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Research Article

Homogeneous Catalytic Transesterification of Renewable *Azadirachta indica* (Neem) Oil and Its Derivatives to Biodiesel Fuel via Acid/Alkaline Esterification Processes

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Abstract

In this work biodiesel was prepared from the crude non-edible oil from the seeds of the neem tree (*Azadirachta indica*) using a two-step reaction systems such as acid and alkaline esterification with short chain alcohol like methanol and its physical and chemical properties of synthesized biodiesel were examined . Important fuel properties of neem oil biodiesel such as kinematic viscosity@ 40°C (5.81 cSt), density @ 15°C (0.898 g/m³), flash point (175°C), pour point (8°C), total sulfur content percent by mass (0.03), ash percent by mass (0.00), carbon residue percent by mass (0.08), copper strip corrosion for 3 hour at 100°C (Not worse than No. 1), sediment percent by mass (0.00), water content percent by mass (0.00) compared well with other methyl esters. Neem oil methyl ester (NOME) has blended with

conventional diesel at various proportions and its physical property of kinematic viscosity was measured at the temperature range up to 95°C. The viscosity of B20 (20% NOME + 80 % ordinary diesel) blended with diesel has 3.81 cSt at 30°C close to conventional diesel and in other blends viscosity slightly close to conventional diesel . The quantification of methyl esters was analyzed by HPLC analysis. A maximum yield of 85% biodiesel was achieved using 1:8 molar ratio of oil to methanol with 0.08% volume of acid catalyst and 1% of NaOH catalyst at 60°C by two step reaction processes.

Keywords: Azardirachta indica, biodiesel, two step esterification process, transesterification.

Introduction

The world has been confronted with the twin crises of fossil fuel depletion and environmental deduction. The indiscriminate extraction and consumption of fossil fuels have led to a reduction in petroleum reserves (Barnwal & Sharma 2005). Fossil fuels especially petroleum diesel have been the prime sources of energy for the purpose of transportation, power generation, agriculture, commercial, domestic and industrial activity for more than a century (Baskar & Samuel 2013). The transport sector faces serious problems as no alternative to petroleum based fuel has been successful so far. An increasing the fossil fuel demand and their cost are the major concern for the developing countries like India as well as petroleum based fuels especially conventional diesel will continue to dominate the transport

sector in the foreseeable future. But its consumption can be minimized by implementation of renewable source like bio-fuel (bio-origin) such as alcohol, vegetable oils, biogas and biodiesel which are becoming important (Barnwal & Sharma 2005). Alternate fuels promise to harmonize sustainable development, energy conservation and management, and environmental preservation (Ramadhas, Jayaraj & Muraleedharan 2005). In recent years, research has been directed to explore plant-based fuels and plant oils and fats as fuels have bright future (Mohibbe, Amtul & Nahar 2005).

Biodiesel is methyl or ethyl ester of fatty acids made from virgin or used edible, non-edible oils and animal fats via transesterification by alcohols (Demirbas 2005). Transesterification reaction helps to alter the chemical structure

of vegetable oils and so reduce their molecular weight and to make their physical and chemical properties comparable to conventional diesel such that they may be used in fossil diesel engines without any engine modifications (Shahid & Jamal 2011). The modified product is called biodiesel and has many advantages such as safe for use in all conventional diesel engines, offers the same performance and engine durability as petroleum diesel fuel, non-flammable, lower emission of carbon monoxide, particulate matter, sulfur compounds, non-toxic and biodegradable. Biodiesel is an efficient, clean and natural energy alternative to petroleum diesel fuels (Bobas 2005; Di Serio et al. 2008; Wang et al. 2009).

Neem oil is a vegetable oil pressed from the fruits and seeds of the neem tree (*Azadirachta indica*, A.Juss), an ever green tree

which is endemic to the Indian subcontinent and has been introduced to many other areas native to India and Burma, growing in tropical and semitropical regions Wikipedia. Neem seeds have the best oil fraction like 40-58.9 wt% compared to other parts of the tree. The kernels yield generally a greenish to brown color oil with simulating garlic which constitutes mainly triglycerides and a large amount of triterpenoid compounds (Soetaredjo et al. 2008, p.45). The azadirachtin content of neem oil varies from 300 ppm to over 2000 ppm depending on the quality of the neem seeds crushed. The oil can be obtained through pressing or through a process incorporating temperature controls. Oil seeds that are to be processed by solvent extraction is usually flaked to increase the exposure of the oil to the solvent. Hexane in a percolation extractor is the most common technology used today. The alternative to solvent

extraction is mechanical extraction or crushing (Kovo 2006). The neem oil yield that can be obtained from neem seed kernels varies from 25% to 45%. The composition of neem oil fatty acids include linoleic acid 6-16%, oleic acid 25-54%, hexadecanoic acid 16-33%, octadecanoic acid 9-24% and others like A-linolenic acid, 9-hexadecanoic acid (Wang, Yu & Luo 2009). The type and quantification of fatty acids contained in vegetable oil depends on the plant species and on the growth conditions of the plant (Ramadhas, Jayaraj & Muraleedharan 2005). Though vegetable oils are of very low volatility in nature, it quickly produces volatile combustible compounds upon heating. Biodiesel preparation is complicated if the oil contains high level of FFA that will produce soap with base catalyst. Acid catalysed transesterification is a classical method for producing biodiesel from high free fatty acids content oil and also requires excess

quantity of methanol and time duration. In this direction, researchers need to develop any method to produce biodiesel from high FFA level oils. A two-step esterification process consists of acid and alkaline esterification process that will help to solve the issue of biodiesel production from high FFA level of oils (Baskar et al. 2013; Ramadhas et al. 2005 Canakci et al. 2001). The purpose of the present study was to develop a method for esterification of high FFA oil considered as a potential feed stock for biodiesel production.

In this present work, biodiesel was produced from the non-edible oil of the neem tree (*Azadirachta indica*) and its properties were analyzed and compared with other methyl esters. Neem oil methyl ester (biodiesel) is blended with conventional diesel at various proportions to determine the kinematic viscosity at the

temperature range up to 95°C. The quantification of methyl esters were analyzed by High Performance Liquid chromatography (HPLC).

Experimental Details

Materials

(Azadirachta indica) Neem oil was purchased from Madurai local market, Tamil Nadu State, India. The oil was extracted by physical method of mechanical oil expeller from the seeds. The age of the extracted neem oil is having long days but the oil has purchased around 40 days after extraction. Average compositions of neem oil have four fatty acids like linoleic acid (6-16%), oleic acid (25-54%), hexadecanoic acid (16-33%) and octadecanoic acid (9-

24%). Sodium hydroxide pellets (NaOH > 99%), methanol (> 99%), conc. sulfuric acid, acetic acid and phenolphthalein indicator (pH 7-10) from SD Fine Chemicals Limited, India were used for the biodiesel production. All chemicals were used as analytical reagent grade without further purification. The conventional diesel was purchased from the commercial petrol bank (Bharath Petroleum Corporation, India) which is nearer to Madurai Kamaraj University, palkalai nagar, Madurai, TamilNadu, India.

Equipment

A hot plate with magnetic stirrer was used to perform the transesterification reaction. Maximum one liter of biodiesel can be produced from this equipment. The kinematic viscosity of

blending ratio biodiesel and diesel was determined by redwood viscometer [Remi equipments private limited].

Density

Density is the weight of a unit volume of fluid. Specific gravity is defined as the ratio of the density of a liquid to the density of water. Density of the sample was measured by specific gravity hydrometers [Koehler Instrument company, inc] (ASTM D287) at the standard temperature range is around 15°C. The length of the tube is 330 mm and the subdivisions like 0.0005. The nominal specific gravity range is 0.850 to 0.900 g/m³.

Kinematic Viscosity

Viscosity means it is a measure of the internal friction or resistance of an oil to flow. The kinematic viscosity of biodiesel was determined by using Saybolt viscometer [Entek Instruments India Pvt Ltd] (ASTM D 88). The operating temperature range is around 20°C to 100°C can be applied for fuel sample analysis.

Flash Point

Flash point means the lowest temperature at which vapors above a volatile combustible substance ignite in air when exposed to flame. Flash point was measured by Pensky-Martens closed cup tester (ASTM D93) [Koehler instrument company, inc]. Flash

point of biodiesel sample was measured within the temperature range of 60 to 190°C.

Pour Point

The pour point of a liquid is the lowest temperature at which it becomes semi solid and loses its flow characteristics. Pour point of biodiesel (ASTM D 97) was measured by Tanaka cloud point tester (Model No:MPC 102A, Cannon instrument company limited). This tester can be measured at the temperature range from +51°C to -30°C.

Copper Strip Corrosion

A qualitative method of determining the corrosivity of a petroleum product by observing its effect on a strip of polished copper suspended or placed in the product which is known as copper strip test. Copper strip corrosion test was conducted by copper strip corrosion tester (Model No: K25330, Koehler instrument company, inc). ASTM D 130 standard has followed for analyzing the samples. The maximum temperature limit 190°C can be applied. A heater maximum range is 0-750W.

Carbon Residue

The carbon residue of a fuel is the tendency of carbon deposits to form under high temperature in an inert atmosphere which is

known that the correlation between carbon residue and diesel engine performance is poor. Carbon residue (ASTM D 524) of biodiesel sample was measured by Ramsbottom carbon residue apparatus (K27100) at frequency 60 Hz and the operating voltage is 115V.

Ash content

Ash content means, the solid residue left when combustible materials is thoroughly burned or is oxidized by chemical reaction systems. The percentage of ash content (ASTM D 482) of biodiesel sample was analyzed by high temperature muffle furnace [Heraeus M 104 Muffle Furnace, Thermo scientific limited]. Muffle furnace consists of programmable temperature controller, upper limit cut-out and exhaust fan. The maximum

temperature is 1000°C can be used. The percentage of ash content of sample was measured at the temperature of 450°C.

Water and Sediment

Water and sediment content can be measured by centrifuge system [TC 450 D, Eltek limited]. This analysis indicates the presence of free water and sediment in biodiesel. This test was performed by spinning a sample in a centrifuge at high speed. The centrifuge system consists of programmable microprocessor control, interchangeable rotor heads and safety lid switch. The maximum speed of rotor is around 5000 rpm.

Total Sulfur Content

This analysis is looking for the presence of sulfur in biodiesel. The sulfur content was measured by UV fluorescence method specified by the ASTM D 5453 standard. UV fluorescence SO₂ analyzer [100 E, Teledyne instruments, Chemtrols industries Ltd] was used and is capable of measuring sulfur content in liquid samples. The detection limit is in the ppb (0 ppm) range at the lower end and up to 1% (20,000 ppb) at the higher end.

Analytical Analysis

The amount of methyl esters in the product of transesterification of neem oil was analyzed using High Performance Liquid Chromatography (HPLC) (SHIMADZU, JAPAN) equipped with

refractive index detector UV-spectrophotometric. The main column ODS-C18 (Analytical-shim-pack CLC-OCTA DECYL SILANE) [4.6 mm 1D * 25 cm] and Guard Column Shim-Pack G-ODS [4 mm 1D * 1 cm] at the temperature of 30°C was used for separation with 1 ml/min flow rate of methanol as a carrier solvent. The sample injection was 20 µl and each constituent was quantified by comparing the peak areas with their respective standards.

Acid and Alkaline Transesterification

The crude neem oil was esterified using acid catalyst and methanol at the temperature of 50°C for 15 minutes and followed by alkaline catalyst like sodium hydroxide with methanol to form sodium methoxide added to oil at the temperature level of 60°C

and stirring during this experiment for 1 hour. Stirring rate was adjusted to form vortex appearing on the oil top surface. The two layers were separated, bottom layer contains impurities and glycerol, top layer is methyl ester (biodiesel). The bottom layer of glycerin is drawn off. The upper of methyl esters is washed with ordinary tap water to remove the catalyst before adding little amount of dilute acetic acid to attain neutral stage ($\text{pH} \approx 7$). The procedure is repeated four or five times. Lower layer is discarded and top layer (biodiesel) is separated. Finally the biodiesel is heated at 130°C for excess methanol recovery and moisture removal.

Biodiesel Production from Azadirachta indica (Neem) Oil

One liter oil was taken in the conical flask. The oil is pre-heated and stirred at 50°C. The oil requires 100 ml of methanol and 0.8 ml conc. sulfuric acid for the acid esterification process. Heating and stirring the mixture were continued for 15-20 minutes at atmospheric pressure for acid esterification. 10 gm of NaOH was added to the 200 ml of methanol to make sodium methoxide. The freshly prepared sodium methoxide solution is added with acid esterifying oil. Heating and stirring were continued for another 1 hour by magnetic stirrer with hot plate at 60°C. A stirring rate can be fixed to form vortex appearing on the top of the oil surface. On completion of this reaction, the mixture was poured into a separating funnel for obtaining the separation of biodiesel and glycerol. There are two layers

were separated due to the density difference between biodiesel and glycerol as shown in Fig.1. The color variation also indicates the layers separation like yellowish brown and dark brown color. The bottom layer of glycerol was drawn off for further purification of the prepared biodiesel. The top layer of biodiesel was washed with usual tap water with little amount of dilute acetic acid for removing the NaOH catalyst to attain $\text{pH} \approx 7$. Finally, the purified biodiesel was heated at 120°C temperature for excess methanol recovery and moisture removal by distillation process.

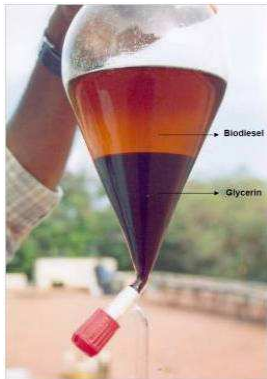


Fig. 1. Transesterification Reaction of Neem Oil Biodiesel Separation

Results and Discussion

Transesterification Reaction of Neem Oil

A broad variety of high free fatty acid content of vegetable oils is available in large quantities like *Pongamia Pinnata* (karanja), *Madhuca indica* (Mahua), *Shorea robusta* (Sal), *Azadirachta indica A Juss* (Neem) and *Hevea brazilienses* (Rubber) which conveys great potential sources for biodiesel production in India (Deepak et al. 2013; Anya et al. 2012). The present study has evaluated for biodiesel production from crude non-edible oil of neem. Neem oil is having high free fatty acid value which is carried with moisture content of seed layer, these two factors have significant effect on the transesterification reaction of triglycerides (Anya, Nwobia & Ofoegbu 2012). Generally, non-edible oils contain high free fatty

acid level. Biodiesel conversion is complicated if the free fatty acid level of oil is above 1% which will form soap with base catalyst and produces low yield of fatty acid methyl esters due to the soap can inhibit the separation of the biodiesel from the glycerin fraction which will be much difficult to isolate the products (Baskar et al. 2013; Sathya et al. 2013). A two-step reaction system consists of acid and alkaline catalyst is recommended for conversion of biodiesel from high FFA oils. Acid esterification process helps to reduce the FFA level of oil and also prevent soap product during alkaline transesterification process (Canakci et al. 2001; Baskar et al. 2013). Deepak et al has considered refined neem oil for biodiesel production. Refined process can be additionally increasing the cost of biodiesel production (Deepak et al. 2013, p.1896). Sathya & Manivannan (2013) have studied about neem oil bidiesel production about

two step transesterification processes. The reaction time duration of acid esterification is around 45 minutes and then 1% of KOH and 30% of methanol were used for alkaline transesterification. The high amount of methanol was used for neem oil biodiesel production. But they have attained a maximum yield of biodiesel was above 90%. Atul, Roblet & Avinash (2012) have reported that the biodiesel from neem oil was synthesized by two stage processes. For the esterification reaction, 4.5% of acid catalyst 6:1 alcohol to oil molar ratio, 90 min reaction time duration and temperature range 45°C. This process was reduced the FFA level from 20.0% to 3.6 %. Acid esterified oil was treated with methanol (6 moles) in the presence of base catalyst 1% at 60°C for 1 hour. The whole process time duration was so long like 9 hours. Elkadi et al. (2013, p. 500) have studied about reaction kinetics of neem oil biodiesel production. The same

method has followed for biodiesel production. But KOH was used for base catalyzed transesterification reaction. Sodium hydroxide is more efficient catalyst and also it requires minimum quantity for transesterification reaction when compared to the KOH catalyst used. In this study, biodiesel produced from neem oil by two step reaction systems. The base catalyst amount was measured by acid base titration method. Titrant value was around 10 which is slightly high value when compared to use refined oil. So the oil has pretreated with acid catalyst (0.8 ml conc. H_2SO_4) with methanol (100 ml) for 20 minutes and then sodium methoxide solution was added to the above mixture while during the reaction. The total time duration of the reaction was 90 minutes (including separation time) only when compared to previous works. The methanol amount, catalyst amount and

reaction time duration are very important factors for economical approach of biodiesel production.

Properties of Methyl Esters of Azadirachta indica (neem oil)

The fuel properties of neem oil methyl esters in comparison with that of oil, other methyl esters and ASTM standard biodiesel (D6751) are presented in Table 1 (Ghadge & Hifjur 2006; Foidl et al. 1996; Tate et al. 2006; Karmee & Chadha 2005; Dorado et al. 2003; Van Gerpen et al. 2004). The results show that the transesterification process improved the fuel properties of the oil with respect to kinematic viscosity@ 40°C (5.81), density @ 15°C (0.898 g/m³), flash point (175°C), pour point (8°C), percent total sulfur content by mass (0.03), ash percent by mass (0.00), percent carbon residue by mass

(0.08), copper strip corrosion for 3 hour at 100°C (Not worse than No. 1), sediment percent by mass (0.00), percent water content by mass (0.00). The kinematic viscosity of biodiesel (5.81 cSt) is close to that of conventional diesel (1.2-3.60 cSt) and other methyl esters. The flash point of neem oil methyl ester (175°C) was lowered by transesterification but it was still higher than that of ordinary diesel.

Table 1. The Properties of Neem Oil Biodiesel Compared with Other Biodiesel and ASTM (D6751) Standard Biodiesel

Please See Table 1 in Full PDF Version

Effect of Dilution and Heating on Kinematic Viscosity of Blends

From the Fig. 2, it can be seen that the viscosity of neem oil methyl esters (NOME) was decreased due to blending with diesel and also heating. The viscosity of neem oil methyl ester has 5.81 cSt, It is slightly higher when compared with conventional diesel. The viscosity can be reduced by increasing the diesel content and temperature of the blend. It has been shown that various blends (80%, 60%, 40%, and 20%) of diesel have viscosity close to that of ordinary diesel. The viscosity of (20% NOME + 80% OD) blended with diesel have 3.81 cSt at 30°C to very close to conventional diesel and (40% NOME + 60% OD, 60% NOME + 40% OD, 80% NOME + 20% OD) the viscosity of other blends slightly varies from that of diesel but close to at 40°C. Heating could be reduced the

viscosity of methyl ester of fatty acids due to viscosity is inversely proportional to temperature. Blends of 20% biodiesel with 80% conventional diesel (B 20) can be used in unmodified diesel engines. Biodiesel can be used in its pure form by many require certain engine modifications to avoid maintenance and performance problems. B 20 (20% Biodiesel + 80% conventional diesel) has a good performance in diesel engines (Shaine & McCormick 2004).

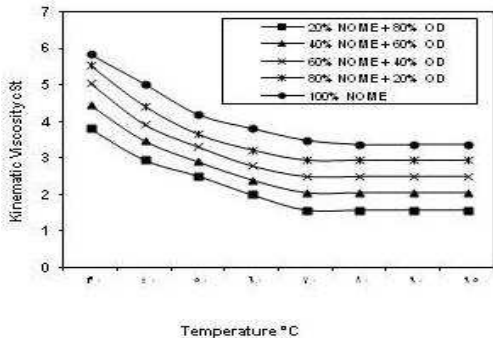


Fig. 2. Comparison of the Kinematic Viscosity of Neem Oil Methyl Esters (Biodiesel) Blending with Ordinary (Conventional) Diesel Blend

Viscosity is one of the most important physical properties of liquid fuels. The effects of viscosity can be observed in the quality of atomization and combustion as well as engine wears (Tate et al. 2006, p.1010). Atomization is the first stage of combustion in the diesel engine. Atmospheric air contains number of oxygen molecules, which will react rapidly with fuel on the outer surface of the oil droplet and produces a large amount of heat to environment. This may create other chemical reactions like charring or coking and polymerization (Krisnangkura et al. 2010, p.2775). The higher viscous is due to the large molecular masses (especially oxygen molecule) and chemical structure of fuels which in turn leads to problems in pumping, combustion and atomization in the injector systems of a diesel engine. The problem of high viscosity of fuels has been approached several ways like preheating the fuels and blending or dilution with

conventional diesel fuel (Pramanik 2003). The present study shows that the important physical property of viscosity of neem oil biodiesel was approached by heating as well as blending method for reducing the viscosity level.

Conventional diesel, neem oil and its methyl esters of kinematic viscosity values were measured at the temperature range up to 95°C is shown in Fig.3. The viscosity of neem oil biodiesel is close to conventional diesel at the temperature above 60°C. The neem oil has higher viscosity than that of its methyl esters (biodiesel).

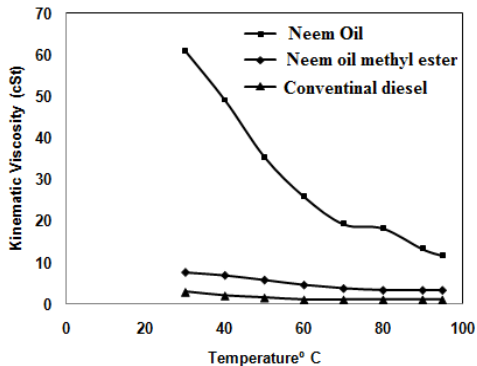


Fig. 3. Comparison of Kinematic Viscosity of Neem Oil, Neem Oil Methyl Esters (Biodiesel) and Conventional Diesel

Quantification of Methyl Esters

The amount of methyl esters in the product after transesterification of neem oil was analyzed using High Performance Liquid chromatography (HPLC) (SHIMADZU, JAPAN), as shown in Figure 4 and its quantification in percentage results are presented in Table 2. Neem oil contains mainly average compositions of four fatty acids like linoleic acid (6-16%), oleic acid (25-54%), hexadecanoic acid (16-33%) and octadecanoic acid (9-24%). There are four peaks have appeared in the HPLC spectrum which are indicating that the corresponding methyl esters of the fatty acids (Anya et al. 2012, p.21).

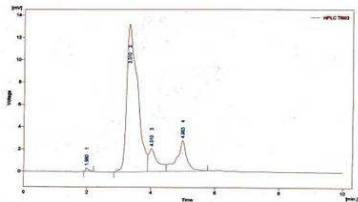


Fig. 4. HPLC Chromatogram of Neem Oil Methyl Esters

Table 2 .Quantification of Neem Oil Methyl Esters

S.No	Retention time [minute]	Area [%]
1	1.980	0.5
2	3.310	72.1
3	4.010	9.7
4	4.983	14.4

Conclusion

The production of biodiesel from the oil through two step processes like acid and alkaline transesterification method has been demonstrated. It was shown that the crude oil from neem is well suited for the production of biodiesel and it has comparable qualities of biodiesel. The advantage of this method is the lower reaction, temperature and duration of time are required. The maximum conversion of 85% was obtained at 60°C with 300 ml methanol for NaOH (14.5g per liter of oil) catalyzed transesterification. The methyl ester fuel properties were found to be close to conventional diesel and compared to other methyl esters. The viscosity of neem oil biodiesel has 5.81 cSt, It is slightly higher than conventional diesel. The viscosity can be reduced by increasing the diesel content and temperature of the

blend. Blends of 20% biodiesel with ordinary diesel are very close to conventional diesel and also the viscosity of neem oil biodiesel is close to conventional diesel at the temperature above 60°C. The methyl esters of neem oil have identified by the HPLC spectrum which illustrated that four fatty acids of neem oil were converted into corresponding fatty acid methyl esters.

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