Experimental Investigation of Biodiesel Production From NaOH-Catalysed Palm Kernel Oil and Ethanol

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Abstract
This experimental work investigated how vegetable oils can be used to produce diesel fuels which can serve the same purpose as petroleum-based diesel. The vegetable oil and alcohol used in this work are palm kernel oil and ethanol respectively. The results showed that the vegetable oil itself cannot be used to serve the same purpose as petroleum-based diesel because of too high viscosity. This problem is solved by making the vegetable oil react with an alcohol (mostly methanol or ethanol), in the presence of a catalyst, to produce an ester of the alcohol used, in a process known as transesterification. The catalyst used is sodium hydroxide (NaOH). The process involves first dissolving the NaOH in the ethanol to produce sodium ethoxide which is then poured into the vegetable oil for reaction. The products of the reaction are biodiesel (ethyl ester) (desired) and glycerol (by-product). Certain reaction conditions (such as concentration) determine the quality and quantity of the products obtained at the end but only the effect of time is considered in this work. The vegetable oil had a viscosity 33.43mm^2/s but at the end a reduction of about 78% was recorded. Also, it was discovered that varying reaction time has not much effect on the yield.

***Key words**: Biodiesel, Palm kernel oil, Sodium hydroxide, ethanol*

1. Introduction

Biodiesel is a biofuel type produced from triglycerides of vegetable oil. Biodiesel, as an alternative fuel, has many merits. It is derived from a renewable and domestic resource, thereby relieving reliance on petroleum fuel imports. It is biodegradable and non-toxic. Compared to petroleum-based diesel, biodiesel has a more favourable combustion emission profile, such as low emissions of carbon monoxide, particulate matter and unburned hydrocarbons. Carbon dioxide produced by combustion of biodiesel can be recycled by photosynthesis, thereby minimizing the impact of biodiesel combustion on the greenhouse effect. Biodiesel has a relatively high flashpoint (150^{0}C) which make it less volatile and safer to transport or handle than petroleum diesel, and it provides lubricating properties that can reduce engine wear and extend engine life (Achanai et. Al., 2009). These merits make biodiesel a good alternative to petroleum-based fuel and have led t its use in many countries, especially in environmentally-sensitive areas.

The most common way to produce biodiesel is by transesterification, which refers to a catalyzed chemical reaction involving vegetable oil (palm kernel oil: PKO, in this project) and an alcohol to yield fatty acid alkyl esters (biodiesel) and glycerol. Triglycerides, as the main component of vegetable oil, consist of three long-chain fatty acids esterified to a glycerol backbone. When triglycerides react with an alcohol (e.g., methanol or ethanol), the three fatty acid chains are released from the glycerol skeleton and combine with the alcohol to yield fatty acid alkyl esters (e.g., fatty acid methyl esters: FAME or fatty acid ethyl esters: FAEE). Glycerol is produced as a by-product. Methanol is the most commonly used alcohol because of its low cost, but methanol is a highly toxic alcohol. Hence, ethanol of choice in the processes developed in this study.

2. Methodology

2.1 Materials

By stoichiometry, 1 mole of PKO is required to react with 3 moles of ethanol to produce 3 moles of the biodiesel and 1 mole of glycerol. Values for different parameters used are results of preliminary experiments and previous works (Lalita et al., 2004; Alamu et al., 2008). Palm kernel oil was purchased at Igbunu Market, along Sapele-Benin Road, Delta State, Nigeria. 200g PKO was used for the transesterification process. The ethanol used (99% pure) is an analytical grade with boiling point of 78^0C; while the NaOH used was also an analytical grade production of Aldrich Chemicals, England. The container used as the reactor is a PET bottle (75 cl). Any capacity of container may be used, as long as it gives enough space for proper agitation. Other materials used include; measuring cylinder; weighing device; spatula; funnel; filter material, thermometer, retort stand, separator, viscometer and heater plus conical flask, beaker, masking tape and PET bottles, for storage. (Tarbuka 2011 p. 25)

2.2 Experimental Design

The experiment was designed to study and investigate the optimal conditions for the production of biodiesel. There are some key variables that affect the yield of methyl ester in this process. They include temperature, reaction time, catalyst concentration, mass ratio of alkanol to oil and agitation speed. This work only looks into the effect of reaction time on the yield of biodiesel (methyl ester). This is studied at low level (30 minutes) and high level (60 minutes). The temperature chosen for the process is room temperature (30^0C).Tthe catalyst concentration chosen was 0.5% NaOH by weight of the palm kernel oil. And the ethanol to oil ratio used was 1/5.

2.2.1 Experimental Procedure

i. 40.0g of ethanol was measured and poured into a PET bottle after which 1.0g of NaOH was carefully added.
ii. The container was agitated thoroughly for about 10 minutes for complete dissolution of NaOH in the ethanol to form sodium ethoxide.
iii. 200.0g of PKO was measured out and poured into a PET bottle.
iv. The PKO, before now, was filtered and heated near boiling to evaporate any water present.
v. Sodium ethoxide from the plastic container was carefully poured into the PKO, the PET lid was secured tightly and agitation was done none stop by hand. The agitation in the container was maintained for 30 minutes in the first case and for 60 minutes in the second case.
vi. The mixture was poured from the PET bottle into a separator held standing by a retort

for setting and the lid was screwed on tightly. Phase separation can be observed within 10 minutes and can be complete within 24 hours after stiring has stopped. Complete setting can take as long as 72 hours, or even longer.

2.3 Recovery of glycerol

A successful reaction produces two liquid phases: ester and crude glycerol. The mixture, after settling, leaves the glycerol at the bottom and methyl ester (BD) is left on top. Crude glycerol, the heavier liquid will collect at the bottom after several hours of settling. The glycerol at the base was collected into a PET bottle by opening the tap.

2.4 Washing

The biodiesel remained and was washed with warm distilled water. The water was poured into the separator making provision of some space for agitation. The agitation is done for about 5 minutes. Water and biodiesel are immiscible and biodiesel floats on water because of its density. The separation content is allowed to settle for 6 – 12 hours and the water is drained out. This is done severally until the water appears clean after settling. The clarity of the water shows that the biodiesel is clear too. Washing the methyl ester is a two-step process which is carried out with extreme care. After settling the aqueous solution is drained. The washing can be done in two ways: either by washing the biodiesel-glycerol mixture after separate or by washing the biodiesel along after the glycerol is drained out. The important parameter in the process can be calculated from the following equations;

PKO-biodiesel yield (%) = [quantity of PKO-BD(g)/quantity of PKO used(g)]x100 (1)

PKO-BD conc. (%) = [quantity of PKO-BD(g)/total production sum(g)] x 100 (2)

Production yield(%)=[total amount of products(g)/total amount of reactants(g)]x100 (3)

3 Results and Discussion

3.1 Product yields

The transesterification process yielded (227 g) PKO biodiesel and (8.4g) glycerol, while (5.51g) of the total reacting masses could not be accounted for. For the second case the process yielded (22.42) PKO biodiesel and (11.12) glycerol with (7.46) loss recorded. These losses have been attributed to some un-reacted alcohol, residual catalyst and emulsion removed during the washing stage of the production process (Alamu et al., 2008 and 2009). Detailed results for each of the experimental runs are as presented in table 1 and the characterization is done in table 2.

Table 1: Reaction Conditions, Production Yield, Methyl Ester Concentration and Methyl Ester Yield.

Run NO	Ethanol:Oil Mass Ratio	NaOH (wt%)	Temperature (^oC)	Reaction Time (mins)	Production Yield (wt%)	Ethyl-Ester Conc. (wt%)	Ethyl-ester Yield (wt%)	Glycerol Yield (wt%)	Loss recorded (wt%)
1	1.5	1.0	30	30	97.71	96.43	94.23	3.57	2.23
2	1.5	1.0	30	60	96.8	95.25	92.23	4.76	3.10

3.2 Fuel Characterization/properties of biodiesel

Samples of the biodiesel were collected and analysis was made for specific gravity, density and viscosity. Specific gravity was measured by comparing the weight of a certain volume of the biodiesel compared to the same volume of water at the same temperature and pressure. Density was determine by measuring a specific volume of and multiplying its inverse by the mass (i.e. density = mass/volume). The viscosity measurement was done using a Viscometer. This was done in a beaker which is wide enough to contain the appropriate *spindle*-a part of the viscometer that rotates when the viscometer is put ON. The spindle used is dependent on how viscous the liquid is. And the one used for this work is *size 03".* The fuel properties of biodiesel obtained in this work are summarized in Table 1. The properties of the PKO-biodesel produced in this work is compared with other biodiesels and standards and showed in table 3. It can be seen that most of the properties of PKO Biodiesel are a little higher than the range of fuel properties as described in the latest Thai and American standard for biodiesel. (Chongkhonget et al., 2007).

Table 2: Characterization results for PKO-biodiesel

Properties/parameters	PKO-biodesel
Viscosity (@ 40°C), (mm²/s) Specific gravity @ 15°C)	7.42 1.176

Table 3: Comparing PKO Bio-disel produced with other diesels and standards (Chongkhonget et al,. 2007)

Properties/parameters	PKO-biodiesel	Petrolem Diesel	EN14214 European BD Standard	Rapeseed Biodiesel (ethylester)	Canola Biodiesel (ethylester)
Viscosity (@ 40°C), (mm²/s)	7.42	2.847	3.50 -	6.170	4.892
Specific gravity @ 15⁰C)	1.176	0.853	5.00	0.876	0.878
Poured point (°C)	_	-16	0.86-0.90	-2	-1
Cloud Point (°C)	_	-12	-	-10	-6
Flash point (°C)		74	>120	124	177

3.3 Effect of reaction time

From analyses of results obtained from the two runs (table 1), the production yield is nearly independent of reaction time. Increasing the reaction time actually brought about decrease in value of ethyl ester concentration, ethyl ester yield and the production yield. But the change affected the methyl ester concentration more. That is to say prolonging the reaction time did not efficiently change conversion of FFA. However, this result is an indication that the reaction should not be for too long a period (Tarbuka, 2011 p. 32 - 33).

3.4 Effect of temperature

The reaction temperature influences the reaction rate and yield of ester (table1). A lot of reports have been made on how the production yield is affected when the temperature is raised. In one of these many reports it was observed that increasing the reaction temperature, especially to supercritical temperatures, had a favourable influence on ester conversion (Demirbas, 2002). The productions yield decreases with increased temperature because the higher solubility of reactant at higher temperature reduced the separation between methyl ester and glycerol phase. The temperature had only a slight effect on the biodiesel (methyl ester) concentration. But then room temperature is considered to be the optimum temperature (Lalita, 2004). In our investigation we also observed that in an attempt to study how temperature affects the yield a very viscous dark-brown substance was produced. This was obtained by first heating the PKO to about 60^{o}C before adding the sodium ethoxide solution. The agitation was for 30 minutes. When this was observed, we thought it was as a result of the fact that the temperature was not uniform for the whole length of time. So, we kept the temperature constant using a water bath. With all other conditions the same as in the first case but the same highly viscous substance was produced. This substance gave out only small amount of liquid (about 3% of the reactant volume), which was too small for analysis of its composition. we were quick to name it a lubricant but there was no test done to confirm what it is and so it is undergoing further investigation.

3.5 Effect of the presence of water in the reactants and catalyst (NaOH/Iye)

In one of our reaction settings, the reaction after being allowed to stay for days did not give any phase of moisture or water in the NaOH. Truly, the container of the NaOH we used had some cracks which would have allowed some absorption of moisture from the atmosphere. This could also be as a result of water in the oil and or ethanol. But we were sure we had heated the oil to evaporate any water present therein. Hence, in the production of biodiesel, the alcohol used has to be very close to 100% alcohol. If exposed to the atmosphere, the Iye and alcohol will absorb water, which will block the biodiesel conversion.

Conclusion

Our investigation showed that diesel fuel can be produced from palm kernel vegetable oil through the process of transesterification. The results also showed that there is no much effect when time is varied. However, keeping the reaction for too long would most likely cause reduction in the biodiesel yield. This decrease though, would most likely increase the quality of the fuel obtained. The study also revealed that the reaction temperature influences the reaction rate and yield of ester. And that ethanol is a preferred alcohol in transesterification reaction compared to methanol (which is very much widely used) because it is derived from agricultural products and a renewable and biologically less objectionable in the environment. However, methanol is preferred because of its low cost and its physical and chemical advantages (polar and shortest chain alcohol). However, for beginners and for home production, ethanol is preferred. This is because methanol is quite toxic and does some danger to the skin. The study also confirmed Tarbuka's report (2011, p. 34) that if the reactants of the transesterrification reaction is exposed to the atmosphere, the Iye and alcohol will absorb water, which may block the biodiesel conversion.

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