



Studies on optimization of transesterification of certain oils to produce biodiesel

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ARTICLE INFO

Article type:

Research article

Article history:

Received June 2015

Accepted September 2015

April 2016 issue

Keywords:

Azadirachta indica

Biodiesel

Pongamia pinnata

GC-MS

FT-IR

Transesterification

ABSTRACT

The oil seed production in the country presently meets only 60-70% of its total edible oil requirements and the rest is met through imports. India also has a potential of collecting 5 million tons of tree-borne oilseeds (TBO) of which only one million tons are being collected presently. The consumption of edible oil is very high in the country and still the indigenous production does not meet the demand and considerable amount of edible oil is imported and it is therefore, not advisable to divert these sources for biodiesel production. On the other hand, the non-edible oil resources can be a solution for biodiesel production. Non-edible oil from the plant seeds is the most promising alternative fuel for internal combustion engine because it is renewable, environment friendly, non-toxic, biodegradable has no sulphur and aromatics, has favourable combustion value and higher cetane number. Extensive work has been done on the transesterification of non-edible oils; however, no significant work has been done on the optimization of transesterification process, oil characterization and fuel analysis of most of the non-edible seed oils. In the present work, optimization of transesterification process and analysis of biodiesel from non-edible oil was done; based on optimized protocol for biodiesel production from non-edible oilseeds of Neem and Pongamia converted into fatty acid methyl esters (FAME) through base catalyzed transesterification using an optimum ratio of 1:6 (Oil : Methanol) at 60°C. Biodiesel from these sources was analyzed for qualitative and quantitative characterization by using, GC-MS and FT-IR techniques. Based on qualitative and quantitative analysis of biodiesel, it is concluded that the biodiesel from these species can be feasible, cost effective and environment friendly.

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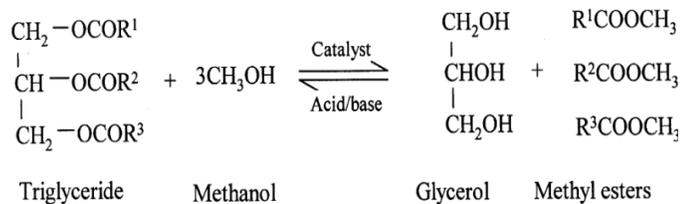
Capsule Summary: Biodiesel production from seed oils of Neem and pongamia was investigated using base catalyzed transesterification process. The process variable molar ratio 6:1 of Methanol and oil, 0.7 g NaOH, 60 °C reaction temperature and 2 h reaction time showed good biodiesel yield.

Cite This Article As: R. Gangadhara and N. B. L. Prasad. 2016. Studies on optimization of transesterification of certain oils to produce biodiesel. Chemistry International 2(2) 59-69

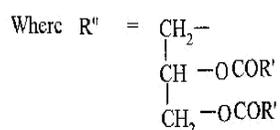
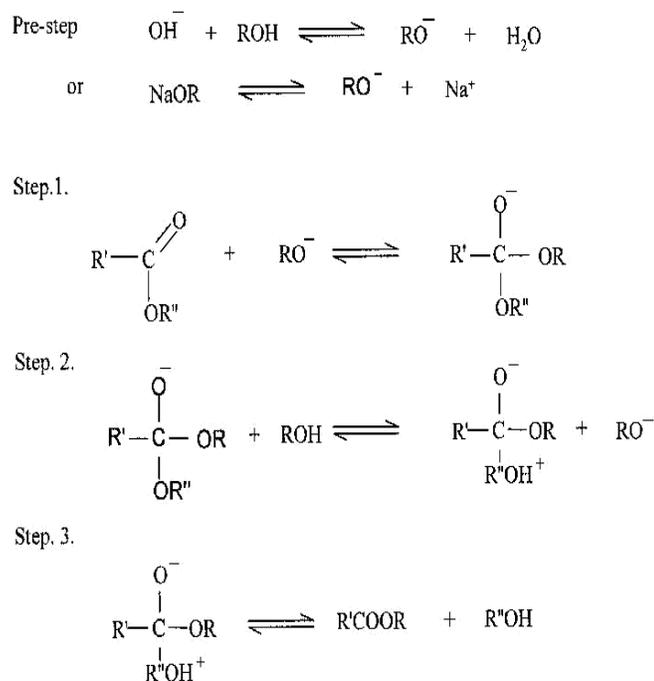
INTRODUCTION

India occupies second place in population and 7th place in area in the World oil production (Abolle et al., 2009). Due to large population and the need of transportation made India top 5th

country in the World in consumption of petroleum products. Yearly consumption of diesel in India is approximately 40 million tones, which constitutes about 40% of the total petro-products consumption. As these carbon sources are limited and the consumption of petro-products is increasing day by day there is a need of alternative resources, which includes solar energy,



Scheme 1: General chemical reaction for methanolysis of triglycerides



$\text{R}' =$ Carbon chain of fatty acid

$\text{R} =$ Alkyl group of alcohol

Scheme 2: Mechanism of transesterification process

thermal energy, hydro energy and bio-energy (Jain and Sharma, 2010). One of the most prominent alternative energy resources, attracting more and more interest in recent years with the price for crude oil reaching record heights, is biodiesel, which is a possible substitute for petroleum-based diesel fuel. Production of biodiesel using plant sources is good alternative resource through which we can meet the demand for petroleum products. Biodiesel is an alternative to diesel which is made from renewable resources such as vegetable oils (or) animal fats. In transesterification reaction base chemical catalyst processes are

more practical compared with the enzymatic method. Alkali process can achieve high purity and yield of biodiesel product in a short time (Dorado et al., 2004; Meher et al., 2006a; Tiwari et al., 2007). Methyl esters are the product of transesterification of vegetable oils with alcohol (methanol) using an alkaline catalyst. In addition, the process yields glycerol which has great applications in the pharmaceutical, food and plastics industries (Bouaid et al., 2005; Meher et al., 2006b; Srivastava et al., 2000).

Biodiesel offers a number of interesting and attractive beneficial properties compared to conventional petroleum-based diesel. Most important, the use of biodiesel maintains a balanced carbon dioxide cycle since it is based on renewable biological materials. Additional environmental benefits are reduced emissions (carbon monoxide, sulphur, aromatic hydrocarbons, soot particles) during combustion. Biodiesel is non-toxic and completely biodegradable. Due to its high flash point, it is of low flammability and thus its use is very safe and non-hazardous. Furthermore, it provides good lubrication properties, thereby reducing wear and tear on engines (Baroutian et al., 2010).

Purpose of the present study is to optimize the processing parameters for improved production of biodiesel by using transesterification process. Pongamia oil and Neem oil are the raw materials with methanol and sodium hydroxide as the catalyst and to evaluate the produced biodiesel as a fuel.

MATERIAL AND METHODS

Experimental work

The seeds of Neem and Pongamia were used as raw material for biodiesel production. Seeds of these plants were expelled by using electric oil expeller (KEK P0015-10127), India. Methanol 99.9% purity, sodium hydroxide (NaOH) and anhydrous sodium sulphate (Na_2SO_4) were of analytical grade obtained from Merck (Germany).

Transesterification

Transesterification is the displacement of alcohol from an ester by another. The process is similar to hydrolysis; in this process alcohol is used instead of water. This process has been widely used to reduce the high viscosity of triglycerides. The transesterification reaction is represented in Scheme 1. Transesterification is one of the reversible reactions and proceeds essentially by mixing the reactants. However, the presence of a catalyst (NaOH) accelerates the conversion. The mechanism of alkali-catalyzed transesterification is described in Scheme 1. First step involves the attack of alkoxide ion to the carbonyl carbon of triglyceride molecule, which results in the formation of a tetrahedral intermediate. The reaction of this intermediate with an alcohol produces alkoxide ion in the second step. In the last step, rearrangement of the tetrahedral intermediate gives rise to an ester and a diglyceride. The same mechanism is applicable to di-glyceride and monoglyceride. The mechanism of transesterification process is shown in Scheme 2.

Experimental procedure

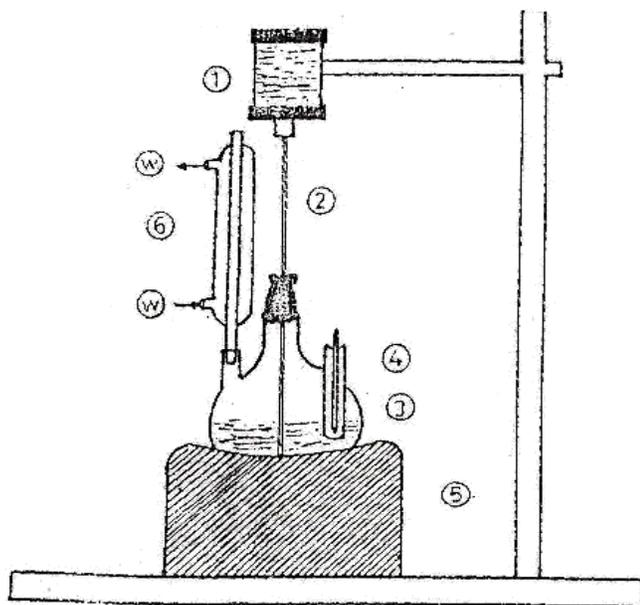


Fig. 1: Experimental setup for preparation of methyl esters from neem and pongamia oil (1-Electric Motor, 2-Stirrer, 3-Three-necked Round Bottom Flask, 4-Thermo-well with Thermometer, 5-Water bath and 6-Condenser)

A 2000 ml three-necked round-bottomed flask was used as a reactor. The flask was placed in a water bath, whose temperature could be controlled within $+ 2^{\circ}\text{C}$. One of the two side necks was equipped with a condenser and the other was used as a thermo well. A thermometer was placed in the thermo well containing little mercury for temperature measurement inside the reactor. A blade stirrer was passed through the central neck, which was connected to a motor along with speed regulator for adjusting and controlling the stirrer speed (Fig. 1).

Esterification

A known amount of Neem/Pongamia oil was taken in the above-mentioned setup. Required amount of sulphuric acid and methanol were added to the oil and stirred continuously maintaining a steady temperature of 64°C . Intermittently samples were collected at regular intervals (30 min) and acid value was determined. After the confirmation of complete reduction of acid value to less than 1.0, the heating was stopped and the products were cooled. The unreacted methanol was separated by separating funnel. The remaining product was analyzed for acid value and it was found that the acid value varied from 1.0 to 0.5. This oil sample was used for transesterification to obtain methyl esters.

TRANSESTERIFICATION

After thorough cleaning of the same setup, known amount of esterified Neem / Pongamia oil was charged. Required amount of catalyst NaOH was dissolved in methanol and the rest amount of methanol along with the catalyst solution was added to the oil sample. After proper closing of the flask it was put on the water bath. The system was maintained airtight to prevent the loss of alcohol. The reaction mixture was maintained at temperature $60-70^{\circ}\text{C}$ to speed up the reaction rate. Excess alcohol was used to ensure total conversion of the oil in to its methyl esters. The formation of methyl ester was monitored by measuring the viscosity of the reaction mixer. Reduction in viscosity confirms the formation of methyl esters. This procedure was followed for all the samples collected at regular intervals of time to check the formation of methyl esters. After the confirmation of complete formation of methyl esters, heating was stopped and the products were cooled and transferred to a separating funnel. Where, the ester layer containing mainly methyl ester and methanol and glycerol layer containing mainly glycerol and water were separated. For neutralization a known amount of sulfuric acid in methanol was added to both the layers separately to neutralize the sodium methoxide present in them. The traces of methanol present in ester layer were recovered in a distillation column under controlled vacuum. Distilled methanol was weighed and stored in sample bottle. Similar procedure was adopted to recover the traces of methanol present in glycerol layer. The methyl ester was washed and dried under vacuum to remove traces of moisture. A sample of esters was analyzed for acid value by using standard AOCS procedure. The sample of glycerol layer was analyzed for glycerol content by using AOCS procedure. The following parameters were studied in order to optimize the transesterification process.

Reaction temperature

The rate of reaction is influenced by the reaction temperature as per kinetics of reaction. Generally the reaction was conducted close to the boiling point of the methanol at the atmospheric pressure. The maximum yield of esters was observed at temperature @ 60°C . Further increase in temperature has negative effect on the conversion. Studies have also indicated that sufficient time for reaction and temperature are important parameters for the better results.

Ratio of alcohol to oil

Higher molar ratio of alcohol to vegetable oil interferes in the separation of glycerol. On the other hand, with lower molar ratio required more reaction time and conversion increases but recovery decreases. It was also found that optimum molar ratio depends upon type and quality of oils. The mixing effect is most significant during the slow rate region of the transesterification reaction. It is the most valuable tool for the transesterification process, while study the kinetics of reaction. In the reaction the different molar ratios of methanol and oil are being used.

Effect of concentration of catalyst and its type

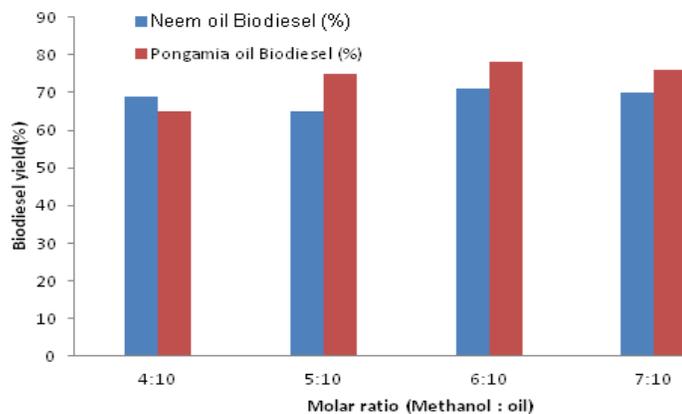


Fig. 2: Biodiesel yield with different ratios of methanol and oil

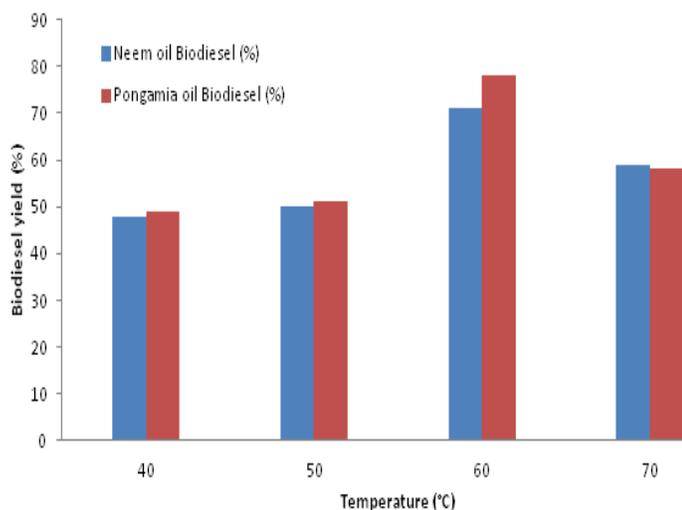


Fig. 3: Biodiesel yields of neem and pongamia with different reaction temperatures

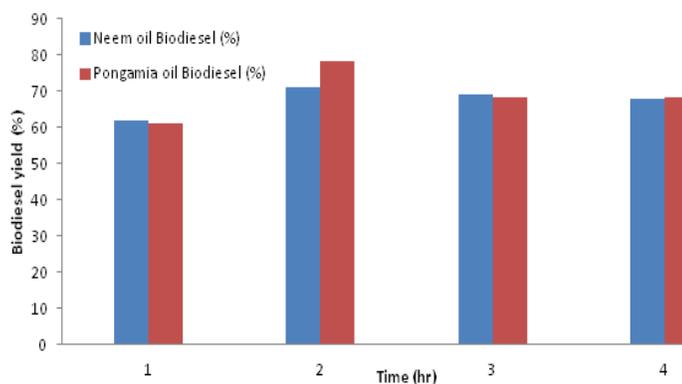


Fig. 4: Neem and Pongamia biodiesel yields with different time interval

Alkali catalysts are the most effective for transesterification compared to others. The NaOH is more frequently used as catalyst in the process than acids and enzymes. The different

amounts of catalyst give different yields of biodiesel and glycerin. The different concentrations of catalyst lead to formation of different yields of biodiesel. If the concentration of catalyst increased then yield of biodiesel decreased.

Time duration

In the present work, effect of reaction time from 60-240 minutes on reaction yield was investigated and it was found that the maximum yield of biodiesel obtained at 120 minutes afterwards the yield is decreases slightly.

Effect of stirring speed (rpm)

The effect of stirring speed of the mixture was varied from 400 rpm to 700 rpm. Based on the experimental observations it can be seen the 600 rpm stirring speed gives maximum product output, therefore this is chosen as the optimum stirring speed for the remaining experiments.

RESULTS AND DISCUSSION

Determination of free fatty acid

The FFA has significant effect on the transesterification of oils with alcohol using catalyst. The high FFA content will cause soap formation and the separation of products will be exceedingly difficult, and as a result, low yield of biodiesel product would be obtained. Hence, it is important to determine the FFA content of oil initially. The FFA of crude Neem and Pongamia oils are 2.5% and 2% respectively. According to Anggraini et al., (1999) the conversion efficiency decreases gradually, if the FFA content is more than 3%.

Experimental results by varying molar ratio of methanol and oil

The yield of methyl esters of Neem and Pongamia oils were investigated by changing molar ratios of methanol/oil (4:1, 5:1, 6:1 and 7:1). The stirring speed, reaction time and temperature and catalyst concentration were kept constant. The highest conversion rate of Neem biodiesel (71%) and Pongamia biodiesel (78.16%) obtained at 6:1 molar ratio of methanol and oil. Similarly, the yield of residual methanol is highest at 6:1 molar ratio (Table 1, Fig. 2).

Experimental results by varying reaction temperature

The yield of methyl esters of Neem and Pongamia oils were investigated by changing temperatures (40 °C, 50 °C, 60 °C and 70 °C). The stirring speed, reaction time, catalyst concentration and molar ratio of methanol/oil were kept constant. The highest conversion rate of Neem biodiesel (71%) and Pongamia biodiesel (78.16%) obtained at 60 °C. The residual methanol recovery at highest yield of Neem and Pongamia oils are 15.6% and 15.8% respectively. The yield of glycerin at highest for Neem and Pongamia oils are 9.2% and 5.5%, respectively (Table 2, Fig. 3).

Table 1: Biodiesel yield with different molar ratios of methanol and oil

Oil type	Methanol : oil ratio	Biodiesel (%)	Glycerin (%)	Residual Methanol (%)
Neem oil sample	4:1	69.2	5.1	13.2
	5:1	65.3	5.8	12.9
	6:1	71.0	7.1	14.1
	7:1	70.2	6.1	12.6
Pongamia oil sample	4:1	65.5	5.5	11.9
	5:1	75.16	7.2	14.5
	6:1	78.16	8.3	15.2
	7:1	76.10	6.5	14.8

Table 2: The yield of Biodiesel with different reaction temperatures

Name of the Bio diesel	Temperature (°C)	Biodiesel (%)	Glycerin (%)	Residual Methanol (%)
Neem oil Bio diesel	40	48	8.0	13.6
	50	50	8.1	14.3
	60	71	9.2	15.6
	70	59	6.5	16.6
Pongamia oil Bio diesel	40	49.10	6.7	14.6
	50	51.10	7.5	14.3
	60	78.16	5.5	15.8
	70	58.10	6.6	13.7

Table 3: Biodiesel yield with different time intervals (h)

Name of the biodiesel	Time (h)	Biodiesel (%)	Glycerin (%)	Residual Methanol (%)
Neem oil biodiesel	1	62	9.0	13.2
	2	71	7.10	17.8
	3	69	6.0	16.5
	4	68	7.0	15.4
Pongamia oil biodiesel	1	61	7.5	14.6
	2	78.16	4.9	9.2
	3	68.16	5.2	11.5
	4	65.70	8.3	11.2

Table 4: Biodiesel yield with different stirring speed (in rpm)

Name of the biodiesel	Velocity(rpm)	Biodiesel (%)	Glycerin (%)	Residual Methanol (%)
Neem oil biodiesel	400	63	8.15	11.1
	500	67	6.10	13.2
	600	71	7.50	16.0
	700	69	6.0	13.1
Pongamia oil biodiesel	400	68.3	6.9	12.1
	500	68.45	7.8	15.3
	600	78.16	5.6	8.6
	700	76.15	6.3	11.3

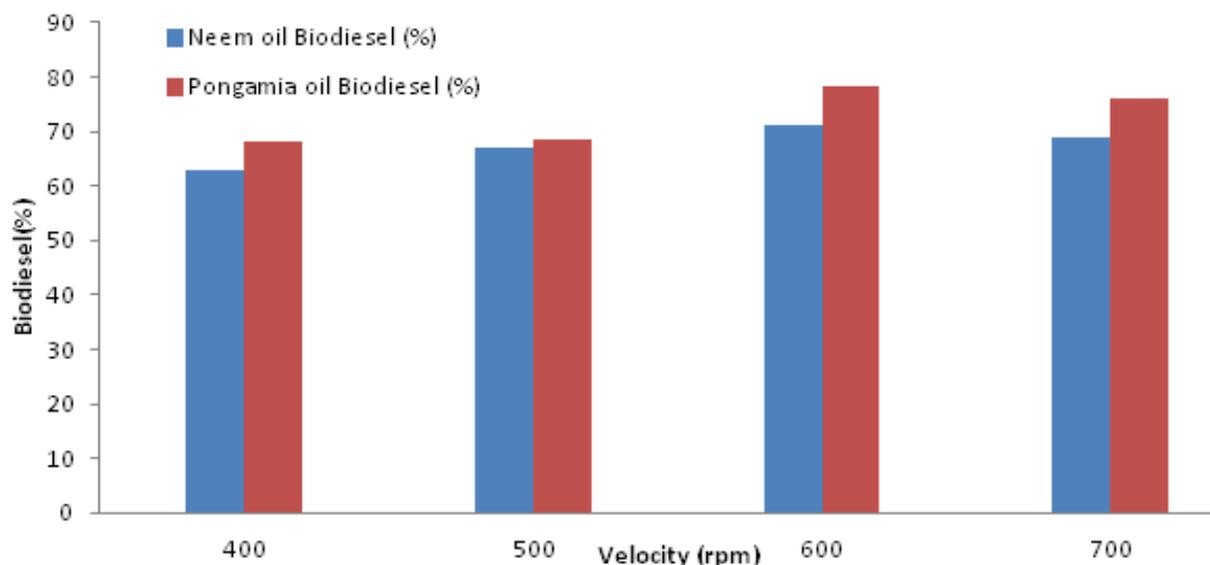


Fig. 5: Neem and Pongamia oil biodiesel yields with different velocities (rpm)

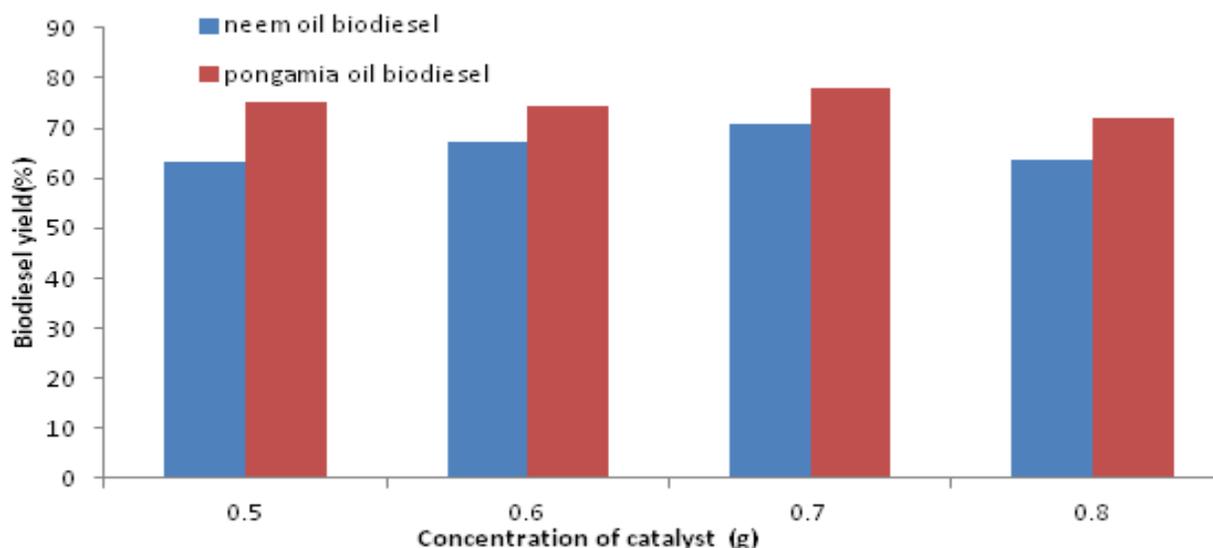


Fig. 6: Neem and Pongamia oil biodiesel yield with various catalyst concentrations

Experimental results by varying reaction time

The yield of methyl esters of Neem and Pongamia oils were investigated by changing reaction time (1hr, 2hrs, 3hrs, 4hrs). The stirring speed, temperature, catalyst concentration and molar ratio of methanol/oil were kept constant. The highest conversion rate of Neem biodiesel (71%) and Pongamia biodiesel (78.16%) obtained in 2hrs. The residual methanol recovery at highest yield of Neem and Pongamia oils are 16.0% and 8.6% respectively. The glycerin yield at highest yield of Neem and Pongamia oils are 7.10% and 4.9% respectively (Table 3, Fig. 4).

Experimental results by varying stirring speed (rpm)

The yield of methyl esters of Neem and Pongamia oils were investigated by changing stirring speed (400 rpm, 500 rpm, 600 rpm and 700 rpm). The time, temperature, catalyst concentration and molar ratio of methanol/oil were kept constant. The highest conversion rate of neem and pongamia biodiesels are (71%) and (78.16%) respectively at stirring speed 600 rpm. The residual methanol recovery at highest yield of Neem and Pongamia oils are 16.0% and 8.6% respectively. The glycerin yield at highest yield of Neem and Pongamia oils are 7.50% and 5.6% respectively (Table 4, Fig. 5).

Experimental results by varying catalyst concentrations

The yield of methyl esters of Neem and Pongamia oils were investigated by changing catalyst concentrations (0.5 g, 0.6 g, 0.7

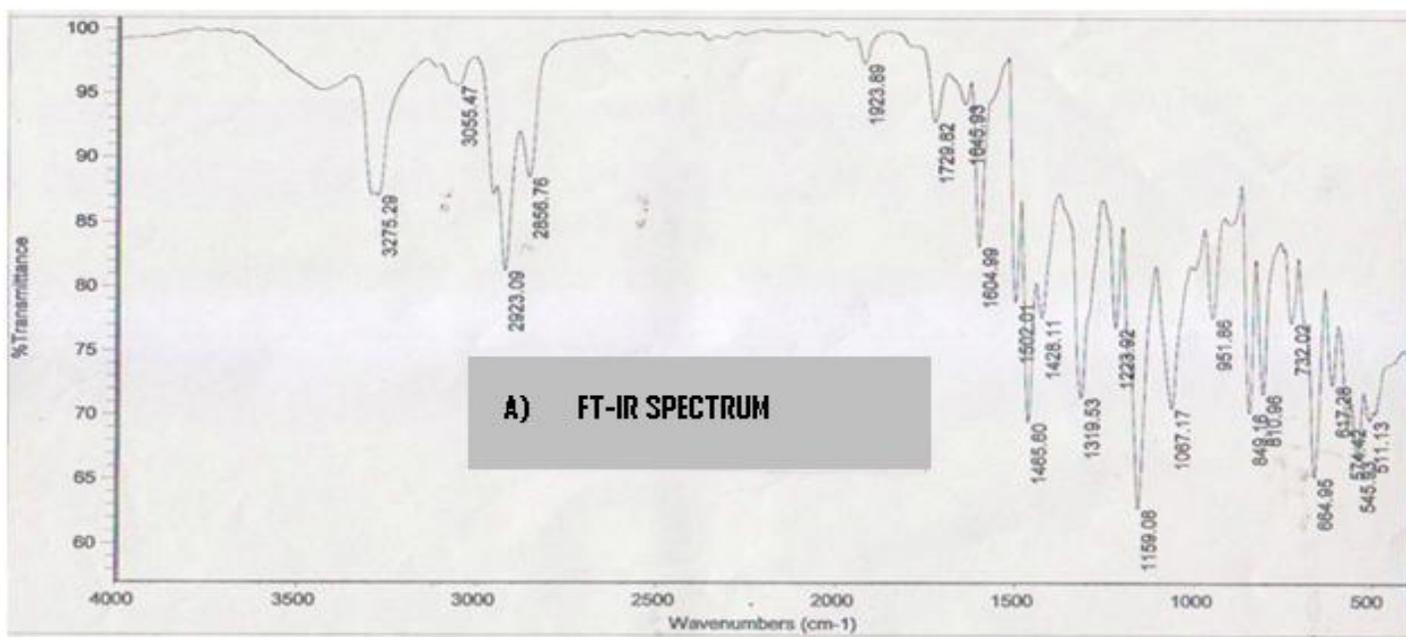


Fig. 7: Neem oil biodiesel chemical profile of FT-IR spectrum

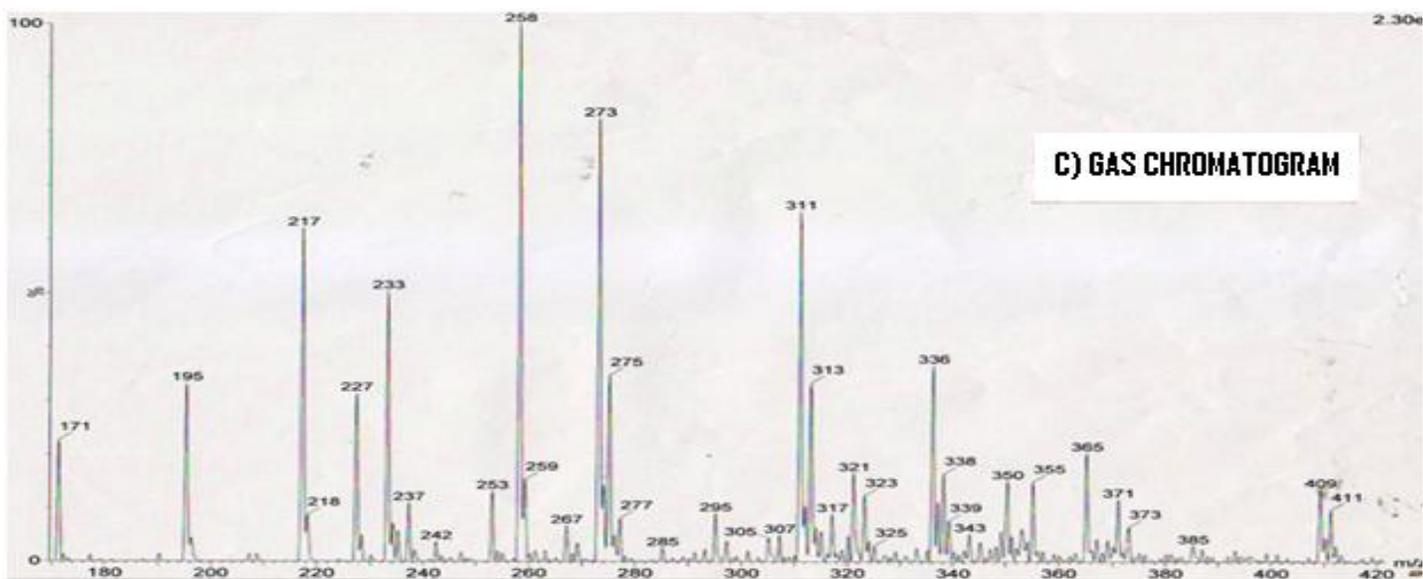


Fig. 8: Neem oil biodiesel chemical profile Gas chromatogram

g, 0.8 g). The time, temperature, stirring speed and molar ratio of methanol/oil were kept constant. The highest conversion rate of Neem biodiesel (71%) and Pongamia biodiesel (78.16%) obtained at 0.7gms. The residual methanol recovery at highest yield of Neem and Pongamia oils are 15.5% and 7.9% respectively. The glycerin yield at highest yield of Neem and Pongamia oils are 7.5% and 6.8%, respectively (Table 5, Fig. 6).

To achieve maximum yield of biodiesel from non-edible seed oils, alkali based transesterification was carried out. Maximum yields of methyl esters of Neem and Pongamia oils were obtained by optimizing parameters like molar ratio (6:1), temperature (60 °C), stirring speed (600 rpm) and reaction time (2 h). The highest conversion rate was obtained with the catalyst

concentration of 0.7 g, under these conditions the biodiesel yield was 71% and 78.16% for Neem oil and Pongamia oils respectively (Table 6).

OIL PROPERTIES

The experiments were carried out for different fuel properties according to ASTM D-445, D-1298, D-93, D-1298, D-1500, and D-4294 methods.

Viscosity

Table 5: Biodiesel yield with different concentrations of catalyst

Name of the biodiesel	Catalyst NaOH concentration (g)	Biodiesel (%)	Glycerin (%)	Residual Methanol (%)
Neem oil biodiesel	0.5	63.25	8.1	12.1
	0.6	67.40	6.1	12.5
	0.7	71.0	7.5	15.5
	0.8	63.50	5.1	12.0
Pongamia oil biodiesel	0.5	75.30	7.1	11.0
	0.6	74.45	7.7	13.0
	0.7	78.16	6.8	7.9
	0.8	72.15	5.4	12.0

Table 6: Comparative analysis for fuel properties of Neem and Pongamia oil biodiesels with High Speed Diesel

Properties	Neem oil Biodiesel	Pongamia oil Biodiesel	High Speed Diesel (HSD)
Kinetic viscosity @ 40 °C cst	4.81	3.83	1.3-4.1
Density @ 30°C Kg/L	1.74	1.72	1.76
Flash point(°C)	124	90	60-80
Sulphur % wt	Nil	0.008	0.05
Pour Point (°C)	9	3	-35 to 15
Cloud point (°C)	12	6	-15 to 6

Table 7: Fatty acid composition of Neem and Pongamia oil

Fatty acids	Carbon number	Neem oil (%)	Pongamia oil (%)
Myristic Acid	C14:0	-	-
Palmitic Acid	C16:0	14.9	10.6
Stearic Acid	C18:0	14.4	6.8
Oleic acid	C18:1	61.9	49.4
Linoleic Acid	C18:2	7.5	19.0
Arachidic Acid	C20:0	1.3	4.1
Eicosenic acid	C20:1	-	2.4
Docosanoic acid	C22:0	-	5.3
Tetracosanoic acid	C24:0	-	2.4

Table 8: Chemical Composition of Neem and Pongamia biodiesel based on FT-IR analysis

Sample type	Catalyst	C=O (cm ⁻¹) Ester Carbonyl	C-O (cm ⁻¹)
Neem oil biodiesel	NaOH	1729.82	1059.08
Pongamia oil biodiesel	NaOH	1610.04	1234.02

High viscosity is the major problem preventing the use of vegetable oils and animal fats directly in diesel engines as it affects the flow of fuel and spray characteristics. High Speed Diesel (HSD) has viscosity of 1.3-4.1 at 40 °c whereas the

viscosities of Pongamia oil biodiesel and Neem oil biodiesel are 3.83 and 4.81 which were closer to HSD.

Density

The density of Pongamia oil biodiesel and Neem oil biodiesels at 30°C was found to be 1.74 Kg/L and 1.72 Kg/L which are closer to the density of diesel 1.76 kg/lit can be used as an alternative fuel.

Flash point

Flash point is the temperature that indicates the overall flammability hazards in the presence of air; higher flash points make for safe handling and storage of biodiesel. The Flash Points

of Neem oil biodiesel and Pongamia oil biodiesels were 124 °C and 90 °C respectively, which are higher than that of HSD (60 °C-80 °C) which makes for safe handling and storage.

Sulphur content

The most valuable result is the reduction and absence of percentage of total sulphur contents in Neem oil biodiesel and Pongamia oil biodiesel that will result in reduction of SO_x in exhaust gases which is one of the reason for acid rain.

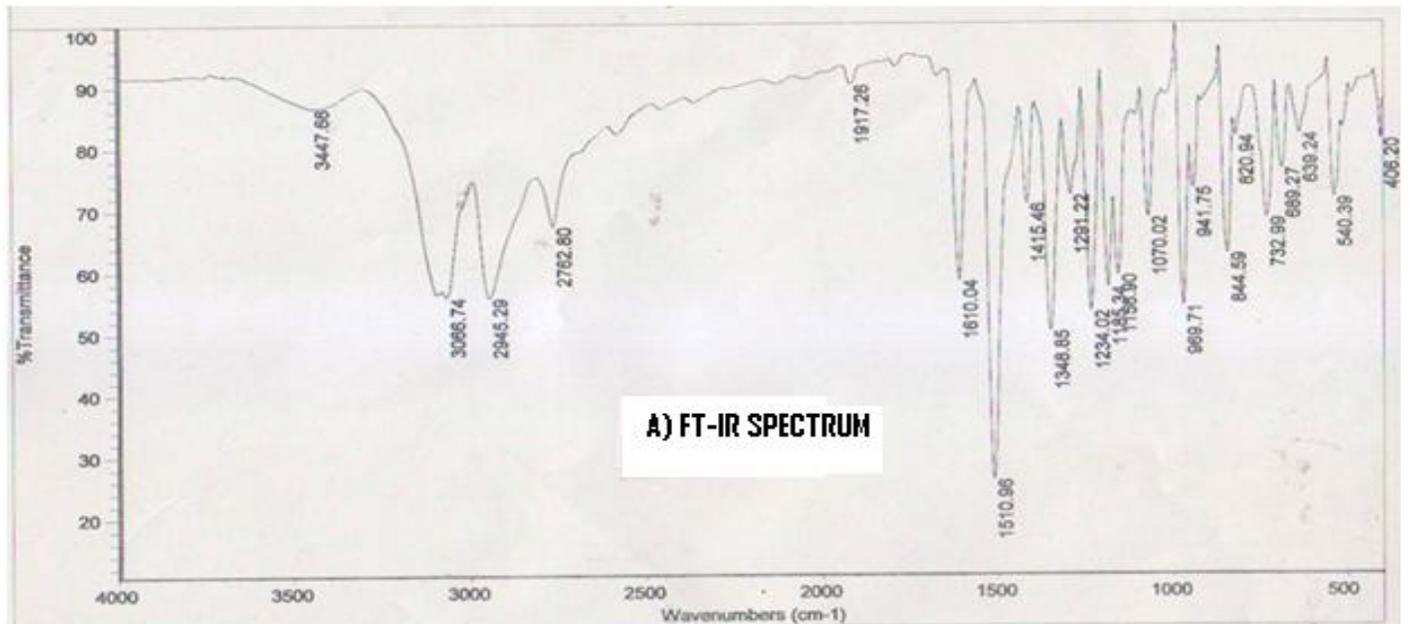


Fig. 9: Pongamia oil biodiesel chemical profile of FT-IR Spectrum

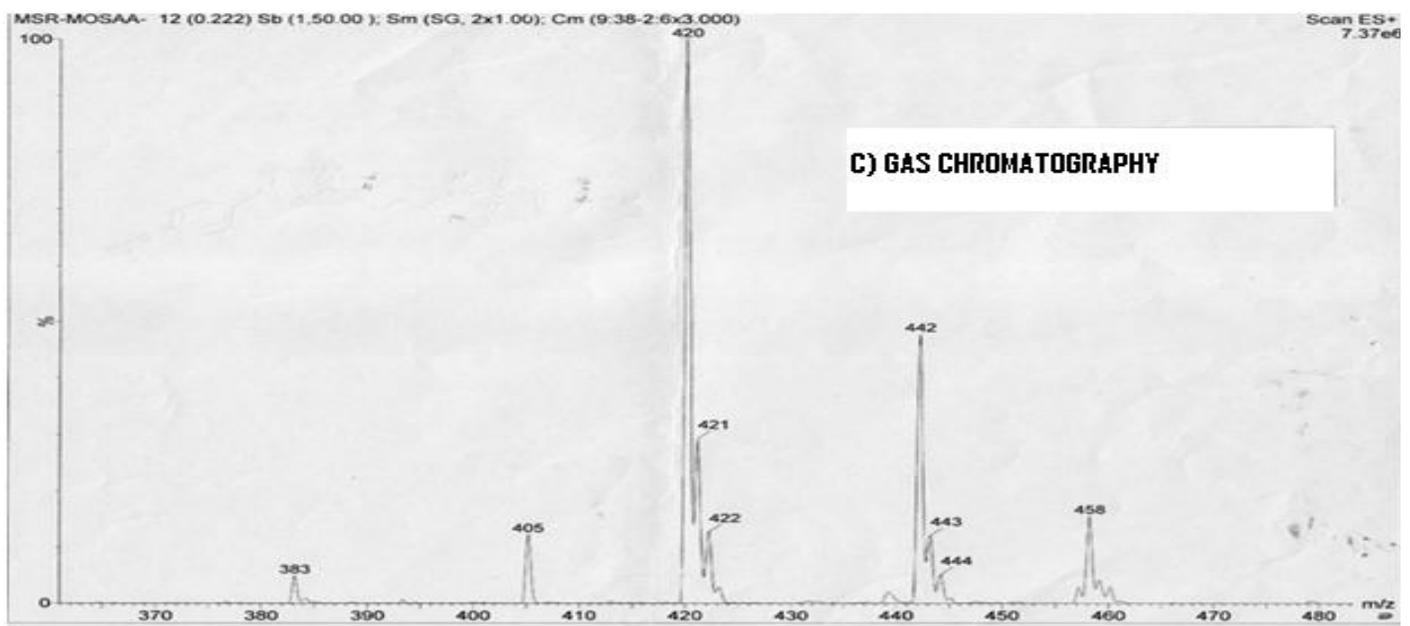


Fig. 10: Pongamia oil biodiesel chemical profile of Gas chromatography

Cloud point and pour point

Cloud point is the temperature at which a cloud of wax crystals first appear in the oil when it is cooled. The pour point is the lowest temperature at which the oil sample can still be moved. These properties are related to the use of biodiesel in colder regions. The cloud points of Neem oil biodiesel and Pongamia oil biodiesel were 12 °C and 6 °C and the pour point are 9 °C and 3 °C respectively.

GC-MS ANALYSIS

The fatty acid methyl esters (FAME) content were determined by gas chromatography, model GC 6890N coupled with mass spectrometer, model MS 5973 MSD (mass selective detector). Separation was performed on a capillary column DB-5MS (30 m×0.32 mm, 0.25µm of film thickness). The carrier gas was helium with flow rate of 1.5 mL/min. The column temperature was programmed from 120-300 °C at the rate of 10°C/min. A sample volume of 0.1µL NOB/POB in chloroform was injected using a split mode, with the split ratio of 1:10. The mass spectrometer was set to scan in the range of m/z 50-550 with electron impact (EI) mode of ionization (Figs. 8 and 10).

The FA compositions of Neem and pongamia oils

The compositions of fatty acids of Neem and Pongamia oils were analyzed by using gas chromatography. The use of mass spectrometer would eliminate any ambiguities about the nature of eluting materials since mass spectra unique to individual compounds would be obtained. Fatty acid (FA) components of Neem and pongamia oils mentioned in the Table 6 and 7.

Oleic acid was the most common FA found in Pongamia oil and Neem oil. Neem oil containing 61.9 % oleic acid, Palmitic Acid (14.9). Stearic Acid (14.4) Linoleic Acid (7.5) Arachidic Acid (1.3).The fatty acids like Eicosenic acid, Docosanoic acid and Tetracosanoic acids were absent in Neem oil . Oleic acid is the major unsaturated fatty acids found in Neem oil.

Pongamia oil contains 49.4% oleic acid, 19.0% linoleic acid, 10.6% palmitic acid, and 6.8% stearic acid Arachidic acid (4.1), Eicosenic acid (2.4), Docosanoic acid (5.3), Tetracosanoic acid (2.4). The amount and type of fatty acids in the oils determines the viscosity. The viscosity is the most important characteristics of biodiesel. Due to the presence of high amount of long chain fatty acids, Pongamia oil may have a slightly higher viscosity compared to Neem oil. The FAMES of these oils also meet the specification of 90/95% boiling point limit of 360 °C specified in ASTM-D6751 and in other biodiesel standards. Generally, the FAMES, which are mainly comprised of carbon chain lengths from 16 to 18, have boiling points in the range of 330–357 °C; thus the specification value of 360 °C is easily achieved. Besides, the concentration of linolenic acid containing four double bonds in FAMES should not exceed the limit of 12% and 1%, respectively. FAMES of all species are within the specified limit, so they are suitable for the production of biodiesel.

FT-IR ANALYSIS

Biodiesel samples were characterized by FT IR, using a Bio-Rad Excalibur Model FTS3000MX in the range 4000 - 400 cm⁻¹. The resolution was 1cm⁻¹ and 15 scans.

FT-IR analysis for fatty acid methyl esters

The FT-IR spectra in the mid-infrared region have been used to identify functional groups and the bands corresponding to various stretching and bending vibrations in the samples of oil and biodiesel. The position of carbonyl group in FT-IR is sensitive to substituent effects and to the structure of the molecule. The ester carbonyl group in Neem oil biodiesel and Pongamia oil biodiesel was appeared at 1729.82 cm⁻¹ and 1610.04 cm⁻¹ respectively. The band appeared at 3465 cm⁻¹ showed the overtone of ester functional group (Table 8, Figs. 7 and 9).

CONCLUSIONS

In this study an optimized protocol for biodiesel production from non-edible seed oils of Neem (*Azadirachta indica* A. Juss.) and pongamia (*Pongamia pinnata* L Pierre.) converted into fatty acid methyl esters (FAME) through base catalyzed transesterification process. It can be concluded that maximum biodiesel yield from Neem and Pongamia oils(with FFA 2%) can be achieved at 6:1 molar ratio of Methanol and oil, 0.7 g NaOH, 60 °C reaction temperature and 2 reaction time followed by 3-4 times gentle washings of the biodiesel with acidified hot water (10% v/v). 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 biofuel as a useful alternative and environmentally friendly energy resource.

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