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Effect of Temperature on the Synthesis of Methyl Ester via the Transesterification of Waste Black Seed Oil and Castor Seed Oil Admixture

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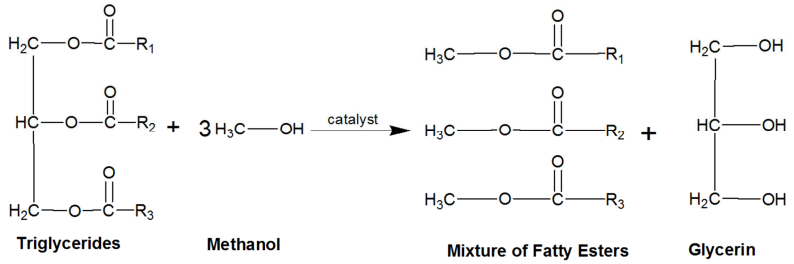
Abstract: There is an increasing attempt in biodiesel production (fatty acid methyl ester) because of the depleting fossil fuel resources as well as the similarity in properties when compared to those of diesel fuels. Engines set off on biodiesel have lower emissions of carbon monoxide, unburned hydrocarbons, and air toxics than engines runned on petroleum-based diesel fuel. We reported the optimization of Coconut oil methyl esters production via methyl ester from black oil/castor seed oils admixture under various operating conditions. The optimum yield, temperature, catalyst concentration and reaction time were found to be 93%, 60°C, 1.0% (wt of crude oil admixture) and 60 minutes respectively. Many fuel properties (viscosity, specific gravity and flash point) as measured according to standard methods, also found to conform to international standard.

Keywords: Black Seed and Castor Oil Admixture, Transesterification, Temperature

1. Introduction

Transesterification is the reaction between ester and alcohol via acid catalysts such as H_2SO_4, a base catalyst such as NaOH or KOH, or enzymes to produce another ester that is Fatty Acid Methyl Ester (FAME). A study on biodiesel production by ethanolysis of mixed castor oil and soybean oils using KOH as catalyst showed no appropriate substrate preference [1-7]. Currently, most of the biodiesel comes up from transesterification of edible resources such as animal fats, vegetable oils, and even waste cooking oil, under alkaline catalysis conditions. However, the high consumption of catalysts, the formation of soaps, and the low yields, make biodiesel currently more expensive than petroleum-derived fuel. The by-product of the transesterification reaction, glycerol, when purified could have other traditional application (pharmaceutical, cosmetics and food industries) [8-12].



R_1, R_2 and R_3 are long hydrocarbon chains, sometimes called fatty acid chains. They usually range from 12 to 20 carbons atoms in Vegetable oils.

Figure 1. *Transesterification reaction.*

Also, the glycerol obtained could have applications in the fields of animal feed, carbon feedstock in fermentations, polymers, surfactants, and lubricants. Biodiesel only refers to 100% pure fuel (B100) that meets the definition above and specific standards given by the American Society of Testing and Materials (ASTM) international (d 6751) [13].

2. Experimental Procedure

2.1. Transesterification of the Oil Admixture

To a stirred solution of methanol, 120mL was added KOH 0.18g until all the KOH had dissolved to allow the formation of methoxide. The resulting methoxide was then transferred to a stirred oil admixture 20mL and heated to 40°C. The reaction was refluxed for 60 minutes while the temperature was kept at 40°C. The resulting mixture was allowed to stand for 1-hr and transferred into separating funnel and allowed to stand for further 8-hrs by which time two layers emerged. The bottom layer (glycerol) was drained off, and the top layer (biodiesel) was collected in a beaker. This procedure was repeated at temperatures of 50°C, 60°C, 70°C, and 80°C.

2.2. Washing of Biodiesel

The product was separated from the glycerol and washed with warm (at about 45°C) deionized water. 30% by volume of the warm deionized water was added to the biodiesel and was left to stand for a further 8–hours by which time two layers emerged. The bottom layer was separated using a separatory funnel.

2.3. Neutralization of Biodiesel

3mL of 0.1M H_2SO_4 was added to the biodiesel. The resulting residue was then drained out; the pure biodiesel was then filtered and collected in an Erlenmeyer flask. The volume of biodiesel before and after purification was measured.

2.4. Determination of Specific Gravity

A 50ml density bottle was washed thoroughly with detergent and then rinsed with clean water and was allowed to stand in oven, dried and weighed (M_0). The container was filled with water and weighed (M_1), then died, filled with the biodiesel produced and considered (M_2), from the theory, and the density of a substance was equal to the mass of a content per unit volume of that substance.

2.5. Determination of pH

The pH of the Biodiesel produced was measured using a digital pH meter. Buffers were prepared by dissolving buffer powders of 4.0 and 7.0 in a 250mL volumetric flask, and were washed, dried and then filled with distilled water to the mark and stirred until the all the powder had dissolved. The electrode of the digital pH meter was then dipped to the buffers (4.0 and 7.0), washed with distilled water and then dropped into the biodiesel produced.

2.6. Calorific Value Determination

The Bomb calorimeter requires the burning a certain mass of oil sample in the presence of oxygen at a certain temperature and time and determination of the heat involved. This heat value is also called the Gross Calorific Value (GCV). 0.28g of oil sample was placed in an adequately washed cup. A thread was fixed at the suspender just a little above the cup holder ensuring the ends of the thread were immersed in the oil. About 3000 kpa of oxygen was pumped inside the tightly fixed vessel and then wait on the calorimeter to display 'insert'. The vessel was inserted, the lid was closed, and after about 15 minutes, the energy content was seen displayed on the screen in mega joules.

2.7. Infrared Study of Biodiesel Sample

IR spectra were recorded on a Perkin Elmer Paragon 1000 or a Perkin Elmer 881 spectrometer as a thin film between sodium chloride plates or as a KBr disk. All absorptions are reported in terms of frequency of absorption (cm^{-1}). To ascertain that the real reaction has taken place and that the biodiesel has been formed, the functional groups must correspond with the known functional groups of methyl esters. The initial background was collected removed from the peak display of the biodiesel. The peaks were analyzed.

3. Results and Discussion

In this work we reported our findings from the effect of temperature in the production of biodiesel from castor and black seed oils admixture; it varies from 40°C, 50°C, 60°C, 70°C, and 80°C. We kept herein the following parameters constant: - methanol / oil molar ratio (6:1), reaction time (60 minutes), catalyst type (KOH), catalyst amount (0.18g 1% w/w), and stirring rate of (300 rpm).

Table 1. *Results of the transesterification reaction(s) under different temperatures.*

Expt. No.	Temperature (°C)	Methanol / Oil molar ratio	Catalyst amount (wt %)	Volume of biodiesel before purification (mL)	Volume of biodiesel after purification (mL)	% Yield of Biodiesel (%)
1	40	6:1	1% w/w	120	117	84
2	50	6:1	1% w/w	122	120	86
3	60	6:1	1% w/w	132	130	93
4	70	6:1	1% w/w	137	128	91
5	80	6:1	1% w/w	110	99	71

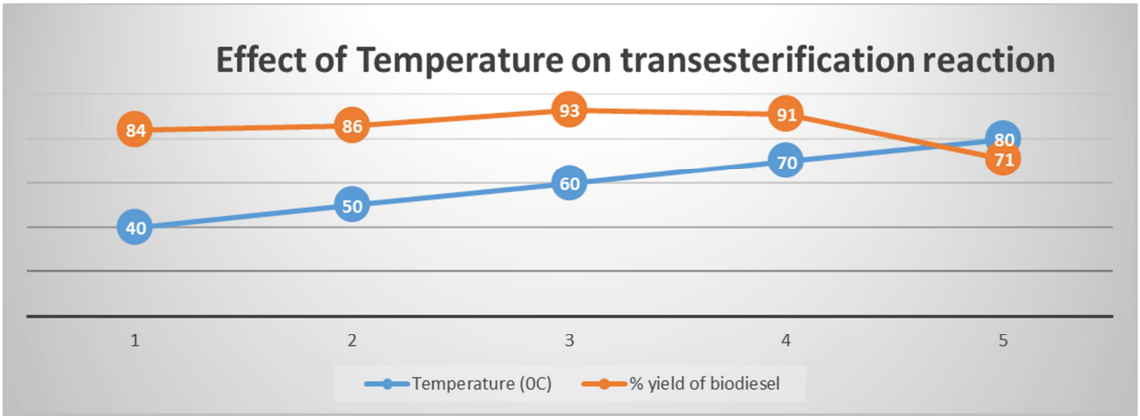


Figure 2. *Line Graph showing the effect of temperature of transesterification reaction(s).*

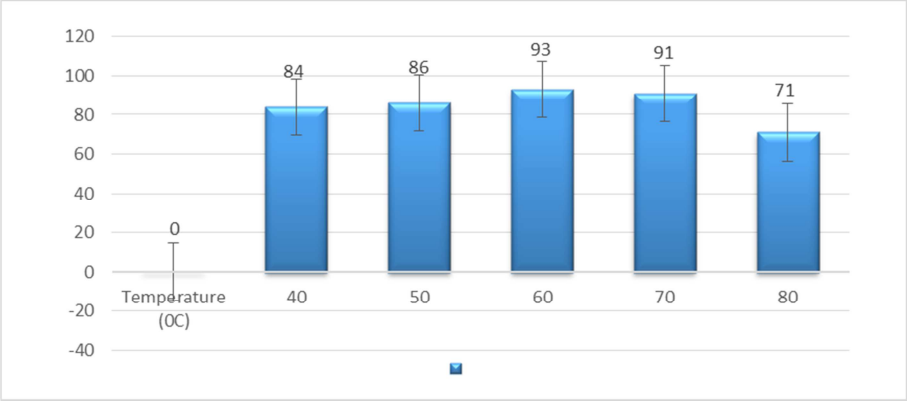


Figure 3. *Bar Graph showing the effect of temperature of transesterification reaction(s).*

From the graphs, the best yield of (93%) was at the temperature of 60°C. It was envisaged that at 60°C the molecules of the oil admixture had high kinetic energy and thus increase the rate of collision hence the overall process favored the formation of methyl ester (biodiesel). At 70°C, there was a slight decrease in the yield; this difference could be because the boiling point of methanol is below 70°C. Hence, the biodiesel produced at this temperature (70 ^{o}C) and above has lower viscosity, and the formation of glycerin is increasing due to the increase in temperature. When the temperature exceeds the boiling point of methanol, some of the methanol will start escaping which decreases the yield of biodiesel. At 40 and 50°C, the oil has almost the same

Table 2. *Results of comparison(s) with standard parameters.*

	PARAMETERS	BIODIESEL	ASTM D6751 STANDARD	EN 14214 STANDARD	UNITS
1	Specific gravity	0.90	0.87 – 0.90	-	g/cm^3
2	pH	7.3	-	-	-
3	Colour	Golden yellow	Golden yellow	Golden yellow	
4	Highest yield (60°C)	93	-	-	% v/v

Table 3. *Results for Calorific and Viscosity determination.*

Temperature (°C)	Calorific Value (MJ/kg)	Viscosity @ 40°C (mm^2/s)
45	33.10	5.02
50	33.45	5.04
60	35.35	5.36
70	36.03	-
75	36.78	-

The result from the IR spectrum was as envisaged and shows convincing evidence of the transesterification protocol. The IR of the pure castor displayed a peak at 1743.84c m^{-1} while the methyl ester of the biodiesel displayed at 1739.79 cm^{-1}. The viscosities at temperatures of 40°C, 50 and 60°C were also found to match the American Standard of Testing materials (ASTM). As it was also envisaged, our biodiesel was found to conform to standard parameters form the results of table 2 above also. Biodiesel is the most suitable alternative substitute of petroleum fuel due to their similarity. Biodiesel is a cleaner fuel, non-toxic, biodegradable, renewable, with less amount of sulfur, CO_2 and CO. it could come from waste vegetable oil, animal fats and fresh vegetable oil. Transesterification process is the most suitable way of converting these oils into biodiesel. It involves the reaction of lower alcohols such as methanol and ethanol with fatty acid triglycerides in the presence of a

catalyst such as NaOH or KOH to produce corresponding fatty acid methyl ester known as biodiesel and by-product is known as glycerol. The crude biodiesel formed was then washed with water and neutralized with 0.1M H_2SO_4. The biodiesel formed can be mixed with petroleum diesel in various ratios, this process is called blending. Black seed oil is pure oil extracted from Black seed plant (botanical name: Nigella sativa). The plant is an annual flowering plant originated from South East Asia, with finely divided linear leaves. The plant itself is non-toxic, its seeds, roots, and bark contains some active glycosides. It has a various medicinal application can cure almost every disease. Castor seed is an annual flowering plant that grows in winters, and it has beautifully mottled seeds which are poisonous. Black seed oil and Castor seed oil were mixed in the ratio of 1:1, the mixture of the oils were transesterified with methanol in the presence of KOH as a catalyst. The reaction time, methanol / Oil molar ration and catalyst amount were kept constant; the only factor that varies was temperature.

4. Conclusion

Herein we reported our experimental findings from varying temperature(s) in the production of biodiesel from the admixture. From the experiment carried out, the highest conversion was at a temperature of 60°C, methanol/oil molar ratio 6:1 and using 1wt% KOH catalyst. It shows that the gradual increase in temperature to the boiling point of methanol increases the production of biodiesel and at a temperature, but above 60°C there is a gradual decrease in the output. Also, the viscosity of the biodiesel produced is lower than that of the oils.

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