**Sudan University of Science & Technology**

**College of Engineering**

**School of Mechanical Engineering**

**Department of Power**

# PRODUCTION AND CHARACTERISTICS ANALYSIS OF BIODIESEL DERIVED FROM USED COOKING OIL

# إنتاج وتحليل خصائص الوقود الحيوي المنتج من مخلفات زيت الطهي

**A Project Submitted in Partial Fulfilment for the Requirements of B.Sc. Degree (Honors) in Mechanical Engineering (Power)**

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بسم الله الرحمن الرحيم

# قال تعالى:

﴿ لِّلَّهِ مَا فِي السَّمَاوَاتِ وَمَا فِي الأَرْضِ وَإِن تُبْدُواْ مَا فِي أَنفُسِكُمْ أَوْ تُخْفُوهُ
يُحَاسِبْكُم بِهِ اللّهُ فَيَغْفِرُ لِمَن يَشَاء وَيُعَذِّبُ مَن يَشَاء وَاللّهُ عَلَى كُلِّ شَيْءٍ قَدِيرٌ (284) آمَنَ
الرَّسُولُ بِمَا أُنزِلَ إِلَيْهِ مِن رَّبِّهِ وَالْمُؤْمِنُونَ كُلٌّ آمَنَ بِاللّهِ وَمَلآئِكَتِهِ وَكُتُبِهِ وَرُسُلِهِ لاَ
نُفَرِّقُ بَيْنَ أَحَدٍ مِّن رُّسُلِهِ وَقَالُواْ سَمِعْنَا وَأَطَعْنَا غُفْرَانَكَ رَبَّنَا وَإِلَيْكَ الْمَصِيرُ (285) لاَ
يُكَلِّفُ اللّهُ نَفْسًا إِلاَّ وُسْعَهَا لَهَا مَا كَسَبَتْ وَعَلَيْهَا مَا اكْتَسَبَتْ رَبَّنَا لاَ تُؤَاخِذْنَا إِن نَّسِينَا
أَوْ أَخْطَأْنَا رَبَّنَا وَلاَ تَحْمِلْ عَلَيْنَا إِصْرًا كَمَا حَمَلْتَهُ عَلَى الَّذِينَ مِن قَبْلِنَا رَبَّنَا وَلاَ تُحَمِّلْنَا مَا
لاَ طَاقَةَ لَنَا بِهِ وَاعْفُ عَنَّا وَاغْفِرْ لَنَا وَارْحَمْنَا أَنتَ مَوْلاَنَا فَانصُرْنَا عَلَى الْقَوْمِ الْكَافِرِينَ
(286) ﴾

صدق الله العظيم

(البقرة)

# DEDICATION

*To the people who are in our hearts, we dedicate this effort*

# ACKNOWLEDGMENT

First of all, our gratitude and thanks goes to ***ALLAH***, for helping us to finish this work. We wish to express our deepest gratitude and sincere appreciation to our project supervisor, Dr. ***Hazir Farouk Abdelraheem Elhaj*** for her continuous guidance

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# ABSTRACT

There is increasing interest on Used cooking oils as cost effective feedstock for biodiesel production. In this study, biodiesel was produced from used cooking oil (Acid value 1.09 mgKOH/g), collected from one of the restaurants at faculty of engineering, Sudan University of Science and Technology, using an alkaline base tranesterfication process. The maximum biodiesel yield obtained was 85.3% in combination of process parameters of 25% (v/v) methanol to oil ratio, 0.8% (w/w) of KOH, at maintained reaction time of 120 min, reaction temperature of 60 $^{o}$C and steering peed of 400 rpm. Optimizing the process parameters such as alcohol to oil molar ratio and catalyst concentration, the results show that the biodiesel yield decreases with the increase in methanol to oil molar ration, and reduces if the alkali catalyst is added above its optimum concentration. The basic physicochemical properties of the biodiesel produced were found to be in agreement within the ASTM D6751 specified limits. Mainly, these parameters were acid value, density, viscosity and flash point. Moreover, the calorific value was found to be 41.05 Mj/kg.

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# الملخص


هنالك إهتمام متزايد لإستخدام مخلفات زيت الطهي كمصدر منخفض التكلفة لانتاج الوقود الحيوي. في هذه الدراسة تم إنتاج الوقود الحيوي من مخلفات زيت الطهي بمستوي حمضية 1.09 mgKOH/g والذي تم جمعه من إحدي مطاعم كلية الهندسة، جامعة السودان للعلوم والتكنولوجيا وذلك عن طريق عملية الأسترة التحويلية بإستخدام كحافز قلوي أساسي. تم الحصول علي أعلي معدل إنتاج للوقود الحيوي 85.3% عند إستخدام 25% (حجم) ميثانول و 0.8% (وزن) هيدروكسيد بوتاسيوم وذلك بإجراء التفاعل عند درجة حرارة 60 درجة مئوية, 120 دقيقة و400 لفة في الدقيقة لسرعة الدوران. كذلك أظهرت النتائج أن معدل إنتاج الوقود الحيوي يتناقص مع زيادة نسبة الميثانول والمحفز القلوي فوق القيمة المثلي. كذلك تم إختبارالخصائص الأساسية للوقود الحيوي المنتج مثل الكثافة, اللزوجة, مستوي الحمضية ونقطة الوميض. وأظهرت النتائج أن جميع القيم المتحصل عليها لتلك الخصائص تتوافق في المدي المحدد لمواصفات الوقود الحيوي طبقا للمواصفة الأمريكية رقم ASTM D6751. إضافة الي ذلك، وجد أن القيمة الحرارية للوقود الحيوي المنتج تساوي 41.05 ميغا جول/كجم.

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| | | |
|---|---|---|
| ASTM | - | American Standard of Testing Materials |
| ASTM D6751 | - | American Standard Specification for biodiesel |
| AV | - | Acid Value |
| B0 | - | Petroleum Diesel |
| B100 | - | 100% UCO Biodiesel |
| CFPP | - | Cold filter plug point |
| DI | - | Direct Ignition |
| °C | - | Degree Celsius |
| CSOME | - | Cottonseed Oil Methyl Ester |
| CME | - | Canola Methyl Ester |
| CN | - | Cetane Number |
| CO | - | Carbon Monoxide |
| CO2 | - | Carbon Dioxide |
| DIN | - | German Institute for Standardization |
| EN14214 | - | European Standard Specification for biodiesel |
| FAME | - | Fatty Acid Methyl Ester |
| FN | - | Flow Number |
| FFA | - | Free Fatty Acid |
| GHG | - | Greenhouse Gases |
| HC | - | Hydrocarbons |
| IC | - | Internal Combustion |
| Kg | - | Kilogram |
| KOH | - | Potassium Hydroxide |
| MeOH | - | Methanol |
| Min | - | Minutes |
| NaOH | - | Sodium Hydroxide |
| NO | - | Nitrogen Oxide |
| NO2 | - | Nitrogen Dioxide |
| N2 | - | Nitrogen |
| NOx | - | Nitrogen Oxides |

| | | |
|---|---|---|
| $O_2$ | - | Oxygen |
| pmm | - | Parts Per million |
| SG | - | Specific Gravity |
| SCM | - | Supercritical Methanol |
| SO | - | Sulphur Oxide |
| SO2 | - | Sulphur Dioxide |
| UCO | - | Used Cooking Oil |
| UHC | - | Unburned Hydrocarbons |

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# CHAPTER ONE

# INTRODUCTION

## 1.1 General Overview

Fossil fuel resources are being used up, and the scarcity of known petroleum reserves will make renewable energy resources more attractive. As one of the most promising fossil fuel alternatives, biofuels have been given priority for development in many countries around the world, with their potential advantages of greatly reduced greenhouse gas (GHG) emissions, and they are generally seen as more environmentally sustainable. Researchers found that the most feasible way to meet this growing demand is by utilizing alternative fuels such as biodiesel which can be produced from vegetable oils or animal fats [1].

Biodiesel gained a lot of interest worldwide, not just because it's a renewable fuel source; it's also an economically feasible one and can be produced in an environmentally friendly way. In most of the developed countries, biodiesel is produced from edible oils from plants such as Soybean, Rapeseed, Sunflower, Cotton and Peanuts. Non-edible oils from plants such as Castor, Pongamia, Jatropha and, Karanja and animal fats are increasingly seen as better options for large scale production of biodiesel because edible oils are already in high demand and are too expensive to be economic feedstocks for making biodiesel [2].

In 2009 European Union (EU) issued a directive for renewable energy which requires that biofuels must supply at least 10% of transportation fuels by 2020. The US Renewable Fuel Standard (RFS) mandates the minimum use of 36 billion gallons of ethanol by 2022. Malaysia, Indonesia, Thailand, and many other countries applied the concept of B5 and B10 (5 and 10% of biodiesel blended with fossil diesel) and developed their own roadmaps for biofuel utilization over the coming decades [3].

### 1.2 Project Background

Sudan is one of the largest countries in Africa with a total area of 1,882,000 $km^2$, and it has a population of about 33.5 million (growth rate of 2.84% per year) [4]. Sudan's energy demand has significantly grown through the past 20 years from 6.8 million tonnes of oil-equivalent (MTOE) to more than 11.MOTE [5]. Crude oil is the main source of fossil energy and its consumption has rapidly increased in recent years owing to the increase in the country's economic and population growth. The transportation sector is the largest user of the refined fuel products, consuming about 61% from the total crude oil volume presently produced. The major fuel form used is diesel. It represents about 50% of the total fuel consumption, whilst gasoline and jet A1 represent 23% [5, 6].

The secession of South Sudan in 2011 with most of the productive oil fields has left Sudan with sharply reduced oil volumes to export (previously the main source of export revenue). Petroleum product subsidies accounted for about 75% of tax revenues in 2011 and have been on the rise as a consequence of this secession and the related rise in petroleum products in the international market. Imports of oil products account for about 20 percent of Sudan's total fuel consumption. Diesel, jet fuel, and LPG are imported to cover the shortfall in domestic production. In 2011, the volume of imports accounted for about 25, 20 and 50 percent respectively of the total consumption of these products, but the share of imports are likely to rise following the secession of South Sudan and the recent events in crude oil production sites [7].

Sudan, while a developing country, has much obvious positive strength that have the potential, if harnessed, for providing great economic benefits. In this context, Sudan's potential for the development of an industrial scale biofuels supply needs to be assessed. These biofuels development actually fits within the development of bioenergy in Sudan – covering the use of biomass of all types, including forest and agricultural residues and biowastes from industry and urban centres, to produce energy. This energy produced may be in the form of heat, electricity, liquid and gaseous transport fuels, or gas or liquid fuels for use in industry and households.

## 1.3 Problem Statement

Development of a large-scale biodiesel production industry in Sudan with aim to reduce dependency on petroleum-sourced diesel fuel requires proper management as well as solution of many critical issues. One significant problem in the production of biodiesel is the availability and cost of feedstock. Optimizing the amounts of the chemicals needed for the production process of biodiesel along with other parameters indicate that real savings can be made in terms of time and chemical cost, which can significantly improve operating economics. However, the use of bidiosel in the transportation system requires meeting the international standards of fuel specification. Thus; the properties of the biodiesel produced and its blend need to be tested to ensure they are within the limits of international standards.

## 1.4 Project Goals and Aim

The aim of this project is to assess the potential of biodiesel production from used cooking oil available from restaurants. The specific objectives are summarized in:

- To produce biodiesel from used cooking oil (UCO) in lab scale.
- To optimize the process parameters of biodiesel production from used cooking oil by using the conventional transesterification method.
- To characterize the basic physicochemical properties of the UCO biodiesel and compare with the ASTM D6751 specification limits.

## 1.5 The Project Scope

The scope of this project is limited to optimizing the main parameters of biodiesel production process from used cooking oil via transesterification reaction. This includes the alcohol-to-oil molar ratio, and concentration of catalyst. The scope also included determination of physical and chemical fuel properties of the produced biodiesel such as acid value, kinematic viscosity, density, calorific value, and flash point.

## 1.6 Project Significance

Sudan has a steadily increasing requirement for fossil diesel for both transport and electricity generation in regional areas beyond the national electricity grid. There is a growing awareness that biodiesel produced in Sudan could be blended with the available fossil diesel, resulting in many positive economic, social and environmental benefits. However, using non-food biomass feedstock could help to achieve sustainable, very low emission and cost-effective bio fuels production through successful development of advanced bio fuels technologies. Use of low cost feed stocks such as used cooking oil (UCO) will increase the competency of biodiesel against petroleum diesel. Moreover, production of biodiesel from used cooking oil is providing alternative opportunity for the management of fats and oils that get put into the city sewage system.

## 1.7 Project Layout

This project is divided into five chapters. Chapter 1 discusses the background of the research, the problem statement and declares the objectives as well as the scope of this study and the significance. Literature review is presented in chapter 2, mainly drawing similar studies by other researchers. The sources are journals, articles and other established sources in internet. Researches done by others are compared in this chapter and several reviews are included. Chapter 3 outlines detailed methods in attaining the objectives of this research including a detailed explanation of the material and process used. The results obtained are analyzed and discussed in chapter 4. Chapter 5 conclude the findings of this report and recommendations are suggested for future research.

# CHAPTER TWO

# LITERATURE REVIEW

## 2.1 Introduction

This chapter presents a detailed literature review with aim to provide a general overview of the potential of uses of biodiesel as a fuel blended with diesel in diesel engine. It includes a detailed review of biodiesel feedstocks, biodiesel production processes, biodiesel standards and policies, properties and qualities of biodiesel and the benefits of using biodiesel. Moreover, this chapter reviews the previous work in the research topic area, and reveals the limitations which are related to the biodiesel industry worldwide.

## 2.2 The Energy Profile Worldwide

During the last Century, the consumption of energy has increased rapidly due to the change in the life style and significant growth population .This increase of energy demand has been supplied by the use of fossil resources. The demand for transport fuel has been increasing and expectations are that this trend will stay unchanged for the coming decades. In the fact, with a worldwide increasing number of vehicles and rising demand of emerging economies, demand will probably rise even harder .Transport fuel demand is traditionally satisfied by fossil fuel demand. However, resources of this fuel are running out, prices of fossil fuel are expected to rise and combustion of fossil fuels has detrimental affecting the climate [8].

The increasing awareness of the depletion of fossil fuel resources and the environmental benefits of biodiesel fuel has made it more attractive in recent times. Its primary advantages deal with it being one of the most renewable fuels currently available and it also non-toxic and biodegradable. It can also be used directly in most diesel engines without requiring extensive engine modifications. However, the cost of biodiesel is the major hurdle to its commercialization in comparison to petroleum-based diesel fuel the high cost is primarily due to the raw material, mostly neat vegetable oil. Used cooking oil is one of the economical sources for biodiesel production. However, the products formed during frying, can affect the transesterification reaction and the biodiesel properties [9].

## 2.3 Definition of biodiesel

Biodiesel is defined as the fatty acid alkyl monoesters derived from renewable feedstocks such as vegetable oils and animal fats [10]. It is a fuel resulting from mixing of vegetable oils with alcohol in the presence of a catalyst. Biodiesel is a biodegradable, non-toxic, almost sulfur-free and non-aromatic, environmentally-friendly alternative diesel fuel. When a diesel engine is operated with biodiesel, exhaust emissions decrease approximately 20% in CO, 30% in HC, 40% in particulate matter (PM), and 50% in soot emission, compared to fossil-diesel fuel. In contrast to these decreases, its nitrous oxides (NOx) emissions increase about 10–15 %. However, the high NOx problem can be overcome by retarding the injection timing. The lubrication effect or lubricity property of biodiesel is much better than that of diesel fuel and especially of low-sulfur diesel fuel [11].

## 2.4 Biodiesel Feedstock

The feedstock for biodiesel production is usually chosen according to the availability and cost in each region or country. Worldwide, there are more than 350 oil-bearing crops identified as potential sources for biodiesel production. The availability of feedstocks for producing biodiesel depends on some factors like the geographical locations and agricultural practices of the country. The more commonly-used feedstocks are soybean, palm, sunflower, safflower, cottonseed, rapeseed and peanut oils [3]. Vegetable oils have the potential to be considered as a possible alternative for petroleum diesel, as they possess fuel properties similar to that of diesel. In general these biodiesel feedstocks can be divided into four main categories; edible vegetable oils, non-edible vegetable oils, waste or recycled oils and animals' fats, and there are other resources available [12]. Some of these feedstocks are presented in Table 2.1.

**Table 2.1: Main biodiesel feedstock [3]**

| Edible oils | Non-edible oils | Animal Fats | Other Sources |
|---|---|---|---|
| Soybeans (Glycine max) | Jatropha curcas | Pork lard | Bacteria |
| Rapeseed (Brassica napus L.) | Mahua (Madhuca indica) | Beef tallow | Algae (Cyanobacteria) |
| Safflower | Pongamia (Pongamia pinnata) | Poultry Fat | Microalgae (Chlorellavulgaris) |
| Rice bran oil (Oryza sativum) | Camelina (Camelina Sativa) | Fish oil | Tarpenes |
| Barley | Cotton seed (Gossypium hirsutum) | Chicken fat | poplar |
| Sesame (Sesamum indicum L.) | Cumaru | | switchgrass |
| Groundnut | Cynara cardunculus | | miscanthus |
| Sorghum | Abutilon muticum | | Latexes |
| Wheat | Neem (Azadirachta indica) | | Fungi |
| Corn | Jojoba (Simmondsia chinensis) | | |
| Coconut | Passion seed (Passiflora edulis) | | |
| Canola | Moringa (Moringa oleifera) | | |
| Peanut | Tobacco seed | | |
| Palm and palm kernel (Elaeis guineensis) | rubber seed tree (Hevca brasiliensis) | | |
| Sunflower (Helianthus annuus) | Salmon oil | | |

Most of the oils used for the production of biodiesel by various researchers were soybean, sunflower, safflower, cotton, rapeseed and palm oil. The production of these has been well established in many countries around the world such as Malaysia, USA and Germany. Currently, more than 95% of the world biodiesel is produced from edible oils such as rapeseed (84%), sunflower oil (13%), palm oil (1%), soybean oil and others (2%) [3]. However the edible oil are in increasing demand, their use is rising and prices increased dramatically during the 10 last years with consequent effect on the biodiesel production industries [13].

One of the possible solutions to reduce the utilization of the edible oil for biodiesel production is by exploiting non-edible oils, waste oils or animal fats. These resources are gaining worldwide attention because they are easily available in many parts of the world. Several studies have been done on the production of biodiesel from waste oils or animal fats such as beef tallow, chicken fat, pork lard and fish oil. They describe the feasibility of making quality biodiesel from this feedstock while identifying the problems with the free fatty acids present in the raw materials. The advantages of using waste cooking oils to produce biodiesel are the potentially lower feedstock cost and prevention of environmental pollution (due to the high cost of disposal, many individuals dispose waste cooking oils directly to the environment especially in rural area [14]. The current feedstocks used for biodiesel production are shown in Table 2.2.

**Table 2.2: Current Potential Feedstocks for Biodiesel Worldwide [10]**

| Country | Feedstock |
|---|---|
| USA | Soybeans / waste oil / Peanut |
| Canada | Rapeseed / Animal fat / Soybeans / Yellow grease and Tallow / Mustard |
| Mexico | Animal fat/ Waste oil |
| Germany | Rapeseed |
| Italy | Rapeseed / Sunflower |
| France | Rapeseed / Sunflower |
| Spain | Linseed oil / Sunflower |
| Greece | Cottonseed |
| UK | Rapeseed / Waste cooking oil |
| Sweden | Rapeseed |
| Ireland | frying oil / animal fats |
| India | Jatropha / Pongamia pinnata (karanja) / Soybean / Rapeseed / Sunflower / Peanut |
| Malaysia | Palm oil |
| Indonesia | Palm oil / Jatropha/ Coconut |
| Singapore | Palm oil |
| Philippines | Coconut / Jatropha |
| Thailand | Palm / Jatropha / Coconut |
| China | Jatropha / Waste cooking oil / Rapeseed |
| Brazil | Soybeans / Palm oil / Castor / Cotton oil |
| Argentina | Soybeans |
| Japan | Waste cooking oil |
| New Zealand | Waste cooking oil / Tallow |

### 2.4.1 Used Cooking Oil

Used cooking oil is a possible low cost feedstock for biodiesel production. The main sources of used cooking oil are household, restaurants, hotels, catering, and camps. This collected wasted cooking oil has variety of qualities and posses properties different from neat vegetable oil [15]. Thus, neat vegetable oil is the best starting material compare to waste cooking oil because of the conversion of triylglycerides to fatty acid methyl ester is high and the reaction time is relatively short. Waste cooking oil contains higher free fatty acid than neat vegetable oil. Researchers reveal stated that the high temperature of typical cooking processes and water from the foods accelerate the hydrolysis of triglycerides and increase the free fatty acid content in the oil [16]. However, the properties of biodiesel produced from used cooking are nearly to that of conventional diesel fuel as shown in Table 2.3.

**Table 2.3: Comparison of fuel properties for a UCO and diesel fuel [17]**

| **Characteristics** | **UCO Biodiesel** | **Diesel Fuel** | **ASTM D6751Limits** |
|---|---|---|---|
| Density at 15ºC ($kg/m^3$) | 875 | 820-860 | 880 |
| Specific gravity at 15.5°C | 0.893 | 0.825 | - |
| Kinematic Viscosity at 40°C($mm^2/s$) | 3.658 | 1.81 | 1.9-6.0 |
| Flash point (°C) | 160 | 53 | 130 minimum |
| Calorific value (MJ/kg) | 39.77 | 42.35 | Report |
| Cetane index | 50.54 | 46.21 | - |
| Cetane Number | 55.45-56.10 | 46 | 47 minimum |
| Pour Point (°C) | -4 to -1 | -20 | -15 to -16 |
| Distillation temperature (°C) | | | |
| 10% recovery temperature | 340 | 165 | 360 |
| 50% recovery temperature | 345 | 265 | |
| 90% recovery temperature | 320 | 346 | |

## 2.5 Biodiesel Production Processes:

The cost of biodiesel is mainly affected by the cost of feedstock, as shown in the Figure 2.1. Feedstock cost represents about 75% of the overall biodiesel production cost, and the rest - 25% - is the combination of the chemicals used, equipment depreciation, labour and other overhead costs. This indicates that selecting of the feed stock should meet two significant requirements: low production cost/unit volume and large production scale [3, 14].

There are four main methods of biodiesel production processes: blending, micro-emulsification, cracking and transesterification. Among these four ways, the most commonly preferred process to reduce the high viscosity of reaction is the transesterification process [18].

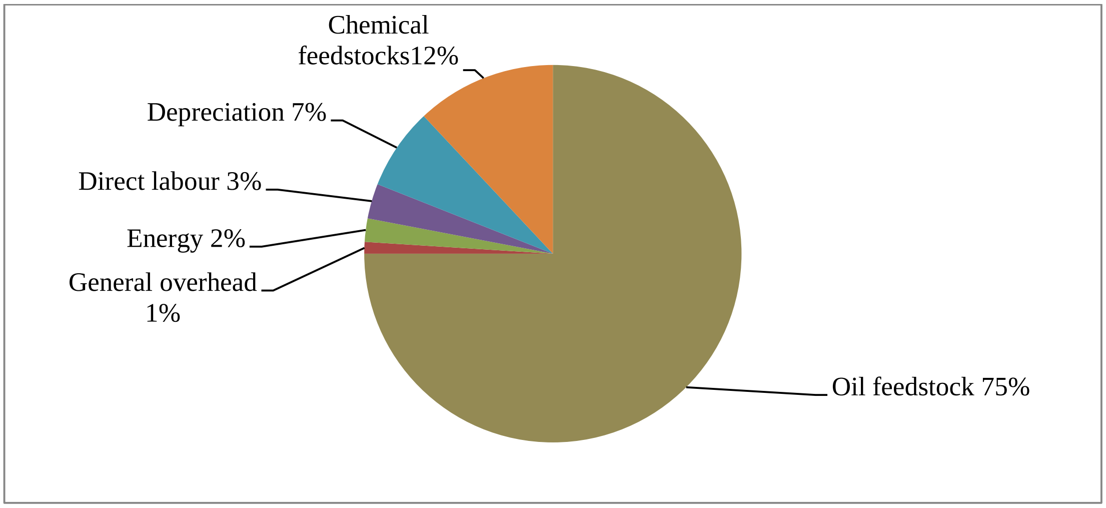


**Figure 2.1: General cost breakdown for production of biodiesel [14]**

### 2.5.1 Blending Process

Blending is the direct mixing of vegetable oil with diesel fuel. However, different chemical nature of biodiesel and diesel may cause differences in the physicochemical properties that will affect the engine performance and pollutant emissions produced [19].

### 2.5.2 Micro-emulsification

Micro-emulsification is another approach to reduce the viscosity of vegetable oils A micro-emulsion is defined as a colloidal equilibrium dispersion of optically isotropic fluid microstructure with dimensions in the 1–150 nm range, formed spontaneously from two normally immiscible liquids. They can improve spray characteristics by explosive vaporization of the low boiling constituents in the micelles [20].

### 2.5.3 Cracking Process

Thermal cracking is the conversion of one substance into another by means of heat in the presence of a catalyst. The thermal cracking material can be vegetable oil, animal fats, natural fatty acids or methyl esters of fatty acids. The thermal cracking of fats has been investigated for more than 100 years, especially in those areas of the world where there is lack of deposit of petroleum. Many investigators have studied thermal cracking of triglycerides to obtain products suitable for diesel engines. Thermal decompositions of triglycerides produce alkadines, alkenes, alkaline, aromatics, and carboxylic acids [21].

### 2.5.4 Transesterification Process

Transesterification is regarded as the best method among the alternative biodiesel production methods, due to its low cost and simplicity. Transesterification is the normal name given to the chemical reaction between triglycerides and alcohol to form an ester and glycerol with or without the presence of catalyst. This process is also called alcoholysis of ester. Generally, the reaction time and yield of transesterification can be enhanced by adding catalyst. The reaction can be represented as in Figure 2.2, where the mechanism of transesterification consists of three reversible reactions, in which the triglycerides are converted into diglycerides followed by conversion to monoglycerides, and then lastly converted into glycerol, producing one ester at each conversion stage [3]. Transesterification can be categorized into two main types, which are the catalytic and non-catalytic methods. Catalytic transesterification includes alkaline-catalyzed reaction, acid- catalyzed reaction and enzyme-catalyzed reaction.

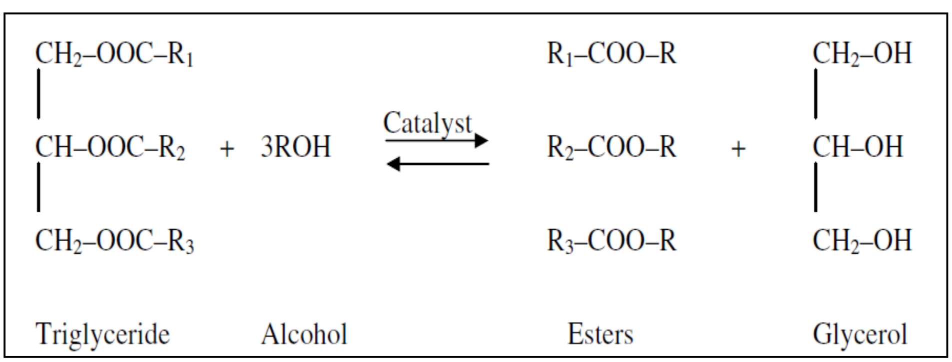


**Figure 2.2: Stoichiometric Transesterification of Triglycerides [3, 17]**

### 2.5.4.1 Parameters Affecting Transesterification Reaction

The transesterification process is influenced by various parameters during the reaction. Some of the imperative factors are reaction temperature, molar ratio of alcohol to oil, concentration of catalyst and reaction time.

#### i. Reaction Temperature

The reaction temperature is limited by the boiling point of the alcohol as temperature beyond the boiling point of the alcohol will vaporize the alcohol, causing lower transesterification yield [18].

#### ii. Molar Ratio of Alcohol to Oil

Stoichiometric transesterification of triglycerides requires ratio of three moles of alcohol and one mole of triglyceride to produce three moles of fatty acid alkyl ester and one mole of glycerol. Normally, excess alcohol is used to transesterify the oil completely to the ester. Base-catalyzed transesterification of oil with FFA less than 1% requires molar ratio of methanol to oil of 6:1. Nevertheless, transesterification of oil with high FFA content by using acid catalyst will require molar ratio up to 24:1 [18].

#### iii. Concentration of Catalyst

Catalyst has an optimum range of concentration that will produce highest yield in transesterification process. Sulphuric acid is a common catalyst that works best in the range of 1.5-2.25 M concentration. Base catalysts, on the other hand, are more effective than acid catalyst as they react faster. High conversion rate of above 90 % for sodium hydroxide occurs at 1.0 to 1.4 % (w/w), whereas that for potassium hydroxide occurs at 0.55 to 2.0 % (w/w) [18].

#### iv. Reaction Time

The conversion rate of oil to alkyl ester increases with time until it reaches maximum yield rate. Usually, optimum reaction time of base-catalyzed reaction is shorter that of acid catalyzed reaction. However, prolonged reaction time will cause backward reaction of transesterification, forming soap [18].

Moreover, there are other variables affecting transesterification process such as free fatty acid and water contents, mixing intensity, and effect of using organic co-solvents. Examples of alkaline- catalyzed process of vegetable oil under optimized reaction conditions are summarized in Table 2.4.

**Table 2.4: Optimized parameters for Alkaline- catalyzed process** (H.Farouk, *et al*. 2014)

| Alkaline- catalyzed Process | | | |
|---|---|---|---|
| **Catalyst amount** | **MeOH to oil ratio** | **Reaction condition** | **Biodiesel yield** |
| KOH, 2.0 wt% | 9:1 mol | 120 min, 60 ℃ | 90-95 % |
| NaOH, 1.4%w/w | 24% w/w | 120 min, 65±0.5 ℃ | 90 % |
| NaOH, 1.0%w/w | 30% v/v | 180 min, 50 ℃ | 90.1 % |
| KOH, 0.55%w/w | 16% v/v | 24 min, 60 ℃ | 99 % |
| KOH,0.55%w/w | 5.41:1 mol | 90 min, 60 ℃ | 95.3% |
| KOH, 8gm/l of oil | 11%v/v | 180 min, 66 ℃ | 93% |
| KOH, 1.0%w/w | 6:1 mol | 30 min, 60 ±0.3℃ | 86.2% |
| KOH, 0.55%w/w | 5.41:1 mol | 90 min, 60 ℃ | 93% |
| KOH, 1.0%w/w | 15%w/w | 40 min, 30 ℃ | 96% |
| KOH, 0.5%w/w | 6:1 mol | 90 min, 60 ℃ | 83% |

## 2.6 Properties and Qualities of Biodiesel

The successful commercialization of biodiesel around the world has led to the development of various national standards which describe the minimum requirement of important biodiesel properties to ensure its suitability for use in diesel engines [21]. The properties of biodiesel are characterized by physicochemical properties. Some of these properties include; cetane number, density ($kg/m^3$), viscosity ($mm^{2/}s$), cloud and pour points (˚C), flash point (˚C), acid value (mg KOH per g-oil), ash content (%), copper corrosion, carbon residue, water content and sediment, sulphur content, glycerine (% m/m), phosphorus (mg/kg) and oxidation stability. The physical and chemical fuel properties of biodiesel basically depend on the type of feedstock and their fatty acids composition [22]. The following section gives explanations of some general properties of biodiesel.

### 2.6.1 Acid Value

The acid number or neutralization number is a measure of the amount of carboxylic acid groups in a chemical compound, such as a fatty acid, or in a mixture of compounds. Free fatty acids (FFAs) are the saturated or unsaturated monocarboxylic acids that occur naturally in fats, oils or greases but are not attached to glycerol backbones. Fatty acids vary in carbon chain length and in the number of unsaturated bonds (double bonds). Acid value is expressed as mg KOH required for neutralizing 1 gm of FAME. Higher acid content can cause severe corrosion in fuel supply system of an engine. The acid value is determined using the ASTM D664 and EN 14104. Both standards approved a maximum acid value for biodiesel of 0.50 mg KOH/g [23].

### 2.6.2 Density

The density of fuel has some effect on the break-up of the fuel injected into the cylinder. In addition, more fuel is injected by mass as the fuel density increases. Higher density of biodiesel allows use of splash blending by adding biodiesel on top of diesel fuel making biodiesel blends or else the fuel will not mix. ASTM Standard D1298 and EN ISO 3675/12185 test method are used to measure the density of the biodiesel. According to these standards, density should be tested at the temperature reference of 15 $^{\circ}$C [24].

### 2.6.3 Kinematic Viscosity

Viscosity is defined as the resistance of liquid to flow. The viscosity of an engine fuel is one of the most critical fuel features. It plays a dominant role in the fuel spray, mixture formation and combustion process. The high viscosity interferes with the injection process and leads to insufficient fuel atomization. It was also stated that higher viscosity leads to poorer atomization of the fuel spray, which affects accuracy of the operation of fuel injectors. However the lower the viscosity of the biodiesel, the easier it is to pump, and it atomizes better and achieves finer droplets. The maximum allowable limit according to ASTM D445 ranges are (1.9-6.0 $mm^2s^{-1}$) and (3.5-5.0 $mm^2s^{-1}$) in EN ISO 3104 [24].

### 2.6.4 Cetane Number

Satisfactory diesel combustion demands self-ignition of the fuel as it is sprayed near TDC into the hot swirling compressed cylinder gas. Long ignition delay is not acceptable as it leads to knock The cetane number is the prime indicator of fuel ignition quality and the opposite of octane number of a gasoline fuel. It can be defined as the measure of knock tendency of diesel fuel. Therefore, the cetane number of the substitute fuel should be high enough, that the knock tendency of the fuel is minimal. Satisfactory fuels must have a cetane number between 40 and 60. Cetane number increases with increasing chain length of fatty acids and increasing saturation. A higher CN indicates shorter time between the ignition and the initiation of fuel injection into the combustion chamber. Fuels with low CN tend to cause diesel knocking and show increased gaseous and particulate exhaust emissions (PM) due to incomplete combustion. Moreover, excessive engine deposits are reported. Generally, biodiesel has higher CN than conventional diesel fuel, which results in higher combustion efficiency. The CN of diesel, specified by ASTM D613 is 47 min and EN ISO 5165 is 51.0 min [25].

### 2.6.5 Heating Value

Although the diesel combustion chamber system can accept wide variations in heating value, practical systems are only suitable when calorific value of the fuel is high. This helps to reduce the quality of fuel handled and maximizes the equipment operating range. It is always desirable for the vegetable oils to have a calorific value nearer to that of conventional diesel [26].

### 2.6.6 Flash Point

The flash point is the temperature at which the fuel will start to burn when it comes to contact with flame or spark. It is an important temperature from the safety point of view during storage and transportation. However, a fuel with high flash point may cause carbon deposits in the combustion chamber. Flash point of biodiesel is higher than the petroleum based diesel fuel. Thus in storage, biodiesel and its blends are safer than conventional diesel. The limit of flash point ranges in ASTM D93 is 93 ˚C and in EN ISO 3679 is 120 ˚C [27].

### 2.6.7 Cloud Point and Pour Point

Cold-flow quality of a fuel is determined by the cloud point and pour point. The cloud point is defined as the temperatures at which when small solid crystal are initially observed as the fuel is being cooled. This is the most conservative measurements of cold flows properties and most fuel can be used without problems below the cloud point but above the cold filter plug point (CFPP). The pour point is the lowest temperature at which the fuel will still flow and can be pumped. All biodiesel regardless of its source have higher cloud and pour points than that of diesel fuel, and this poor cold flow property is one of the most critical obstacles against the widespread biodiesel usage, and particularly of higher blends or of pure biodiesel. In general, biodiesel has higher CP and PP than diesel fuel. Cloud and pour points are measured using ASTM D2500 EN ISO 23015 and D97 procedures [28].

### 2.6.8 Oxidation Stability

The oxidation of biodiesel fuel is one of the major factors that helps assess the quality of biodiesel. Oxidation stability is an indication of the degree of oxidation, potential reactivity with air, and can determine the need for antioxidants. Oxidation occurs due to the presence of unsaturated fatty acid chains and the double bond in the parent molecule, which immediately react with the oxygen as soon as it is being exposed to air [28]. The chemical composition of biodiesel fuels make it more susceptible to oxidative degradation than fossil diesel fuel. The Rancimat method (EN ISO 14112) is listed as the oxidative stability specification in ASTM D6751 and EN 14214. A minimum IP (110 °C) of 3 h is required for ASTM D6751, whereas a more stringent limit of 6 hours or greater is specified in EN 14214 [29].

## 2.7 Biodiesel Specifications and policy

Different countries having different mandates for biodiesel blends .In the EU it is a legal undertaking by each country to achieve an overall 10% biofuel use by 2020 .The Federated States in place are Thailand B5, Argentina B7, Belgium B4, Columbia B20, Indonesia B2.5, Malaysia B5 (B7 last month), Philippines B2, Peru B5, Sth Korea B2.5, Spain B7, This is usually as a blending mandate or an overall biofuel use mandate. In the USA it is a state by state biofuel use or blending authorization (some states are up to B10. Some blending targets are raising to B20 in step with state production targets). The USA has an overall biofuel use target of over 100 billion liters by 2022. Thailand target is 6 million liters a day of biodiesel by 2022. Moreover many countries have additional national targets. This include Italy which targets 2,899 ktoe biodiesel uses in transport by 2020, Denmark targets 100% of transport by biofuels and electricity by 2050, France targets10.5% by 2020, Spain targets 2,313 ktoe of biodiesel uses by 2020, and Sweden targets 100%biofuels by 2030 [30].

The properties and qualities of biodiesel must adhere with the international biodiesel standard specifications. These specifications include the American Standards for Testing Materials (ASTM 6751-3) or the European Union (EN 14214) Standards. However, there are some other standards available globally such as in Germany (DIN 51606), Austria (ON) and Czech republic (CSN). Biodiesel (B100) specifications ASTM D6751 and EN 14214 standards are shown in Table 2.5 [30]

**Table 2.5:** ASTM D975, ASTM 6751 and EN 14214 specifications of diesel and biodiesel

| Property specification | Unit | Diesel ASTM D975 | | Biodiesel | | | |
|---|---|---|---|---|---|---|---|
| | | | | ASTM D6751 | | EN 14214 | |
| | | Test method | Limits | Limits | Test Method | Limits | Test Method |
| Flash point | ˚C | ASTM D975 | 60 to 80 | 130 minimum | ASTM D93 | 101 minimum | EN ISO 3679 |
| Cloud point | ˚C | ASTM D975 | -15 to 5 | -3 to -12 | ASTM 2500 | - | - |
| Pour point | ˚C | ASTM D975 | -35 to -15 | -15 to -16 | ASTM 97 | - | - |
| Cold filter plugging point (CFFF) | ˚C | EN 590 | -8 | Max +5 | ASTM D6371 | - | EN 14214 |
| Cetane number | | ASTM D4737, EN 590 | 46 | 47 minimum | ASTM D613 | 51 minimum | EN ISO 5165 |
| Density at 15ºC | $kg/m^3$ | ASTM D1298 | 820-860 | 880 | D1298 | 860-900 | EN ISO 3675/12185 |
| Kinematic viscosity at 40ºC | $mm^2/s$ | ASTM D445 | 2.0 to 4.5 | 1.9-6.0 | ASTM D445 | 3.5-5.0 | EN ISO 3104 |
| Iodine number | g $I_2$/100 g | - | - | - | - | 120 | EN 14111 |
| Acid number | mg KOH/g | - | - | 0.5 maximum | ASTM D664 | 0.5 maximum | EN 14104 |
| Oxidation stability | | ASTM D2274 | 25 mg/L maximum | - | - | 3 hours minimum | EN 14112 |
| Carbon residue | % m/m | ASTM D4530 | 0.2 maximum | 0.050 maximum | ASTM D4530 | 0.3 maximum | EN ISO 10370 |
| Copper corrosion | | ASTM D130 | Class 1 max | No.3 maximum | ASTM D130 | class 1 | EN ISO 2160 |
| Distillation temperature | ˚C | ASTM D86 | 370 maximum | 360 | ASTM D1160 | - | - |
| Lubricity (HFRR) | | IP 450 | 0.460 mm (max) | 520 maximum | ASTM D6079 | - | - |
| Ash content | %mass | ASTM D482 | 100 maximum | - | - | - | - |
| Water and sediment | | ASTM D2709 | 0.05 maximum | 0.005 %v maximum | ASTM D2709 | 500 mg/kg maximum | EN ISO 12937 |

| Property specification | Unit | Diesel ASTM D975 | | Biodiesel | | | |
|---|---|---|---|---|---|---|---|
| | | | | ASTM D6751 | | EN 14214 | |
| | | Test method | Limits | Limits | Test Method | Limits | Test Method |
| Moisture | wt % | - | - | - | - | 0.05 maximum | EN 1412 |
| Monoglycerides | %mass | - | - | - | - | 0.8 maximum | EN 14105 |
| Diglycerides | %mass | - | - | - | - | 0.2 maximum | EN 14105 |
| Triglycerides | %mass | - | - | - | - | 0.2 maximum | EN 14106 |
| Free glycerine | %mass | - | - | 0.02 maximum | ASTM D6584 | 0.02 maximum | EN 1405/14016 |
| Total glycerine | %mass | - | - | 0.24 | ASTM D6548 | 0.25 | EN 14105 |
| Phosphorous | %mass | - | - | 0.001 maximum | ASTM D4951 | 0.001 maximum | EN 14107 |
| Calcium | %mass | - | - | - | - | - | - |
| Magnesium | %mass | - | - | - | - | - | - |
| Sulphur (S 10grade) | | ASTM D5453 | 10 maximum | - | - | - | - |
| Sulphur (S 15 grade) | ppm | - | - | 150 maximum | ASTM D5453 | - | - |
| Sulphur (S 50 grade) | ppm | ASTM D5453 | 50 maximum | - | - | - | - |
| Sulphur (S 500 grade) | ppm | ASTM D5453 | 500 maximum | 500 maximum | ASTM D5453 | - | - |
| Carbon | wt% | ASTM D975 | 87 | 77 | ASTM PS 121 | - | - |
| Hydrogen | wt% | ASTM D975 | 13 | 12 | ASTM PS 121 | - | - |
| Oxygen | wt% | ASTM D975 | 0 | 11 | ASTM PS 121 | - | - |
| Sodium and potassium | mg/kg | - | - | - | - | 5 maximum | EN 14108, EN 14109 |
| Methanol content | %mass | - | - | - | - | 0.2 maximum | EN 14110 |
| Ester content | %mass | - | - | - | - | 96.5 minimum | EN 14103 |
| Linolenic acid methyl ester | %mass | - | - | - | - | 12 maximum | EN 14103 |
| Polyunsaturated (³ 4 double bonds) methyl esters | %mass | - | - | - | - | 1 maximum | EN 14104 |
| Alkaline metals (Na+ K) | mg/kg | - | - | - | - | 5 maximum | EN 14108, EN 14109, EN 14538 |

| Property specification | Unit | Diesel ASTM D975 | | Biodiesel | | | |
|---|---|---|---|---|---|---|---|
| | | | | ASTM D6751 | | EN 14214 | |
| | | Test method | Limits | Limits | Test Method | Limits | Test Method |
| Alkaline metals (Ca+ Mg) | mg/kg | - | - | - | - | 5 maximum | EN 14538 |
| BOCLE scuff | g | ASTM D975 | 2000 to 5000 | >7000 | ASTM PS 121 | - | - |
| Conductivity @ ambient temp | pS/m | ASTM D2624 | 50 m minimum @ambient temp (all diesel held by a terminal or refinery for sale or distribution) | - | - | - | - |
| Polyaromatic hydrocarbons (PAHs) | % m/m | IP391 | 11 maximum | - | - | - | - |
| Total contamination | mg/kg | - | - | 24 | ASTM D 5452 | 24 | EN 12662 |

## 2.8 Benefits of biodiesel uses

Biodiesel has several significant advantages over conventional diesel fuel. Biodiesel is available from many renewable sources which make it an environmentally friendly source of energy. It is easy to store and safer to transport compared to conventional diesel fuel, and can be used alone or blended in diesel engines.

Biodiesel as alternative fuel for petroleum can significantly reduce global warming emission gases such as carbon dioxide (CO2). The biofuels producing plants such as soybeans or palm oils breathe the CO2 from the air when they grow. Meanwhile the biodiesel during combustion releases CO2 and other emissions to atmosphere. This cycle ideally does not add the net CO2 concentration in the air because the next soybean or palm oil crop will reuse the CO2 in order to grow [31].

Biodiesel has higher combustion efficiency due to better homogeneity of oxygen/fuel during combustion and it will improve the lifespan of diesel engines owing to better lubricating properties as compared to diesel fuel. The combustion of biodiesel generally can reduce emissions and it charted 90% drop in total unburned hydrocarbons, and a 75–90% drop in polycyclic aromatic hydrocarbons. Low sulphur content of biodiesel apparently reduces the SO2 emissions [31].

Biodiesel is classified as biodegradable, meaning that it is easy to decompose. It is safer to transport and safe to handle because its biodegradability is the same as sugar as and ten times less toxic than table salt. Most importantly it is less flammable. Biodiesel is less flammable compared to regular petroleum diesel. This is because biofuels has a higher flash point compared to petroleum diesel. Theoretically, biodiesel has flash point of around 160$^{o}$C whereas petroleum diesel has much lower flash point of near 55$^{o}$C. This characteristic makes a vehicle fueled with neat biodiesel far safer in an accident than one powered by petroleum diesel [32].

If the features of biofuels are looked into profoundly, they are shown as able to create new markets for agricultural products that will be able to boost intense development in these sectors. It is because biodiesel feedstocks are mainly produced from agricultural or horticultural crops. For the developing countries where up to two-thirds of the economic growth is concentrated around agriculture, biodiesel would be an economical boost in the near future. At the national level, producing more biofuels will generate new industries, new technologies, new jobs and new markets.

## CHAPTER THIRD

## METHODOLOGY

### 3.1 Introduction

This chapter explains the method that was used in this project work. It details the procedure adopted for biodiesel production process and for determination of physical and chemical properties of biodiesel produced .The equipment and the apparatus used in this project are also shown in this chapter. The methodology flowchart of this project work is shown in Figure 3.1.

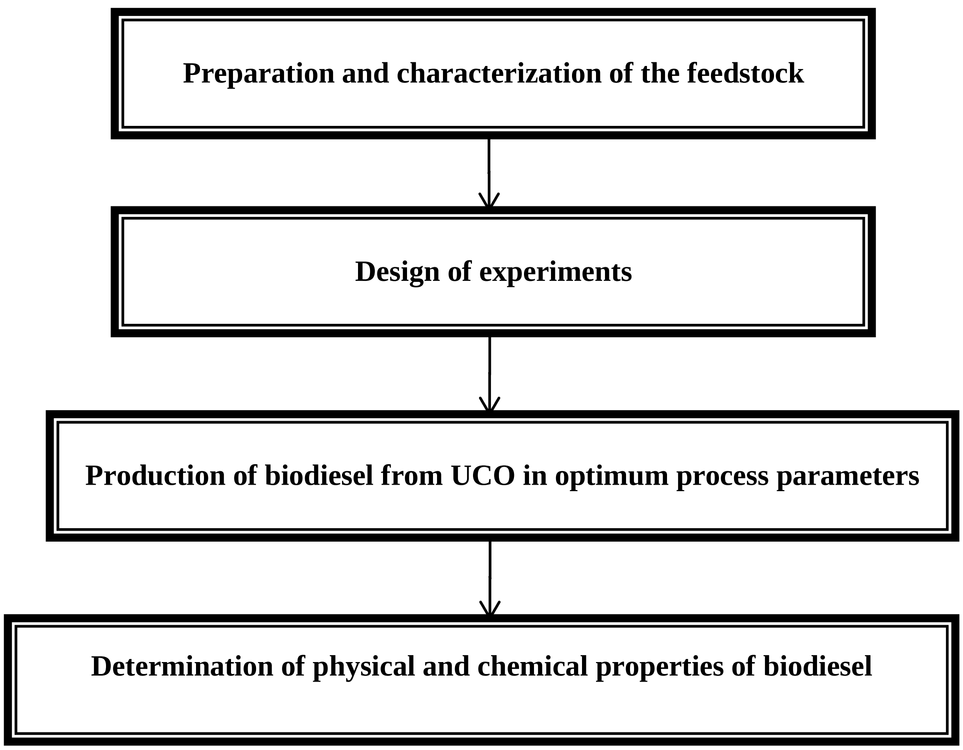


**Figure 3.1: Flow chart of the research methodology**

### 3.2 Biodiesel Production

### 3.2.1 Material and Chemicals

Used cooking oil (UCO) was used to produce biodiesel in this project work. The oil was provided from a restaurant located at Faulty of Engineering – Sudan University of Science and Technology. The oil was processed for experimentation in the Laboratory of chemistry, faculty of science. All chemicals used in the production process were obtained in their analytical grade. Potassium hydroxide (KOH) used to enhance the reaction for the transesterification process. 2-Propanol (iso-propyl alcohol), potassium hydroxide solution (0.1 N), and double rings qualitative filter paper of 125 mm size were also used in this project work. The main physiochemical properties of the UCO sample were determined as per standard methods and reported in Table 3.1.

**Table 3.1: Physiochemical properties of used cooking oil (UCO)**

| Properties | Value |
|---|---|
| Acid value (mgKOH/g) | 1.09 |
| Density @ 20ºC (g/ml) | 964 |
| viscosity@37ºC ($mm^2/s$) | 44.6 |

### 3.2.2 Experimental Procedure

Alkaline-catalysed transesterification is the most widely used process for biodiesel production because it is very fast and yield high amount of biodiesel. However, to use alkaline catalysts, the free fatty acid (FFA) level should be below a desired limit (rang between 0.5% and 3%). Most of used cooking oils have high FFA values. Therefore, transesterification with alkali based catalyst yield a considerable amount of soap which are emulsifiers that make the separation of glycerol and ester phases very difficult. Acid-catalysed esterification was found to be a good solution to this problem. However the oil used for biodiesel production in this project was found to be with FFA value less than 3%. Therefore, Alkaline-catalysed process was used in one step. Refer to APPENDIX (A) for the sample calculation of reaction parameters like amount of methanol and catalyst.

### 3.2.2.1 Alkaline Base-catalyzed Step

The used cooking oil was poured into the round-bottomed flask, then (1% w/w) amount of catalyst KOH was weighed and dissolved completely in methanol (25 % v/v) to form potassium methoxide. Meanwhile, the oil was warmed up, and the prepared methoxide was added into the oil at 60 $^{o}$C. The reaction conducted in vigorous mixing at 400 rpm and for 120 min then, it has been allowed to separate and settle in a funnel for 30 min to remove the glycerol layer which was formed in the bottom of the separation funnel. Levels of the two parameters optimized are shown in Table 3.2.

**Table 3.2: Levels of the alkaline base-catalyst process variables**

| Variables | Low Level | High Level |
|---|---|---|
| MEOH Ratio (% v/v) | 15 | 50 |
| KOH Ratio (%w/w) | 0.8 | 2 |

### 3.2.2.2 Sample Treatment

In this step, the produced methyl ester from the first step washed three times with warm water in 50 °C till the pH of the water was less than 8. To remove the moisture, the final product was heated up to 70 °C for 30 min then filtered with filter papers. This resulted in a clear light liquid which is biodiesel. Table 3.3 lists the equipment and apparatus used in the biodiesel production process.

**Table 3.3: Equipment and apparatus used in the biodiesel production**

| No. | Equipment/ apparatus | Purpose |
|---|---|---|
| 1. | Hot plate magnetic stirrer | To provide heat and steering for the reaction |
| 2. | Two-necked round-bottomed flask | To hold the mixture of oil and methoxide for the reaction |
| 3. | Thermometer with cork | To maintain the temperature of the reaction |
| 4. | Water-cooled Condenser | To condense the evaporated methanol back into two-necked round-bottomed flask |
| 5. | Separation funnel | To separate glycerol from biodiesel by gravity |
| 6. | $P_H$ indicator papers | To measure the $P_H$ of washing water of biodiesel |

## 3.3 Properties Analysis and Equipment

The quality of any fuel is expressed in terms of the fuel properties such as kinematic viscosity, calorific value, CCR, flash point and cold filter plugging point. In this project, the important physical and chemical properties of the biodiesel produced and blends were tested according to ASTM D 6751 standard. The properties determined for the UCO biodiesel produced include; acid value, viscosity, density, calorific value, and flash point. .

### 3.3.1 Determination of Acid Value

Acid value defined as the quantity of potassium hydroxide (KOH) in milligrams which is required to neutralize the free acids in 1 gram of sample. The following sections explain in details the procedure conducted in this project to measure the acid values of UCO biodiesel.

#### 3.3.1.1 Procedure to prepare phenolphthalein indicator (phph)

Phenolphthalein is a chemical compound with the formula $C_{20}H_{14}O_4$. In titration, it turns color less in acidic solutions and pink in basic solutions. To prepare titration solution, 0.5 g of phenolphthalein was dissolved in 50% ethanol (ethyl alcohol) solution consisting of 50ml ethanol and 50 ml water. The solution was stored in a stopper bottle.

#### 3.3.1.2 Titration Procedure

Firstly, a measured amount of the biodiesel was poured into a beaker or Erlenmeyer flask. This was followed by adding 50 ml of 2-Propanol to the measured amount of crude oil. The mixture was then heated to around 50-55 °C to make sure that the oil was well diluted in 2-Propanol. Before the titration is started, 1-3 drops of phph indicator were then added to the mixture. The titration begins with adding the base titrant (KOH solution) with a known normality (0.1 N) from the burette drop by drop to the mixture until the indicator changes, reflecting arrival at the endpoint of the titration. Depending on the endpoint desired, single drops or less than a single drop of the titrant can make the difference between a permanent and temporary change in the indicator. When the endpoint of the reaction is reached, the volume of reactant consumed is measured and used to calculate the acid value of the crude oil. Therefore, acid value can be calculated using the following equation:

$$AV = \frac{MW \times N \times V}{W} \tag{3.1}$$

Where

MW ≡ Molecular weight of potassium hydroxide.

N≡ Normality of potassium hydroxide solution (0.1 N).

V≡ Volume of potassium hydroxide solution used in titration.

W ≡ Weight of oil sample.

Figure 3.2 shows the procedure adopted for titration.

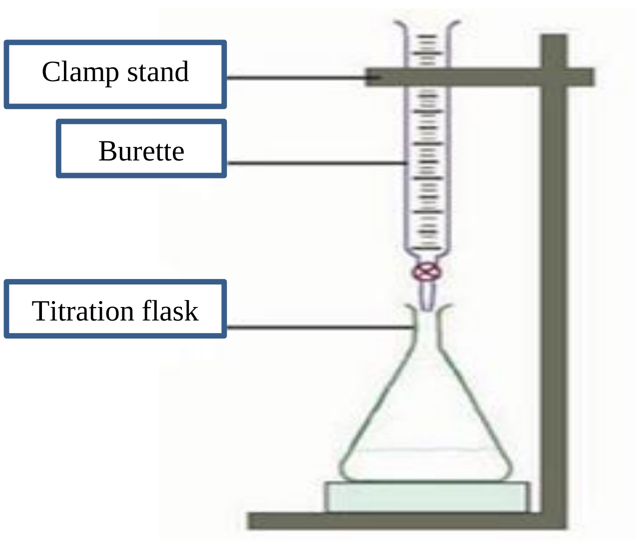


**Figure 3.2: Experiment set up of acid value titration**

**Table 3.4: Equipment list for analyzing biodiesel properties**

| No. | Property | Equipment |
|---|---|---|
| 1. | Density |  Pycnometer |
| 2. | Kinematic viscosity |  Sunrim |
| 3. | Flash point |  Pensky martens |
| 4. | Caloric value |  Lab Calorimeter |

Figure 3.3: Shows the equipment used to analyze the biodiesel properties.

# CHAPTER FOUR

# RESULTS AND DISCUSSION

## 4.1 Introduction

This chapter presents the experimental results obtained using the apparatus and procedures described in chapter 3. The production process of biodiesel is discussed in section one. Section two analyzes the results obtained from the optimization of the alcohol to molar ratio and catalyst concentration and their effects on the biodiesel yield. The third section summarises the properties of UCO biodiesel produced compared to biodiesel standard specification ASTM D6751.

## 4.2 Production of Biodiesel

In the transesterification process, alkaline base catalyst was used for biodiesel production. The final product was a clear light liquid which is biodiesel with a viscosity close to conventional diesel fuel. The sequences of the biodiesel production steps are shown in Figure 4.1.


(a) (b) (c) (d) (e) (f)

**Figure 4.1: Transesterfication process steps of used cooking oil**

(a) reaction of UCO with methoxide. (b) separation of glycerol (c) biodiesel clear from glycerol (d) washing of biodesel (e) filtration of biodiesel (f) final biodiesel

### 4.2.1 Alkaline Base-catalyzed Step

In this process, KOH was used as an alkaline base catalyst in order to enhance the reaction. The process parameters optimized were alcohol to oil molar ratio, and the catalyst concentration. However, the reaction temperature maintained to be less than the boiling point of alcohol which is between 60–70°C at atmospheric pressure, to ensure the alcohol will not be lost through vaporization.

#### 4.2.1.1 Effect of Alcohol Molar Ratio

The alcohol to oil molar ratio is one of the most important factors that can affect the yield of esters. Thus, the effect of methanol molar ratio (15 - 50 % v/v) in biodiesel yield is investigated in this project. The other process parameters remain constant at 120 min of reaction time and 1% concentration of KOH. The results obtained are summarized in Table 4.1 and Figure 4.2. It observed from the Figure that the biodiesel yield is decreased with the increase in methanol to oil molar ration. Excess alcohol is used during transesterification to ensure that the oils will be completely converted to ester due to the forward reaction being more favorable. However, more alchole will negatively affect the reaction and lead to decrease the biodiesel yield. From this experiment, the maximum yield of biodiesel obtained was 83.7% for methanol/oil volume ratio of 25% after 120 min of reaction time.

**Table 4.1: Effect of methanol to oil molar ratio in biodiesel yield**

| No. | Alchol to oil ratio (%v/v) | Volume of biodiesel (ml) | Volume of glycerol (ml) | Biodiesel yield (%) |
|---|---|---|---|---|
| 1 | 15 | 244 | 50 | 81.33 |
| 2 | 25 | 251 | 70 | 83.70 |
| 3 | 40 | 249 | 80 | 83.00 |
| 4 | 50 | 216 | 100 | 72.00 |

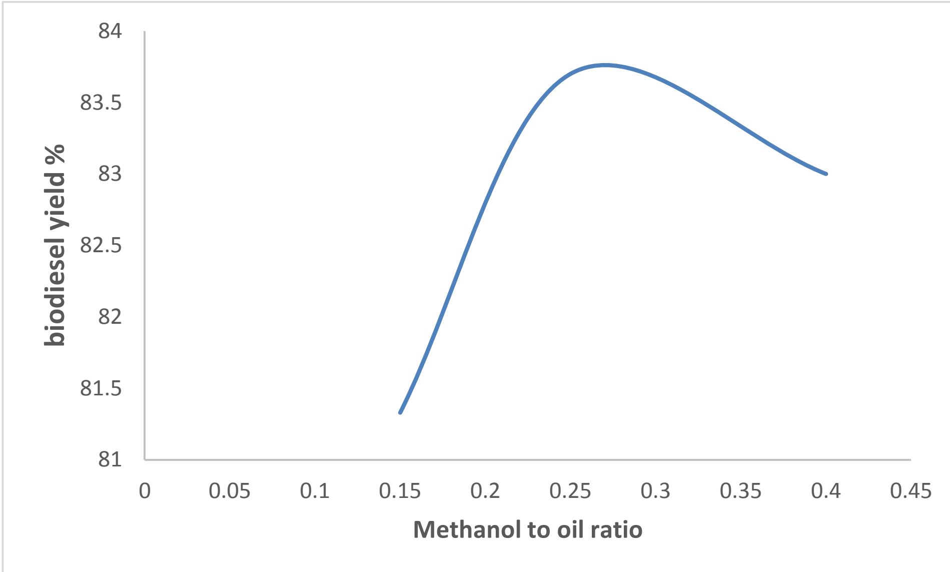


**Figure 4.2: Effect of methanol to oil molar ratio on biodiesel yield**

#### 4.2.1.2 Effect of Catalyst Concentration

The effect of KOH concentration on the biodiesel yield is investigated in Table 4.2 and Figure 4.3 with its concentration varying from 0.8 to 2% w/w. The other process parameters were fixed at 120min of reaction time and alcohol to oil ratio of 25% v/v. As mentioned earlier, basic catalysts are usually preferred to acid catalysts in this transesterfication process, because of the higher reactivity and the lower process temperature required.

**Table 4.2: Effect of KOH concentration in biodiesel yield**

| No. | Catalyst ratio (%w/w) | Volume of biodiesel (ml) | Volume of glycerol (ml) | Biodiesel yield (%) |
|---|---|---|---|---|
| 1 | 0.8 | 256 | 50 | 85.33 |
| 2 | 1.0 | 251 | 70 | 83.70 |
| 3 | 1.5 | 238 | 90 | 79.33 |
| 4 | 2.0 | 202 | 125 | 67.33 |

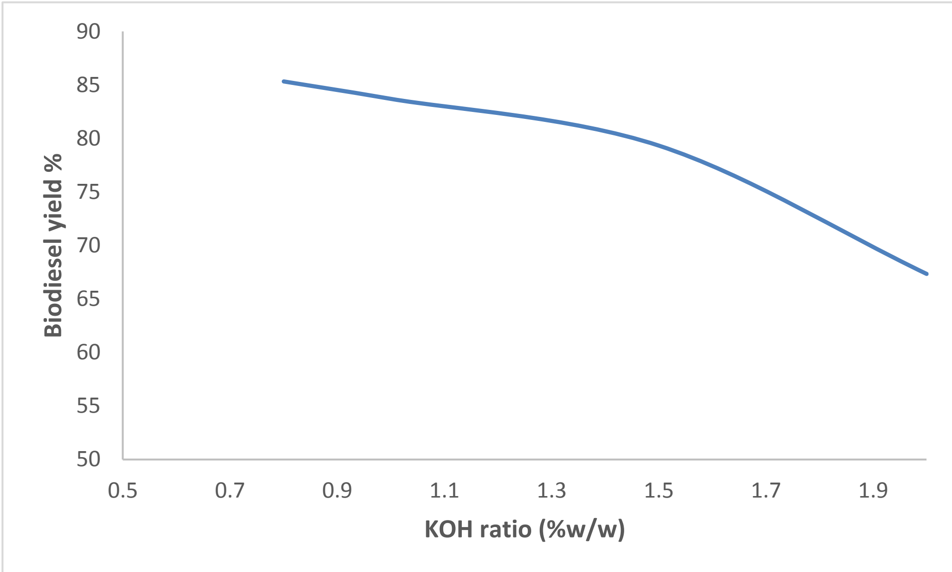


**Figure 4.3: Effect of KOH concentration on biodiesel yield**

As can be observed from Figure 4.3, the biodiesel yield is decreased with the increase in catalyst concentration to reach the minimum value at higher concentration of 2.0% of catalyst. However, the yield of biodiesel is reduced if the alkali catalyst is added above its optimum concentration as this causes more soap formation. The maximum biodiesel yield achieved in the experiment was 85.33% at catalyst concentration of 0.8%.

The optimum condition of the transesterfication process was found to be 25 % (v/v) methanol to oil ratio, 0.8% (w/w) of KOH, at maintained reaction of time 120 min and reaction temperature of 60$^{o}$C. The results obtained from this process, was biodiesel free from glycerol.

## 4.3 Biodiesel Properties Specifications

The basic biodiesel parameters were tested to ensure the fuel quality according to the standard specification. Table 4.3 shows some of physical and chemical fuel properties of the produced biodiesels from used cooking oil in this project work. It can be observed that all properties are in agreement with the standard specification of biodiesel ASTM D6751.

**Table 4.3: Physical and chemical properties of the produced biodiesel**

| Property | UCO Biodiesel | ASTM D6751 limit |
|---|---|---|
| Acid Value mg (KOH/g) | 0.056 | 0.5 max |
| Viscosity at 40 °C ($mm^2/s$) | 2.15 | 1.9-6.0 |
| Density at 20 °C ($kg/m^3$) | 875 | 880 at 15°C |
| Caloric value (MJ/kg) | 41.05 | Report |
| Flash point °C | 186 | 130 min. |

The kinematic viscosity at 40 °C of the UCO biodiesel produced found to be 2.15 $mm^2/s$. This is an expected finding since biodiesel molecules are single, long chain fatty esters with higher mobility than the bigger and bulkier triglyceride molecules as revealed by some researcher like Sanford, *et al*., 2009.

The flash point of the UCO biodiesel produced found to be 186 $^oC$ which is high compare to the flash point of diesel fuel is 55–66 $^oC$. This means that biodiesel is safe for storage purpose, handling and transport. This high value flash point of biodiesel is due to the presence of unsaturated acid chain length of C18:1 and C18:2 in the vegetable oil (Silitonga *et al*., 2013).

Moreover, the calorific value was found to be 41.05 Mj/kg which is more than that value of used cooking oil obtained by Enweremadu, *et al*., 2010 which was 39.77 Mj/kg. The calorific value is not specified in ASTM D6751 and EN 14214 biodiesel standards but it is prescribed in EN 14213 (biodiesel for heating purpose) with a minimum value of 35 MJ/ kg

# CHAPTER FIVE

# CONCLUSION AND RECOMMENDATIONS

This chapter summarizes the work done in this research with the set objectives and several recommendations that are stated for future research.

## 5.1 Conclusion

i. Selection of a transesterification process steps depends on certain parameters including the amount of free fatty acid content of the feedstock. However, base alkaline catalyzed transesterification process is most effective in converting triglycerides into esters when free fatty acid level is less than 1%.

ii. Adopting an alkaline base tranesterfication process, the maximum final biodiesel yield of 85.3% was achieved from used cooking oil in combination of process parameters of 25% (v/v) methanol to oil ratio, 0.8% (w/w) of KOH, at maintained reaction time of 120 min, reaction temperature of 60 $^{o}$C and steering peed of 400 rpm.

iii. The biodiesel yield is decreased with the increase in methanol to oil molar ration. On other hand, the biodiesel yield is reduced if the alkali catalyst is added above its optimum concentration as this causes more soap formation.

iv. The basic physicochemical properties of the UCO biodiesel produced in this project work were found to be in agreement within the ASTM D6751 specified limits. Mainly, Acid value, density, viscosity and flash point. Moreover, the calorific value was found to be 41.05 Mj/kg.

v. Overall, the results obtained from this project reveal that biodiesel produced from used cooking oil is a promising alternative for Sudan energy sector.

## 5.2 Recommendations

Several recommendations are suggested, as following;

i. Some other biodiesel properties should also be analysed against the ASTM D6751 specification limits. These include oxidation stability, cloud and pour point, free fatty acid composition and total glycerol and free glycerol,

ii. The effects of other process parameters like reaction time and reaction temperature on the biodiesel yield should also be optimized.

iii. Investigate the potential and the economics of producing biodiesel from used cooking oil with high level of free fatty acid.

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# APPENDIX A

## Calculations of Biodiesel Conversion Parameters

### Calculation of molar ratio and volume of methanol

From general stoichiometric of transesterification reaction,

1 mol of triglyceride reacts with 3 mol of methanol to produce 3 mol of biodiesel and glycerol.

volume $_{\text{of MeOH}} = \frac{\text{No.of mol} \times \text{molar ratio} \times \text{MeOH molecular weight}}{0.7918}$

or 25% of oil volume used

### Calculation of catalyst used

Concentration of catalyst to jatropha oil in wt% can be obtained by calculation mass of catalyst used in the reaction.

Catalyst wt% $= \frac{\text{Mass of catalyst} \times 100}{\text{Mass of uco oil}}$