Optimized Continous Flow Production of Bio-Diesel Using Domestic Microwave

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Abstract
Continuous flow production of Biodiesel from vegetable oil using a domestic microwave oven was investigated as an alternative to batch processing at 50^0 C for one hour with continuous heating and stirring and approximately 14 hours of time for separation. Trans-esterification of vegetable oil with methanol from microwave heating in the presence of catalyst-potassium hydroxide yields methyl-ester or bio-diesel as a product and glycerin as a by-product. Experiments conducted in the microwave indicated that the continuous production of biodiesel can be accomplished by taking vegetable oil and methanol in the ratio of 1:5 with the micro wave irradiation of the mixture at its exit power of 1200 W. Optimization of extraction techniques has also been discussed.

Keywords
Biodiesel, Diesel, Renewable Fuel, Alternative Fuel. Animal Fats, Designated B100, Microwave Heating or Irradiation, Trans-Esterification and Catalyst

I. Introduction

Fossil fuels continue to be our mainstay in the near future; with the diminishing resources of fossil fuel day by day and increased population [1] and usage of fuels, there is a constant need for long-lasting fuel resource to the human kind. Apart from the growing demand for energy, there have also been problems associated with vehicular emissions and environmental damage due to massive usage of fossil fuels. This has brought pressure on many countries of the world to reduce their CO_2 emissions and go for the use of cheaper and nonpolluting energy sources [2].

Renewable energy technologies such as Biodiesel usage, solar energy, fuel cell energy are receiving increased attention as attractive energy supply options for meeting human needs like electric utility, heating, industrial power usage etc. These are less polluting and contribute less to the global warming problem. However, they do not solve the demand problem created by exponential population growth [3]. The momentum for this growing attention is coming from the actions of national and state governments, environmental advocacy groups, state regulatory organizations, and from within the utilities themselves.

This paper discusses the need for alternative and renewable energy source specifically about biodiesel, its production practices, environmental and health effects of biodiesel highlighting continuous production and analysis of biodiesel using microwave heating instead of manual heating.

Alternative, renewable energy sources are indigenous, and can therefore contribute further to reducing dependency on energy imports and increasing security of supply. Renewable energy sources still make an unacceptably modest contribution to the Community's energy balance as compared with the available technical potential.

A. Bio-energy

Bioenergy is Biomass (burning biological and biodegradable materials to generate heat), biofuels (processing biological materials to generate fuels such as biodiesel and ethanol), and biogas (using anaerobic digestion to generate methane from biodegradable. One of the prominent features of biodiesel is that, its usage significantly reduces greenhouse gas emissions and serious toxic air pollutants. Biodiesel provides numerous environmental and economic benefits. It gives us energy independence and it endows us with many environmental benefits. Biodiesel is a domestically produced, renewable alternative fuel that can be manufactured from vegetable oils, animal fats, or recycled restaurant greases which is safe and biodegradable [4-5].

1. Biodiesel Compared to Petroleum & Diesel

Biodiesel possesses several distinct advantages over petro-diesel in the following safety, biodegradability and environmental aspects such as (Guo and Young et al.2006) [6], biodiesel yields 3.2 units of fuel energy for every unit of fossil fuel consumed in its life cycle, where as Petroleum diesel yields 0.83 units of fuel energy per unit of energy consumed, Higher Flash point makes it safer in transport and storage. Biodegrades as fast as dextrose and reduces Polycyclic Aromatic Hydrocarbons (PAH) and nitro PAH; Heating value is usually expressed as British thermal units (Btu) per pound or per gallon at 60°F (International metric [SI] units are kilojoules per kilogram or per cubic meter at 15°C). The energy content of biodiesel is much less variable than that of petro diesel, and with biodiesel meeting D 6751 standards the energy content is more dependent upon the feed stocks used than the particular process [7]. Liquid biofuels have bulk densities comparable to those for fossil fuels. Biodiesel is the only alternative fuel to have fully completed the health effects testing requirements of the Clean Air Act. [8].

Table 1: Biodiesel Properties

Fuel Properties	Biodiesel
Fuel Standard	ASTM 6751-02
Fuel composition	C12-C22 FAME
Lower Heating Value, Btu/gal	117,093
Kinetic Viscosity,@40 deg C.	1.9-6.0
Specific Gravity kg/1@ 60 deg F.	0.88
Density, lb/gal @15 deg C.	7.0328
Water ppm by wt.	.05% max
Carbon, wt %	77
Hydrgen, wt%	12
Oxegen, by dif wt. %	11
Sulfer, wt %	0.0-0.0024
Boiling point, deg C.	182-338
Flash Point, deg C.	100-170
Cloud Point deg C. -	3 to 12
Pour Point deg C.	-15 to 10
Cetane Number	48- 65
Stoichiometric Air/Fuel Ratio	13.8

Biodiesel is often blended with petroleum diesel to produce a fuel that is compatible with diesel engines. Biodiesel blends reduce harmful emissions. Biodiesel blends will become more common as drivers are made aware of the many benefits. All blends can be used in the vehicles without modifications.

II. Materials and Methods

A. Biodiesel Production

1. Biodiesel Processing Techniques

There are three basic routes to ester production [9] from oils and fats:

- Base catalyzed trans-esterification of the oil with alcohol.
- Direct acid catalyzed esterification of the oil with methanol.
- Conversion of the oil to fatty acids, and then to alkyl esters with acid catalysis.

The most commonly used catalyst in transesterification reactions are NaOH, KOH and H_2SO_4.The majority of the alkyl esters produced today is done with the base catalyzed reaction because it is the most economic for several reasons:

- Low temperature 50^{0}C and atmospheric pressure processing.
- High conversion (98%) with minimal side reactions and reaction time.
- Direct conversion to methyl ester with no intermediate steps.

B. Microwave-Batch and Continuous

By using microwave heating as a fast simple way to produce biodiesel continuously for transesterification we get high grade biodiesel [10]. The three steps that occur in biodiesel transesterification are:

- Transesterification
- Phase separation
- Washing

1. Experimental Design

(i). Materials

Fresh vegetable (canola) oil, anhydrous Potassium Hydroxide, Methyl alcohol of laboratory grade from Fischer used as purchased without any further purification.

Fig. 1: Experimental Setup

Fig. 2: Tubular Reactor

A poly propylene tubing of internal diameter of 0.25 in was taken and tested for the flow rates. We obtained varying flow rates with pumping water through the pump. Then after getting a consistent set of results, we used vegetable oil and optimized the flow rates

The reaction mixture of 500 ml vegetable oil is mixed with 100 ml of methanol that has pre dissolved amount of 2.9 g KOH. The contents were transferred into a conical flask and heated for 5 minutes in microwave at 30^{0}C.Similarly heating time of 10, 15 minutes was tried but was unable to reach the desired 50^{0}C for optimum seperation.When tested for the quality of biodiesel it was not up to the desired consistency which implied that mixing and more heating was required. Then another sample was sent with continuous pre-mixing of the contents before entering into the microwave by means of a pump and obtained good quality fuel.

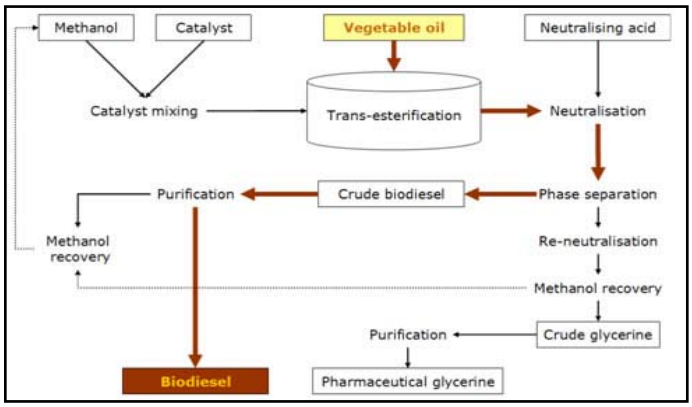

Fig. 3:

This was repeated several times for the optimization of the pump settings, temperature, and catalyst amount and mainly for the residence time inside the tubular reactor contained in microwave by varying each parameter in different combinations [11]. The optimum conditions obtained were giving the good quality fuel. It was observed that the batches which produces limited amount of biodiesel, take long time for each batch to separate and more labor. Continuous heating of vegetable oil mixture in the microwave oven through pumping seemed to be more efficient in terms of productivity, time, quality and labor [12]. So we chose to go ahead with the continuous production of biodiesel by microwave heating. So far we achieve only separation of bio diesel and glycerin, to ensure efficient separation and more bio diesel optimization is required as explained in the subsequent section.

2. Glycerin byproduct

So we got second layer separated as glycerin, what we can use glycerin in the following ways.

Fig. 4:

Fig. 5:

Fig. 6:

- Further processing it into soap
- Separating the lye and re using it as catalyst again will improve the efficiency of the process.
- Can be used as fuel for wood stove.

Most of the glycerin can be recovered from the byproduct by using a technique called acidulation. This process uses phosphoric acid (H_3PO_4) to separate the soap into free fatty acids and salts. This method results in separation of the byproduct into three stinct layers: an upper layer of (Free Fatty Acids) FFAs, a middle layer of glycerol, and a bottom layer of salts.

Biodiesel Blend, n—a blend of biodiesel fuel meeting ASTM D 6751 with petroleum-based diesel fuel, designated BXX, where XX represents the volume percentage of biodiesel fuel in the blend.

Fig. 7: Various Biodiesel Blends

The 3 common blends are:

- B2 - 2% biodiesel and 98% diesel
- B5 - 5% biodiesel and 95% diesel
- B20 - 20% biodiesel and 80% diesel

3. Biodiesel Extraction and Optimization

There are different ways to determine the best procedure to optimize biodiesel extraction.

The step that takes the longest is washing.

Washing is a process that removes unwanted contaminants from biodiesel by making biodiesel to come in contact with water to transfer hydrophilic compounds from the biodiesel to the water. It can be achieved by the following processes.

(i). Temp Effects on Emulsion Separation

In this three emulsion samples are taken and one is placed in water bath at 60°C, the next in a water bath at 40°C, and the last is left in ambient air and these samples are left for at least six hours. It was observed that higher the temperature, the faster the sample should separate.

(ii). Salting to Bust Emulsion

Start with emulsion samples made from the wash test. Weigh and add a small amount of salt to the sample then shake it again. If it doesn't separate, weigh and add more salt and then shake again. Repeat until the emulsion breaks solid salt particles become visible in the samples. One emulsion sample for each salt is used. The salts tested in increasing price order are NaCl, $CaCl_2$, and KI. NaCl is the best choice if it works because it is by far the cheapest.

Solubility testing of several contaminants was performed with the ASTM certified biodiesel. We found that glycerol and sodium hydroxide would not appreciably dissolve in biodiesel. Testing was conducted by adding a small amount of contaminant and stirring to see if it would dissolve. Methanol did dissolve at first, but then separated out to the top of the biodiesel.

A single batch wash of biodiesel was carried ouy. This is where we first encountered our emulsion problem. Initially the biodiesel would separate after a few minutes, but the emulsion layer remained and was completely stable.

Fig. 8 & 9: Left Shows the Biodiesel and Water Samples Taken 2 Minutes Into the Continuous Washing, Right Shows the Effluent Samples Taken at the 10 Minute Mark

Salting

Fig. 10: Emulsions Left Over With Different Levels of Salt Concentration (Increasing From Left to Right).

III. Results and Discussions

By using domestic microwave for continuous production of biodiesel, we are able to produce good quality biodiesel. The optimum conditions were 7.5 ml/min of biodiesel flow at 50°C. The analysis of the biodiesel is as followed.

From the experiments it was observed that at 50°C the vegetable oil conversion is good making it the optimum temperature for

the transesterification reaction and also the obtained feasible conditions like a catalyst requirement of 2.9g KOH, residence time of 8 min for the vegetable oil inside the tubular reactor in the micro oven suggest that a good quality biodiesel can be produced provided the optimum conditions of the reaction exist. So we can process the vegetable oil to get biodiesel in the large scale also by configuring the conditions.

A. Bio Diesel Analysis

In order to assess the quality which is of primary concern when we use bio diesel in the vehicles, the product was tested. The obtained biodiesel was taken in small amount and divided into two equal parts. One half was washed with a 10% acetic acid solution, to be followed by the water washing using de-ionized water. The other half was washed with neat acetic acid followed by washing with a 10% acetic acid solution.

Subsequent washings with de-ionized water until the ester layer becomes clear and the washings were neutral to litmus. Addition of a 10% acetic acid solution to the ester layer resulted in emulsion formation that was enhanced on addition of the de- ionized water. However, initial washing of the ester with neat acetic acid resulted in complete separation of the ester from the washings because no emulsions were formed on the later addition of the 10% acetic acid solution and water washing without emulsion formation was achieved. The improved method aims at solving the problem of emulsion formation during the washing stage. The reaction of the ester and water gives fatty acid and alcohol. There may also be fatty acids present because of incomplete reaction of the tri-acyl-glycerol.

Fig. 11: Quality Test for Biodiesel

Fig. 12: Fresh and Ready to Use Bio- Diesel

The fatty acid may then react with the excess base to give soaps. Therefore neutralizing the base after the reaction time will reduce the occurrence of these reactions. In the method, acetic acid was used to neutralize the excess catalyst so as to achieve water washing without emulsion formation. The acetic acid neutralizes the excess catalyst and prevents the further reaction of the un reacted oil or incomplete reaction products with the base to form soaps.

IV. Conclusions and Recommendations

Investigation of continuous microwave heating of vegetable oil to produce biodiesel suggests that it is possible to produce biodiesel in better and faster way than using conservative manual heating which takes long time and which produces biodiesel in batches that is on smaller scale. The problem of NO_x can be solved by using technologies like Selective Catalytic Reduction (SCR) or Exhaust Gas Recirculation (EGR).

There is scope for large scale production biodiesel from used vegetable oil obtained from restaurants or vegetable oil produced from the biodiesel seed crops like rape seed or palm trees especially grown for industrial use of biodiesel production. Efficient research work to investigate the technologies and conditions capable of handling various feed-stocks including waste vegetable oil, poultry fat, and refined vegetable oils such as corn, canola and soybean oils that can produce the best quality biodiesel in an economic and environmental friendly way.

Alternative fuels and energy sources provide an excellent opportunity to yield considerable benefit to the general public like shifting from petroleum fuels to domestically produced biofuels would create millions of jobs, improve our economy, reduce pollution enormously, and eliminate a key strategic concern for all countries - the dependence on foreign fuels.

In summary, we were able to develop a continuous- flow production of biodiesel from vegetable oil by domestic microwave heating, which offers clean burning, economic and pollution free fuel that can be prepared fast and easily.

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