil especially in countries where this feedstock is abundant.

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