PRODUCTION OF BIODIESEL FROM NON-EDIBLE TREE-BORNE OILS AND ITS FUEL CHARACTERIZATION

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ABSTRACT

This article intends to extract oil from non-edible seeds like neem, polanga, mahua and simarouba in the form of crude vegetable oil. The extracted oil was used for biodiesel production by following esterification and transesterification process. Different methods were adopted for the determination of fuel properties namely density, viscosity, calorific value, flash point, fire point, cloud point, poor point, cetane no, carbon residue, copper strip corrosion, iodine value, saponification value and moisture content of sample fuels following the standards procedure. The result of fuel properties obtained for the sample fuels are compared with diesel fuel. Pertaining to fuel properties, the tested fuels show akin fuel properties as diesel fuel.

KEYWORDS: Biodiesel, Diesel, Fuel Properties, Vegetable Oil

India being a developing country requires much higher level of energy to sustain its rate of progress. India's energy demand is expected to grow at 5.2%. India is the world's 5th largest energy consumer for about 4.1% of the world's energy and moving fast enough to become the 3rd largest consumer after US and China. India's import bill on import of petroleum products is 5,27,765 crore in 2015-16.

Many research works have been carried out on edible and non-edible methyl ester and various conclusions were drawn [1-3]. The use of edible vegetable oils for biodiesel production is been a great concern because they compete with food materials. Hence, the contribution of non-edible oils such as neem, mahua, polanga and Simarouba oil will be significant as a non-edible plant oil source for biodiesel production [4-6]. Considering the oil contents of seeds and availability potential of neem, mahua, polanga and simarouba oils, the present investigation on biodiesel production and determining the physical and chemical properties of the said biodiesel and comparing with diesel were undertaken with the following specific objectives given below.

The main objectives of the study can be stated as the following

- To extract vegetable oil from the seeds
- To convert the vegetable oil to biodiesel.
- To prepare the blends of biodiesel (B10).
- To determine some important fuel properties of the sample fuel and compared with diesel.

MATERIALS AND METHODS

Oil Extraction

Considering the number of non-edible oil seed bearing tree species having tremendous potential of biodiesel production in Odisha, Neem(Azadirachta Indica), Mahua (Madhuca Indica), Polanga (Calophyllum inophyllum) and Simarouba (Simarouba glauca)tree borne oils were selected for present investigation. Details of some of the tree-borne oil seed plants are given in Table 1 referred from [7-8].

Table 1: Oil Extractions of selected Oil Seed Plants

Sl. No	Botanical Name	Common Name	Oil (%)
1	Azadirachta indica	Neem	20-50
2	Madhuca indica	Mahua	35
3	Calophyllum inophyllum	Polanga	50-73
4	Simarouba glauca	Simarouba	60-75

The extraction of vegetable oil from seeds is commonly categorized as:

- Mechanical extraction by expeller.
- Chemical extraction by solvent extraction.

For mechanical expeller, the dry fruits were collected in a drum, and the kernels were separated. Later the kernels were dried and then fed into the oil extraction machine. The pure vegetable oil obtained by pressing followed by filtration and is collected in a drum.

In solvent extraction, oil from solid material was extracted by repeated washing with an organic solvent which was hexane. In this method 50 gram of kernel was dried, ground into small particles and placed in the thimble. The thimble was placed in an extraction chamber, which was suspended above a flask containing the solvent. The flask was heated and the solvent evaporates and moves up in to the condenser where it was converted in to a liquid and

trickles into the extraction chamber containing the sample. When the solvent surrounding the sample exceeds a certain level it overflows and trickles back down into the boiling flask. At the end of the extraction process which lasts for 7 hours. The solvent was removed by rotary evaporator to recover the required oil. The collected oil from expeller was tested for FFA (Free fatty acid).

The FFA of oil was found to be more than 1.5. Therefore, a two-step transesterification process was required for these feed stocks. Line diagram of biodiesel production is shown in Fig. 1.
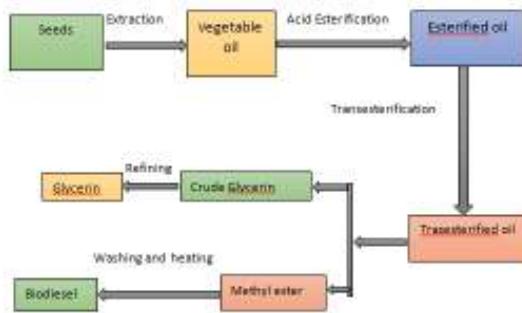

Figure 1: Line diagram of biodiesel production

Preparation of Biodiesel

Esterification Reaction

For the pre-treatment, 5 litre of the above oil was heated up to 60^0C, and then methanol (6:1 molar ratio of methanol: oil) and acid catalyst (0.5% v/v) were added. The reactants were stirred at a speed of 1600 revolution per minute at a temperature of 60^0C for 2 hours. This decreased the acid value significantly. The top layer methanol was separated out by decantation process and the oil layer was taken for transesterification. Once the reaction was completed, it was dewatered by passing over a hydrous Na_2SO_4 and then fed to the transesterification process.

Transesterification Reaction

The above esterified oil were filtered and pre-processed to remove water and contaminants if any and then fed directly to the transesterification process along with any product of the acid esterification process. The oil was pre-heated to 65^0 C and a mixture of methanol and the catalyst KOH was added to the oil. The molar ratio of MeOH / oil was 6:1 and catalyst concentration was 1% w/w of oil. A part of the alkali catalyst was used to neutralise the residual amount of acid and the remaining as catalyst for transesterification. Once the reaction was completed, the product was allowed to stand overnight to separate

the layers. The upper biodiesel layer was washed with hot distilled water to remove the excess methanol, catalyst and traces of glycerol. The washed ester layer was dried under the vacuum to remove the moisture and methanol, and again it was passed over a hydrous Na_2SO_4. The biodiesel so obtained were designated as methyl ester. Laboratory Scale-Biodiesel Production Unit is shown Fig. 2.
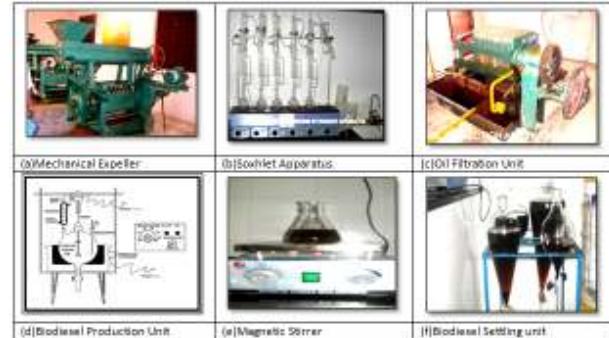
Figure 2: Biodiesel production unit

Preparation of Biodiesel Blends

Fuel blends were prepared from above prepared biodiesels and diesel. Diesel was blended with biodiesel in different proportions to form B10(10% biodiesel + 90 % diesel). Photograph of sampleoil with biodiesel blends is shown in Fig. 3.

Figure 3: Sample Oil and Biodiesel

Fuel Properties

Fatty Acid Profile

Fatty acid profile of oil samples is given in Table 2.

Table 2: Fatty Acid Profile of Sample Oils as shown in [9]

Fatty Acid	Neem oil	Mahua oil	Polanga oil	Simarouba oil
C_{16}	14.9	24.5	12.01	12.3
C_{17}	-	-	-	0.1
C_{18}	14.4	22.5	12.95	27.3
$C_{18:1}$	61.9	37.5	34.09	54.6
$C_{18:2}$	7.5	14.3	38.26	2.3
$C_{18:3}$	-	-	0.3	0.2
C_{20}	1.3	1.5	-	1.2
$C_{22:1}$	-	-	-	0.4

Flash Point Fire Point

A Pensky Martens closed cup type apparatus was used in the study for determination of flash point and fire point shown in Fig. 4 a. In the experiment, the sample was filled in the test cup up to the specified level and heated with the help of a heater in such a way that the temperature rise was approximately 5^0C per minute with uniform stirring. At every 1^0C rise in temperature, the flame was introduced for a moment with help of a shutter. The fire point was recorded at that temperature when the fuel gives sufficient vapor that catches fire at least for five seconds.

Cloud Point and Pour Point

The apparatus mainly consists of 12 cm high glass tubes of 3 cm diameter. The tubes were enclosed in an air jacket, which is filled with a freezing mixture of crushed ice and sodium chloride crystals. The glass tube consists of fuel sample was taken out from the jacket at every 1^0C interval as the temperature falls, and inspected for cloud formation. The point, at which a haze was first observed at the bottom of the sample, was taken as the cloud point.

For determining the pour point, the sample was pre-heated to 48^0C and then cooled to 35^0C in air before it was filled in the glass tube. Thereafter, the cooled samples were placed in the apparatus and withdrawn from the cooling bath at 1^0C interval for checking its flow ability. The pour point was taken to be the temperature 1^0C above the temperature at which no motion of fuel was observed for five seconds on tilting the tube to a horizontal position. Three replications were made for the fuel.

Density

Density is defined as mass per unit volume. The relative density of the selected fuels at 15^0C was determined. The empty pycnometer of 50 ml capacity was weighed by an electronics balance. The pycnometer was then filled with fuel sample and weighed. The samples were maintained at 15^0C by keeping them in a temperature control chamber. The weight of the empty pycnometer was subtracted from the weight of the filled ones to get the weight of the fuel sample. Three replicates were taken for each sample and their mean was calculated. This value when divided by the volume of the fuel sample gave the density of the fuel sample at 15^0C. The density of the distilled water at 15^0C was also determined. The Pycnometer is shown in Fig. 4b. Density was then calculated from the given Eqn 1.

$$Relative\ density = \frac{Density\ of\ the\ fuel\ at\ 15^0C}{Density\ of\ the\ water\ at\ 15^0C} \quad (1)$$

Viscosity

Kinematic Viscometer is used for measuring viscosity shown in Fig. 4 c. The sample was introduced into the viscometer, invert the viscometer, immerse tube into the liquid and suction is applied which causes the sample to rise to etched line. The tube was inserted into a holder and placed at constant temperature bath for about 15 minutes to reach the equilibrium temperature. Vertical alignment of the tube may be accomplished in bath by suspending a plumb bob in the tube. Suction was applied to the tube so that the sample was raised a short distance above the mark. Same procedure was repeated to get the exact data. The viscometer constants vary for different size of the tubes and are given inn Appendix-I. Kinematic viscosity in centistokes was then calculated by using Eqn. 2.

Kinematic Viscosity = Viscometer constant X *t*   (2)

Where,

t = flow time, s

Vis constant of the viscometer= 0.0336 cSt/s

Calorific Value

Calorific value of fuel was determined by a closed vessel called bomb calorimeter shown in Fig. 4 d.

Sample fuel (1 gram) was placed in the crucible. Pure oxygen was then admitted through the oxygen valve till pressure inside the bomb rises to 30 atmospheres. The bomb was then completely submerged in a known quantity of water contained in a large copper vessel. When the bomb and its contents had reached steady temperature, fuse wire was heated up electrically. The fuel ignited and continued to burn till whole of its burnt. The heat, liberated by the combustion of fuel, is absorbed by this water and apparatus. The gross calorific value was calculated using Eqn. 3.

$$H_c = \frac{(m_w + m_e)C_w(t_2 - t_1)}{m_f} \quad (3)$$

Where,

m_f= Mass of fuel sample burnt in bomb, kg.; m_w= Mass of water filled in the calorimeter, kg.; m_e= Water equivalent of apparatus, kg ; C_w = Calorific value of water, kJ/kg K; t_2 = Final temperature of water, ^{0}C ; t_1 = Initial temperature of water, ^{0}C

Cetane No.

Cetane Number indicates the readiness of the fuel to self-ignite when exposed to the high temperatures and pressure in the diesel engine combustion chamber. Higher the cetane number, better its ignition properties. It affects engine performance parameter like combustion, stability, smoke, noise and emission of CO and hydrocarbons.

The cetane number is an indicator of the quality of fuel's combustion during ignition while it is under compression. It is one of several important measures of fuel's quality and specifically indicates the fuel's ignition delay. This is the period of time that elapses between a fuel's injection into the combustion chamber and the start of its combustion. A higher cetane number means that fuel has a shorter ignition delay. Cetane index is given in Eqn 4.

$$Cetane\,Index = 46.3 + \frac{5458}{s.v} - (0.225 \times i.v) \quad (4)$$

Where,

s.v= Saponification value. i.v = Iodine value

Cetane number is nearly equal to cetane index as per the equation given below also reported by [10]:

Cetane Number = Cetane Index – (1.5 to +2.6)

Carbon Residue

The test was conducted by using Conradson's apparatus shown in Fig. 4 e. In this method, 10 g weight of each fuel sample was weighed free of moisture and other suspended matter in to an iron crucible. The crucible was then placed in the centre of skid more crucible of the apparatus and the sand was levelled in the large sheet iron crucible and then the skidmore crucible was set on it in the exact centre of the iron crucible. Thereafter, the covers were applied to both skidmore and iron crucible loosening the latter fitting to allow free exit to the vapour as it formed. The fuel sample was then heated with a high flame from gas burner for 20 minutes. When the smoke appeared on the chimney, immediately the burner was moved or tilled so that the gas flame placed on the sides of the crucible for the purpose of igniting the vapour, after that the ignited vapour was burnt uniformly with the flame above the chimney. When the vapour ceased to burn and no further smoke was observed, the burner was adjusted and the heat was held as at the beginning to make the bottom and the lower part of the sheet iron crucible a cherry red appearance about 15 minutes. The cover of skidmore was then removed with a tong and it

was cooled and weighed. The percentage of carbon residue on the original sample was then calculated using the equation 5given below:

$$Carbon\,residue\,(\%) = \frac{mass\,of\,carbon\,residue, g}{mass\,of\,test\,sample, g} \times 100$$
(5)

Copper Strip Corrosion Test

The corrosiveness of a fuel is measured using the copper strip corrosion test. A polished copper strip was immersed in a specific volume of the sample being heated under specific temperature and time. At the end of the heating period the copper strip foil was removed, washed and the colour tarnish level were assessed against the ASTM copper strip corrosion standards. The model is shown in Fig. 4 f and Fig. 4 g.

Iodine Value

Iodine value, also called Iodine number, is the mass of iodine in grams absorbed by 100 g of the oil/fat. The iodine value is a measure of the degree of un-saturation of an oil or fat. It is a constant for particular oil or fat. It is an useful parameters in studying oxidative rancidity of oils since higher the un-saturation, greater is the possibility of the oil to become rancid.

Halogens add across the double bonds of unsaturated fatty acids to form addition compounds. Iodine mono chloride (ICL) was allowed to react with the fat in the dark. Iodine gets incorporated into the fatty acid chain wherever the double bond exist. The amount of iodine consumed was then determined by titration of the iodine released (after adding KI) with standard sulphate and comparing with a blank in which the fat was omitted. Hence the measure of iodine absorbed by an oil or fat gives the degree of un-saturation. The reaction in the test is given below as per [11]:

The iodine value of the sample is determined by the Eqn. 6 given below.

$$Iodine\,Value = \frac{V \times N \times 0.1269}{Mass\,of\,all\,ng\,gram} \times 100 \quad (6)$$

Where,

V = Volume of $Na_2S_2O_3$ (blank experiments - test experiments);   N= Normality of $Na_2S_2O_3$ solution

1 ml of 0.1N $Na_2S_2O_3$ = 0.1269gm. of Iodine

Saponification Value

Saponification value represents the number of milligrams of potassium hydroxide required to saponify 1 g of fat under the conditions specified.

Saponification is the process by which the fatty acids in the glycerides of the oil/fat are hydrolyzed by an alkali. The resultant salts of fatty acids are called soaps. When the oil or fat (triglyceride) is heated with KOH (alkali) it is saponified (hydrolyzed) and release fatty acids and glycerol. It is the number of milligram of KOH required to neutralise the fatty acids resulting from the complete hydrolysis of 1 g of fat or oil. This value gives an indication of the nature of fatty acids constituent of the fat and depends upon the average molecular mass of the fatty acid constituent of fat. Thus this value is useful for a comparative study of the fatty acid chain length in oils. The greater the molecular mass (the longer the carbon chain) the smaller the number of fatty acids is liberated per gram of fat hydrolysed and therefore the smaller the saponification number and vice versa. A blank experiment was similarly treated in the absence of oil. 5 gm of oil was refluxed with 25 ml of alcoholic KOH. After saponification, remaining KOH was estimated by titrating it against standard HCl. Clarity and homogeneity of the test solution are indicators of complete saponification.

$$Saponification\ Value\ of\ oil (mgKOH) = \frac{(Blank\ experiment - Titrate\ experiment)}{Mass\ of\ oil(g)} \times 23.06$$

(7)

Where,

1 ml of 0.5 N HCl = 28.06 mg KOH

Moisture Content

Karl Fischer Moisture Analyzers shown in Fig. 4 h is used for determination of oil or water content in crude oils. In this experiment, methanol was poured into the titration vessel in such a way that the upper level of solvent was just in contact with platinum electrode. Titration was carried out by KF reagent to moisture free the vessel with continuous stirring by magnetic stirrer. Then 10 µl distilled water was added to the vessel and titration was performed to determine the titre value in mg of water consumed per ml of KF reagent. After the determination of titre value, a known quantity of sample was added to the vessel and titrated against the KF reagent which was contained in burette. The burette was driven by microprocessor controlled stepper motor with continuous stirring by magnetic stirrer. As the end point was approached, the reagent delivery was slowed down and finally stopped when the end point was reached. The amount of water present in the sample was calculated by the Eqn. 8 as per [12] given below:

$$Moisture\ Content, \% = \frac{Volume\ of\ KF\ reagent\ consumed, ml \times titre\ value, \frac{mg}{ml}\ of\ titre}{Sample\ volume} \times 100$$

(8)
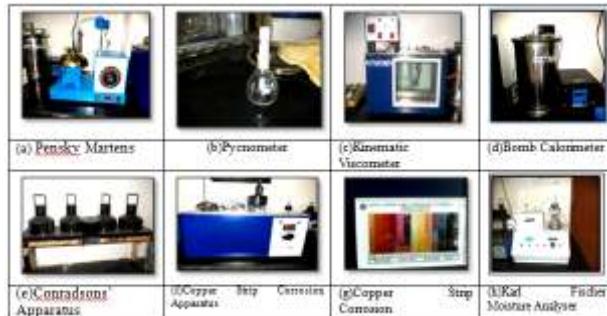
Figure 4: Apparatus used for Determination of Fuel Properties

RESULTS AND DISCUSSION

The oil explored from kernels by soxhlet apparatus and expeller is shown in Table 3.

Table 3: Oil Productions by Soxhlet Apparatus and Expeller

Vegetable Oils	Oil Production by Soxhlet Apparatus (%)	Oil Production by Expeller (%)
Neem (Azadirachta indica)	40-45	35
Mahua (Madhuca indica)	50	37
Polanga (Calophyllum inophyllum)	60	50
Simarouba (Simarouba glauca)	70	55

Table 3 shows that the oil production by soxhlet apparatus was 25%-40 % more than mechanical expeller, can be compared with the result given in [13]. The FFA of Neem, Mahua, Polanga and Simarouba at different conditions, are summarized in Table 4.

Table 4: FFA values of the Sample Oils

Oil samples	Before esteri-fication	After esteri-fication	After trancesteri-fication
Neem	45	3.1	0.40
Mahua	34	2.3	0.23
Polanga	42	3.2	0.39
Sima-rouba	10	1.5	0.20

From the above table the FFA of all these oil samples after transesterification were within the limit. The acid value (FFA *2) of the above tested samples were within the prescribed limits. Density, Viscosity and Calorific value of sample fuels is mentioned in Table 5.

Table 5: Densities, Viscosity and Calorific Value of Sample Fuels

Sl. no.	Name of Oil/Bio diesel	Density (gm/cc)		Kinematic Viscosity (cSt)		Calorific value (MJ/Kg)
		Oil	Bio diesel	Oil	Bio diesel	Bio diesel
1	Neem	0.90	0.88	37.87	4.5	39.81
2	Mahua	0.90	0.88	37.18	4.98	39.10
3	Polanga	0.89	0.87	35.61	4.00	41.38
4	Simarouba	0.85	0.86	44.95	4.68	41.20
5	Diesel	0.82		2.85		45.339

The density of sample crude oil was found to be around 2-5% higher than that of biodiesel and was 5-12% higher than that of diesel. The density of tested biodiesel samples were within the limits (0.86-0.90 gm/cc) prescribed in the biodiesel standards.

The kinematic viscosity values of neem, mahua, polanga and simarouba crude oil were 13 to 16 times that of diesel.

The calorific values of neem, mahua, polanga and simarouba biodiesel were 13.8%, 15.93%, 9.5%, and 10.02 % lower than that of diesel respectively. The oxygen molecule present unites with hydrogen of the oil for combustion reacts with hydrogen as reflected by [12].

Flash Point, Fire Point, Cloud Point and Pour Point of sample fuels are given in Table 6.

Table 6: Flash Point, Fire Point, Cloud Point and Pour Point of Sample Oil/Biodiesel

Sl. no.	Bio-diesel	Flash Point (^{0}C)	Fire Point (^{0}C)	Cloud Point (^{0}C)	Pour Point (^{0}C)
1	Neem	160	170	9	2
2	Mahua	168	180	14	3
3	Polanga	148	160	13	4
4	Simarouba	146	156	19	4
5	Diesel	60	65	6.5	-20

The flash point of sample crude oil was found to be more than that of biodiesel. Also the flash point of sample biodiesel was quite high compared to 60^0C for the diesel.

The fire point of oil was higher than that of biodiesel.

The cloud point of biodiesel was more than that of oil. The pour point of sample oil was quite higher than that of diesel. In general the cloud point and pour point of non-edible biodiesels were higher than that of diesel

Cetane No. and Carbon residue of sample fuel is mentioned in Table 7

Table 7: Cetane No. and Carbon Residue of Sample Biodiesel

Sl. No	Biodiesel	Cetane No	Carbon residue, %
1	Neem	52	0.21
2	Mahua	55	0.22
3	Polanga	56	0.20
4	Simarouba	54	0.21
5	Diesel	50	0.15

The cetanes no of biodiesels were more than that of diesel which led to better combustion.

The carbon residue values of biodiesel were found more than diesel. Carbon residue of sample biodiesel was within the specified limit of 0.3%.

The copper strip corrosion test results of sample fuel are given in Table 8

Table 8: Copper Strip Corrosion Test Results of Sample Oils

Sl. No.	Name oil	Copper strip corrosion
1	Neem	Slight Tarnish
2	Mahua	No corrosion
3	Polanga	Slight Tarnish
4	Simarouba	No corrosion
5	Diesel	No corrosion

Copper-strip corrosion test indicates the presence of sulphur compounds.

Iodine value, Saponification value and Moisture Content of sample fuel are mentioned in Table 9.

Table 9: Iodine Value, Saponification Value and Moisture Content of Sample Fuels

Sl. no.	Sample Biodiesel	Iodine Value (g I_2/100g)	Saponification Value (mg KOH/g)	Moisture content (%)
		Bio-diesel	Bio-diesel	Biodiesel
1	Neem	80	204	0.03
2	Mahua	70	194	0.04
3	Polanga	75	187	0.03
4	Simarouba	83.4	185.9	0.03
5	Diesel	-	-	0.02

The iodine value, which is a measure of the degree of un-saturation of oil, was found to be within the limit (≤ 115) of the standard specification.

Saponification number refers to the milligram of KOH required for neutralizing the fatty acid resulting from the complete hydrolysis of 1 g of fat or oil. It affected the quality of biodiesel.

The moisture contents of seed given above were removed during esterification and transesterification process.

CONCLUSION

- Oil production by soxhlet apparatus was 25%-40 % more than mechanical expeller.
- The density of sample crude oil was found to be around 2-5% higher than that of biodiesel and was 5-12% higher than that of diesel.

- The kinematic viscosity values of neem, mahua, polanga and simarouba crude oil were 13 to 16 times that of diesel.
- The calorific values of neem, mahua, polanga and simarouba biodiesel were 13.8%, 15.93%, 9.5%, and 10.02 % lower than that of diesel respectively.
- The flash point of sample crude oil was found to be more than that of biodiesel. The fire point of oil was higher than that of biodiesel. The cloud point of biodiesel was more than that of oil. The pour point of sample oil was quite higher than that of diesel.
- The cetane no of biodiesels was more than that of diesel which led to better combustion.
- The carbon residue values of biodiesel were found more than diesel. Carbon residue of sample biodiesel was within the specified limit of 0.3%. Sample oils shows slight tarnish or no corrosive properties.
- The iodine value, was found to be within the limit ($\leq$ 115) of the standard specification. Saponification number gives an indication of the nature of fatty acids constituent of the fat.
- The moisture contents of seed given above were removed during esterification and transesterification process.

To end with, it can be concluded that pertaining to fuel properties, the tested fuel shows akin fuel properties as diesel fuel which can be an alternative for diesel fuel.

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