

# Production and Characterization of Biofuel Produced by Non-Edible Oil

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**Abstract:** The present-day internal combustion engines are operating essentially on petroleum-based fuels, which are non-renewable in nature and lead to depletion in short period due to its indiscriminate use in different fuels. Renewable agriculture based, non-edible oils like pongamia, mahua (*Maduca Indica*), neem, *jatropha* oils etc. can be used as an alternative fuel in CI engines. Biodiesel production from Mahua seed was experimentally investigated in the present study. Expeller method was employed to extract mahua oil from its seed and was subjected to two stage transesterifications due to the presence of more than 18% of free fatty acid content. In the primary stage, the FFA content was reduced to less than 2% by acid esterification using concentrated H<sub>2</sub>SO<sub>4</sub> and methanol and followed by base catalyzed transesterification to convert the mahua oil into biodiesel. The properties like density, viscosity, and Calorific value, flash and fire point were analyzed and compared with another prominent biodiesel.

**Index Terms** - *Maduca Indica*, Transesterification, Free Fatty Acid.

## I. INTRODUCTION

Biodiesel is a form of fuel which is produced from vegetable oil by transesterification process. Inventor of biodiesel is G.Chavanne in 1937 from palm oil, and the inventor of ethanol is Johann Tobias Lowitz in 1796. Feedstock includes vegetable oil such as waste vegetable oil and other oils like soya, cotton seed, rice bran etc. In America already 30 to 40% transportation dependence is on biofuel so that they are reducing the pollution day by day. Efficiency and the flash point of the biofuel is more compared to diesel and gasoline. Use of biofuel will provide up to 12% GST benefits. APJ Abdul kalam has started the Biofuel park in 2007 at Hassan. Henry Ford told "We can get fuel from fruits" in 1925. World BIOFUEL DAY is celebrated annually on 10<sup>th</sup> august to create awareness of various sustainable, biodegradable and renewable fuels and the theme of 2020 was "Biofuels towards Atmanirbhar Bharat".

Biodiesel is an alternative fuel made from renewable biological sources such as vegetable oils both (edible and non-edible oil) and animal fats According to the US standard specification for biodiesel (American Society for Testing and materials (ASTM) 6751), biodiesel is defined as a fuel comprised of mono alkyl esters of long chain fatty acids from vegetable oils or animal fats [1]. The dominant bio-diesel production process, namely transesterification, typically involves the reaction of an alkyl-alcohol with a long chain ester linkage in the presence of a catalyst to yield mono-alkyl esters (biodiesel) and glycerol.

There are three basic routes to ester production from oils and fats:

- i. Base catalyzed transesterification of the oil with alcohol.
- ii. Direct acid catalyzed esterification of the oil with methanol.
- iii. Conversion of the oil to fatty acids, and then to alkyl esters with acid catalysis.

The majority of the alkyl esters produced today is done with the base catalyzed reactor because it is the most economic for several reasons:

- I. Low temperature (150 F) and pressure (20 psi) processing.
- ii. High conversion (98%) with minimal side reactions and reaction time.
- iii. Direct conversion to methyl ester with no intermediate steps.
- iv. Exotic materials of construction are not necessary.

## II. LITERATURE REVIEW

Ulf Schuchardt et. al. (1998) reviewed the transesterification of vegetable oils with methanol as well as the main uses of the fatty acid methyl esters. The general aspects of this process and the applicability of different types of catalysts (acids, alkaline metal hydroxides, alkoxides and carbonates, enzymes, and non-ionic bases, such as amines, amidines, guanidine's and triamino(imino)phosphoranes) are described. Special attention is given to guanidine's, which can be easily heterogenized on organic polymers. However, the anchored catalysts show leaching problems. New strategies to obtain non-leaching guanidine containing catalysts are proposed. Finally, several applications of fatty acid esters, obtained by transesterification of vegetable oils, are described.

A.S Ramadhas, S.Jayaraj, C Muraleedharan(2004) Currently, most of the biodiesel is produced from the refined/edible type oils using methanol and an alkaline catalyst. However, large amount of non-edible type oils and fats are available. The difficulty with alkaline-esterification of these oils is that they often contain large amounts of free fatty acids (FFA). These free fatty acids quickly react with the alkaline catalyst to produce soaps that inhibit the separation of the ester and glycerine. A two-step transesterification process is developed to convert the high FFA oils to its mono-esters. The first step, acid catalysed esterification reduces the FFA content of the oil to less than 2%. The second step, alkaline catalysed transesterification process converts the products of the first step to its monoesters and glycerol. The major factors affect the conversion efficiency of the process such as molar ratio, amount of catalyst, reaction temperature and reaction duration is analysed. The two-step esterification procedure converts rubber seed oil to its methyl esters. The viscosity of biodiesel oil is nearer to that of diesel and the calorific value is about 14% less than that of diesel. The important properties of biodiesel such as specific gravity, flash point, cloud point and pour point are found out and compared with that of diesel. This study supports the production of biodiesel from unrefined rubber seed oil as a viable alternative to the diesel fuel.

**Shashikant Vilas Ghadge, Hifjur Rahman (2005) IIT Kharagpur** A technique to produce biodiesel from mahua oil (*Madhuca indica*) having high free fatty acids (19% FFA) has been developed. The high FFA level of mahua oil was reduced to less than 1% by a two-step pre-treatment process. Each step was carried out with 0.30–0.35 v/v methanol-to-oil ratio in the presence of 1% v/v H<sub>2</sub>SO<sub>4</sub> as an acid catalyst in 1-hour reaction at 60°C. After the reaction, the mixture was allowed to settle for an hour and methanol–water mixture that separated at the top was removed. The second step product at the bottom was transesterified using 0.25 v/v methanol and 0.7% w/v KOH as alkaline catalyst to produce biodiesel. The fuel properties of mahua biodiesel were found to be comparable to those of diesel and conforming to both the American and European standards.

**Rushang M.Joshi Michael J.Pegg(2006)**The dynamic viscosities of biodiesel derived from ethyl esters of fish oil, no. 2 diesel fuel, and their blends were measured from 298 K down to their respective pour points. Blends of B80 (80 vol.% biodiesel–20 vol.% no. 2 diesel), B60, B40 and B20 were investigated. All the viscosity measurements were made with a Bohlin VOR Rheometer. Cloud point and pour point measurements were made according to ASTM standards. Arrhenius equations were used to predict the viscosities of the pure Biodiesel (B100), no. 2 diesel fuel (B0) and the biodiesel blends (B80, B60, B40, and B20) as a function of temperature. The predicted viscosities agreed well with measured values. An empirical equation for calculating the dynamic viscosity of these blends as a function of both temperature and blend has been developed. Furthermore, based on the kinematic viscosity and density measurements of B100 up to 573 K by Tate et al. [Tate RE, Watts KC, Allen CAW, Willkie KI. The viscosities of three biodiesel fuels at temperatures up to 300 °C. Fuel 2006; 85:1010–5; Tate RE, Watts KC

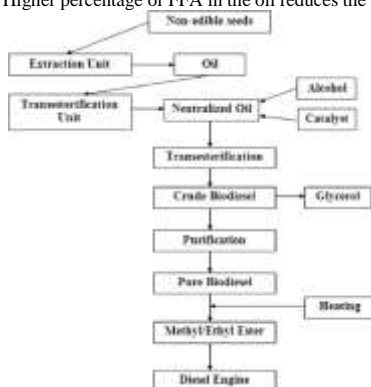
### III. METHODOLOGY

**Transesterification** (alcoholysis) is the chemical reaction between triglycerides and alcohol in the presence of catalyst to produce mono-esters. The long and branched chain triglyceride molecules are transformed to mono-esters and glycerin. Transesterification process consists of a sequence of three consecutive reversible reactions. That is, conversion of triglycerides to diglycerides, followed by the conversion of diglycerides to monoglycerides. The glycerides are converted into glycerol and yielding one ester molecule in each step. The properties of these esters are comparable to that of diesel. The overall transesterification reaction can be represented by the following reaction scheme.



#### Production of Biodiesel from Neem oil

Stoichiometrically, three moles of alcohol are required for each mole of triglyceride, but in practice a higher molar ratio is employed in order to displace the equilibrium for getting greater ester production. Though esters are the desired products of the transesterification reactions, glycerin recovery also is important due to its numerous applications in different industrial processes. Commonly used short chain alcohols are methanol, ethanol, propanol and butanol. The yield of esterification is independent of the type of alcohol used. Therefore, the eventual selection of one of these three alcohols will be based on cost and performance considerations. The methanol is used commercially because of its low price. Alkaline hydroxides are the most effective transesterification catalysts as compared to acid catalysts. Potassium hydroxide and sodium hydroxide are the commonly used alkaline catalysts. Alkaline catalyzed transesterification of vegetable oils is possible only if the acid value of oil is less than 4. Higher percentage of FFA in the oil reduces the yield of the esterification process.



**Processing of Biodiesel from non edible oil (Neem Oil):**

Free fatty acid (FFA) percentage in neem oil is very high. There are many methods to find out the free fatty acid percentage content in oil. Simple titration with the KOH is a simple method.

For titration first 0.1 to 10 g of oil was weighed and dissolved in about 10 ml of a suitable solvent. Iso Propyl Alcohol is normally used solvent. It was heated gently for some time. A small drop of indicator was added. Phenolphthalein was used as indicator. Then the solution was titrated with KOH. The amount of KOH required, in milligram (mg) to neutralizing the free fatty acid in one gram of oil expressed as a number is known as acid number. From acid number the free fatty acid present in the oil can be calculated.

**Esterification**

The objective of this study is to develop a process for producing biodiesel from non-edible neem oil. The process consists of two steps namely, acid esterification and alkaline esterification.

**(a) Acid Esterification:** The first step reduces the FFA value of crude neem oil to about 2% using acid catalyst

**(b) Alkaline Esterification:** After removing the impurities of the product of first step, it is transesterified to monoesters of fatty acids using alkaline catalyst. The parameters affecting the process such as alcohol to oil molar ratio, catalyst amount, reaction temperature and duration are analyzed.

**Esterification Setup:**

A round bottom flask is used as laboratory scale reactor for these experimental purposes. A hot plate with magnetic stirrer arrangement is used for heating the mixture in the flask. The mixture is stirred at the same speed for all test runs. The temperature range of 50-60 °C is maintained during this experiment.

**Acid Esterification**

One litre of crude neem oil requires 250 ml of methanol for the acid esterification process. The neem oil is poured into the flask and heated to about 50 °C. The methanol is added with the preheated neem oil and stirred for a few minutes. 6ml of sulphuric acid is also added with the mixture. Heating and stirring is continued for 3 hr at atmospheric pressure. On completion of this reaction, the product is poured into a separating funnel for separating the excess alcohol. The excess alcohol, with sulphuric acid and impurities moves to the top surface and is removed. The lower layer is separated for further processing (alkaline esterification).

**Effect of reaction temperature**

At room temperature the conversion efficiency is noted to be very low, even after 2 hrs of stirring. With increase in temperature the conversion takes place at a faster rate. The optimum temperature for this reaction is found to be in the range of 50-55 °C. At higher reaction temperatures, there is a chance of loss of methanol and increase in darkness of the product. High reaction temperature increases the production cost of biodiesel also.

**Alkaline Esterification**

Alkaline catalyzed esterification process uses the experimental setup of acid catalyzed pretreatment process. The products of first step are preheated to the required reaction temperature of 50±5 °C in the flask. Meanwhile, 10 gm of KOH is dissolved in 250 ml methanol and is poured into the flask. The mixture is heated and stirred for 2 hr. The reaction is stopped, and the products are allowed to separate into two layers. Glycerin, which is heavier, deposits at the bottom and the esterified neem oil is obtained at the top portion. The esterified neem oil is separated from the funnel and now it is ready for blending with diesel.

**Effect of reaction temperature**

The maximum yield of ester is obtained at the temperatures of 50±5 °C. The decrease in yield is observed when the reaction temperature goes above 55 °C. The reaction temperatures greater than 60 °C should be avoided, in the case of neem oil, because they tend to accelerate saponification of the glycerides by the alkaline catalyst before completion of the alcoholysis.

Commented [KS1]:

**IV. EXPERIMENTAL RESULTS**

Comparisons of Properties of neem biodiesel, mahua biodiesel & diesel :

	DIESEL	NEEM BIO DIESEL	MAHUA BIO DIESEL
Flash Point (0c)	66	165	162
Fire Point (0c)	79	172	170
Density (kg/m <sup>3</sup> )	866.4	949 kg/m <sup>3</sup>	898.6 kg/m <sup>3</sup>
Viscosity At 40 C	0.96X10 <sup>-5</sup> m <sup>2</sup> /sec	1.306X10 <sup>-5</sup> m <sup>2</sup> /sec	0.962X10 <sup>-5</sup> m <sup>2</sup> /sec

Blending :

TYPE OF BLEND	EMISSION TEST			
	CO	HC	CO <sub>2</sub>	NO
B10 [540ml D + 60ml N]	0.42	61.2	4.72	108.8
B10[540ml D + 60ml M]	0.46	74.4	5.78	128.8
B10[540ml D+30ml N+30ml M]	0.536	88	5.5	134
B20[480ml D + 120ml N]	0.48	94.2	5.26	142.4
B20[480ml D + 120ml M]	0.54	102.4	5.76	155.8
B20[480ml D+60ml N+60ml M]	0.48	90.2	5.34	131.8
B30[420ml D+90ml N+90ml M]	0.48	82.8	4.92	120.6
B0[600ml D]	0.786	80.2	5.04	125.2

**V. FURTHER IMPROVEMENTS**

Biodiesel has indeed a promising future in the Moroccan market and fits properly its ambitious renewable energy goals. The pilot design has been successfully made as well as the attempted automation of its flow. The feasibility study of our project have made us strongly believe in its success. Future work would mainly consist of designing a glycerol valorization plant to produce combustible pellets that can substitute coal. Moreover, rather than water washing, other biodiesel purification methods could be implemented in the future. In addition, an acid esterification prior to transesterification can be integrated in our design as well, in case of using WCO with a higher FFAs content (>2%).

**VI. CONCLUSION**

It is observed that there is no variation in peak pressures for neem oil blends and mahua biodiesel blends. But in case of neem oil blends, the peak pressures is highest at 200 bar. In case of neem biodiesel blends the peak pressure at 200 bar and 220 bar is almost same.

**VII. ACKNOWLEDGEMENT**

With great pleasure we were able to bring about this project on the "Production And Characterization Of Biofuel Produced by Non Edible Oil". We would like to put forth our heartfelt gratitude to all those responsible in making our project come into fruition. We are eternally grateful to our guide Prof. Karthik S without whose guidance and constant support this project would not have been accomplished. We also wholeheartedly thank our Director Dr. Narayana Swamy and Assistant Director Dr. Mahesh for this opportunity and for their encouragement. Finally, we are thankful to the entire staff membership of our Mechanical Engineering department for their valuable suggestion and encouragement.

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