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Synthesis and Physicochemical Study of Methyl Ester from Black and Castor Seed Oil Admixture

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Abstract There is increasing effort in biodiesel production (fatty acid methyl ester) because of the depleting fossil fuel resources as well as similarity in properties when compared to those of diesel fuels. Diesel engines operated on biodiesel have lower emissions of carbon monoxide, unburned hydrocarbons and air toxics than those operated on petroleum-based diesel fuel. Herein we reported the optimization of Black and Castor oils methyl esters production via KOH catalyzed transesterification under various superintended conditions. The optimum yield, temperature, catalyst concentration and reaction time found to be 97%, 60°C, 1.0% (wt of crude black/castor oil) and 45 minutes respectively. A number of the fuel properties (viscosity, specific gravity and flash point) were measured according to standard methods, and were found to conform to international standards.

Keywords: *methyl ester, black seed oil, castor oil, physicochemical parameters, statistics*

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1. Introduction

Biodiesel is the mono-alkyl ester of vegetable or animal oils. [1,2,3] It has very similar physical properties to diesel fuel and even higher cetane number, which allows it to be used directly as substitute fuel for diesel engines without any modifications or as a blending agent for diesel fuel. Biodiesel is a cleaner burning fuel than diesel and could be a suitable replacement. Since it is produced from renewable and domestically grown feed-stocks, it can reduce the demand for petroleum based fuels and possibly lower the overall cost of diesel fuel. Biodiesel contains no or very little sulfur and thus offers promise to reduce particulate and toxic emissions. Example of such feed-stock is castor seed oil that is obtained from castor plant *Ricinus Communis* which belongs to the family of euphorbiaceae and black seed oil that is obtained from black seed plant Nigella sativa which belongs to the family of ranunculaceae. Biodiesel contains long-chain fatty acid esters, and is synthesized by transesterification reaction of vegetable oils with short chain alcohols. [4,5,6] It is compatible with conventional diesel fuel and already constitutes a commercial fuel in Europe. [7,8,9,10]

1.1. Experimental Methods

Apparatus and equipment in our experiments include; 400 mL beakers, pipette, pipette filler, 50 mL beakers, test tubes, measuring cylinders, magnetic stirrers, petri-dishes, distillation apparatus, separating funnel, clamp stand, spatula. Heating plates, regulated stirrer, thermometer, chemical balance, stop watch, and oven.

Viscosities were recorded as the product of the calibration constant of the viscometer and the time (seconds) it takes the fluid to travel between the calibrated marks. All results are in mm^2/s.

pH was recorded using Exstik pH meter, model PH100.

1.2. Preparation of the Biodiesel

Black seed castor oil admixture (20ml) 1:1 ratio was poured into a 250ml conical flask and was preheated to 60°C. To a stirred solution of methanol (120ml) 6:1 molar ratio was added 0.5 wt % KOH and the solution was stirred vigorously until the KOH had fully dissolved. The resulting solution was poured into the preheated oil admixture and the stirring continues for 60minutes by which time TLC analysis indicated the presence of a new spot. The mixture was transferred into a separating funnel and allowed to settle overnight. Two layers were observed, which were separable, the top layer (biodiesel) was collected, measured. The biodiesel was washed with warm distilled water (10ml) to remove the residual catalyst. The biodiesel recovered was neutralized with 0.1M H_2SO_4 (1ml). The black suspensions formed was allowed to settle and decanted gently into a clean beaker. The same experimental protocol was repeated for all the different catalyst concentrations (1.0%, 1.5%, 2.0% and 2.5%) keeping methanol/oil molar ratio at 6:1 and a constant stirring rate of 250rpm. The yield of the biodiesel for each of the experiments was calculated

1.3. Determination of the Specific Gravity

The temperature of the biodiesel recovered was found to be 34°C when the specific gravity was determined. The cleaned, dried and weighed density bottle (M_o) was filled with distilled water to the mark ($50cm^3$) and the weight of the bottle was taken as M_1. The same bottle was then emptied and dried. It was then filled with the recovered biodiesel to the mark ($50cm^3$) and the weight of the bottle was taken as M_2. The specific gravity of the biodiesel was calculated using

$$\frac{M_2 - M_0}{M_1 - M_0} = \text{Specific gravity.}$$

1.4. Determination of the pH

The biodiesel sample was agitated thoroughly. Then, the pH value was determined by using Exstik pH meter, model PH100. The Exstik pH meter has a 20ml flat surface pH electrode inform of a container into which the sample to be tested is poured in. It also has a simulated analog bargraph which indicates whether the tested sample is acidic (< 7.0), neutral (7.0) or basic (> 7.0). It measures pH from 0.0 to 14.0 pH with high resolution and accuracy of 0.01 pH. The biodiesel sample was poured into the 20ml flat surface pH electrode, and then the pH meter was inserted into it. This was observed carefully until a steady point was observed in the simulated analog bargraph. The pH value was noted and recorded.

1.5. Determination of Viscosity

Four Uppelhode viscometers of sized 3C were used in the viscosity measurement. 10 ml of each sample to be analyzed was added through the open end. The viscometer was maintained on its stand and then immersed in a viscometer bath that was already thermostat at 40°C and the viscometer was allowed to stay for 2 hours. By which time, the oil had assumed the temperature of its environment. A pipette filler is then placed at on open end and by closing the other one, oil is sucked up above the upper boundary line. Oil was allowed to flow under gravity to the upper mark where the stop watch was counting. The watch was stopped as the liquid reached the lower boundary and the viscosity was obtained in mm^2/s.

1.6. Calorific Value Determination

Oil sample (0.34g) was placed in a cup after which had been properly cleaned. A thread was fixed at the suspender just a little above the cup holder making sure the ends of the thread are 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, lid was closed and after about 15 minutes, the energy content was obtained in mega joules.

1.7. Results and Discussion

Statistical graphical techniques have been used to analyze the recorded data, namely scatter diagram and bar chart plots. [11,12].

Table 1. Transesterification by Varying the Catalyst Concentration

Experiment No:	Temperature (°C)	Castor seed oil-to-black seed oil molar ratio	Methanol-to-oil admixture molar ratio	KOH catalyst amount (% w/w)	Volume of biodiesel before purification (mL)	Volume of biodiesel after purification (mL)	% Yield of biodiesel (%)
1	60	1:1	6:1	0.5	118	107	76.4
2	60	1:1	6:1	1.0	137	136	97.1
3	60	1:1	6:1	1.5	125	118	84.3
4	60	1:1	6:1	2.0	120	114	70.1
5	60	1:1	6:1	2.5	117	77.5	55.4

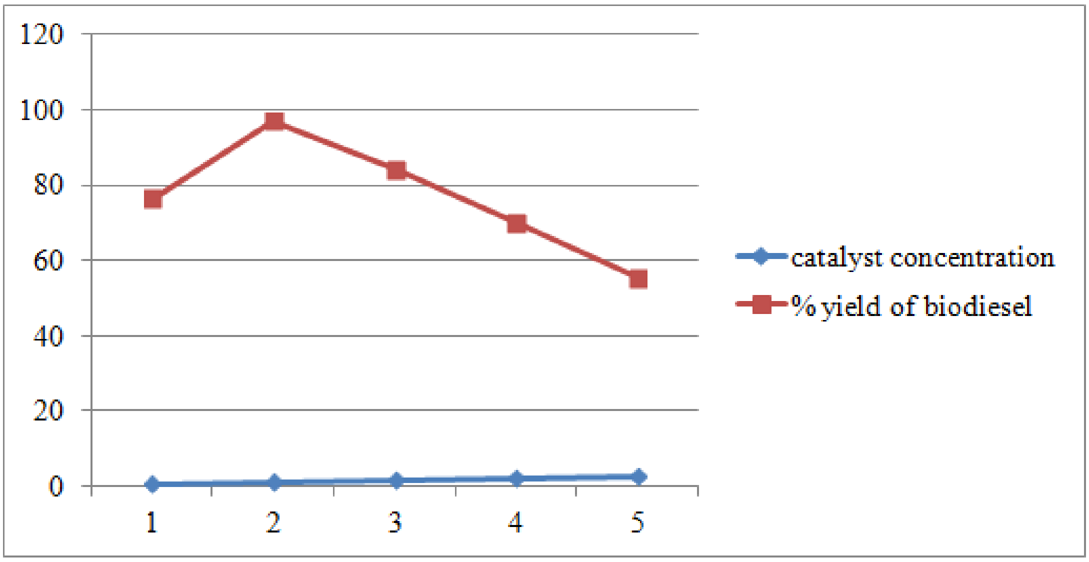

Figure 1. A Graph showing the Effects of Catalyst in the Production of Biodiesel from Castor Seed Oil and Black Seed Oil Admixture

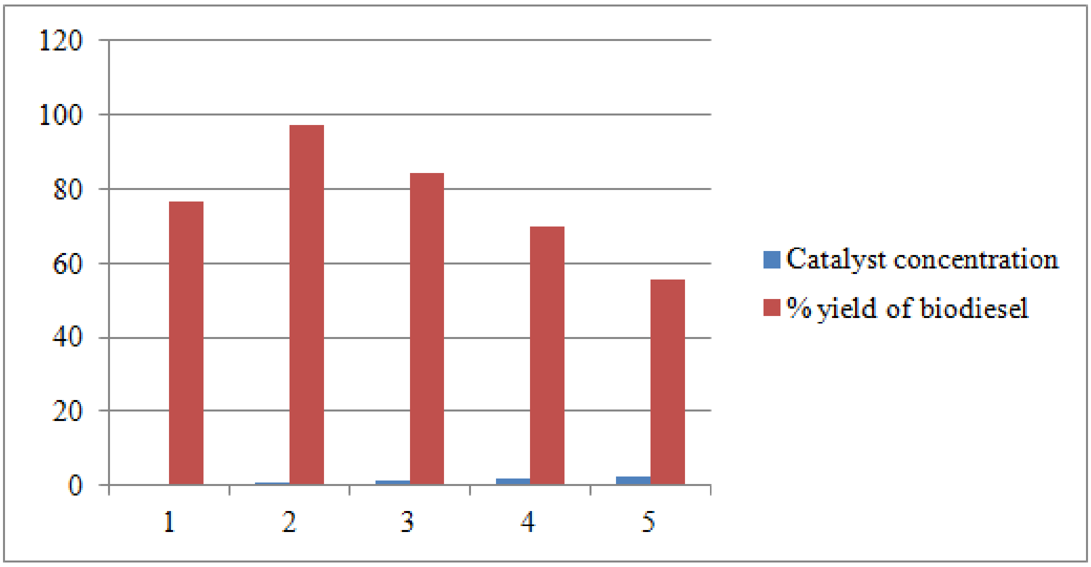

Figure 2. Bar chart showing the Effects of Catalyst in the Production of Biodiesel from Castor Seed Oil and Black Seed Oil Admixture

Table 2. Physicochemical Parameters of the Biodiesel in Comparison with ASTM 6751 and EN 14214 Standards

S/No	Parameters	Biodiesel	ASTM 6751 Standard	EN 14214 Standard	Units
1.	Colour	Golden yellow	Golden yellow	Golden yellow	-
2.	pH	7.42	-	-	-
3.	Specific gravity	0.88	0.86 - 0.90	-	g/cm^3

Table 3. Results for Calorific Value and Viscosity

Temperature (^0C)	Calorific Value (MJ/kg)	Viscosity @ 40°C (mm^2/s)
45	33.10	11.02
50	33.45	11.04
60	35.35	12.36
70	36.03	-
75	36.78	-

As it was hoped, the results in the Table 1 and Table 2 showed consistency with standards.

2. Conclusion

It was noted that biodiesel production could be affected by the amount of catalyst concentration used in the reactions. In this preliminary studies, we reported our findings from five different concentrations of KOH; of 0.5w/w %, 1.0w/w %, 1.5w/w %, 2.0w/w % and 2.5w/w %. All the reactions were carried out by keeping other parameters constant. Temperature (60^oC), methanol-to-oil molar ratio (6:1), castor seed oil-to-black seed oil molar ratio (1:1), reaction time (60mins), and stirring rate (250rpm) constant. Figure 1 and Figure 2 shows biodiesel yield using different concentration of KOH. The optimum value for the investigated KOH catalyst amount variation in the production of biodiesel was found to be 1.0w/w % KOH with the highest yield of 97.1%. As it was envisioned, the yield increases as the concentration of catalyst increased up to 1.0% but beyond this value, the yield starts to drop. It was envisaged that, increasing the catalyst concentration help to fasten the reaction and give better yield. However, every reaction has its optimum catalyst concentration value. Beyond this value, KOH will participate in reacts with triglyceride to form soap and water reducing the yield.

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