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Original Research Article

Preparation and Characterization of Biodiesel (Fatty Acid Methyl Ester) From a Commercial (Soya Bean) Oil

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ABSTRACT

In recent times, the world has been confronted with an energy crisis due to depletion of resources and increased environmental problems. This situation has led to the search for an alternative fuel, which should be not only sustainable, but also environmentally friendly. Biodiesel is a clean burning renewable fuel made from vegetable oils, animal fats and recycled cooking oils and greases. It is an amber yellow liquid. The manufacturing process for biodiesel combines oils and fats with methanol and a catalyst to produce Fatty Acid Methyl Esters (FAME) which is commonly referred to as biodiesel. Vegetable oils such as rapeseed, canola, soybean and palm oils are the most common raw materials for biodiesel production. The aim of this work was to prepare and characterize biodiesel from soya-bean (a commercial vegetable) oil. To achieve this aim, the following objectives were focused on: To prepare biodiesel from a refined commercial oil, To separate the by-product (glycerol) from the biodiesel in a biodiesel-glycerol mixture, To purify the biodiesel with water washing method, To characterize the refined biodiesel by determining the density, viscosity, flash and point value of the biodiesel. This project data shown above shows that biodiesel can be a viable replacement for the now dwindling fossil fuel diesel. It is even observed that due to its low emissions; its renewability, and its ease of preparation; it will be a better fuel in combating environmental pollution.

Keywords: Biodiesel, Fossil Fuel, Fatty acids.

INTRODUCTION

In recent times, the world has been confronted with an energy crisis due to depletion of resources and increased environmental problems. This situation has led to the search for an alternative fuel, which should be not only sustainable, but also environmentally friendly. [1] Biodiesel is a clean burning renewable fuel made from vegetable oils, animal fats and recycled cooking oils and greases. It is an amber yellow liquid. The manufacturing process for biodiesel combines oils and fats with methanol and a catalyst to produce Fatty Acid Methyl Esters (FAME) which is commonly referred to as biodiesel. Vegetable oils such as rapeseed, canola, soybean and palm oils are the most common raw materials for biodiesel production. About forty years ago, in 1973, the organization of Petroleum Exporting Countries (OPEC) voted to raise the posted price of oil by 70%. Soon afterwards, several Arab Oil producers decided to impose an embargo on oil sales to the United States of America to punish her for supporting Israel in the Israeli-Arab War. In-fact, the organization of Arab Petroleum

Exporting Countries (OAPEC) consisting of Asian members of OPEC plus Egypt and Syria announced, as a result of the war, that they would no longer ship oil to the nations that had supported Israel in its conflict with Syria and Egypt. The countries include Japan, Netherlands, United States and some of their allies in Western Europe. [2] Thus began what is now known as the 1973 world oil crisis-panic by western nations led oil prices to be greatly inflated. By 1974 January, world crude oil prices were four times higher than they were in October when the crisis began. Although the fighting ended in late October, OPEC, and more significantly OAPEC over the coming years continued to use the oil weapon throughout the next decades. For example, during a revolution against Iran, petroleum exports were diminished to virtually negligible level causing crude oil prices to be raised exorbitantly. Further, Iraq's invasion of Kuwait in the 1990's also inflated the prices of petroleum produce. By putting an end to decades of cheap energy, the 1973-74 oil crises and its aftermath exacerbated the economic difficulties facing many industrialized nation and forced developing countries to finance their energy imports through borrowing. In addition, there is the environmental impact of petroleum and other fossil fuels. Gas flaring in developing countries like Nigeria results in problems such as thermal pollution, noise pollution, land pollution not to mention the enormous release of the greenhouse gases and other environmental hazards. [3,4] Automobile exhaust has been reported as one of the leading anthropogenic sources of greenhouse gases (GHG) which has resulted in a wide variety of problems such as rising sea levels, ozone layer depletion, global warming and a significant increase in health problems resulting from inhalable and respirable particle. [5] These disturbing trends reinforce and accentuate the world's dependence on fossil fuel especially from the Middle East and argue for the development of new energy sources not tied to any geographical region of the earth. These facts also converge in a search for renewable energy sources with a reduced propensity for pollution, thus making developments sustainable. Rudolf Diesel proposed in the year 1900 at an exhibition in Paris the first diesel engine made to run on peanut oil. That idea though brilliantly plausible was never fully utilized. And even when the talk of biodiesel has been raised; the biofuels, in most cases-ethanol have been with low energy content (Volumetric Energy Density) compared with conventional hydrocarbon petroleum fuels and neutral gas. A desirable and efficient fuel is expected to have a high volumetric Energy Density and should blend easily with existing fuels and yet require little or no engine modification. The aim of this work was to prepare and characterize biodiesel from soya-bean (a commercial vegetable) oil. To achieve this aim, the following objectives were focused on: To prepare biodiesel from a refined commercial oil, To separate the by-product (glycerol) from the biodiesel in a biodiesel-glycerol mixture, To purify the biodiesel with water washing method, To characterize the refined biodiesel by determining the density, viscosity, flash and point value of the biodiesel. Biodiesel is a renewable source of energy which serves as an alternative to petroleum diesel used to power diesel engines. This study therefore serves as a means of reducing the greenhouse gases (GHG) emitted from petroleum diesel engines. This will consequently reduce the global warming which has caused adverse effect on climate change in the world.

MATERIALS AND METHODS

Since the free fatty acid value of the oil was less than 0.5%, there was no need to pretreat the oil by esterification reaction. However, the oil was heated to about $60^{O}C$ for 25 minutes to dry up the any available water that could be present in the oil. This helps to avoid the hydrolysis of the triglyceride if allowed to stay in the oil. This reaction would have resulted in the formation of soaps and the reduction of

yield. Heating, however, evaporate a large volume of the water. [6] Approximately 1% of catalyst based on the mass of oil was added to a certain amount of methanol to give a 6:1 methanol to oil molar ratio. In this study, 0.55g of Sodium hydroxide (NaOH) pellets was weighed and added to 31.25g of methanol. The mixture was shaken vigorously to form sodium methoxide ($NaOCH_3$ as the resulting solution. [6] Precautions were taken as the chemicals involved here are toxic. Hence, thick rubber gloves were worn and a facemask was used to prevent inhalation of either the methanol or methoxide fumes which are very toxic. Sodium hydroxide does not readily dissolve in methanol. The mixture was thus agitated continuously. The beaker was observed to be warm which signify the reaction being exothermic. The prepared sodium methoxide solution was added to about 125ml of oil to prepare the biodiesel fuel. It should be noted that the oil was heated to $60^{O}C$ -$65^{O}C$ before the sodium methoxide was added. The mixture was agitated continually for about 90minutes in covered glass bottle as the reactor placed in a temperature controlled magnetic stirrer/ hot plate. [7] The diagram of the biodiesel production process i illustrated in appendix A. After the reaction time has been completed, the mixture was transferred to a separating funnel which separated into two layers. This heavier glycerol settled at the bottom while the less dense biodiesel was at the top. The setting began almost immediately and lasted for about 4 hours. The glycerol was then removed through the tap and the biodiesel was also taken into another beaker. The diagram for the separation process is shown in appendix A. After separation of the biodiesel, it was washed with hot water to remove unreacted methanol and NaOH. The biodiesel was washed by adding water to the drained biodiesel and vigorously shaking the mixture. The mixture was then transferred into the separation funnel where it is allowed to settle. It is observed that the bottom aqueous layer became milky signifying that some NaOH and methanol as well as very little unreacted FFA have been washed off. After the completion of purification process the biodiesel layer may contain some amount of water and methanol. This was removed before the analyses were carried out on the biodiesel. Because methanol reduces the flash point of fuel and it have corrosive nature to fuel hoses. Hence the biodiesel was heated in hot plate with stirrer at 100°C for 15 to 30 min to remove the water and methanol content in the product (biodiesel). Finally the dried biodiesel was in a glass container for further analysis.

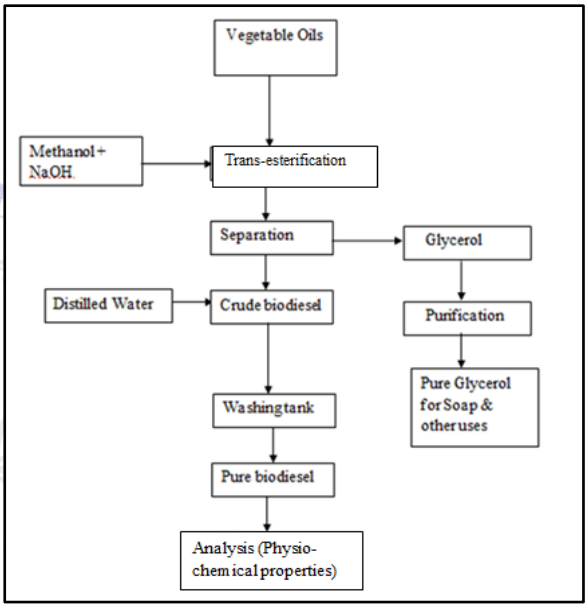

Figure 3.1: Flow chart for the preparation of Biodiesel

Density bottle was used to determine the density of the oil. A clean dried density bottle of 25ml capacity was weighed (W_0), it was then filled with the oil, stopper was inserted and reweighed to give (W_1). The oil was then substituted with water after washing and drying the bottle and weighed to give (W_2). The expression for specific gravity was calculated using the following formula:

$$Density = \frac{Mass\ of\ substance}{Mass\ of\ equal\ volume\ water} = \frac{W_1 - W_0}{W_2 - W_0}$$

[8]

The viscosity of the biodiesel was carried out using Brookfield Digital Viscometer. About 20ml of biodiesel placed into a sample container. A standard spindle

attached to the viscosity was immersed into the sample, the viscometer was switched on and the spindle was rotated at 260rev/min. The viscometer was read on the displayed screen. An open cup Kochler flash point tester equipped with a motor driven stirrer and gas cylinder was used for this study. Biodiesel was poured into the cup up to the marked position. The cup was covered with the lid, and immersed into an in-built heater. The heater and the stirrer were switched on. The two nozzle connected to the gas cylinder and placed above the sample cup were lit with a lighter. At an interval of 5 minutes, a knob controlling the opening of the lid of the sample cup and one of the nozzles was used to carry out flash point testing. Flash point is reached when a light sound explosion is observed, and at this point the biodiesel will ignite. The temperature at which this phenomenon occurred was taken as the flash point of the biodiesel. The cloud point was determined by visually inspecting for a haze in the normally clear fuel. The biodiesel was placed in a test tube and the test tube was place in an ice bath with a thermometer dipped right in the middle of the biodiesel. The biodiesel was observed and the temperature at which the clear amber yellow developed its first haziness was recorded as the cloud point. The apparatus used is the same as that for cloud point. The procedure is that given by the process standard ASTMD97 at every $3^{O}C$ of cooling the sample was inspected and when no movement was detected after 5minutes, the test was stopped where no movement was observed and this point was recorded as the pour point. A measure of 5g of biodiesel was weighed into a conical flask. A quantity of 50ml of solution made up of equal volume of 95% ethanol and diethyl ether was added and then gently mixed to dissolve the oil. The mixture was heated to enhance homogenization and was then titrated against 0.1N KOH in methanol, using l ml of phenolphthalein indicator, until a slight pink colour persisted for 15 seconds. The acid value was computed using the equation below

$$Saponification\ Value = \frac{T \times N \times 56.1}{m}$$

Where T is the titre value in mL;
N is normality of methanolic KOH
m is mass of sample in gm (Aransiola, 2013).

3.3.7 %FFA COMPOSITION

The %FFA composition was obtained using the equation given by [9] as shown below:

$$\%FFA = \frac{AN}{1.99}$$

Where AN is the acid number. [9]

1g of the sample was weighed into a conical flask; 25ml of 0.1N ethanoic potassium hydroxide was then added. The content which was constantly stirred was allowed to boil gently for 60min. A reflux condenser was placed on the flask containing the mixture and a few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5 M HCl to the end point until the pink colour of the inculcator just disappeared. The same procedure was used for other samples and a blank.

The expression for saponification value (*sv)* is given by

$$SV = \frac{N \times 56.1 \times (V_0 - V_1)}{M}$$

Where:
Vo- the volume of the solution used for the blank test,
Vi- the volume of the solution used for determination,
N- actual normality of HCL used,
M- mass of the sample. [10]

A certain amount of biodiesel (0.3g) was taken in a 200mL conical flask with stopper. 10mL of cyclohexane was added and shaken to dissolution. Wijs reagent (0.lmol/L iodine monochloride acetic acid solution) was added and stirred, the flask was stoppered and the solution left for about 1 hour in a dark cupboard. Then 20mL of 10% potassium iodine solution and l00mL distilled water were added. The mixture was then titrated with titrate with 0.1M sodium thiosulfate solution, and iodine value

obtained. A blank titration was carried out as well.

The Iodine value of the biodiesel was obtained using the following equation:

$$IV\ (g/100g) = \frac{(BV - TV) - 1.003 \times CF}{m}$$

where

BV = blank volume

TV =Titration volume

CF = Concentration conversion coefficient (1.269)

m = Sample weight (g)

3.3.10. CETANE INDEX

This was obtained using the correlation given by Krisnangkura (1986) using the saponification and iodine values

$$\boldsymbol{CI = 46.3 + (5458/SV) - 0.225 \times IV}$$ [11]

RESULTS

The biodiesel produced and the by-product (Glycerol) are shown in Figure 4.1

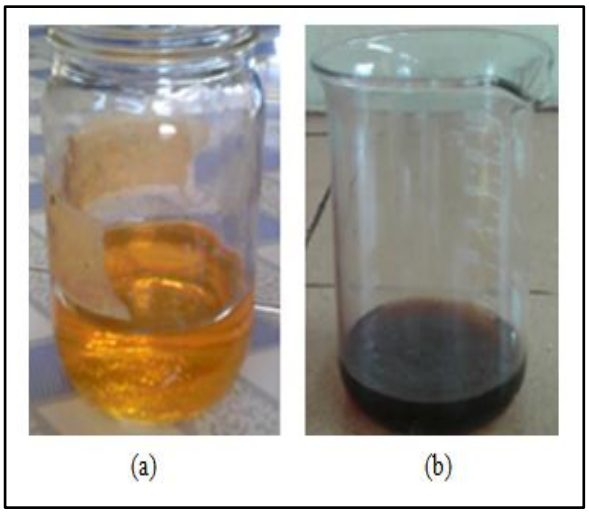

Figure 4.1: (a) Refined Biodiesel (b) Glycerol

The values obtained from analysis are presented in Table 4.1

Table 4.1: PHYSIO-CHEMICAL PROPERTIES OF BIODIESEL

PROPERTY	VALUES OBTAINED	STANDARD VALUES	
		ASTM D6751	EN 14214
Density at 25°C (kg m^{-3})	877		860-900
Specific Gravity	0.877		
Pour point. (°C)	13.2	-5 to 15	
Cloud point. (°C)	10.4	-3 to 10	
Saponification value (mgKOH/g)	142.5		
Iodine Value (g/100g)	118		120(max)
Cetane index	58.05		
Cetane number.	56	48-65	51(min)
Acid value (mgKOH/g)	0.3	0.8(max)	0.5 (max)
FFA (%)	0.15		
Viscosity (mm^2/s)	5.30	1.9 -6.0	3.5 -5.0
Flash point (^oC)	132	130 min.	101min.

DISCUSSION

Viscosity is one of the most important properties to be analyzed in a refined Biodiesel. Viscosity is a measure of resistance of the fuel to gradual deformation by shear stress. It is usually measured as Kinematic viscosity which is the ratio of absolute viscosity of a fluid to the fluid density. According to literature, viscosity of a fuel is related to its lubricity. [12] It was stated that low viscosity fuels are unlikely to provide sufficient and appropriate lubrication to the injection pumps which often lead to seepage and increase in wear. On the other hand, high viscosity in fuel results to incomplete combustion and increased exhaust emissions, choking of the injections thereby forming larger droplets on injector, ring carbonization and accumulation of the fuel in the engine. [12,13]

The viscosity obtained in this study was 5.30mm^2/s as shown in Table 3.1. The result fell within the ASTM 6751 standard range though it was slightly higher than the EN14214 upper range. However, following the ASTM range of viscosity, this biodiesel produced from commercial soya-bean oil can be accepted as an alternative to petroleum diesel. Flash point is the minimum temperature at which a fuel must

be heated for it to ignite air -vapor mixture. The U.S.Department of Transportation specified 90^{o}C as the flash point for non-hazardous fuel. [12] The flash point determined for this soya-bean oil based biodiesel was 132^{O}C. From the result, it shows that the high flash point is an indication that essentially the entire methanol used in the production of the biodiesel was wash-out during purification. It has been ascertained from other researchers that small quantity of methanol can reduce the flash point of the biodiesel thereby affecting engine parts negatively. The relatively high pour and cloud points could suggest that the starting oil contains more of saturated or monounsaturated fatty chains. This suggestion however may have its own flaws and may not be absolutely correct. The cetane number obtained was well above the ASTM and EN standards. This suggests that the fuel will have very good fuel ignition properties. It also means that the fuel will offer easier starting and quieter operation in automobile engines. The Acid Value of the produced biodiesel is comfortably lower than the maximum standard values. This could be because it was freshly prepared as acid numbers are used to determine the degradation level of fuels. The very low acid number also means that it has very good storage stability and this was proven as the fuel was still good even after over six months of storage. Several researchers have found that temperature increase clearly influences the reaction rate and biodiesel yielding a positive manner. This was also observed as the biodiesel did not react at first due to a process error of not heating before reaction; but on heating the mixture the reaction was kick-started. The iodine value for the biodiesel product is high though exceeding the ASTM standard. This shows that the volume of unsaturated fatty chains is within standards and can be used. The 0.15FFA recorded shows that the product will have a lesser tendency to cause rupture and degradation in hoses and will have very good cleaning properties. It is generally observed that most of the properties fall within standard or vary slightly to either the increase or the decrease. The density, pour point, iodine value, and cetane number all fall within standard, while the cetane index higher heating value and cloud point fell slightly out of proportion. A likely reason for the difference in cloud point could be the presence of unsaturation or impurities in the start oil which were not removed in or reacted all through the reaction. Specific gravity of the fuel is very important in diesel engine because fuel injection system operates on a volume metering basis. The values of specific gravity obtained for this biodiesel was 0.877. The obtained density value of 877kg/m^3 lies within the standard range of 860-900kg/m^3.

CONCLUSION

This project data shown above shows that biodiesel can be a viable replacement for the now dwindling fossil fuel diesel. It is even observed that due to its low emissions; its renewability, and its ease of preparation; it will be a better fuel in combating environmental pollution.

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