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Producing Biodiesel from Neem Oil Using a Two-Step Transesterification Process

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Abstract: As a renewable, biodegradable, and non-toxic fuel, biodiesel has attracted a lot of attention during the past century. It is a methyl ester of a fatty acid made from triglycerides by the methanol transesterification process. Due to the alarming rise in automobile ownership over the past year, there is now more focus on finding alternatives to diesel fuel as both demand and costs for petroleum goods have increased. The majority of biodiesel is currently made from refined or oil seeds using an alkaline catalyst and methanol. Large quantities of oil and fat, however, cannot be ingested. However, it is realistic to use a lot of non-edible oil and fat in our nation. Neem oil is utilized in this study as an alternate fuel to create biodiesel. By combining high-free fatty acid (FFA) oil with the best methanol and H2SO4 acid catalyst properties, biodiesel is created utilizing this process using methanol and KOH catalyst.
Keywords: Neem Oil, fatty acid, biodiesel, biodegradable, transesterification, methanol.

I. INTRODUCTION

The huge increasing growth in the number of cars over the past few years has resulted in an extremely high demand for petroleum products. Since it is predicted that crude oil reserves would run out within a few decades, attempts are currently being conducted to look for practical alternatives to diesel [1]. Any nation or region's technological, industrial, social, and economic progress depends heavily on its access to energy. The primary commercialized energy sources worldwide are coal, gas, electricity, and petroleum oil. Coal, petroleum-based products, and natural gas are the main sources of fossil fuels that India now uses to meet its energy needs. The need for diesel is five times greater than the demand for gasoline in India, where 90 percent of the country's transportation energy comes from oil. The need for diesel is five times greater than the demand for gasoline in India, where 90 percent of the country's transportation energy comes from oil. According to estimates, India currently needs to produce 2- to 3-fold more energy in order to support its rising population and maintain its average annual profitable growth of 7 percent [2].

In India, research, production, and commercialization of biodiesel are still in their infancy. Jatropha cruces have been identified as the more suitable (TBO) tree-borne oilseed for the production of biodiesel in India, both in terms of its non-edible oil and its widespread distribution. Farmers already extract Jatropha oil in a few regions of India, combining it with filtered diesel oil after it has settled and been decanted. In 2003, a biofuels mission was initiated in India. The government launched its biofuel policy in September 2008. They have been given the go-ahead to create a national Coordinating Committee for Biofuels and a Steering Committee for Biofuels with legal authority.

The current study is based on the production of biodiesel, which involves an alcohol-based reaction between two ester compounds. Here, the vegetable oil's triglyceride content is called the ester. There are four ways to use good vegetable oils in diesel engines [10–13].

1) Micro-emulsions of diesel fuel.
2) Oil separation by thermal means.
3) The direct use of diesel fuel and its combination.
4) Transesterification.

Out of the four processes, transesterification is the most favored and effective way to use smooth vegetable oils [14]. As early as 1853, researchers E carried it out. Duffy and J. Patrick, several years before the first diesel engine was operational [15]. This technique was developed to enhance the removal of glycerin for use in making soap in the 1940s [16]. The transesterification process in the esterification method uses both acid and alkali catalysts (KOH or NaOH).

Vegetable oil transesterification requires mass-based raw ingredients, 15% methanol, and 5% sodium hydroxide in order to produce methyl esters. One mole of glyceride reacts with three moles of methanol to form methyl esters in the presence of a catalyst (KOH or NaOH). For the equilibrium scenario, a thought process temperature of 55–65 °C is necessary. To avoid pressurizing the reactor, the temperature is often kept below methanol's typical boiling point (65 ° C) [17].

II. EXPERIMENTAL PROCEDURE

The local market is where neem oil was purchased. Chemicals used in procedures like 88 percent pure potassium hydroxide pellet and methanol (99 percent purity). All chemicals, equipment, and analytical reagents are purchased from Hind Scientific Industries Pvt Ltd (Jhansi).

The set-up consists of a one-liter flask with a circular bottom and three necks. The first neck of the flask is used to insert the thermometer, the second neck is used to enter the condenser, and the main neck is used to insert the stirrer for continuous agitation. To maintain a constant temperature, about two-thirds of the flask is submerged in the water bath using a relay, immersion rod, and thermometer. The mixes are swirled at the same speed throughout all test sessions. In this test, a thermometer maintains and controls a temperature range of 50–60°C. Following acid pretreatment and transesterification, the mixture of methanol and water is entirely separated using a separation funnel.

Neem oil can be converted in a variety of ways to biodiesel or alkyl esters. In our work, we primarily used three techniques to produce ester from oils and fats.

1) Direct esterification of methanol oil using an acid catalyst.
2) Oil conversion from alkyl ester after oil has been transformed into fatty acids that act as an acid catalyst.
3) Base crude oil transestrification reaction with alcohol as the catalyst.

From an economic standpoint, base catalyzed reactions currently yield the most significant production.

The flask is filled with 220 ml of pure neem oil and heated to 50 ° C. Neem oil was heated in a 50 ml addition of methanol, which was then stirred for a few minutes. 2.08 gram of KOH should be added to the mixture. For around 90 minutes, heated stirring should be done at atmospheric pressure. After this reaction, the fluid is poured into a different funnel to separate the KOH, extra alcohol, and contaminants. Extra alcohol, KOH, and contaminants accumulate on the top layer, which is then discarded. The lowered layer is split for additional transesterification into methyl ester processing. Neem oil's acid value is decreased with this technique to less than 1% of FFA.

At the end of the acid pretreatment in the flask, the oil is esterified and heated to (50-60) ° C. 45 cc of methanol dissolves 1.5 gram of KOH. The dissolved solution is poured into the flask.

The mixture is stirred and cooked for two hours. After the reaction is finished, the mixture is placed into a 48-hour separation funnel. In the lowest layer, impurities and glycerol sink and are discarded. The top layer still contains impure biodiesel. To clean the mechanism, combine 2/3 th of methyl ester with warm distilled water while gently stirring. The bottom layer is removed, leaving the top layer of regular biodiesel.

The amount of methyl alcohol was initially determined using the author's equations in the most recent study of the transesterification response to the preparation of methyl/ethyl ester from the experimental vegetable oil/non-edible plants [41].

The methanol and KOH required for the response will be determined by the formulas that is given below:

$$\text{Needed methanol} = 0.2235*\text{oil} \qquad (1)$$

Where oil in each experiment shows the real amount of experimental oil taken in grams.

The equation (2) calculated the catalyst KOH / NaOH .

$$\text{Needed KOH / NaOH} = \frac{\text{oil}}{100} \qquad (2)$$

Where the amount of vegetable oil / non-edible oil taken in the transesterification method.

III. RESULTS AND DISCUSSION

Various physico-chemical qualities were discovered through various experiments. The neem oil is described as follows:

A. *Characterization of the oil used in the Experiment*

The point of pour is the temperature at which a liquid loses its flow characteristics and solidifies to a semi-solid state. The paraffinic concentration will rise as the pour point rises. The point of pour is determined using the ASTM D5853 test. The specimen is placed in a cooling bath and allowed to produce paraffin wax crystals.

The temperature at which wax haze forms after cooling oil in the bottom of the container is known as the cloud point. It explains the temperature at which we may safely manage oil without worrying about clogging the filter.

The temperature at which enough fumes are created to ignite the oil and immediately cause flames to appear is called Flash Point. To determine the flash point of fuel oil, the Abel and Pensky-Martens devices are employed.

Mass per volume per unit equals density. Denser oils provide more energy since they contain more energy due to the fact that diesel is denser than gasoline. Density is measured using the hydrometer.

The diesel engine's fuel ignition is delayed, as shown by the cetane number. 15 heptamethylnonane and 100 hexadecane were each assigned in the calculation of the cetane quantity. The fuel's ignition quality is indicated by the cetane number indicated in Equation 3.

Cetane number = percent n-cetane + 0.15x (percent heptamethylnonane) (3)

The internal resistance that oil fluid layers provide is called viscosity. A redwood viscometer is used to measure viscosity. Equation for measuring centi stokes of kinematic viscosity as shown in equation 4.

$$(V = A*t - B / t) \qquad (4)$$

Where,

A = Instrument calibration constant

B= Instrument type constant

t = Efflux time, s

Acid value is the quantity KOH needed to neutralize 1 gram with chemical compound in milligrams. It's a measure of the number of substance groups of carboxylic acids.

$$\text{Acid value} = \frac{\text{VeqxNx55.1}}{\text{Woil}} \qquad (5)$$

Where,

Veq = Titrant volume in ml consumed by biodiesel

Woil = Biodiesel mass in grams.

N = KOH normality

Another significant biofuel property is about 138F flash point. It is greater than petrol and diesel, so the storage and using of biodiesel is safer. If the point of the flash is small, low temperature fuel is going to ignite and become extremely hazardous for use and storage.

Viscosity and density are other fuel characteristics. Due to greater viscosity value, clogging happens in to the fuel engine issue. However, in the ASTM range standards, the biodiesel generated has viscosity. Density impacts engine efficiency. If the fuel density is high, pumping the fuel will become harder. In our research, the standard range is approximately 0.88.

B. *Neem Oil And Its Biodiesel Have Physicochemical Characteristics*

Table 1 shows the Neem oils and their ester properties as discussed in the work [48-52].

Neem biodiesel has a variety of properties that are analogous to those discovered by other scientists.

Table 1 Neem oils and their ester properties.

Parameters	Diesel	Neem oil	Neem biodiesel(step1)
Flash point ° C	60	214	77
Cloud point ° C	-12	19	-13
Pour point ° C	-16	10	-11
Kinematic Viscosity (cst)	4.7	20.5-47.7	4.76
Cetane Number	45	40-50	57
Acid Number mg of KOH/gm	ND	ND	1.06
Oxidation Stability, (h)	3-6 min	12.4 min	8.1 min
Density (kg/m^3)	830	910-960	838
Water content (%)	0.02	0.098	1.13

ND- Not determined

Figure 1 shows the physiochemical characteristics obtained at different temperature.

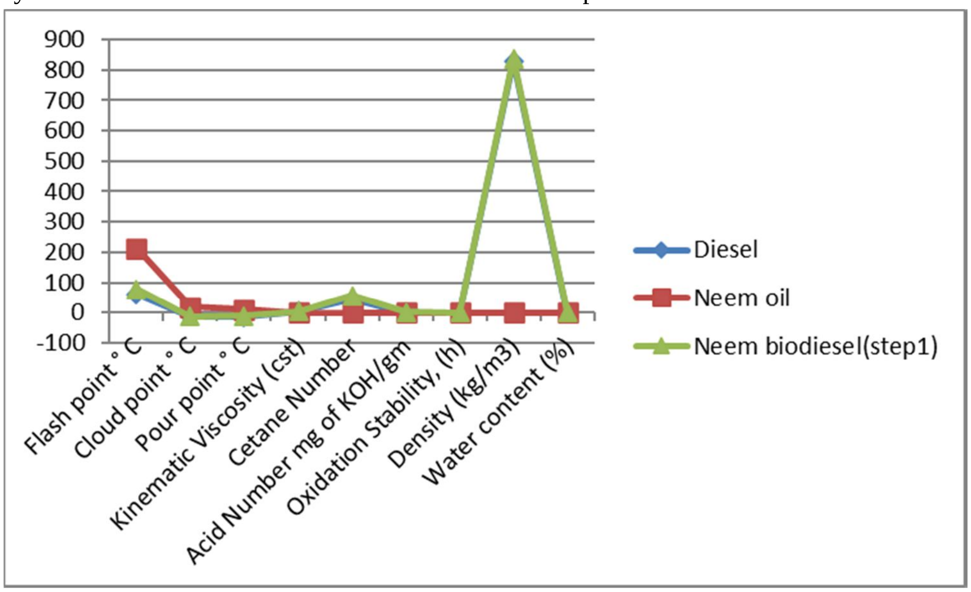

Figure 1: Physiochemical characteristics obtained at different temperature

Table 2 shows the Neem oil and their ester properties obtained in the recent work.

Table 4. 2 Neem oils and their ester properties.

Parameters	Diesel	Neem oil	Neem biodiesel(step2)
Flash point ° C	60	214	70
Cloud point ° C	-12	19	-12
Pour point ° C	-16	10	-12
Kinematic Viscosity (cst)	4.7	20.5-47.7	4.60
Cetane Number	45	40-50	50
Acid Number mg of KOH/gm	ND	ND	1.09
Oxidation Stability, (h)	3-6 min	12.4 min	7 min
Density (kg/m^3)	830	910-960	835
Water content (%)	0.02	0.098	1.03

Figure 2 shows the physiochemical characteristics obtained at different temperature and at different parameters.

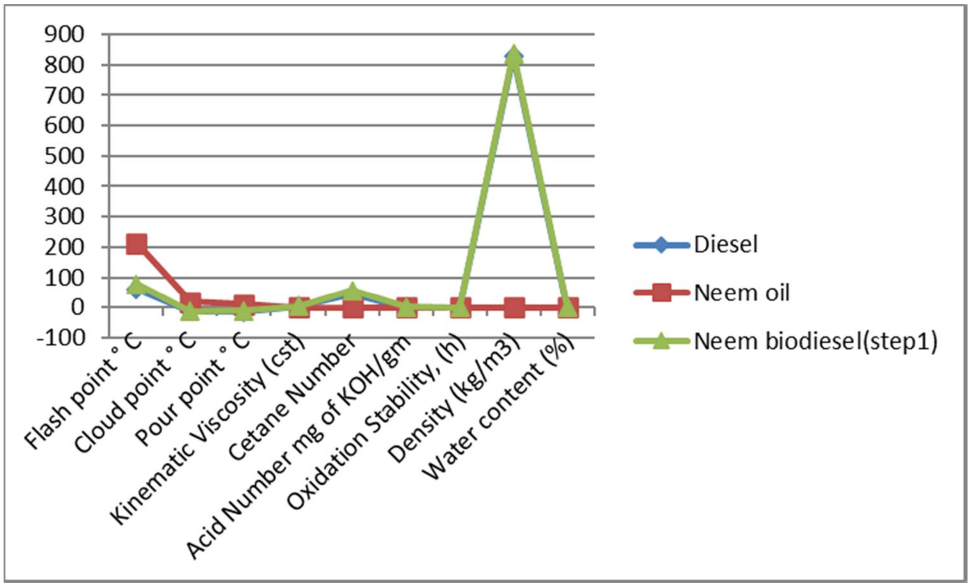

Figure 2: Physiochemical characteristics obtained at different temperature and parameters

IV. CONCLUSION

Renewable energy sources including biodiesel, bioethanol, biomethane, biomass from waste, and hydrogen have gained a lot of attention in the current fossil fuel energy scenario. These fuels assist in lessening reliance on fossil fuels. Additionally, the usage of fuels that cause environmental degradation and may become rare in the future could be partially replaced by energy sources like these. They are referred to as "alternative fuels" for these reasons. Because of its higher density, higher flash point, higher viscosity, and lower calorific value, vegetable oil cannot be utilized directly in a diesel engine. In order to match the fuel qualities of diesel, it must be transformed into biodiesel.

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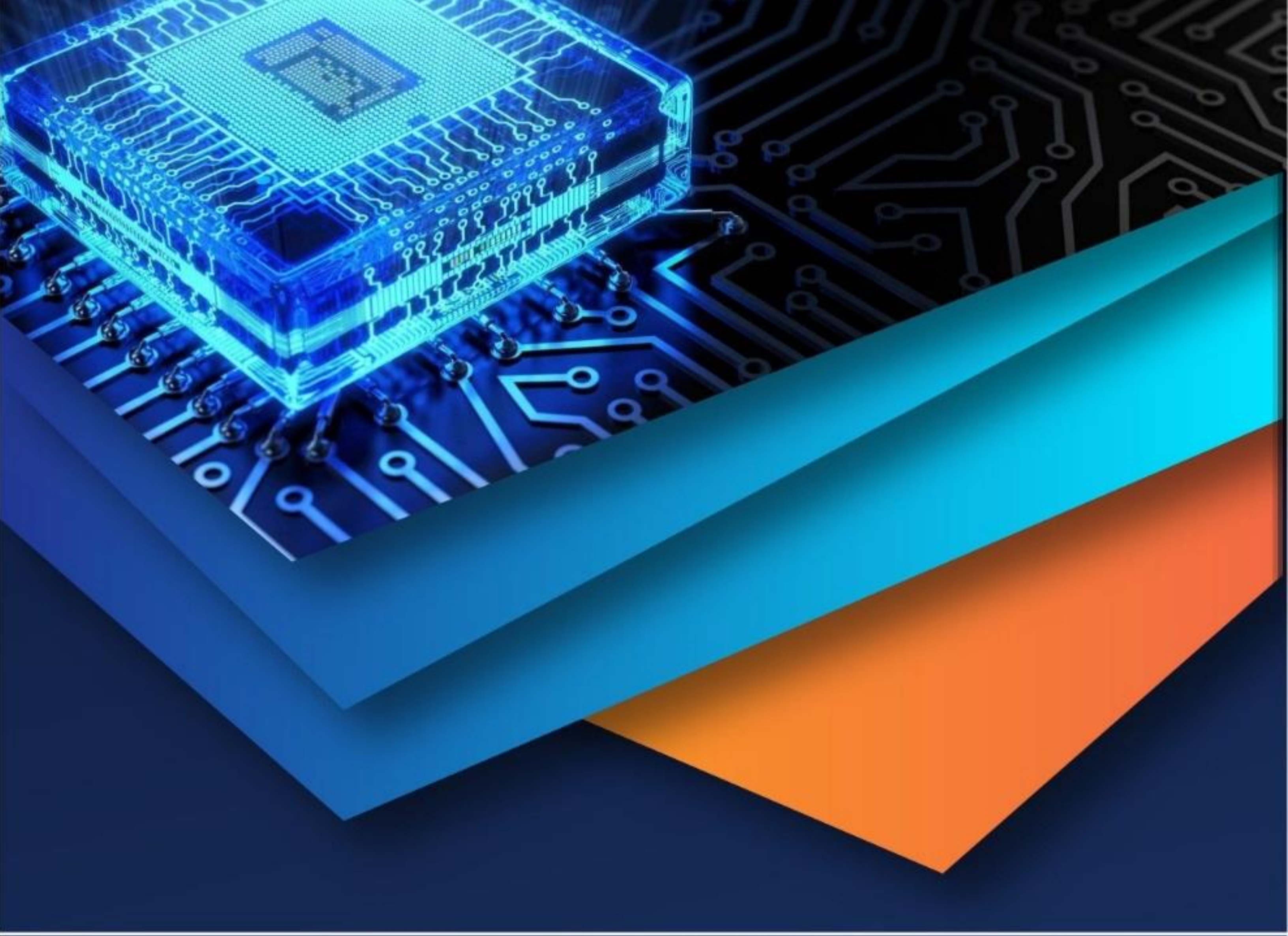

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