IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE)
e-ISSN: 2278-1684,p-ISSN: 2320-334X,
PP 77-83
www.iosrjournals.org

Bio-Diesel Production from Mahua (Madhuca Indica) and its Characterization through a TLC Analysis

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Abstract: *In search of a suitable fuel alternative to fast depleting fossil fuel and oil reserves and in serious consideration of the environmental issues associated with the extensive use of fuels based on petrochemicals, research work is in progress worldwide, Non edible mahua oil was also used as a SGBF candidate in the present study. Double stage transesterification was used for Non Edible Mahua Oil. Through a TLC analysis is found that the FFA and TG are totally converted in to the FAME. The optimum combinations for reducing the acid level of mahua oil to less than 1% after pretreatment was 0.32% v/v methanol to oil ratio, 1.24% v/v H2SO4, catalyst and 1.26 hours reaction time at 65 C. After the pretreatment of mahua oil, transesterification reaction was carried out with 0.25 v/v methanol to oil ratio (6:1 molar ratio) and 0.7% w/v KOH as an alkaline catalyst to produce biodiesel. The fuel properties of mahua biodiesel so obtained compiled the requirements of both the ASTM and EN standards for biodiesel.*

Keywords: *Alternative fuel, Bio-Diesel, mahua oil, TLC Analysis, Transesterification.*

I. Introduction

Bio fuels are drawing increasing attention worldwide as substitutes for petroleum-derived transportation fuels to address energy cost, energy security and global warming concerns associated with liquid fossil fuels. The term bio fuel is used here to mean any liquid fuel made from plant material that can be used as a substitute for petroleum-derived fuel. Second-generation fuels are generally those made from non-edible lignocellulosic biomass, either non-edible residues of food crop production (e.g. corn stalks or rice husks) or non-edible whole plant biomass (e.g. grasses or trees grown specifically for energy) or waste product of refinery oil industry. Biodiesel is defined by ASTM International as a fuel composed of monoalkyl esters of long-chain fatty acidsderived from renewable vegetable oils or animal fats meeting the requirements of ASTM D6751 (ASTM 2008a). Vegetable oils and animal fats are principally composed of triacylglycerols (TAG) consisting of longchain fatty acids chemically bound to a glycerol (1, 2,3- propanetriol) backbone. The chemical process by which biodiesel is prepared is known as the transesterification reaction.

II. Materials And Method

Mahua oil is obtained from the kernel of mahua tree (Madhuca indica), which is a medium to large tree found in most parts of India. Mahua seed contain 30-40 percent fatty oil called mahua oil. Mahua oil was obtained from Agrawal oil mills near Udaipur, Rajasthan, India. Before preparation of bio diesel the property of oil likes chemical composition of oil, acid value, iodine value and FFA etc should be known for establishing procedure for bio diesel. Mahua biodiesel was prepared using two step processes, in which initial step is acid esterification process and second one is transesterification process. Experiments were conducted in a laboratory-scale setup. All chemicals including methanol (99.5%) and sulfuric acid (99% pure) were of analytical reagent (AR) grade. The KOH in pellet form was used as a base catalyst for transesterification reaction. Crude unrefined mahua oil was brownish yellow in colour. This oil had an initial acid value of 20.34 mg KOH/gm of oil corresponding to a FFA level of 10.17%.

III. Preperation And Chracterrization

"Biodiesel is defined as mono-alkyl ester of long chain fatty acid derived from vegetable oil or animal fats." Non edible Mahua oil and acid oil (waste product) from ground nut oil industry is used as a second generation bio fuel. Mahua biodiesel was prepared using two step processes. Crude unrefined mahua oil was brownish yellow in colour. This oil had an initial acid value of 20.34 mg KOH/gm of oil corresponding to a FFA level of 10.17%.

3.1 Transesterification process:
a) Preheating of esterified oil:
b) Transesterification process using alkaline catalysis
c) Separation of Glycerol from Transesterified oil (FAME)
d) Neutralizing Transesterified oil (FAME)

3.2 Acid esterification:
a) Preheating of raw oil
b) Esterification process using acid catalysis
c) Separation of Methanol from Esterifies oil
d) Neutralized Esterifies oil

Pre-heated raw mahua oil mixed with methanol (0.32% v/v) using H_2SO_4 as catalyst (1.24% v/v) for 1.45 hours reaction at 65^0 C temperature.

Fig. 1 Pre-heating of Raw oil

Fig. 2 Esterification Process Using Acid Catalysis

After esterification process 94.14% reduction in acid value of mahua oil which is suitable for the main transesterification reaction.

Fig. 3 Transesterification process

Fig. 4 Neutralized Esterified Oil

Alkaline-catalyzed transesterification was run using a pre-treated sample that had an acid value of 1.19 mg KOH/gm of oil. A 0.25 %v/v methanol-to-oil and an alkaline catalyst (0.5% w/v KOH) were used for the reaction for 45 minute.

Fig.5 Separation of Methanol from Esterifies Oil

Fig.6 Neutralizing Transesterified Oil (FAME)

IV. Tlc Analysis Of Bio Diesel With Respect To Mahua Oil

TLC (Thin Layer chromatography) is commonly used for qualitative analysis. It is fast and effective, and also can be used to verify the conversion of the oil during the reaction. Some solvent systems and detecting reagents commonly used for evaluation of starting material conversion during the reaction are shown below.
Solvent systems and detecting reagents parameters by (%v/v/v) for TLC [15]:

(a) Hexane: ethyl acetate: acetic acid (90:10:1) iodine vapour
(b) Iso hexane: diethyl ether: acetic acid (80:20:1) ultraviolet light
(c) Chloroform: petroleum ether (1:3)
(d) Petroleum ether: diethyl ether: acetic acid (85:15:1) iodine vapour
(e) Hexane: ethyl acetate: acetic acid (90:10:1) sulphuric acid: methanol 1:1

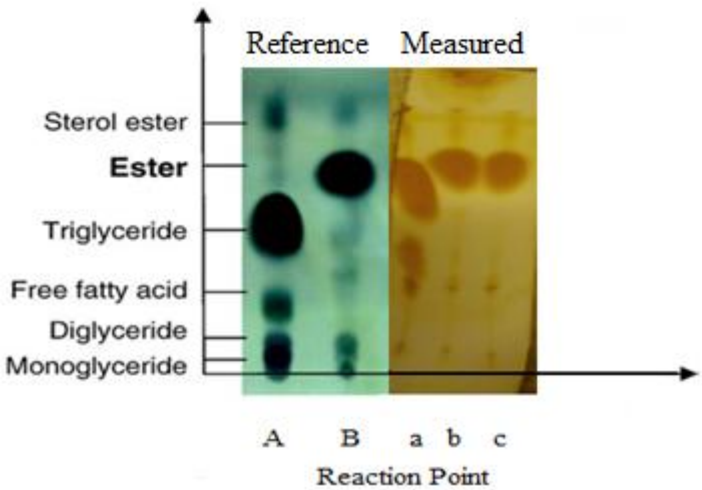
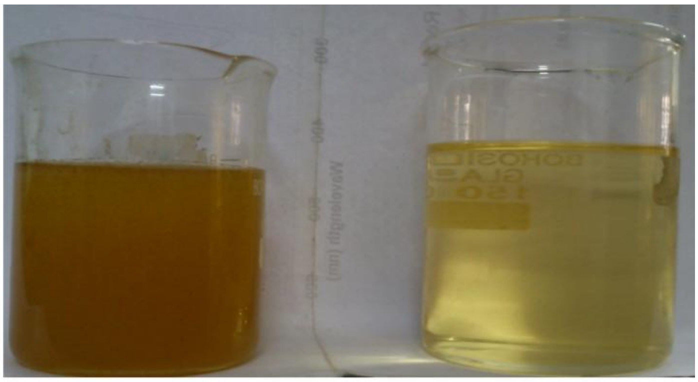


Fig. 7 TLC results of product composition at different reaction point for transesterified mahua oil.

The results analyzed by TLC for neat mahua oil were shown in figure 7. The changes in product compositions with reaction during the transesterification of the oils and the distribution of various components in the reaction system can be clearly seen. In above figure point A and B is consider as a reference point and point a show initial mahua oil point, b indicates after esterification point.



(A) Raw mahua oil (B) Mahua Bio diesel

Fig 8. Raw Mahua Oil and Mahua Oil Bio Diesel

V. Characterisation of second generation bio diesel

5.1 Acid value

A value indicating the amount of free acid present in a substance, equal to the number of milligrams of potassium hydroxide needed to neutralize the free fatty acids present in one gram of fat or oil also called acid number. Figure 9-10 shows acid value of different Oils and biodiesel.

AV = (v - b) x N x 56.1 / w

Where, v is the titration volume in ml
b is the blank in ml
N is the normality of the KOH solution
w is the weight of sample in gm

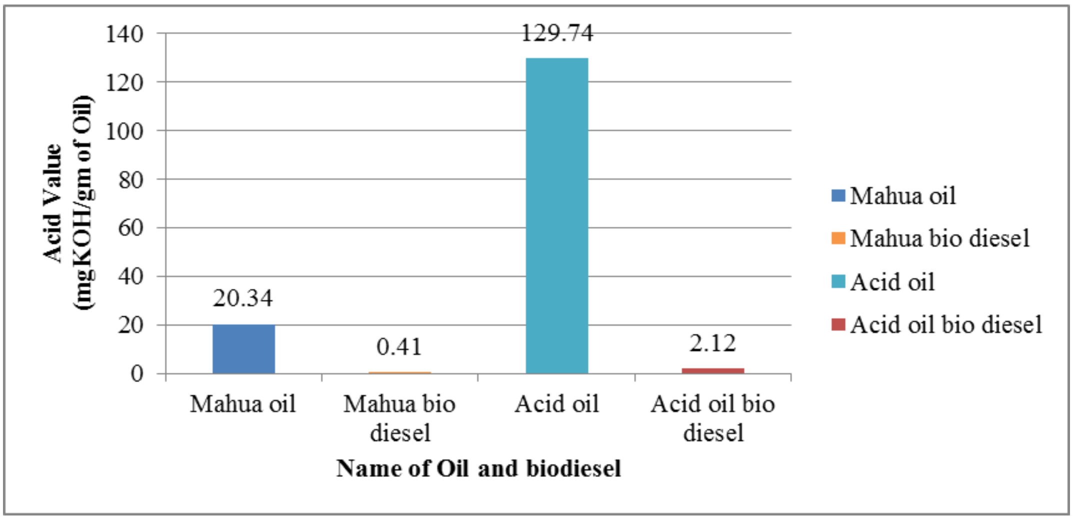


Fig. 9 Acid Value of different Oils and biodiesel

5.2 Free fatty acid (FFA)

Figure FFA of different Oils and biodiesel. FFA% is the weight to weight ratio of FFA found in an oil sample. The weight of an oil sample divided into the weight of the FFA in that sample. To calculate FFA% from an acid value the formula is:

AV = 1.99 FFA%

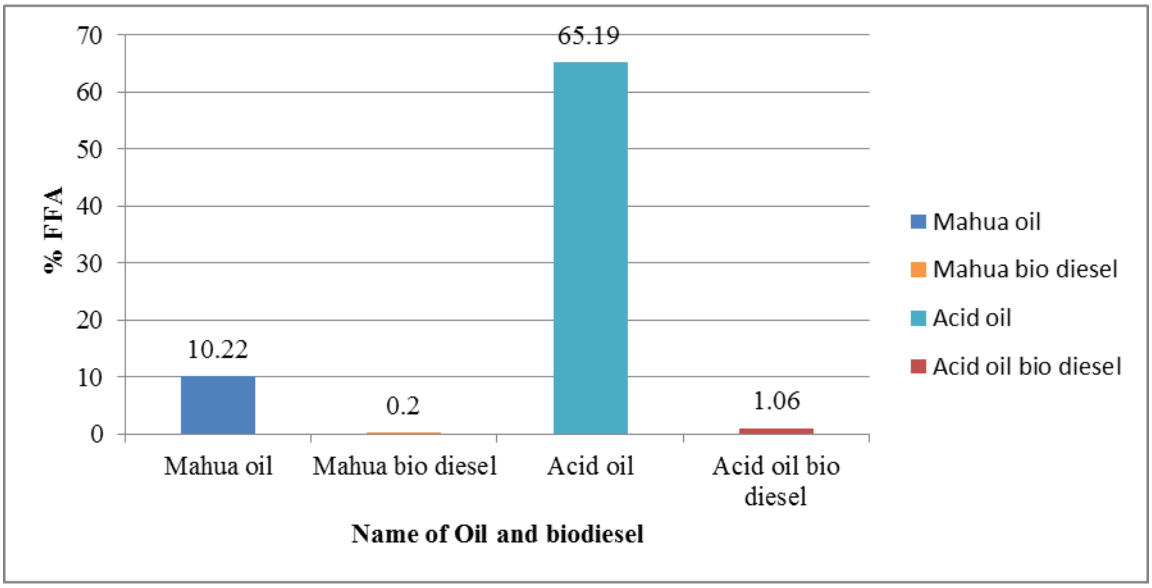


Fig. 10 FFA of different Oils and biodiesels

5.3 Kinematic Viscosity

For present work Viscosity is measure with help of redwood viscometer which shows on figure 11-12. In red wood viscometer cup is fill up to oil and which is surrounded by water bath. Kinematic viscosity is measure at a 400C. With the help of heater water is heated and reached temperature around 40-450C. Oil was stop through needle in cup pin hole when temperature is reached 400C needle is remove from the pin hole and collect in to collector for 50 ml and time was measured with the help of stop watch.

Fig. 11 Red wood Viscometer

The viscosity of an engine fuel is one of the most critical fuel features. It plays a dominant role in the fuel spray, mixture formation and combustion process. The high viscosity interferes with the injection process and leads to insufficient fuel atomization. Moreover, the mean diameter of the fuel droplets from the injector and their penetration increases with increasing fuel viscosity. The inefficient mixing of fuel with air contributes to incomplete combustion in the engine. In addition to all these, high viscosity can cause early injection due to high line pressure, which moves the combustion of the fuel closer to top dead centre, increasing the maximum pressure and temperature in the combustion chamber. Figure 3-22 shows kinematic viscosity of different oils and biodiesel obtained in our laboratory.

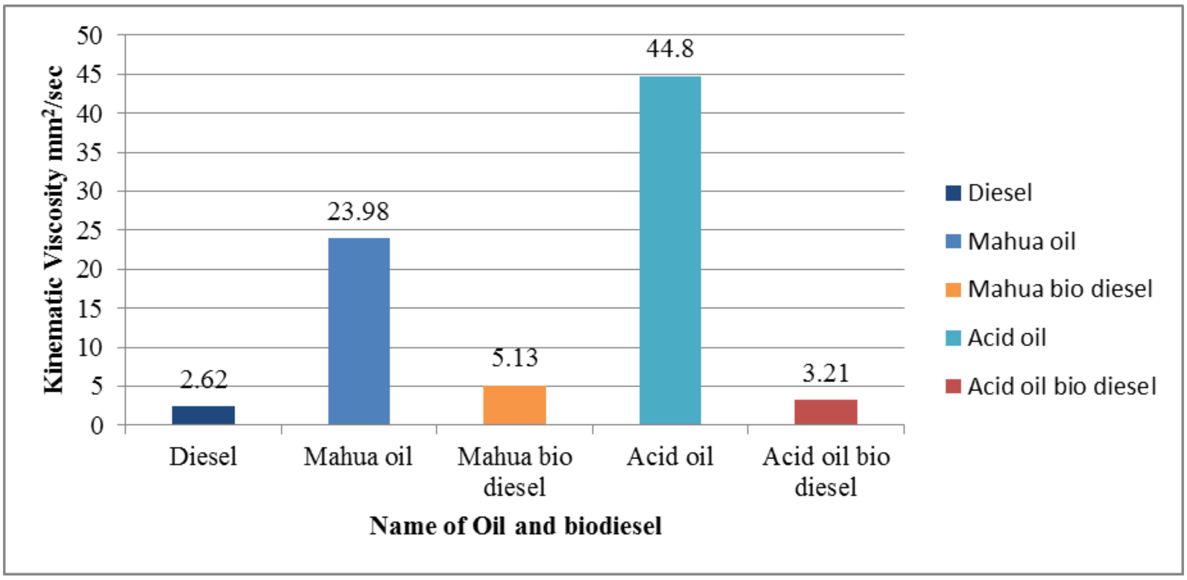


Fig. 12 Kinematics Viscosity of different Oils and biodiesels

Viscosity of any fuel is related to its chemical structure. Viscosity increases with the increase in the chain length and decreases with the increase in the number of double bonds (un-saturation level) [20].

VI. Conclusions

Biodiesel from Mahua oil was obtained by two stage transesterification process. In order to synthesize acid oil no standardize method was available in the literature. The conventional method fails to form ester using acid oil as its FFA value is too high. A suitable solid acid catalyst was developed with the help of chemistry department to successfully synthesize the acid oil for biodiesel formation. It is important to note that acid oil is much cheaper as compared to any other available feedstock for biodiesel and is generally considered as a waste product of oil refinery. In view of increased cost of first generation biofuel and also some non-edible oil candidate like Pongamia and Jatropha, the waste product of oil industry i.e. acid oil which is available at much cheaper cost and its utility as a biofuel for CI engine promises better prospectus for biodiesel industry. Mahua oil biodiesel too have better prospectus as its tree has abundant seeds and oil seed yield is also very high.

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