

Biodiesel Production from Neem Oil Using Two Step Transesterification

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Abstract

Currently, most of the biodiesel is produced from the edible/refined type oil using methanol and alkaline catalyst. However, large amount of non-edible type oils and fats are available in our country. In this study, crude neem oil is used as alternative fuel for biodiesel production. The difficult with alkaline transesterification of these oils have contained 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 glycerin. A two-step transesterification process is developed to convert the high FFA oils to its mono-esters. Using 100 ml of oil, the optimum combination of parameters for pretreatment were found to be .45 v/v methanol-oil-ratio, 0.5% v/w H₂SO₄ acid catalyst, 50°C and 45 min reaction time. After pretreatment of neem oil, transesterification reaction was carried out with 0.3:1 methanol-to oil ratio, 1% KOH as alkaline catalyst, 1hr reaction time and 55°C reaction temperature to produce the fatty acid methyl ester. This two step process gave maximum average yield of 90±2%.

Keywords: Biodiesel, free fatty acid, Neem oil, pretreatment, transesterification.

1. Introduction

The tremendous increase in number of automobiles in recent years have resulted in greater demand of petroleum products. The depletion of crude oil reserves are estimated for few decades, therefore, effort are on way to research now alternatives to diesel[1]. Due to increasing price of petroleum product and environment concern about emission from the car exhaust, the biodiesel has become an area of high concern[2]. The biodiesel is an alternative

feedstock for depletion of fossile fuel. It has derived from renewable resources such as vegetable oil, which could either be fresh or waste vegetable oil are find useful in Europe, America and Asia as a feedstock in production of biodiesel, as a consequently, biodiesel derived from a wide variety of sources can be used as a direct substitute for petro-diesel fuels[3]. They are several non-edible oil seed such as thevetia(thevetia peruviana), karanja (pongomia pinnate), jatropha (jatropha curca), neem (azadirachta india) etc. Among these, azadirachta. indica seed which contain 25%-45% oil on dry matter basis is non edible oil that can be use in biodiesel production [3]. Neem tree is evergreen tree growing in almost every state of India. It is native to Indian subcontinent, South_East Asian countries and it grows all over India. It grows in all kinds of soil and in drier areas [1]. The oil contain three bitter compound that can be extracted from it which are nimbin, nimbinin and nimbidin which are mostly responsible for it medicinal value, it can also be use in cosmetics preparation and act as an effective mosquito repellent. Neem oil have high free fatty acid value which is accompanied with moisture content and some other impuritiest, this two have prime effect on the trans-esterification of glycerides with alcohol using catalyst. However most non-edible oil have high free fatty acid content which it leads to high production cost through trans- esterification, an FFA content more than 2% will form soap and the separation of the product will be very difficult, it produces low yield fatty acids methyl esters[3]. This present investigation is intended to consider

aspects related to the feasibility of the production of biodiesel from neem oil. The variables affecting the yield and characteristics of the biodiesel made were studied. The obtained results were analyzed and compared with conventional diesel fuels.

2.2. MATERIALS AND METHODOLOGY.

2.1 Materials

Crude neem oil was purchased from local market. The magnetic stirrer with hot plate was purchased from Taleco Chemicals Pvt. Ltd at Tirunelveli. Chemicals are used in transesterification process like Potassium hydroxide pellet (88% purity), Methanol (99% purity), Concentrated sulphuric acid and Sodium sulphate. All the chemicals are purchased from Himedia and Nice chemicals Pvt Ltd and its analytic reagent grade.

2.2 Equipment

A round bottom conical flask is used as reactor for these experimental purposes. A magnetic stirrer with hot plate 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 40–60°C is maintained during this experiment and is monitored by thermometer. The separating funnel is used to separate the methanol-water mixture after acid pretreatment and the glycerol after transesterification. Four sets of trial runs are carried out for each combination of parameters.

2.3 Methodology

The aim of this study is to improve the process for producing biodiesel from crude neem oil. There are three processes such as oil filtration, acid esterification and alkaline transesterification.

2.3.1 Oil filtration

Neem oil has higher moisture content and some other impurities. So, in order to remove the moisture and impurities from the neem oil it

should be refined. The purification can be done by boiling oil with about 20% of water. The boiling should continue until no bubbles of water vapor anymore. After one hour the oil then becomes clear. This refined neem oil is taken as raw material for transesterification process.

2.3.2 Acid esterification

100 ml of refined neem oil is poured into the flask and heated up to 60°C. The 45% v/v methanol is added with the preheated neem oil and stirred for a few minutes. 0.5% of sulphuric acid is added with the mixture. Heating and stirring should continue about 45 min at atmospheric pressure. After completion of this reaction, the mixture is poured into a separating funnel for separating the excess alcohol, impurities and sulphuric acid. The excess alcohol, sulphuric acid and impurities move to the top layer and is discarded. The lower layer is separated for further processing of transesterified into methyl ester. This process reduces the acid value of refined neem oil to less than 1% of FFA. Viscosity reduction increases with increase in methanol to oil ratio.

2.3.3 Alkaline transesterification

After acid pretreatment the esterified oil is taken in flask and heated up to 60°C. 1% of KOH is dissolved in 30% (6:1 M) methanol. The dissolved solution is poured into flask. The mixture is heated and stirred for 1 hr. On completion of reaction, the mixture is poured into separating funnel over 12 hr. The glycerol and impurities are settled in lower layer and is discarded. The impure biodiesel remains in upper layer. It contains some trace of catalyst, glycerol and methanol. The washing process can be done by the 3/4th of hot distilled water added with methyl ester and gently stirred. The upper layer is pure biodiesel and lower layer is drawn off.

3. RESULTS AND DISCUSSION

3.1. Acid esterification

3.1.1 Effect of methanol-to-oil ratio

Two important parameters significantly affected the acid value. These are sulphuric acid concentration and methanol quantities. Molar ratio is defined as the ratio of number of moles

of alcohol to number of moles of vegetable oil. Theoretically, transesterification reaction requires 3 moles of alcohol for each mole of oil. However, in practice, molar ratio should be higher than stoichiometric ratio. By varying methanol proportion such as 0.35:1, 0.40:1, 0.45, 0.50:1, 0.55:1 methanol-to-oil ratio, among these 0.45:1 gave higher yield. The last two 0.50:1, 0.55:1 have only slight variation. In economic view 0.45:1 M is selected for reaction condition. The effect of methanol variation is shown in fig.1. From the figure conversion efficiency is slightly increase upto 0.45:1 methanol-to-oil ratio. After that conversion efficiency is decreased with increase in methanol-to-oil ratio because it leads to increasing the acid value. It has been determined that in viscosity reduction increases with increase in methanol-to-oil ratio

3.2.2 Effect of alkaline catalyst

The amount of catalyst variation affects the conversion efficiency. The catalyst proportion is varied from 0.5- 2% KOH. The effect of catalyst variation on conversion efficiency is shown in fig.4. From the figure yield is slightly increased upto 1% KOH and after that yield is decreased due to reverse reaction taking place (emulsion formation). The maximum yield is achieved of 88% at 1 KOH

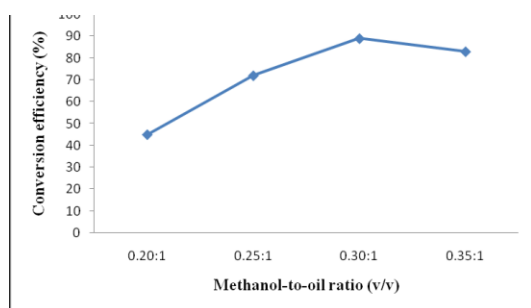


Fig.3. Effect of methanol on conversion efficiency

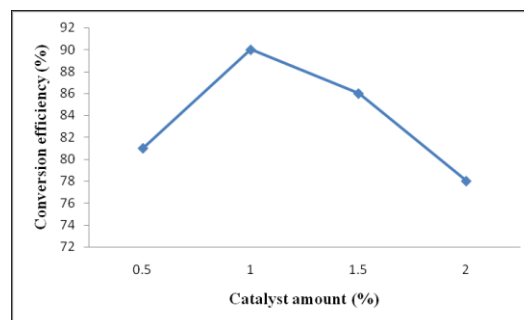


Fig.4. Effect of alkaline catalyst amount on conversion efficiency

4.3. Effect of reaction temperature

The reaction temperature has an important role in alkaline-catalyst transesterification. At room temperature no significant yield is notified for even 2hr reaction. The yield is increased with increase in reaction temperature. The effect of temperature variation on conversion efficiency is shown in fig.5. By varying temperature in four different levels such as 45, 50, 55 and 60°C among these 55°C gave maximum methyl ester yield. If greater than 60°C, there is a chance for loss of the methanol. The maximum ester efficiency is achieved at 55°C.

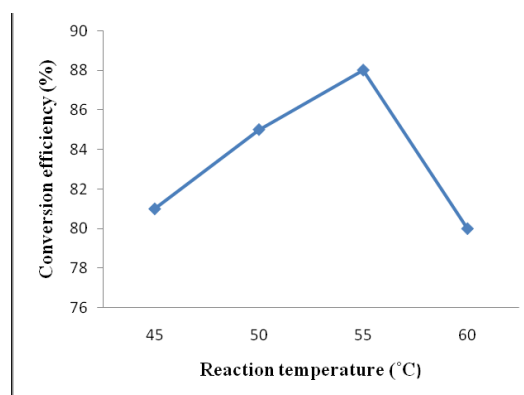


Fig.5. Effect of reaction temperature on conversion efficiency

4.4 Effect of reaction time

The conversion rate is increased with increase in reaction time. In this experiment, reaction time varying from 0.5-2 hr. The effect of reaction time variation on the conversion efficiency is shown in fig. 6. From the figure yield was slightly increased upto 1hr reaction and after that yield is decreased. The maximum efficiency is achieved of 90% for 1 hr reaction. From these experiment the optimum yield is obtained at 1 hr reaction.

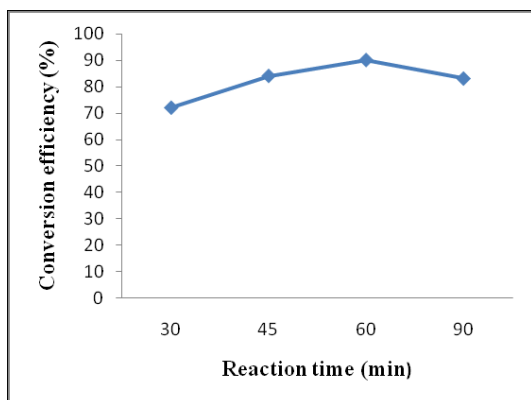


Fig.6. Effect of reaction time on conversion efficiency

5. Conclusion

The high FFA (6%) content neem oil has been investigated for the biodiesel production. It has been found that the feedstock with high FFA its could not be transesterified with alkaline catalyst because the alkaline catalyst react with FFA to form soap. So in this study, two step process was developed to convert FFA to its methyl ester. The first step is acid treatment it reduces the FFA content of oil to less than 1% using acid catalyzed (0.5 % v/v H₂SO₄) reaction with methanol (0.45 v/v) at 50°C temperature and 45 min reaction time. After acid treatment alkaline transesterification reaction was carried out at 1% KOH, 30% methanol, 55°C and 60 min. The maximum yield is 90±2%. The effect of molar ratio, catalyst, reaction temperature and reaction

time are analyzed in each step process. Excess addition of sulphuric acid darkens the product it leads to more production cost.

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