

EVALUATION OF THE TECHNOLOGY FOR BIODIESEL PRODUCTION FROM *JATROPHA CURCAS* OIL AND ACID OIL

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Abstract

In recent times, the world has been confronted with an energy crisis due to depletion of resources and increased environmental problems. The situation has led for search for an alternative fuel, which should be not only sustainable but also environmental friendly. Due to increase in the petroleum and the environmental concerns about pollution coming from the car gases, Biodiesel is becoming a developing area of high concern. For developing countries, fuels of bio-origin such as alcohol, vegetable oils, biomass, biogas, synthetic fuels etc. are becoming important. Biodiesel is now mainly being produced from soybean, rapeseed, *Jatropha curcas* oil (JCO), Palm oil and Acid oil. Alkali base catalyzed transesterification being the most commonly used method of Biodiesel production. Optimum conditions for production of Biodiesel from JCO and Acid oil was obtained at reaction temperature of 55 ± 1 °C, methanol to oil molar ratio of 4:1, NaOH concentration 4% for JCO and 8% for Acid oil and the reaction time of 6-8 hour. JCO contains 15% free fatty acids (FFA) and acid oil contains 78% FFA. So two step acid base catalyzed transesterification is used to reduce FFA contents, because that reduces the yield of biodiesel. The results were obtained 85% for JCO and 90% for Acid oil for one-step alkali base catalyzed transesterification and 75% for JCO and 80% for Acid oil for two step acid-base transesterification reactions.

Key words: *Jatropha curcas* oil (JCO), free fatty acids (FFA), Acid oil, Fatty Acid Methyl Esters (FAMES)

Introduction: Biodiesel production is a very modern and technological area for researchers due to the relevance that it is winning everyday because of increase in the petroleum price and the environmental advantages¹. Biodiesel is an eco-friendly, alternative diesel fuel prepared from domestic renewable resources i.e. vegetable oils (edible or non- edible oil) and animal fats. These natural oils and fats are made up mainly of triglycerides. These triglycerides when reacted chemically with lower alcohols in presence of a catalyst result in fatty acid esters. These esters show striking similarity to petroleum derived diesel and are called "Biodiesel". For this purpose *Jatropha curcas* oil and acid oil considered as most potential source for it². A variety of Vegetable oil can be used to produce Biodiesel. These are:

Virgin vegetable oil feedstock; rapeseed and soybean oils are most commonly used, through other crops such as mustard, palm oil, sunflower, hemp and even algae show promise.

Waste vegetable oil (Refinery waste).

Animal Fats; including tallow, lard and yellow grease.

Non-edible oils; such as *Jatropha curcas*, neem oil, castor oil, tall oil etc³.

***Jatropha curcas* as a Biodiesel Source:** *Jatropha curcas*, belonging to the family Euphorbiaceae, is a low-growing tree, native to South America, but widely cultivated also throughout Central America, Africa and Asia. The popularity of *Jatropha* is also based on the use of its oil and other derivatives. *Jatropha* is unique renewable energy sources in terms of the number of potential benefits that can be expected to result from its widespread cultivation. Its cultivation requires simple technology. The seed yield for *Jatropha* varies from 0.5 to 12 tons /year/ha-depending on the soil, nutrient and rainfall conditions- and the trees have a protective life of over 30 years. An average annual seed production of about five tons/ha can be expected on good soil when rainfall is 900-1,200mm. The seeds contain about 30% oil that can be converted into biodiesel by a process called Transesterification, in which a simple alcohol (e.g. methanol) replaces glycerol from the vegetable oil molecules (these are triglycerides, i.e.; three molecules of fatty acid molecules are attached to a glycerol molecule). The suitability of *Jatropha* seed oil for transesterification into biodiesel has also been clearly demonstrated⁴.

Vegetable oils as biodiesel source: The use of vegetable oils, such as palm, soyabean, sunflower, peanut, and olive oil as alternative fuel for diesel engines, depending upon the climate and the soil condition, different countries are looking for different types of vegetable oils as substitutes for diesel fuels. For example, soybean oil in the U.S, rapeseed and sunflower oils in Europe, palm oil in South East Asia, and coconut oils in the Philippines are being considered⁵.

Waste vegetable oil (Refinery waste) as a Biodiesel source:

Acid oil, which is a by-product in vegetable oil refining, mainly contains free fatty acids (FFAs) and acylglycerols, and is a candidate of material for Biodiesel fuel. In order to provide FAMES at a reasonable price, production of FAMES not only from refined vegetable oils, but also from crude or waste material and from by-products of oil processing has been attempted; one of the materials is acid oil.

Alkali deacidification, one of the steps in vegetable oil refining, by-produces soap stock that mainly contains soap and water.

Acid oil is obtained by acidulation of the soap stock, and contains free fatty acids (FFAs), acylglycerols, and other lipophylic compounds,

It is reproduced currently as industrial FFAs, although their demand is almost in saturation. Conversion of the acid oil to Biodiesel fuel is thus expected to avoid oversupply of the industrial FFAs and their price down.

A mixture (Acid oil model) or refined FFAs was recently reported to be converted to fatty acid methyl esters (FAMES) at >98% conversion by two step reaction system comprising methyl esterification of FFAs and methanolysis acylglycerols using immobilized *Candida antarctica* lipase⁶.

Material and Methods:

TRANSESTERIFICATION PROCESS – Steps involved in the transesterification process are-

Purification- This step includes washing or pretreatment and filtration and heating of oil to remove impurities.

Neutralization of the free fatty acids (Titration)- A sample of the cleaned oil is titrated against a standard solution of base in order to determine the concentration of free fatty acids (RCOOH) present in the *Jatropha curcas* oil and Acid oil sample.

Transesterification-

One-step alkali base catalyzed transesterification- One step alkali base catalyzed transesterification was carried out for methyl ester production process from *Jatropha curcas* oil and Acid oil. Firstly, in the transesterification process, different catalyst NaOH-to-oil ratios (0.5%-10% w/v) and different oil-methanol ratios (1:1, 1:2, 1:3, 1:4, 1:5 and 1:6 v/v) were used to investigate their influence on the methyl ester yields of the oils at fixed reaction temperature $55\pm 1^{\circ}\text{C}$. All the reactions were carried out in the reaction glass tubes, which were immersed in a glass water bath placed on the plate of magnetic stirrer of 400 rpm. After the reaction, the mixture was allowed to settle for 2 h.-overnight before separating the glycerol layer and the top layer including methyl ester fraction was removed.

Two-step acid-base catalyzed transesterification- In this process the first step was acid esterification or pretreatment for removing FFA in the oil, which is mainly a pretreatment process, which could reduce the FFA. The process was intended to convert FFA to esters using an acid catalyst (H_2SO_4 1% V/V) to reduce the FFA concentration of *Jatropha curcas* oil and Acid oil. Second step was alkali base catalyzed transesterification.

First step: Acid pretreatment- On this step, the *Jatropha curcas* oil and Acid oil was poured into the reaction glass tubes and heated. The solution of concentration H_2SO_4 acid (1.0% based on the oil weight) in methanol was heated at $55\pm 1^{\circ}\text{C}$ and then added into the reaction glass tubes. Different methanol to oil ratios by weight were used, namely at 1:1, 2:1, 3:1, 4:1, 5:1, 6:1 were investigated. After one hour of reaction, the mixture was allowed to settle for 2 h and the methanol–water fraction at the top layer was removed. The optimum condition having the lowest acid value was used for the main transesterification reaction.

Second step: Base catalyzed transesterification- Firstly, the oil product that has been pretreated from the first step was poured into the reaction glass tubes and heated at $55\pm 1^{\circ}\text{C}$. The solution of NaOH in methanol at 0.5%-10% v/v of the oil were heated to $55\pm 1^{\circ}\text{C}$ prior to addition and then added to the heated oil. The reaction mixture was heated and stirred again at $55\pm 1^{\circ}\text{C}$ and 400 rpm for 2 h. The mixture was allowed to settle 2 h or overnight before separate the glycerol layer to get the methyl ester layer of fatty acids on the top.

Down stream processing

Glycerol and esters separation- After giving the optimum conditions like reaction temperature and reaction time etc the reaction products were allowed to separating funnel. The products of the transesterification process i.e. methyl ester and glycerol form the upper and lower layers, respectively. The bottom layer of glycerol was removed, and upper layer of Biodiesel was mixed with warm distilled water (10% v/v) in order to remove the impurities like unreacted methanol, unreacted oil and catalyst. The mixture was again allowed to settle down under gravity for 6 h, and the lower layer of water containing impurities was drained out.

Alcohol Removal- Once the glycerin and biodiesel phases have been separated, the excess alcohol in each phase is removed with a flash evaporation process or by distillation or treating at water bath.

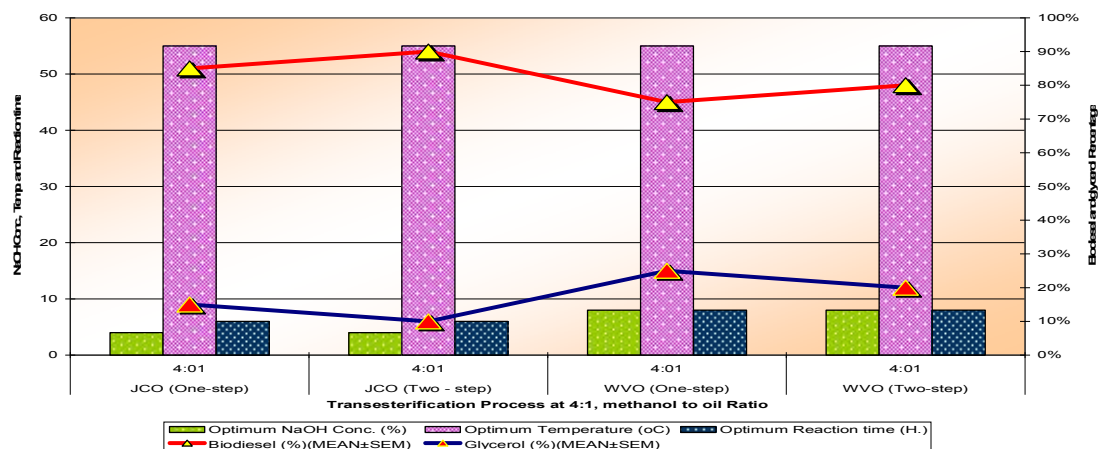
Results and Discussions: The above studies shows that for one- step alkali base catalyzed transesterification reaction under optimum conditions Fatty acid methyl esters yield was 85% for *Jatropha curcas* oil and 75% for Acid oil. For two-step Acid- Base

catalyzed Transesterification reaction, the fatty acid methyl ester yield was 90% for *Jatropha curcas* oil and 80% for Acid oil.

Effect of Molar ratio of Methanol to oil on Transesterification: One of the most parameter affecting the yield of esters is the molar ratio of Methanol to oil. Methanol was used in the range of 1:1 to 5:1 (molar ratio of methanol to oil), keeping other parameters fixed. The reaction temperature was kept constant at $55\pm 1^{\circ}\text{C}$, and reaction was performed with for 6-8h. The reaction was performed with different concentrations of NaOH. The results are shown in table 3.3. The max. Conversion was obtained at the ratio of 4:1, methanol to oil ratio for one-step base catalyzed transesterification of *Jatropha curcas* oil and Waste vegetable oil, and also for two-step process of acid-base catalyzed transesterification of *Jatropha curcas* oil and Waste vegetable oil. The reason behind using 4:1 ratio of methanol to oil rather than 3:1 for max. conversion, was that excess quantity of alcohol or methanol is required to 'drives' the reaction closer to the 99.7% yield, we need to meet the total glycerol standard for fuel grade biodiesel.

Effect of Catalyst Concentration on Transesterification: The effect of NaOH concentration was studied in the range of 0.5%-10% (weight of NaOH/Volume of oil). The reaction temperature and reaction time were kept constant. It was found that the ester yield decreases as the amount of catalyst increased from 4% for *Jatropha curcas* oil and 8% for Waste vegetable oils and reduces the almost 50% of yields of methyl esters. This lesser yield at high NaOH concentration may possibly be due to soap formation. These viscosities first decreases up to 2.5% NaOH concentrations and after that it is almost constant. Excess NaOH reduces the yield and leads to undesirable extra processing cost because it is necessary to remove it from the reaction products at the end.

Effect of Reaction Temperature on Transesterification: Reaction temperature is also an important variable that affected the transesterification reaction. For studying the effect of reaction temperature on the transesterification reaction, the reaction temperature was varied as 35, 40, 50, 55, and 60°C , while the other parameters such as molar ratio of methanol and NaOH concentration were kept constant. It was found that the ester yield increases as the reaction temperature increases till the $55\pm 1^{\circ}\text{C}$ and then ester yield decrease as the reaction temperature increases above $55\pm 1^{\circ}\text{C}$. The optimum conversion was obtained at $55\pm 1^{\circ}\text{C}$. The reason behind the maximum conversion obtained at $55\pm 1^{\circ}\text{C}$ reaction temperature was that the transesterification reaction be conducted close to the boiling point of methanol ($55-65^{\circ}\text{C}$) at atmospheric pressure.



Effect of Reaction Time on Transesterification:

Reaction time is also an important variable that affect the reaction very much. It was observed that the ester yield increases as the reaction time increases. Reaction starts very fast and almost 80% of the conversion takes place in first 5 minute and after 1 hour almost 90- 93% conversion of triglycerides into esters takes place but it take 6-8 hours in finishing.

Conclusion: The focus of the investigation carried during this work was oriented towards the conversion of high viscous oils to Biodiesel or Fatty acid methyl esters. Vegetable oils can be used as diesel fuel but due to high viscosity they cause many problems like poor atomization, incomplete combustion, leading to heavy smoke emissions and high flash point of oil attributes to lower volatility characteristics. The indiscriminate extraction and consumption of fossil fuels have led to a reduction in petroleum reserves, so alternative fuels, energy conservation and management, energy efficiency and environmental protection have become very important in recent years. There are four ways in which oils and fuels can be converted into Biodiesel, namely Blending, Pyrolysis, Micro emulsion and Transesterification. Alkali or Base catalyzed transesterification is the promising area of research for production of Biodiesel.

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