

**PRODUCTION OF BIODIESEL FROM USED VEGETABLE OIL**

**BY**

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**2006/24030EA**

**BEING A FINAL YEAR PROJECT REPORT SUBMITTED IN  
PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE  
AWARD OF BACHELOR OF ENGINEERING (B. ENG.) DEGREE  
IN AGRICULTURAL AND BIORESOURCES ENGINEERING,  
FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA, NIGER  
STATE**

**FEBRUARY, 2012.**



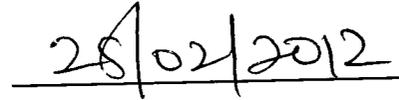
## CERTIFICATION

This is to certify that the project entitled "Production of Biodiesel from Used Vegetable Oil" by Godwin, James Akor meets the regulations governing the award of the degree of Bachelor of Engineering (B. ENG.) of the Federal University of Technology, Minna, and it is approved for its contribution to scientific knowledge and literary presentation.



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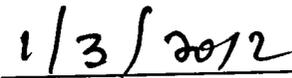


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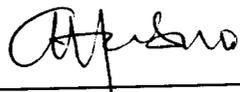


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## DEDICATION

This project work is dedicated to God Almighty for his protection, guidance and wisdom granted to me. This project is also dedicated to my parents Mr Godwin Akor and Mrs Mary Godwin for their tremendous support. And to my brother Joseph Godwin, my sister Justina Godwin, my sister Helen Ada Godwin for their extreme support towards my work and Prof. Itodo for his support.

## ACKNOWLEDGEMENTS

In the name of God Almighty, the creator of heaven and earth be all the glory forever. Thanks Be to God Almighty for giving me the strength, wisdom, knowledge and guidance through this piece of work successfully.

My sincere appreciation goes to Engr. (Prof).B.A. Alabadan who professionally supervised this project work. I humbly appreciate the efforts of hard-working Lecturers of the Agricultural and Bioresources Engineering Department especially, Engr. Dr. P.A. Idah(HOD), Engr. Prof. B.A. Alabadan, Engr. Dr. O. Chukwu, Engr. Dr. A.A. Balami, Engr. Dr. Z.D. Osunde, Dr. N.A Egharevba, Dr. I.E. Ahaneku, Engr. M.E. Sadeeq, Engr. (Mrs) H.I. Mustapha, Engr. (Mrs) Orhevba, Engr. P. Adeoye, Engr. Adamu Halilu, Engr. A.O Fabunmi, Engr. S.E. Adebayo, Mal Zaggi, Alh. M.S. Suleiman, Mal. Ishyaku, Engr. Mal Dada, and all the laboratory technicians for their contribution towards the successful completion of my programme.

My profound gratitude goes out to my parents Mr Godwin Akor and Mrs Mary Godwin and my brother Joseph Godwin, my sisters Justina Godwin, and Helen Ada Godwin for their moral and financial support during my study at the University. I am also grateful to all my friends and well wishers for their encouragement.

Finally, this work will not be complete without giving appreciation to my lovely friends: Solomon Paul, Clement Yari, Stephen Popoola, Samuel Augustine, Ayangbile Olufemi, Olakunle Lemah, Emmanuel Abi, Diana Kadiri, Francis S. Onoja, Michael K. Opeyemi, Onu B. Ogbonnaya and all those not mentioned but highly recognized may God Almighty grant you all the very best life has to offer.

## ABSTRACT

Biodiesel is a clean burning diesel fuel processed from natural and renewable biological sources such as waste cooking oil, rape seed, jatropha seed, animals fats and refined bleached deodorized palm oil. The objective of this work was to produce biodiesel from used vegetable oil and characterization of the biodiesel produced. The scope of this work entails the produce of biodiesel, characterization of the diesel produced and compare the properties with that of diesel produced from fossil fuel. The type of method used to produce biodiesel is called transesterification. The transesterification of used cooking oil with short-chain alcohols, in the presence of base catalyst sodium hydroxide (NaOH) and methanol as solvent, by means of single step batch transesterification process in order to obtain biodiesel fuel was studied using a reaction ratio of 6:1 for alcohol to oil ratio. The oil was heated in a water bath. The process variables that were investigated are catalyst concentration and reaction time. The variable that is fixed throughout the whole experiment is quantity of used vegetable oil used, mixing degree of mechanical stirrer at 1300 rpm and the alcohol to oil ratio. This paper also studied the combustion characteristic which is the carbon monoxide emission of UCO to be compared with conventional diesel, and determined the optimal transesterification reaction conditions that produce the maximum methyl ester content or purity and biodiesel yield. The oil was split into three samples and the results obtained for all the samples are; sample 1: biodiesel yield of 58ml and glycerine yield of 19ml. Sample 2: biodiesel yield of 79ml and glycerine yield of 19ml. Sample 3: biodiesel yield of 70ml and glycerine yield of 20ml. The reaction times for the three samples are 60, 90 and 120 minutes respectively. The best result for highest yield and highest purity is at 90 minutes reaction time and using 1.5g catalyst concentration and this is because, after characterisation of the biodiesel produced, sample 2 was found to have the highest cetane rating closer to the ASTM standard which implies that sample 2 will be a more efficient fuel than the other two samples, guarantee smooth running of the engine as well as burn cleaner.

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## LIST OF ABBREVIATIONS

°C - Degree Celsius

FFA - Free fatty acid

h - Hour

Min - minutes

g - Gram

kg - kilogram

L - Litre

ml – Millilitre

ppm - Parts per million

NaOH - Sodium Hydroxide

UCO - Used Cooking Oil

UVO - Used Vegetable Oil

FAME - Fatty acid methyl ester

NO - Nitrogen Oxide

CO<sub>2</sub> - Carbon Dioxide

HC – Hydrocarbon

ND – Not Determined

## CHAPTER ONE

### 1.0 INTRODUCTION

#### 1.1 Background of Study

The world consumption of fuels is undoubtedly unstable causing world economic crisis; the worst compared to other economic recession that took place at different era. This factor has urged all nations especially the government and the academics to find another alternatives to replace the usage of petroleum. Therefore there is a rising demand to globally provide renewable energy by means of a sustainable and ethical approach (Meher *et al.*, 2006). Sustainable development is a concept that has become significant and increases the awareness of its necessity. There are many alternatives nowadays. There are three generations of biofuels. First-generation biofuels are biofuels made from sugar, starch, vegetable oil, or animal fats using conventional technology. These feedstocks could instead enter the animal or human food chain, and as the global population has raised their use in producing biofuels has been criticised for diverting food away from the human food chain, leading to food shortages and price rises. Second generation biofuel production processes are in development. These allow biofuel to be derived from any source of biomass, not just from food crops such as corn and soy beans but also from waste cooking oil (Felizardo *et al.*, 2006). Algae fuel, also called oilgae or third generation biofuel, is a biofuel from algae.

Biodiesel is a fuel made from natural, renewable sources, such as new and used vegetable oils and animal fats, for use in a diesel engine. Biodiesel has physical properties very similar to petroleum-derived diesel fuel, but its emission properties are superior (Demirbas, 2008). Using biodiesel in a usual diesel engine substantially reduces emissions of unburned hydrocarbons, carbon monoxide, sulphates, polycyclic aromatic

hydrocarbons, nitrated polycyclic aromatic hydrocarbons, and particulate matter (Lakshmi *et al.*, 2010). Diesel blends containing up to 20% biodiesel called B20 can be used in nearly all diesels powered equipment, and higher-level blends and pure biodiesel, B100 can be used in many engines with little or no modification. Lower-level blends are compatible with most storage and distribution equipment, but special handling is required for higher-level blends. One of the most used renewable energy is biodiesel which is the most common biofuel in Europe. It is produced from oils or fats using transesterification and is a liquid similar in composition to mineral diesel. Its chemical name is fatty acid methyl ester (FAME). In my study, oil is mixed with Sodium hydroxide NaOH as catalyst and methanol and the chemical reaction produces biodiesel (FAME) and glycerol. 1 part glycerol is produced for every 10 parts biodiesel. Biodiesel can be used in any diesel engine when mixed with mineral diesel. In some countries manufacturers cover their diesel engines under warranty for 100% biodiesel use. Many people have run their vehicles on biodiesel without problems. However, the majority of vehicle manufacturers limit their recommendations to 15% biodiesel blended with mineral diesel. In many European countries, a 5% biodiesel blend, B5 is widely used and is available at thousands of gas stations. Biodiesel can be made from waste and virgin vegetable and animal oil and fats (lipids) (Ramadhas *et al.*, 2009). Virgin vegetable oils can be used in modified diesel engines. In fact the diesel engine was originally designed to run on vegetable oil rather than fossil fuel. There are also studies and efforts to commercialize biodiesel from algae.

## **1.2 Statement of the Problem**

Because of the incessant oil spillage experienced during drilling of the crude from beneath the earth surface as well as the gas flaring which has threatened the health of human beings living in the areas where these crude is being refined through global warming and climate change which could also affect the socioeconomic activities of people living there especially in the area of agriculture, portable drinking water as well as aquatic life in that area, it has become necessary to sought for alternative means to source for energy.

## **1.3 Objective of the Study**

Aim of this project is the production of high quality methyl ester and environmental friendly biodiesel, from Used Cooking Oil by using single step batch transesterification process with good reaction ratio and catalyst concentration.

## **1.4 Justification of the Study**

Petroleum price is undoubtedly agreed to be very unstable. At some point the price roars to maximum price at \$145 per barrel. Then it decrease till \$40 per barrel. This sort of trend has affected the world economic growth. Each human being in this mother earth can feel the effect of economic crisis that is mainly caused by unstable price of petroleum. Thus, by using biodiesel as alternative, the problem could be tackle. Malaysia is famously known as one of the net producer of palm oil. It has the potential to lead the way in biofuel production looking at its vast production of palm oil. By using Refined-Bleached-Deodorized (RBD) palm oil, Malaysian would have a consistent supply and provision to replace the usage of diesel petroleum. Besides, palm oil is also one of the most highly efficient feedstock for biodiesel compared to other vegetable oils

(Catharina and Keshun, 1999). The raw material that will be used in this research is used cooking oil. It seems as a practical way for used cooking oil (UCO) to be converted as biodiesel as it will give a comparable and cheap price than subsidized diesel. Furthermore, the usage of used cooking oil will promote into a cleaner environment because the water and land will be less polluted by used cooking oil. But the main problem of using used cooking oil is the existence of free fatty acid as the by product in transesterification process (Cvengros and Cvengrosova, 2004). Thus the aim of this project is to produce biodiesel as diesel substitute at a feasible way. The research of which types of feedstock is the best have to be conduct. The result of the study will provide a clear picture as to the yield gotten from used vegetable oil as a feedstock and will be sustainable in alleviating the energy crisis. Single step transesterification process will be used in synthesizing raw material to methyl ester. Single step transesterification process provides less time in reaction, lower temperature and pressure, and hence will result in less cost of production (Ramadhas *et al.*, 2009). The high content of free fatty acid in Used cooking oil need to be synthesize by using homogenous catalyst, Sodium Hydroxide (NaOH). Even though the use of 4 homogenous catalysts resulted in higher formation of soap, homogenous catalyst provides shorter reaction time compare to heterogeneous. In the transesterification process, methanol will be use as alcohol solvent because of its price is cheaper among other alcohol solvent.

### **1.5 Scope of the Study**

In order to achieve the objective, this project is focused on how to produce biodiesel from used cooking oil (UCO) by using single step transesterification process and to compare the combustion characteristic of biodiesel produced from used cooking oil (UCO) with standard diesel from fossil fuel. In the research that will be conducted, we

will fix the volume at 100ml. While the catalyst concentration used is between 5, 15, and 20g and the time that be conducted is from 60, 90, and 120 minutes.

## CHAPTER TWO

### 2.0 LITERATURE REVIEW

#### 2.1 Biodiesel

Biodiesel is defined as mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats which conform to American Society for Testing and Materials, ASTM D6751 specifications for use in diesel engines. It is a clean burning alternative fuel, produced from domestic, renewable resources. Biodiesel contains no petroleum, but it can be blended at any level with petroleum diesel to create a biodiesel blend. Biodiesel is simple to use, biodegradable, nontoxic, and essentially free of sulphur and aromatics. Biodiesel is made through a chemical process called transesterification whereby the glycerine is separated from the fat or vegetable oil (Fukuda *et al.*, 2001). The process leaves behind two products: methyl esters or biodiesel and glycerine. Biodiesel is much cleaner than fossil-fuel diesel. It can be used in any diesel engine with no need for modifications. In fact diesel engines run better and last longer with biodiesel. And it can easily be made from common waste product, used cooking oil. Biodiesel is better for the environment because it is made from renewable resources and has lower emissions compared to petroleum diesel (Ramadhas *et al.*, 2005). It is less toxic than table salt and biodegrades as fast as sugar.

##### 2.1.1 Biodiesel emission and engine relation

A major benefit of biodiesel is lower emissions. The use of biodiesel reduces emission of carbon monoxide and other hydrocarbons by 20 to 40%. Biodiesel burns up to 75% cleaner than conventional petroleum diesel fuel. Biodiesel reduces unburned hydrocarbons, carbon monoxide and particulate matter in exhaust fumes, as well as cancer-causing PAH and nitrated PAH compounds. Sulphur dioxide emissions are

eliminated as biodiesel contains no sulphur. Biodiesel is plant-based and using it adds no extra CO<sub>2</sub> to the atmosphere. The ozone-forming (smog) potential of biodiesel emissions is nearly 50% less than petro-diesel emissions. Nitrogen oxide (NO) emissions may increase or decrease but can be reduced to well below petrol-diesel fuel levels. Biodiesel exhaust is not offensive and does not cause eye irritation (International journal of energy and environment, 2010).

Biodiesel is environmentally friendly, it is renewable, "more biodegradable than sugar and less toxic than table salt" (US National Biodiesel Board, based on US Environmental Protection Agency studies). Biodiesel is a much better lubricant than petro-diesel and extends engine life. Even a small amount of biodiesel means cleaner emissions and better engine lubrication. 1% biodiesel added to petro-diesel will increase lubricity by 65%. Biodiesel can be mixed with petrol-diesel in any proportion, with no need for a mixing additive. Biodiesel has a higher cetane number than petroleum diesel because of its oxygen content. The higher the cetane number the more efficient the fuel, the engine starts more easily, runs better and burns cleaner. With slight variations depending on the vehicle, performance and fuel economy with biodiesel is the same as with petrol-diesel.

### **2.1.2 Biodiesel Properties**

Table 2.1 shows a comparison of chemical properties and fatty acid composition (%) of UCO. Fatty acid is a carboxylic acid often with a long unbranched aliphatic tail (chain). These long-chain fatty acids generally have an even number of carbon atoms; unbranched chains predominate over branched chains. They may be saturated (e.g. palmitic (hexadecanoic) acid and stearic (octadecanoic) acid) or unsaturated, with one double bond (e.g. oleic (cis-octodec-9-enoic) acid) or two or more double bonds, in

which case they are called polyunsaturated fatty acids (e.g. linoleic acid and linolenic acid). As for specific gravity, UCO has higher specific gravity because it has much residue such as water as water has mixed inside the UCO as a result of condensation of cooking process. So, the density of UCO is high.

**Table 2.1:** Chemical properties and fatty acid composition (%) of UCO

Properrty	UVO
Palmitic acid C16:0	16
Stearic acid C18:0	5.21
Oleic acid C18:1	34.28
Linoleic acid C18:2	40.76
Specific gravity	0.92

Table 2.2 shows major properties comparing between premium diesel and B100. The density for biodiesel is 0.92 g/ml for UCO. Flash point is the temperature at which the vapour above a volatile liquid forms a combustible mixture with air. At the flash point the application of a naked flame gives a momentary flash rather than sustained combustion, for which the temperature is too low. So from the table shown, biodiesel has higher flash point and it makes as an advantage because it would not easily vaporize to air compared to premium diesel. This will save the cost of fuel for transport especially if the ambient temperature is high. As for cetane number, a higher cetane number indicates greater fuel efficiency (Ya-fen Lin *et al.*, 2006). In this case, B100 scores higher cetane number and it shows that the performance rating of a diesel fuel, corresponding to the percentage of cetane in a cetane-methylnaphthalene mixture with the same ignition performance.

**Table 2.2:** Major properties of premium diesel and biodiesel used in this study

	Premium		Test method
	Diesel	B100	
Density at 20 °C (g/mL)	0.826	0.86	ASTM D 1298
Kinematic viscosity at 40 °C (cSt)	2.73	4.49	ASTM D 445
Cetane index	46.2	48.05	EN ISO 4264
Flash point (°C)	89	122	ASTM D 93
Water and sediment (vol.%)	<1	0.22	ASTM D 2709
Gross Heating Value (cal/g)	11411.4	9850.6	ASTM D 240

From Table 2.3, some significant differences were found between Reference (REF) and biodiesel fuel. These differences are within the daily differences typically found in samples taken from urban collectors, and, in any case, they did not lead to any significant difference in ultimate composition and to very small differences in the unsaturation level (iodine number). The reference fuel (REF) is a typical low sulphur diesel fuel similar to those available in Spanish petrol stations in winter. It was supplied by Repsol YPF, and fulfils the current European norm EN-590. (Magín Lapuerta *et al.*, 2008)

**Table 2.3: Specifications of biodiesel fuels****Properties REF UCO**

<b>Properties</b>	<b>REF</b>	<b>UCO</b>
Density at 15 °C (kg/m <sup>3</sup> )	834	887
Kinematic viscosity at 40 °C (cSt)	2.72	5.16
Gross heating value (MJ/kg)	45.54	39.26
Lower heating value (MJ/kg) <sup>a</sup>	42.49	36.59
Acid number (mg KOH/g)	0.10	0.55
% C (wt.)	86.13	76.95 <sup>b</sup>
% H (wt.)	13.87	12.14 <sup>b</sup>
% O (wt.)	0	10.91 <sup>b</sup>
Sulphur content (ppm wt.)	34	0 <sup>b</sup>
Water content (ppm wt.)	57	466
IBP (°C)	172	320
T10 (°C)	211	325
T50 (°C)	270	333
T90 (°C)	340	356
Molecular weight	211.7 <sup>c</sup>	293.2 <sup>b</sup>
Stoichiometric fuel/air ratio	1/14.67	1/12.55
CFPP (°C)	-18	-6
Iodine number <sup>b</sup>	-	97.46
Renewable fraction	0	90.11 <sup>d</sup>

a- Calculated from composition and gross heating value.

b- Calculated from speciation.

c- Calculated by Aspen-Advisor software.

d- Calculated from used cooking oil composition.

From Table 2.4, it shows that biodiesel sample meet EN14214 standards for density, kinematic viscosity, copper corrosion, acid value, cetane number, free glycerol and total glycerol. There was slight difference in density and viscosity compared to diesel but completely acceptable. The higher flash-point of biodiesel sample is beneficial in safety aspect, and the low sulphur content is the reason for the extremely low SO (sulphur oxide) emission associated with its use as fuel. The cetane number is higher than diesel resulting in a smoother running of the engine with less noise. Biodiesel sample is an oxygenated fuel naturally with oxygen content about 10% which contributes to the favourable emission, but leads to a little bit low caloric value compared with petro-diesel. Biodiesel nearly meets all the properties of normal diesel fuel, according to diesel and EN14214 standards, which indicates that Biodiesel derived from UCO has adequate values compared to diesel fuel (Xiangmei Meng *et al.*, 2008).

**Table 2.4:** The properties of biodiesel sample compared to diesel fuel and EN14214 biodiesel standard

Parameter	Samples	Diesel fuel	EN14214
Density (15 °C, kg/m <sup>3</sup> )	890	NA	860–900
Flash point (°C)	171	> 65	> 101
Kinematic viscosity (40 °C, mm <sup>2</sup> /s)	4.23	3.0–8.0	3.5–5.0
Sulfur content (%wt.)	0.007	< 0.05	< 0.01
10% Conradson carbon residue	0.2	0.3	0.3
Copper strip corrosion (3 h, 50 °C)	1a	class1	class1
Water content (mg/Kg)	150	NA	< 500
Cold filter plugging point (°C)	1	≤ 4	NA
Free glycerol (%)	0.008	NA	0.02
Total glycerol (%)	0.21	NA	0.25
Acid value (mg KOH/g)	0.48	< 0.1	≤ 0.5
Cetane number	54.5	> 49	≥ 51
Caloric value (MJ/kg)	32.9	41.8	NA

### 2.1.3 Green House Gases and Global Warming Impacts and Benefits

Human-caused global warming is one of the greatest and most urgent challenges facing humanity and life on earth today. The main culprit is the enormous amount of the potent greenhouse gas carbon dioxide (CO<sub>2</sub>) released into the atmosphere by the burning of fossil fuels (petroleum, coal, natural gas). Burning fossil fuels releases more than 6 billion tons of CO<sub>2</sub> per year, twice as much as the biosphere can absorb (International Energy Agency, 2006). The excess CO<sub>2</sub> is clogging the atmosphere, with the result that

less solar heat is reflected away, more heat reaches the earth's surface, and global temperatures rise. Using vegetable oils or animal fats as fuel for motor vehicles is in effect running them on solar energy.

All biofuels depend on the conversion of sunlight to energy (carbohydrates) that takes place in the green leaves of plants. Plants use water and CO<sub>2</sub> from the atmosphere as the raw materials for making carbohydrates. Burning plant (or animal) products in an engine releases the CO<sub>2</sub> back into the atmosphere, to be taken up again by other plants. The CO<sub>2</sub> is recycled. Natural mechanisms work to hold the amount of CO<sub>2</sub> in the atmosphere at a stable level, maintaining a balance between the CO<sub>2</sub> removed from the atmosphere to be "fixed" into growing organic matter and the CO<sub>2</sub> released back into the atmosphere when the organic matter burns or dies and decays. The net amount of CO<sub>2</sub> in the atmosphere stays the same. Activities that don't disrupt this balance are described as carbon-neutral. In fact, there's no actual reduction in the amount of CO<sub>2</sub> produced when biodiesel is burned instead of petrol-diesel. The same amount of CO<sub>2</sub> will come out of the exhaust pipe with either fuel. But the CO<sub>2</sub> released by burning biodiesel is part of the current natural cycle; it does not raise the level of CO<sub>2</sub> in the atmosphere and does not act as a greenhouse gas. Biodiesel is carbon-neutral and does not increase global warming. Petrol-diesel is not carbon-neutral. Burning petrol-diesel unleashes CO<sub>2</sub> that has been trapped beneath the earth for millions of years, upsetting the natural balance and raising the level of CO<sub>2</sub> in the atmosphere, causing global temperatures to rise. Fossil-fuel CO<sub>2</sub> is an active greenhouse gas. In practice however, not all biodiesel is carbon-neutral. It depends how it's produced. "Life-cycle" studies of the whole production process from sowing the seed to filling the fuel tank can show a different picture.

Industrialised agricultural production of oil crops like soy or rapeseed depends heavily on fossil-fuel inputs which must be included in the equation, and biodiesels made from these Crops are not carbon-neutral. But petrol-diesel is a lot worse. Organic farms don't use fossil-Fuel based chemical fertilizers and their fossil-fuel inputs are much lower, shrinking to zero when they produce their own fuel and energy on-farm, as a growing number of organic farmers are doing. Biodiesel made from used vegetable oil (UVO) should also qualify. Most UVO ends up in the sewers and landfills where it does no good and doesn't offset any fossil-fuel use. Converting it to biodiesel is a much better option, a social service. Reduce, reuse, and recycle. The US produces an estimated 4.5 billion gallons a year of used cooking oil, and most of it goes to waste. By comparison, US commercial production of biodiesel in 2006 was only 250 million gallons, most of it made from new soy oil, very little from used oil. 13 According to a model developed by the USA's Argonne National Laboratory (ANL), neat (100%) biodiesel from soybeans can cut global warming pollution by more than half relative to conventional petroleum based diesel. The emissions benefits are higher for canola oil. In the future, non-conventional sources like algae may have the potential to provide dramatic (90%) reductions in global warming pollution. However, significant technological hurdles remain before algae and other advanced feed stocks can be processed into biodiesel for commercial purposes. It is important to note that the ANL model of global warming impacts does not take into account changes in land use. When soybeans are used for fuel, they are taken out of the market for food. This increases prices and stimulates demand that farmers around the world respond to by bringing more land into cultivation. With soybean production increasing in the Amazon, it is possible that the lifecycle global warming pollution of soybean biodiesel is even higher than petroleum diesel,

once indirect land use changes are considered. When biodiesel is made from recycled food oil or other waste products these land use considerations do not apply.

Also advanced technologies including biomass gasification may allow the use of other waste streams to be converted to synthetic diesel fuels, expanding the pool of potentially low carbon diesel. In addition to land use, there is also some controversy over the emissions impact of fertilizer use and other land use practices, such as tillage practices. As a result, the estimated emissions from biodiesel can be expected to change as our understanding of the lifecycle improves. Large scale production of biodiesel would require more virgin plant oils or other waste stream sources to meet larger demands. However, such large-volume biodiesel use could raise concerns about genetically modified crops, pesticide use, and land-use impacts common to ethanol and all other plant-based fuels. Crops for biodiesel must be grown in a manner that supports wildlife habitat, minimizes soil erosion, avoids competition for food crops, and does not rely on the use of harsh chemicals and fertilizers (Kaieda *et al.*, 2000).

## **CHAPTER THREE**

### **3.0 MATERIALS AND METHOD**

#### **3.1 Materials**

##### **3.1.1 Raw Materials**

- Waste vegetable oil

##### **3.1.2 Chemical Reagents**

- Sodium hydroxide
- Methanol

##### **3.1.3 Equipments**

- Pipette
- Pycnometer bottle
- Electric weighing balance
- Beaker
- Measuring cylinder
- Magnetic hot plate
- Water bath

Using the above materials/apparatus, three samples of the used vegetable oil were put in three different beakers to produce the biodiesel varying temperature, time and quantity of catalyst used using the procedure below.

## **3.2 Methodology**

### **3.2.1 General Process**

This section provides detailed description of the starting material/source, chemicals used, equipment and experimental procedures for various processes and characterization for this study. The major starting material used in this work is used groundnut oil which was sourced from Grand local bean-cake sellers at bosso market along bosso road minna, Nigeria. Chemicals used like methanol, manufactured by Aldrich Chemicals Co. Ltd, England has a boiling point of 65<sup>0</sup>C and with 99.5% purity, the sodium hydroxide (catalyst) used was also manufactured by Aldrich Chemicals Co. Ltd, England.

### **3.2.2 Experimental Procedure**

The used vegetable oil was bought from local bean-cake sellers at Bosso market along Bosso road Minna, Niger state. The following physical and chemical properties of the oil were determined:

### 3.2.2.1 Characterization of used vegetable oil

#### ❖ Specific gravity

This experiment was carried out in accordance with description reported by Onwuka in 2005. A 50ml pycnometer is washed thoroughly with detergent, water and petroleum ether, it is then dried and weighed. The bottle is filled with water and weighed, the bottle is then dried and filled with groundnut oil sample and weighed, from theory, the density of a substance is equal to mass of a substance per unit volume of that substance. The temperature at which the density is being measured is known, as density changes with changes in temperature (Ibitoye, 2005).

$$\text{Specific gravity} = \frac{\text{weight of groundnut oil (ml)}}{\text{weight of water (ml)}}$$

$$\text{Density} = \frac{\text{weight of oil (g)}}{\text{volume of oil (ml)}}$$

#### ❖ Refractory Index:

A few drops of the oil will be placed on the face of the prism of the refractometer and allowed to gently spread close and it will be tightened for sometimes so as to allow for the oil and the prism to attain a steady temperature. The refractive index will be read from the demarcation line after adjusting to where it coincides with diagonal crossing (Ibitoye, 2005).

❖ **Acid Value (AN)/Free Fatty Acid (FFA) (ASTM D 664):**

The free fatty acid is estimated by titrating 10g of waste vegetable oil against sodium hydroxide (NaOH) using phenolphthalein as indicator (Ibitoye, 2005). The acid value is mg NaOH required for neutralizing the free fatty acids present in one gram of the sample. It is expressed as oleic acid (octadec-9-enoic acid) equivalent. The acid value in mg/NaOH/g oil is gotten by using the equation given in the appendix A.

$$AN = \frac{56.1 \times V \times N}{\text{weight of oil (g)}}$$

Where;

V= volume of standard alkali used

N= normality of standard alkali used

W<sub>oil</sub>= weight of oil used

$$FFA = \frac{AN}{2}$$

#### ❖ Iodine Value (IV):

This is the measure of the degree of unsaturation in a given volume of oil. 0.25g of the oil will be weighed into a glass stopper bottle and 10cm<sup>3</sup> will be added and it will be shaken so as to dissolve the oil. 25cm<sup>3</sup> of iodine monobromide will be added from a burette to stopper and then kept in the dark for exactly 30mins. After 30mins during which iodine absorption must have taken place, 15cm<sup>3</sup> and 1000cm<sup>3</sup> of distilled water will be added. The mixture will be titrated against standard sodium sulphate solution until the colour becomes faint yellow. At this point, 1cm<sup>3</sup> of starch solution will be added and titration will be completed and simultaneously, a blank titration will be carried out using the same reagents and distilled water in exactly the calculation but with the oil (Ibitoye, 2005).

$$\text{Iodine Value} = \frac{0.1269 \times (B-A) \times N \times 100}{W_{OIL}}$$

Where;

B= volume of standard Na<sub>2</sub>CO<sub>3</sub> used in blank titration.

A= volume of standard Na<sub>2</sub>CO<sub>3</sub> used in titration with oil.

N= normality of standard Na<sub>2</sub>CO<sub>3</sub>.

W<sub>oil</sub>= weight of oil used.

### ❖ Saponification value

Saponification value is the amount (mg) of alkali required to neutralise definite quantity (1g) of an oil. A known quantity of oil is refluxed with an excess amount of alcoholic KOH. After saponification, the remaining KOH is estimated by titrating it against a standard acid. The saponification value of the groundnut oil was determined in accordance to the experimental procedure reported by Onwuka in 2005. The alcoholic KOH is freshly prepared by dissolving KOH pellet in ethanol. 2g of groundnut oil is measured and poured into a conical flask. 25ml of the alcoholic KOH is added to it, a blank is used. The sample is well covered and placed in a steam water bath for 30minutes shaking it periodically, 1ml of phenolphthalein is added to the mixture and titrated against 0.5M HCl to get the end point (Ibitoye, 2005).

$$\text{Saponification value} = \frac{(B-A) \times 28.05}{\text{weight of groundnut sample (g)}}$$

#### ❖ Peroxide value

The peroxide value of the groundnut oil was determined in accordance to the experimental procedure reported by Onwuka (2005). 1g of groundnut is weighed into a clean drying boiling tube, 1g of powdered potassium iodide and 20ml of solvent mixture (2volume of glacial acetic acid + 1volume of chloroform) is added, the tube is placed in boiling water so that the liquid boils within 30seconds and is also allowed to boil vigorously for not more than 30seconds. The content is quickly poured into a flask containing 20ml of potassium iodide solution; the tube is washed out with 25ml of distilled water and is titrated with 0.002M sodium thiosulphate solution using starch as indicator (Ibitoye, 2005). A blank is also carried out at the same time.

$$\text{Peroxide value} = \frac{T \times M \times 1000}{\text{weight of sample (g)}}$$

#### 3.2.2.2 Filtration

The used vegetable oil collected from the bean-cake fryers was first properly filtered using a filter paper and transferred into a clean beaker so as to remove completely every food particle present in the oil and obtain a very clean, clear and particle free oil which will guarantee a good and acceptable result.

#### 3.2.2.3 Transesterification Process

##### ❖ Sample 1

1.0g of NaOH crystals was put into a beaker and 100ml of methanol was used to dissolve the NaOH crystals by heating it on a magnetic hot plate with a magnetic stirrer inside the mixture to obtain a meth oxide solution. 100ml of the used vegetable oil was measured and poured into a separate beaker and was purified by heated in a water bath

at a temperature of 45 for 60 minutes. 25ml of the meth oxide solution was then mixed with the 100ml of purified oil and the mixture was heated for 30 minutes on the magnetic hot plate with a magnetic stirrer inside the mixture. (Schuchardt *et al.*, 1998)

#### ❖ Sample 2

1.5g of NaoH crystals was put into a beaker and 100ml of methanol was used to dissolve the NaoH crystals by heating it on a magnetic hot plate with a magnetic stirrer inside the mixture to obtain a meth oxide solution. 100ml of the used vegetable oil was measured and poured into a separate beaker and was purified by heated in a water bath at a temperature of 65 for 90 minutes remove the free fatty acid content which may alter the result. 25ml of the meth oxide solution was then mixed with the 100ml of purified oil and the mixture was heated for 30 minutes on the magnetic hot plate with a magnetic stirrer inside the mixture (Schuchardt *et al.*, 1998).

#### ❖ Sample 3

2.0g of NaoH crystals was put into a beaker and 100ml of methanol was used to dissolve the NaoH crystals by heating it on a magnetic hot plate with a magnetic stirrer inside the mixture to obtain a meth oxide solution. 100ml of the used vegetable oil was measured and poured into a separate beaker and was purified by heated in a water bath at a temperature of 70 for 120 minutes remove the free fatty acid content which may alter the result. 25ml of the meth oxide solution was then mixed with the 100ml of purified oil and the mixture was heated for 30 minutes on the magnetic hot plate with a magnetic stirrer inside the mixture (Schuchardt *et al.*, 1998).

### 3.2.2.4 Settling

#### ❖ Sample 1

The result was kept in desiccators for 48 hours to allow for separation of the biodiesel from glycerine after which the biodiesel gotten was separated by a decantation process and was washed with water and heated to obtain pure biodiesel.

#### ❖ Sample 2

The result was kept in desiccators for 48 hours to allow for separation of the biodiesel from glycerine after which the biodiesel gotten was separated by a decantation process and was washed with water and heated to obtain pure biodiesel.

#### ❖ Sample 3

The result was kept in desiccators for 48 hours to allow for separation of the biodiesel from glycerine after which the biodiesel gotten was separated by a decantation process and was washed with water and heated to obtain pure biodiesel.

### 3.2.3 Analysis

#### 3.2.3.1 Characterisation of the Biodiesel Produced

##### ❖ **Determination of Specific Gravity: ASTM D1298 — Density, Relative Density (Specific Gravity) determination, by Hydrometer Method**

This procedure is used to measure of specific gravity of the biodiesels. A clean dry empty 50ml density bottle is to be weighed and the mass recorded as  $M$ , it is then filled up with distilled water and subsequently with the samples. The mass of the bottle and water is taken and recorded as  $M_1$  and that of biodiesel as  $M_2$  respectively hence, the

specific gravity is evaluated. This procedure is used to determine the specific gravity of the sample.

$$\text{Specific gravity (biodiesel produced)} = \frac{m_1 - m}{m_2 - m}$$

❖ **Determination of Flash Point: ASTM D 93 — Flash-Point by Pensky-Martens Closed Cup Tester:**

A sample of the biodiesel is heated in a close vessel and ignited. When the sample burns, the temperature is recorded; the pensky-martens cup tester measures the lowest temperature at which application of the test flame causes the vapour above the sample to ignite. The biodiesel is placed in a cup in such quantity as to just touch the prescribed mark on the interior of the cup. The cover is then fitted onto the position on the cup and Bunsen burner is used to supply heat to the apparatus at a rate of about 5°C per minute. During heating, the oil is constantly stirred. As the oil approaches its flashing, the injector burner is lighted and injected into the oil container after every 12 second intervals until a distinct flash is observed within the container. The temperature at which the flash occurred is then recorded, it is repeated three times and the average taken (Van Gerpen, 2005).

❖ **Determination of cloud point: ASTM D 2500 — Cloud Point of Petroleum Products**

A sample of the biodiesel is placed in a test jar to a mark and then placed inside a cooling bath. The temperature at the bottom of the test jar that is the temperature at which the biodiesel starts to form cloud is taken as the cloud point (Van Gerpen, 2005).

❖ **Determination of kinematic viscosity: ASTM D 445 — Kinematic Viscosity**

A viscometer is inserted into a water bath with a set temperature and left for 30minutes. The sample is added to the viscometer and allowed to remain in the bath as long as it reaches the test thermometer. The sample is allowed to flow freely and the time required for the meniscus to pass from the first to the second timing mark is taken using a stop watch. The procedure is repeated a number of times and the average value are taken which is then multiplied with the viscometer calibration to give the kinematic viscosity (Van Gerpen, 2005).

❖ **Determination of pour point: ASTM D 97 — Pour Point of Petroleum Products**

A sample of the biodiesel is kept in the freezer to about 50<sup>0</sup>C then placed in a heating mantle to melt. The temperature at the bottom of the test jar that is the temperature at which the biodiesel starts to pour is taken as the pour point (Van Gerpen, 2005).

❖ **Cetane Number of Diesel Fuel Oil ASTM D 613**

Is a measure of the fuel's ignition delay, Higher Cetane numbers indicate shorter times between the injection of the fuel and its ignition. Higher numbers have been associated with reduced engine roughness and with lower starting temperatures for engines (Van Gerpen, 2005).

#### ❖ Acid Number of Petroleum Products by Titration ASTM D 664

The biodiesel sample is measured and poured in a beaker. A neutral solvent (a mixture of petroleum ether and ethanol) is prepared and 50ml of it is taken and poured into the beaker containing the biodiesel. The mixture is stirred vigorously for 30minutes. 0.56g of potassium hydroxide (KOH) pellet is measured and placed in a separate beaker and 0.1M KOH is prepared, 3drops of phenolphthalein indicator is added to the sample and is titrated against 0.1M KOH till the colour change observed turned pink (Van Gerpen, 2005).

## CHAPTER FOUR

### 4.0 RESULTS AND DISCUSSION

#### 4.1 Results

##### 4.1.1 Characterization of used vegetable oil

Table 4.1 Characterization of used vegetable oil

Sample name	Used Vegetable oil
Acid value	17.391 mg KOH/g
FFA value	8.70 mg/g
Iodine value	119g
Peroxide value	10
Saponification value	191.388 mg/g
Specific gravity	0.912
Refractive index	1.463

##### 4.1.2 Biodiesel produced

Table 4.2 Biodiesel produced and its yield

Quantity of oil (ml)	Quantity of catalyst (g)	Temperature (°C)	Time (Minutes)	Biodiesel produced (ml)	Glycerine produce (ml)
100	0.5	45	60	58	19
100	1.5	65	90	79	19
100	2.0	70	120	70	25

### 4.1.3 Characterization of Biodiesel Produced

**Table 4.3** Characterization of Biodiesel produced (Sample 1)

TEST	UNIT	TEST METHOD		LIMIT	RESULT
		IP	ASTM		
Specific gravity	kg/l	160	D1298	0.95 max	0.88
Total sulphur	% wt	107	D4294	0.5max	0.006
Flash point	°C	54	D93	150 min.	ND
Pour point	°C	219	D97	70 max	ND
Kinematic viscosity	c.s.t	71	D445	26 max	4.65
Diesel index	°C	21	-	47 min.	18.0
Cetane number		-	D975	40 min.	52.5
Free Glycerine	%mass	-	-	0.02	0.019
Total Glycerine	%mass	-	-	0.24	0.17
Cloud point	°C	219	D2600	40 max	20
Water by Distillation	%vol.	53	D95	0.5 max	Trace
Acid value	mg KOH/g	65	D108	0.5 max	0.43

**Table 4.4** Characterization of Biodiesel produced (Sample 2)

TEST	UNIT	TEST METHOD		LIMIT	RESULT
		IP	ASTM		
Specific gravity	kg/l	160	D1298	0.95 max	0.89
Total sulphur	% wt	107	D4294	0.5max	0.006
Flash point	°C	54	D93	150 min.	ND
Pour point	°C	219	D97	70 max	ND
Kinematic viscosity	c.s.t	71	D445	26 max	4.65
Diesel index	°C	21	-	47 min.	19.0
Cetane number		-	D975	40 min.	53.5
Free Glycerine	%mass	-	-	0.02	0.019
Total Glycerine	%mass	-	-	0.24	0.18
Cloud point	°C	219	D2600	40 max	20
Water by Distillation	%vol.	53	D95	0.5 max	Trace
Acid value	mg KOH/g	65	D108	0.5 max	0.46

**Table 4.5** Characterization of Biodiesel produced (Sample 3)

TEST	UNIT	TEST METHOD		LIMIT	RESULT
		IP	ASTM		
Specific gravity	kg/l	160	D1298	0.95 max	0.88
Total sulphur	% wt	107	D4294	0.5max	0.006
Flash point	°C	54	D93	150 min.	ND
Pour point	°C	219	D97	70 max	ND
Kinematic viscosity	c.s.t	71	D445	26 max	4.65
Diesel index	°C	21	-	47 min.	19.0
Cetane number		-	D975	40 min.	53.1
Free Glycerine	%mass	-	-	0.02	0.019
Total Glycerine	%mass	-	-	0.24	0.17
Cloud point	°C	219	D2600	40 max	20
Water by Distillation	%vol.	53	D95	0.5 max	Trace
Acid value	mg KOH/g	65	D108	0.5 max	0.45

## 4.2 Discussion

### 4.2.1 Effect of Catalyst concentration

#### 4.2.1.1 Effect of Catalyst concentration on purity

From the three samples of biodiesel produced as seen in the results, the concentrations of catalysts used vary for the three samples with the same quantity of oil. When a little quantity of the catalyst was used for the first sample, it was discovered that the purity of the biodiesel obtained did not conform to the acceptable standard as seen in most literatures and this could prove costly for its use in automobiles using diesel engines. This is because the viscosity of the diesel, cetane rating among other properties are below the recommended value and this could lead to excessive use of the diesel by automobiles, smoky exhaust, e.t.c which is not advisable as smoky exhaust causes the release of carbon monoxide to the environment which could prove harmful to the people

living in that area and the excessive increase in the cost of maintaining the vehicle for the user (Jose *et al.*, 2005).

#### **4.2.1.2 Effect of Catalyst Concentration on Yield**

From the three samples of biodiesel produced, the yields obtained are of three different volumes despite using the same quantity of oil for the production. The yield of the first sample was smallest of the three; this was followed by the third sample with a catalyst concentration of 2.0g and then the second with a catalyst quantity of 1.5g. The second sample produced more biodiesel because of the reaction ratio of the oil sample to the catalyst. From this, It can be deduced that a biodiesel produced using this ratio will yield more product and less glycerine (Jose *et al.*, 2005).

#### **4.2.2 Effect of reaction of reaction time**

##### **4.2.2.1 Effect of Reaction Time on Purity**

From the results obtained, the reaction time of 90 minutes produced the biodiesel that is in its purest state than the other two at 60minutes and 120 minutes. This implies that at a lower reaction or higher reaction time than 90 minutes, the biodiesel produced may likely be of low quality as well as contain some form of impurities and this can be checked by ensuring that diesel produced at acceptable reaction ratio are allowed to completely react at corresponding time which will enhance the purity of the diesel produced (Jose *et al.*, 2005).

##### **4.2.2.2 Effect of Reaction Time on Yield**

The result obtained shows that the highest quantity of biodiesel produced was at a reaction time of 90 minutes. This implies that the biodiesel produced at 60 minutes and 120 minutes have lower yield and which suggest that with a reaction time of 90minutes, the second sample produced more biodiesel with good combustion properties and less glycerine than the other two (Jose *et al.*, 2005).

## CHAPTER FIVE

### 5.0 CONCLUSIONS AND RECOMMENDATIONS

#### 5.1 Conclusions

From the results shown, the objective of the project was achieved. The production of quality biodiesel was done using single step transesterification process with three (3) different samples of the same oil but with different quantity of catalyst and methanol solution as well as the time of heating and the heating temperature. This was done to ascertain an acceptable reaction ratio to be used for production of high quality biodiesel. After the production, the biodiesel produced at a temperature of 65°C, time of heating as 90minutes and quantity of catalyst as 1.5g (NaOH) was found to be of higher quality and generated more yield than the rest with a good reaction ratio(oil : catalyst). From this, it can be conclusively said that producing biodiesel using the above mentioned ratio, will result in high yield of quality biodiesel using used vegetable oil as feed stock.

#### 5.2 Recommendations

- a) I recommend that safety precautions should be carried out when producing biodiesel in the laboratory so as to avoid health hazards.
- b) The laboratory in the department should be well equipped to enhance research on renewable energy.
- c) The use of renewable energy should be encouraged due to its environmental friendly nature and reduce over-dependence on energy from fossil fuel.
- d) The department should encourage students working on renewable energy by exposing them to advanced technologies through effective participation in conferences, seminars and symposium with players already in the field.

- e) I recommend that when carrying out such a research work next time, the free fatty acid present in the used vegetable oil should be removed so as to obtain a higher yield of biodiesel as its presence affects the yield of biodiesel.
- f) I also recommend that the single step transesterification method should be used in subsequent research work on biodiesel production because it saves time, is reduces production cost, better understood for first time researchers and also produces pure biodiesel with good combustion characteristics.

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## APPENDIX

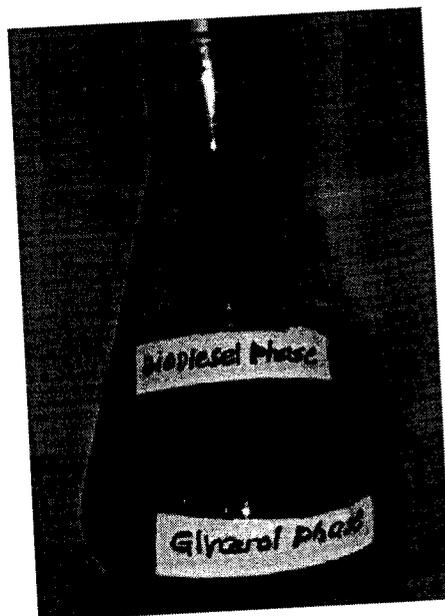


Plate 2: Biodiesel using NaOH catalysts

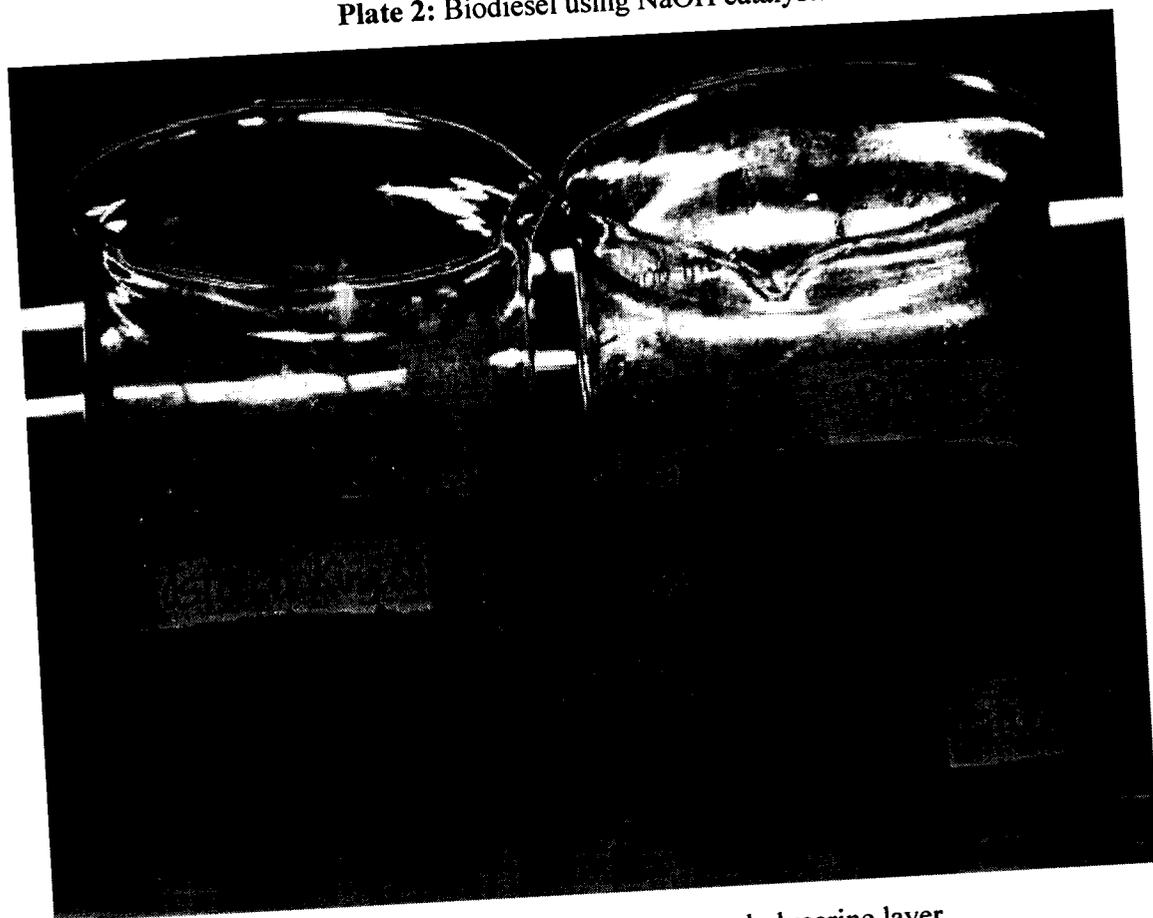
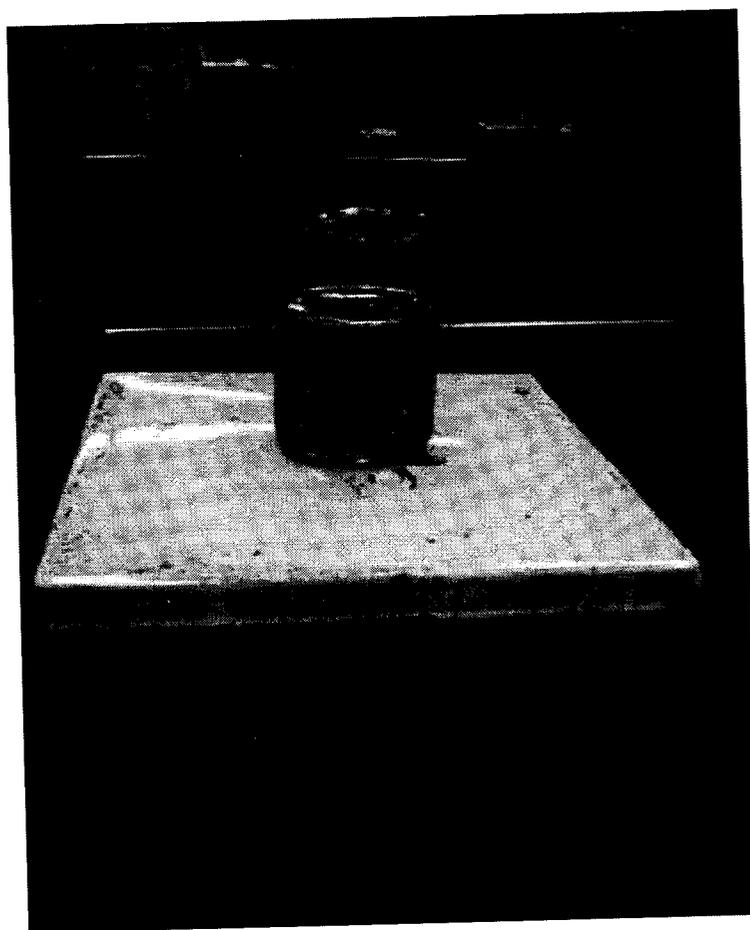


Plate 3: Biodiesel ester and glycerine layer



**Plate 1:** Biodiesel washing using distilled water



**Plate 4:** Glass reactor with heating plate and magnetic stirrer