
# Addis Ababa institute of Technology

## School of Chemical ad Bioengineering

**Production and Characterization of Biodiesel from Moringa Stenopetala Seed Oil via Catalytic Transesterification Process**

By

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A thesis submitted to school of graduate studies of Addis Ababa University in partial fulfillment of the requirement of Masters of Science in Chemical Engineering (Process Engineering stream)

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# Addis Ababa University

## Addis Ababa institute of Technology

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This is to certify that the thesis prepared by Gemechu Yadeta, entitled: **Production and Characterization of Biodiesel from Moringa Stenopetala seed oil via catalyzed transesterification process** and submitted in partial fulfillment of the requirement for Masters of Science in Chemical engineering (process engineering stream) complies with the regulations of the university and meets the accepted standards with respect to quality and originality

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## Declaration

I declare this thesis entitled ‘’ Production and characterization of biodiesel from moringa stenopetala seed oil via catalyzed transesterification process’’ for M.Sc. degree at Addis Ababa University, here submitted by me, is my original work and has not been submitted for any degree, diploma or an award at any university or institution. Information and citations taken from published and unpublished work of others have been duly acknowledged in the text and list of reference is given. This work was directed under the guidance of Dr. Eng. Abubeker Yimam, instructor at Addis Ababa University and Dean of School of Chemical and Bioengineering.

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**Abbreviations and Symbols**

MSSO…….Moringa Stenopetala Seed Oil

RSM……Random Surface Method

FAME…….Fatty Acid Methyl Ester

IEA…….International Energy Agency

CI…….....Compression Ignition

ASTM……American Standard for Testing Materials

AC…….Acid Value

CV…….....Calorific Value

CCD……. Central Composite Design

FFA…….. Free Fatty Acid

ANOVA……...Analysis of Variance

VM……Volatile Matter

FC……..Fixed Carbon

Mc…….Moisture Content

EU…….European Union

USA……..United States of America

LPG……..Liquefied Petroleum Gas

SV ……Saponification Value

AV……Acid Value

I.V ……....Iodine Value

C.N ……..Cetane Number

Mwt……..Molecular weight

S.G ……..Specific Gravity

MS ……...Moringa Stenopetal

ISO……International Standards Organization

CaO……...Calcium Oxide

KOH…….....Potassium Hydroxide

CCl4……..Carbon tetra chloride

HCl……Hydrochloric acid

EN……..European Standards

EPA……Environmental Protection Agency

$Na_2S_2O_3$…….....Sodium Thiosulfate

KI……Potassium Iodide

SCFs……...Super Critical Fluids

AAiT……..Addis Ababa institute of Technology

AAU……...Addis Ababa University

SNNPE……Southern Nations, Nationalities and Peoples of Ethiopia

Rpm……Revolution per minute

ha…….Hectare

hr……..hour

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**Abstract**

*The energy demand of the world is increasing from time to time. Depletion of petroleum reserves, the rising price of petroleum fuels and environmental pollution are the major problems of the countries including Ethiopia. These problems pushed the researchers to focus on renewable energy sources such as biodiesel. Biodiesel is a clean and alternative energy source derived from natural fats or vegetable oils. This study investigated production of biodiesel from moringa stenopetala seed oil via catalyzed transesterification process. The optimum process parameters for soxhlet extraction of oil were: 80 °c, 5 hours and 5:1 for temperature, extraction time and hexane to sample ratio respectively. Biodiesel was produced from purified oil by catalyzed transesterification reaction. Experimental design and statistical analysis was done by Design Expert 11.0 software. The optimization of process parameters of transesterification reaction: methanol to oil ratio, temperature and catalyst load was carried out by response surface method (RSM). The experimental result showed that the maximum biodiesel yield was obtained at optimum conditions of temperature 55°c, methanol to oil ratio 6:1 and catalyst loading 1.0 %. The main physicochemical properties of oil and biodiesel were determined according to international ASTM and EN standards. It was found that MSSO biodiesel moisture content 1.4 %, pH 7.9, specific gravity 0.874 g/mole, kinematic viscosity 4.8 mm2/s ,acid value 0.4, saponification value 196 mg KOH/g, free fatty acid 0.2 %, iodine value 104,2 g I2/100 g , calorific value 43 MJ/KG, flash Point 184 °c , fire point 190 °c ,pour point 1 °c ,cloud point 10 °c and cetane number 53. Whereas, moisture content, density, kinematic viscosity, acid value, saponification value and free fatty acid of oil were 6.54%, 840 kg/m3, 9.4 mm2/s, 1.6 mg KOH/g. 189 mg KOH/g and 0.8 % respectively. These results agreed with international standards values and previous research works.*

Keywords: Moringa stenopetala seed oil, Biodiesel, Transesterification, Catalyzed, optimization

# 1. Introduction

## 1.1 Background of the study

Energy plays a crucial role in the socio-economic development of any country. The world is highly dependent on fossil fuels for energy source. Current concerns such as global warming, depletion of fossil fuels and fluctuating price of petroleum-based fuels have pushed researchers toward finding new alternative, cleaner, renewable and sustainable energy resources, such as solar energy, hydroelectric energy, wind energy, and biomass-derived energy (Chu and Majumdar, 2012).

Energy demand is expected to increase due to rapid population growth, expanding urbanization and better living standards (Khan et al, 2014). According to Serrano-Ruiz JC *et al.* (2010) the world energy consumption would increase by 35% over the next 20 years in to meet the increasing energy demand of industrialized countries. Currently fossil fuel is the dominant sources of energy in the world and makes up around 80% though no renewable and has negative impact on global climate (IEA, 2009). Rapid growth in both global energy demand and carbon dioxide emissions associated with the use of fossil fuels has driven the search for alternative energy sources which are renewable and has a lower environmental impact (Atabani et al, 2013).

Biodiesel is a promising biofuel which is renewable, biodegradable and nontoxic. Biodiesel is gaining importance as one of the most important clean energy source. There are many advantages of using biodiesel as an alternative form of energy. It can be used as such in the diesel engine without any engine modification indicating that it has similar physical and chemical properties as conventional diesel fuel. The combustion properties of biodiesel are also very close to those of petroleum diesel (Piyushi et al, 2013).

The Interest of using biodiesel has been expanding recently due to high petroleum prices. The reserves of fossil fuel are being depleted, while the environmental problems caused by their use have become serious. Thus, the renewable energy is the best alternative energy source to be used (Eman and Cadence, 2012).

There are several potential feed stocks that are used for the production of biodiesel. The first generation feed stocks (edible) and Second-generation feed stocks are non-edible feed stocks, waste oil and animal fats. First-generation biofuels are directly related to a feed stocks that are

generally edible, and they are usually obtained from edible oils, such as soybeans, palm oil, sunflower, safflower, rapeseed, coconut and peanut. Second-generation biofuels are fuels that are produced from a wide range of different feed stocks, ranging from lignocellulose feed stocks to municipal solid wastes. Third-generation biofuels are related to algae species which have been considered as emerging non-edible oil sources of growing interest because of their high oil content and rapid biomass production. However, the first generation biofuels seems to create some skepticism to scientists due to food versus fuel crisis (Marchetti and Yadessa, 2017).

Globally, there are more than 350 oil-bearing crops identified as potential feed stocks for biodiesel production. The availability and variety of biodiesel feed stocks is one of the most significant factors that enables the sustainable production of biodiesel (Marchetti and Yadessa, 2017). Satisfactory replacement of petroleum diesel with biodiesel depends on two basic requirements: first is its easy availability and environmentally acceptability, and the second is being economically reasonable (Avhad and Marchetti, 2015).

Moringa seed oil have been proposed as an alternative potential feed stock to produce biodiesel and complement with other feed stocks by researchers. Moringa seed concentrates an average of 35 %- 45 % oil content with high oleic content of greater than 73 % based on the type of the tree. Moringa oil is considered as a great natural cosmetic emollient and has high oxidative stability (Ayerza, 2012).

Moringa is particularly suitable for dry regions, since it can be grown using rainwater without expensive irrigation techniques. Flowering begins within the first six months. In seasonally cool regions, flowering only occurs once a year between April and June. In constant seasonal temperatures and constant rainfall, flowering can happen twice or all year-round. Moringa fruit is a hanging, three-sided brown capsule of 20–45 cm size which holds dark brown, globular seeds with a diameter around 1 cm. The seeds have three whitish papery wings and are dispersed by wind and water (H.Sreepada and H.Vijayalaxmi, 2013).

Ethiopia has a comfortable environment for moringa tree plantation and it has been promoted across the country. In most parts of Ethiopia, particularly in southern parts of the county, moringa tree plantation is increasing due to multipurpose use of the plant. Many people of southern Ethiopia has planted it on their garden and farm land (H.Sreepada and H.Vijayalaxmi, 2013).

## 1.2 Statement of the problem

Ethiopia has faced serious problems in energy supply and utilization. Energy needs will continue to grow despite the increase in oil prices. Ethiopia is an energy importer and is viewed as the top energy poor country in the world. According to International Energy Agency (IEA) report only 23% of households have electricity connection in 2012, with this rate being 11% in rural areas. Looking at public investments, Ethiopia suffers from a persistent lack of infrastructural development, particularly in the sector of energy supply. Fossil fuels cannot satisfy the world's demand for energy in the long run, especially with the concern that these fuels are the main cause of climate change. Fossil fuels such as coal, oil, and natural gas are basically valuable mineral resources of many countries which could fuel for human activities.

Using non renewable energy sources (fossil fuels) become potential danger for environment. Burning coal, oil and natural gas releases mostly carbon dioxide, which adds to the greenhouse effect. In addition, such fuels also release Sulfur dioxide and oxides of nitrogen, which are causing acid rain. Furthermore, Fossil fuels like oil do also imply serious environmental consequences especially, the serious issue is of accumulating concentrations of atmospheric CO2 which most researchers by now agree on implies greenhouse effects and climate changes. A very large share of the CO2 released in atmosphere can be derived from gasoline or diesel fueled vehicles. As a result, increasing CO2 concentrations are expected to lead to a warmer global climate, resulting in higher sea levels and severe consequences for ecological systems, including human beings.

In order to reduce the dependency on liquid petroleum fuel, fuel cost, fuel scarcity and environmental problems, Ethiopia is striving to find sustainable and renewable fuel alternatives such as biogas, syngas, bioethanol, and biodiesels that are more environmentally friend and can be produced from locally available biomass feed stocks. There are a wide range of feed stocks for the production of biodiesel. Most of them are edible oil bearing crops and vegetables. Production of biodiesel from this edible feed stocks caused the problem of food versus energy crisis (food security problem). Hence, many researchers are looking for the other non-edible feed stocks for biodiesel production. There is a wide range of non-edible feed stocks like Moringa seed and jatropha in Ethiopia. Moringa stenopetala seed oil is one of the promising non edible feed stocks for production of biodiesel as it does not compete food security.

## 1.3 Objectives of the study

### 1.3.1 General objective

The general objective of this study was Production and characterization of Biodiesel from Moringa stenopetala seed oil by catalytic transesterification process as an alternative energy source

### 1.3.2 Specific objectives

The specific objectives includes the following

- To determine the physical and chemical properties of Moringa stenopetala seed
- To extract and determine physical and chemical properties Moringa stenopetala seed oil
- To determine and analyze physical and chemical properties of biodiesel
- To investigate the main effects of transesterification process parameters on yield and fuel quality of biodiesel and find the optimum operating parameters using Response Surface Method (RSM)

## 1.4 Significance of the study

The most significance of this study was providing an alternative renewable energy source which can be used in diesel engine without engine modifications. Now days, the use of fossil fuels causes global warming and air pollution since it releases greenhouse gases to the environment. This increases the interest of development in renewable energy sources such as biofuel, solar, wind, geothermal, and hydrogen energies. Cleaner energy technologies like biodiesel production from locally available moringa seed oil can substitute the use of depleting petroleum fuels.

This study has a great significance in terms of assuring the production of clean alternative energy source from locally available feed stock. Next to jatropha and castor, moringa seed is a promising non edible feed stock for the production of biodiesel in Ethiopia. Even though it has a tremendous benefits, Moringa is a neglected, miracle and less utilized tree species in Ethiopia.

Moringa particularly Moringa stenopetala is an indigenous, neglected, miracle and underutilized tree species in Ethiopia. Moringa stenopetala has been promoted for long period of time in different parts of the country mainly in drought prone areas. It has a wider adaptability and tolerance to drought. Moringa tree seed is a second generation feed stock (non-edible) and has high oil content

similar to jatropha. This will make moringa seed environmental friend plant and promising source for biofuel besides other feed stocks.

This study also introduced the new potential feed stock (moringa stenopetala) that did not discover in the previous and extend the choice for alternative fees stocks. Moreover, this study provided eco-friend, underutilized tree seed, oil rich, fast growing and higher yield feed stock for biodiesel production. Hence, production of biodiesel from indigenous moringa stenopetala has a great significance in energy sector as well as for environmental protection.

## 1.5 Scope and limitation of the study

The study included feed stock collection, preparation, characterization, oil extraction and characterization of it, biodiesel production and characterization of it and optimization of process parameters (factors) using standard procedures and test methods. Comparison and evaluation of physiochemical properties of biodiesel with an international standard such as ASTM and European methods.

Besides to characterization of different basic fuel properties of it, Biodiesel can be blended with petrol-diesel at different percentage and apply in compression ignition (CI) engine to evaluate the effect of using blend of biodiesel by using different parameters such as engine power, brake thermal efficiency, and specific fuel consumption, exhaust gas temperature and greenhouse gas emission. However, the performance of analysis of blended biodiesel with petrol diesel in CI engine did not done due to lack of laboratory set up and equipment required to measure these parameters. This study did not compare biodiesel with conventional fuels by using in diesel engines. Moreover, it did not characterize the blend of biodiesel with other petroleum fuels and did not compare biodiesel produced from other feed stocks other than moringa stenopetala.

### 1.6 Organization of the paper

Chapter one deals with introduction part of the study. Chapter two discusses review of related literatures to the study while chapter three is about materials and methods of the study. Chapter four deals with results and discussions. Chapter five is about conclusions and recommendations.

# 2. Literature Review

## 2.1 The current trends of Biodiesel production

### 2.1.1 Worldwide trends of Biodiesel production

Biodiesel production is dominated by European Union (EU) countries followed by USA, Argentina and Brazil. Globally, ethanol consists of 80 % of liquid biofuels produced while 20 % is biodiesel. EU is the world major biodiesel manufacturer with a share of 57 % of total world production in 2009. Currently, the production capacity of European biodiesel has reached approximately 22 million tons. Within the EU, the first four largest biodiesel-producing countries share two-thirds of total production are Germany (33 % of total European production), France (18 %), Spain (7 %), and Italy (5.6 %) (Padula, 2014).

Currently, majority of the world countries produce biodiesel from edible oil resources which affects the food security by increasing price of food and land. For these reasons the world is looking for biodiesel oil resources which are non-edible, biodegradable and environmentally friend. In Australia, 90 % of biodiesel are produced from non-edible oil resources and 10 % from edible oil resources. Australia started producing biodiesel in 2004 and the potential production capacity reached 9100 barrels per day in 2011 (A.K Azad et al., 2014).

Biodiesel production increased rapidly starting from 2002 to 2006 with 40% increase annually. According to the United States Environmental Protection Agency (EPA), the total diesel demand in US and EU was 147 billion gallons. The following table shows summary of biodiesel production in USA. As it can be seen from the table, production rates are increasing every year and the target of EPA for 2020 was to ramp up the production to 12 billion gallons in USA.

*Table 1: Biodiesel production summary in 2009 - 2011 in USA (million gallons)*

| Year | 2009 | 2010 | 2011 |
|---|---|---|---|
| Consumption | 326 | 263 | 878 |
| Production | 516 | 343 | 967 |
| Gross imports | 77 | 23 | 36 |
| Gross exports | 266 | 105 | 73 |
| Consumption (% of distillate fuel by volume) | 0.6 | 0.5 | 1.5 |

IEA, *Monthly Energy Review,* August 2012. Biodiesel Production and Consumption:

### 2.1.2 Biodiesel production trends in Ethiopia

Ethiopia is one of the energy poorest countries in the world. In Ethiopia the largest energy consumption rely on traditional biomass fuel and petroleum fuels such as gasoline, kerosene, fuel oil and LPG. 77 % of total export earning directly goes to meet national fuel demand. This is the great economic challenge for the country. This socio-economic problems can be minimized by developing alternative renewable energy sources. According to Ethiopian Biofuel development strategy of 2007, biodiesel production from alternative feed stocks such as jatropha curcas, castor oil and moringa are one of the main sustainable energy development pillars in the country.

Ethiopia is a net energy importer and the top energy poorest country in the world. According to IEA only 23 % of Ethiopian households have access to electricity in 2012. Moreover, the country suffers from lack of infrastructure development particularly in the energy sector. Most energy supply is dependent on locally available traditional biomass energy and expensive imported fossil fuels to some extent. Ethiopian government launched an extensive bio- fuels expansion strategy and investment promotion program in 2007 for two selected biodiesel feed stocks: castor and jatropha. These two feed stocks has been promoted especially for their expected adaptability and capacity to grow on marginal lands and under drought conditions (O. Riera, 2014).

From 2007 onwards, the Government has specifically sup- ported the economic attractiveness of biofuel production and the expansion of investments in the sector by providing incentives to investors which include tax holidays, low-cost land leases, and long term credit facilities, among others. The government's interest in biofuels was later reemphasized five year period (2010–2015). During these years, ethanol production was increased to 194.9 million liters and biodiesel to 1.6 million liters. The blending facilities were increased to 8 million liters for ethanol and to 72 million liters for biodiesel (Martha. N and O. Riera (2014).

There are various non-edible oil bearing feed stocks such as jatropha, castor bean, moringa, pongamia and croton grown in Ethiopia. Although castor and jatropha are the most known and cultivated crops, other feed stocks such as moringa are promising for Ethiopia. Jatropha and castor

are the primary feed stocks selected by government. Ethiopia is one of African countries by planting jatropha on large area, approximately 20,000 ha.

*Table 2: Biodiesel production projects in Ethiopia in 2012*

| Type of business model | Cultivation Scheme | No projects | Type of feedstock specified | Total area (ha) | |
|---|---|---|---|---|---|
| | | | | Total allotted (1000 ha) | Under cultivation (1000 ha) |
| Corporate centered | Large scale plantation(a) | 3 | Jatropha, pongamia, castor | 66.7 | 8 |
| | Out growers (b) | 1 | Castor | Not applicable | 15 -30 |
| | Mixed out grower with plantation | 2 | Castor | 3 | 3 |
| Private centered | Mixed out grower with plantation | 1 | Jatropha, candlenut, croton | 15 | 7 |
| Environment centered | Afforestation | Not identified | Jatropha | Data not available | Data not available |

a = all are foreign firms. b = Cultivation by farmers. c = Jatropha planted by farmers as fence
Source: Martha. N and O. Riera (2014).

**2.2 Feed stocks (Raw materials) used for Biodiesel production**

There are various feed stocks for biodiesel production. Oil crops are the common pillar raw materials for biodiesel production. It is very important to select the suitable feedstock for biodiesel production since feed-stock alone takes 75% of biodiesel production cost. Currently, there are about 350 oil-bearing crops that have been identified for biodiesel production globally. They are categorized as edible and non-edible oil sources. The main edible oil sources are: peanut oil, soybean oil, sunflower oil, corn oil, rice bran oil, palm oil, coconut oil, olive oil, rapeseed oil, castor oil, milkweed seed oil and linseed oil. Non-edible oil sources are: Jatropha curcas, Pongami, Madhuca indica, Salvadora oleoid, cotton seed oil, Tobacco, Calophyllum inophyllum, Eruca Sativa Gars, terebinth, rubber seed oil, desert date, fish oil, Jojoba, neem oil, apricot seed, Pistacia chinensis, Bunge Seed, Moringa olifera (moringa stenopetala native to Ethiopia) and croton

megalocarpus. Besides to this oil crops, microalgae, terpenes, waste cooking oil and animal fats are also alternative feed stocks for biodiesel production (H.M Mahamudul et al., 2017).

Generally edible feed stocks such as sunflower, coconut oil and palm oil which are used for biodiesel production are called first generation feed stocks while non-edible vegetable oils are second generation. Feed stock alone takes the highest of overall production cost of biodiesel. Hence, it is important to employ inexpensive feed stocks to be competitive with petroleum derived fuels.

*Table 3: Alternative feed stocks for biodiesel production*

| Edible oils | No-edible oils | Animal fats | Other sources |
|---|---|---|---|
| Barley | Camelina sativa | Beef tallow | Cyanobacteria |
| Canola | Cotton seed | Chicken fat | Bacteria |
| Coconut | Croton megalocarpus | Fish oil | Cooking oil |
| Soybeans | Jatropha curcas | Pork lard | Fungi |
| Ground nut | Jojoba | Poultry fat | Latexes |
| Sorghum | Karanja (pongamia pinnata) | Waste salmon | Microalgae (Chlorella vulgaris) |
| Peanut, wheat | Mahua (Madhuca indica) | | Miscanthus |
| Pumpkin seed | Moringa seed | | Pomace oil |
| Rape seed | Neem ( Azadirachta indica) | | Poplar |
| Rice brain oil | Passion seed (Passiflora adulis) | | Soap stocks |
| Sunflower Sesame, corn | Rubber tree seed (Hevea brasiliensis), Tobacco seed | | Switch grass, Tall oil, Tarpenes |
| Sesame ,palm | | | |

Source: (M.Marchetti and Yadessa G (2017).

The yield of biodiesel production highly depends on the oil content of the selected feed stocks. The higher the oil content of the feeds stock, the higher yield of biodiesel produced from the corresponding feed stock.

### 2.3 Status of Moringa tree and its utilization in Ethiopia

*Moringa tree* is an evergreen, fast growing, deciduous and widely cultivated tree species. It has some other common English names such as, drumstick tree, horseradish tree and benzoic tree. It grows in tropical and subtropical areas. It requires rainfall about 250-2000 mm depending on soil condition. It grows best in dry sandy soil and tolerates poor soil with *pH* range 5 to 9. Moringa is distributed from Africa, Asia, Latin America and Oceana countries including Australia. Moringa plant can be grown by direct seeding and cuttings. If water is available for irrigation, moringa trees can be seeded directly and grown anytime during the year. On the other hand, cuttings should be 45cm to 1.5m long and 10cm thick can be planted directly or planted in sacks in the nursery. The tree can reach a height of 10 to12m and the trunk can reach a diameter of 30 to 45cm. When matured, each moringa tree can produce a high yield of seed pod from 300 up to 1000 seed pods. (A.K. Azad et al., 2014).

In Ethiopia, Moringa has been promoted for long time. However, its utilization is limited to specific areas. Moringa stenopetala is native to Ethiopia. It is grown in southern parts of the country and in other areas. Moringa is a multipurpose and miracle tree. Its leaves are used for medicinal, food, cosmetics production and many other applications. The seed of moringa has high oil yield of 35-45 %. Moringa is not commercially utilized in Ethiopia Currently, moringa seed oil is not used for food purpose and utilization of it for renewable energy production does not affect food security of the country.

The native Moringa species in Ethiopia is Moringa stenopetala. Moringa stenopetala is often referred as African Moringa tree because it is native to only Ethiopia and Northern Kenya. Moringa stenopetala belongs to the family of Moringa cease which is represented by single genus ‘’Moringa’’. The genus has 14 species to which Moringa stenopetala belongs (Mark, 1998). It can grow in elevations between 1000 – 1800 m in Ethiopia. It is also extremely a fast growing tree and can continue growing during the exceptionally long dry season. Moringa stenopetala is one of the most widely cultivated indigenous tree species in Ethiopia. It is widely distributed in southern parts of Ethiopia such as: Wolayta, Gamo Gofa, Derashe, Konso, Sidama, Bale, Arba Minch and Borana. Although MS is widely cultivated and has got research and development attention worldwide, it has introduced introduce to Ethiopia very recently and much more research has to be conducted to evaluate and investigate its species, benefits and biomass production (Petr Nemec

et al., 2020). The leaf of Moringa is very popular vegetable in Southern Nations, Nationalities and peoples Regional state of Ethiopia and valued for its special flavor. It is grown as backyard tree for daily use in more than six million households.

Ethiopia has more than 23 million hectare potential land for feed sock production of biodiesel and 700,000 irrigable potential land for ethanol production. Thus, about 1 billion liters of ethanol can be produced per year and this excess ethanol can be used as substitute of methanol for biodiesel production (Ethiopian Ministry of mines and energy, 2007).

**Fatty acid composition of Moringa seed oil**

Fatty acid composition of any feed stock of biodiesel is an important parameter which helps to select the efficient method to produce biodiesel. The available fatty acids contained in vegetable oils are palmitic (16:0) and stearic (18:0) acids. Some of the vegetable oils also contain other fatty acids such as lauric (12:0), myristic (14:0), Palmitoleic (C16:1), Oleic (18:1), linoleic (18:2), linoleic (18:3), arachidic (20:0), beheric (22:0) and lignoceric (24:0). It affects the fuel properties of biodiesel such as calorific value, cetane number, viscosity, melting point and flash point (H. M. Mahmudul et al., 2017).

*Table 4: Fatty acid composition of Moringa seed oil.*

| Feed stock | Fatty acid content | | | | | | |
|---|---|---|---|---|---|---|---|
| Moringa seed oil | C16:0 Palmitic acid | C16:1 Palmitoleic acid | C18:0 Stearic acid | C18:1 Oleic acid | C18:2 Linoleic acid | C18:3 Linolenic acid | C20:0 Arachidic acid |
| | 7.6 | 1.4 | 5.5 | 66.1 | 8.1 | 0.2 | 5.8 |

Source: Kafuku G, Mbarawa M (2010).

**2.4 Extraction methods of seed oils**

There are three main methods that have been identified for oil extraction. These are: mechanical extraction, chemical or solvent extraction and enzymatic extraction. In addition to this, accelerated solvent extraction, supercritical fluid extraction and microwave assisted extraction methods are

frequently used; however, they are not as common as the first three mentioned methods. It has been identified that mechanical pressing and solvent extraction are the most commonly used methods for commercial oil extraction (Atabani AE et al., 2013).

### 2.4.1 Mechanical oil extraction

Oil seed is fed to mechanical press machine which exerts high pressure. When high pressure is applied on the required amount of seed, the solid cake is removed at one side while oil and some impurities are collected on another side. Finally, oil and impurities are separated using gravity settling. Mechanical oil extraction method is most applicable in industrial scale. These technique is used for particular feed stocks which has high oil yield (40% -70%). Oil extracted by mechanical technique has large amount of gum, and wastes and needs extra treatments of purification and degumming. The problem of conventional mechanical press is it is designed for particular seeds and not suitable for other seeds as it affects the yield. The advantageous of mechanical extraction technique are: can be done by simple equipment and low investment, low operating cost and oil need not undergo separation process (Ong et al., 2013).

### 2.4.2 Soxhlet oil extraction

Soxhlet oil extraction is also known as chemical or solvent extraction method. Soxhlet oil extraction technique is done using soxhlet apparatus which contains a glass extractor, condenser at top, thimble holder, round bottom distillation flask initially contained an extracting solvent and hot plate. The most common type of solvent used is Hexane due to its low cost and low toxicity. Initially the extraction solvent is poured round bottom distillation flask and it is heated up. When the solvent vapor goes up to the condenser and condensate returned to the glass in container. Then contacted with seed oil and oil is filtered out from the seed due to mass transfer. After 3 – 4 hours of extraction, the mixture is allowed to recover under vacuum at 65 °C with rotational evaporator to remove the solvent (Amin et al., 2010).

### 2.4.3 Enzymatic oil extraction

Enzymatic oil extraction technique is a promising oil extraction method that uses suitable enzymes to extract oil from crushed seeds. The main advantages associated with this method are: it is environmentally friend and does not produce volatile organic compounds. Some disadvantages of this technique are long extraction time and operation cost (A.E. Atabani et al., 2012).

*Table 5: Physicochemical properties of crude oils*

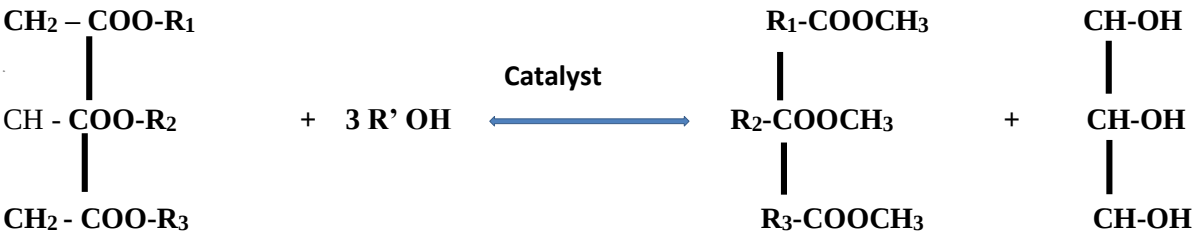
| Vegetable oil | Kinematic viscosity (mm2/s) | Cetane number | Heating value(MJ/KG) | Flash point( °c) | Density (Kg/m3) | Pour point (°c) | Cloud point (°c) |
|---|---|---|---|---|---|---|---|
| Coconut | 27.64 | 51.5 | 37.80 | 264.5 | 0.9089 | - | - |
| Corn | 34.9 | 37.6 | 39.5 | 277 | 0.9095 | -1.1 | -40 |
| Cottonseed | 33.5 | 41.8 | 39.5 | 234 | 0.9148 | 1.7 | -15 |
| Linseed | 27.2 | 34.6 | 39.3 | 241 | 0.9236 | 1.7 | -15 |
| Peanut | 39.6 | 41.8 | 39.8 | 271 | 0.9026 | 12.8 | -6.7 |
| Rapeseed | 37.0 | 37.6 | 39.7 | 246 | 0.9115 | -3.9 | -31.70 |
| Sesame | 35.5 | 40.2 | 39.3 | 260 | 0.9144 | -3.9 | -9.4 |
| Soya bean | 32.6 | 37.9 | 39.6 | 254 | 0.9138 | -3.9 | -12.2 |
| Sunflower | 33.9 | 37.1 | 39.6 | 274 | 0.9161 | 7.2 | -15 |
| Palm | 39.6 | 42.0 | 39.9 | 267 | 0.9180 | 31 | - |
| Jatropha | 48.09 | - | 38.96 | 258.5 | 0.9150 | - | - |
| Croton | 29.84 | - | 39.33 | 235 | 0.9100 | - | - |
| Moringa | 43.46 | - | 39.76 | 263 | 0.8971 | - | - |
| Polanga | 55.67 | - | 38.51 | 236.5 | 0.9510 | - | - |
| Diesel | 3.06 | 50 | 43.8 | 76 | 0.855 | -16 | - |

Source: H. M. Mahmudul et al., 2017).

The above data (table 6) is for crude oils only. The value varies for purified oils. For instance, the kinematic viscosity for crude moringa oil is 43 $mm^2/s$ while kinematic viscosity of purified moringa oil is 9 $mm^2/s$.

**2.5 Chemistry of Biodiesel production**

Biodiesel is a fatty acid methyl esters produced by reaction of triglycerides with light alcohols. Triglycerides are chemicals found in oil rich substances such as vegetable oils, plant seed oils and animal fats. They are known as lipids. The transesterification reaction takes place with catalyst or without catalyst. The fatty ester is released as product with the reformation of the OH group in glycerol. The overall reaction occurs in three stages is controlled by chemical equilibrium.




Triglyceride + Alcohol Biodiesel + Glycerol

Most transesterification reaction use ethanol or methanol alcohol. Many studies show that Methanol is more practically used alcohol than ethanol. Methanol is preferable due to its low cost and its physical and chemical advantageous. Another advantage of using methanol is the ease of separation of glycerin in which it can be achieved by simple decantation (Eman N. Ali and I.Tay, 2012).

### 2.6 Biodiesel production technologies

There are various production technologies of biodiesel. The most common production technologies are: Pyrolysis (thermal cracking), micro-emulsion and transesterification reaction. Transesterification reaction. Currently, biodiesel is manufactured at pilot scale and at industrial level by transesterification reaction method (S.Raj and M.Bahandari, 2018).

#### 2.6.1 Direct use and blending

Crude vegetable oils has been found not much encouraged to be used directly as diesel fuels in diesel engines. Hence, to improve the viscosity of vegetable oils for direct use or blending, it is mixed directly or diluted diesel fuel to use in diesel engines. Energy consumption in this case were found equivalent to Energy consumption in diesel fuels. The optimum used ratios of vegetable oil to diesel fuel were 1:10 – 2:10 and was found successful (Fukuda H et al., 2001). But at the end, the use of direct oils or blend of oils was not satisfactory with regard to direct or indirect usage as

diesel fuels due to the major problems encountered such as polymerization during storage and combustion, carbon deposits and lubricating oil thickening.

### 2.6.2 Micro-emulsions of oil

Micro - emulsion method of biodiesel production is defined as a colloidal equilibrium dispersion of optically isotropic fluid microstructures with dimension range of 1-150 nm formed from two normally immiscible liquids and one more ionic or non-ionic amphiphiles spontaneously. Fuels of this type are known as hybrid fuels. They exist as three components namely an oil phase, an aqueous phase and a surfactant. The maximum viscosity limitation for diesel engines can be met by micro-emulsions with butanol, hexanol and octanol (Jain S and Sharma MP, 2010).

### 2.6.3 Pyrolysis (thermal cracking)

Pyrolysis process is the conversion of organic compound or biomass in to another compound by using heat energy with or without the help of catalyst. Animal fats, vegetable oils, natural fatty acids or methyl esters of fatty acids can be processed by pyrolysis or can be taken as pyrolyzed material. In the case of animal fats and vegetable oils conversion, triglycerides play an important role to undergo a thermal cracking reactions (pyrolysis). Pyrolysis is a promising technology for biodiesel production. Pyrolysis (thermal cracking reactions) can be divided in to catalytic and non-catalytic reactions. Pyrolysis reaction requires expensive equipment for the process and all possibilities of more gasoline production than diesel fuel (Maher KD and Bressler DC, 2007).

### 2.6.4. Transesterification process

Transesterification is a reaction process that transforms triglycerides in oil rich substances into a mixture of fatty acid esters using alcohol and catalyst to speed up this reaction to the right side and to obtain biodiesel with high yields. Methyl or ethyl esters are obtained with much more similar properties to those of conventional diesel fuels as main product and glycerol as byproduct. The most common alcohol used for biodiesel production is methanol because of its low price and high conversion rates. Other alcohols such as plant based ethanol, propanol, isopropanol, and butanol also can be used. In presence of excess alcohol, the foreword reaction extends beyond the reverse reaction. Many catalysts could be utilized in the process; however, it was confirmed that transesterification is completed faster using an alkali catalyst. The mechanism of transesterification process shows some challenges such as limitation of reaction rate by mass

transfer between the immiscible oil and alcohol besides the reversibility of the transesterification itself which limits the conversion and consequently increases the reaction time and cost (M.F Elkady et al., 2015).

Transesterification is the reaction of triglycerides found in oil with alcohols such as methanol and ethanol in the presence of catalyst to give fatty acid methyl esters and glycerol. Fatty acid methyl ester (FAME) is the main product (biodiesel) while glycerol is byproduct.

After transesterification process, the mixtures of esters, glycerol, catalyst, alcohol and tri-di – mono glycerides are separated from the wanted product (biodiesel). Transesterification process brings variations in viscosity, boosts cetane number and drop in flash point. The yield of biodiesel produced is affected by aspects such as moisture content, free fatty acid (FFA), reaction time, reaction temperature, catalyst and molar ratio of alcohol to oil (S. Raj and M. Bhandari, 2017).

### 2.7 Catalysts for transesterification process and their role on yield of Biodiesel

Catalyst plays a significant role to fasten the rate of chemical reaction in transesterification process. Catalysts break the bond of triglyceride which is reacted with alcohol to separate fatty acid methyl ester from glycerol. Catalysts increase the yield of biodiesel by fastening the rate of the transesterification reaction. The main catalysts used for biodiesel production are categorized as chemical and biological catalysts. Chemical catalysts comprise of homogeneous catalyst, heterogeneous catalyst and super critical fluids (SCFs) (Thangaraj et al., 2018).

#### 2.7.1 Chemical catalysts

**A) Homogeneous catalysts**

Homogeneous catalysts include basic and acidic catalysts. The basic catalysts commonly used are NaOH and KOH since they are relatively cheap and widely available. It was reported that the rate of base catalyzed transesterification is 4000 times faster than that of the acid-catalyzed one. Currently, most biodiesel production is carried out using homogenous base catalysts of sodium hydroxide (NaOH) or potassium hydroxide (KOH). These two base catalyst are commonly used for the reasons: they are able to catalyze at lower reaction temperature and atmospheric pressure, high conversion (high biodiesel yield) can be obtained in a minimal time, cheap and available.

Now day, industrial scale biodiesel production use base catalysis (Eman N. Ali and Cadence I. Tay, 2012).

Transesterification can be conducted under homogeneous or heterogeneous catalysis. In homogeneous catalysis, the chemical reaction is faster and require lower catalyst loadings than in the heterogeneous one. However, the used catalyst cannot be reused or recycled. Therefore, it needs several washings for product separation (Lucia de Lima et al, 2016). Transesterification reactions that are homogeneously catalyzed for biodiesel production results with high yields in a relatively short time. However, biodiesel does not compete favorably with fossil fuels because of the catalyst cannot be reused (Colombo et al., 2016).

**B) Heterogeneous catalysts**

In heterogeneous catalysis, the catalyst and the reaction are in different phases. This has advantage of easily removal of the catalyst and its reuse without intense washing. However, leaching of the active phase may be a problem. Therefore, the synthesis of active reusable heterogeneous catalysts is a great challenge in biodiesel production. They are still not currently viable for wide industrial scale production, as most of these catalysts are expensive, lower activity and lower yield when compared to homogeneous catalysts (Lucia de Lima et al, 2016).

Environmental issue have led to search for solid heterogeneous catalyst since they are eco-friend and effective. The chemical process to produce biodiesel using solid heterogeneous catalyst in catalyzing the reaction has advantages of reducing time and cost of the process through the reuse of the catalyst, decreasing the level of impurities in the reaction products and conducting the operation in a continuous fixed bed (Colombo et al., 2016).

**C) Supercritical fluids (SCFs)**

Supercritical fluids are used to produce biodiesel through transesterification process in the absence of catalysts. Researchers have proposed biodiesel fuels could be produced from vegetable oils via non-catalyzed transesterification reaction with supercritical methanol. Supercritical techniques offer various advantageous such as easy separation of products, higher reaction rate, maximum yield and it is also environmentally benign compared to conventional catalytic techniques. It has also advantage of solving problems associated with the two phase nature of methanol and oil

mixtures by creating a single phase as a result of dielectric constant of methanol in the supercritical state (Thangaraj et al., 2018).

### 2.7.2 Biological catalysts (Enzymes)

Enzymes have received increased attention because of their high selectivity, biocompatibility, biodegradability, environmental acceptability and forming few or no by-products. Enzymatic transesterification has environmental advantage over other catalysts (Kalantari et al., 2013). However, enzymes are not widely used in commercial biodiesel production processes as they are very expensive and show longer reaction time (Lucia de Lima et al, 2016). Free lipase, traditional immobilized lipase, lipase immobilized on magnetic nanoparticles are commonly used biological catalysts. They have their own merit and demerit during their application. Among biological catalysts immobilized lipase are more preferable than others (Thangaraj et al., 2018).

## 2.8 Parameters that affect Biodiesel production

Biodiesel is produced from alcohol and triglyceride by transesterification reaction. During this reaction, there are different factors that affects this process such as alcohol to oil ratio, catalyst weight, reaction temperature, mixing intensity and time of reaction. These parameters affects the fuel property, quality and yield of biodiesel product (S.Raj and M.Bahandari, 2018).

### 2.8.1 Alcohol to oil ratio

The most effective variable that affects biodiesel production during transesterification reaction is molar ratio of alcohol to oil .Since transesterification is an equilibrium reaction, large and excess amount of alcohol is required for the reaction to move forward and avoid the reversible reaction. From the stoichiometry of transesterification reaction, 3 moles of alcohol reacts with 1 mole of triglycerides to yield 3 moles of fatty ester and 1 mole of glycerol. Since transesterification is an equilibrium reaction, large excess alcohol is required to accelerate the reaction. Lower molar ratio decreases reaction rate and longer time is needed to complete the reaction. However, excess molar ratio beyond the optimum level makes separation of glycerol difficult. In the case of transesterification of moringa oil, the range of alcohol to oil ratio is 10 wt. % of oil to 50 wt. % of oil (G. Kafuku and M.Mbarawa, 2010).

### 2.8.2 Catalyst loading (catalyst weight)

The most common catalysts used for transesterification reaction are the alkali catalysts such as sodium hydroxide and potassium hydroxides since they both react with the triglycerides of oil to break them apart so that methanol can bond with the fatty acids to produce fatty acid methyl ester (biodiesel). For moringa oil transesterification reaction the range of catalyst loading is 0.5 % to 1.5% of oil (G.Kafuku and M.Mbarawa, 2010).

### 2.8.3 Reaction temperature

Reaction temperature affects the yield biodiesel produced via transesterification process. Higher reaction temperature increases reaction rate by reducing viscosity of oils. This shortens the transesterification reaction and higher biodiesel yield is obtained. Lower reaction temperature decreases the rate of reaction and decreases the yield of desired product (biodiesel). However, the increase of reaction temperature beyond the optimum accelerates the saponification of triglycerides of oils which causes methanol to vaporize and results in decreasing the yield of biodiesel. To prevent the evaporation of alcohol (methanol), transesterification reaction temperature should be maintained below the boiling point of alcohol. In the case of biodiesel production from moringa seed, the transesterification reaction temperature could be maintained in the range of 30 °c to 60 °c. However, good oil yield is obtained after 40 °c (Ogbu et al., 2013).

## 2.9 Biodiesel as a sustainable fuel and its benefits

Biodiesel is a fuel that is technically feasible, economically competitive, environmentally friend, socially acceptable and easily available in the word. Biodiesel is insoluble in water. It has a physical appearance of light to dark yellow and clear liquid .Biodiesel fuel is attracting and increasing attention worldwide as blending component or direct replacement for the diesel fuel in diesel engine. Biodiesel comprises alkyl fatty acid (Chain length C14 – C22). This structure is esters of short chain alcohols, mainly methanol or ethanol. Biodiesel can be defined as a renewable fuel composed of mono - alkyl esters of long chain fatty acids derived from sustainable energy source such as vegetable oil and animal fats. It must meet the requirements of American Society of Testing and Materials (ASTM D 6751) standards and European Nations (EN 14214) standards (Siraj et al., 2013).

Some of technical properties of biodiesel are similar to that of fossil fuels and some are slightly different from that of fossil fuels. The density of biodiesel is in the range of 860 – 900 kg/m3 as per ASTM D 6751 standard and fossil fuel has 800 – 860 kg/m3 as per ASTM D 975 standard. Biodiesel has a kinematic viscosity of 1.9 – 6.0 mm2/s as per ASTM D6751 standard and 3.5 – 4.1 mm2 /s as per EN 14214 standards and for fossil fuel the range is 1.3 -4.1 mm2/s as per ASTM D6751 and 3.5 -5.0 mm2/s as per EN 14214 standards. The flash point of biodiesel is 130°*c* minimum as per ASTM D 6751 standard and for fossil diesel is 52°*c* as per ASTM D975 standard. The Cetane number is higher for biodiesel that is 47 minimum as per ASTM D6751 Standard and for fossil fuel 40 minimum as per ASTM D 975. The Higher Heating or Gross Heating Values of Biodiesel is relatively high in the range of 39 – 41 MJ/kg (Siraj et al., 2013).

*Table 6: Specification and Test Methods of Biodiesel as per ASTM D6751 & EN 14214 Standards.*

| Property | Units | Limits | | Test methods | |
|---|---|---|---|---|---|
| | | ASTM D6157 | EN 14214 | ASTM D6751 | EN 14214 |
| Flash points | °c | 130 min | 101 min | D 93 | ISO CD 3679e |
| Kinematic viscosity | mm2/s | 1.9 – 6.0 | 3.5 – 5.0 | D 445 | EN ISO 3104 |
| Cetane number | - | 47 min | 51 min | D 613 | EN ISO 5165 |
| Copper strip corrosion | - | No.3 max | Class 1 | D 130 | EN ISO 2160 |
| Acid value | mg KOH/g | 0.80 max | 0.5 max | D 664 | EN 14104 |
| Free glycerol | % (m/m) | 0.020 max | - | D 6584 | EN 14106 |
| Total glycerol | % (m/m) | 0.240 max | 0.25 max | D 6584 | EN 141101 |
| Carbon residue | % (m/m) | 0.05 max | 0.3 max | D 4530 | EN ISO 10370 |
| Cloud point | °c | | | D6950 | |
| Density at 15 °c | Kg/m3 | - | 860 – 900 | - | EN SIO 3675 |
| Sulfur content | mg/kg | - | 10 max | - | - |
| Water content | mg/kg | - | 500 max | - | EN ISO 12937 |
| Iodine value | - | - | 120 max | - | EN 14111 |

Source: Demirbas (2009)

# 3. Materials and Methods

## 3.1 Materials and chemicals used

The main chemicals and equipment used in this study were the following

*Table 7: Main chemicals and equipment required*

| Equipment | Purpose |
|---|---|
| Miller | To mill feed stock sample |
| Oven dry | To dry samples |
| Soxhlet extractor | To extract oil |
| Measuring cylinder | To measure volume of liquids |
| Vibrio viscometer | To measure viscosity |
| Furnace | To burn or heat samples |
| Refrigerator | To determines pour point |
| Digital balance | To measure mass |
| Heating mantle | To heat samples |
| Rotary evaporator | To separates Hexane from crude oil |
| Condenser | To condense (cool) methanol and to recover hexane |
| Water bath | To heat reactants during transesterification reaction |
| Stirrer | To agitate reactants |
| pH meter | To measure pH |
| Pycnometer | To determine specific gravity |
| Bomb calorimeter | To determine calorific value of biodiesel |
| Burette | Used for titration |
| Separator funnel | To separate crude biodiesel and glycerol |
| Thermometer | To measure temperature |
| | |
| **Chemicals** | **Purpose** |
| Hydrochloric acid (HCL) | Used as titrant for determining saponification value |
| Potassium hydroxide (KOH) | Used as catalyst for reaction |
| Methanol | Used as reactant |

| | |
|---|---|
| Ethanol | Used to dissolve oil during titration |
| Hexane | Used solvent for oil extraction |
| Acetone | For drying purposes |
| Sodium thiosulfate ($Na_2S_2O_3$ ) | Used as titrant for Iodine value determination |

## 3.2 Experimental methods

### 3.2.1 Raw material collection and preparation

The seeds of moringa stenopetala was collected at the harvesting time (in January) from Arba Minch which is located in Southern Nations, Nationalities and Peoples of Ethiopia (SNNP). The collected seed samples were transported to Addis Ababa University, where the study was conducted. The outer layer of the seed was removed to expose the kernel. The moisture of seed was removed by sun drying. The dried sample size was reduced by milling machine. Size reduction (agglomeration) is used to create high surface area for the reaction. After size reduction the sample was sieved to appropriate mesh size. The prepared sample was stored by packing in plastic bag for the next analysis.

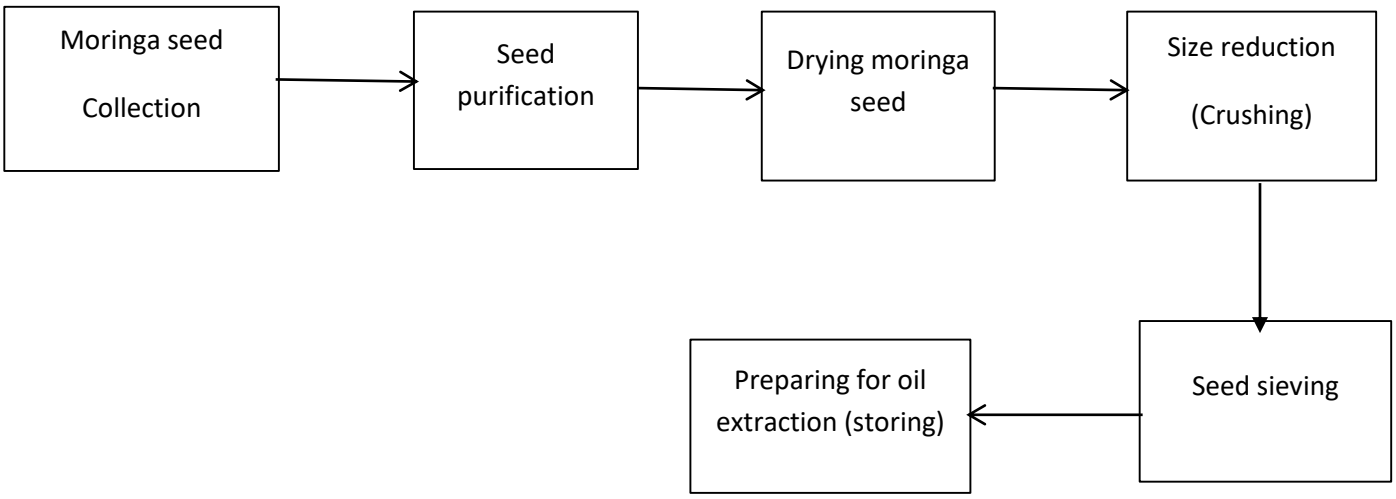



*Figure 1: Feed stock preparation*

### 3.2.2 Raw material (feed stock) proximate analysis

Proximate analysis is characterization that consists of determining physical and chemical properties which includes moisture, ash, volatile matter and fixed carbon content based on ASTM standards.

The proximate analysis of a substance is a simple means of determining the distribution of substances obtained when the sample is heated under specified conditions. As defined by ASTM D 121, proximate analysis separates the products into four groups: (1) moisture, (2) volatile matter, consisting of gases and vapors driven off during pyrolysis, (3) fixed carbon, the nonvolatile fraction of coal, and (4) ash, the inorganic residue remaining after combustion. Proximate analysis is the most often used analysis for characterizing a material in connection with their utilization.

**A) Determination of moisture content**

Moisture content (Mc) affects the quality and combustibility of the final product. High moisture decreases the heating (calorific) value of the product. Moreover, it forms glycerol and decreases yields of biodiesel during transesterification process. The appropriate range of moisture content of feed stock is 0.5 to 10% (A.K.Shaha, 2011). The moisture content of the sample was determined according to EN 14774-1. The sample was conditioned by putting it in desiccator for 24 hours to attain its equilibrium condition. 2g of the sample was oven dried for 2 hours at 105°C. After 2 hours drying, the sample was cooled in desiccator for 30 minutes and weighed. This was continued until the constant weight was registered.

The percentage weight was calculate as

$$Wt.\%\, moisture = \frac{W1 - W2}{W1} * 100 \quad \text{..... (1)}$$

Where; $W_1$ is the original weight of the sample before oven drying and
W2 is the weight of the sample after oven drying

**B) Determination of ash content**

Ash is the remained impurity after the burned fuel. High ash content affects the combustion efficiency of fuels by causing slagging and clinkering. Average ash content of the fuel is 3 % - 10 % (A.K. Shaha, 2010).

The ash content was determined according to EN 14775. 2 g of sample was weighed in crucible and burned at 650 °C for 2 hours in muffle furnace.

$$Ash, \% = \frac{W2}{W1} * 100 \ldots\ldots\ldots\ldots (2)$$

Where, W1 is weight of sample initially taken and W2 is weight of Ash cooling after heating

**C) Determination of volatile matter**

The content of volatile matter (VM) affects the ignitability of biofuels. Volatile matter was determined using high temperature furnace. 2 g of the sample was weighed in crucible and heated at temperature of 925 °C in a closed crucible for 8 minutes.

$$Volatile\ matter, \% = \frac{W1-W2}{W1} * 100\ \% \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Where, w1 is weight of sample before drying and W2 is weight of sample after drying

**D) Determination of fixed carbon**

Fixed carbon (FC) determines the estimate of heating value of fuels. Fuels that have high carbon content have higher flame length and easy to ignite. Carbon content of fuels was obtained by subtracting the percentage composition of ash, moisture and volatile matter from 100.

Fixed carbon content = 100 – (ash, % + M.c, % + V.M, %)…….( 4)

Where, ash, % = ash content

Mc, % = Moisture content and V.M, % = Volatile matter content

### 3.2.3 Extraction of Moringa seed oil

Extraction of moringa seed oil was carried out by soxhlet extraction method. The solvent used for the extraction is Hexane. The extraction was done using 500 ml capacity soxhlet apparatus using Hexane as a solvent. 80 g of sample was put in to timble and placed in the Soxhlet chamber.

400 ml Hexane was used for the extraction at different temperatures for 3 hrs to 5 hrs. The extraction was conducted at 70 °C, 75 °C and 80 °C by varying extraction time to get the maximum possible oil yield. As the solvent evaporates and passes through the sample, the oil is extracted and goes to the flask (Nur'Atiqah et al, 2015). Soxhlet extraction (solvent) extraction method was selected due to various factors. For instance, soxhlet extraction method results in high oil yield, simple and quick, solvent can be reused, and low cost (Bhuiya et al., 2016).

The main factors that affect oil extraction process are: Temperature, particle size, extraction time, moisture content of sample, solid to solvent ratio and type of solvent used. To have high oil yield the particle size should be maintained in the range of 0.2 mm to 5 mm). For this study the smallest particle size (less than 1 mm) was used since oil yield increases with decreasing particle size. As the particle size decreases, the high surface area is created for the extraction.

As time of extraction increases, the oil yield is increased and it starts after the peak value of time. The range of extraction time to obtain high oil yield is 3 hrs to 5 hrs since below 3 hrs and above 5 hrs, no more oil can be extracted (Tesfaye and Tefera, 2017). The maximum temperature at which high oil yield is obtained is in the range of 70 °C -80 °C since the boiling point of Hexane varies between 70 °C – 80 °C based on its product grade. Therefore, oil extraction was conducted at this range of temperature. The experiment of oil extraction was carried out at the following parameters.

*Table 8: Oil extraction range of parameters*

| Test No | Time (hrs.) | Temperature (°C) | Particle size (mm) | Oil yield (%) |
|---|---|---|---|---|
| 1 | 3 | 70 | < 1 | |
| 2 | 3 | 75 | | |

| | | | | |
|---|---|---|---|---|
| 3 | 3 | 80 | | |
| 4 | 4 | 70 | | |
| 5 | 4 | 75 | | |
| 6 | 4 | 80 | | |
| 7 | 5 | 70 | | |
| 8 | 5 | 75 | | |
| 9 | 5 | 80 | | |

$$Oil\ yield, \% = \frac{W1-W2}{W1} * 100 \ldots\ldots\ldots\ldots\ldots\ldots \text{(5)}$$

$$\text{Oil yield (\%)} = \frac{mass\ of\ oil\ extracted}{mass\ of\ sample\ used\ in\ feed} * 100 \ldots\ldots\ldots\ldots \text{(Iloamaeke at al., 2016)}$$

$$Oil\ yield(\%) = \frac{gram\ of\ oil\ extracted}{gram\ of\ sample\ used} * 100$$

W1 is original mass of sample before extraction

W2 is mass of sample after extraction

W1 –W2 is mass of oil extracted.

The range of the above factors was taken because high yield of oil is obtained in the range of these parameters.

### 3.2.4 Purification of extracted oil

The extracted oil contains undesired substances like: hexane, gum, moisture, wax substance and phospholipids. These unwanted substances decreases the quality of the oil as they increases formation of soap during transesterification process. The extracted oil was purified by the following selected methods (evaporation and degumming).

**A) Evaporation**

Evaporation process is used to separate n-Hexane from oil. The oil and solvent (n-Hexane) were separated by evaporating at 80 °C using rotary evaporator. The mixture of extracted oil and Hexane was separated based on their boiling point differences. The boiling point of Hexane varies from 70 °C – 80 °C while that of oil is greater than 200 °C. Hexane solvent is evaporated at 80 °C.

**B) Degumming**

Degumming is used separate the unwanted mixtures of gum and wax from extracted crude oil. There are two types of degumming methods. Acid degumming and water degumming. Water degumming was selected since it is cost effective. Distilled water was heated at 50 °C for 30 minutes. Then, about 1/3 ratio of hot distilled water to crude oil was mixed and stirred for 7 minutes. When the mixture is completely settled in separatory funnel, oil and unwanted substances were separated by decantation.

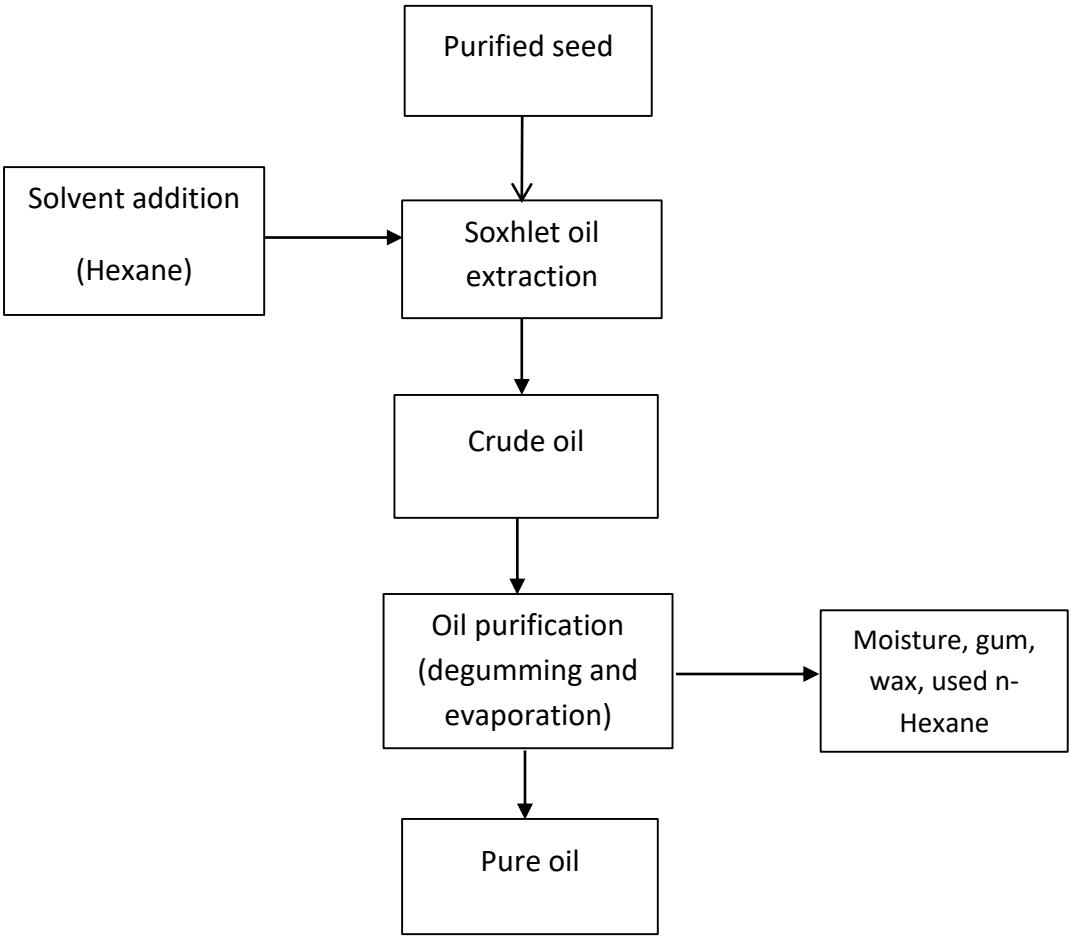



*Figure 2: Block flow diagram of soxhlet oil extraction*

### 3.2.5 Characterization of the oil (determination of physico-chemical properties)

A) ***Determination of moisture content***

Moisture content of the sample was determined according to EN 14774-1 standard. Empty dish was conditioned for 24 hours and weighed. 2 g of oil was taken on crucible and weighed. Then, the oil sample was oven dried for two hours until constant weight is obtained. Moisture content of sample was determined using equation (1).

$$Moisture\ content, \% = \frac{W1 - W2}{W1} * 100$$

Where, W1 = original mass before oven drying

W2 = weight after oven drying

**B) Determination of Acid value**

Acid value is the measure of free fatty acids (FFA) present in the extracted oil. Acid value is expressed in terms of milligram of base (KOH) per gram of oil to neutralize free fatty acids. Higher acid value reduces the quality of the oil since it cause corrosion to oil. 5 g of oil sample was dissolved in 250 mm conical flask with 25 mm ethanol. After adding few drops of phenolphthalein (2 drops) and boiled for few minutes (5 minutes), it was titrated with 0.1 N of KOH until the end point of colorless to pink was recognized.

The acid value is calculated using the following equation (Iloamaeke et al., 2016)

$$A.V = (V * N * 56.1)/W \ldots\ldots\ldots\ldots\ldots (6)$$

Where, V is volume expressed in milliliter of 0.1 N ethanol KOH solution

N is concentration of ethanol KOH solution

W is mass of oil taken in gram

**C) Determination of specific gravity**

A stopper density bottle (pycnometer) was filled with distilled water. The weight is taken after losing away any water drops on the bottle. After drying, the bottle is filled with the extracted oil and weighed.

The specific gravity was calculated as follows:

$$Specific\ gravity\ = \frac{a-c}{b-c} \text{ …….. (7)}$$

Where, c is weight of empty density bottle or pycnometer

a is weight of bottle with oil

b is weight of bottle with water

Density of oil (ρ) = (specific gravity ∗ density of water)

**D) Determination of kinematic viscosity**

Kinematic viscosity of oil was determined by vibrio - viscometer. The sample was put in the water thermostat until it reaches the equilibrium temperature of 20 °C. The tip of the vibrio-viscometer was inserted in the sample and measures the viscosity of the oil. Viscosity decreases as temperature increases. Hence, kinematic viscosity is the ratio of dynamic viscosity to the density of oil.

$$\mu = \frac{v}{\rho} \text{ …….. (8)}$$

Where μ=kinematic viscosity, mm2/s
N =dynamic viscosity of oil, mPa.s (measured by using vibrio-viscometer) and
P =density of oil, g/cm3

**E) Determination of density**

Density of oil was determined from the following relation

$$Density\ of\ oil\ (\rho)\ =\ (specific\ gravity\ *\ density\ of\ water) \text{ ……. (9)}$$

**F) Determination of saponification value**

Saponification value is the number of milligrams of base required for saponification. 3 g of the sample was weighed into a conical flask. 25 ml of 0.5 M KOH was added from burette by allowing it to drain for a definite period of time. Then a reflux condenser was connected to the flask and boiled gently for 30 minutes. The flask and condenser were allowed to cool and rinsed with little distilled water and the condenser was removed. 1ml of indicator (5 drops) was added and tittered with 0.5M HCl until the pink color is disappeared. Finally volume of titrant (V1) was recorded.

The blank was determined with the same quantity of KOH solution under the same conditions.

The final result was calculated using the following equation:

$$S.V = \frac{56.1 * N * (V2 - V1)}{W} \quad \ldots\ldots\ldots\ldots\ldots\ldots (10)$$

Where, W = Weight of oil taken

N = Normality of HCL solution

V1 = volume of HCL solution used in the test in milliliter

V2 = volume of HCL solution used in the blank in milliliter and S.V = Saponification value

**G) Determination of pH**

pH value is the measurement of acidity or basicity of oil

Standard pH meter was used to determine the pH of the oil

**H) Determination of free fatty acid (FFA)**

The content of free fatty acid of the oil was determined using the following equation

$$\% FFA = \frac{A.V}{2} \quad \ldots\ldots\ldots\ldots\ldots\ldots (11)$$

Where, A.V is acidic value of oil

**I) Determination of molecular weight**

Molecular weight of oil was determined according from the following equation according to Indhumathi et al., 2014.

$$Mwt = \frac{168300}{(S.V - A.V)} \quad \ldots\ldots\ldots\ldots\ldots\ldots (12)$$

Where, S.V = Saponification value of oil

A.V = Acid value of oil

### 3.2.6 Biodiesel production process and experimental design

There are many methods that have been used to produce biodiesel from various non-edible feeds tocks. These methods are pyrolysis, micro-emulsification, dilution, and transesterification. The most common way to produce biodiesel is transesterification process. Transesterification process is a chemical reaction between one mole triglycerides and three mole alcohol to separate fatty acid from glycerol to produce Fatty acid methyl ester (FAME) and glycerin in the presence or no catalyst. Alcohols used in transesterification include methanol, ethanol, propanol, butanol, and amyl alcohol. Methanol and ethanol are used most frequently used in both laboratory and the biodiesel industry scale. Methanol becomes the first choice for transesterification reaction as its low cost (Ulfah et al., 2017).

Now days, most of biodiesel is produced with base catalyzed reaction for several reasons such as it is a low temperature and low-pressure reaction. It results in high conversion and yield (Marchetti et al., 2017) with minimal side reactions and short reaction time. It is a direct conversion to biodiesel with no intermediate compounds. Hence, base catalysis was selected for this study. For this study catalyzed transesterification process was selected for biodiesel production from moringa oil seed. Methanol was selected alcohol due low cost and its popularity. The base catalyzed reaction was carried out between extracted oil sample and methanol. The major reaction parameters considered in this study were: Methanol to oil ratio, temperature and catalyst concentration. The other parameters, reaction time and mixing intensity were kept constant and set at 1 hr. and 500 rpm for all test runs according to (Mathiyazhagan, M, 2011).

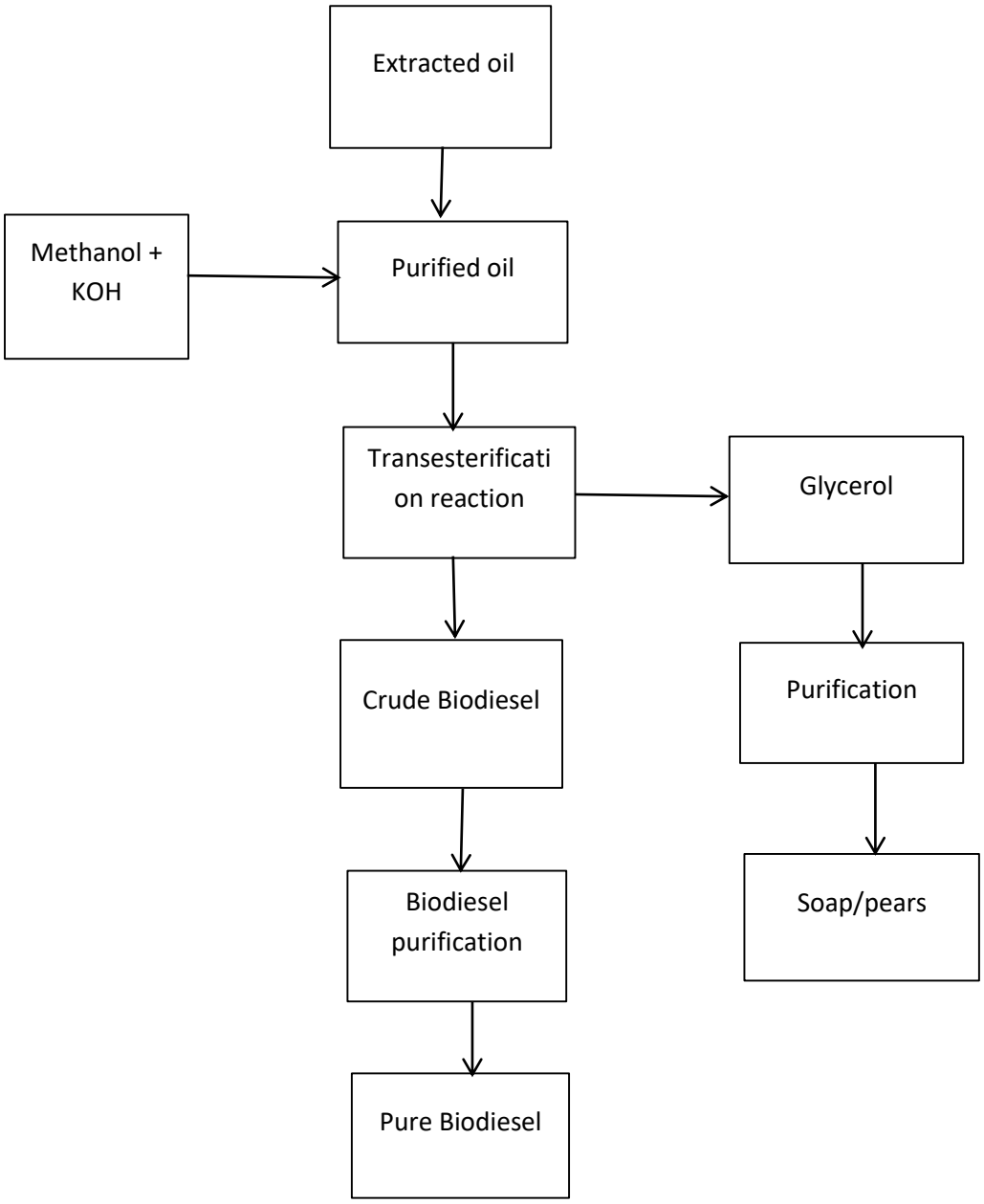



*Figure 3: Block flow diagram of Biodiesel production*

**Experimental set up and procedures of activity**

I. The experimental set up was set and adjusted
II. Methanol and catalyst was mixed in a beaker based on the provided methanol to oil ratio and catalyst concentration
III. KOH catalyst and methanol were well mixed in the beaker on water bath (5 °C) until complete solution was observed
IV. The purified moringa oil was added to methanol and catalyst solution in 500 ml three neck flask and stirred vigorously for one hour at 500 rpm by mechanical stirrer (reaction)
V. The mixture obtained from the reaction was settled for 24 hours in separating funnel
VI. The upper layer (biodiesel) was separated from glycerol, soap and other residuals and the quantity of biodiesel obtained was measured. The above procedure was repeated for all experimental runs by varying methanol to oil ratio, catalyst concentration and reaction temperature.

The catalyst concentration used in the experimental runs varies from 0.5 wt. % of oil to 1.5 wt. % of oil; the methanol to oil ratio varies from 10 wt. % of oil to 50 wt. % of oil (2: 1 to 10: 1); the temperature of the reaction varies from 40 °C to 65 °C. The reaction time varies from 30 min to 90 min and agitation speed varies from 200 rpm to 800 rpm. The optimal value of methanol to oil ratio, catalyst loading and reaction temperature is determined while for the remaining parameters, average value is taken and kept constant. Finally, the overall production is achieved using the optimal parameters obtained (G. Kafuku and M.Mbarawa, 2010). Finally, the overall production is carried out at the obtained optimum parameters. For this study, 0.5 – 1.5 Wt.%, 45 – 65 °C and 3:1 – 9:1 range of parameters were taken for catalyst load, temperature and methanol to oil ratio respectively.

**Experimental design**

The experimental design used for this study was Box-Behnken design standard. The response measured was the yield of biodiesel, fatty acid methyl esters (FAME). The experimental design was done using three-variable and three levels for response surface method. This enables to study the combined effects of catalyst concentration, methanol to oil molar ratio and reaction

temperature on the amount of biodiesel produced over three levels. Box-Behnken design is suitable for the exploration of quadratic response surfaces and generates a second-degree polynomial model and is used for optimization of process with small experimental runs.

*Table 9: Independent variables and level of main factors that affect transesterification reaction*

| Variables | Unit | Level | |
|---|---|---|---|
| | | -1 | +1 |
| Methanol to oil ratio | - | 3 | 9 |
| Catalyst loading | Wt.% | 0.5 | 1.5 |
| Reaction temperature | °C | 45 | 65 |

A combination of three factors, three levels and selected experimental runs by design expert was conducted. The following 17 experimental runs (table10) was selected by DESIGN EXPERT 11.0 using Box-Behnken standard design method where central points per block is five.

*Table 10: Box Behnken design Arrangement (experimental design matrix)*

| Run | Factor 1<br>A: Methanol to oil ratio | Factor 2<br>B: Temperature | Factor 3<br>C:Catalyst loading | Yield response (%) |
|---|---|---|---|---|
| 1 | 6 | 55 | 1.0 | |
| 2 | 6 | 65 | 0.5 | |
| 3 | 6 | 45 | 0.5 | |
| 4 | 3 | 55 | 1.5 | |
| 5 | 9 | 45 | 1.0 | |
| 6 | 9 | 65 | 1.0 | |
| 7 | 3 | 65 | 1.0 | |
| 8 | 6 | 55 | 1.0 | |
| 9 | 6 | 55 | 1.0 | |
| 10 | 9 | 55 | 0.5 | |
| 11 | 3 | 55 | 0.5 | |
| 12 | 6 | 55 | 1.0 | |

| | | | | |
|---|---|---|---|---|
| 13 | 3 | 45 | 1.0 | |
| 14 | 9 | 55 | 1.5 | |
| 15 | 6 | 45 | 1.5 | |
| 16 | 6 | 65 | 1.5 | |
| 17 | 6 | 55 | 1.0 | |

$$Yield = \frac{Weight\ of\ Biodiesel}{Weight\ oil\ used} * 100\%$$

The above range of parameters was taken because of best product with high yield was obtained within these ranges.

### 3.2.7 Separation (purification) process of biodiesel

After the transesterification reaction is completed, the product formed is the mixture of biodiesel, glycerin, alcohol, catalyst, water and other unreacted residuals. To separate biodiesel from this mixture, different separation techniques can be used.

When the reaction reached the preset reaction time, heating and stirring was stopped. All the products of the experiments were allowed to settle overnight for 24 hours to enhance separation of biodiesel and unwanted substances. Two distinct liquid phases were formed during separation, with the crude ester phase at the top and the glycerol phase at the bottom. The bottom glycerol phase was removed and the crude methyl esters layer at the top was then washed with 1/3 of warm deionized (distilled) water repeatedly until the water becomes clear.

The excess methanol and water in the ester phase was then removed by drying. Drying is conducted by heating the product to 105 -110 °C for some minutes until water is removed from the desired product (G. Kafuku and M.Mbarawa 2010). For this study, drying of biodiesel was conducted at 105 °C for 30 minutes using oven until some water evaporates and removed. After drying of final product is completed, the measurement was taken and yield of pure biodiesel was determined for each experimental runs.

The yield of biodiesel is the amount of purified biodiesel obtained per oil feed to the reactor. Yield of biodiesel can be calculated from the following equations:

a). Yield (%) = $\frac{gram\ of\ biodiesel\ produced}{gram\ of\ oil\ used} * 100$ (Pyushi et al., 2013)

b). Yield (%) = $\frac{Weight\ of\ biodiesl\ produced}{Weight\ of\ oil\ used} * 100$ (Iloamaeke et al., 2016)

c). Yield (%) = $\frac{Volume\ of\ biodiesel\ produced}{Volume\ of\ oil\ used} * 100$ (Jibril et al., 2012)

$$Yield, \% = \frac{Oil\ used}{Pure\ biodiesel} * 100$$

### 3.2.8 Characterization (determining physical and chemical properties) of biodiesel

#### A) Specific gravity

Specific gravity of biodiesel was determined according to EN 14214 standard. An improvised specific gravity bottle was washed and dried in the oven. The bottle was cooled at room temperature in a desiccators and the weight of the empty bottle was taken. Then, the bottle was filled with water and its weight was taken. Water was poured out and the bottle was rinsed with acetone and oven dried. Then, it was filled with biodiesel and weighed.

$$S.G = \frac{w3 - w2}{w1}$$

Where, W3 is weight of bottle and biodiesel

W2 is weight of empty bottle W1 is weight of equivalent volume of water (weight of water)

#### B) Kinematic viscosity

Viscosity is the measure of resistance to flow of fluids. Viscosity affects the quality of biodiesel. Kinematic viscosity is resistance to flow of fluid against gravity while dynamic viscosity (μ) is the ease with which the fluid flow. Dynamic viscosity of oil (biodiesel) was determined by viscometer according to ASTM D445 standard. Kinematic viscosity of biodiesel was determined using the following equation.

$$\nu = \frac{\mu}{\rho} \ldots\ldots\ldots\ldots\ldots\ldots (12)$$

Where, v is kinematic viscosity (mpa.sec)

ρ is density (kg/m3)

**C) Density**

Density of biodiesel was determined according to EN 14214 standard. Density of biodiesel influences the efficiency of fuel combustion. High density fuel has higher energy content (calorific value). Density of biodiesel was determined from the following equation

Density = specific gravity * density of water ……. (13)

**D) Saponification value**

3g of biodiesel sample was placed in a 250ml conical flask and 25ml of 0.5M ethanol potassium hydroxide solution added. A reflux condenser was attached and the flask content refluxed for 30 min on a water bath with continuous swirling until it simmered. The excess potassium hydroxide was titrated with 0.5 hydrochloric acid adding phenolphthalein indicator. A blank determination was carried out under the same condition and the saponification value was calculated from the following equation.

$$S.V = (B - R) * Mwt.C)/m \ldots\ldots\ldots\ldots\ldots\ldots (14)$$

Where, B = blank titration Mwt = molecular weight of KOH

R = real titration of oil (biodiesel) C = Concentration of KOH solution

m = weight of oil

**E) Acid value (acid number)**

Acid number is the amount of free fatty acid present in biodiesel. Acid value is expressed in milligrams of KOH base per gram of sample required to titrate to a specified point. Acid value was determined according to EN14104 standard. 5 gram of biodiesel was added to the mixture of 25 ml of ethanol and 1ml (5 drops) of phenolphthalein in 250 ml conical flask. The mixture was heated for about 7 minutes. Then, the mixture was titrated with 0.1 M of KOH with consistent

shaking until dark pink color is observed (colorless to pink). Acid value was determined using the following equation according to ASTM D664 standard.

$A.V = (V * C * Mwt)/m$ …….. (15)

Where, V = volume of titrated solution Mwt = molecular weight of KOH

C = concentration of KOH

m = mass of biodiesel taken

**F) Calorific value (High Heating Value)**

Heating value or calorific value (CV) is determined by burning biodiesel is determined by burning biodiesel in bomb calorimeter according to ASTM D240 protocol (E.I. Bello and F.Otu, 2014). Benzoic acid is used to standardize the calorimeter. 1 gram of sample was measured and taken in a crucible and made into a pellet, after taking the initial weight, it was placed in the bomb, which was pressurized to 18atm (18.2385 bar) of oxygen. Then, the bomb was placed in a vessel containing 2000 gram of water. The ignition circuit was connected and the temperature of water was taken. Ignition temperature rise was taken every minute until a constant temperature was attained. The pressure was released and finally, the length of unburned fuse wire was measured.

**G) Flash point**

Flash point of biodiesel was determined according to ASTM D93 standard. 40 ml of the biodiesel was poured into an open metal cup and put on heat source. A thermometer was put into it by clamping it to a retort stand. A test flame applicator was used to pass flame across it with continuous smooth motion and the temperature at which the sample ignited was recorded as the flash point with the help of the thermometer.

**H) Free fatty acid value (FFA)**

Free fatty acid of biodiesel was determined from acid value from the following equation.

$\% FFA = (acid\ value)/2$ …….. (16)

**I) Determination of pH**

pH is the measure of acidity or basicity of sample which is measured by pH meter (Orhevba et al., 2014). 30 ml of the biodiesel sample was poured into 50 ml beaker. The standard pH electrode with buffer solution and the electrode was immersed into the sample and the pH value was taken and recorded.

**J) Determination of moisture content**

Moisture content of biodiesel was determined according to EN 14774-1 standard method. Moisture content of pure biodiesel was determined by oven drying process according to ASTM standard. Empty crucible was desiccated for 24 hrs and weighed. 3 gram of biodiesel was weighed on crucible and dried in oven at 105 °C for two hours until constant weight was obtained.

$$Mc,\% = \frac{W1-W2}{w1} * 100 \ldots\ldots\ldots\ldots\ldots (17)$$

Where, W1 = weight before drying

W2 = weight after drying

**K) Determination of Iodine value**

Iodine value was determined according to EN 14111standard. 0.3 g biodiesel was dissolved with 20 ml of Carbon tetra chloride ($CCl_4$) in 250 ml conical flask. 20 ml of 15 % KI and 100 ml distilled water was added after one hour. Then, it was titrated against 0.1 M Sodium thiosulfate by adding 2ml indicator until yellow color was disappeared. Iodine value was calculated using the following equation according to Atabani et al., (2012).

$$I.V = ((B-S) * 0.1 * 12.69))/m \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (18)$$

Where, B = Blank titration

S = Sample titration and m = molecular weight of Iodine

**L) Determination of Cetane number**

Cetane number is a relative measure of interval between the beginning of injection and ignition quality of fuel. Theoretically, Cetane number is defined in the range of 15 -100. The minimum value of cetane number of biodiesel is 47 according to ASTM D6751.The higher Cetane number indicates that it takes shorter time between injection of fuel and its ignition in engine. Cetane number of biodiesel was determined using the following empirical formula.

$$C.N = 46.3 + \frac{54.58}{S.V} - 0.225 * I.V \ldots\ldots\ldots\ldots\ldots (19)$$

Where, S.V = saponification value

I.V= Iodine value

C.N = Cetane number

**M) Determination of fire point**

Fire point is the temperature at which the fuel starts to create a fire. Fire point of biodiesel was determined using the same procedure with that of flash point determination.

**N) Determination of cloud and pour point**

Cloud and pour point are properties of fuels that determines their performance at low temperature to identify whether the flue can flow before it changed in to cloud of crystal wax. The solidification of fuels can block fuel filters, fuel lines and damage fuel engine due to insufficient lubrication. Biodiesel has higher pour and cloud point than diesel fuels because of its higher fatty acid. This makes the performance of biodiesel in cold weather conditions lower (Das I.M and Agarwal A.K, 2001).

Cloud point is the temperature at which the first crystal wax of biodiesel is formed when cooled. Cloud point was determined according to ASTM D6950. To determine cloud point of biodiesel, 40 ml of biodiesel was measured and poured in to empty jar. Then, the jar was sealed by cork and thermometer was inserted in to the sample through small hole. The sample was put on water bath and cooled for minutes. The temperature of the biodiesel when the first cloud crystal wax was observed was measured.

Pour point is the temperature at which the liquid form of sample starts to flow or stop to flow. Pour point was determined according to ASTM D5771. To determine the pour point of biodiesel, 40 ml of biodiesel was placed in empty jar and sealed by cork after inserting thermometer in it. Then, the jar was placed in refrigerator. The temperature was inspected at five minutes interval by fitting it horizontally. The temperature of the sample was taken when the surface of biodiesel does not sag for five seconds.

### 3.2.9 Statistical analysis of experimental results

Data analysis of this study was carried out by DESIGN EXPERT 11.0.0 software using Box-Behnken standard design method with three factors, three levels and five center points per block to determine the experimental results that maximize the synthesis of biodiesel from the different reaction conditions such as temperature, catalyst loading and methanol to oil ratio. To get maximum conversion, the reaction speed and time were kept constant at 500 rpm and 1 hr. respectively. The transesterification process parameters were optimized using Response Surface Method (RSM) and the significance of the experimental result was determined from analysis of variance (ANOVA).

The individual effect of process parameters on yield of biodiesel was analyzed both numerically and graphically using Box Behinken design method. The response surface plot of experimental variables was also discussed using 3D plot. The interaction effect of the process parameters on the yield of biodiesel; effect of methanol to oil ratio and temperature at constant catalyst loading, effect of methanol to oil ratio and catalyst loading at constant temperature and effect of temperature and catalyst loading at constant methanol to oil ratio were discussed using 3D plot.

# 4. Results and Discussions

## 4.1 Feed stock proximate analysis

*Table 11: Feed stock proximate analysis*

| No | Property | Result (%) |
|---|---|---|
| 1 | Moisture content | 3.12 |
| 2 | Ash content | 4.52 |
| 3 | Volatile matter | 76.73 |
| 4 | Fixed carbon | 15.63 |

### Ash content

The average ash content obtained (4.52 %) agreed with the range of biomass ash content 3 – 10 % according to Shaha, 2010.

### Moisture content

Higher moisture content affects the fuel quality of biodiesel. It decreases the calorific value and yield of biodiesel by forming more glycerol. The final average moisture content of the sample (3.12 %) agreed with the literature value; 0.5 - 10 % which was reported by Shaha, 2010.

### Volatile matter

Volatile matter affects the ignitability of the biodiesel. The average volatile matter content of the sample (76.73 %) agreed with literature value and range of volatile matter of biomass which is 70 -80 % according to B.M .Jenk et al., (1989).

### Fixed carbon

The percentage of fixed carbon was obtained by subtracting the percentage of ash content, moisture satisfied and volatile matter from 100. The result was 15.63 %. This result indicated that the feed stock has good fuel properties and is promising resource for biofuel production since it has high carbon content, low ash and moisture content.

### 4.2 Determination of yield of oil extracted from moringa seed

The maximum oil yield was obtained at 80 °C and 5 hours extraction time and the yield was 39.86 %. This oil yield agreed with the literature data which was 35% - 45 % oil yield according to Ayerza, 2012. As extraction time and temperature increased, the oil yield also increased. The average oil yield 39.86 % obtained also agreed with 37 % which was reported by Orhevba et al., (2013) and 38 % moringa oil yield which was reported by A.K Azad et al., (2014).

### 4.3 Physicochemical properties of extracted oil

#### Moisture content

Moisture content of oil was determined by drying 3 g oil in oven at 105 °C for two hours and weighing it repeatedly until constant weight was obtained. The result of moisture was 6.54 %. This result agreed with the previous work of Schinas P.et al (2008) which was 5 %. Higher moisture content forms more soap during transesterification reaction and it is difficult for separation process. Moreover, higher moisture of oil decreases the yield of biodiesel produced.

#### pH

The pH of oil was determined with pH meter and the result was 6.7. This agreed with the range of MSSO pH which is 5 – 7. Lower pH value makes the oil more acidic which creates corrosion and higher pH value makes the oil more basic and this makes soap during transesterification reaction. Formation of more soap on the other hand decreases yield and quality of biodiesel. The result obtained was good since it meets the requirement for production of quality biodiesel and also agreed with the previous report of Orhevba et al (2013) which was 5.9

#### Kinematic viscosity

The dynamic viscosity of purified oil 7.9 mpas was obtained from experimental result. Kinematic viscosity of oil was determined using equation (8) and the result was 9.4 mm2/s .This result agreed with the previous report of H.M Mahamudul (2017) which was 9 mm2/s. Viscosity of oil affects the storage and handling of fuel oil. Highly viscous oil is difficult for pumping and to operate in CI engine. In this study, the result showed that moringa stenopetala oil has lower viscosity and it can be used in CI engines.

**Specific gravity (S.G)**

Specific gravity of oil was determined using 50 ml pycnometer or density bottle. The result obtained was 0.840 g/mol. This result agreed with literature value.

**Density (ρ)**

The density of oil was 840 kg/m3. This value is in range of density of vegetable oils (600 -900 kg/m3).

**Acid value (A.V)**

Acid value of oil was 1.63 mg KOH/g oil. This result agreed with literature value. The maximum acid value for alkaline transesterification of oil is two. If acid value is greater than this value, it needs further acid treatment method since it affects the fuel quality of biodiesel produced from it. Acid value is the measure of number of carboxylic acid groups such as free fatty acid.

**Saponification value (S.V)**

The saponification value of oil was 189.2 mg KOH/g oil. Higher saponification value of oils is good for production of quality biodiesel. The saponification value of moringa stenopetala oil is 175 -195 mg alcohol/g oil. This result agreed with literature value.

**Free fatty acid**

Free fatty acid of oil was 0.815 % .This result agreed with literature value.

**Molecular weight of oil**

Molecular weight of oil which was calculated from equation (12) was 895.5 g/mol. This result meets the range of molecular mass of vegetable oils (600 -900 g/mole).

Generally, acid value, free fatty acid, saponification value, specific gravity of moringa seed oil agreed with the previous report of G.Kafuku et al (2010) which was 1.2 mg KOH/g, 0.6%, 192 mg KOH/g, 0.90 g/mole respectively.

### 4.4 Determination of yield of Biodiesel from transesterification reaction

The maximum yield of biodiesel was 94.0 % and it was obtained at reaction temperature of 55 °c, catalyst loading of 1.0 % and methanol to oil ratio of 6:1. The yield of biodiesel increased as reaction temperature, catalyst loading and methanol to oil ratio increases until optimum point. Then, the yield decreased as these parameters increased beyond peak value. Lower value of these process parameters also decreased the yield of the biodiesel.

### 4.5 Physicochemical properties of Biodiesel

#### Specific gravity or Density

Specific gravity of biodiesel obtained was 0.874 g/mol. This value was in range of standard value of 0.86 – 0.9 set by EN 3675. It also agreed with 0.85 g/mole which was reported by Bala BK (2005). Density affects fuel quality and fuel injection system. Higher density fuels are highly pressurized (atomized) and fuel pumps can easily inject higher mass of biodiesel.

#### Kinematic viscosity

Kinematic value of biodiesel was 4.8 mm2/s. This value agreed with the range of standard ASTM D445 which was 1.9 – 6.0 mm2/s. Low viscosity fuels cannot provide good lubrication of fuel injection pumps and cause leakage and wear. On the other hand, higher viscosity fuels forms large droplets in fuel injection and cause poor combustion, exhaust smoke and waste emissions.

#### Acid value

Acid value of biodiesel was 0.4 mg KOH/g. This value agreed with the range of international standard 0.8 maximum set by ASTM D664. Acid value is the measure of number of carboxylic groups such as free fatty acids. Higher acid value forms corrosion of fuel pumping system and internal combustion engines. Lower acid value has lower free fatty acid.

#### Iodine value

The Iodine value of biodiesel was 104.5g I2/100g. Since the maximum Iodine value of biodiesel is 120 g I2/100g, this result agreed with the standard specification EN 14111. Iodine value of biodiesel affects oxidation stability, polymerization and degree of unsaturation. Highly saturated fuels form deposits in internal combustion engines.

**Calorific value**

Calorific value determines the ease of flammability of fuels. Thus, it is used as a regulation mechanism for transport and storage of fuels. Calorific value of biodiesel was 43 MJ/KG. This value is above the standard ASTM value which is 39 - 42.5 MJ/KG. This result indicated that MSSO biodiesel has the highest calorific value of all vegetable oils and almost equal value with that of diesel fuel calorific value; 43.8 MJ/KG. This result also agreed with the previous report of H.M Mahamudul (2017) and Siraj et al (2013) which was 39 MJ/Kg.

**Pour and cloud point**

Pour and cloud point of biodiesel were 1 °c and 10 °c respectively. The cloud point of many feed stocks' biodiesel is less than 14 °c according to ASTM D6950.This result showed that biodiesel produced meets the range of ASTM D5771 and ASTM D6950 standards respectively. This result agreed with the previous report of (G.Kafuku and Mbarawa, 2009) which was 3°c and 10 °c for pour and cloud point respectively. Higher cloud and pour points of biodiesel is the major complications for operating in cold weather conditions.

**Flash and fire point**

Flash and fire point of biodiesel were 184°c and 190 °c respectively. Flash and fire point of fuels are determinants for the flammability, storage and transportation of fuels. This result agreed with the ASTM D93 (minimum flash point of biodiesel is 130 °c).

Physicochemical properties of biodiesel produced was summarized in the following table

Table 12: Biodiesel physicochemical properties result.

| Property | Result | Unit | Standard method |
|---|---|---|---|
| Moisture | 1.4 | % | |
| Ph | 7.9 | - | |
| Density at 15 °C | 874 | Kg/m3 | EN 3675 |
| Kinematic viscosity at 22 °C | 4.8 | mm2/s | ASTM D445 |
| Acid value | 0.4 | mg KOH/g | ASTM D664 |
| Saponification value | 196 | mg KOH/g | |

| | | | |
|---|---|---|---|
| Free fatty acid | 0.2 | % | |
| Iodine value | 104.5 | g $I_2$ /100 g | EN 14111 |
| Calorific value | 10,287 | Cal/g | |
| Flash point | 184 | °C | ASTM D93 |
| Cetane number | 53 | - | |
| Fire point | 190 | °C | ASTM D93 |
| Specific gravity | 0.874 | g/mole | |
| Cloud point | 10 | *°c* | *ASTM D6950* |
| Pour point | 1 | *°c* | *ASTM D5771* |

The above result (table14) agreed with ASTM D6157 and EN 14214 standards of biodiesel. Moisture content, PH, density, Kinematic viscosity, acid value, saponification value and free fatty acid of biodiesel were determined following the same procedure with oil while Iodine value and cetane number were determined using equation (18) and (19). The value and Cetane number was 104.5 g I2/100 g and 53 respectively.

The above physicochemical properties of Biodiesel agreed with ASTM and EN standard values.

### 4.6 Statistical analysis of experimental results

#### 4.6.1 Analysis of variance (ANOVA) for quadratic model

Table 13: Analysis of variance (ANOVA) for quadratic model

| Source | Sum of Squares | Df | Mean Square | F-value | p-value | |
|---|---|---|---|---|---|---|
| Model | 1316.65 | 9 | 146.29 | 380.98 | < 0.0001 | Significant |
| A-Methanol to oil ratio | 35.15 | 1 | 35.15 | 91.55 | < 0.0001 | |
| B-Temperature | 332.56 | 1 | 332.56 | 866.06 | < 0.0001 | |
| C-Catalyst loading | 7.55 | 1 | 7.55 | 19.65 | 0.0030 | |
| AB | 0.6972 | 1 | 0.6972 | 1.82 | 0.2198 | |
| AC | 0.3481 | 1 | 0.3481 | 0.9065 | 0.3727 | |
| BC | 0.0380 | 1 | 0.0380 | 0.0990 | 0.7622 | |
| $A^2$ | 393.68 | 1 | 393.68 | 1025.23 | < 0.0001 | |

| | | | | | | |
|---|---|---|---|---|---|---|
| $B^2$ | 458.66 | 1 | 458.66 | 1194.44 | < 0.0001 | |
| $C^2$ | 16.58 | 1 | 16.58 | 43.18 | 0.0003 | |
| Residual | 2.69 | 7 | 0.3840 | | | |
| Lack of Fit | 2.18 | 3 | 0.7259 | 5.69 | 0.0631 | not significant |
| Pure Error | 0.5101 | 4 | 0.1275 | | | |
| Cor Total | 1319.33 | 16 | | | | |

Factor coding is coded**.**

Sum of squares is Type III - Partial

The **Model F-value** of 380.98 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

**P-values** less than 0.0500 indicate model terms are significant. In this case A, B, C, $A^2$, $B^2$, $C^2$ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 5.69 implies there is a 6.31% chance that a Lack of Fit F-value this large could occur due to noise. Lack of fit is bad -- we want the model to fit. This relatively low probability (<10%) is troubling.

**Fit Statistics**

***Table 14: Model adequacy measures***

| | | | |
|---|---|---|---|
| Std. Dev. | 0.6197 | $R^2$ | 0.9980 |
| Mean | 83.28 | Adjusted $R^2$ | 0.9953 |
| C.V. % | 0.7441 | Predicted $R^2$ | 0.9730 |
| | | Adeq. Precision | 59.4041 |

The **Predicted $R^2$** of 0.9730 is in reasonable agreement with the **Adjusted $R^2$** of 0.9953; i.e. the difference is less than 0.2.

**Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 59.404 indicates an adequate signal. This model can be used to navigate the design space.

**Coefficients in Terms of Coded Factors**

*Table 15: Regression coefficients and significance of response surface quadratic model*

| Factor | Coefficient Estimate | df | Standard Error | 95% CI Low | 95% CI High | VIF |
|---|---|---|---|---|---|---|
| Intercept | 93.67 | 1 | 0.2771 | 93.02 | 94.33 | |
| A-Methanol to oil ratio | 2.10 | 1 | 0.2191 | 1.58 | 2.61 | 1.0000 |
| B-Temperature | 6.45 | 1 | 0.2191 | 5.93 | 6.97 | 1.0000 |
| C-Catalyst loading | 0.9713 | 1 | 0.2191 | 0.4532 | 1.49 | 1.0000 |
| AB | 0.4175 | 1 | 0.3098 | -0.3151 | 1.15 | 1.0000 |
| AC | -0.2950 | 1 | 0.3098 | -1.03 | 0.4376 | 1.0000 |
| BC | 0.0975 | 1 | 0.3098 | -0.6351 | 0.8301 | 1.0000 |
| $A^2$ | -9.67 | 1 | 0.3020 | -10.38 | -8.96 | 1.01 |
| $B^2$ | -10.44 | 1 | 0.3020 | -11.15 | -9.72 | 1.01 |
| $C^2$ | -1.98 | 1 | 0.3020 | -2.70 | -1.27 | 1.01 |

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-colinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

### *4.6.1.2 Development of regression model equations*

**I). Final Equation in Terms of Coded Factors**

$$Yield = +93.67 + 2.10 * A + 6.45 * B + 0.9713 * C + 0.4175 * AB - 0.2950 * AC + 0.0975 * BC - 9.67 * A2 - 10.44 * B2 - 1.98 * C2$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. Thus, the above equation correlates the response (yield of biodiesel) to transesterification process variables in terms of coded factors.

Where, A = Methanol to oil ratio

B = Temperature

C= Catalyst loading

**II). Final Equation in Terms of Actual Factors**

Yield = -305.77250 + 13.02267 * Methanol to oil ratio + 12.02245 *Temperature +17.92600 * Catalyst loading +0.013917 *Methanol to oil ration * Temperature - 0.196667 *Methanol to oil ratio * Catalyst loading - 0.019500 *Temperature *Catalyst loading - 1.07439*Methanol to oil ratio $^{2}$ - 0.104370 *Temperature $^{2}$ - 7.93800 *Catalyst loading $^{2}$.

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

### *4.6.1.3 Diagnosis of Model adequacy test*

As shown in the following figure (18), shows normal probability distribution. The points on the graph are fitted to the straight line. This indicates that the quadratic model satisfies the assumption of analysis of variance. The distribution of the error is approximately normal.

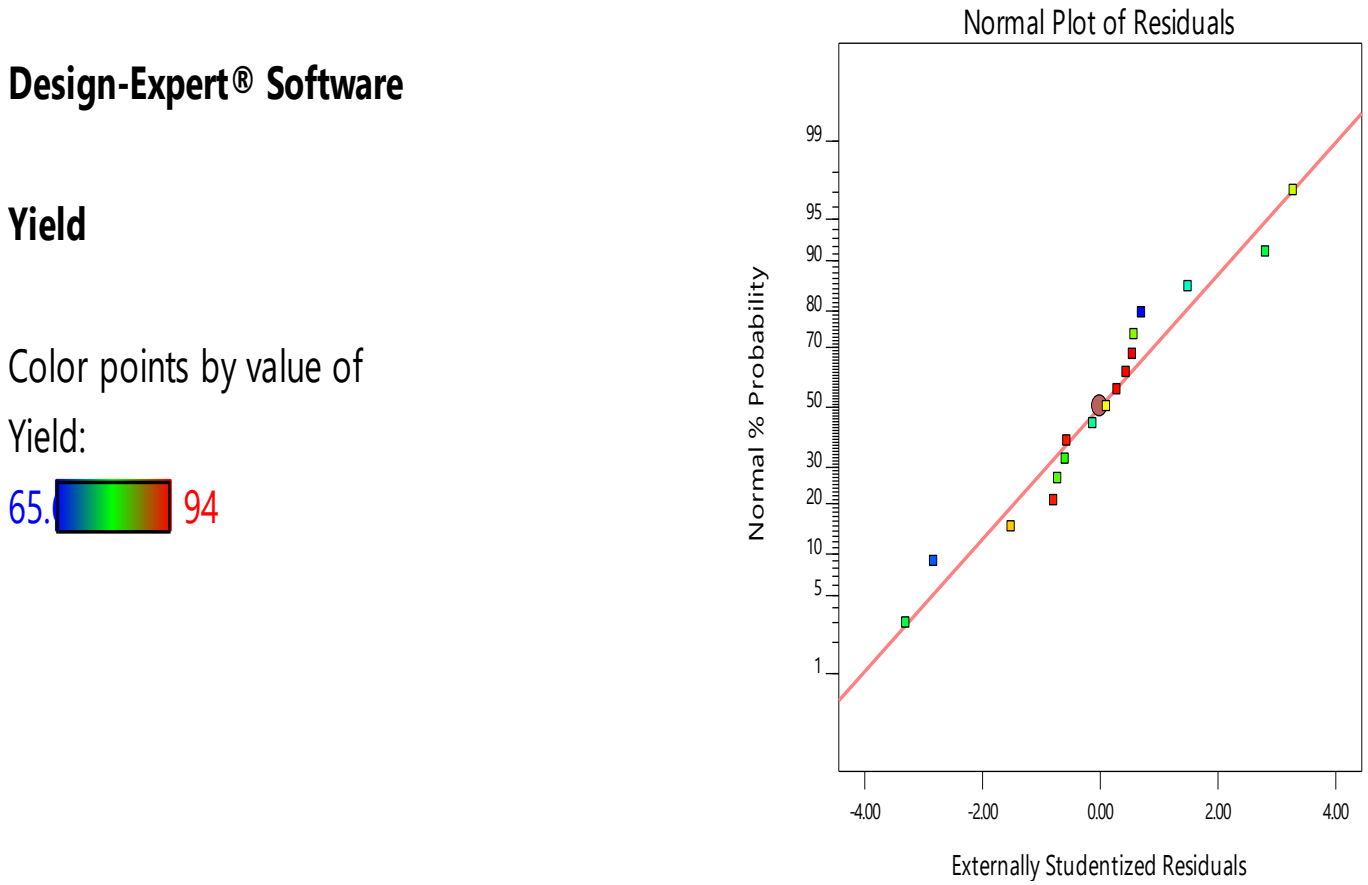



*Figure 4: Normal probability plot of residuals*

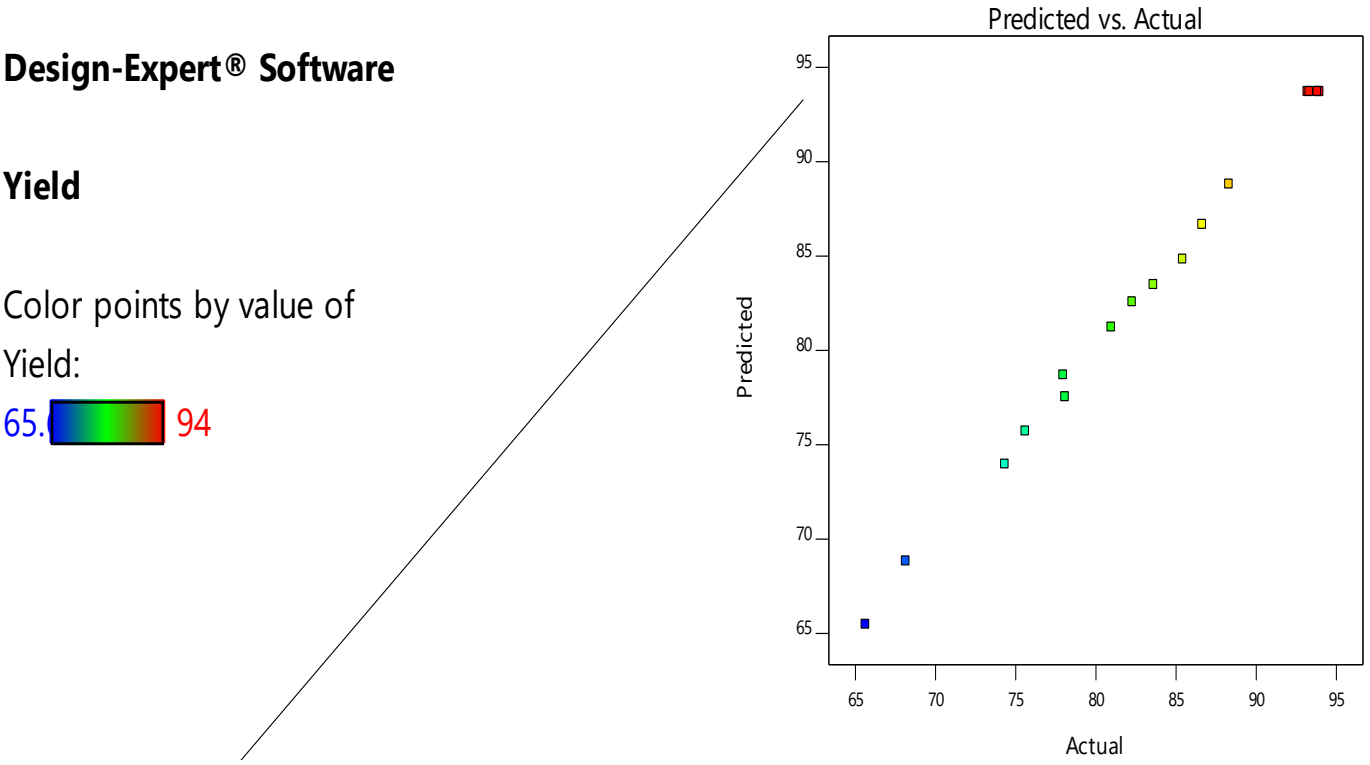



*Figure 5: Predicted versus actual value plot*

The graph of Predicted versus actual plot (fig. 18) is the correlation between actual experimental data and predicted value. The line on the plot has a unit slope (a line of perfect fit.). The points on the line corresponds to zero error between actual and predicted value. Therefore, the regression model equation granted accurate description of experimental data.

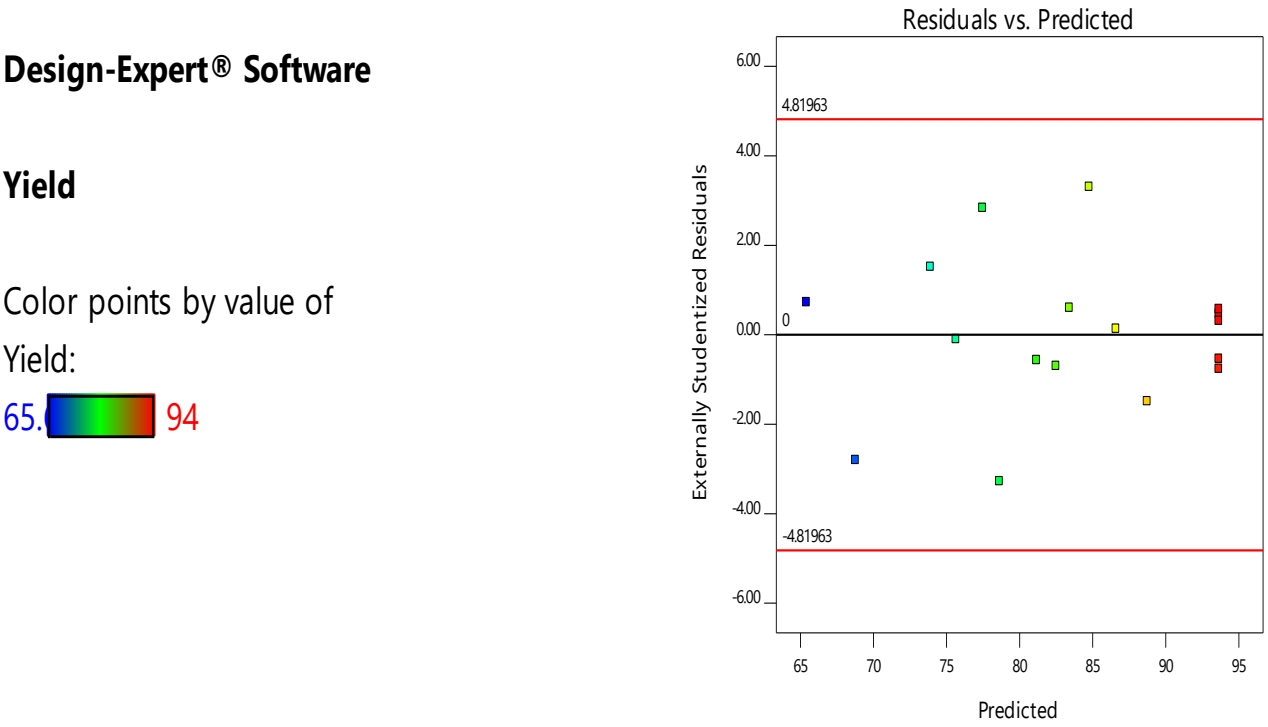



*Figure 6: Residual versus predicted value plot*

Residuals versus predicted value plot shows that the residuals are less structured and they are unrelated to any of other variables including the predicted value. Hence, figure (20) showed a random scatter distribution of points and justifying that no need for alteration of minimizing personal error.

### 4.7 Individual effect of experimental variables on yield of biodiesel

#### A) Methanol to oil ratio

Methanol to oil ratio is one of the main factors that affect transesterification reaction. Figure. (16) Shows the effect of methanol to oil ratio on biodiesel yield at constant temperature and catalyst loading. It indicated that the yield of biodiesel increases as methanol to oil ratio increases. Increasing methanol to oil ratio beyond optimum value results in excess methanol which decreases the yield of biodiesel and increase the cost of post -production treatment. Furthermore, excess methanol makes the separation process difficult. The maximum yield of biodiesel (94.0 %) was obtained at methanol to oil ratio of 6: 1. Therefore, this ratio was taken as an optimum value of methanol to oil ratio. On the other hand, decreasing methanol to oil ratio below the optimum value

(3:1), decreased the conversion efficiency of the transesterification reaction and resulted in lower biodiesel yield.

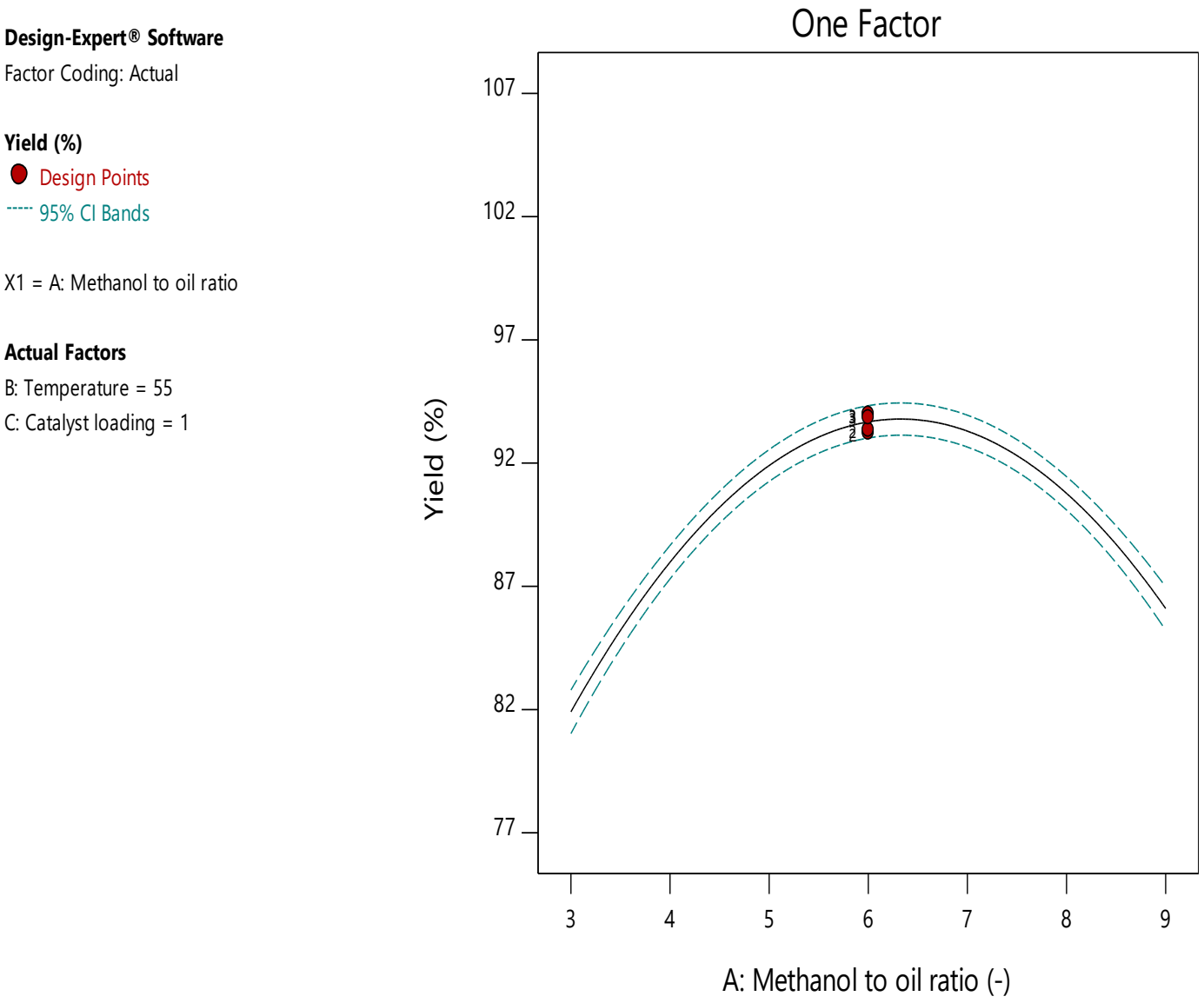



*Figure 7: Effect of methanol to oil ratio on yield of biodiesel*

**B) Effect of Temperature**

Figure (17) shows the effect of reaction temperature on yield of biodiesel at constant methanol to oil ratio and catalyst loading. The rate of transesterification reaction is highly influenced by reaction temperature. The rate of the reaction and the yield increased as temperature increased from 45 °C to 55 °C. Maximum yield (94 %) was obtained at 55 °C and the yield started to

decrease slightly from 55 °C to 65 °C due to the vaporization of methanol results in soap formation. The boiling point of methanol is 64.7 °C. Increasing reaction temperature above this point leads to methanol loss by evaporation and decreases the conversion efficiency of reaction. The lowest biodiesel yield (65.67 %) was obtained at lowest temperature of 45 °C.

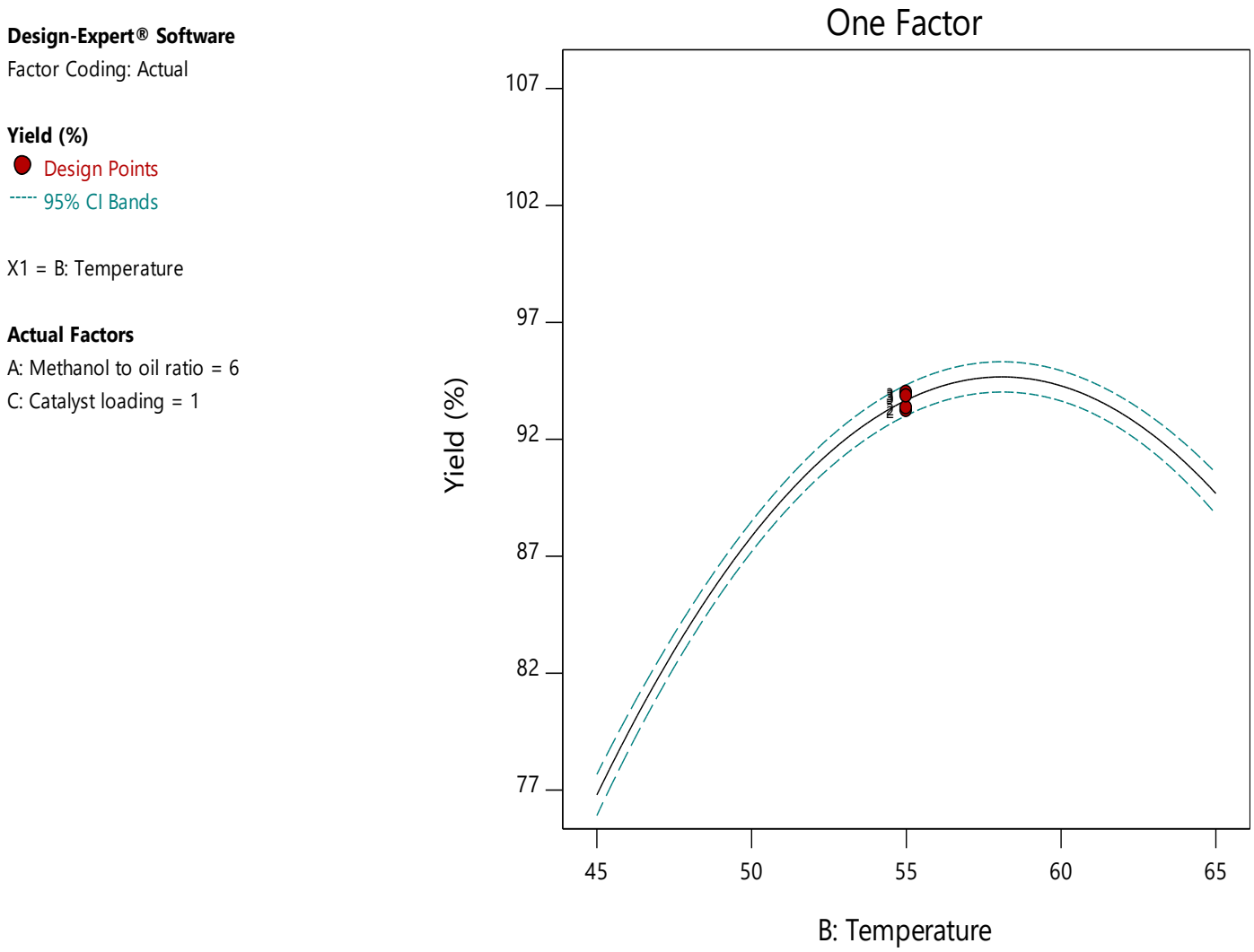



*Figure 8: Effect of temperature on yield of biodiesel*

### C) Effect of catalyst loading

Figure (18) shows the effect of catalyst loading on yield of biodiesel at constant methanol to oil ratio and temperature. As the amount of catalyst increased from 0.5 % to 1%, the yield of biodiesel increased from 89% to 94 % and the yield starts to decrease slightly as amount of catalyst increased from 1.0 % to 1.5% due to the formation of soap and emulsion. At lower amount of catalyst (0.5%),

the yield of biodiesel decreased due to incomplete conversion of triglycerides of oil to fatty acid methyl ester.

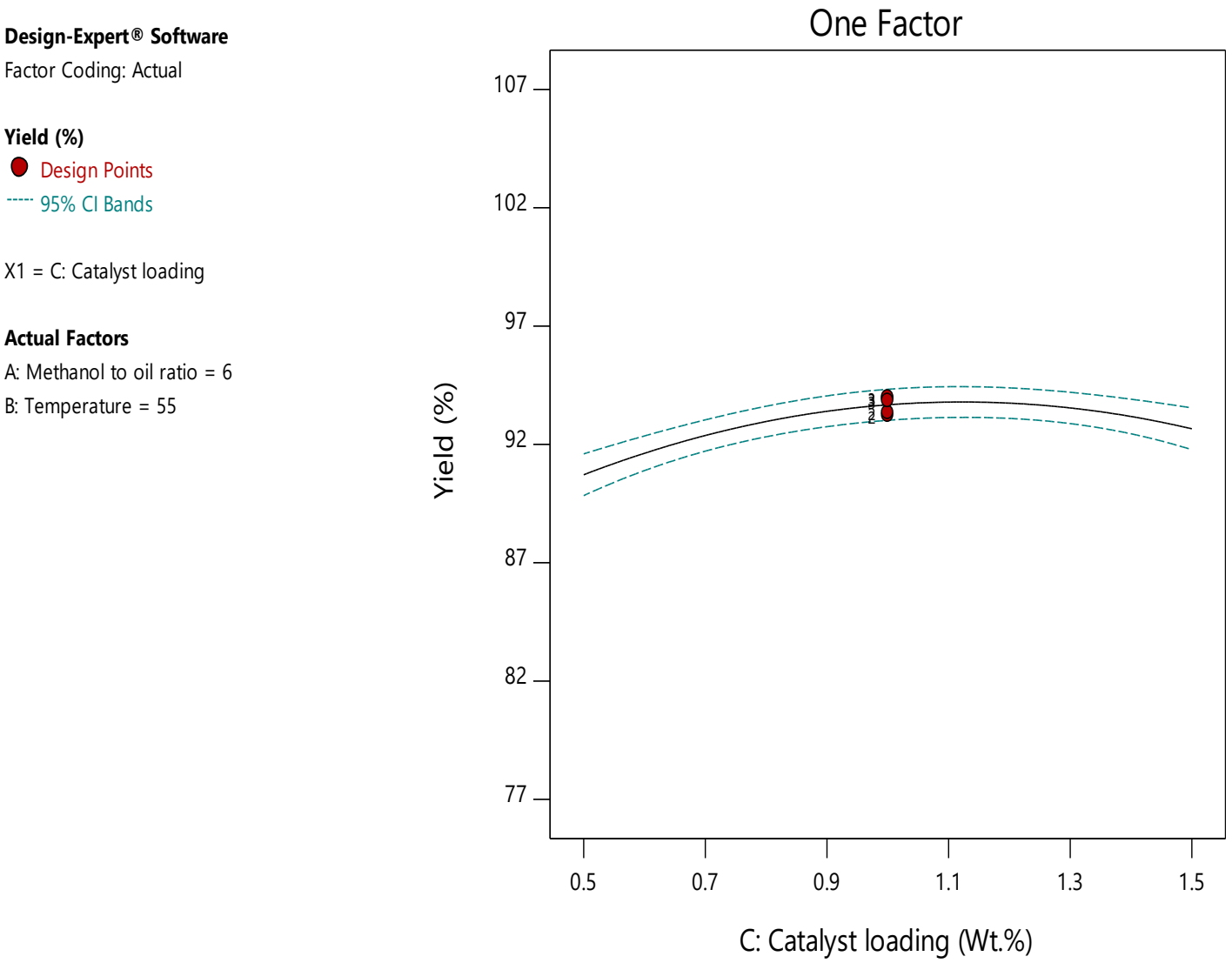



*Figure 9: Effect of catalyst loading on yield of biodiesel*

The above effects of methanol to oil ratio, temperature and catalyst load on yield of biodiesel agreed with the previous report of Eman N. Ali and Cadence Isis Tay (2012) And G.Kafuku (2010).

### 4.8 Response surface plot of experimental variables

Response surface plots are used to analyze the regression model. These plots are created to analyze the change in response surface. Figure (19) shows the respone surface plot of methanol to oil ratio and temperature on biodiesel yield at fixed catalayst loading of 1%. As the temperature increases from 45 °C to 55 °C, the yield increased from 68 % to 94 %. This indicated that yield of biodiesel was very sensitive to temperature change. As methanol to oil ratio increased from 3 to 6, the yield of biodiesel increased and reach peak value (94%).

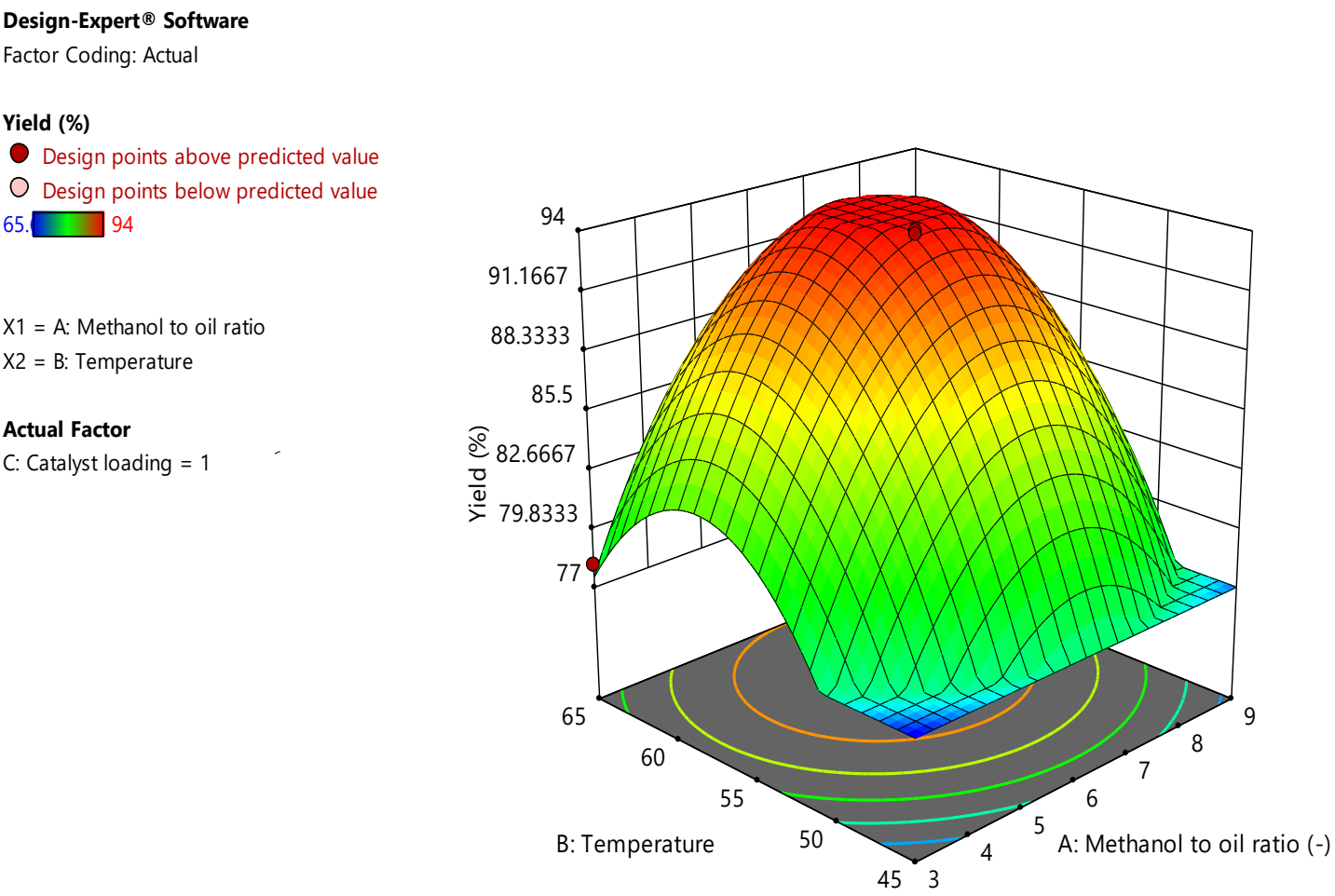



*Figure 10: Response surface plot of effect of methanol to oil ratio and temperature at constant catalyst loading*

Figure (20) shows the response surface plot as function of methanol to oil ratio and catalyst load at conistant temperature of 55°C. As methanol to oil ratio increased from 3:1 to 6:1 with an increase of catalyst load from 0.5 to 1.0, the yield of biodiesel increased rapidly and reach its peak value

(94%). Increasing methanol to oil ratio from 6:1 to 9:1 with increasing catalyst load from 1.0 % to 1.5 %, decreases the yield of biodiesel because the formation of soap.

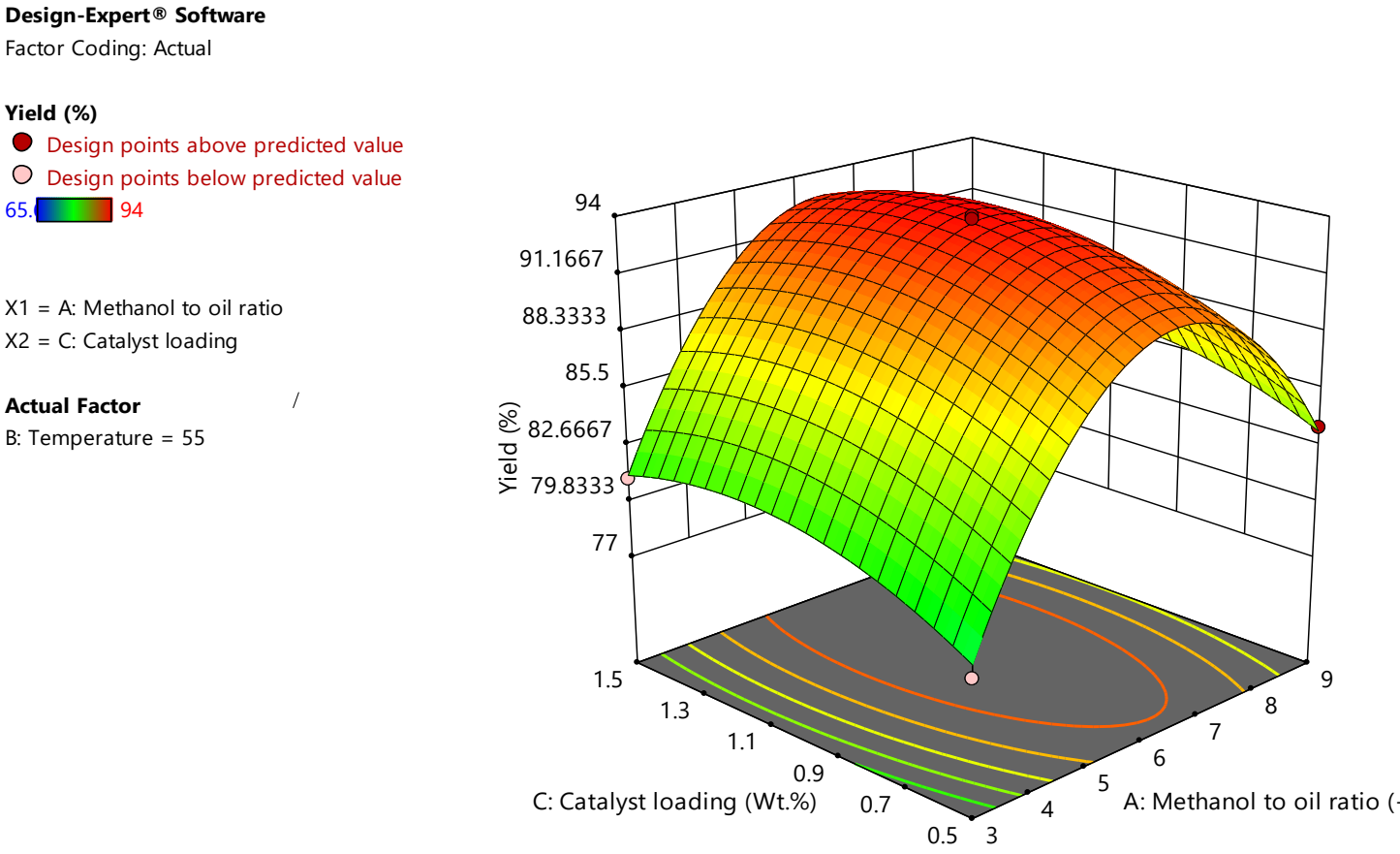



*Figure 11: Response surface plot of effect of methanol to oil ratio and catalyst loading at constant temperature*

Figure (21) show the response surface plot of temperature and catalyst load at constant methanol to oil ratio of 6:1. Upon increasing catalyst load from 0.5 % to 1.0 % with increasing temperature from 45 °C to 55 °C, the yield of biodiesel increases slightly and close to the optimum value (94 %). As temperature increases from 55 °C to 60 °C, the yield is nearly the same with peak value however, the yield slightly decreases as temperature increases from 60 °C to 65 °C due to methanol evaporation.

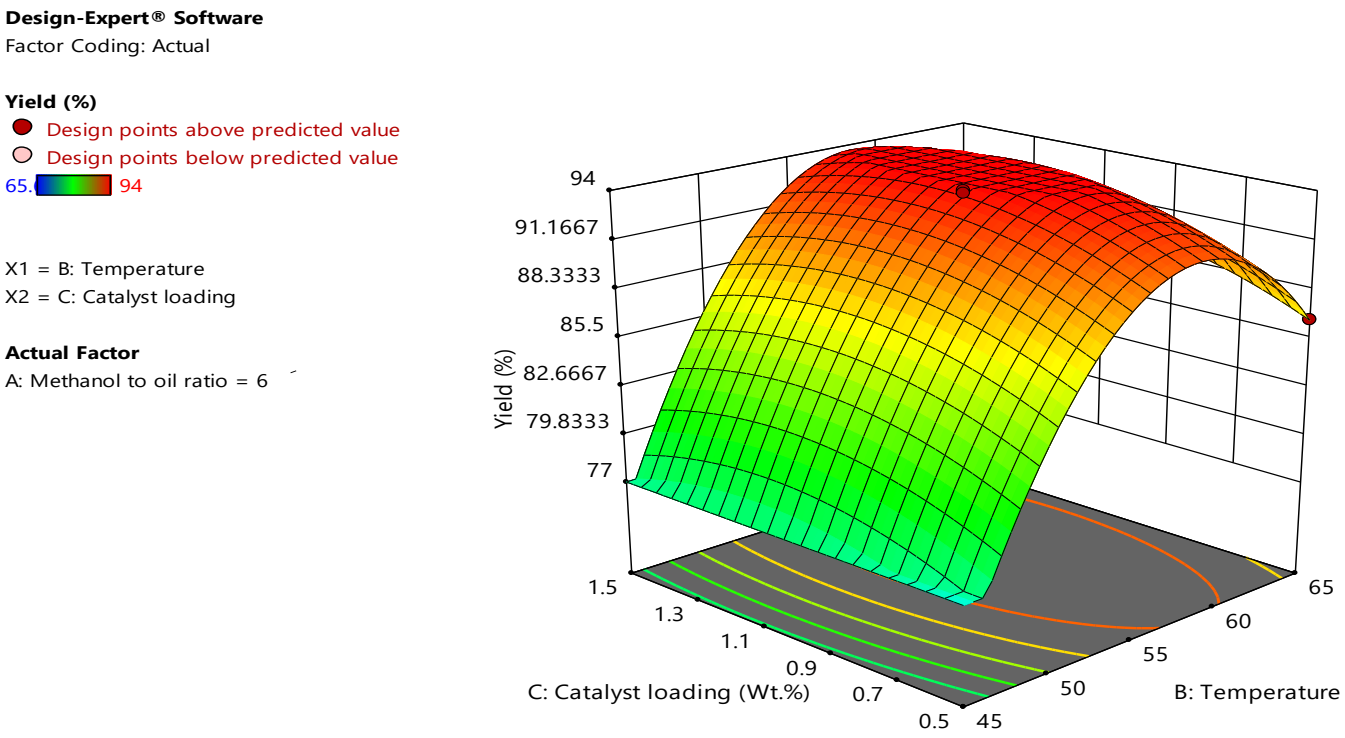



*Figure 12: Response surface plot of effect of temperature and catalyst loading at constant methanol to oil ratio*

### 4.9 Optimization of process parameters of transesterification reaction using RSM

Response surface method (RSM) is collection of statistical and mathematical techniques used for developing, improving and optimizing various processes. It is also useful for designing, developing and formulation of new products and for improvement of the existing product designs. Optimization of process parameters of biodiesel production from MS seed oil via transesterification reaction was discussed in the following table (15).

*Table 16: Optimization goal and limit of process variables*

| Name | Goal | Lower Limit | Upper Limit | Lower Weight | Upper Weight | Importance |
|---|---|---|---|---|---|---|
| A:Methanol to oil ratio | minimize | 3 | 9 | 1 | 1 | 3 |
| B:Temperature | minimize | 45 | 65 | 1 | 1 | 3 |
| C:Catalyst loading | minimize | 0.5 | 1.5 | 1 | 1 | 3 |
| Yield | maximize | 65.67 | 94 | 1 | 1 | 3 |

*Table 17: Optimum numerical solutions by RSM*

| Number | Methanol to oil ratio | Temperature | Catalyst loading | Yield | Desirability | |
|---|---|---|---|---|---|---|
| **1** | **4.489** | **51.537** | **0.500** | **83.683** | **0.753** | **Selected** |
| 2 | 4.511 | 51.501 | 0.500 | 83.722 | 0.753 | |
| 3 | 4.513 | 51.518 | 0.500 | 83.753 | 0.753 | |
| 4 | 4.487 | 51.430 | 0.500 | 83.529 | 0.753 | |
| 5 | 4.500 | 51.630 | 0.500 | 83.850 | 0.753 | |
| 6 | 4.444 | 51.617 | 0.500 | 83.610 | 0.753 | |
| 7 | 4.521 | 51.382 | 0.500 | 83.599 | 0.753 | |
| 8 | 4.450 | 51.419 | 0.500 | 83.369 | 0.753 | |
| 9 | 4.419 | 51.558 | 0.500 | 83.428 | 0.753 | |
| 10 | 4.512 | 51.729 | 0.500 | 84.028 | 0.753 | |
| 11 | 4.422 | 51.412 | 0.500 | 83.243 | 0.753 | |
| 12 | 4.472 | 51.818 | 0.500 | 83.981 | 0.753 | |
| 13 | 4.421 | 51.813 | 0.500 | 83.769 | 0.753 | |
| 14 | 4.773 | 50.864 | 0.500 | 83.781 | 0.751 | |
| 15 | 5.068 | 53.527 | 0.500 | 87.898 | 0.737 | |

Response surface method found 15 solutions and selected the optimum values to minimize the economic cost of temperature, methanol and catalyst required for transesterification reaction and to maximize the yield of biodiesel. The selected optimum yield of biodiesel was observed at methanol to oil ratio of 4.489, Temperature of 51.537 °C and catalyst loading of 0.500 Wt.%. This yield was confirmed experimentally at specified methanol to oil ratio, temperature and catalyst loading.

# 5. Conclusion and Recommendations

## 5.1 Conclusion

In this research the production and characterization of biodiesel from moringa stenopetala seed oil via catalyzed transesterification process was studied. The process parameters that affect transesterification reaction; methanol to oil ratio, temperature and catalyst loading have been investigated. The statistical analysis and experimental design was done using Box Behnken standard design and optimization of process parameters was done by response surface method (RSM). The physicochemical properties of biodiesel was determined according to ASTM D6751 and EN 14214 standard methods and all the results obtained meet these standards.

The experimental result showed that the highest yield of biodiesel was 94.0 % and this value was obtained at optimum conditions: temperature 55 °C, methanol to oil ratio of 6:1 and catalyst load of 1.0 %. Increasing methanol to oil ratio, temperature and catalyst load up to the optimum point (middle point) increased the yield of biodiesel. However, further increase of the parameters above the optimum point decreased the yield of biodiesel. On the other hand, insufficient amount of the process parameters resulted in incomplete conversion of the reactants and decreased the yield of biodiesel. The response surface optimization method indicated that all process parameters affects the yield and fuel quality of biodiesel. Temperature has a highest significant effect while catalyst loading has lower effect.

Generally, it can be concluded that moringa stenopetala is a promising alternative feed stock for biodiesel production for the reasons: it does not affect the food security, has a good oil content and can be harvested throughout the year. The biodiesel produced meet the standard fuel quality since it has high calorific value (43 MJ/KG), low kinematic viscosity, and low acid value. Moreover, biodiesel can directly be used in diesel engine without further engine modifications.

## 5.2. Recommendations

Moringa stenopetala is a miracle and multipurpose tree grown at different parts of Ethiopia. However, it is ignored and underutilized for long time. Moringa stenopetala can be grown all over the parts of the country. It is dry resistant and can be grown in any type of soil. This research confirmed that it is a promising alternative feed stock for biodiesel production since it has high oil yield. Currently, moringa stenopetala is not used for direct food purpose and does not affect the food security of the country.

The biodiesel produced from this feed stock showed higher calorific value when compared with other vegetable oil fuels and it also meet all the international fuel standards. Biodiesel is non-toxic, biodegradable and environmentally friend fuel which can be used as a substitute for petroleum fuels. Ethiopia has high potential of moringa stenopetala to produce biodiesel. Therefore, it is highly recommended to use this resource for biodiesel production.

The following points are recommended for the concerned stake holders

- Make awareness for farmers to plant and grow moringa stenopetala on their land and encourage them to practice agroforestry on their farmland
- Aware urban community to grow moringa stenopetala on their garden
- Government and concerned body should give training on plantation, utilization and multipurpose use of moringa stenopetala
- Further study should be done on biodiesel production from other non-edible feed stocks
- Further advanced experiments should be done on biodiesel fuel properties of refractive index, melting point and ash content
- Further study should be done on engine test to determine fuel consumption efficiency, brake thermal efficiency and exhaust gas emissions
- Further research should be done on non - catalytic transesterifiication process of biodiesel production and compare the result with catalyzed process

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## Appendices

### Appendix A

### Yield calculation of oil and biodiesel at optimum conditions

Yield of oil (%) $= \frac{mass\ of\ oil\ produced}{mass\ of\ sample\ used}$ *100

$32.184/80 * 100$

Yield of oil (%) = 40.23

Yield of biodiesel (%) = $\frac{volume\ of\ biodiesel\ produced}{volume\ of\ oil\ used}$*100

$51.7/55$ *100

94

Amount of seed sample used = 80 g

Amount of oil extracted = 32.184 g

Amount of oil used for transesterification reaction = 55 ml

Amount of biodiesel produced = 51.7 ml

| Level | Yield (%) of oil |
|---|---|
| 1 | 39.71 |
| 2 | 39.64 |
| 3 | 40.23 |
| Average | 39.86 |

| Level | Yield (%) of biodiesel |
|---|---|
| 1 | 93.90 |

| | |
|---|---|
| 2 | 94.0 |
| 3 | 94.10 |
| Average | 94.0 |

**Appendix B**

**Actual biodiesel yield from experimental design of transesterification reaction**

| | Factor 1 | Factor 2 | Factor 3 | Response 1 |
|---|---|---|---|---|
| Run | A:Methanol to oil ratio | B:Temperature | C:Catalyst loading | Yield |
| | - | | Wt.% | % |
| 1 | 6 | 55 | 1 | 93.23 |
| 2 | 6 | 65 | 0.5 | 86.67 |
| 3 | 6 | 45 | 0.5 | 74.36 |
| 4 | 3 | 55 | 1.5 | 81 |
| 5 | 9 | 45 | 1 | 68.18 |
| 6 | 9 | 65 | 1 | 82.3 |
| 7 | 3 | 65 | 1 | 78.12 |
| 8 | 6 | 55 | 1 | 93.35 |
| 9 | 6 | 55 | 1 | 93.94 |
| 10 | 9 | 55 | 0.5 | 83.63 |
| 11 | 3 | 55 | 0.5 | 78 |
| 12 | 6 | 55 | 1 | 93.85 |
| 13 | 3 | 45 | 1 | 65.67 |
| 14 | 9 | 55 | 1.5 | 85.45 |
| 15 | 6 | 45 | 1.5 | 75.64 |
| 16 | 6 | 65 | 1.5 | 88.34 |
| 17 | 6 | 55 | 1 | 94 |

## Appendix C

### Calculations of physico-chemical properties of oil / Biodiesel

**Acid value of oil using equation (3)**

$$A.V = V * N * 56.1/w$$

$$1.45 * 56.1 * \frac{0.1}{5}$$

$$A.V = 1.63\ mg\frac{KOH}{g}\ oil$$

**Saponification value of oil using equation (4)**

$$S.V = Mwt * (Vb - Vb)/w$$

$$\frac{0.5 * 56.1 * (55 - 20.2)}{3\ g}$$

S.V = 189.2 mg KOH/g oil

**Free fatty acid of oil**

FFA = A.V/2

= 0.815 %

Molecular weight of oil using equation (12)

$$Mwt = \frac{168300}{(S. - A.V)}$$

985.5 g/mole

**Kinematic viscosity of oil using equation (1)**

$$v = (\mu)/\rho$$

$$v = 7.9 * 0.001/840 = 9.4\ mm2/s$$

**Iodine value of Biodiesel using equation (18)**

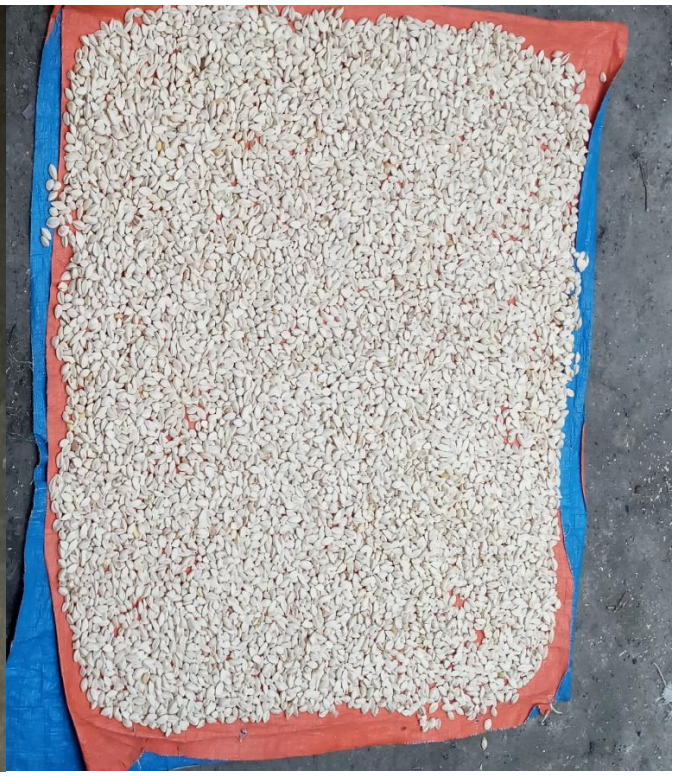
$$I.V = \frac{B - S}{0.3\ g} * 0.1 * 12.69$$

$$\frac{(27 - 2.3) * 0.1 * 12.69}{0.3}$$

$$I.V = 104.5\ \frac{I2}{100} g$$

**Appendix D**

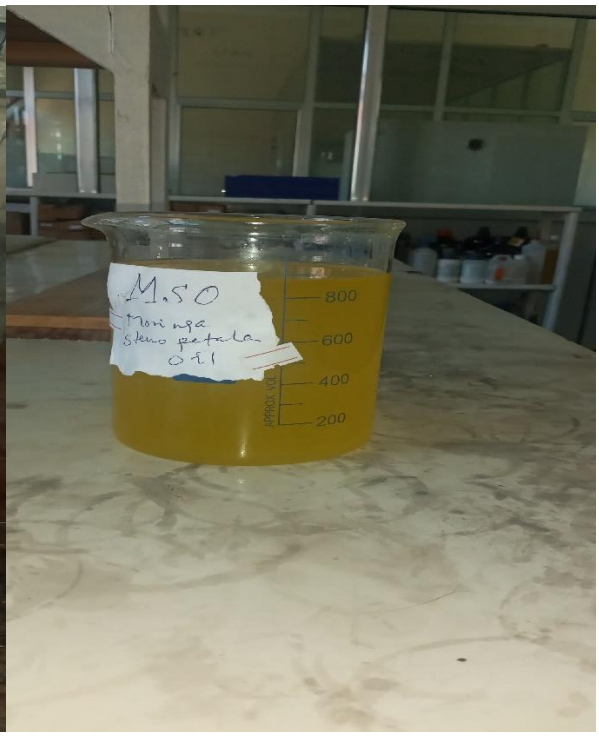
**Feed stock and experimental set up pictures**



**Moringa stenopetala seed**

**Soxhlet oil extraction method**


**Crude oil**




**Purified oil**


**Transesterification reaction**

**Separation of Biodiesel and glycerol**

## Appendix E

## Characterization of moringa stenopetala seed oil and biodiesel pictures

Kinemayic viscosity


Titration and specific gravity determination

Open cup Flash point determination

## Appendix F

## Calorific value of Biodiesel (FAME)

| | GEOLOGICAL SURVEY OF ETHIOPIA | Doc.Number: GSE/F 5.10-2 | Version No: 1 |
|---|---|---|---|
| | GEOCHEMICAL LABORATORY DIRECTORATE | | Page 1 of 1 |
| Document Title: | Hydrocarbon Laboratory Analysis Report | Effective date: | May, 2017 |

Issue Date: - 27/04/2021
Request No: - GLD/RN/934/21
Report No: - GLD/TR/386/21
Sample Preparation : - 60 Mesh
Number of Sample: -One (01)

Customer Name: -Gemechu yadeta

Sample type: -Biodiesel
Date Submitted: - 19/04/2021
Elements to be determined:- Calorific Value.
Method of analysis:- Adiabatic Calorie Metter.

| Collectors' Code | Calorific Value Cal/gm. |
|---|---|
| MSSO | 10287.03 |

Note: - This result represent only for the sample submitted to the laboratory.

Analysts
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Approved By
Alemnesh Abate

Quality Control
Gosa Haile

Geological Survey of Ethiopia
Central Geological Lab Team