PRODUCTION OF BIODIESEL FROM SOYABEAN OIL.

BY

HALILU, NDAASEBE MOHAMMED 75%

(2004/ 18500EH)

A PROJECT SUBMITTED TO

THE DEPARTMENT OF CHEMICAL ENGINEERING

SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY

FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA
NIGER STATE, NIGERIA

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IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE
AWARD BACHELOR ENGINEERING
(B.ENG)

DECEMBER, 2009.

DECLARATION

ALILU, NDAASEBE MOHAMMED hereby declare that this research project ,"Production Biodiesel From Soya bean Oil" carried out under the Supervision of Engr.A.G Isah and ented in partial fulfillment of the requirement for the Award of Bachelor of neering.(B.Eng)degree in chemical Engineering as not been presented for any degree where, to the best of my knowledge. All literature cited have been duely acknowledged in eference.

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21/01/2020
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Date

CERTIFICATION

The undersigned certify that this work was carried out in the department of Chemical Engineering, Federal University of Technology, Minna, and has been read and recommended to the School of Engineering and Engineering Technology , Minna, for acceptance, these titled "Production of Biodiesel from Soyabean Oil". Submited by Halilu Ndaasebe Mohammed in partial; fulfilment of the award of the degree of Bachelor of Engineering (B. Eng) in chemical Engineering.

13/11/09
Signature and Date

Engr A.G. Isah
Supervisor

....
Signature and Date

Dr J.Okafor
Head of Department

Prof. A.J. Kehinde
External Examiner

5/03/2010
Signature and Date.

DEDICATION

This project is dedicated to God who sustained my life until this day. The work is whole – heartedly dedicated to my father and mother, Mr. and Mrs. Halilu who bore the material and non – material burden of my educational career.

ACKNOWLEDGEMENT

It is impossible to thank individually, all those who have contributed to the success of this work. First and foremost, I am grateful to Almighty God not only for the inspiration he has given me to pursue higher studies, but for his enormous generosity throughout the hard and deplorable conditions of heavy academic work, he has granted me victory and survival. I am happy to acknowledge the work of my supervisor Engr A.G Isah for guiding me throughout this work. I wish to record my indebtedness to my Father Commander Halilu.B.H, whose financial assistance to my educational upbringing cannot be overemphasized. My appreciation goes to Engr Manasseh, the H.O.D. and all lecturers of chemical Engineering. I sincerely appreciate the effort of the following during my study; Udo Gudluck, Oguche Benjamin,Amozua Geofrey , mathew Afolayan, Halilu Mohd,Bola,Tope,Joseph, Sule Mohammed, Luca panthong ,Abubakar Bake, not forgetting Mallam Kudu of NCRI Laboratory Badegi for his assistance during my practical's .Finally my profound appreciation goes to my family, Mohammed Halilu,Hauwa Jibrin Khadijat Halilu, Fatima,Alhassan,Ramatu, Abdullah and Sadiq Halilu For there moral support throughout my studies.

ABSTRACT

The biodiesel sample was prepared from extracted soyabean oil at over different reaction times. Methyl alcohol with potassium hydroxide as catalyst was used for the transesterification process. The preliminary investigation of the soyabean oil was conducted and the acid value was obtained to be 3.4 mgKOH/g, viscosity at 32 °C was 35 mm^2/sec, saponification value was 172.30 mgKOH/g iodine value was 107.10 gramsIodine/100g and pH was 4.1 The maximum yield of methyl ester (biodiesel) ~~was~~ obtained at 35 minutes of reaction time was (87.10g) while the minimum yield obtained at 25 minutes was (70.75g). The biodiesel produce was characterized for flash point which was gotten as 147 °C, Viscosity 4.7 mm2 and Specific gravity 0.87 which fall within the range of values expected for biodiesel.

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CHAPTER ONE

1.0 INTRODUCTION

Biodiesel is obtained from transesterification of animal fats or vegetable oils. It is a non fossil fuel which is clean burning biodiesel is safe non toxic, biodegradable, renewable fuel and high quality and can be used in any diesel motor engines with little to no modifications to the engine. Biodiesel is compatible with it and can be blended with petroleum diesel in any ratio and will reduce emission in proportion to the percentage of biodiesel used. Biodiesel fuel consists of methyl esters of vegetable oils and belongs to ecological fuels because of its qualitative compositions such as carbon 77%, hydrogen 12%, oxygen 11% and traces of sulfur and nitrogen. Biodiesel as a fuel of biological origin. It is recommended by the European Union and has been classified as the future fuel, (Jordanov et al, 2007).

Biodiesel may contain small but problematic quantities of water.Although it is not miscible with water,it is like ethanol,hygroscopic (absorb water from atmospheric moisture).One of the resins biodiesel can absorb water is the persistence of mono and diglycerides left over from an incomplete reaction.The molecules can act as emulsifier allowing water to mix with biodiesel. (Wikipedia, 2008).

For engine designed to burn biodiesel fuel the viscosity of soyabean oil must be lowered to allow for proper atomization of fuel otherwise incomplete combustion and carbon build up will ultimately damage the engine.

The biodiesel has the following advantages

- Blends of 20% biodiesel with 80% petroleum diesel can be used in unmodified diesel engines. Biodiesel can be used in its pure form but many require certain engine modification to avoid maintenance and performance problems.
- It was stated that about half of the biodiesel industry can use recycled oil or fats. The other half being soybeans or rapeseed oil according to the origin of this feedstock's

- Biodiesel is non toxic, biodegradable it reduces the emission of harmful pollutants (mainly particulate) from diesel engine 80% less CO2 emission, 100% percent sulphur dioxide. But emissions of nitrogen oxide (precursor of ozone) are increased.
- Biodiesel has a high cetane number (above 100 compared to only 40 for diesel fuel).Cetane number is a measure of a fuel ignition quality. The high cetane numbers biodiesel can contribute to easy cold starting and low idle noise.
- The use of biodiesel can extend the life of the diesel engine because it is more lubricating and further more power out put are relatively unaffected by biodiesel.
- Biodiesel replaces the exhaust odor of petroleum diesel with a more pleasant smell of pop corn or French fries (M.Canakci, 1999).

1.2 AIMS AND OBJECTIVES OF THE STUDY

The aim of this research is to produce biodiesel which is clean burning, non-toxic,biodegradable and renewable fuel.

OBJECTIVES

The work will be carried out through the following objectives:

- Extraction of oil from soyabean using soxhlet extractor.
- To determine the optimum time required for the transesterification of soybean oil using constant reactant ratio and temperature.
- To investigate the use of potassium methoxide for transesterification of soyabean oil.

1.3 SCOPE OF WORK

This work is focus on the use of soxhlet extraction to produce oil from soya bean with later application of potassium hydroxide as the catalyst at a temperature of 70°C and transesterification was carried out

1.4 JUSTIFICATION

These research works is of significance in the sense that biodiesel is going to be produced from soyabean oil and its quality is going to be improved upon so that it could have uses in wide range of application. Biodiesel from soya bean oil approximately reduced the emission of harmful pollutant.

In the long run we would have a healthier environment cheaper biodiesel and more vibrant agricultural sector saddle with the responsibility of producing soyabean to meet the anticipated high demand. In essence our gross domestic product (GDP) will increased appreciably.

CHAPTER TWO

2.0 LITERATURE REVIEW

Soyabeans (U.S) or soybean (U.K) is species of crop native to East Asia.Ancient Chinese document state that soyabean agriculture began during the Zhou dynasty in 664BC and was introduced to the Zhou heartland from the Manchurian plains (Britannica Concise Encyclopedia) Archaeologists recovered soya beans from a site in Jili province and dated charcoal associated with it to cal. 900 – 520BC.

Reports indicate that the Eighteenth Century Missionaries returning from china brought the first soyabeans to Western Europe in the early 1700s (Britannica Concise Encyclopedia). Soyabeans were first introduced to the United States in 1760s where it was grown for hay. American took interest in the crop around 1952 and since then, soya beans acreage has grown tremendously. Today the USA produces 83.9millions metric tons of soyabeans and this represent about 40% of the worlds supply (Britannica Concise Encyclopedia). In Nigeria today, it is grown in most northern states with Benue being the chief producer. With encouragement from the Federal Government in area of providing subsidy and modern agricultural inputs. Nigeria is poised to join the largest producers in the world (Free Soyabean Encyclopedia).

2.2 PHYSICAL CHARACTEISTICS

Soyabeans exist in various sizes and in several coat colors including black brown yellow and mottled. The seed coat of mature bean is hard, water resistance and protects the cotyledon hypocotyls from damage. If the seed coat is cracked, the seed will not germinate. The sear visible on the seed coat is called the helium. At one end of the helium is the seed coat which can allow the absorption of water. Remarkably, seed such as soyabeans containing very high level of protein can undergo desiccation yet survive and receive after water absorption (Britannica concise Encyclopedia).

2.3 CHEMICAL COMPOSITION

The oil and protein content together account for about 60% of dry soyabeans by weight, protein at 40% and oil at 20%.The remainder consist of 35% carbohydrate and about 5% ash (Britannica Concise Encyclopedia).

Majority of soy protein is a relatively heat stable storage protein. This heat stability enables soy food products requiring high temperature cooking such as soy milk and soy flour to be made without destroying the protein content.

2.4 BIODIESEL

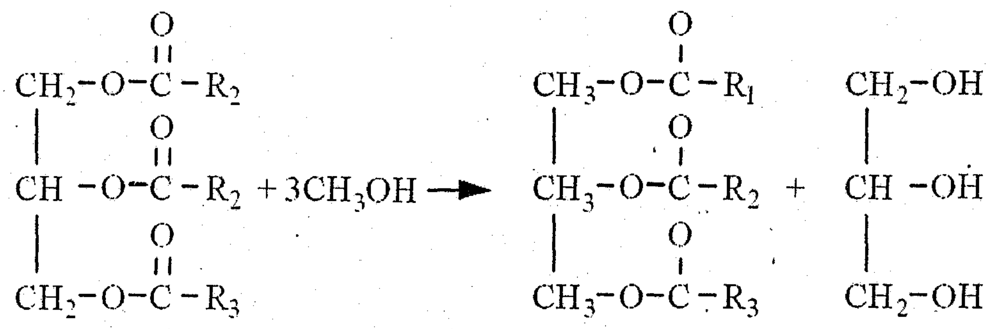
Biodiesel is the name for a variety of ester based fuels generally defined as the monoalkyl esters made from vegetable oils such as soybean oil, canola or hemp oil or sometime from animals fats through a simple transesterification process. This renewable source is as efficient as petroleum diesel in powering unmodified diesel engine.

The methyl esters derived from vegetable oil (biodiesel) have good potential as an alternative diesel fuel. The cetane number energy content, viscosity and phase changes of biodiesel are similar to those of petroleum based diesel fuel.

Article released by Stidham W.D and Dnzer M.F. 2007, outline that Animal and plant fats and oils are typically made of triglycerides which are esters of free fatty acids with the trihydric alcohol, glycerol in the transterification process, the alcohol is deprotonated with a based to make it a stronger nucleophile.Commonly ethanol or methanol are used as can be seen the reaction has no other inputs than the triglycerides and the alcohol. Normally this reaction will proceed more quickly. It is important to note that the acid or base are not consumed by the transesterification reaction Thus, they are not reactants but catalyst almost all biodiesel is produced from virgin vegetable oils requiring only low temperature and pressure and producing over 98% conversion yield (provided the starting oil is low in moisture and free fatty acids) (Stidham and Danzer, 2007).

Graboski and McCormick have summarized several experimental studies of biodiesel characteristics they report that the cetane number for biodiesel ranges from 45.8 - 56.9 for soya beans oil methyl esters with an average of 50.9 In comparison the cetane index for petroleum diesel ranges from 40 to 52.They imply that careful production control could result in biodiesel products with cetane numbers in the high end of the range whereas petroleum diesel tends towards the low end of the range. United State refiners use the catalytic cracking and cooking process to increase gasoline output from oil refineries yielding high octane gasoline material but low cetane diesel material. The alkyl esters of fatty oils (biodiesel) produced by transesterification of the oil.

A typical stoichiometry of such reaction is given as follows



$$\begin{array}{l} CH_2-O-\overset{O}{\overset{\|}{C}}-R_2 \\ | \\ CH-O-\overset{O}{\overset{\|}{C}}-R_2 \\ | \\ CH_2-O-\overset{O}{\overset{\|}{C}}-R_3 \end{array} + 3CH_3OH \longrightarrow \begin{array}{l} CH_3-O-\overset{O}{\overset{|}{C}}-R_1 \\ | \\ CH_3-O-\overset{O}{\overset{|}{C}}-R_2 \\ | \\ CH_3-O-\overset{O}{\overset{|}{C}}-R_3 \end{array} + \begin{array}{l} CH_2-OH \\ | \\ CH-OH \\ | \\ CH_2-OH \end{array}$$

2.2 BIODISEL PROCESSING

The process is called transesterification similar to saponification.Saponification is soap making to make soap you take a transfatty acid or triglyceride (oil) and blend it with a solution of sodium hydroxide (NaOH or caustic soda) and water. This reaction causes the ester chains to separate from the glycerin these esters chains are what becomes the soap. They are also called lipids their unique characteristics of being attracted to polar molecules such as water on one end and to non polar molecules like oil on the other end is what makes, them effective as soap.In transesterification caustic soda are mixed to create sodium methoxide ($Na^+CH_3O^-$) when mixed in with the soybean oil this strong polar bonded chemical breaks the transfatty acid into glycerin and also ester chains (biodiesel) along with some soap if you are not painstaking or vigilant (more on that later) the esters become methyl esters they would be esters if reacted with ethanol instead of methanol.

For the transesterification process

Fats/oil + NaoH + H_2O → esters chain +glycerin

Glycerin CH_3OH water soap
Sodium methoxide

Other important operational process of biodiesel production involves filtering heating titration and so on more of these procedures will be discussed in chapter 3 of the work.

2.3 BIODIESEL PRODUCTION PROCESS

Biodiesel are commonly produced by the transesterification of the feedstock. There are several methods for carrying out this transesterification reaction including the common batch process, supercritical process ultrasonic methods and microwave method (Wikipedia, 2008.)

2.3.1 SUPERFICIAL PROCESS

An alternative catalyst free method for transesterification uses .superficial methanol at high temperatures and pressures in a continuous process in the superficial state the oil and methanol are in a single phase, and reaction occurs spontaneously and rapidly. The process can tolerate water in the feedstock free fatty acids are converted to methyl esters instead of soap so a wide variety of feedstock can be used. Also the catalyst removal step is eliminated high temperatures and pressures are required but energy costs of production are similar or less than catalytic production routes.

2.3.2 ULTRA AND HIGH SHEAR IN LINE AND BATCH REACTORS

Ultra and high shear in line or batch reactors allow production of biodiesel continually semi continuously and in batch mode. This drastically reduces production time and increases production volume. The reaction takes place in the high energy shear zone of the ultra and high shear mixer by reducing the droplet size of the immiscible liquid such as oil or fats and methanol. Therefore the smaller the droplet size the larger the surface area the faster the catalyst can react.

2.3.3 ULTRASONIC REACTOR METHOD

In the ultrasonic reactor method the ultrasonic waves cause the reaction mixture to produce and collapse bubbles constantly this cavitations' provides simultaneously the mixing and heating required to carry out the esterification process thus using an ultrasonic reactor for biodiesel production drastically reduces the reaction time, reaction temperature and energy input hence the process of transesterification can run inline rather than using the time consuming batch processing. Industrial scale ultrasonic devices allow for the industrial scale processing of several thousand barrels per day.

2.3.4 MICROWAVE METHOD

Current research is being directed into using commercial microwave ovens into provide the heat needed in the transesterification process. The microwave provide intense localized heating that may be higher than the recorded temperature of the reaction vessel a continuous flow process producing 6litres/minutes at a 99% conversion rate has been developed and shown to consume only one-fourth of the energy required in the batch process although it is still in the laboratory scale development stage the microwave method holds great potential to be an efficient and cost comparative method for commercial scale biodiesel production

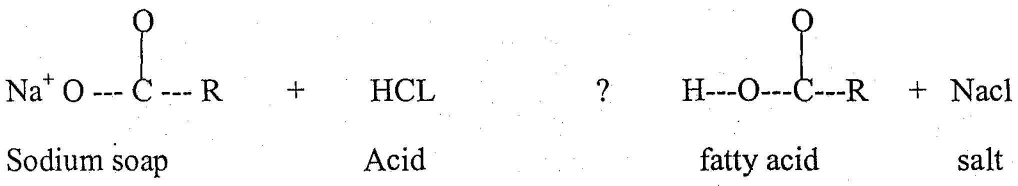
2.3.5 BATCH PROCESS

In the batch process care must be taken to monitor the amount of water and free fatty acids in the incoming biolipds (oil and fat). if the free fatty acid level or water level is too high it may cause problems with soap formation (saponification) and the separation of the glycerin by product downstream Catalyst is dissolved in the alcohol using a standard agitator or mixer. The alcohol/catalyst mix is then charged into a closed reaction vessel and the biolipid (vegetable oil or animal oil or fat) is added the system from here on is totally closed to the temperature of the atmosphere to prevent the loss of alcohol The reaction mix is kept just above the boiling point of the alcohol (around 70%, 1580F) to speed up the reaction through some systems recommend the reaction take place anywhere from room temperature to 55^{0}C (131^{0}F) for safety reasons. Recommended reaction time varies from 30 min to 8 hours under normal conditions the reaction rate will double with every 10^0 C increase in reaction temperature excess alcohol is normally used to ensure total conversion of the fat or oil to its esters the glycerin phase is much denser than biodiesel phase and the two can be gravity separated with glycerin simply drawn off the bottom of the setting vessel. In some cases a centrifuge is used to separate the two materials faster. Once the glycerin and biodiesel phases have been separated the excess alcohol in each phase is removed with a flash evaporation process or by distillation. In other systems the alcohol is removed and the mixture neutralized before the glycerin and esters have been separated. In either case the alcohol is recovered using distillation equipment and is reused. Care must be taken to ensure no water accumulates in the recovered alcohol stream. The glycerin by product contains unused catalyst and soaps that are neutralized with an acid and sent to storage as crude glycerin (water and alcohol are removed later, chiefly using evaporation to produce 80 - 88% pure glycerin)

Once separated from the glycerin, the biodiesel is sometimes purified by washing gently with warm water to remove residual catalyst or soaps dried and sent to storage An alcohol catalyst and oil are combined in a reactor and agitated for approximately one hour at 60 ^{0}C smaller plants often use batch reactors (W.D Stidham et al, 2000) but larger plants (> 4 million litres/yr) use continuous flow processes involving continuous stirred tank reactors (cstr) or plug flow reactors (G.assman et al, 1996,).

The reaction is sometimes done in two stages where approximately 80% of the alcohol and catalyst is added to the oil in a first stage CSTR then the product stream from the reactor goes through a glycerol removal step before entering a second a second CSTR the remaining 20% of the alcohol and catalyst are added in this second reactor this system provides a very complete reaction with the potential of using less alcohol than single step systems following the reaction the glycerol is removed from the methyl esters due to the low solubility of the glycerol in the esters, this separation generally occurs quickly and may be accomplished with either a settling tank or a centrifuge the excess methanol tends to act as a solubilizer and can slow the separation. However this excess methanol is usually not removed from the reaction stream until after the glycerol and methyl esters are separated due to concern about reversing the transesterification reaction water may be added to the reaction mixture after the transesterification is complete to improve the separation of glycerol. (W.D Stidham et al, 2000 and T.Wimmer et al, 1999)

After the separation from the glycerol the methyl esters enter a neutralization step and then pass through a methanol stripper, usually a vacuums flash process or a falling film evaporator before water washing acid is added to the biodiesel to neutralize any residual catalyst and to split any soap that may have formed during the reaction. Soaps will react with the acid to form water soluble salts and free fatty acids as shown in the reaction



$$Na^{+}O{-}{-}{-}\overset{O}{\overset{|}{C}}{-}{-}{-}R \quad + \quad HCL \quad ? \quad H{-}{-}{-}O{-}{-}{-}\overset{O}{\overset{|}{C}}{-}{-}{-}R \quad + \quad Nacl$$

The salt will be removed during the water washing step and the free fatty acids will stay in the biodiesel. The water washing step is intended to remove any remaining catalyst soap salt methanol or free glycerol from the biodiesel Neutralization before washing reduces the water required and minimizes the potentials for emulsion to form when the wash water is added to the biodiesel. Following the wash process any remaining water from the biodiesel by a vacuum flash process. The glycerol stream leaving the separator is only about 50% glycerol it contains some of the excess methanol and most of the catalyst and soap. In this form the glycerol has little value and disposal may be difficult. The methanol content requires the glycerol to be treated as hazardous waste. The first step in refining the glycerol is usually to add acid to split the soaps into free fatty acids and salts. The free fatty acids are not soluble in the

Glycerol and will rise to the top where they can be removed and recycled the salts remain with the glycerol although depending on the economical compounds present some may precipitate out. One frequently touted option is to use potassium hydroxide as the reaction catalyst and phosphoric acid for Neutralization so that the salt formed is potassium phosphate which can be used for fertilizer. After acidulation and separation of the free fatty acids the methanol in the glycerol is removed by a vacuum flash process or another type of evaporator. At this point the glycerol should have a purity of approximately 85% and is typically sold to a glycerol refiner. The glycerol refining process takes the purity up to 99.5 to 99.7% Using vacuum distillation or ion exchange process the methanol that is removed from the methyl ester and glycerol streams will tend to collect any water that may have entered the process the water should be removed in a distillation column before the methanol is returned to the process this step is more difficult if an alcohol such as ethanol or isopropanol is used that forms an azeotrope with water then a molecular sieve is used to remove the water.

2.5 PERFORMANCE AND EMISSION CHARACTERISTICS OF BIODIESEL

One of the most important characteristics of diesel fuel is its ability to auto ignite a characteristic that is quantified by a fuels cetane number or cetane index, where a higher cetane number or index means that the fuel ignites more quickly United States petroleum diesel typically has a cetane number index in the low 40s and European diesel typically has a cetane index in the low 50s

i. Graboski and McCormick have summarized several experimental studies of biodiesel characteristics they report that the cetane number for biodiesel ranges from 45.8—56.9 for soya beans oil methyl esters with an average of 50.9 in comparison the cetane index for petroleum diesel ranges from 40 to 52.They imply that careful production control could result in biodiesel products with cetane numbers in the high end of the range whereas petroleum diesel tends towards the low end of the range. United State refiners use the catalytic cracking and cooking process to increase gasoline output from oil refineries yielding high octane gasoline material but low cetane diesel material.

ii. Lubricity another important characteristic of diesel fuel is a measure of lubricating properties. Fuel injectors and some types of fuel pumps rely on fuel for lubrication. One study published in 1998 and cited by the national diesel biodiesel board, found that one half of samples of petroleum diesel sold did not meet the recommended minimum standard for lubricity .biodiesel has better lubricity than current low sulphur petroleum diesel, which contains 500 parts per million (ppm) sulphur by weight the petroleum diesel lubricity problem is expected to get worse when ultra low sulphur petroleum diesel

(15ppm sulphur by weight) is introduced in 2006 A 1 or 2 percent volumetric blend of biodiesel in low sulphur petroleum diesel improves lubricity substantially it should be noted however that the use of other lubricity additives may achieve the same effect at lower cost.

iii. Biodiesel also has some performance disadvantages the performance of biodiesel in cold condition is markedly worse than that of petroleum diesel and biodiesel made from yellow grease is worse than soya bean biodiesel in this regard. At low temperature diesel fuel forms wax crystals which can clog fuel limes and filters in a vehicle fuel system. The cloud point is the temperature at which a sample of the fuel starts to appear cloudy indicating that wax crystals have begun to form. At even lower temperature diesel fuel becomes a gel that cannot be pumped. The pour point is the temperature below which the fuel will not flow the cloud and pour point for biodiesel are higher than those for petroleum diesel.

iv. Vehicles running on biodiesel blends may therefore exhibit more drivability problems at less severe winter temperatures than those vehicles running on petroleum diesel. This is a potential concern during the winter in much of the United States. The solvent property of biodiesel may be incompatible with the seals used in the fuel systems of older vehicles and machinery, necessitating the replacement of those parts if biodiesel blends are used. The initial used of B20 or B100 in any vehicle or machine required core petroleum diesel forms deposits in vehicular fuel systems and because biodiesel can loosen those deposits they can migrate and clog fuel lines and filters.

v. Another disadvantage of biodiesel is that it tends to reduce fuel economy energy efficiency is percentage of the fuels thermal energy that is delivered as engine output, and biodiesel has shown no significant effect on the energy efficiency of any test engine volumetric efficiency a measure that is more familiar to most vehicle users usually is expressed as miles travelled per gallon of fuel (or kilometers per litre of fuel). The energy content per gallon of biodiesel is approximately 11Percent lower than that of petroleum diesel vehicles running on B20 are therefore expected to achieve 2.2 Percent (20 percent * 11percent) fewer miles per gallon of fuel.

vi. About 11 percent of the weight of B100 is oxygen the presence of oxygen in biodiesel improves combustion and therefore reduces hydrocarbon carbon monoxide and particulate emissions but oxygenated fuels also tends to increase nitrogen oxide emissions. Engine test have confirmed the expected increases and decreases of each exhaust component from engines without emission.controls biodiesel users also note that the exhaust smells better than the exhaust from engine burning conventional diesel.

vii. The increase in nitrogen oxide emissions from biodiesel can be reduced by adding cetane number enhancers di-tert-butyl peroxide at 1 percent or 2 ethylhexylnitrate at 0.5 percent can reduce nitrogen oxide emissions from biodiesel and reducing the aromatic content of petroleum diesel from 31.9 percent to 25.8 percent is estimated to have the same effect in the case of petroleum diesel the reduction in aromatic content can be accomplished by blending fuel that meets environmental protection agency specification.

viii. Nitrogen oxide emission from biodiesel blends could possibly be reduced by blending with kerosene or Fischer tropsch diesel kerosene blended with 40% biodiesel has estimated emissions of nitrogen oxide no higher than those of petroleum diesel as doe's fisher tropsch diesel or kerosene could be used to reduce nitrogen oxide emissions from blends containing 20% biodiesel.

ix. Oxides of nitrogen and hydrocarbons are ozone precursors carbon monoxide is also an ozone precursor but to a lesser extent than unburned hydrocarbons or nitrogen oxides. Air quality modeling is needed to determine whether the use of biodiesel without additives to prevent increase in nitrogen oxide emissions will increase or decrease ground level ozone on balance.

2.6 STANDARDS OF VEGETABLE OIL IN BIODIESEL PRODUCTION

THE PROPERTIES OF VEGETABLE OIL

Property	Soya bean oil	Sunflower oil	Rapeseed
Density (kg/m^3)	922	933	921
Viscosity (mm^2/s)	31.7	64.1	35.9
Cetane index	47.8	49.1	47.5
Acid value(mgKOH/g)	2.67	3.82	2.88
Saponification value (mgKOH/g)	169	196	177
Iodine value (g)	106	117	109

The effects of some of these properties are discussed below

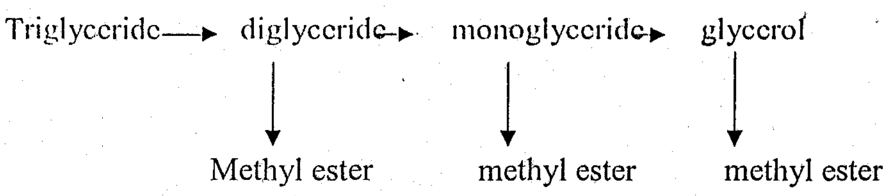
- The acid value is also called "Neutralization number" or Acid number and it is the mass of potassium hydroxide (KOH) in milligrams (mg) that is required to neutralize one gram of the oil. On a general note the more the oil is used the more acidic it becomes which means a higher acid value .The effect of this on the biodiesel production is that more quantity of the base will be required to neutralize the acidic content of the oil to be transesterified resulting in a higher cost of production of biodiesel.
- Cetane number which is a measurement of the combustion quality of diesel fuel during compression ignition it is a significant expression of diesel fuel quality among a number of other measurements that determine overall diesel fuel quality. Cetane number of a fuel is defined as the percentage by volume of normal cetane in a mixture of normal cetane and alpha methyl naphthalene which has the same ignition characteristics (ignition delay) as the test fuel when combustion is carried out in a standard engine under specified operating conditions. Generally diesel engines run well with a cetane number from 40 to 55 Fuels with higher cetane number which have shorter ignition delays provide more time for the fuel combustion process to be completed. Hence higher speed diesels operate more time effectively with higher cetane number fuels (Wikipedia, 2008)

- The iodine value is the mass of iodine in grams that is consumed by 100g of a substance the higher this value the more unsaturated fat it contains this then means that for a waste vegetable oil the iodine value will be lower than the range given in the figure on the table except for sunflower which is a used oil.
- The viscosity can be perceived as "thickness" or resistance to flow for most vegetable oil the viscosity is higher than that of used vegetable oil in biodiesel production a used vegetable oil is often preferred because of its low viscosity which is as a result of several uses allowing the fatty acid chain of the oil to be broken resulting in high free fatty acid of the oil. The effect of high viscose oil on final biodiesel is that it makes the oil congeal at low temperature(pour point) this is a general problem of biodiesel but it is compounded with the use of high viscose oil so low viscose oil is often used for transesterification.
- Saponification value is the measure of the mean molecular weight of fatty materials present in the oil it is expressed as the numbers of milligrams of potassium hydroxide required to saponites one gram of the oil. The process of saponites is the hydrolysis of triglycerides into glycerol and potassium salts of the fatty acid using a solution of potassium in alcohol the determination of the saponification value is a reasonably reliable means of characterizing the fat.
- The colour of the feedstock (vegetable oil) sometimes determine the colour of the biodiesel that will be obtained from the oil, this difference in colour cannot be easily detected because no chemical change that occurs during the transesterification reaction affects the colour of the product the noticeable difference will be just the viscosity of both as discussed above.

2.7 PRODUCTION PROCESS FACTORS

2.7.1 COMPLETION OF REACTION

The most important issue during biodiesel production is the completion of the transesterification reaction. The basic chemical process that occurs during the reaction is indicated by the following sequence of events.



The triglycerides are converted to triglycerides, which in turn are converted to monoglycerides and then to glycerol each step produces a molecule of a methyl ester of a fatty acid if the reaction is incomplete then there will be triglycerides diglycerides and monoglycerides left in the reaction mixture each of these compounds still contain a glycerol molecule that has not been released. The glycerol portion of these compounds is referred to as bound glycerol when the bound glycerol is added to the free glycerol the sum is known as the total glycerol. The ASTM specification requires that the total glycerol be less than 0.24% of the final biodiesel product as measured using a gas. Chromatographic method described in ASTM D 6584 since the glycerol portion of the original oil is usually about 10.5% this level of total glycerol corresponds to 97% reaction completion (G.V Gerpen, 1999).

2.7.2 FREE GLYCEROL

Free glycerol refers to the amount of glycerol that is left in the finished biodiesel. Glycerol is essentially insoluble in biodiesel so almost all of the glycerol is easily removed by settling or centrifugation. Free glycerol may remain either as suspended droplets or as the very small amount that is dissolved in the biodiesel. Alcohols can act as cosolvents to increase the solubility of glycerol in the biodiesel. Most of this glycerol should be removed during the water washing process. Water washed fuel is generally low in free glycerol, especially if hot water is used for washing. Distilled biodiesel tends to have a greater problem with free glycerol due to glycerol carry over during distillation. Fuel with excessive free glycerol will usually have a problem with glycerol settling out in storage tanks, creating a very viscose mixture that can plug fuel filters and cause combustion problems in the engine (J.V Gerpen, 1999).

2.7.3 RESIDUAL ALCOHOL AND RESIDUAL CATALYST

Since methanol (and ethanol) and alkaline catalyst are more soluble in the polar glycerol phase most will be removed when the glycerol is separated from the bio diesel however the biodiesel typically contains 2.4% methanol after the separation which may constitute as much as 40% of the excess methanol from the reaction. Most processors will recover this methanol using a vacuum stripping process. Any methanol remaining after this stripping process should be removed by the water washing process. Therefore the residual alcohol level in the biodiesel should be very low. A specific value for the allowable alcohol level is specified in European biodiesel standards (0.2% in EN 14214) tests have shown that as little as 1% methanol in the biodiesel can lower the flashpoint of the biodiesel from 170^{0}C to less than 40°C. Therefore by including a flash point specification of 130^{0}C the ASTM standard limits the amount of alcohol to a very low level (< 0.1%) residual alcohol left in the bio diesel will generally be too small to negatively impact the fuels performance. However lowering the flashpoint presents a potential safety hazards as the fuel may need to be treated more like gasoline which also has a low flash point than diesel fuel.

Most of the residual catalyst is removed with the glycerol like the alcohol remaining catalyst should be removed during the water washing. Although a value for residual catalyst is not included in the ASTM standard it will be limited by the specification on sulfated ash Excessive ash in the fuel can lead to engine deposits and high abrasive wear levels. The European standards EN 14214 places limits on calcium and magnesium as well as the alkali metals sodium and potassium (J.V Gerpen, 1999).

2.8 BIODIESEL SPECIFICATION

The standards ensure that the following important factors in the fuel production process are satisfied

- Acid value
- Complete reaction
- Removal of glycerin
- Removal of catalyst
- Removal of alcohol
- Absence of free fatty acids
- Low sulphur content

2.11 BIODIESEL PROPERTIES

A property of biodiesel mentioned in the following table must meet the specification mentioned for the property from the date mentioned for the property.

Item	Properties	Specification
1	Density at 50°C	860 – 890Kg/m3
2	Distillation at T90	360°C
3	Viscosity	3.5 – 5.0mm^2/40°C
4	Flash point	120°C(min)
5	Copper stripper corrosion	No.3max
6	Ester content	96.5 % (m/m)(min)
7	Acid value	0.80 mgKOH/g(max)
8	Total contamination	24 mg/kg(max)
9	Cetane number	51.0 (min)
10	Oxidation stability	6 hrs at 110°C (min)

(Biodiesel fact sheet, 2003).

2.10 ENVIRONMENTAL AND SAFETY INFORMATION OF BIODIESEL

- Skin irritation in humans: A 24hours human patch test indicated that undiluted biodiesel produced very mild irritation. The limitation was less than the result produced by a 4% soap and water solution.
- Biodegradability: The biodiesel degrades about four times faster than petroleum diesel. Within 28 days. Pure biodiesel degrades 85 to 88 percent in water. Dextrose (a test sugar used as the positive control when testing biodegradability) degraded at the same rate. Blending biodiesel with diesel fuel accelerates its biodegradability. For example, blends of 20 percent biodiesel and 80 percent diesel fuel degrade twice as fast as diesel alone

- Flash point: The flash point of a fuel is defined as the temperature at which it will ignite when exposed to a spark or flame .Biodiesel flash point is over 125°C well when above petroleum based diesel fuels flash point of around 58°C testing has shown the flash point of biodiesel blends increases. Therefore biodiesel and blends of biodiesel with petroleum diesel are safer to store handle and use than conventional diesel fuel (Biodiesel fact sheet, 2003).

CHAPTER THREE

3.0 RESEARCH METHODOLOGY

3.1 RAW MATERIALS USED

- Methanol
- Potassium hydroxide
- Hydrochloric Acid
- Ethanol Potassium Hydroxide
- Sodium Thiosulphate Solution
- Carbon Tetrachloride
- DAM'S Reagent
- n-Hexane
- Soya bean
- Phenolphthalein Indictor

3.2 EQUIPMENT TO BE USED

- Grinding Machine
- PH Meter
- Beaker
- Magnetic Stirrer
- Measuring Cylinder
- Weighing Balance
- Glass Stirrer
- Viscometer
- Soxhlet Extractor

- Conical Flask
- Water Bath

3.3 EXPERIMENTAL PROCEDURE FOR OIL EXTRACTION FROM SOYABEANS (SOXHLET EXTRACTION)

The soxhlet apparatus is designed to extract oil from materials by the use of hot solvent. This grind soyabean powder was transferred into a thimble and placed inside a receiver portion of the extactor.250nm of n-hexane was poured into a round bottom flask whose weight had earlier been taken the flask was then placed in a heating mantle. The soxhlet apparatus fixed with a condenser was mounted on the flask. The condenser water inlet was connected to tap by a rubber tube and the water outlet was channeled to the sink. The set up was held in position by a retort stand and the vent of the condenser was made air tight to prevent the escape of the solvent in the vapor state. The heating mantle was switched on and regulated to 8°C to commence the extraction process. The extraction was allowed to run for six hours the n-hexane was later observed to boil at about 72°C the vaporized n-hexane percolated into the thimble through. The tiny pore spaces to the sample the oil was extracted (leached) by the vaporized n-hexane. The n-hexane is then condensed through the constant flow of water into the soxhlet extractor. The oil extract containing n-hexane is then carried down to the round bottom flask as it refluxes the above process of reflux continues until a total extraction of oil was observed. To recover the solvent the mixture contained in the flask was continually heated and the solvent evaporated out of the mixture and condensed into the thimble chamber until it remains the pure oil in the flask. The condensed solvent in the thimble chamber was poured into a measuring cylinder to measure the solvent recovered while the flask containing the oil was also weighed to determine the weight of oil. This process was repeated for five more extraction runs to obtain a reasonable quantity for this research work.

3.4 PRELIMINARY ANALYSIS OF SOYBEAN OIL

3.4.1 Determination of acid value

The mixture of ethanol and toluene in ratio 1:1 by volume was first neutralized prior to use by using ethanol potassium hydroxide solution in the pressure of 0.3ml of indicator per 100ml of the mixture.2g of sample was weigh into 250ml conical flask 50ml of previously neutralized mixture of toluene and ethanol was added to the flask. A few drops of phenolphthalein indicator were added and the contents were titrated against 0.1ml/L solution until the indicator changes pink colour.

Calculation:

$$Av = \frac{56.10 \times C \times V}{m}$$

Where;

C= the exact concentration of hydrochloric acid

m= the mass of test sample

V = Volume of ethanol potassium hydroxide solution

3.4.2 DETERMINATION OF SAPONIFAICATION VALUE

2g of the soyabean oil was weighed into a conical flask; 25ml of ethanol potassium hydroxide was then added by the acid of pipette. A reflux condenser was placed on the electric heater. The content was allowed to boil gently for 60 minutes with shaking from time to time. 1ml of phenolphthalein indicator was then added to the flask and the content of the flask was titrated with 0.5ml/L hydrochloric acid until the pink colour of the indicator just disappeared. The same procedure was used for other samples and blank which contain no sample. Two determinations were carried out in each test sample.

Calculations:

$$S-\frac{(V_0-V_1)\times C\times 56.1}{m}$$

Where:

V_0– the volume of 0.5mol/l hydrochloric acid used for blank test

V_1= the volume of hydrochloric acid solution used for determination

C= the exact concentration of hydrochloric acid

m= the mass of test sample

3.5.3 DETERMINATION OF IODINE VALUE

2g of sample was weighed into conical flask. 20ml of carbon tetrachloride and 25ml of DAMS reagent was added to the flask. Stopped was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 1hours 30 minutes. At the end of the time, 20ml of potassium iodine solution and 150ml of water were added. The content of each flask was titrated with 0.1ml/L sodium thiosulpahte solution until the yellow colour due to the iodine has almost disappeared. Few drops of starch were then added and titration continued until the blue colour disappears after vigorous shaking.

Calculations;

The Iodine value is given by the expression $12.69C\,\frac{V_1-V_2}{m}$

Where;

C= concentration of Sodium Thiosulphate used

V_1= volume of the Sodium Thiosulphate solution used for determination

V_2= Volume of the sodium Thiosulphate solution used for determination

m= mass of the test sample

3.4.4 SPECIFIC GRAVITY

The specific gravity of the oil was determined by the use of the relationship between the weight of the oil and weight of an equal volume of water.' The density bottle was weight empty with weight (W_1) the density bottle was filled with oil and weighed (W_2). The bottle was emptied, washed and rinsed several times with water, and then filled with water with volume equal to that of the oil and weighed (W_1).

3.5 STUDY OF THE TRANSESTERIFICATION REACTIONS

100ml of soyabean oil is transferred into the 1000ml beaker serving as the reactor. At exactly 70°C, the potassium methoxide was added to the soyabean oil on the magnetic stirrer and heater, this is to enable uniform agitation and to maintain the temperature at 70°C while the reaction is carried out for a specified time.The amounts of reactants used are given below.

100ml of soyabean oil

25ml of methanol

2g of KOH

The reaction was carried out in four different batches using the same rates of reactants, at a temperature of 70°C but with varying reaction time. Time was set at 25, 35, 45 and 55 minutes. The product which was weighed was transferred into a separating funnel and left for about 16hours to allow for complete separation of methyl ester from by product (glycerine).

3.5.1 SEPERATION OF GLYCERINE (BY PRODUCT)

After about 16 hours the product has formed two distinct layers. A dark brown semi liquid bottom layer of glycerin and top layer of methyl ester (biodiesel). These were separated with the separated funnel by allowing the glycerine to flow out of the funnel.

3.5.2 WASHING OF METHYL ESTER

The purpose of washing the biodiesel is to remove soluble impurities basically the remnant of KOH in the form of soap from the biodiesel. This was done by adding distilled water to the biodiesel (since is insoluble in water) and shaking, then left to settle into layer of denser soapy Water, and a lighter biodiesel at the top which was separated to obtain the washed biodiesel.

3.6 CHARACTERIZATION OF THE BIODIESEL

The pure biodiesel obtained through the above procedure was characterized to test for the following fuel physical properties.

3.6.1 Flash Point

The flash point was determined using the pesky-marten equipment. The apparatus consist of a small cup into which the samples is put in the cup at regular intervals the cup was opened and open flame was directed into it when the vapour in the cup was able to produce a momentary flame the temperature range that produced the flame is the flash point.

3.6.2 VISCOSITY

The common Fensky Viscometer was used to determine the viscosity of the sample. Some quantity of the sample was put in the viscometer so that it just conceded with the level of the upper mark on the viscometer. The time taken for the meniscus of the sample to fall upper meniscus of the bulb was noted and viscosity was calculated.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

This chapter presents the results of the laboratory experiment conducted on sample at various stages of the project. Results obtained are presented in the form of tables and various observation from the study conducted are discussed.

4.1 RESULTS

Table 4.1 Result of Extraction

Sample	Amount of solvent (ml)	Weight of dish (g)	Weight of samples (g)	Weight of dish +oil (g)	Weight of oil extracted (g)	% of oil extracted
A	150	54.56	15	72.56	14.00	93
B	150	94.26	30	121.26	27.00	90
C	150	98.09	45	130.09	32.00	71
D	150	61.04	30	90.14	29.10	97
E	150	98.09	50	138.09	40.00	80

Table 4.2 Soya bean oil characteristics

Property	Value
pH	4.1
Saponification value	172.30 mgKOH/100g
Iodine value	107.10 giodine/100g
Acid value	3.4 mgKOH/g
Viscosity	35 mm^2/s
Specific gravity	0.911

Table 4.3 Experimental results

Reaction time	25 min	35 min	45 min	55 min
Yield of methyl ester before washing %	55.71	68.58	65.98	62.24
Yield of methyl ester after washing %	54.43	66.91	64.57	60.86

TABLE 4.4 PHYSICAL PROPERTIES AND YIELD OF BIODIESEL SAMPLES

Property	Biodiesel	Conventional diesel	ASTM-D
Viscosity	4.70mm^2/s	2.96mm^2/s	1.9 – 6.5
Flash point	147°C	70°C	150°C
Specific gravity	0.87	0.85	0.86 – 0.90
Appearance	7.3	8.0	7.5
Odour	Mild odour		Mild odour
Flammability	Non-sooty flame	Sooty flame	Non-sooty flame

4.2 DISCUSSION OF RESULT

The amount of oil extracted is shown in table 4.1 above it was observed that the amount of oil extracted with different sieve confirm that the rate of chemical reaction increases with increase in surface area. This is due to the fact that the particle size decreases the solute available for the solvent attach is increasing and hence more oil extracted.

Considering table 4.2: The property of the oil is necessary in the production of biodiesel to determine the nature of pretreatment needed for conversion of oil to esters. The pH of oil which was determine to be 4.1 .It shows how acidic the oil is the viscosity and specific gravity of the oil were also determine to be 35mm^2/s and 0.911 respectively. The effect of this on the final biodiesel obtained is that it prevents biodiesel from coagulating at a low temperature.

Considering table 4.3, which present the characteristics of biodiesel, at 25 minutes of reaction time, yield of methyl ester obtained before washing was 55.71% and after the washing process of methyl ester, the yield reduced to 54.43% it can further be said that the weight of glycerin obtained (48.20g) as the final byproduct, there is still some reacted methanol and potassium hydroxide that if more time, its yield will increase. Also, the 1.28% reduction in the yield after the washing process is as a result of side reactions during the transesterification process, this soluble soap in the methyl ester was then removed during the washing of the methyl ester with water.

At 35 minutes of reaction time, yield of methyl ester increased abruptly to 68.58% compared with 54.43% of the yield of 25min of reaction time. This sharp increase is as a result of the prolonged time of reaction, and to allow excess unreacted reactants to further undergo transesterification. This yield is the highest among three runs of the experimented carried out: The value then reduced to 66.81% after washing due to the removal of soluble soap in the biodiesel. Variations in values obtained as compared with literature may be due to free glycerol which is the amount of glycerol that is left in the finished biodiesel. Glycerol is essentially insoluble in biodiesel so almost all of the glycerol is easily removed by setting. Free glycerol may remain either as suspended droplets or as the very small amount that is dissolved in the biodiesel, because the methanol used can act as solvents to increase the solubility of glycerol in the biodiesel.

At 45 minutes of reaction time, yield obtained before washing of methyl ester 65.98% which is about 2.60% less than the yield of 35min. Also after washing, the yield is still considerably less than the Percentage yield obtained for 35min. The reasons for this slight decline may be because of the residual methanol and catalyst (KOH) since methanol and alkaline catalyst are more soluble in the polar glycerol phase most will be removed when the glycerol is separated from the biodiesel.

At 55 minutes of reaction time, there is also a decline in both percentage yield of methyl ester before washing with values 62.24% and 60.86% respectively. The possible reason for this drop in yield of methyl ester after 55min of reaction time could be the continuous reaction of excess methanol and some content in the reaction mixture.

Considering table 4.3, the physical properties of the biodiesel obtained from different batches are based on visual observations and laboratory procedures for the determination of specific gravity and viscosity. The specific gravity for all samples of biodiesel was all gotten as 0.87 which is almost the same with the standard value, therefore conforming to the standard. The viscosity value was 4.7mm^2/s which falls in the range from the literature review. The mild odour and pale yellow appearance of the biodiesel samples confirms with the expected findings.

CHAPTER FIVE

1.0 CONCLUSIONS AND RECOMMENDATIONS

1.1 CONCLUSION

The following conclusion could be drawn from the experimental analysis and results of this project work

The amount of oil extracted with difference sieve conforms that the rate of chemical reaction increases with increase in surface area.

Methanol transesterification of soyabean oil for biodiesel production is another possibility of producing cheap alternative fossils fuels, which could reduce pollution and protect the environment. The methyl ester obtained through the method outlined in this work can be used as a fuel in diesel engines, because of its satisfying physical properties that could be compared with standard methyl esters.

Biodiesel of good quality can be produced using potassium methoxide, at reaction temperature of 70°C and for 35minutes to obtain a maximal yield of methyl ester.

1.2 RECOMMENDATIONS

- Though it was expected that the potassium Hydroxide pellet used as a catalyst would yield more biodiesel than glycerin but it did not. I would recommend that subsequent experiment incorporate the use of Sodium Hydroxide as a catalyst.
- The chemical engineering departmental laboratory of Federal University of Technology Minna should be well equipped with biodiesel production facilities to enable students taking on this kind of project or related topic to be able to monitor the quality of biodiesel product and also that of the feedstock rather be seen as an outsider in other laboratories which sometimes slows the work and makes it hard for easy monitoring.
- This project has shown that soya bean oil can be converted to wealth through the production of biodiesel The use of soybean oil to produce biodiesel can only account for

a very low percentage of diesel consumption so farmers should be encouraged to concentrate on the production of non edible oils like rapeseed palm oil soybean oil, a well coordinated soyabean development programme should be set up in the country so as to fully utilize the benefit that can be derived from it.

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APPENDIX

EXPERIMENTAL CALCULATION

eight of the soya bean before drying =500g

eight of soya bean after drying =450g

noisture content $=\frac{500 - 450}{500}$

e weight of the sample before the extraction =54.56g

e weight of sample + dish =68.56g

e amount of oil extracted =68.56 – 54.56 =14

oil extracted = (14/15) ×100%=93.33%

e weight of the sample before the extraction = 94.26g

e weight of sample + dish = 121.26g

amount of oil extracted = 121.26 – 94.26=27

il extracted = (27/30) ×100%=90%

weight of sample before the extraction = 98.09g

weight of sample + dish = 130.09g

amount of oil extracted = 130.09 – 98.09=32

oil extracted = (32/45) ×100%=71%

e amount of sample before extraction = 61.04g

e weight of sample + dish = 90.14g

e amount of oil extracted = 90.14 – 61.14=29.1

oil extracted = (29.1/30) ×100=97%

e amount of sample before extraction = 98.09g

e weight of sample + dish = 138.09g

e amount of oil extracted = 138.09 – 98.08=40

oil extracted = (40/50) ×100=80%

termination of iodine value

mple	Mass of sample used (g)	1st titre value (ml)	2nd titre value (ml)	Average titre value (ml)
a bean oil	2.0	4.00	4.20	4.10
nk	-	25.60	25.50	25.5

rmination of saponification value

ple	Mass of sample used (g)	1st titre value (ml)	2nd titre value (ml)	Average titre value (ml)
bean oil	2.0	4.00	3.50	3.75
k	-	16.40	16.20	16.30

mination of acid value

e	Mass of sample used (g)	1st titre value (ml)	2nd titre value (ml)	Average titre value (ml)
ean oil	2.0	2.00	2.00	2.00

CALCULATION OF PERCENTAGE YIELD OF METHYLESTER

The composition of reactants for the transesterification at 70^{0}C is given below:

Weight of methanol - 25g

Weight of SBO - 100g

Weight of KOH - 2g

So, total weight of reactant = (100 + 25 + 2) = 127g

SAMPLE A (REACTION TIME 25 minutes)

Wt of flat bottom flask = 180.5g

Wt of product + flask = 299.45g

Wt of product = (299.45 – 180.5) = 118.95g

Wt of glycerin = 48.20g

Wt of methyl ester = (118.95 – 48.20g)g = 70.75g

% yield of methyl ester before washing = $\frac{70.75 \times 100}{127}$ = 55.71%

of methyl ester after washing = 69.12g

% yield of methyl ester after washing = $\frac{69.12 \times 100}{127}$ = 54.43%

SAMPLE B (REACTION TIME 35 minutes)

Wt of conical flask = 163.2g

Wt of product + flask = 274.68g

Wt of product = (274.68 – 163.2)g = 111.48g

Wt of glycerine = 24.38g

Wt of methyl ester = (111.48 – 24.38)g = 87.10g

% yield of methyl ester before washing = $\frac{87.10 \times 100}{127}$ = 68.58%

Wt of methyl ester after washing = 84.98g

% yield of methyl ester after washing = $\frac{84.98 \times 100}{127}$ = 66.91%

SAMPLE C (REACTION TIME 45 minutes)

Wt of conical flask = 163.2g

Wt of product + flask = 274.45g

Wt of product = (274.45 – 163.2)g = 111.25g

Wt of glycerin = 27.45g

Wt of methyl ester = (111.25 – 27.45)g = 83.80g

% yield of methyl ester before washing $= \frac{83.80 \times 100}{127} = 65.98\%$

Wt of methyl ester after washing = 82.01g

% yield of methyl ester after washing $= \frac{82.01 \times 100}{127} = 64.57\%$

SAMPLE D (REACTION TIME 55 minutes)

Wt of flat bottom flask = 180.5g

Wt of product + flask = 293.30g

Wt of product = (293.30 – 180.5)g = 112.8g

Wt of glycerin = 33.75g

Wt of methyl ester = (112.8 – 33.75)g = 79.05g

% yield of methyl ester before washing $= \frac{79.05 \times 100}{127} = 62.24\%$

Wt of methyl ester after washing = 77.29g

% yield of methyl ester after washing $= \frac{77.29 \times 100}{127} = 60.86\%$