International Journal of Students' Research in Technology & Management
Vol 2 (04), June-July 2014, ISSN 2321-2543, pg 153-156

Study of Microwave Radiation on Transesterification of Jatropha Oil in Presence of Alkali Catalyst

Nadira Hassan Mohammed Al Balushi[#1], Priy Brat Dwivedi[*2]

[1]*Student*, [2]*Project Guide, Mechanical & Industrial Engineering Department*

Caledonian College of Engineering, Muscat, Oman

Corresponding author: nadraalbalushi@hotmail.com

***Abstract*— The objectives of this study is to produce biodiesel from Jatropha oil using microwave radiation in presence of alkali catalyst and designing suitable batch reactor for lab scale production. Cost effectiveness of the project is also being studied. This paper outlines studies done to find the optimal method for converting Jatropha oil to useable biodiesel using microwave irradiation. The amount of acid catalyst is 0.4w % and ratio of methanol to oil is 6:1 w/w for the optimal trans-esterification.**

Keywords: **Jatropha oil, Biodiesel, Catalyst, Microwave radiation, Trans-esterification**

I. Introduction

Oil is running out. In the short term it will continue to go up in price and in the middle distant future it will be too expensive to burn. As the world energy demand and consumption increases every day, we need to focus on the use of biofuels that will help extend the lifetime of our oil supply, but eventually we will need to replace oil. Whatever that replacement is it needs to be sustainable.

By 2030, global energy consumption is projected to grow by 36% [1] and, in our view; demand for liquid transport fuels will rise by some 16 million barrels more a day. With the world's population projected to reach 8.3 billion by then, an additional 1.3 billion people will need energy. To meet this demand a diverse energy mix is needed. This is where biofuels can help; in the next two decades, biofuels is expected to provide some 20% (by energy) of the growth in fuel for road transport [2]. The possibility of deriving bioduesel from locally grown sources and using them as alternatives to petro diesel products is attractive for many countries, including the Sultanate of Oman, that currently depend largely on fossil fuels.

Biodiesel is fuel that is similar to diesel fuel and is derived from usually vegetable sources. Biodiesel refers to a vegetable oil- or animal fat-based diesel fuel consisting of long-chain alkyl (methyl, ethyl, or propyl) esters. Biodiesel is typically made by chemically reacting lipids (e.g., vegetable oil, animal fat (tallow) with an alcohol producing fatty acid esters (FAE).

Biodiesel helps reduce greenhouse gas emissions (GHGs) because it comes from animal or plant biomass with a lifecycle of a few years. On the other hand, petro diesel is a fossil fuel that releases into the atmosphere carbon that has been tied up for hundreds of millions of years, and all of it adds to GHGs. Fossil fuels also release more tailpipe emissions than does biodiesel. Biodiesel is a liquid which varies in color between golden and dark brown depending on the production feedstock. It is slightly miscible with water, has a high boiling point and low vapor pressure. The flash point of biodiesel (>130 °C, >266 °F) is significantly higher than that of petroleum diesel (64 °C, 147 °F) or gasoline (−45 °C, -52 °F). Biodiesel has a density of ~ 0.88 g/cm³, higher than petrodiesel (~ 0.85 g/cm³). Most diesel engines are warranted to run on anywhere between B5 (5% biodiesel) to B20 (20% biodiesel). [3] Have discussed few chemical and physical properties of jatropha oil. (Table 1).

Kapilan [5] has used microwave radiation for two step transesterification in his work and reported successful production of biodiesel from jatropha oil grown in Indian soil. Antony Raja, et al. [6] reported that Jatropha oil is converted into jatropha oil methyl ester known as (biodiesel) prepared in the presence of homogeneous acid catalyst. The same characteristics study was also carried out for the diesel fuel for obtaining the base line data for analysis.

TABLE I

CHEMICAL AND PHYSICAL PROPERTIES [4]

Parameter	Value
% FFA as oleic acid	2.23±0.02
Iodine value	103.62±0.07
Saponification value	193.55±0.61
Peroxide value	1.93±0.012
Percentage oil content (kernel)	63.16±0.35
Density at 20° C (g/ml)	0.90317
Viscosity at room temperature (cp)	42.88
Physical state at room temperatur	Liquid

A Value is mean ± standard deviation of triplicate determinations.

Marchetti, et al. [7] concluded that there are different ways of production, with different kinds of raw materials: refine, crude or frying oils. Also with different types of catalyst, basic ones such as sodium or potassium hydroxides, acids such as sulfuric acid and ion exchange resins. One of the advantages of this fuel is that the raw materials used to produce it are natural and renewable. Also of this process, the free fatty acid will be changed completely in to esters. Bojan, et al. [8] carried out his work to produce biodiesel from crude Jatropha Curcas oil (CJCO) with a having high free fatty acid (HFFA) contents (6.85%) and also the crude Jatropha Curcas oil was processed in two steps. During the first step the free fatty acid content of crude Jatropha Curcas oil was reduced to 1.12% in one hour at 60°C using 9:1 methanol to oil molar ratio. The second step was alkali catalyzed transesterification using methanol to oil molar ratio of 5.41:1 to produce biodiesel from the product of the first step at 60°C.The maximum yield of biodiesel was 93% v/v of crude Jatropha Curcas oil which was more than the biodiesel yield (80.5%) from the one step alkali catalyzed transesterification process. Temu, et al. [9] reported that the quality of biodiesel is influenced by the nature of feedstock and the production processes employed. The physico-chemical properties of jatropha and castor oils were assessed for their potential in biodiesel. The properties of jatropha and castor oils were compared with those of palm from literature while that of biodiesel were compared with petro-diesel. Results showed that high amounts of FFA in oils produced low quality biodiesel while neutralized oils with low amounts of FFA produced high quality biodiesel.

In current study locally grown jatropha oil was taken as feed stock and two step transesterification was done by microwave radiation.

Antony, et al. [10] reported that all countries are at present heavily dependent on petroleum fuels for transportation and agricultural machinery. The fact that a few nations together produce the bulk of petroleum has led to high price fluctuation and uncertainties in supply for the consuming nations. This in turn has led them to look for alternative fuels that they themselves can produce. Among the alternatives being considered are methanol, ethanol, biogas and vegetable oils. Vegetable oils have certain features that make them attractive as substitute for Diesel fuels. Vegetable oil has the characteristics compatible with the CI engine systems. Vegetable oils are also miscible with diesel fuel in any proportion and can be used as extenders. Ronnie, et al. [11] concluded that the benefits of jatropha as biodiesel include the reduction of greenhouse gas emissions, as well as the country's oil imports. Local production of jatropha is also practical because as a non-food crop, it will not compete with food supply demands. It can also grow on marginal degraded land, leaving prime agricultural lots for food crops while at the same time restoring the marginal and degraded land's fertility. All of these benefits can possibly be achieved by the presence of this locally fabricated high efficiency jatropha oil extractor equipment.

This mixture was heated in LG make domestic microwave oven with occasional shaking for 60 seconds. Power level was set at 160 W. This pretreatment was done with every set before mixture was set for transesterification. This pre-treated jatropha oil was used in base catalysed second-step transesterification.

In the second step, transesterification was carried out at with various methanol-to- oil ratio, at various catalyst strength, and various time duration. In this step also power supply 160 W. Results of variations are summarized in table 2. After the reaction, the excess methanol was removed by vacuum distillation and then the trans-esterification products were poured into a separating funnel for phase separation. After phase separation, the top layer (biodiesel), was separated and washed with distilled water in order to remove the impurities. Then the biodiesel was heated above 1000C, to remove the moisture.

Fig 1: Conventional Heating

II. MATERIALS

For current study, Jatropha oil was purchased from local market in Salalah, Oman. This oil was filtered and then used for the production of biodiesel. Sulphric acid (H_2SO_4) is used as acid catalyst in first step and KOH was used as catalyst in second step. In our study we used Methanol for transesterification. Because methanol is cheaper and has better physical and chemical properties (polar and shortest chain alcohol). Potassium hydroxide, methanol and sulphuric acid were purchased from Schalau, Chemie S.A, Spain. All the chemicals used for transesterification were of analytical reagent grade. Study was done in LG domestic microwave oven at 160 W power levels.

Fig 2: Microwave-assisted biodiesel production units.

III. Biodiesel Production

Acid value of Jatropha oil was determined by standard method and it was found as 9 mg KOH per g of oil. Since acid value is higher than 1 mg KOH, acid catalyzed trans-esterification is necessary in first step. Acid catalyzed trans-esterification is good if oil is having high free fatty acid content. It avoids possibility of soap formation like in case of alkali catalyst. In this pretreatment, methanol-to-oil ratio was taken as 4:1 w/w and 0.4 w% of H_2SO_4 was

IV. Results and Discussion

Conventional heating set was also studied (Figure 1) with 5g of jatropha oil, 40 ml methanol and 5 hrs of refluxing. Biodiesel yield was 3.09g. From table 2 it is clear that microwave radiation is one of the best tools for transesterification of Jatropha oil. During experiment various ratios of methanol to jatropha oil was tested. Results are summarized in table 2 (entries 1, 2 and 3). Optimum yield was found when methanol to oil ratio was 6:1. Later yield was decreasing with increasing the amount of methanol. More study is required in this area to find the reasons behind this observation. In case of alkali catalyst variation, (entries 4, 5 and 6 in table 2) biodiesel yield was increasing with increase in alkali catalyst concentration. But due to of possibility of soap formation and difficulty in product separation, catalyst ratio was not studied beyond 0.8 w%. During this study, effect of time was also studied and results are summarized in table 2 (entries number 7, 8, and 9). Yield of biodiesel was found to be increasing with time. But to avoid bumping and overheating, no study was done after 200 seconds.

Biodiesel production by microwave irradiation was due to direct adsorption of the radiation by the polar group (OH group) of methanol. It is speculated that the OH group is directly excited by microwave radiation, and the local temperature around the OH group would be very much higher than its environment. Hence, microwave assisted transesterification is a way of reducing the reaction time, the electrical energy and labor costs as compared to the conventional method.

TABLE II

Summary of Microwave Heating Variation

No.	Oil(g)	Methanol (g)	Catalyst (g)	Time(s)	Yield (g)
1	5g	30g	0.02g	80s	4.8
2	5g	40g	0.02g	80s	4.68
3	5g	50g	0.02g	80s	3.97
4	5g	40g	0.01g	140s	4.06
5	5g	40g	0.02g	140s	4.10
6	5g	40g	0.04g	140s	4.21
7	5g	50g	0.02g	80s	3.9
8	5g	50g	0.02g	140s	4.18
9	5g	50g	0.02g	200s	4.26

After variation, biodiesel properties were tested as per ASTM D 6751, for various parameters as given in table 3.

TABLE III

Fuel Properties

Property	ASTM D6751	Biodiesel	Diesel
Flash point (°C)	> 130	128	68
Pour point (°C)	-	– 7	−15
Calorific Value (MJ/kg)	-	39.9	42.71
Viscosity at 40 °C (mm2/sec)	1.9–6	4.20	2.28
Density at 15 °C (kg/m3)	–	901	846
Water content (mg/kg)	< 500	99	102
Acid number (mg KOH/g)	< 0.50	0.80	0.34
Copper strip corrosion	>No. 3	1	1
Ash Content (%)	< 0.02	0.01	0.01

Table III compares the properties of jatropha biodiesel produced in this study with the properties of diesel. The flash point of biodiesel satisfies the fuel standards and is better than the flashpoint diesel. This is an important safety consideration when handling and storing flammable materials. The important cold flow properties of biodiesel are the cloud and pour point.

According to ASTM standard D 6751, no limit is given for pour point and suggested "report" in the fuel standard. The calorific value is an important property of biodiesel that determines its suitability as an alternative to diesel. As per European standard, EN 14214, the approved calorific value for biodiesel is 35 MJ per kg. The table shows that the calorific value of jatropha biodiesel is close to that of diesel. According to the ASTM standards, the acceptable viscosity range for biodiesel is between 1.9–6.0 mm^2/s at 40^0C, and jatropha biodiesel satisfies the biodiesel standard. The density of jatropha biodiesel is close to that of diesel and satisfies the ASTM standard. ASTM standard approves a maximum acid value for biodiesel of 0.5 mg KOH/g, but jatropha biodiesel produced in this study has acid value 0.80 mg. The degree of tarnish on the corroded copper strip correlates to the overall corrosiveness of the fuel sample. The copper strip corrosion property of jatropha biodiesel is within the specifications of ASTM. Another important factor of biodiesel is the ash content estimation. The ash content of jatropha biodiesel satisfies the ASTM standard.

V. Conclusions

In this work, biodiesel was produced from jatropha oil using microwave radiation and with the help of two-step transesterfication. It was observed that microwave radiation helps the synthesis of methyl esters (biodiesel) from non-edible oil, and higher biodiesel conversion can be obtained within a few minutes, whereas the conventional heating process takes more than 5 hrs.

In the current investigation, it has confirmed that Jatropha oil may be used as resource to obtain biodiesel. The experimental result shows that alkali catalyzed transesterification is a promising area of research for the production of biodiesel in large scale. Effects of different parameters such as time, reactant ratio and catalyst concentration on the biodiesel yield were analyzed. The best combination of the parameters was found as 6:1 w/w ratio of Methanol to oil, 0.8 w% of KOH as catalyst and 200 seconds of reaction time. The viscosity of Jatropha oil reduces substantially after transesterification and is comparable to diesel. Biodiesel characteristics like density, viscosity, flash point, and pour point were studied and are found as comparable to diesel.

Acknowledgement

Authors are thankful to Caledonian College of Engineering, Muscat, for supporting this work.

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