International Journal of Engineering Research and Application www.ijera.com ISSN : 2248-9622, Vol. 8, Issue 2, (Part -3) February 2018, pp.90-100

RESEARCH ARTICLE OPEN ACCESS

Production and Characterization of Mahua Biodiesel

Dr Nabnit Panigrahi[1],Mir Naseer Hussain[2], Manoj Kumar Behera[3]

[1] Professor, Department of Mechanical Engineering, Gandhi Institute For Technology (GIFT), Bhubaneswar
[3]Assistant Professor, Department of Mechanical Engineering, Gandhi Institute For Technology (GIFT), Bhubaneswar
[2] Assistant Professor, Department of Mechanical Engineering, Gandhi Engineering College, Bhubaneswar

ABSTRACT-- The oils obtained from expeller were used for biodiesel production. Degumming process was carried out for Neem and Polanga oil to nullify the gum effect. Then the oils were analysed for determination of their acid values by titrating against a known strength of KOH solution.

In this study, biodiesel is produced from Madhuca indica seeds commonly known as Mahua by using transesterification process using a low capacity pressure reactor and by-product of transesterification is glycerol, which is used in preparation of soaps. Mahua Oil Ethyl Ester (MOEE) was produced from the Mahua oil and is mixed with diesel to get different ratios of blends. biodiesel fuels are characterised by a number of physiochemical properties. Some of these are FFA, density, kinematic viscosity, calorific value, flash point , fire point, cloud point, pour point, cetane no, carbon residue, copper strip corrosion, iodine value, saponification number and moisture content. Although most of the biodiesel properties are similar to those of diesel, there are considerable differences in some of the basic fuel properties such as calorific values, density and viscosity. Biodiesel properties should meet the IS 15607, ASTMD-6751 and EN 14214 specifications.

Index Terms—Mahua, biodiesel, fuel properties, mahua oil methyl ester

I. INTRODUCTION

NE of the most essential preconditions for the overall development of a society is the efficient use of energy resources. The conventional energy sources like fossil fuels are widely used for production of energy. However, in the present day scenario, the attention has been shifted to non-conventional energy sources considering the pros and cons of

conventional energy resource utilization. The sources of such non-conventional energy are renewable in nature, and hence origin cyclically, safeguarding the sustainability of the globe, driven majorly by modernized needs of human beings.

Today worldwide energy demand is the most critical challengedirectly or indirectlyin the entire process of evolution, growth and survival of all living beings. It plays a vital role in the socio-economic development and human welfare of a country. Energy has come to be known as a 'Strategic Commodity' and any uncertainty about its supply can threaten the livelihood and economy of a country. The world is using energy at a rate of 2.4 kW per person as per [1]. Energy demand appears to have gradually gained momentum. The world energy consumption is increasing day by day and will be double in a period of 20 years by 2035 as also stated in [2]. Fossil fuel still represents over 80% of total energy supplied in the world. The increasing industrialisation and motorisation of the world has led to a steep rise in the demand of petroleum products. There are limited resources and the petroleum fuels are irreplaceable.

These finite resources and high consumption of petroleum fuel has given rise to frequent disruptions and uncertainties in its supply as well as hike in price.This higher international oil prices lead to domestic inflation increased input costs, an increase in the budget deficit which invariably drives up interest rates and slows down the economic growth. Geologists are searching for the unexplored deposits of petroleum resources. The new resources appear to grow arithmetically while the consumption is progressing geometrically. Under this situation, when consumption overtakes discovery, the world will lead to an ecological disaster.In this paper mahua biodiesel is prepared from mahua oil by esterification and transesterification method. Fuel properties are determined and compared with diesel.

II. METHODOLOGY

Mahua oil is extracted from mahua seed by mechanical expeller. First 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 oil obtained by pressing is collected in a drum. Then filtration is done to remove the unwanted particles left in the extruded oil in order to obtain the pure vegetable oil.

The oils obtained from expeller were used for biodiesel production. Degumming process was carried out for Neem and Polanga oil to nullify the gum effect.. Then the oils were analysed for determination of their acid values by titrating against a known strength of KOH solution.

The methods adopted for degumming were treating the crude mahua oil first with immobilized phospholipase and then extracting the phospholipase-treated crude oil with pure water. 1% phosphoric acid was added to 1200 ml of crude neem/polanga oil. The mixture was heated and stirred at 90^0 C. Then the mixture underwent settling for 24 hours resulting in gum free mahua oil.

A. Determination of Acid Value of Crude Sample Oils

The acid value of oil is defined as the number of milligrams of potassium hydroxide required to neutralize the free acid present in 1 gram of oil. It is the measure of amount of FFA present in the oil. Higher acid value leads to corrosion besides gum and sludge formation. The acid value or FFA is determined by a standard Titration method. The reagents used are N/10 KOH, Neutral ethyl alcohol and Phenolphthalein indicator. A solution was prepared by adding 1.4 gm KOH to 250 ml of water. 1gm sample oil was added to 20 ml ethyl alcohol, stirring it for 2 minutes without heating. Then 4 drops of phenolphthalein indicator was added into the solution. The solution underwent titration with the KOH solution. Titration indicating FFA is shown in Fig. 1.

$$\mathbf{AcidValue} = \frac{\mathbf{Burrettereading \times 5}}{\mathbf{Weightofsample}} (1)$$

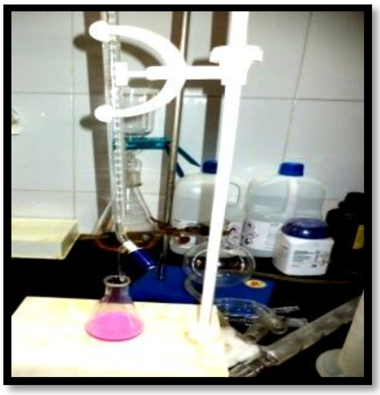


Fig.1. Titration of mahua oil

The FFA of oil was found to be more than 1.5. Therefore, a two-step transesterification process was required for these feed stocks. Initially, the FFA of these were converted to fatty acid methyl esters by an acid-catalysed pre-treatment and in the second step, transesterification was completed by using alkaline catalyst to complete the reaction.

B. Esterification of Crude Sample Oils

The reaction was carried out in the reactor. For the pre-treatment, 5 litre of each 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. Now, the pre-treated oil became suitable for alkali-catalysed transesterification.

C. Transesterification Reaction of Sample Oils

The above obtained oil feed stocks 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 major co-products, biodiesel and glycerine, were separated into two layers. 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 Mahua oil methyl ester (MOME).

Chemicals, including methanol, acid catalyst (H_2SO_4), alkali catalyst (KOH) etc used for the production of biodiesel were available in the Biodiesel Testing Lab and were of analytical grade.

The fuel properties of the purified biodiesels were characterised based on kinematic viscosity, flash point, acid value, calorific value, water and sediments, and were compared with ASTM and BIS standards. Laboratory Scale-Biodiesel Production Unit is shown Fig. 2. The Biodiesel Preparation Equipments in Biodiesel Testing Lab. is shown in Fig. 3.

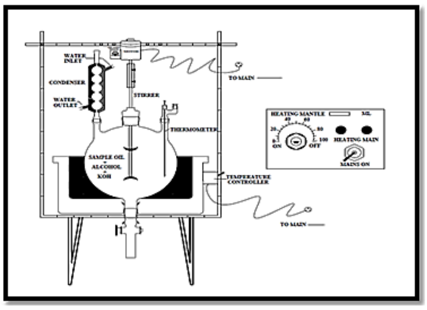


Fig. 2. Laboratory Scale-Biodiesel Production Unit

Fig.3. Magnetic stirrer with hot plate

The mahua oil and biodiesel is shown in Fig.4

Fig. 4. Mahua oil and biodiesel

D. Determination of Fuel Properties

FFA

Potentiometric titration is a technique similar to direct titration of a redox reaction. No indicator is used; instead the potential across the analytic, typically an electrolyte solution is measured. In this experiment a mixture of two acids was titrated with standard base. Since the pH did not change abruptly enough at either equivalence point to permit estimation with visual indicators, a potentiometric titration with a pH meter and glass pH electrode –reference electrode pair was employed. Potentiometric Titrator is shown in Fig. 5

$$\mathbf{FFA(FreeFattyAcid)} = \frac{\mathbf{Acidvalue}}{\mathbf{2}}$$
(2)

Fig. 5. Potentiometric Titrator

Density

Density is defined as mass per unit volume. It is an essential measurement of how tightly the matter is crammed together. The relative density of the selected fuels at 15^0C was determined as per **ASTM D 4052**. 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. A mercury thermometer was used to measure the temperature of fuels kept inside the 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 Density was then calculated from the given Equation 3.

$$\mathbf{Relativedensity} = \frac{\mathbf{Densiyofthefuelat15^0C}}{\mathbf{Densiyofthewaterat15^0C}}$$
(3)

Kinematic Viscosity

Viscosity is a measure of the internal resistance to motion of a fluid and is mainly due to the forces of cohesion between the fluid molecules. Absolute viscosity may be defined as the tangential force per unit area required to maintain unit velocity gradient between two parallel planes in the fluid at unit distance apart. The S.I. unit of kinematic viscosity is m^2/s. The CGS physical unit of viscosity is the stokes (st), also expressed in centistokes (cSt).

$1 \text{ St} = 1 \text{ cm}^2.\text{s}^{-1} = 10^{-4} \text{ m}^2.\text{s}^{-1}$

$1 \text{ cSt} = 1\text{mm}^2.\text{s}^{-1}=10^{-8} \text{ m}^2\text{s}^{-1}$

The viscosity of oils is the function of temperature. As the temperature increases the viscosity decreases. Too high viscosity can cause excessive heat generation in the injection equipment, owing to viscous shear in the clearance between pump plunger and cylinder. On the other hand, too low viscosity would lead to leakage through that clearance. Increasing viscosity reduces the injector spray cone angle, fuel distribution and penetration, besides increasing droplet size thus affecting fuel injection.

For determination of viscosity in the laboratory Kinematic Viscometer used was of Alliance Teknologies, K V Bath Type model. The Kinematic Viscometer is shown in Fig. 6. **ASTM D445** is a standard test procedure for determining the kinematic viscosity of liquids. The instrument measures the efflux time of flow freely in seconds of a fixed volume of the fluid through specified marks given on the Cannon-Fenske routine type viscometer tube. The viscometer tube was cleaned with chromic acid to remove any possible traces of organic deposits. 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 viscometer was turned to normal position and cleaned. 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. 4.

$$\mathbf{KinematicViscosity} = \mathbf{Viscometerconstant} \times \mathbf{t} \quad (4)$$

Where,

t = flow time, s

Vis constant of the viscometer= 0.0336 cSt/s

Fig. 6 Kinematic viscometer

Calorific Value

The Calorific value (energy value or heating value) of a substance is the amount of heat released during the combustion of an unit weight.. The heat value is an important characteristic of a fuel since it is the heat produced by the fuel within the engine cylinder which enables the engine to work. **ASTM D240** is a standard test procedure for determining the heating values.

Higher calorific value of fuel was determined by a closed vessel called bomb calorimeter shown in Fig. 7. The bomb is made of acid-resisting stainless steel which is capable of withstanding high pressure, heat and corrosion. The cover of the bomb carries the oxygen valve for admitting oxygen and a release valve for exhaust gases. A carrier ring, carried by the ignition rods supports the silica crucible, which in turn holds the sample of fuel under test. There was an ignition wire which dips into the crucible. It was connected to the battery, which was heated by passing current through it and the fuel was ignited. The fuel was burnt at a constant volume and under a high pressure.

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. 5.

$$\mathbf{H_c} = \frac{\mathbf{(m_w+m_e)C_w(t_2-t_1)}}{\mathbf{m_f}} \quad (5)$$

Where,

H_c= Heat of combustion of the fuel sample, kJ/kg;

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

Fig 7 Bomb Calorimeter

Flash Point and Fire Point

Diesel fuel flashpoints are measured with **ASTM D93**. Flash point of a fuel is defined as the lowest temperature at which a combustible mixture can be formed above the liquid fuel. The flash point is the only property, which is considered in assessing the overall flammability hazard of a material. It indicates the possible presence of highly volatile and inflammable materials in relatively non-volatile or non-flammable material. A material with about 90^0 C or higher flash point is considered as non-hazardous from storage and fire-hazard point of view. A Pensky Martens closed cup type apparatus was used in the study for determination of flash point is shown in Fig. 8

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 temperature at which it gives a flash of light was recorded as flash point. At the flash point (a lower temperature) a substance will ignite briefly, but vapour might not be produced at a rate to sustain the fire.

The fire point was recorded at that temperature when the fuel gives sufficient vapour that catches fire at least for five seconds. In general the fire point can be assumed to be about 10^0 C higher than the flash point.

Fig. 8 Bomb Calorimeter

Cloud Point and Pour Point

Cloud point refers to the temperature at which a cloud or haze of wax crystals appear at the bottom of the test jar when chilled under prescribed conditions. The pour point is defined as the temperature at which the fuel ceases to flow.

The presence of solidified waxes thickens the oil and clogs fuel filters and injectors in engines. The wax also accumulates on cold surfaces and forms an emulsion with water. Therefore cloud point indicates the tendency of the oil to plug filters or small orifices at cold operating temperatures. In crude or heavy oils cloud point is synonymous with wax appearance temperature or wax precipitation temperatures.

The standard used for cloud point and pour point of fuel were determination as per **ASTM D 97.** Theapparatus 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.The apparatus and the procedure for the pour point where same as the cloud point only 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. The Cloud point and pour point determination apparatus is shown in Fig. 9.

Fig, 9. Cloud point and pour point apparatus

Cetane Number

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[1-2].

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.

The Cetane number was determined by using **ASTM D 613** methods.

$$\mathbf{CetaneIndex} = \mathbf{46.3} + \frac{\mathbf{5458}}{\mathbf{s.v}} - (\mathbf{0.225\ x\ i.v})(6)$$

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

It is often difficult to measure the cetane number for vegetable oil and biodiesel. Cetane number is nearly

equal to cetane index as per the equation given below.:
Cetane Number = Cetane Index – (1.5 to +2.6)

Carbon Residue

Carbon residue is the non-volatile residue left when fuel is heated to a high temperature in the absence of air. It gives an indication of the coke forming tendencies of the fuel at higher temperature. The carbon residue of the fuel correlates with the amount of carbonaceous deposits formed in the combustion chamber of the engine[3]. Higher carbon residue value indicates more carbon deposits in the combustion chamber. So, fuels having less carbon residue are preferred for engine operation. The test was conducted by using **ASTM D 189** procedures and the Conradson's apparatus shown in Fig. 10.

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 skidmore 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. Thereafter, the burner was removed and allowed to cool until no smoke appeared. 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 given below:

$$\mathbf{Carbonresidue}\ (\%) = \frac{\mathbf{massofcarbonresidue,g}}{\mathbf{massoftestsample,g}} \times \mathbf{100} \quad (7)$$

Fig. 10. Conradsons' Apparatus

Copper Strip Corrosion Test

Many of the compounds in diesel fuel can be corrosive. The corrosiveness of a fuel is measured using the copper strip corrosion test, which follows **ASTM D 130**. 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 used in the laboratory was Copper Strip with heating bath [4-5].
Copper Strip Classification is given in TABLE I.

TABLE I Copper Strip Classifications

Classification	Designation	Description
1	Slight Tarnish	**1a** Light orange, almost the same as freshly polished strip **1b** Dark orange
2	Moderate Tarnish	**2a** Claret red **2b** Lavender **2c** Multi colored with lavender blue or silver, or both, overlaid on claret red **2d** Silvery **2e** Brassy or gold
3	Dark Tarnish	**3a** Magenta Overcast on brassy strip **3b** Multicoloured with red and green showing (peacock), but not gray

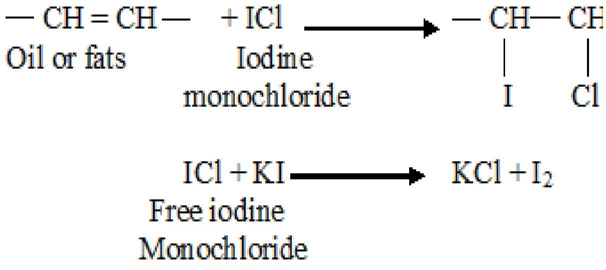
4	Corrosion	**4a** Transparent black, dark gray or brown with peacock green barely showing **4b** Graphite or lustreless black **4c** Glossy or jet black

Fig. 11. Copper strip corrosion apparatus

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 [6]

The oil contains both saturated and un-saturated fatty acids. 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 thio 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 :



$$-CH=CH- + ICl \longrightarrow -\underset{I}{CH}-\underset{Cl}{CH}-$$

$$ICl + KI \longrightarrow KCl + I_2$$

$$Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + NaI$$

Sodium thiosulphate　Sodium tetrathionate

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

$$\mathbf{IodineValue} = \frac{\mathbf{V \times N \times 0.1269}}{\mathbf{Massofoilingram}} \times \mathbf{100} \qquad (8)$$

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 Number

Saponification value or saponification number (also referred to as “sap”) 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.

$$\mathbf{SaponificationValueofanoil(mgKOH)} = \frac{\mathbf{(Blankexperiment - Titrateexperiment)}}{\mathbf{Massofoil\,(g)}} \times \mathbf{28.06} \qquad (9)$$

Where,
1 ml of 0.5 N HCl = 28.06 mg KOH

Moisture Content

Karl Fischer Moisture Analyzers totally eliminate the troublesome procedures involved with conventional water content. in particular the determination of oil or water content in crude oils. Since they incorporate a special electrolysis current control system, moisture analyzers ensure the fast and accurate measurement of even trace water content. Moisture analyzers are available in laboratory (LKF) and portable models (PKF). The instrument used for this test in the laboratory is of Veego Instruments Corporation, Mumbai, of Veego/Matic D model [7]

The fundamental principle is based on the modified Bunsen reaction between iodine and sulphur dioxide in a non-aqueous medium using primary alcohol (methanol) as a solvent and a base as the buffering agent. The tritrant generally consists of an alcohol (ROH), base, SO_2 and a known concentration of I_2. This composition is commonly known as Karl Fischer (KF) reagent. Due to carcinogenic effect of pyridine the reagents most frequently used are pyridine free and contain imidazole or primary amines instead. When the titrant is added to the solvent (methanol) in the titration vessel oxidation of SO_2 by I_2 occurs. In the course of reaction water and iodine is consumed in a 1:1 ratio. Once all the water present is consumed the presence of excess iodine is detected voltametically by the titrator's indicator electrodes. This indicates the end point of titration.

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. The signal from the electrode was continuously compared between end point voltage, which was prefixed and the voltage when the sample solution was free from moisture. 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. as per [56] given below:
.

$$\mathbf{MoistureContent, \% = \frac{VolumeofKFreagentconsumed, ml \times titrevalue, \frac{mg}{ml} oftitre}{Samplevolume} \times 100}$$
(10)

III. RESULTS AND DISCUSSIONS

Density, Viscosity and Calorific value of sample fuels is mentioned in TABLE II.

TABLE II Densities, Viscosity and Calorific Value of Sample Fuels

Sl.no.	Name of Oil/Biodiesel	Density (gm/cc)		Kinematic Viscosity (cSt)		Calorific value (MJ/Kg)
		Oil	Biodiesel	Oil	Biodiesel	Biodiesel
1	Mahua	0.90	0.88	37.18	4.98	39.10
2	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 higher densities of crude oil and biodiesel compared to diesel may be attributed to the higher molecular weights of triglyceride molecules present in them. If the specific gravity was more, the fuel was more concentrated and more fuel was likely to be delivered on mass basis, which led to higher fuel consumption. The density of tested biodiesel samples were within the limits (0.86-0.90 gm/cc) prescribed in the biodiesel standards.

The high value of kinematic viscosity of crude oil hinders its smooth flow in the injectors of the engine. The **kinematic viscosity values** of crude oil were 13 to 16 times that of diesel. The higher viscosity of these oils in contrast to diesel may be due to the greater inter molecular attraction of the long chains of its glyceride molecules. The kinematic viscosity of biodiesels was within the limits prescribed by the ASTM, EN, and BIS standards.

The **calorific values** of, mahua, biodiesel were 15.93% lower than diesel. This might be due to the difference in chemical composition or the difference in percentage of carbon and hydrogen content or the presence of oxygen molecule in the molecular structure of biodiesels. The oxygen molecule present unites with hydrogen of the oil to form water vapour even before secondary air or oxygen supplied for combustion reacts with hydrogen.

Flash Point, Fire Point, Cloud Point and Pour Point of Sample Oil/Biodiesel is shown in Table III

TABLE III Flash Point, Fire Point, Cloud Point and Pour Point of Sample Oil/Biodiesel

Sl. no.	Name of Oil/ Biodiesel	Flash Point (^{0}C)		Fire Point (^{0}C)		Cloud Point (^{0}C)		Pour Point (^{0}C)	
		Oil	Bio-diesel	Oil	Bio-diesel	Oil	Bio-diesel	Oil	Bio-diesel
1	Mahua	234	168	245	180	2	14	-3	3
2	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^{0}C for the diesel. The flash point of the sample biodiesel obeys the ASTM, EN and BIS standard. Generally, a material with about 90^0 C or higher flash point was considered as non-hazardous from storage and fire-hazard points of views.

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

The cloud point and pour point were of concern in order to measure the performance of fuels under cold temperature conditions. However, 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. This might be due to the presence of more saturated fatty acids (palmitic and stearic acid) or wax, which begins to crystallize with decrease in temperature.

Cetane No. and Carbon residue of sample fuel is mentioned in TABLE IV.

TABLE IV Cetane No. and Carbon Residue of Sample Biodiesel

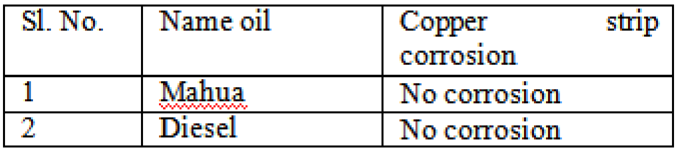
Sl. No	Biodiesel	Cetane No	Carbon residue, %
1	Mahua	55	0.22
2	Diesel	50	0.15

Cetane number is the most important measure of ignition characteristics of diesel and /or biodiesel fuels, since it directly pertains to ignition within compression ignition engines. The cetanes no of biodiesels were more than that of diesel which led to better combustion. The Cetane No. of sample biodiesel follows the **ASTM, EN, and BIS** standards

The carbon residue is non-volatile when fuel is heated to a high temperature in the absence of air. The higher this value, the greater the carbon deposits in the combustion chamber. The carbon residue values of biodiesel were found more than diesel. Carbon residue of sample biodiesel was within the specified limit of **0.3%.**

Copper strip corrosion test results of sample fuel are given in TABLE V

TABLEV Copper Strip Corrosion Test Results of Sample Oils



Sl. No.	Name oil	Copper strip corrosion
1	Mahua	No corrosion
2	Diesel	No corrosion

Copper-strip corrosion test measures relatively the degree of corrosion. It indicates the presence of sulphur compounds. Copper and copper compounds tend to be particularly susceptible to chemical attack. The corrosiveness of a fuel has implications on storage and usage of the fuel. Iodine value, Saponification value and Moisture Content of sample fuel are mentioned in TABLE VI.

TABLE VI Iodine Value, Saponification Value and Moisture Content of Sample Fuels

Sl. no.	Name of sample fuel	Iodine Value (g I_2/100g)	Saponification Value (mg KOH/g)	Moisture content (%)
		Biodiesel	Biodiesel	Biodiesel
1	Mahua	70	194	0.04
2	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. This value gives an indication of the nature of fatty acids constituent of the fat. Biodiesel and its blends were susceptible to growing microbes when water was present in fuel. It affected the quality of biodiesel.

The **moisture contents** of seed given above were removed during esterification and transesterification process. The moisture content of oil should be within the limit i.e., **< 0.05 w/w%.**

ASTM D975, ASTM6751, EN 14214 and BIS 15607 specifications of diesel and biodiesel fuels are given in TABLE VII

TABLE VII ASTM D975, ASTM6751, EN 14214 and BIS 15607 Specifications of Diesel and Biodiesel Fuels

Property Specification	Unit	Diesel	Biodiesel		
		ASTM D975	ASTM D6751	EN 14214	BIS 15607
		Limits	Limits	Limits	Limits
Acid value	mg/KOH/g	-	0.5 max	0.5 max	0.5 max
Density at 15^0C	kg/m^3	820-860	880	860-900	870-900
Kinematic viscosity at 40^0C	mm^2/s	2.0 to 4.5	1.9-6.0	3.5-5.0	3.5-5.0
Calorific value	kJ/kg	-	-	-	-
Flash Point	^{0}C	60 to 80	130 min	101 min	120 min
Fire point	^{0}C	-	-	-	-
Cloud point	^{0}C	-15 to 5	-3 to -12	-	-
Pour point	^{0}C	-35 to -15	-15 to -16	-	-
Cetane No.	-	46	47 min	51 min	51 min
Carbon residue	%	0.2 max	0.050 max	0.3 max	0.05 max
Copper corrosion	-	Class 1 max	No. 3 max	Class 1	
Iodine value	g I_2/100g	-	-	120 max	115
Saponification value	-	-	-	-	-
Moisture Content	-	-	-	0.05 max	-

IV. CONCLUSION

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 crude oil were 13 to 16 times that of diesel.

The **calorific values** of, mahua, biodiesel were 15.93% lower than diesel.

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 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.

No corrosion was found in the tested fuels.

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.

The moisture content of oil is within the limit i.e., < **0.05 w/w%.**

REFERENCES

[1]. K. Krisnangkura, “A Simple Method for Estimation of Cetane Index of Vegetable Oil Methyl Esters,”JAOCS, vol. 63, no. 4, pp. 552-553, 1986.

[2]. M. Jesikha, “Fatty Acid Methyl Esters Characteristic and Esterification of Some Vegetable Oils for Production of Biodiesel,” International Journal of Engineering and Science, vol. 1, no. 12,pp. 50- 53, Dec.2012.

[3]. V. Patel, “Cetane Number of New Zealand Diesel Report,” Office of Chief Gas Engineer, Energy Inspection Group, Ministry of Commerce Press, Wellington, New Zealand, 1999.

[4]. S. S. Ragit, “Process standardization, characterization and experimental investigation on the performance of biodiesel fuelled C.I engine,” Ph.D. dissertation, Dept. Mechanical Engineering, Thapar University, Patiala, 2011.

[5]. J. V. Gerpen et al., “Biodiesel Analytical MethodsAugust 2002–January 2004,” NREL 1617 Cole Boulevard, Golden, Colorado, July. 2004. http://www.osti.giv/bridge.

[6]. M. Jesikha, “Fatty Acid Methyl Esters Characteristic and Esterification of Some Vegetable Oils for Production of Biodiesel,” International Journal of Engineering and Science, vol. 1, no. 12, pp. 50- 53, Dec.2012.

[7]. P. C. Jena, “Production of biodiesel from mixture of vegetable oils and its performance in dieselengine”. Ph.D. dissertation, Dept. Agricultural and food engineering department, IIT, Kharagpur, 2011.