Article

Organic Synthesis of Bio-Diesel from Waste Cooking Oil by Microwave Irradiation

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Abstract

In recent times, environmentally benign and economically viable alternatives to fossil derived fuels are seriously being explored due to increasing global demand for energy, coupled with the threats posed by the recent climate changes. With the increasing concern of the environment and more stringent regulations on exhaust emissions, so an experiment was made to reclaimed vegetable oil from restaurants and use it as a fuel for vehicles. Biodiesel is well-accepted alternative to diesel fuels as they are economically feasible, renewable, environmental-friendly and can be produced easily in rural areas where there is an acute need for modern forms of energy. This environmentally threatening problem could be turned into both economical and environmental benefit by proper utilization and management of waste cooking oil as a fuel substitute. Biodiesel is a mixture of fatty acid methyl esters produced from the transesterification of plant oils or animal fats with methanol over alkali or acid catalysts. Energy utilization and specific energy requirements for Microwave based biodiesel synthesis are reportedly better than conventional techniques. Alkali process yields high quantity and high purity biodiesel in shorter reaction time In this research, around 50^{o}C reaction temperature, reaction time of 5 minutes, 6:1methanol to used cooking oil ratio, 1000W microwave power output and NaOH catalyst has been used to yield 93% of bio diesel. The determined specifications of obtained biodiesel according to ASTM D6751 and EN14214 standards were in accordance with the required limits. The results showed that application of radio frequency microwave energy offers a fast, easy route to this valuable bio-fuel with advantages of enhancing the reaction rate and improving the separation process usage.

Introduction

Biodiesel is defined by ASTM as “a fuel comprised of monoalkyl esters of long-chain fatty acids derived from vegetable oils or animal fats, designated B100”. Biodiesel is produced by a chemical process known as transesterification, by which the triglycerides are reacted with alcohols, in the presence of a catalyst, to produce fatty acid alkyl esters. The byproduct of transesterification is glycerin, also known as glycerol. Since the most common alcohol used to produce biodiesel is methanol, another name for biodiesel is fatty acid methyl esters (FAME). The physical and chemical properties of biodiesel are determined by the compositional profiles. Biodiesel properties can vary substantially from one feedstock to the next. The properties of individual fuels can vary because of its considerable oxygen content (typically 11%), biodiesel has lower carbon and hydrogen contents compared to diesel fuel, resulting in about a 10% lower mass energy content. However, due to biodiesel's higher fuel density, its volumetric energy content is only about 5–6% lower than petroleum diesel [1]. The quality of biodiesel can be influenced by a number of factors: the quality of the feedstock, the fatty acid composition of the parent vegetable oil or animal fat, the production process and the other materials used in this process, handling and storage. Given the fact that most current diesel engines are designed to be powered by diesel fuel, the physicochemical properties of biodiesel should be similar to those of diesel oil [2].Besides these aspects, biodiesel can absorb a certain amount of water during storage. Another example is the susceptibility, of linoleic acid esters especially, to oxidation. The storage parameters also can affect fuel quality [3]. In a conventional transesterification process, Sunflower seed oils, methanol and NaOH in various concentrations were refluxed together equipped with a glass anchor shaped mechanical stirrer, a water condenser and funnel. After the complete conversion of the vegetable oil, the reaction was stopped and the mixture was allowed to stand for phase separation. The ester mixture formed the upper layer and glycerin formed the lower layer. The residual catalyst and alcohol were distributed between the two phases [4].The study of transesterification reaction of fresh cooking oil showed that the main factors affecting the process are the molar ratio methanol / oil, the catalyst amount, and the reaction time. The result also showed that application of radio frequency microwave energy offers a fast, easy route to this valuable biofuel with advantages of enhancing the reaction rate and improving the separation process. The reaction time was reduced to 3 minutes for waste cooking oil instead of 150 minutes in conventional experiment, because, by using the microwave technique, no pretreatment is required[5].A diesel engine works by creating heat by compressing air followed by the injection of fuel which burns, increasing pressure to drive the pistons. Early diesel engines were intended to run on peanut oil and were later adapted to run on the lower viscosity and cheaper petroleum diesel. Today, diesel engines are used in cars, buses and trucks. Biodiesel can be used directly in diesel engines or blended with petroleum based diesel fuel. Vegetable oils are esters of glycerin, commonly called triglycerides, with different fatty acids with the structure [6]

Table 1: palm oil standard values

Name of the Oil	Analysis			
	Specific Gravity	Saponification Value	Iodine value	Melting point

Palm Oil	0921-0.925/15°c	196-205	48-58	42-45

Table: 2 kinematic viscosities of organic liquids

Liquid	Temperature		Kinematic Viscosity
	(°F)	(°C)	Centistokes
Alcohol - ethyl (grain) C_2H_5OH	68 100	20 37.8	1.52 1.2
Alcohol - methyl (wood) CH_3OH	59 32	15 0	0.74 1.04
Diesel fuel 2D	100 130	37.8 54.4	2-6 1.-3.97
Diesel fuel 3D	100 130	37.8 54.4	6-11.75 3.97-6.78
Diesel fuel 4D	100 130	37.8 54.4	29.8 max 13.1 max
Diesel fuel 5D	122 160	50 71.1	86.6 max 35.2 max
Palms oil	100 130	37.8 54.4	47.8 26.4
Petroleum ether	60	15.6	31(est.)

Experimental

Measure out 10 ml of methanol and put into the 100 ml Erlenmeyer flask. Weigh out 0.5g of sodium hydroxide pellets. Crush these pellets and transfer the crushed solid into the flask containing the methanol. Place a stir bar into the flask, place it on a magnetic stirrer and stir for 5-10 minutes until the NaOH dissolves in methanol.

Vegetable oil	Used cooking oil (palm oil)
Alcohol	Methanol (CH_3OH) absolute
Catalyst	sodium hydroxide (NaOH)

Measure out 10 ml of vegetable oil, determine the mass of the oil and add this to the reaction flask containing methanol and catalyst. Place the solution in a microwave oven and heat to a temperature 50^{o}C for 5 minutes with continuous stirring. After heating for the required time, pour the mixture into a separator funnel and allow the solution to cool until the mixture separates into two layers. The upper layer will be the biodiesel and the lower layer will be mostly glycerin. Do not let it stand for too long as the lower layer may solidify.

Carbon residue Test

The estimation of carbon residue is carried out by conradson method , a weighed quantity of bio diesel is taken in a silica crucible which is placed in a wrought iron crucible having a cover with a small opening for the escape of volatile matter , these are then placed in a third iron crucible covered with a chimney shaped iron hood and heated slowly for 10 mins till flame appears finally strong heating Is done for about 15mins till the substance burns completely , the crucible is then cooled and the residue left out is weighed

$$\text{Carbon residue} = \frac{\text{weight of carbon left out} \times 100}{\text{Weight of bio diesel taken}}$$

Density

Density was measured using the standard method (BIS, 1972), capillary stopper relative density bottle of 50 ml capacity were used to determine density of biodiesel.

Determination of pH

Add 5 drops of your biodiesel to 1 ml of distilled water and mix thoroughly. Using pH paper, estimate the aqueous pH of your biodiesel.

Cloud and pour point

Cloud point is the temperature at which biowax in biodiesel forms cloudy in appearance and pour point is the temperature at which biodiesel becomes semi solid and loses its flow characteristics.

Flash and fire point

The lowest temperature at which bio diesel gives of vapors which will flash if brought in to contact with a flame is called the flash point of biodiesel. The lowest temperature will give enough vapors which on rising will begin a continuous flame above the biodiesel for at least 5 seconds is called fire point of bio diesel which is done by abel's apparatus

Acid value

In this process 1 gm of bio diesel is allowed to react with known concentration of KOH using phenolphthalein as indicator and measure the amount of KOH consumed.

Viscosity (Redwood Viscometer)

Clean the cylindrical oil cup and ensure the orifice tube is free from dirt. Close the orifice with ball valve. Place the 50 ml flask below the opening of the Orifice. Fill oil in the cylindrical oil cup and then fill the water in the water bath. Later insert the thermometers in their respective places to measure the oil and water bath temperatures. By heating the water bath, stirr the water bath and maintain the uniform temperature. At 40^{0}C temperature lift the balance valve and collect the heated oil in the 50 ml flask and note the time taken in seconds for the collecting the oil . A stop watch is used to measure the time taken. This time is called Redwood seconds. .

Result and discussion

Density

Density of the oil at 40^{o}C is 0.85 g/cc.

PH test:

aqueous pH of our biodiesel 10

Viscosity test

Table 3.Kinematic viscosity of biodiesel

Temperature of oil ^{0}C	Density of oil gm/ml	Time for collecting 50cc of oil in seconds	Kinematic viscosity ν Centi stokes
40	0.86	138	3.5

Table:4 conversion values of redwoods viscosity to kinematic viscosity

Viscometer Name	Range of flow time, T seconds	Kinematic viscosity, ν Stokes
Redwood I	T=34 to 100	0.00260T-1.79/T
	T≥ 100	0.00247T-0.50/T

Kinematic viscosity of the oil is given by, $\nu = At - B/t$
t - Time of flow in seconds
ν - Kinematic viscosity of the oil in centistokes
A & B - instrument constants

Conclusion

This research revealed that biodiesel could be produced successfully from waste cooking oil by alkali- catalyzed transesterification. The reaction time has been reduced by using a microwave oven for heating up the oil. The effects of different parameters such as reaction time, temperature, catalyst concentration and reactant ratio on the biodiesel yield were analyzed. The good combination of the parameters were found as 6:1 molar ratio of oil to ethanol, 0.5% KOH catalyst, 50°C reaction temperature and 2-5 minutes of reaction time. The viscosity of cooking oil reduces substantially after transesterification and is comparable to petro-diesel and the physical and chemical properties of biodiesel produced confirm to available standards.

Table 5. Comparison of fuel properties of biodiesel and Petro-diesel

Properties	Waste oil biodiesel	Petro-diesel	Biodiesel	Biodiesel
		ASTM D0975	ASTM D6751	EN 14214
Density at 40°C (kg/m3)	0.85	0.876	0.875- 0.90	0.86-0.90
Viscosity at 40°C	3.5	1.9-4.1	1.9-6.0	3.5-5.0
Specific gravity (kg/m3)	0.85	0.870	0.88	…..
Flash point °C	63	60-80	100-170	>120
Fire point °C	73			
Pour point	-4	-35 to -15	-15 to 16	…..
Cloud point	17	-15 to 5	-3 to 12	….
Acid value (KOH mg/g)	0.36	0.35	<0.8	<0.5
Ash content (%)	0.16	0.01	<0.02	<0.02
Water content (%)	0.004	0.02	0.03	<0.05

ASTM = American Society for Testing and Materials, EN= European Organization

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