JIMMA UNIVERSITY

SCHOOL OF GRADUATE STUDIES

JIMMA INSTITUTE OF TECHNOLOGY

FACULTY OF MECHANICAL ENGINEERING

SUSTAINABLE ENERGY ENGINEERING STREAM

Synthesis and Optimization of Biodiesel from Bracissa Carinata A.Braun Seed by using Calcium Oxide as Catalyst

A Research Thesis submitted to the School of Graduate Studies of Jimma University, Jimma Institute of Technology, Faculty of Mechanical Engineering in Partial Fulfillment of the Requirements for the Degree of Master of Science in

Sustainable Energy Engineering

BY**:** Asegu Regesa

March, 2023

JIMMA, ETHIOPIA

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Main Advisor: Balewgize Amare Zeru (Ass. Prof.)

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DECLARATION

I, the under signed, declare that this research entitled "Synthesis and Optimization of Biodiesel from Bracissa Carinata A.Braun Seed by using Calcium Oxide as catalyst" is my original work and has not been submitted as requirement for an award of any degree in Jimma University or elsewhere and all sources of materials used for the thesis have been properly acknowledged.

Name: Asegu Regesa____________________ ____________________

Signature Date

As a research Advisor, I here certify that I have read and evaluate this thesis paper prepared under my guidance, entitled Synthesis and optimization of biodiesel from Bracissa Carinata A.Braun Seed by using Calcium Oxide as catalyst" and recommend and to be accepted as a Thesis in fulfilling requirement for the degree Master science in Sustainable Energy Engineering.

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Advisor: Signature Date:

Mr. Demeke Girma (Msc) ______________________ ______________________

Co-Advisor: Signature Date

Approved by Board of Examiners

External Examiner ____________________ Signature _______ Date_______

Internal Examiner ____________________ Signature _______ Date_______

Chairperson ____________________ Signature _______ Date_______

ABSTRACT

Biodiesel is simply the monoalkyl esters of vegetable oils, animal fats, or cooking grease .The majority of energy demand is met by conventional fuels, but they are depleting at an alarming rate. However, researchers have only focused on energy source production which has resulted in developing high erucic acid varieties. This results partly in limited studies on synthesis of biodiesel from Bracissa Carinata A.Braun. The adoption and scaling-up of this promising crop as an oilseed crop in developing countries and Mediterranean conditions can sustain the impact of climate change with the demand for food and energy debate concepts. This study paper assesses the seed of Bracissa Carinata A.Braun was utilized for characterization, optimization, fatty acid composition analysis of the seed and oil extraction. The Gomenzer seed was extracted by using normal hexane (62—78°C) by Soxhlet apparatus.The result showed that the seed kernels consisted of: 12% (w/w) oil, moisture. After optimization using Design-Expert has been carried out, optimal conditions of temperature (70-80°C), methanol to oil ratio (12:1), and (1:3) catalyst concentration have been obtained and an oil yield of 97.65% by wt. was found. After the optimum values have been obtained, an experiment has been conducted and an oil yield of 98.166% by wt. was found which is closely in agreement with the result obtained from the model and the experimental observation and hence validated the findings of the optimization. Physicochemical characterization of the optimized oil yield revealed that it has the following most important properties: moisture (0.154% w/w), kinematic viscosity (40.8116mm2) at 40℃, specific gravity (196) at 40℃, density (882.1kg/m3) at 40℃, free fatty acid value (0.00123 % by wt.), saponification value (182.325 mg KOH g-1 oil), iodine value (84.389 g I2/100 g), From the Gas chromatography analysis result of Gomenzer oil showed that the presence of the following major fatty acids: Erucic acid (0.360%),Palmitic acid (0.0171%), Oleic acid(0.393%),Stearic acid(0.0222%),Linoleic acid (1.339%), Linolenic acid (0.104%), Myristic acid (2.257%).Generally this Paper presents the results of an engine test, characterization of Gomenzer seed, optimization of biodiesel with Physicochemical characterization of the optimized oil yield . Therefore biodiesel can use as an alternative fuel for diesel engine .The diesel-biodiesel fuel can improve combustion and emission characteristics of diesel engine

Key Words: Biodiesel, Characterization, brassica carinata, Optimizatio*n*

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ABBREVIATION/NOMENCLATURE

FFA	Free fatty acid
Cao	Calcium oxide
FAAE	Fatty acid alkyl esters
FAME	Free fatty acid methyl ester
ANOVA	Analysis of variance
XRD	X-ray diffraction
RSM	Response surface methodology
ASTM	American Society for Testing and Materials
TAN	Total acid number
IEA	International Energy Agency
EU	European Union
CP	Cloud point
CN	Cetane number
GSO	Gomenzer seed oil

CHAPTER ONE

1. INTRODUCTION

(Brassica carinata A. Braun) is mainly originated in the highlands of Ethiopia .It is locally known as "Gomenzer" in Amharic. It can well be adopted in the Mediterranean climate areas and it is a drought and heat tolerant oilseed crop. It is used as oilseed crop, especially in the arid and semi-arid areas (Alam, Masum, and Dwivedi 2021).To meet the growing demand for high erucic oil has industrial feedstock, researchers have developed cruciferous varieties with higher concentrations of erucic acid and ultra-long-chain fatty acid.Brasica carinata are also expected to be feedstocks for hydro treated renewable Jet fuel (HRJ).

Therefore, it is critical to advocate for and develop Brassica carinata as a bio refinery and bio industrial oil platform using traditional and molecular breeding techniques and instruments. Brassica carinata is a green vegetable crop that produces a moderate amount of commercial oil in Ethiopia. Wealthier countries, on the other hand, have commercialized it as a biofuel product. Therefore, EM is an important feedstock for the production of sustainable biofuels. It is emerging as an intriguing new source of biofuels and industrial feedstocks, helping to mitigate the impact of global climate change on sustainable agricultural development by reducing pollution caused by the over use of petroleum in developed countries(Benti et al. 2023).

1.1 Background of the studies

Biodiesel is simply the monoalkyl esters of vegetable oils, animal fats, or cooking grease. It has shown great promise for compression-ignition engines and has piqued the interest of numerous commercial fuel manufacturers(Hagos et al. 2020). The majority of energy demand is met by conventional fuels, but they are depleting at an alarming rate. Because of the scarcity of petroleum-based fuels, researchers have been looking into biodiesel and its properties to find out why it has such striking similarities to diesel (Demirbas 2018).

Because of modernization and population growth, total primary energy consumption is rapidly increasing in today's scenario. At the moment, fossil fuels account for roughly 80% of total global energy demands, and diesel fuel consumption is expected to reach 5.7 million gallons by the year 2035, liters per day (Nuradila et al., 2017).As a result, there is a global race to explore potential renewable resources to meet the ever-increasing demand for fuel. To meet rising energy demands while reducing environmental pollution caused by fossil fuels, a paradigm shift toward renewable biofuels has occurred. Biodiesel (mono alkyl fatty acid ester) has the potential to be a renewable resource that can be used as an alternative to petroleum-based fuels.

From 2016 to 2020, global biodiesel production is expected to increase by 14 %(Hymavathi, Prabhakar, and Sarath Babu 2014). Because of its high energy density due to the presence of C14-C20 long carbon chain triglycerides, biodiesel is thought to be more suitable for engines. Due to the ethical constraints associated with first-generation biofuels derived from edible biomass, there has been increased interest in second and third-generation biodiesel, particularly from microalgae (Behera et al., 2019).

Biodiesel, which is a fatty acid methyl ester, can be produced starting from non-edible agricultural seeds with high oil content such as Jatropha, Castor, etc., and alcohol in presence of a catalyst. As an alternative to Jatropha and Castor, Brassica carinata seed can be a potential and promising feedstock for the production of biodiesel with extra environmental and socioeconomic importance in Ethiopia. The expansion of the biodiesel sector in Ethiopia has been aided by private investment (both domestic and foreign), with 25 projects approved so far

1.2 problem statements

Ethiopia's economy is moving forward and striving to change from an agricultural led economy to an industrial led economy. Hence, has achieved rapid economic growth. However, this development requires a huge amount of fuel energy sources. Currently, the country is completely dependent on imported oil and a large sum of the country's earnings are used to pay for the imported petroleum fuel. Because they produce less gas and particle matter than petro diesel, the burning of biodiesel and biodiesel blends has been shown to significantly reduce exhaust emissions and particulate matter.

Additionally, biodiesel does not include the hazardous component Sulphur, unlike petroleum-based diesel, which may do so. When used as fuel in a diesel engine, biodiesel performs similarly to petro-diesel or, in some situations, even outperforms it. Biodiesel and petroleum-based diesel fuel blend nicely together. These blends are stated along with how much biodiesel they include (for instance, "B20" has 20% biodiesel and 80% petroleum diesel). A blend's properties frequently fall in the middle of those of biodiesel and petroleum diesel. To create petroleum diesel, mixes are sometimes employed.

The combustion of biodiesel and biodiesel blends has demonstrated a considerable reduction in particulate matter and exhaust emissions because they emit particulate matter and gases that are lower than petrol diesel. In addition, unlike petroleum-based diesel, which may contain Sulphur, biodiesel does not contain this harmful ingredient. Biodiesel when applied as a fuel in a diesel engine mechanisms like petro-diesel or in some cases even gives better results related to petro-diesel fuel. Diesel fuel composed of petroleum and biodiesel combines quite well. The amount of biodiesel in these blends is listed (for example, "B20" has 20% biodiesel and 80% petroleum diesel). The characteristics of a blend will often fall halfway between those of biodiesel and petroleum diesel. Sometimes blends are used to make petroleum diesel more lubricious or to lower the amount of sulfur in it. Probably the best benefit of blending for a biodiesel producer would be to enhance cold-operating properties (Ciolkosz 2015).

Ethiopia is one of Africa's fastest-growing countries, aiming to achieve lower-middle-income status in the coming years. Maintaining the nation's fuel security is required to ensure the country's continued development program. Approximately half of the population lacks access to electricity, and the only potential fuel source is wood. Ethiopia also spends $ 2.5 billion on imported petroleum products each year, accounting for 20% of the country's foreign exchange. Thus, to address the aforementioned issues and achieve fuel security, it is critical to increasing the production and use of locally produced fuels such as bio-based fuel (Feyisa 2021).

Nowadays, energy has become a critical component of long-term economic growth and a high standard of living for humanity. Even though fossil fuels play an important role in global energy needs, they are not sustainable and their prices are not stable. Furthermore, the most serious global environmental issues caused by the use of fossil fuels are now affected by climate change, acid rain, and pollution (Benti et al. 2023).

To meet the growing energy demand, and support and stabilize the economy locally produced cheap, renewable, clean, and efficient source of energy is necessary. Globally, conventionally grown non-edible oils are the major feedstocks used for biodiesel production. The current study is intended to investigate the feasibility of producing biodiesel from non-edible oil sources such as gomenzer seed oil as well as optimization of the transesterification process using calcium oxide as catalysts. The success of the research will increase the value of biodiesel production since non-edible oil feeds will be employed and can be produced locally.

1.3 Objectives of Research

1.3.1 General Objective

The general objective of this study is the Synthesis and Optimization of Biodiesel from Bracissa Carinata (Gomenzer) Seed by using Calcium Oxide as a Catalyst.

1.3.2 Specific Objectives

- To extract biodiesel from Brassica carinata seed (gomenzer)
- To optimize the process for maximum biodiesel yield
- To Study the factors that affect liquid fuel production through transesterification
- To characterize biodiesel
- To test the performance of biodiesel blend with diesel fuel using Engine test and Exhaust emission analyzer
- To compare the performance of gomenzer biodiesel with other feedstock (jatropha, castor) from the literature

1.4 Scope of the study

There are more than 350 oil-bearing crops acknowledged as potential sources for biodiesel production. Based on this fact, the scope of this research aims mainly to identify and select some potential oil-bearing plants (non-edible) for biodiesel production.

Ethiopia is of the developing countries which produced an abundance of renewable resources such as agricultural oilseeds. Also, gomenzer is one of the agricultural oilseeds and is widely produced in Ethiopia. So these oilseeds help us to have alternative ideas of energy to bring into technology to improve and save our country from foreign currency. In the laboratory analysis, it will study both the ultimate and proximate analysis of Brassica carinata (with local name gomenzer) and liquid fuel, chemical Composition, and physical properties. In addition, to simulate Design expert software Feedstock characteristics, physical and chemical analysis of liquid fuel depend on the value obtained from the experiment. Finally, engine performance and emissions analysis of gomenzer feedstocks of only B_{100} and B_5 fuel blends are presented.

1.5 Significance of the study

The outcome of this research can potentially lower (reduce) the dependence on imported fuels which means, shifting the need for some foreign–produced oil to domestically-produced energy sources. This means, almost all fuels consumed in Ethiopia are imported, and the displacement of oil-based fuels to biodiesel produces a net shift from foreign to domestic energy sources.

The government of Ethiopia is considering jatropha, castor, and palm as alternative sources of biodiesel feedstock. This thesis considers gomenzer seed as an additional alternative source with extra socioeconomic and environmental importance for farmers and the community around them. Moreover, this thesis provides an environmentally safe and closed-loop industrial production system from high-free fatty acid feedstock through the use of the heterogeneous catalyzed processes to enhance the yield of FAME. Generally, Small scale and large-scale biodiesel production companies can use the material as a reference.

CHAPTER TWO

2. LITERATURE REVIEW

Around 25 billion hectares are suitable for biodiesel feedstock production, among the countries all-out land areas of 1.2 million km^s. Given the global prices of fossil fuels, the biofuels business has grown to be a major national player. As a result, biofuel investment activities are underway in several locations in Ethiopia, with a particular emphasis on biodiesel and bioethanol production. Moreover, Ethiopia, began using a 5% bioethanol blend in transportation fuel in 2008, which was later increased to 10%.Despite Ethiopia's recently announced climate resilient green economy policy calling for a 5% biodiesel blend in transportation gasoline by 2030, biodiesel mixing in transportation fuel has yet to begin (Asnake et al. 2020).

2.1 Background on Gomenzer (with local name brassica carinata)

Brassica carinata A. Braun, also known as "Ethiopian mustard," "Ethiopian rape," "Abyssinian mustard," or "carinata," is being developed as a non-food oilseed feedstock with a low carbon intensity to produce advanced drop-in renewable fuels and bio products. It has a desirable oil profile, broad adaptability, and productivity even under suboptimal conditions (Ramdeo Seepaul et al. 2021).

Carinata production as a winter crop represents a once-in-a-lifetime opportunity for growers in the Southeast United States to produce a significant amount of biofuel feedstock to help meet domestic energy needs. Carinata fits into remaining cropping systems as a winter crop, providing opportunities to farm over 1.4 million hectares of winter fallow land, resulting in over 1224 million liters of jet fuel, displacing 1.4 percent –2.33 percent of petroleum-based jet fuel in the United States (Alam, Masum, and Dwivedi 2021).

Carinata seed contains 18.7 percent to 28.3 percent protein and 42 percent to 52 percent oil, with a well-balanced fatty acid profile. The primary fatty acid component is erucic acid (41%–43%), followed by linoleic, linolenic, and oleic ac (Kumar et al.,2020)(Aslan and Eryilmaz 2020).

2.1.1 Characteristics of Gomenzer (with local name brassica carinata) seed

Carinata contains less than 7% saturated fatty acids and is strong in erucic and linoleic acids. These qualities make it an attractive oil that can be converted into a high-quality, "drop-in" biofuel with no processing. The oil's high erucic acid content (around 36%) makes it a non-food oil. Since of its ability to split into two biofuel hydrocarbons from its 22-carbon chain, carinata oil is appealing as a biofuel because it effectively doubles the amount of fuel produced from each molecule of erucic acid. Without replacing feed and food crops, carinata has the potential to assist the US and other countries meet their demand for renewable energy(R Seepaul et al. 2019).

Carinata is a very shallow-planted crop (Seepaul, Small, Mulvaney, et al., 2019) and germinates in the top portion of the soil that usually experiences moisture deficit (Patane&Tringali, 2011). Carinata should be established not more than 1.3 cm deep because of its small seed size; however, deeper depths may be considered when planting in sandy soils. Early season moisture availability in the 0.64–1.3 cm inches of topsoil is critical for uniform and vigorous seed germination (Patane&Tringali, 2011). Maximum seed yields (1140–1492 kg ha-1) also occurred at a relatively high seeding rate (9–13.5 kg ha-1) in South Dakota (Alberti, 2017). Similarly, carinata planted at 10 kg ha-1 produced 2890 kg seed ha-1 (Bozzini et al., 2007) in Italy, while 8 kg ha-1 produced 1592 kg ha-1 in Ethiopia (Tadesse et al., 2012). In Florida, the maximum seed Yield (2761 kg ha–1) was produced at a 3 kg ha–1 seeding rate (Mulvaney et al., 2019). Higher seeding rates may reduce stem diameter and increase accommodation Potential but may be necessary for no-till systems to compensate for increased

Seedling mortality (Alberti, 2017). Changing the plant geometry through seeding rate and row spacing modifies the leaf arrangement and upper most manner, which controls light interception and photosynthetic productivity (Sarlikioti et al., 2011). Carinata has phenotypic plasticity to modulate plant style to optimize light interception, especially at low plant populations, by producing more branches, racemes, and pods. This phenotypic plasticity is modulated by environmental conditions and resource availability (Hossain et al., 2018). Carinata growth and yield are influenced more by row spacing than by seeding rate (Mulvaney et al., 2019) in Florida.

2.2 Current status of biodiesel production

As an alternative fuel, biodiesel is made from various vegetable oils, oil seeds, animal fat, and used cooking oil. Biodiesel has gained popularity since the 1970 oil crisis, which accelerated the development of biodiesel as an alternative fuel. Today, biodiesel is primarily used is created through the transesterification of vegetable and animal fats. Currently, among the various vegetable oils, seeds, and other products used in the production of biodiesel are: Cotton seed oil, sunflower oil, neem oil, rapeseed oil, soybean oil, waste cooking oil, and various alcohols (Behera et al. 2020)

Recent development has also envisaged the potential of using jatropha seeds for the production of biodiesel. There are more than 350 oil-bearing crops have been identified till now however sunflower, safflower, soybean, rapeseed, cottonseed, and peanut oil are identified as potential alternative fuels for diesel engines

2.2.1 Global biodiesel production

Today, global biodiesel production ranges between 35 and 45 million tons, with the absolute figure varying depending on the source (F. O. Licht, (2018).

In 1992, the European Union began industrial-scale production of biodiesel, largely in response to positive signals from EU institutions. In 2003, the EU launched a biodiesel promotion campaign, setting clear targets for biodiesel consumption in the transportation sector and allowing member states to provide various fiscal and regulatory incentives to entice individual investors.

The European Union will likely continue to dominate biodiesel markets, followed by the United States, Germany, and Asia. In 2018, the United States was the leading producer, accounting for 6.9 billion liters of biodiesel, followed by Brazil with 5.4 billion liters. Germany was the leading producer on the European continent in 2018, with a total output of 3.5 billion liters. Thailand led the Asian province in biodiesel production in 2018, with approximately 1.6 billion liters produced. According to the statistics, these figures are expected to rise between 2019 and 2024.

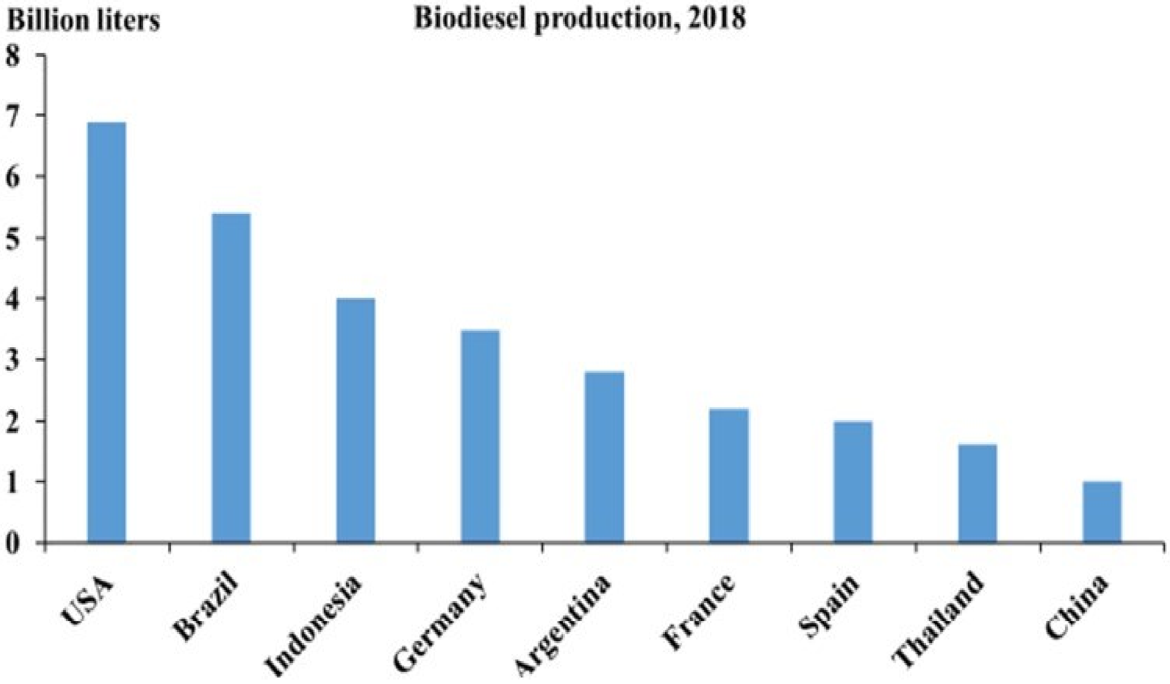


Figure 1: Global leading biodiesel-producing countries by the year 2018

(Source:https://www.statista.com/statistics/271472/biodiesel-production-inselected-countries/Ce :)

2.2.2 Current status of biodiesel production in Ethiopia

Biodiesel production in Ethiopia is still in its early stages. The country currently has two functioning production plants located in Amhara and Oromia regions. There is also a planned plant for construction in Benishangul Gumuz region. In addition, several other projects are under development, including an oilseeds project from the World Bank to create more biodiesel plants in Ethiopia.

Despite the potential for growth, there are still challenges facing Ethiopian biodiesel producers. These include limited access to finance and technology, inadequate infrastructure, and insufficient supply of raw materials such as oilseeds. There are also regulatory challenges that need to be addressed before further progress can be made in advancing the sector. In order to spur progress in this area, various government initiatives have been launched to promote the use of environmentally friendly energy sources like biodiesel.

2.3 Feedstocks for Biodiesel Production

Biodiesel feedstocks are classified into first, second, and third generations based on the stage at which they were discovered for use in biodiesel production. The first generations are edible oils, which have sparked numerous heated debates about energy and food security (Ibrahim 2018).Corn, soybeans, sunflower seeds, cottonseeds, canola, crambe, rapeseeds, safflowers, flaxseeds, rice bran, mustard seeds, and camelina are among the first-generation biodiesel feedstocks, as are animal fats (Hymavathi, Prabhakar, and Sarath Babu 2014).

Virgin vegetable oils that can be used in cooking are classified as first-generation feedstocks. Rapeseed oil, soybean oil, groundnut oil, palm oil, and cottonseed oil are among them. Second-generation oils are those that have been used for frying and are no longer fit for human consumption. Vegetable oil derived from non-edible plants, such as Jatropha curcas, castor, and neem, is also included in this category. Microalgae are third-generation biodiesel feedstocks. Algae produce oil, which can be easily refined into diesel or even specific components of gasoline. It can be genetically modified to produce anything from ethanol and butanol to gasoline and diesel fuel (Ibrahim, 2018) (Barreiros et al. 2020). Fourth-generation biofuel, which are still in an early developmental stage, use bioengineered microorganisms such as bioengineered algae or crops that are genetically different to consume more CO_2 from the environment than they emit when they are disbursed (burning). Biofuel are used to produce various fuels including ethanol, butanol, hydrogen, methane, vegetable oil, biodiesel, isoprene, gasoline, and jet fuel.

In Europe, rapeseed oil remains the dominant biodiesel feedstock, accounting for 45 percent of total biodiesel feedstocks in 2017, followed by used cooking oil (UCO), which accounted for 21 percent of total feedstock production. Rapeseed oil, palm oil, soybean oil, sunflower oil, used cooking oil, animal product, Jatropha, brown grease, soap stock acid oil, and refining residue with rapeseed account for 55% of total feedstock used in the European Union. Soybean oil is the most common feedstock for biodiesel production in the United States (Branco, Serafim, and Xavier 2019).

2.4 Chemistry of biodiesel production

Transesterification is used to create biodiesel. Transesterification is the reaction of a fat or triglyceride with an alcohol in the presence of an acid or base catalyst to form esters (typically methyl or ethyl esters) and glycerol as a byproduct. Alcoholysis is, in standard, the action of one alcohol transferring another from an ester (P. Kumar, Kumar, and Yadav 2018).

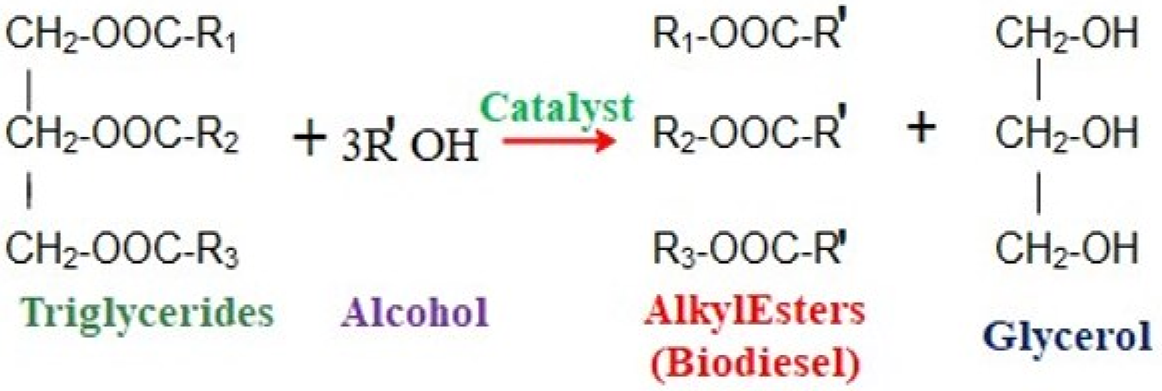


Figure 2: General Transesterification reaction in the presence of a catalyst

2.5 Physio chemical properties and specifications of biodiesel

The fuel requirement describes and sets the quality standards for biodiesel. It is based on the standard ASTM D6751. The biodiesel standard in Brazil and the U.S. are appropriate for both fatty acid methyl ester (FAME) and fatty acid ethyl ester (FAEE), whereas the current European biodiesel standard is only applicable for fatty acid methyl esters (FAME) (Jazie, Pramanik, and Sinha 2012). The ASTM and EU biodiesel property specifications with the recommended test methods are given in Table 1.

To ensure quality biodiesel, there are standard for testing the fuel properly to see that it happens expressions for use. ASTM (an international standards and testing group) has a method to officially define biodiesel for use in diesel engines, labeled ASTM D6751. Tables. 1 show the test procedures necessary for all the predictable standards for biodiesel.

Table 1: ASTM Standards for Biodiesel

Property	Test Method	Limits	Units
Ca and mg combined	EN14538	5max.	ppm
Flash point	ASTM D93	93.0 min	℃
Alcohol control: one of the following must be seen : 1. method content 2. flash point	EN14110 ASTM D93	0.2 max. 130 min.	Vol % ℃
Water and sediment	ASTM D2709	0.050 max.	Vol %
Kinematic viscosity @40℃	ASTM D445	1.9-6.0	mm^2/s
sulfated	ASTM D874	0.020	% mass
sulfur	ASTM D5453	0.0015 max(S15) 0.05 max(S500)	-
Coper strip corrosion	ASTM D130	No.3max	-
Acid number	ASTM D664	0.50 maximum	mg KOH/g
Cetane number	ASTM D613	47min	-

Table 2: Standards specification of petro diesel

Property	Value
Specific gravity	0.82-0.845
Flash point	50℃
Cetane number	50
Distillation point	350℃
Kinematic viscosity	2.7c.s

A.viscosity

The viscosity of the oil is a measure of its internal fluid friction or resistance to flow which tends to oppose any dynamic change in a fluid motion. The viscosity of oil decreases as the temperature rises. The less viscous the oil, the easier it is to pump, atomize, and achieve finer droplets. Viscosity in fuels reduces combustion efficiency by lowering the heating value.

B.Iodine value

This determines the level of unsaturation in the oil as well as the amount of iodine absorbed per 100 grams of fuel. The higher the iodine value, the higher the oil's unsaturation.

C.Specific gravity

The relative density about water is referred to as specific gravity. The specific gravity of most oils and their methyl ester is higher than that of diesel fossil fuel. This is due to the higher molecular mass and chemical structure of vegetable oils.

D.Total acid number (TAN)

This is the amount of potassium hydroxide (KOH) required to neutralize one gram of oil or biodiesel in milligrams. The TAN value in a refinery indicates the possibility of a Corrosion problem. TAN values can be calculated using color indicator titrations. Biodiesels with acid numbers less than three (mg OH/g oil) are preferable.

E. Flash point

Atmospheric pressure, is the lowest temperature at which a fuel must be heated to vaporize and form a combustible mixture. It is one of the distinguishing features of fossil fuels and biofuels. According to the literature, liquids with a flash point less than 60 °C are flammable, while those above this temperature are combustible.

F. Cetane number (CN)

The cetane number measures the fuel's ability to self-ignite when injected into the engine. Many performance characteristics, such as density and heating value, are related to the Cetane number. Cetane number is a parameter used to determine biodiesel quality; it is proportional to in CI engines, the time it takes for the fuel to ignite. To determine ignition, the CN rating of fuel can be used for biodiesel fuel characteristics.

G. Calorific value

The calorific value of a fuel is the amount of thermal energy released per unit quantity of fuel when the fuel is completely burned and the combustion products are cooled to the initial temperature of the combustible mixture. It determines a fuel's energy content. The ASTMD240 standard method was used to determine the calorific value of vegetable oils and their methyl esters using a bomb calorimeter.

2.6 Biodiesel production process system

Biodiesel is produced from vegetable oils or animal fats and alcohol, through a transesterification reaction (Oniya, Adebayo, and Ogunkunle 2016). This chemical reaction converts an ester (vegetable oil or animal fat) into a mixture of esters of the fatty acids that makes up the oil (or fat). Biodiesel is obtained from the purification of a mixture of fatty acid methyl esters (FAME).

2.6.1 Batch process

In a batch method, refined vegetable oil is added to a reactor for transesterification together with surplus alcohol and a catalyst. To maintain the reaction mixture's viscosity low and ensure that all of the triglycerides are soluble, an excessive amount of alcohol is required. For changing the chemical balance we can say that the Trans esterification reaction is complete. When the highest conversion rate is reached, Ester and glycerol are separated from each other by decantation. The ethanol is then recovered using a rotary evaporator and used in the subsequent batch. The ester is dried and then washed with hot water (Aranda Moraes et al. 2008).

2.7 Biodiesel production process methods

Triglycerides and other minor ingredients such as free fatty acids, gums, waxes, etc. make up the complex mixture of triglycerides found in vegetable oils and animal fats. Triglycerides are glycerol esters containing three chains of varying lengths of aliphatic or olefinic FFAs (12–24 carbons). Transesterification of the triglycerides appears to be the best option out of all the suggested ways to turn oils into biodiesel since the physical properties of fatty acid esters (biodiesel) are quite similar to those of diesel fuel(Wang et al. 2021).

Several generally accepted technologies have been well-established for the production of biodiesel fuel. Vegetable oils and animal fats are appropriate to be modified to reduce their viscosities so that the product obtained has suitable properties to be used as diesel engine fuels. There are many methods for this modification to produce a better quality of biodiesel such as direct use and blending, micro-emulsions, pyrolysis of vegetable oil, and transesterification (Kaisan et al. 2017). In this section, an investigation was carried out to briefly review these methods.

2.7.1 Transesterification reactions

Transesterification of oils (triglycerides) with alcohol is the most prevalent biodiesel manufacturing process, which produces biodiesel (fatty acid alkyl esters, FAAE) as the main product and glycerin as a byproduct. The conversion of triglycerides to diglycerides is the initial step, followed by the conversion of diglycerides to monoglycerides and monoglycerides to glycerol, with each glyceride providing one methyl ester molecule. Transesterification, sometimes known as alcoholics, is the process of exchanging alcohol from one ester for another alcohol like hydrolysis, except that instead of water, alcohol is employed. The most relevant operating variables affecting the transesterification process are reaction temperature, reaction time, reaction pressure, the ratio of alcohol to oil, concentration, type of catalyst, mixing intensity, and kind of feedstock (S. Kumar et al. 2020).

2.7.1.1 Types of transesterification reactions

- **Homogeneous catalytic transesterification**

Homogenous catalyst is classified into basic and acidic catalysts. The homogenous transesterification process especially the basic type requires a high purity of raw materials and post-reaction separation of catalyst, by-product, and product at the end of the reaction. Both of these necessities rise up the cost of biodiesel.

- **Supercritical alcohol transesterification**

The supercritical alcohol method is a non-catalytic method for biodiesel production in which instead of using catalysts, high pressure and temperature are used to carry out the transesterification reaction. The reaction is fast and conversion raises 50–95% for the first 10 min but it requires a temperature range of 250–400 C. The transesterification of triglyceride by supercritical methanol, ethanol, propanol, and butanol has demonstrated to be the most promising process. The vegetable oils were Trans esterified 1:6–1:40 vegetable oil-alcohol molar ratios in supercritical alcohol conditions. The disadvantages of the supercritical methods mostly are high pressure and temperature requirement and high methanol-to-oil ratios (usually 42) that renders the production expensive.

- **Heterogeneous solid-acid catalytic transesterification**

Despite the lower activity, heterogeneous solid acid catalysts have been used in many industrial processes because they contain a variety of acid sites with different strengths of Bronsted or Lewis acidity, compared to the homogenous acid catalysts. Using solid acid catalysts has the following advantages: insensitive to FFA content, simultaneous esterification and transesterification removal of the purification step of biodiesel, easy separation of the catalyst from the reaction products, and reduction of the corrosion tricky, even with the presence of acid species. Heterogeneous Solid acid catalysts, such as Nafion-NR50, sulfated zirconia, and tungstate zirconia have been chosen to catalyze biodiesel-forming transesterification due to the presence of sufficient acid site strength. Among the solid catalysts, Nafion- NR50 demonstrated higher selectivity towards the production of methyl ester and glycerol due to its acid strength. However, Nafion - NR50 has the drawbacks of high cost and lower activity compared to liquid acids.

2.8 Parameters affect Biodiesel production

Transesterification has been described as a chemical reaction between triglycerides and alcohol in the presence of a catalyst to produce mono-esters that are termed biodiesel. The reaction is either partial or the yield is reduced to a significant extent if the parameters are not optimized. The important process parameters, which affect the yield of the transesterification process are the Alcohol to Oil Molar Ratio, Catalyst concentration, Reaction time, Reaction Temperature, Mixing Intensity, and Purity of Reactants (Barreiros et al. 2020).

2.8.1 Alcohol to oil molar ratio

The stoichiometric transesterification needs 3 moles of the alcohol per mole of the triglyceride to yield 3 moles of the fatty esters and 1 mol of the glycerol. However, the transesterification reaction is an equilibrium reaction in which a huge excess of alcohol is required to initiative the reaction close to achievement in a forward direction. Lower molar ratio need a longer time to complete their action. Excess molar ratios increase the conversion rate but the separation of glycerol is difficult. A molar ratio of 6:1 is commonly used in industrial processes to obtain methyl ester yields higher than 98% (w/w)

2.8.2 Catalyst concentration

A catalyst is required to recover the transesterification reaction and yield. Alkaline catalysts such as sodium hydroxide and potassium hydroxide are the most widely used. These catalyst increase the reaction rate several times faster than acid catalyst. Alkaline catalyst concentration in the range of 0.5 to 1% by weight yields 94 to 99% conversion efficiency. Further increase in catalyst concentration does not increase the yield, rather it adds to the cost and makes the separation process more complicated.

2.8.3 Reaction Temperature

The proportion of the transesterification reaction is strongly influenced by the reaction temperature. Generally, the reaction is carried out close to the boiling point of methanol (60 to 70°C) at atmospheric pressure. With further increase in temperature, there is more chance of loss of methanol. Transesterification reaction has been reported to be influenced positively by the increase in temperature.

2.8.4 Purity of Reactants

Impurities present in vegetable oil also affect ester conversion levels significantly. The free fatty acid and moisture content are key parameter for determining the viability of the vegetable oil transesterification process. The starting materials used for base-catalyzed alcoholysis should meet certain specifications which are that a free fatty acid (FFA) value lower than 3% is needed to carry the base-catalyzed reaction to completion (Repeat et al.). If the reaction situations do not meet the above requirements, ester yields are significantly reduced

2.9 Latest literature review

(Vicente, Martinez, and Aracil 2015) studied biodiesel production from Brassica carinata and sunflower oils, using methanol at low temperatures ranging between 20 and 32 °C. They found high biodiesel yield as a maximum yield of 91.31% for high erucic acid Brassica carinata oils (HEBO). They emphasized that the effect of the temperature had a negative impression on the process for HEBO owing to the fatty acid Composition of the oil and because of the formation of soaps.

(Boye et al. 2015) investigated the effect of reaction time on biodiesel yield. Theyused100g-waste frying oil (WFO), 22% methanol, and 1.0% KOH (wt. %WFO), reaction temperature of 60 ºC. The Reaction time varied from 30 to 75 every 5 min. intervals under these conditions. The maximum yield of biodiesel was acquired (97.20%) at a reaction time of 60 min.

Furthermore, Gomes Filho et al. (2015) Reported Sterculia striata (Chicha) oil transesterification with methanol at the reaction temperatures which were used at 30 °C and 70 °C. They concluded that the best biodiesel yield is (98.5%) at 30 °C. They perceived that alcohol evaporated at 70 °C. Also, Valle et al. (Silveira et al. 2019) used FFD (Fractional Factorial Design) from RaphanussativusL. var. Oleifera Stokes oil, for biodiesel making at low and high temperatures. They obtained the best result at 30 °C and claimed that increasing reaction temperature had a negative influence due to saponification with the temperature rising.

Samuel, Giwa, and El-Suleiman (2016) obtained coconut oil ethyl ester at the maximum yield of 97.20 using the KOH catalyst. Rashid and Anwar (MomarTallaDieng et al.(2019)discussed and optimized the transesterification of rapeseed oil for the production of biodiesel by using NaOH, KOH, CH3ONa, and CH3OK. They resulted that the best Catalyst was KOH at a high yield in their study. Tomesevic and Siler-Marinkovic (Topi 2020) Singh et al. (Chanakaew somboon et al. 2020) and Al-Sakkari et al. (Al-Sakkari et al. 2018).

Examined biodiesel production using different alkali catalysts which had NaOH and KOH. They specified that KOH was a more efficient catalyst than NaOH. The dissolution of KOH was better than NaOH in methanol. If the dissolution is not fully realized, saponification may occur due to triglyceride reacting with the excess amount of NaOH catalyst (Chhetri, Watts, and Islam 2008), (Suresh, Jawahar, and Richard 2018).

Because of this situation, the biodiesel yield of NaOH might have decreased. On the Other hand, experiments with ethanol observed that the NaOH catalyst had the highest Yield.

Kucek et al. (2017) made refined soybean oil biodiesel using NaOH and KOH (0.3and 1.0 wt. %) as catalysts, with ethanol to oil molar ratio of 6:1 and 12:1 at the temperature between 30 and 70 °C. Encinar et al, Anastopoulos used NaOH and KOH (0.25–1.50 (Kucek et al. 2007) to produce biodiesel from CynaracardunculusL. oil with ethanol. In both studies, the highest biodiesel yield was achieved with NaOH. KOH is more effective than NaOH in ethanol solution on saponification. The saponification can take place more in ethanol solution than in methanol solution due to the lower acidity and higher quantity of hydroxide ions of ethanol Eze, Harvey, and Phan (2015).

Hafizil et al. (2015) studied how methanol-diesel mixes performed in a diesel engine. 10%, 20%, and 30% volume quantities of methanol were added to diesel as additives (methanol (10%)-diesel (90%), 20%-diesel (80%), and 30%-diesel (70%). Overall findings indicate that methanol-diesel blends significantly outperform mineral diesel in terms of effective brake power and torque. Methanol (10%) and diesel (90%), the fuels with the lowest fuel mixing ratio, produced the lowest exhaust temperature. In contrast to methanol-diesel blends, mineral diesel produced the highest exhaust temperature. The mineral diesel produced a lower brake-specific fuel consumption (bsfc) than other methanol-diesel blends, whereas the maximum bsfc was obtained by a blend of 30% methanol and 70% diesel.

2.10 Engine performance and emissions production of various feedstocks of some selected

Due to their miscibility with pure biodiesel, alcohol additions like methanol and ethanol are highly useful in biodiesel blends. Alcohol additions are highly useful for lowering biodiesel's viscosity and density, which are higher than those of conventional mineral diesel. When used to fuel diesel engines, alcohol additions increase combustion efficiency and result in fewer exhaust emissions. When compared to mineral diesel, ethanol and methanol have bases of around 35% and 30% more oxygen, respectively, which aids diesel engines in more thorough combustion. Fuels with greater oxygen will result in more thorough combustion.(Jazie, Pramanik, and Sinha 2012).

2.10.1 Palm oil

The Authors studied the engine performance using neat palm oil methyl ester, $B_{50,}$ and neat diesel (B_0) at different speeds and load conditions. The result showed that SFC for palm oil methyl ester is higher than diesel fuel due to lower energy content. They also found that neat palm oil methyl ester (B_{100}) reduces tailpipe NO, UHC, and smoke opacity by 5.0%, 26.2%, and 66.7%, respectively due to improved combustion, higher cetane number, and oxidation of soot. However, it was found that PME content in the fuel blend did not considerably affect tailpipe CO emission, with only a maximum 0.89% reduction attained with the B_{50} blend. The authors concluded that despite the defect of PME in its higher specific fuel consumption, its overall reduction of controlled tailpipe emissions makes PME a green technically feasible alternative to fossil diesel in both neat and blended forms for use in light-duty diesel engine (Zahedi and Mirnezami 2020).

2.10.2 Jatropha curcas

Chauhan evaluated the performance and exhaust emissions using 5%, 10%, 20%, and 30% Jatropha biodiesel blends with diesel fuel on an unmodified diesel engine. The experimental results show that engine performance with biodiesel of Jatropha and its blends was comparable to the performance of diesel fuel. In the case of all fuel blends, brake thermal efficiency, HC, CO, CO_2, and smoke density were lower while BSFC and NO_x were higher than that of diesel. The authors decided that biodiesel derived from Jatropha and its blends could be used in a conventional diesel engine without any modification. However, various parameters can be evaluated in the future such as the prediction of the best blend concerning the various engine parameters by varying the spray time of fuel using common-rail fuel injection(Chauhan, Kumar, and Cho 2012).

2.10.3 Canola (Rapeseed)

The Authors studied the effect of canola oil biodiesel and its blend on a low heat loss diesel engine emission and performance with and without coating situations. The author found out that the increase in power for biodiesel is between 1.6% and 3.5% related to 8.4% for diesel. While the drop in SFC for biodiesel is between 4.7% and 8% compared to 4.9% for diesel. There is a significant reduction in exhaust gas emission CO (22-24%) compared to 25% for diesel, and smoke (4.7-8.2%) compared to 9% for diesel. However, NO_x emission increases (4.8-7.3%) compared to 4.9% for diesel. This is recognized to the higher oxygen content and increase in after-combustion temperature due to the ceramic coating. Exhaust gas temperature increase by between 5.4% and 2.6% for biodiesel while it increases by 11.4% for diesel (Anastopoulos et al. 2019).

2.11 summary of literature review

Overview of previous studies by researchers in the fields of sustainable, Renewable Engineering, and chemical and to understand the techniques and key studies that were conducted.

The main review was as follows:

- History of Biodiesel Production System
- Engine performance and emissions production of various feedstocks of some selected, transesterifications processes of biodiesel Parameters that influence the biodiesel production system Optimum Temperature Ranges of biodiesel

Table 3: A brief review of different kinds of literature related to biodiesel production

Authors	Title	Task performed	Methodology used
Vicente, Martinez, $ Aracil (2015)	➢ Biodiesel production from brassica carinata and sunflower oil	➢ By using ethanol at low temperatures ranged b/n 20 and 32 °C and found optimum biodiesel yield 91.3%	➢ Analytical methods and Statistical analysis used
Boye et al.(2015)	Optimization of Coconut Oil Ethyl Ester Reaction Variable and Prediction Model of Its Blend	✓ the effect of reaction time on biodiesel yield ✓ Use American Oil Chemists' Society (AOCS) test methods for oil and fats analysis	➢ Transesterification process ➢ Blends and model development
Gomes Filho et al. (2015)	Effects of biodiesel on low heat loss diesel engine emission and performance with and without coating condition	omparison and Characteristics of coated and uncoated engines used for engine test	Transesterification with methanol at the reaction temperatures which were used 30 °C and 70 °C
Chauhan et al.(2015)	A study on the performance and emission of a diesel engine fueled with Jatropha biodiesel oil and its blends	The performance and exhaust emissions using 5%, 10%, 20%, and 30% Jatropha biodiesel blends with diesel fuel on an unmodified diesel engine	✓ Data collection and analysis by Mathematical or size and select. ✓ Experimental and the simulation was done in summer and winter time

CHAPTER THREE

3. METHODOLOGY

3.1 Structure of research methodology

Research methodology is a crucial factor to bring in effective research with accredited results. It can be defined in many ways such as procedure, way, method, and technique that are applied to incorporate and gather all relevant information for the research.

This chapter explains how the whole research was conducted and shows the methods by which seeds collection, crude oils extraction, characterization of biodiesel, and engine test.

Therefore, Figure: 3 gives a summary of the implemented flow chart of this research.

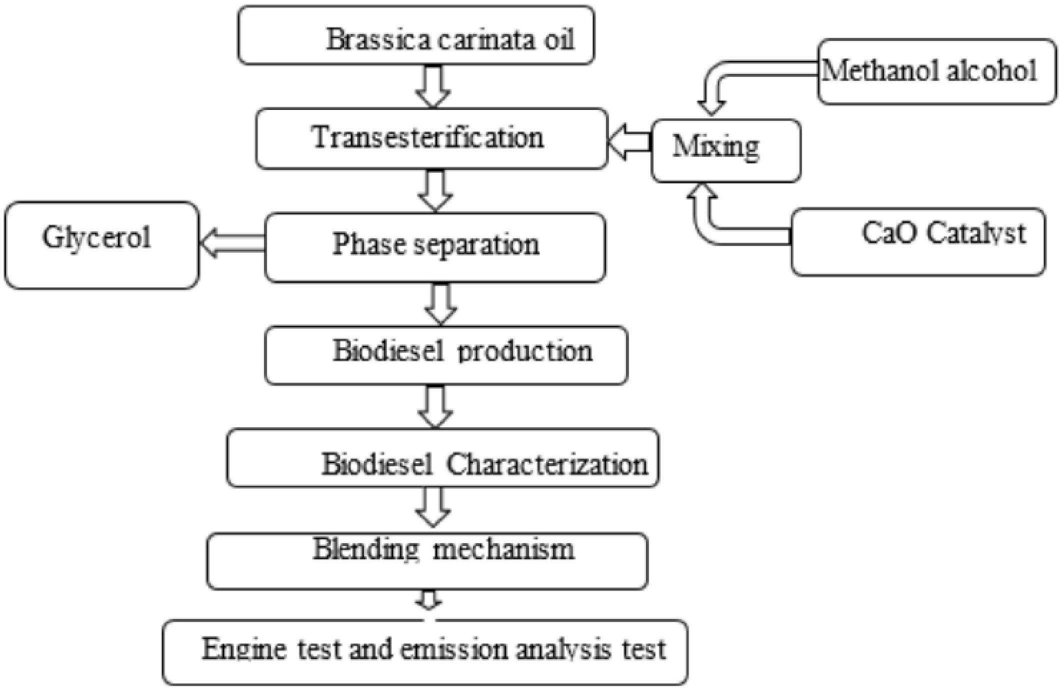


Figure 3: flow chart of research methodology

3.2 Materials and equipment used

3.2.1 Chemicals and Material used

The different chemicals and materials that have been used during the experimental analysis of the extraction of gomenzer seed oil are listed as follows of the reagents are analytical and some chemicals are potassium hydroxide and dimethyl ether, normal hexane (99%), methanol (99.8%), Phenolphthalein, hydrochloric acid, chicken eggshell as catalyst and Brassica carinata (with the local name of gomenzer) seed.

3.2.2 Equipment used

The instruments and their purpose are listed in the Table below.

Table 4: List of laboratory equipment

Material/equipment	**Use**
Soxhlet apparatus	to extract oil
Oven	to dry sample
Coffee grinder	to crush the sample
Muffle furnace	To calcined ground eggshell
Rotary Evaporator	To separate hexane from oil
Vibro viscometer	To measure the heating value
Gc-ms apparatus	To determine the fatty acid composition of oil
Filter paper	To put the sample in Soxhlet
Graduated cylinder	To measure different volumes
Digital balances	To weigh the samples
Separating funnel	to separate biodiesel from glycerol

3.3 Experimental Methods

3.3.1 Calcium oxide catalyst preparation

A.Egg shell (Cao) catalyst Preparation

The basic steps for catalyst preparation from waste chicken egg shells are Sample Preparation and Calcination.

I. Catalyst Sample preparation

To make chicken eggshell powder, start by submerging the collected eggshells in hot water for 5 to 10 minutes and washing with tap water multiple times to remove any impurities. Afterwards, dry the shells in an oven at 105°C for approximately 24 hours. Finally, grind the dried shells using a coffee grinder to attain a uniform size and sift the powder through a 200 mesh sieve.

II. Calcination process

The crushed eggshell powder is put into an alumina crucible and calcined in a furnace at different temperatures such as 900℃ and 1000℃ for 2 hr to convert the calcium species present in the shells into CaO particles**.** After calcination, the white powder is stored in a desiccator for 24hr**.** Then the calcined CaO is kept in a closed vessel to avoid a reaction with CO2.

a) Oven dried egg shell b) calcined eggshell

Figure 4: Catalyst preparation from waste chicken eggshell

3.3.2 Catalyst characterization

I. Determination of crystalline size by using XRD analysis

The structure of the calcium oxide calcined at a temperature of 900°C was characterized using X-ray Diffraction (XRD) (Drawell XRD-7000) and the crystallinity size of a particle was also determined by the Scherrer equation. XRD spectrum of calcined eggshell sample were Obtained with Cu-ka1 (λ=0.154056 nm), a scan speed of 2 °/ minute, and a scan range of 5 to 75°.

Scherrer equation:

$$\tau = \frac{k\lambda}{\beta \cos\theta} \quad \text{...Equation} \quad (3.1)$$

Where:

- τ is the mean size of the crystalline domains
- K is the dimensionless form factor, with a value close to unity
- λ is the X-ray wavelength;
- β ($\Delta(2\theta)$) is the line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening, in radians.
- θ is the Bragg angle in degree.

3.4 Raw material Preparation

3.4.1 Preparation of Gomenzer Seed

The Gomenzer seeds have been purchased from the Jimma town market in Mercato. To remove any foreign elements, the acquired samples are thoroughly cleaned by hand. The Gomenzer seed is then dried in an oven at 105 degrees Celsius for 12 hours, to a moisture content of 12 percent. The dried gomenzer seed is then crushed by using a coffee grinder to 0.50mm to 1mm and ready for extraction by 0.50 mm sieve size. Then the grounded seeds are stored in a plastic bag until the next process.

a) Raw gomenzer seed

b) dried gomenzer seed in oven

c) grounded gomenzer seed

Figure 5: raw material preparation

3.4.2 Determination of Moisture content

The cleaned sample was weighed 100g,150g, and 200g and dried in an oven dryer and the weight was measured every 2hrs.The procedure was repeated until a constant weight was obtained. The percentage of moisture in the gomenzer was calculated using the following:

% condition continent = ((W1-W2))/W1×100 ....equation 3.2

Where

W1= the initial Weight of the sample before drying

W2 =the final weight of the sample after drying

3.4.3 Size reduction and sieve analysis

After the moisture is removed by placing the sample in an oven at 105 degrees Celsius for 12 hours, the dried Gomenzer seed was first fed to a coffee grind machine and the seeds have been crushed hence the size has been reduced into smaller pieces.

3.5 Oil extraction using Soxhlet apparatus

In the solvent extraction procedure, Soxhlet extraction using normal hexane as solvent has been used.

3.5.1 Experimental procedures of solvent extraction method by using hexane

The sample of gomenzer seed is first packed with filter paper and placed on the extraction Soxhlet. The packed filter paper is a sample holder used for the solvent extraction system and it is suited for a powdered sample of 100-gram grounded seed with 300 ml of hexane the extraction takes place at 80°C for 3 hours. After the solvent has added, the temperature on the water bath was turned on .when it is heating the solvent was evaporated into a vapor, and it was then cooled down into a liquid in a condenser and passed into the sample collection tube. This continues for several hours until the liquid was removed from the sample. After the mixture of extracted oil and the solvent were collected in a flask, the oil has been separated from the solvent using conventional distillation. Hundred grams in powder form of the sample Gomenzer seed with particle size .After extraction the hexane and oil were separated by a rotary evaporator at 70℃. Next, the solid compound was separated by centrifugation.

The yield of oil gained was calculated by:

% oil = $(\frac{mass\ of\ oil\ obtained}{mass\ of\ \ sample})*100$..eq.3.3

3.5.2 Crude Gomenzer seed oil refining

3.5.2.1 Degumming

The crude Gomenzer seed oil contains gums and phospholipids which can increase the free fatty acids of the oil. Therefore, these are removed by degumming. During the process initially, the crude oil is heated at 70ºC stirring under 1000 rpm in a beaker. Then 3wt% of distilled water (which is first heated to 90 ºC) and 0.2wt% of 85% purity of phosphoric acid is added. After that allow the mixture to stir for 1h. The precipitate is separated by centrifugation at 400 rpm for 30min and the degummed oil is dried at 100 ºC for 30 min under reduced pressure using a rotary evaporator (fractional condenser).

Figure 6: Gomenzer seed Oil degumming

3.5.2.2 Neutralization of oil

During neutralization the degummed oil was poured into a beaker and heated to 80°C, then 2 ml of 0.1M NaOH is added for each 3 g of degummed oil and stir it to a uniform solution. Next 10% of the weight of the oil Sodium chloride, was added to settle out the soap formed. This was poured into a separating funnel and allowed to stand for 1h to separate the soap formed. Hot water was added repeatedly to the oil solution until the soap remaining in the solution was removed. The neutralized oil was then strained off into a beaker.

(a)Crude GSO (b) separation of GSO and (c) neutralized

Figure 7: Gomenzer seed Oil refining

3.6 Characterization of Gomenzer Oil

3.6.1 Determination of Moisture

The cleaned sample was weighed 100g,150g, and 200g and dried in an oven dryer and the weight was measured every 2hrs. The procedure was repetitive until a constant weight was gained.

The percentage of moisture in the gomenzer seed oil was calculated using the following:

% moisture continent = ((W1-W2))/W1×100 ....equation 3.4

Where

W1= the initial Weight of the sample before drying

W2 = the final weight of the sample after drying

3.6.2 Determination of Specific Gravity (SG)

A density bottle (volumetric cylinder) is used to determine the density of the oil. A clean and dry bottle of 50ml capacity was weighed (W0) then it was filled with the oil, insert stopper, and reweighed to give (W1). The oil was replaced with water after washing and drying the bottle and weighed to give (W2). Then the specific gravity was determined by using the formula.

3.6.3 Determination of Kinematic Viscosity

45m1 of the gomenzer seed oil was poured into a test tube and a viscometer was used to measure the viscosity at a temperature of 35°C, the kinematic viscosity was determined after determined the dynamic viscosity of the gomenzer seed oil. The dynamic viscosity of the gomenzer seed oil was determined below;

The kinematic viscosity was obtained by the following formula:

V = $t_{av*}\,(d_b - d_{fo}) * K * F$ .... equation 3.5

Vd = volume displaced during measuring

Mb = mass of the ball

K = ball constant = 6.799mpa/ (cm3/gm)

F= multiplication factor at viscosity measurement at viscometer at 70°inclination 0.952

Scherrer equation: v = μ/ρ .... equation 3.6

Where: v=kinematic viscosity (m2/s)

μ=absolute or dynamic viscosity (N s/m2)

ρ = density (kg/m3)

3.6.4 Determination of Acid Value and Free Fatty acid

The acid value of the oil was considered using the method described by IUPAC (1979) and modified by Egan et al. (1981). The acid value of the oil was calculated using the method described by IUPAC (1979) and modified by Egan et al. (1981). 25ml of diethyl ether and ethanol mixture was added to 5gm of oil in a 250ml conical flask and the solution was titrated with 0.1N ethanoic KOH solution in the presence of 5 drops of phenolphthalein as indicator until the endpoint (colorless to pink) was recognized with consistent shaking. The volume of 0.1 N ethanoic KOH (V) for the sample titration was recording. The total acidity of oil in mg KOH/ gram was intended using the following equation:

$$\mathrm{AV} = \frac{56*\mathrm{N}*\mathrm{V}}{\mathrm{W}} \text{ .... Equation 3.7}$$

Where, V =the volume expressed in a milliliter of 0.1N solution of ethanolic KOH

W = the weight of the oil sample (the mass in grams of the test portion)

N = concentration of ethanolic KOH

56 = molar mass of KOH

Then, the % FFA value was calculated from the acid value as followed:

$$\%\mathrm{FFA} = \frac{\mathrm{AV}}{2}$$

3.6.5 Determination of Saponification Number (SN)

2g of the sample was weighed and added to a conical flask then 25 ml of 0.1N ethanolic KOH solution was also added. Next, the sample was constantly stirred, and allowed to boil at 70 ^{O}C for 60min and a reflux condenser was connected to the flask containing the mixture. After that,3 to 5 drops of phenolphthalein indicator were added to the warm solution and then titrate with 0.5M HCl (volume Va was recorded) to the endpoint until the pink color of the indicator disappeared. Then a blank was prepared following the same procedure and (volume Vb was recorded). Then the SN was calculated using the equation below:

$$SN = \frac{56.1*N(V_{B-}V_{S)}}{W} \text{ .... Equation 3.8}$$

Where, W= weight of oil taken in grams, N= normality of HCl solution,

VS= volume of HCl solution used in the test in ml,

VB= bulk of HCl solution used in blank in ml

3.6.6 Determination of Iodine Value (IV)

Weigh approximately 2 g of the oil into a250 mL conical flask and Add 10 ml of chloroform and 30 ml of Hanus solution, then close the flask completely with Para layer and leave the solution for 30 minute with shaking continuously. Next to this add 10 ml of 15% potassium iodide solution and 100 ml of distilled water then shake it. Finally, the solution was titrated against 0.1 N Sodium thiosulfate solution till yellow color formed, then add 2-3 drops of starch solution where the blue solution formed and then continue with titration till the blue color disappeared (Volume (ml) of Na2S2O3 at endpoint represents S). For blank, the same procedure was conducted as above but without a sample (Volume (ml) of Na2S2O3at endpoint represents B). Then the iodine value (IV) was obtained by:

$$IV = \frac{12.69*N(V_{B}-V_{S})}{M} \text{ .... Equation 3.9}$$

Where, N = normality of sodium thiosuphate,

M = Mass of the sample

VS = Volume of sodium thiosuphate used for a sample test

VB = Bulk of sodium thiosuphate used for blank

3.6.7 Determination of HHV

The higher heating value (the energy content per unit quantity) of acid-treated oil and fatty acid methyl ester was estimated from their saponification value and iodine value using the following correlation:

HHV (energy content per unit quantity) = 49.43-[0.041(S.V) +0.015(I.V)].......................Eq.3.10

3.6.8 Determination of Cetane number (C.N)

Cetane number, which is the ignition delay after injection of the fuel of gomenzer seed oil and Fatty acid methyl ester, was estimated based on saponification value and Iodine value using the following correlation.

C.N = 46.3+ (5458/S.V)-0.225(I.V).. Eq.3.11

3.6.9 Determination of the flash point of the gomenzer seed oil

The flash point of gomenzer seed oil was determined using the empirical formula by (Kaisan et al. 2017). The equations between FP and HHV of oil are shown in the equation below. The flash point of the gomenzer seed oil is calculated from the equation below.

HHV = 0.021FP+32.12

3.7 GC-MS Analysis of Brassica carinata (Gomenzer seed) oil

The fatty acid composition of the Gomenzer seed oil was determined by GC-MS at Jimma University, main campus. The oil physicochemical properties were conducted in JiT in the chemistry lab.

3.7.1 Characterization of Brassica carinata (Gomenzer seed) oil fatty acids using GC-MS Analysis

GC–MS machine was used for the characterization of biodiesel. In the conduct of the analysis, 2 µl (microliters) of the biodiesel sample was injected into the gas chromatograph with its oven temperature programmed between 60 °C and 280 °C at 5 °C/min holding time. The injected sample in the gas chromatograph was separated into a column. Helium gas was used as a carrier gas and a mass-selective detector was used for the analyses. The analyses were done in scan mode and the components were identified based on software matching with standard mass spectra.

3.8 Transesterification and Production of biodiesel from gomenzer seed oil

3.8.1 Biodiesel production process

The experiment was conducted in a 250 mL two-necked flat-bottom flask equipped with a reflux condenser and a mechanical stirrer. During the reaction, initially, the neutral oil was measured and poured into a conical flask and it was pre-heated for 30 min at 50 °C. Then a specified amount of calcium oxide was weighed and dissolved in the required amount of hydrous methanol (99.8%). Next the calcium oxide solution was added to the pre-heated oil and placed in a hot water bath with a constant agitation speed of 400 rpm. After the reaction took place for 3hr it was completed and stopped. Followed to this the product was poured into a separating funnel and it was kept for 24 hr until phase separation is formed. Finally, the catalyst was removed by filtration and the biodiesel was also purified by washing it with hot distilled water.

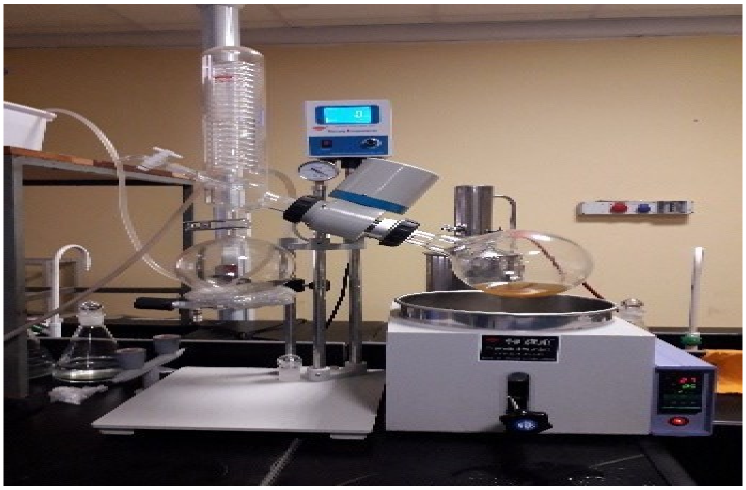


Figure 8: Experimental set-up for batch transesterification reaction

3.9 Characterization of Physico-chemical properties of biodiesel

The physicochemical properties of FAEE, such as specific gravity, density, kinematic viscosity, acid value, saponification value, and iodine value and were determined following the same procedure as used for Gomenzer seed oil. These tests were conducted in JiT in the chemistry laboratory according to the ASTM D6751 procedure

3.9.1 Determination of Acid Value and Free Fatty acid

The acid value of the oil was considered using the method described by IUPAC (1979) and modified by Egan et al. (1981). 25ml of diethyl ether and ethanol mixture was added to 5gm of oil in a 250ml conical flask and the solution was titrated with 0.1N ethanoic KOH solution in the presence of 5 drops of phenolphthalein as indicator until the endpoint (colorless to pink) was recognized with consistent shaking. The volume of 0.1 N ethanoic KOH (V) for the sample titration was recorded.

The total sourness of oil in mg KOH/ gram was calculated using the following equation:

$$AV = \frac{56*N*V}{W} \quad \text{... Equation 3.12}$$

Where, V =the volume expressed in a milliliter of 0.1N solution of ethanolic KOH

W = the weight of the oil sample (the mass in grams of the test portion)

N = concentration of ethanolic, KOH, 56 = molar mass of KOH

Then, the % FFA value was calculated from the acid value as followed:

$$\%FFA = \frac{AV}{2}$$

3.9.2 Determination of Saponification Number (SN)

The saponification value was determined using the indicator method according to ISO 3657 (1988). 2g of the sample was weighed and added to a conical flask then 25 ml of 0.1N ethanolic KOH solution was also added. Next, the sample was constantly stirred, and allowed to boil at 70 ^{O}C for 60min and are flux condenser was connected to the flask containing the mixture. After that,3 to 5 drops of phenolphthalein indicator were added to the warm solution and then titrate with 0.5M HCl (volume Va was recorded) to the endpoint until the pink color of the indicator disappeared. Then a blank was prepared following the same procedure and (volume Vb was recorded). Then the SN was calculated using the equation below:

$$SN = \frac{56.1*N(V_{B-V_{S)}}}{W} \text{ ....Equation 3.13}$$

Where, W= weight of oil taken in grams, N= normality of HCl solution,

VS= volume of HCl solution used in the test in ml,

VB= number of HCl solution used in blank in ml

3.9.3 Determination of Iodine Value (IV)

Weigh approximately 2 g of the oil into a250 mL conical flask and Add 10 ml of chloroform and 30 ml of Hanus solution, then close the flask completely with Para layer and leave the solution for 30 minutes with shaking continuously. Next to this add 10 ml of 15% potassium iodide solution and 100 ml of distilled water then shake it. Finally, the solution was titrated against 0.1 N Sodium thiosulfate solution till yellow color formed, then add 2-3 drops of starch solution where the blue solution formed and then continue with titration till the blue color disappeared (Volume (ml) of Na2S2O3 at endpoint represents S). For blank, the same procedure was conducted as above but without a sample (Volume (ml) of Na2S2O3at endpoint represents B). Then the iodine value (IV) was considered by:

$$IV = \frac{12.69 \times N(V_{B-V_{S)}}}{M} \text{ ....Equation3.14}$$

Where, N = normality of sodium thiosuphate,

M = Mass of the sample

VS = Volume of sodium thiosuphate used for a sample test

VB = size of sodium thiosuphate used for blank

3.10 Optimization of the process parameters

The most important factors affecting the yield and quality of extraction of the seed are extraction temperature, type of solvent, methanol to oil ratio, catalyst concentration, and extraction time. For this thesis work, the effect of three main factors which are temperature, methanol-to-oil ratio, and catalyst concentration were investigated to optimize the extraction operating conditions for achieving maximum oil yield. 200g of ground powder was extracted with n-hexane. The parameters temperature (70-80°C), methanol to oil ratio (1:12), and catalyst concentration (1:3) on gomenzer seeds were used for the extraction. At the end of the extraction, the solvent was separated from the oil using a rotary evaporator and was collected in the receiving flask. The oil which will be retained in the sample flask has been weighed after the process is completed. The percentage of extracted oil was then calculated by dividing then the amount of oil obtained by the amount of the seeds multiplied by 100.

Table 5: Ranges of applicability of extraction parameters

No	Parameters	Range
1	Extraction temperature	(70-80°C),
2	methanol to oil ratio	(1:12)
3	catalyst concentration	(1:3)
4	Type of solvent	Normal hexane
5	Extraction time	3hours

3.11 Design of Experiments using Central Composite Experimental Design

In this study, the effect of three factors on Gomenzer seed oil extraction including temperature, methanol to oil ratio, and catalyst concentration was selected as factors. As a response, a percentage oil yield was chosen; a total number of experiments were employed for the response surface modeling. The Response Surface Methodology (RSM) based on Central Composite Design (CCD) was used to evaluate and optimize the effect of the factors on the response function. The interaction effects, optimal parameters, and the coefficients of the second-order fitting equation were obtained by using Design Expert software. The significance of the independent variables and their interactions were tested utilizing analysis of variance (ANOVA) with a 95% confidence level. The model suitability was tested using the ANOVA test.

The results were assessed with various descriptive statistics such as the p-value, F-value, and the degree of freedom (df); the determination coefficient (R2) of each coefficient which was determined by Fisher's F-test and values of probability >F. As shown in Table 16, a small probability value ($p < 0.001$) indicates that the model was highly significant and could be used to predict the response function accurately. Goodness-of-fit for the model was also evaluated by coefficients of determination R2 (correlation coefficient) and adjusted coefficients of determination R. If the value of the correlation coefficient R2 is large, then it will indicate high reliability of the model in predicting of getting the best operating parameters for the oil extraction. Finally, regression models for the model were established for the dependent variables to fit the experimental data for the response function. The factors and stages that were selected for the experiment were:

- Temperature (70-80°C)
- methanol to oil ratio (1:12), and
- catalyst concentration (1:3)

3.12 Engine test and emission analyzer

The experiments were conducted by considering various parameters. The test was conducted for gomenzer seed oil and its blends at different proportions (5% and pure diesel) for a conventional engine.

The test was conducted from no load to maximum load conditions. The readings such as fuel consumption, speed of the engine, temperatures, etc, were noted. The observations were recorded in tabular columns and calculations are made using appropriate equations. The experiments were conducted on a 7.5kW (type CT 110) single-cylinder diesel engine. The general specifications of the engine are given in the Table below.

3.12.1 Blending Mechanism

The test fuels were blended using a magnetic stirrer (IKA® C-MAG HS 7) at 1200 rpm for half an hour and a shaker (IKA® KS 130 basic) at 400 rpm for half an hour to formulate sure the blends were well blended.

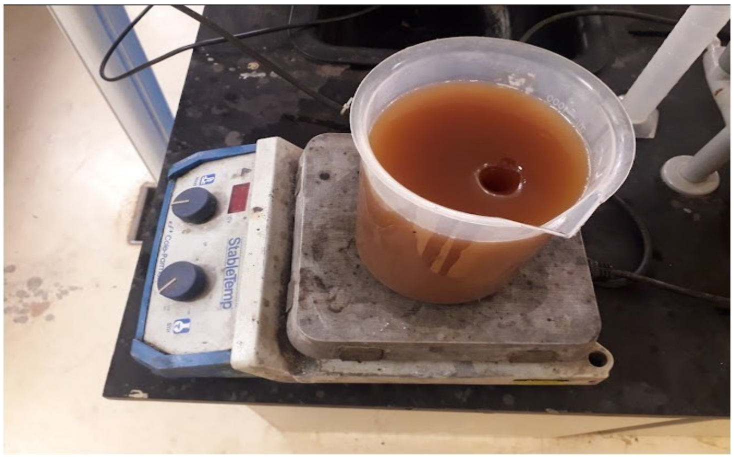


Figure 9: blending the mechanism

In this research, the effect of biodiesel-biodiesel and biodiesel-diesel blending by different percentage on some physical and chemical properties has been studied and presented.

3.12.2 Engine and emissions tests

The engine utilized in this study is a 7,5kW (type CT 110) single-cylinder diesel engine tested on a single-cylinder engine stand. The engine's technical specifications are displayed in Table: 6 Figure: 11 depicts the engine's test rig. The engine setup's schematic diagram is displayed in Figure: 11. A Infralyt smart exhaust gas analyzer (model V1.011) was used to measure the exhaust emission gases of NO, HC, and CO. In this section, one fuel mixture consisting of diesel fuel Brassica carinata (gomenzer) biodiesel and B0, B5 were tested on a single-cylinder diesel engine as a result of the experiments' performance and emission characteristics are obtained.

The experimental investigation was carried out using diesel (B_0), B_5 (95% diesel and 5% biodiesel) and B_{100} (100% diesel and 0% biodiesel) blends for fuel blends. Firstly, the engine was run with diesel for 15 minutes to warm up the engine before running with B_0 and B_5 blends. Again, the engine was run on diesel before the engine was shut down. The same procedure was maintained for all fuel blends this is very important to make sure that the engine was free from biodiesel blends before proceeding to the next test. The engine was run at various speeds ranging from 1000 rpm to 4000 rpm at full load condition.

Table 6: Engine specification

1. Engine data		
Bore	89	Mm
Stroke	63	Mm
Crank radius	31.5	Mm
Length of connecting rod	110.0	Mm
Connecting rod ratio	0.29	
Compression ratio	8.5	
Combustion surface chamber	6221	$\mathrm{m}m^2$
Swept volume	391932	$\mathrm{m}m^3$
Compression chamber	52258	$\mathrm{m}m^3$
Combustion chamber	444189	$\mathrm{m}m^3$
Strokes	4	
2. operating data		
Speed	0	1/min
Initial pressure	NaN	bar
Fuel consumption	200	Kg/hr
Calorific value	43.5	MJ/kg
Mass fuel/stroke	Inf	g
Thermal input/stroke	Inf	J
3. complementary heating process		
isentropic exp.compresion	1.40	
isentropic exp.expansion	1.40	
Indicated efficiency	0.46	
Converted heat	Inf	J
Percentage conversion process	100	%

Converted heat comb proc 1	Inf	J
Converted heat comb proc 2	NaN	J
Initiation of combustion 1	20	“
Combustion period 1	40	“
Form factor 1	1.9	
Initiation of combustion 2	20	“
Combustion period 2	40	“
Form factor 2	1.9	

Figure 10: Engine test device

Table 7: Details of the exhaust gas analyzer

Equipment	Infralyt smart exhaust gas analyzer (model V1.011)
Measuring components and ranges	CO 0-10.00 % vol CO2 0-20.00 % vol HC 0-2500 ppm vol(C_6H_{14}) O_2 0-22.00 % vol NO 0-5000 PPM volume (available as an option) NOx 0-5000 PPM volume (available as an option)
Measuring components' absolute tolerances	CO ± 0.03%vol CO2 ± 0.5%vol HC ± 10 ppm vol(C_6H_{14})O_2 O_2 ± 0.1% vol NO ±25 PPM vol (available as an option) NOx ±25 PPM vol (available as an option)
Measuring components' relative tolerances	±5%

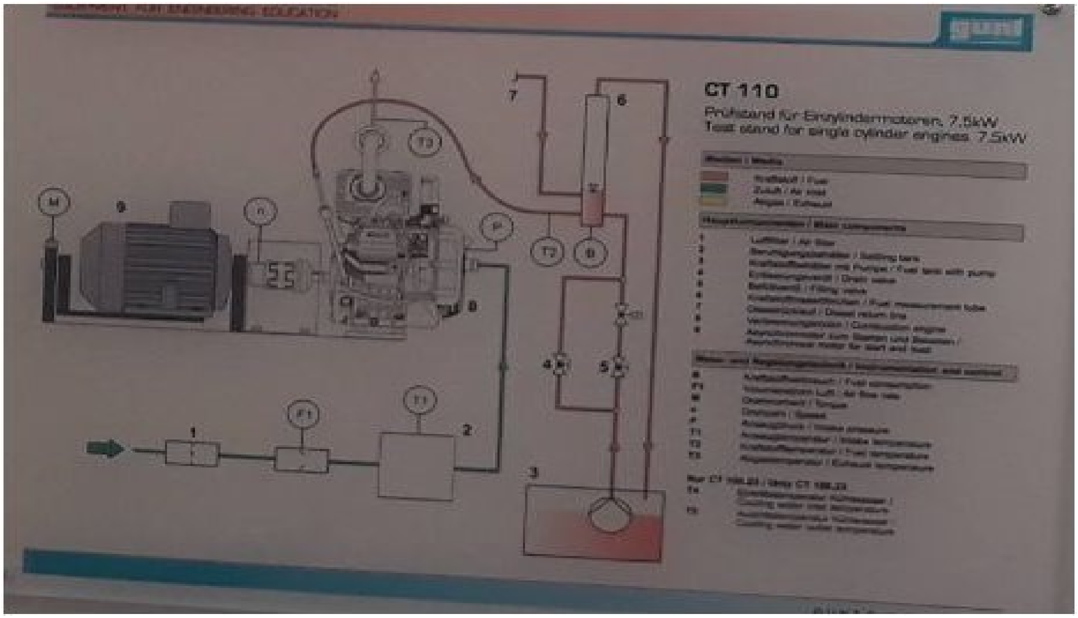


Figure 11: Schematic diagram of experimental set-up for engine test rig

Table 8: Properties of Diesel, gomenzer seed biodiesel

Properties	Diesel	gomenzer seed biodiesel
Calorific value (kJ/kg)	42000	41839
Density at 30 0C (kg/l)	0.85	0.882
Viscosity at 40	2.7	2.2
Flash point	52	46
Cetane number	50	62.69

Table 9: Proportions of Diesel, gomenzer seed biodiesel blends

S.NO		Diesel ,% Vol	gomenzer seed biodiesel ,% Vol
Diesel fuel		100	0
B5		95	5

CHAPTER FOUR

4. RESULTS AND DISCUSSION

4.1 Catalyst Characterization

The amount of chicken eggshell used for calcination was 100g. This powder was calcined at different temperatures such as 900°C and 1000°C with a constant time of 2hr. During calcination weight loss was observed.

4.1.1 XRD Analysis of CaO

As observed from (fig: 12) Based on the XRD analysis the powder calcined at 900 °C and 1000 °C shows a well-crystallized structure. The diffraction pattern of eggshell samples at various calcination temperatures showed a slight characteristic variation in CaO at 900 ºC and 1000 ºC. XRD spectra of calcined eggshell samples were obtained with Cu-ka1 (λ=0.154056 nm) at 37.5^{o,} a scan speed of 2 °/ minute, and a scan range of 5 to 80°. Fig: 12 shows 100% decomposition of CaCO3 to CaO.

The sharp peaks resulting at 2θ were 29.5 º, 34 º, 37.5 º, 45 º, and 50 º. So the catalyst used for transesterification was selected by testing their conversion efficiency. Therefore, the CaO calcined at 900ºC was used to produce biodiesel, since the catalyst that has a small particle size gives a higher yield. As (Asri et al. 2017) studied the yield obtained by using CaO calcined at 900°C was increased by 5% when CaO calcined at 1000°C was used. This may be due to the exposition of the catalyst with air because when the CaO contacts air it adsorbs CO2 and this leads to CaCO3 formation thereby decreasing the biodiesel yield.

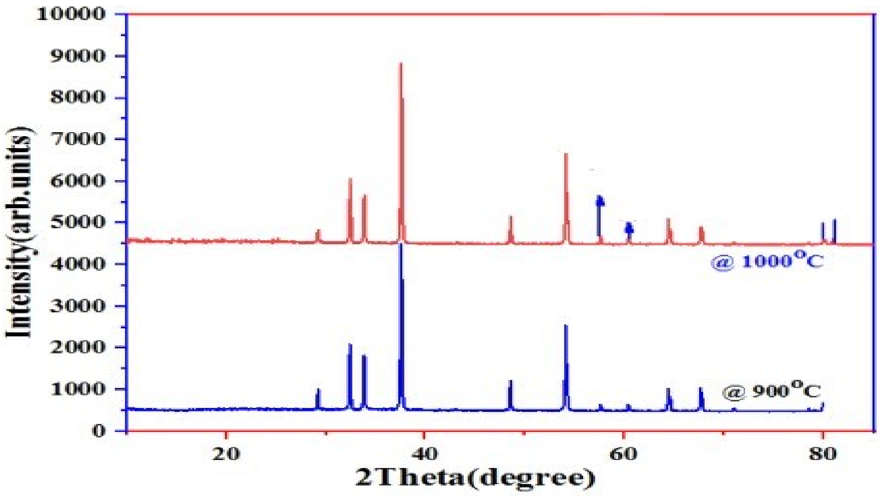


Figure 12: XRD pattern of calcium oxide powder calcined at different temperatures

4.2 Gomenzer seed oil characterization

4.2.1 Gomenzer Seed preparation, Oil extraction, and refining

4kg of Gomenzer seed was purchased from the market and dried using oven at a temperature of 102℃. Then, the dried Gomenzer seed is purifying from dust and the Gomenzer seed was powdered manually. The Gomenzer seed powders obtained were used for oil

Preparation. The amount of sample was weighted using a balance for each experiment. Then; it was dried in an oven at 102℃ for 12 hours. Again, the weight of the sample after drying was measured. Six experiments were conducted and the moisture content was determined for each of them and averaged to give the Gomenzer moisture content of 72.87%.

Table 10: the moisture content determination of the gomenzer conducted laboratory result

	Drying of time (hours)							% of moisture content
	0	2	4	6	8	10	12	
Sample weight (gram)	100	42	36	30	29.71	29.21	29.21	70.71
	150	54	38	32	30.62	29.98	29.98	80.01
	200	62	42	40	36.42	36.42	36.41	67.89
Average weight (gram)	150	52.67	38.67	34	32.25	31.87	64.21	72.87
% Average moisture content	-	64.89	26.58	12.01	5.15	1.18	50.37	-

The moisture content of the gomenzer of a sample with 100,150 and 200 grams was 70.71, 80.01and 67.89% respectively. Thus, the average moisture content of the three samples would be 72.87%.

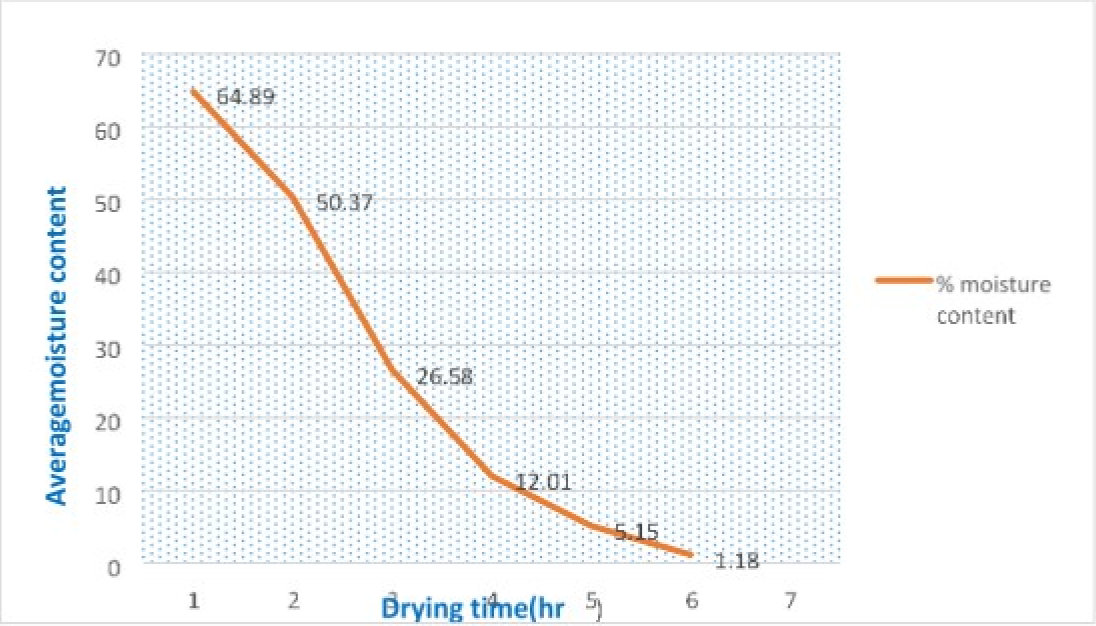


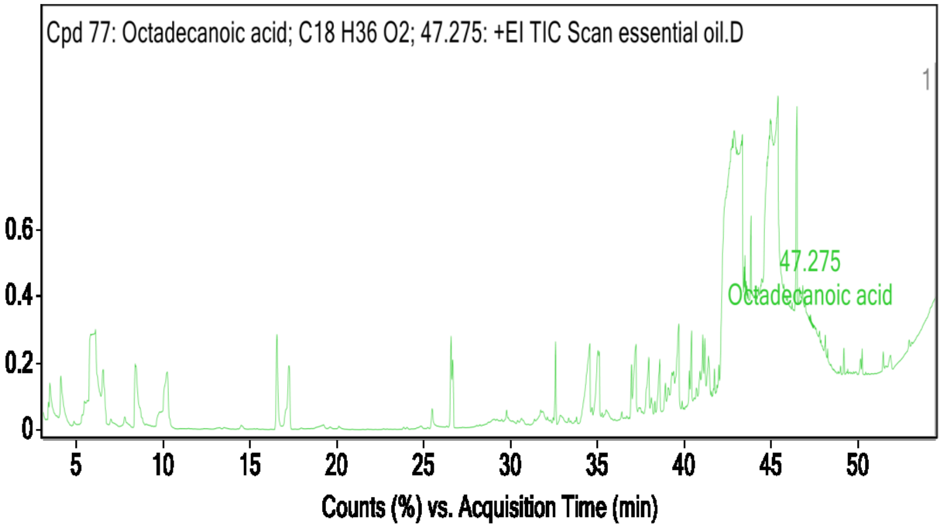
Figure 13: Average moisture content versus drying time

4.2.2 Fatty acid composition of gomenzer oil from GC-MS Analysis

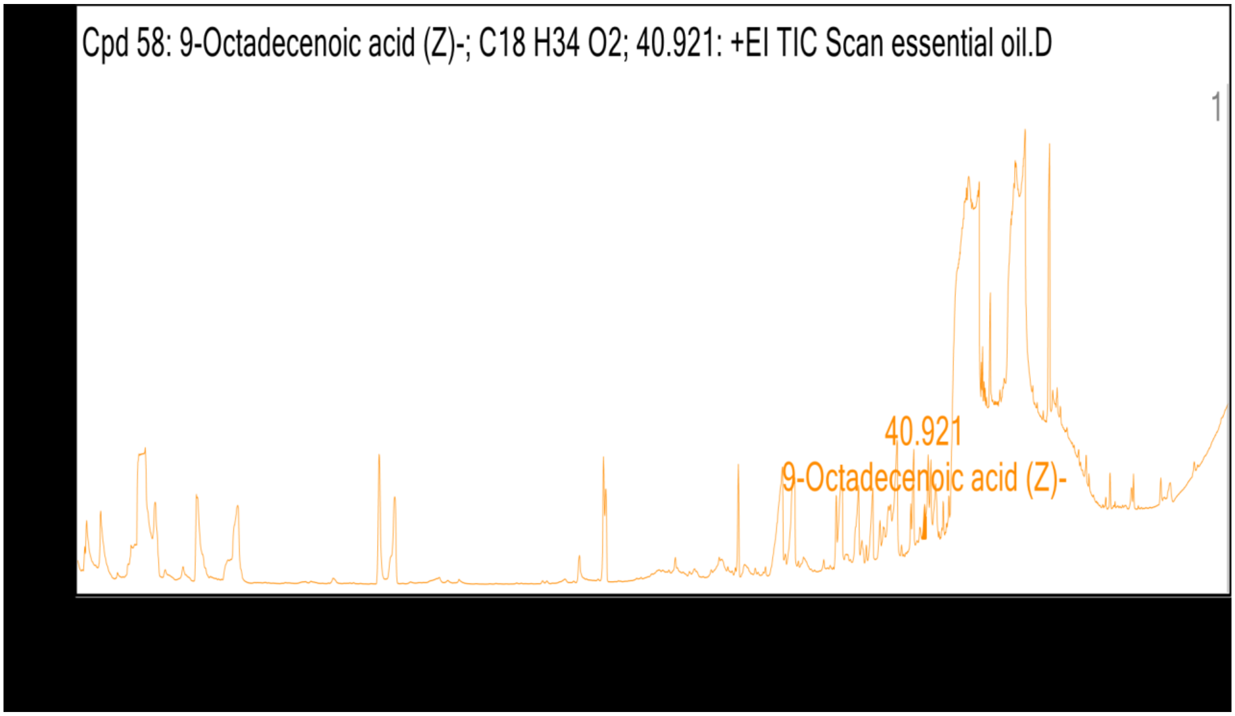
The fatty acid composition of gomenzer oil influences the cetane number and cold flow properties of biodiesel. The fatty acid composition of the oil was determined using GC-MS. The peaks obtained were identified using the standards of fatty acids. Based on GC-MS analyses from fig.14 the highest peaks that contain above 90% qualities were selected as the dominant composition of gomenzer oil. The calculated Percentage composition for the selected fatty acids was listed in table: 11.

The gomenzer seed oil was found to contain both saturated and unsaturated fatty acids. Linoleic acid was found which was contained the highest composition followed by Octadecanoic acid (0.223018%), Tridecanoic acid (0.089452966%), Octadecanoic acid (0.102971501%), etc. as shown in Table: 11. Except palmitic acid all values well comparable to the compositions founded by gs-ms.

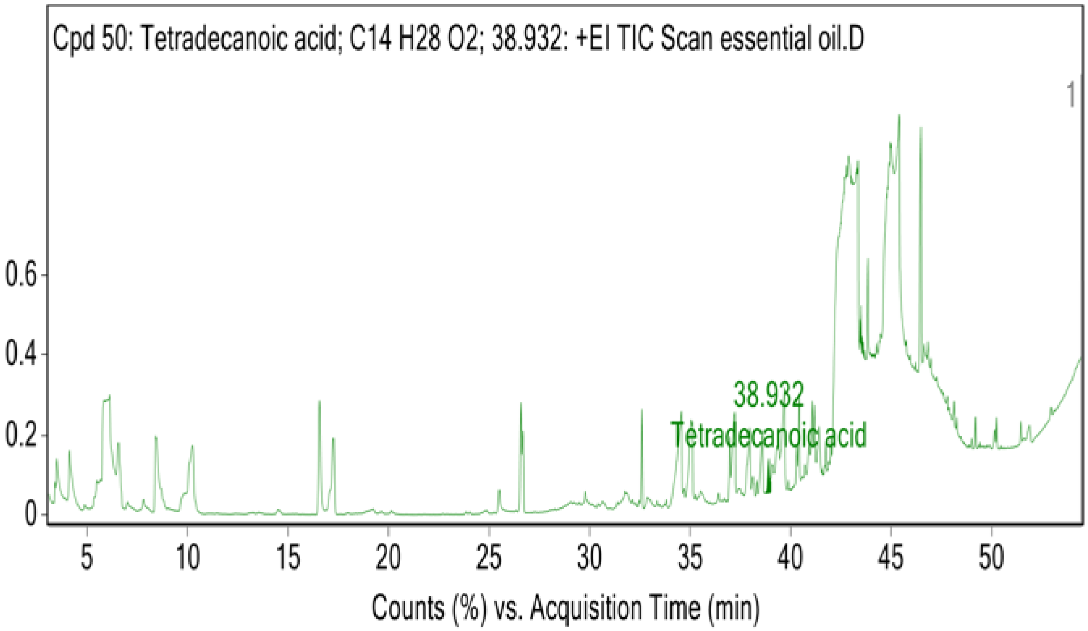
The low percent of palmitic acid may be due to the gomenzer growing soil nature and fertilizer used, temperature, and seed variety.



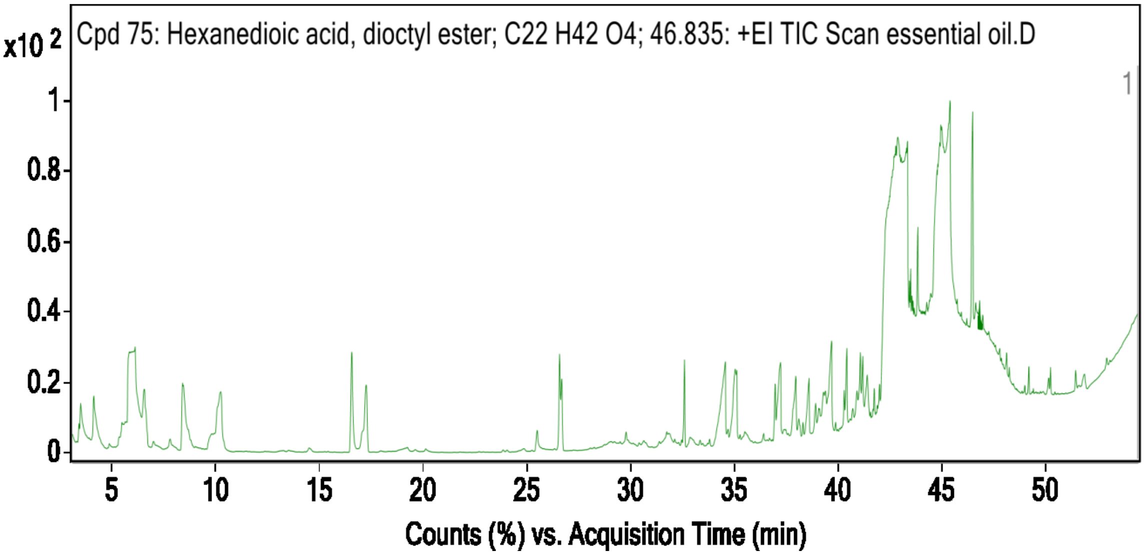
a) Octadecanoic acid



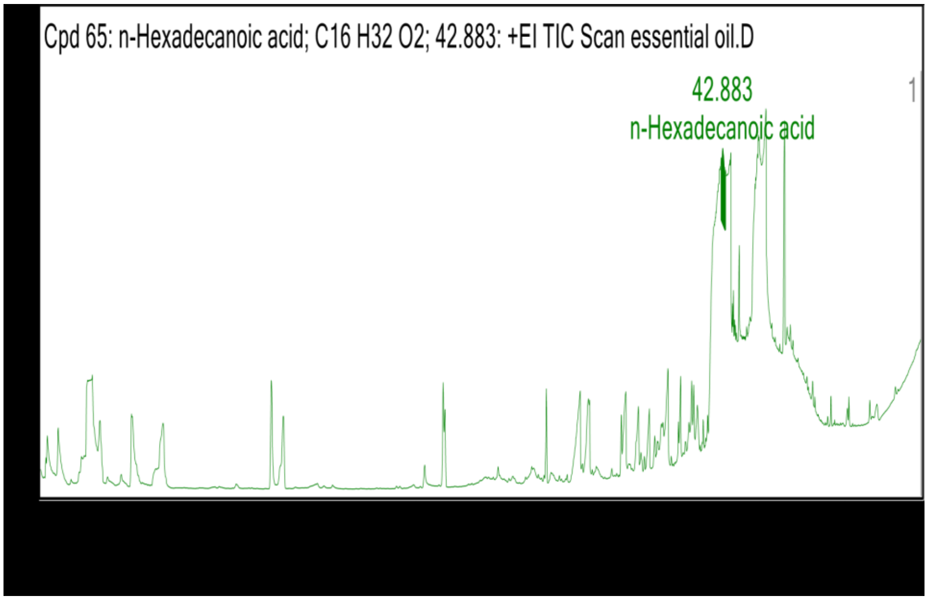
b) 9-Octadecenoic acid



c) 9-Tetradecanoic acid



d) Hexanedioic acid, dioctyl ester



e) n-Hexadecanoic acid

Figure 14: GC-MS Analysis of Fatty acid composition of gomenzer oil

Table 11: Fatty acid composition of gomenzer oil from GC-MS Analysis

Common Name	Systematic name	Area	structure	Wt%	M.weight in g/mol	RT
Erucic acid	Hexanedioic acid, dioctylester	3382385	C22 H42 O4	0.360018669	370	46.835
Palmitic acid	n-Hexadecanoic acid	160785	C16 H32 O2	0.017113842	256	43.057
Oleic acid	9-Octadecenoic acid (Z)-	3687700	C18 H34 O2	0.392516182	282	40.921
Stearic acid	Octadecanoic acid	208253	C18 H36 O2	0.022166302	284	48.263
Linoleic acid	Octadecadienoic acid	12585617	C18 H32 O2	1.33960418	284	37.97
Linolenic acid	octadecatrienoic acid	980227	C18 H30 O2	0.104334669	284	47.275
Myristic acid	Tetradecanoic acid	21188386	C14H28 O2	2.255276834	228	39.692

4.3. Characterization of Gomenzer oil

4.3.1 Determination of acid value

25ml of diethyl ether and ethanol mixture was added to 5gm of oil in a 250ml conical flask and the solution was titrated with 0.1N ethanoic KOH solution in the presence of 5 drops of phenolphthalein as indicator until the endpoint (colorless to pink) was recognized with consistent shaking. The volume of 0.1 N ethanoic KOH (V) for the sample titration was recorded in below.

Table 12: Determination of the acid number

Run	Titration volume(ml)	Color change
1	1.9	Pink to red
2	1.8	Pink to red
3	2	Pink to red
Average of this component	1.93	

The total acidity of oil in mg KOH/ gram was intended using the following equation:

$$\mathrm{AV} = (56 * \mathrm{N} * \mathrm{V})/\mathrm{W}$$

Where

V = Titration volume of potassium hydroxide (ml) = 1.93ml
N=Concentration of potassium hydroxide = 0.1M

W = the weight of oil sample (the mass in gram of the test portion) = 5 gram

Molecular weight of potassium hydroxide = 56 gram/ mol.L

$$\mathrm{AV} = \frac{56*\mathrm{N}*\mathrm{V}}{\mathrm{W}} = \frac{0.00193*0.1\frac{mol}{L}56\frac{gram}{mol}}{5\mathrm{gram}}$$

$$= 0.0021616$$

The percentage free fatty acid (FFA) content of the oil was calculated empirically using the acid value perversely determined as follows:

$\%\mathrm{FFA} = \frac{\mathrm{AV}}{2}$, Where = % FFA =percentage free fatty acid content

AV= acid value of oil

$$\%\mathrm{FFA} = \frac{0.0021616}{2} = 0.0010808$$

4.3.2 Determination of saponification value

2g of the sample was weighed and added to a conical flask then 25 ml of 0.1N ethanolic KOH solution was also added. Next, the sample was constantly stirred, and allowed to boil at 70 °C for 60min and a reflux condenser was connected to the flask containing the mixture. After that,3 to 5 drops of phenolphthalein indicator were added to the warm solution and then titrate with 0.5M HCl (volume Va was recorded) to the endpoint until the pink color of the indicator disappeared. Then a blank was prepared following the same procedure and (volume Vb was recorded). Then the SN was calculated using the equation below

$$SN = \frac{56.1 * N(V_{B-V_{S)}}}{W}$$

Where, W= weight of oil taken in gram = 2 gram

N= normality of HCl solution=0.5mol/l

VS= volume of HCl solution used in test in ml = 20ml

VB= volume of HCl solution used in blank in ml=34ml

Therefore,$SN = \frac{56.1 * o.5mol/l(34_{-20)}ml}{2gram}$

$= 0.196\frac{gram\ of\ KOH}{gram\ of\ oil}$

$= \frac{196\ mg\ of\ KOH}{gram\ of\ oil}$

4.3.3 Determination of specific gravity

After the sample was filled into a graduate cylinder at 50ml and its temperature was recorded; a hydrometer was used to measure the specific gravity of the oil at 15°C specified. Hence, the density of the oil was determined using specific gravity. In this measurement, the specific gravity of the gomenzer oil was 0.8105.

4.3.4 The density of the gomenzer seed oil

The specific gravity of the oil was measured by hydrometer to be 0.8105 and after multiplying the specific gravity of the oil with the water density, and then gained the density of the gomenzer oil.

Density of oil = the specific gravity of oil * the density of water ....eq. 3.6

The specific gravity of oil = 0.8105

The density of water = 1000kg/m^3 by subsisting all these values we would gain the density of the oil

Therefore, Density of oil = 0.8105*1000kg/m3

= 810.5 kg/m3

4.3.5 Kinematic Viscosity the gomenzer seed oil

45m1 of the gomenzer seed oil was poured into a test tube and a viscometer was used to measure the viscosity at a temperature of 35°C, the kinematic viscosity was determined after determined the dynamic viscosity of the gomenzer seed oil. The dynamic viscosity of the gomenzer seed oil was determined below;

Table 13: kinematic viscosity measuring data

The time that taken the ball reached the bottom of the viscometer	Time(s)
T1	9
T2	8
T3	7
Average of time(s)	8

Other data

Vd = volume displaced during measuring =3.45ml

Mb = mass of the ball = 5 gram

K = ball constant = 6.799mpa/ (cm3/gm)

F = multiplication factor at viscosity measurement at viscometer at 70°inclination 0.952

The density of ball was = db =$\frac{\text{Mb}}{\text{Vd}}$ = 5g/3.45ml = 1.4493 g/ml

$V = t_{av*}(d_b \; - d_{fo}) * K * F$

= 8(1.4493 g/ml -0.8105) 6.799*0.952mpa/ (cm3/gm

= 33.0778 m pas =33.0778*10^{-3} kg/s

Kinematic viscosity = 33.0778*10^{-3} kg/s/810.5 kg/m3

= 40.8116 mm^2/s

4.3.6 Determination of Iodine value (I.V) of gomenzer seed oil

The iodine value of the gomenzer seed oil was determined experimentally by titration described in equation (3.9) above.

Where

12.69 = factor proceeding from the reason between the number of transferred electrons and the molar mass of iodine values.

$$IV = \frac{12.69 * N(V_{B-v_{S)}}}{M}$$

Where, N = normality of sodium thiosuphate = 0.1

M = Mass of the sample = 0.125

VS = Volume of sodium thiosuphate used for a sample test

VB = size of sodium thiosuphate used for blank

Table 14: Titration result of iodine value of gomenzer seed oil

Factor proceeding	Mass of the Sample of gomenzer seed oil	The concentration of sodium thiosulfate (c)	Volume of sodium thiosuphate used for blank (VB)ml	Volume of sodium thiosuphate used for a sample test	I.V(gI2/100g) gomenzer seed oil during experimental titration
12.69	2	0.1	158	14	91.368
12.69	2	0.1	161	15	92.637
12.69	2	0.1	156	13.5	90.416

The I.V. was a measure of the total unsaturated (double bond) within the FAME product of the gomenzer seed oil. There iodine absorption occurs at the double bond position thus a higher quantity of the double bonds in the gomenzer seed oil sample and greater potential to polymerize in the engine and hence less stability. The process of transesterification reduces the iodine value to a small extent. The iodine value of this brassica carinata seed oil is shown in the above table 14, that was fitted the ASTM requirement (i.e., the iodine value of the gomenzer seed oil is less than 115). From the above table, the maximum amount of I.V. of the gomenzer seed oil was 92.637and the minimum amount of (I.v) gI2/100g of oil. There which used the maximum amount of I.v for other experimental analyses.

4.3.7 Determination of HHV

The determination of the heating value(calorific value) of the gomenzer seed oil was by using the empirical formula suggested above in equation(3.8)(Sivaramakrishnan and Ravi Kumar 2011). Higher heating value (the energy content per unit quantity) of acid-treated oil and fatty acid m ethyl ester was estimated from their Saponification value and iodine value using the following correlation:

HHV (energy content per unit quantity) = 49.43-[0.041(S.V) +0.015(I.V)]

= 49.43-[0.041(196) +0.015(92.637)]

= 45.297 MJ/Kg

Therefore the energy content of the gomenzer seed oil was 45.297 MJ/Kg

4.3.8 Determination of Cetane number (C.N)

The determination of Cetane number was used for knowing the ignition delay after injection of the fuel, of gomenzer seed oil. From equation (3.9) above determine the Cetane number of the gomenzer seed oil by correlation of the S.V and I.V suggested by(Nguyen n.d.) as shown below.

C.N = 46.3+ (5458/S.V)-0.225(I.V)

= 46.3+ (5458/196)-0.225(92.637)

= 53.28

4.3.9 Determination of the flash point of the gomenzer seed oil

The flash point of gomenzer seed oil was determined using an empirical formula by (Kaisan et al. 2017). The equations between FP and HHV of oil are shown in Eq (3.10) above. The flash point of the gomenzer seed oil is calculated from the equation below.

$$HHV = 0.021FP+32.12$$

$$45.297 = 0.021FP+32.12$$

$$FP = [(45.297 -32.12)/0.021]$$

$$= 627.476\ ℃$$

The flash point of the gomenzer seed oil was 627.476℃, that were fitted to the ASTMD93

4.3.10 Moisture content of gomenzer seed oil

The empty measuring cylinder was weighed with and without the amount of the gomenzer seed oil and dried in an oven at 105 ℃ for 6hr, weighing each 2hr till the constant weight was obtained, and finally, the weight 24gram was taken and compared with the initially recorded weight. Then, the percentage of the moisture content of the gomenzer seed oil was determined by the equation below,

The moisture content of the oil (%) = [redacted] $\times 100\%$

$$= 0.154\%$$

4.4 Statistical Analysis of the Experimental Results

4.4.1 Analysis of variance (ANOVA)

The Model F-value of 6.21 implies the model is significant. There is only a 1.25 % chance that a "Model F-value" this large could occur due to noise. P-Value of "Prob> F" less than 0.0500 shows model terms are significant. In this case, A, AB, and A^2 are significant model terms. The values greater than 0.1000 specify the model terms are not significant. If there are numerous insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. The lack of Fit F-value of 1.38 implies the lack of Fit is not significant relative to the pure error. There is a 36.94% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant nonexistence of fit is good - -we want the model to fit.

Table 15: The Analysis of variance (ANOVA) for Response Surface Quadratic Model

Source	Sum of squares	df	Mean square	F-value	P-value	
Model	105.08	9	11.68	6.21	0.0125	significant
A-temperature	22.45	1	22.45	11.94	0.0106	
B-molar to gomenzer oil ratio	1.50	1	1.50	0.7956	0.4020	
C-catalyst to gomenzer oil	1.36	1	1.36	0.7251	0.4226	
AB	41.53	1	41.53	22.08	0.0022	
AC	1.17	1	1.17	0.6202	0.4568	
BC	0.3911	1	0.3911	0.2079	0.6622	
A^2	42.47	1	42.47	22.58	0.0021	
B^2	1.94	1	1.94	1.03	0.3441	
C^2	1.91	1	1.91	1.01	0.3476	
Residual	13.17	7	1.88			
Lack of Fit	6.70	3	2.23	1.38	0.3694	Not significant
Pure Error	6.46	4	1.62			
Core Total	118.25	16				

The lack of Fit F-value of 1.38 implies the lack of Fit is not significant relative to the pure error. There is a 36.94% chance that a Lack of Fit F-value this large could occur due to noise. The non-significant lack of fit is good - -we want the model to fit.

✓Model adequacy checking

The negative "Pred R-Squared" indicates that the overall mean may be a better predictor of your response than the current model. In some cases, a higher order may also predict better. Adeq Precision measures the signal-to-noise ratio. A ratio greater than 4 is desirable. In this study 8.3512 indicates an adequate signal. This model can be used to conclude the design space.

Table 16: Model adequacy measures

Std.Dev.	**1.37**	**R-Squared**		**0.8887**
Mean	94.43	Adj R-Squared		0.7455
C.V.%	1.45	Pred R-Squared		-0.0013
		Adeq Precision		8.3512

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

Table 17: Regression coefficients and the corresponding 95% CI High and Low

	Factor	coefficient estimate	df	Standard error	95% CI Low	95% CI High	VIF
	Intercept	96.47	9	0.6110	95.03	97.92	
	A-temperature	1.07	1	0.4913	0.5357	2.86	1.03
	B-molar to gomenzer oil ratio	-0.4970	1	0.5572	-1.81	0.8206	1.13
	C-catalyst to gomenzer oil	0.4184	1	0.4913	-0.7435	1.58	1.03
	AB	3.60	1	0.7661	1.79	5.41	1.07
	AC	0.5400	1	0.6857	-1.08	2.16	1.0000
	BC	0.3493	1	0.7661	-1.46	2.16	1.07
	A^2	-3.18	1	0.6697	-4.77	-1.60	1.01
	B^2	-0.8012	1	0.7898	-2.67	1.07	1.20
	C^2	-0.6742	1	0.6697	-2.26	0.9095	1.01

Final Equation in Terms of Coded Factors:

Yield = + 96.47 +1.07* A -0.4970* B+ 0.4184* C +3.60 * A * B+ 0.5400* A * C +0.3493* B * C-3.18*A^2-0.8012* B^2 -0.6742* C^2

Final Equation in Terms of Actual Factors:

Biodiesel yield = – 474.04399+17.05924 * temperature –16.79484 * molar to gomenzer oil ratio –6.03268* catalyst to gomenzer oil +0.239982*temperature* molar to gomenzer oil ratio +0.108000*temperature * catalyst to gomenzer oil +0.116443* molar to gomenzer oil ratio* catalyst to gomenzer oil -0.127304$*temperature^2$ * -0.089023* molar to gomenzer oil ratio^2-0.674226 * catalyst to gomenzer oil ^{2}

As shown in fig.15 below, the normal probability plot indicates the residuals followed by the normal % probability distribution, in the case of this experimental data the points in the plots show fitted to the straight line in the figure, this shows that the quadratic polynomial model satisfies the assumptions analysis of variance (ANOVA) i.e. the error distribution is approximately normal.

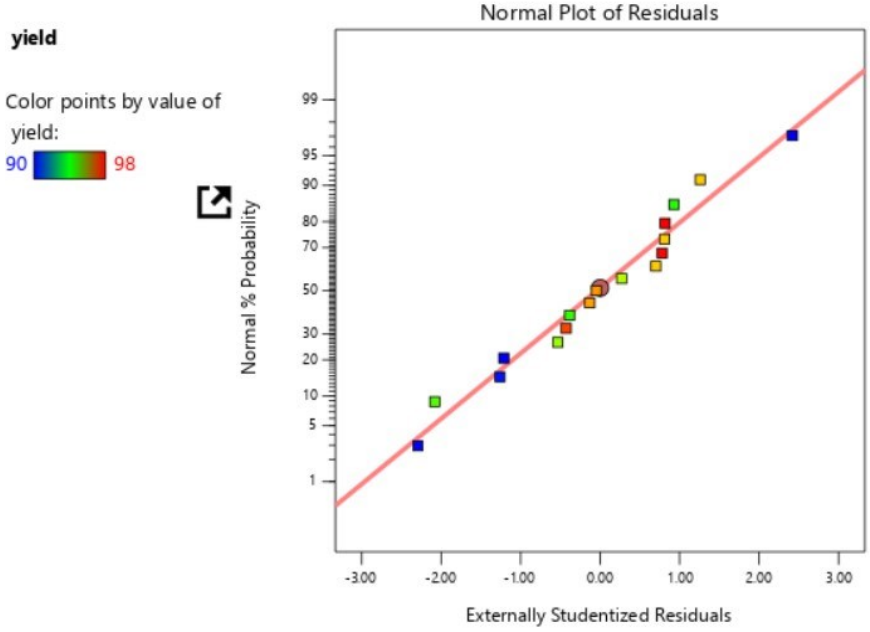


Figure 15: Normal probability plot of residuals

If the model is correct and the assumptions are satisfied, the residuals should be structured less; in particular, they should be unrelated to any other variable including the predicted response. A simple check is to plot the residues versus the fitted (predicted) values. A plot of the residuals versus the rising predicted response values tests the guess of constant variance. Fig 15 shows that random scatter modifying no need for an alteration to minimize personal error.

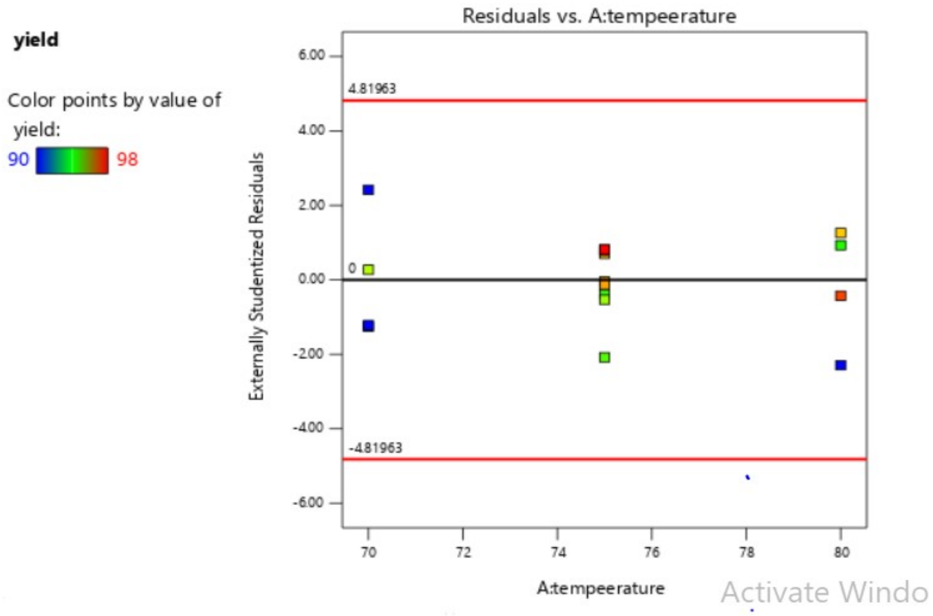


Figure 16: Residual versus predicted values plot

4.4.2 Response Surface plot for the Experimental Variables

To analyze the regression equation of the model, three-dimensional surface and 2D contour plots were obtained by plotting the response (yield of biodiesel) on the Z axis against any three variables while keeping the other variable at zero level.These plots are created to analyze the change in the response surface. The conical shape response surface plot indicates optimum operating conditions. The response-optimized value for the production of biodiesel was based on the three process variables described on the response surface plot. The response surface plots of the quadratic model are shown in Figures (16, 18, and 20). It was reported that Response surface plots provide a method to predict the yield of biodiesel for different parameter values of the tested variables and the interaction plots help in the identification of the type of interactions between these variables. The axes of the contour plot are the experimental variables and the area within the axes is termed the response surface.

Figure (16) shows the response surface plots developed as a function of temperature and methanol to oil ratio, while the catalyst load. Was kept constant at 2%. It was observed that the yield of biodiesel was more sensitive to temperature change when the temperature changed from 70 to 75 °C the yield of biodiesel reached the peak and beyond 70 °C the yield slightly decreased. The reason for this observation is that when the temperature approaches the boiling point of methanol, loss of alcohol would be occurred and this reduces the yield of biodiesel found in(Jazie, Pramanik, and Sinha 2012).

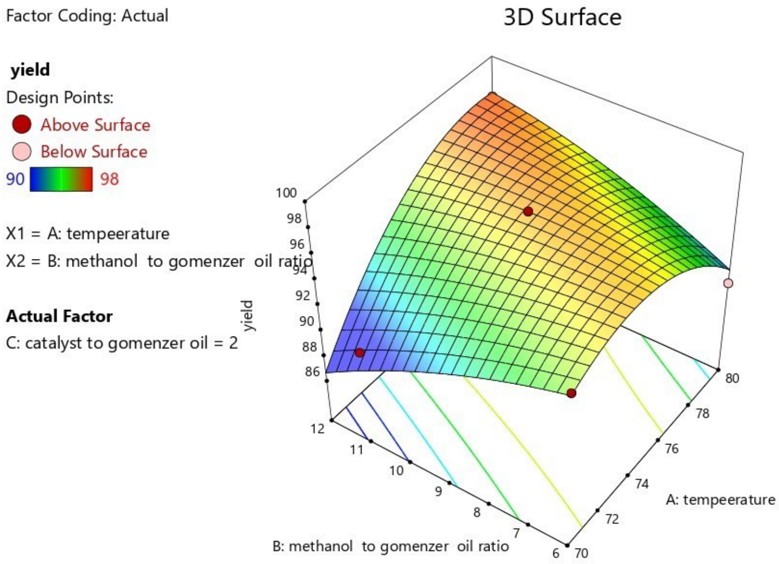


Figure 17: Response surface plots of the effect of methanol to oil ratio and temperature on the yield of biodiesel at fixed catalyst load

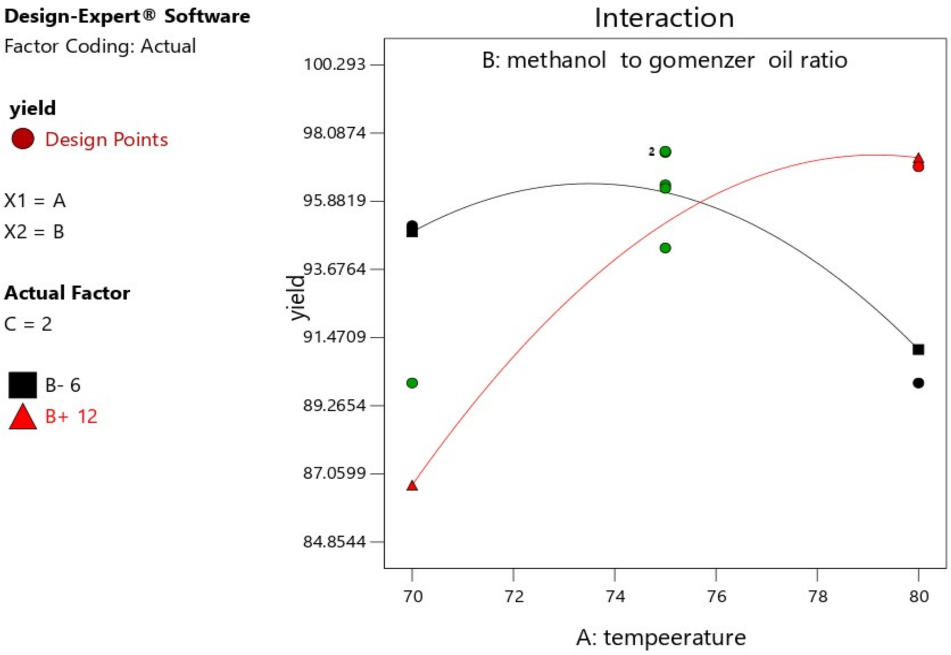


Figure 18: Effect and interaction of temperature and methanol to oil ratio (fixed) on the yield of biodiesel at the center of catalyst loading

As we observed from the figure (17) there is no interaction effect between the methanol to oil ratio and temperature at the given interval, but a Positive yield was observed at 6:1 methanol to oil ratio and around 75 ºC reaction temperature. The yield of biodiesel at a 3:1 methanol to oil ratio is less than at a 6:1 methanol to oil ratio at the same temperature.

As we can see below Figure (18) represents the response surface plots developed as a function of catalyst concentration and temperature, while the methanol to oil ratio was kept constant at 3:1. At a definite catalyst concentration, the yield of biodiesel increased slightly with reaction temperature from 70 to 75oC and nearly reached a peak. However, upon increasing the reaction temperature beyond 75 oC, there was a gradual decline in the yield, because as the reaction temperature closes to the boiling point of methanol that could lead to loss of methanol as reported by Musa Umaru and Aboje Audu Alechenu, 2015(Boey, Maniam, and Hamid 2009) As shown from the figure (20) the maximum yield of biodiesel was occurring in the region of 75 ℃ temperature and 2% catalyst loading.

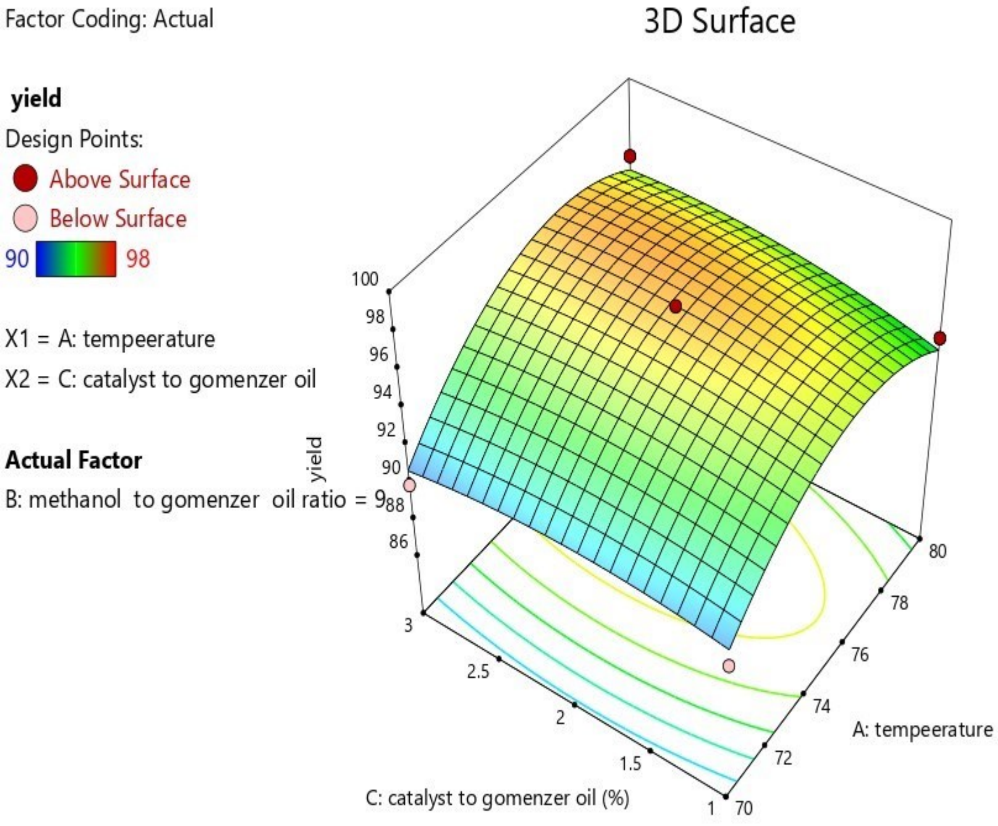


Figure 19: Response surface plots of the effect of catalyst load and temperature on the yield of Biodiesel at a fixed methanol-to-oil ratio.

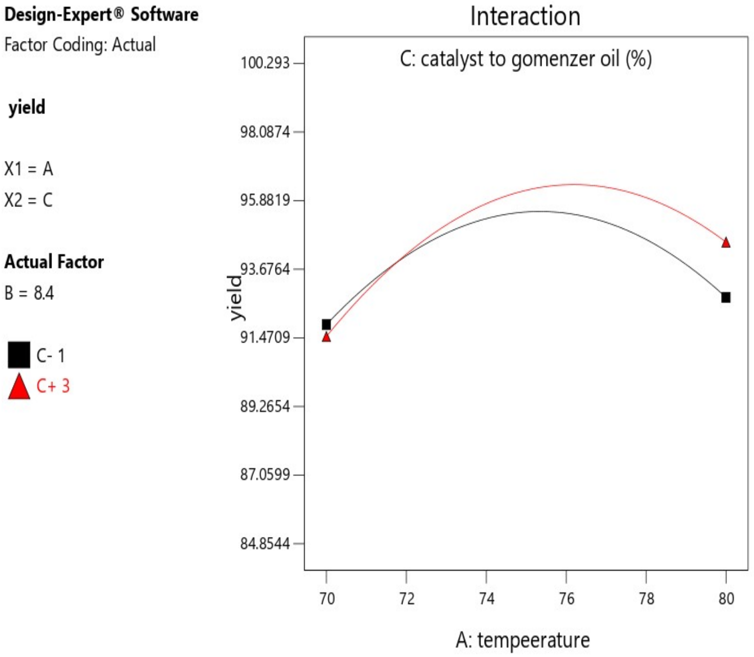


Figure 20: Effect and interaction of temperature and catalyst load (fixed) on the yield of biodiesel at the center of methanol to oil ratio

As we observed from figure (19) there was no interaction between catalyst loading and temperature, but Positive yield was observed at low temperature and high catalyst loading and also at high temperature and low catalyst loading. Because at low catalyst loading high temperature is required to obtain the positive result the same is true at low temperature and high catalyst loading. Low temperature and catalyst loading might not be enhanced the transesterification reaction to convert the oil into FAME. Too much catalyst used might initiate a saponification reaction and higher temperature also may cause methanol vaporization and solubility of reactants which causes difficulties in glycerol separation. The highest yield of biodiesel was observed at 75°C reaction temperature and 2% catalyst loading.

As we can see below Figure (20) shows the response surface plots developed as a function of methanol to oil ratio and catalyst loading, while the temperature was kept constant at 75°C. Upon increasing the catalyst loading from 1 to 2% with an increase of methanol to oil ratio from 6:1 to9:1, the yield of biodiesel increased highly. Beyond this ratio, the yield of biodiesel gradually decreased. The highest yield was obtained at a 9:1 methanol-to-oil ratio and 2% catalyst loading. The decrement in biodiesel yield with increasing catalyst loading from 2% - 3% may due to the formation of complexity during glycerol separation. The addition of an excess amount of catalyst beyond the optimum point increases the formation of an emulsion and the viscosity. This also retards the glycerin separation (Boey, Maniam, and Hamid 2009).

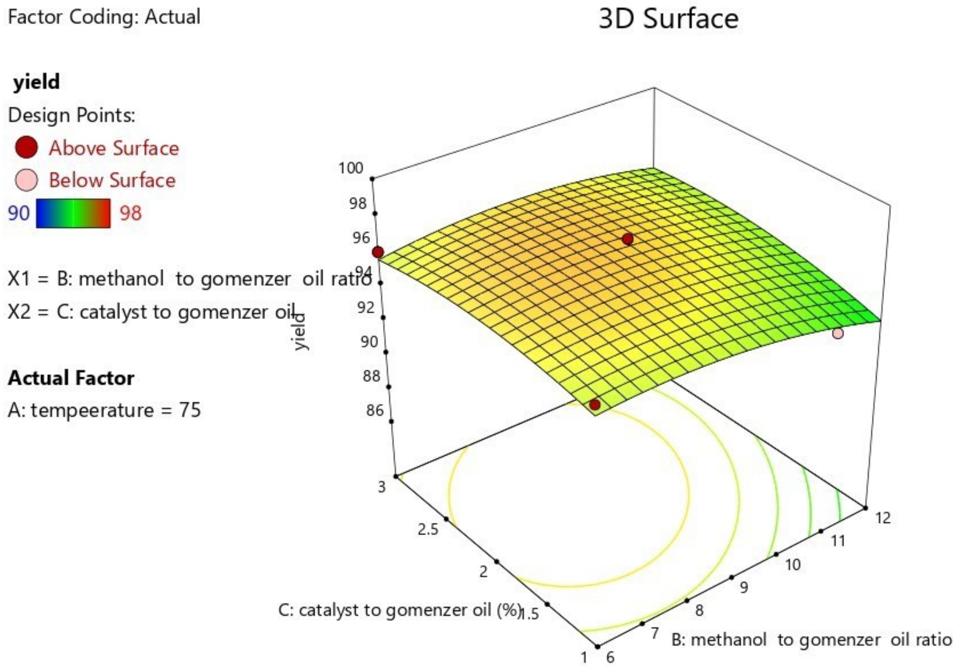


Figure 21: Response surface plots of the effect of catalyst load and methanol-to-oil ratio on the yield of biodiesel at a fixed temperature

As observed from figure:21 the methanol to oil ratio and catalyst load has small interaction before 12:1 and it has a positive effect on the yield of biodiesel at high catalyst loading until the methanol to oil ratio reaches 9:1. But, Beyond 9:1 methanol to oil ratio at a fixed catalyst loading the yield of biodiesel slightly decreases since emulsion is formed (difficulty during product separation).

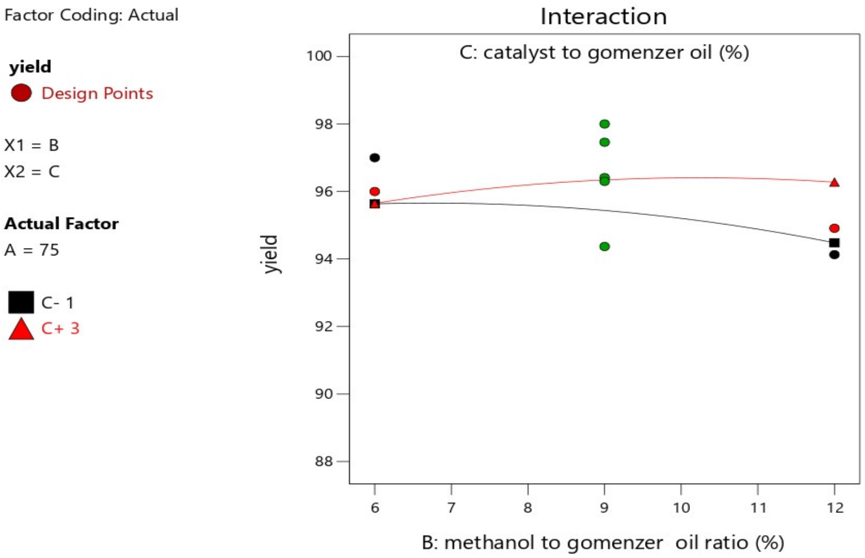


Figure 22: Effect and interaction of methanol to oil ratio and catalyst loading (fixed) on the yield of biodiesel at the center of temperature

4.4.3 Individual effect of experimental variables on the yield of biodiesel

4.4.3.1 Effect of temperature

Figure: 26 represents the effect of reaction temperature on the yield of biodiesel at a constant methanol-to-oil ratio and catalyst concentration at the center point. As shown in figure: 26 the yield of biodiesel was very sensible to the reaction temperature. The yield was highly increased as temperature increased from 70°C to 75°C. The optimum yield of biodiesel was obtained at around 75°C. When the reaction temperature was increased from 75°C to 80°C the biodiesel yield decreased gradually due to the vaporization of methanol because as methanol loses its amount in contact with a reactant in transesterification reaction decreased, as studied in(Jazie, Pramanik, and Sinha 2012)(Rezki et al. 2020).

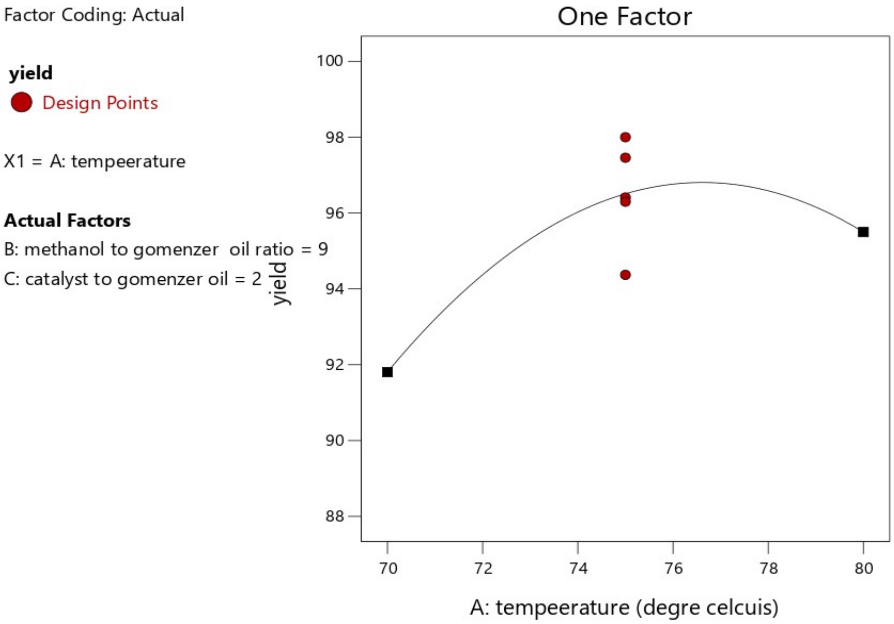


Figure 23: Effect of reaction temperature on biodiesel yield

4.4.3.2 Effect of methanol to oil molar ratio

Figure: 27 shows the effect of the methanol to oil molar ratio on the yield of biodiesel at constant temperature and catalyst concentration in the center point. In addition, the figure shows that the yield of biodiesel is highly affected by the methanol-to-oil ratio, as the methanol-to-oil ratio increases from6:1 to 9:1 the yield sharply increases. Whereas, beyond 9:1 the yield of biodiesel to some extent diminished. The decrease in biodiesel yield at a high molar ratio beyond 9:1 could be due to the dissolving of glycerol in the methanol and this hinders the interaction of methanol with the catalyst (Tan et al. 2015).

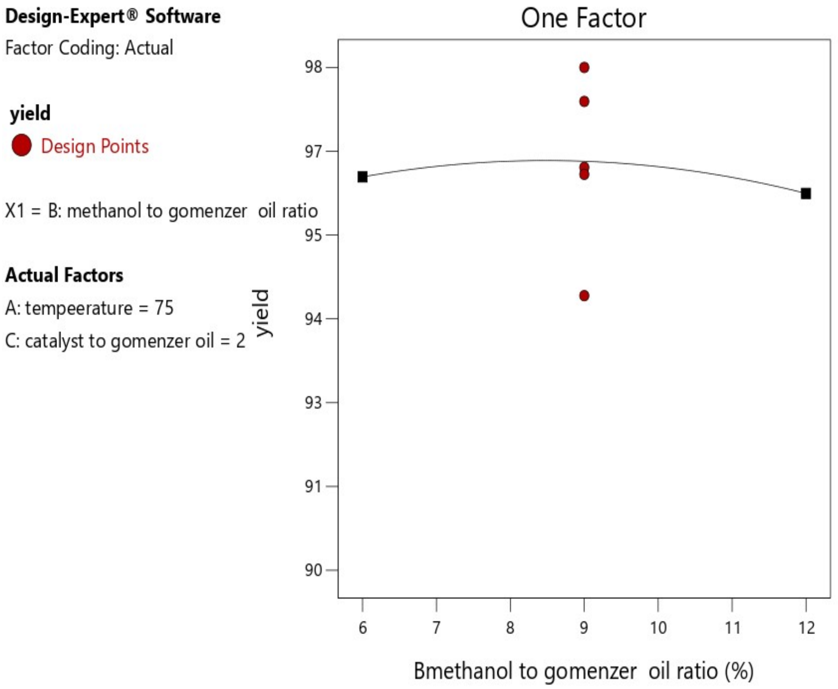


Figure 24: Effect of methanol to oil ratio

4.4.3.3 Effect of Catalyst loading

Figure: 28 shows the effect of catalyst loading on the yield of biodiesel at constant temperature and methanol to oil ratio in the center point. As shown in the figure the yield of biodiesel was affected by catalyst loading, as the amount of catalyst increased from 1% to 2% an increase in biodiesel Yield was observed, beyond 2 % the biodiesel yield was slightly decreased, because of the increase in loading might cause viscous nature of catalyst and reactants, this increases the difficulties in mixing and separation process (Bello et al. 2013). Insufficient amount of catalyst results in an incomplete reaction so this reduces the yield.

In this study, the biodiesel yield of 98% was obtained at a reaction time of 2 hr, 2 wt. % of CaO derived from chicken-eggshell catalyst, the temperature of 75 °C, and methanol/oil molar ratio of 9:1, while the previous work reported by (Tan et al. 2015), the highest yield of biodiesel production was 94% and 96%, obtained using CaO derived from ostrich- eggshell and chicken eggshell, respectively. The optimum operating conditions employed in the previous study were at 2hr reaction time, a catalyst to oil ratio of 2 wt. %, temperature of 75°C, and methanol/oil molar ratio of 9:1. The change may be due to better catalyst preparation.

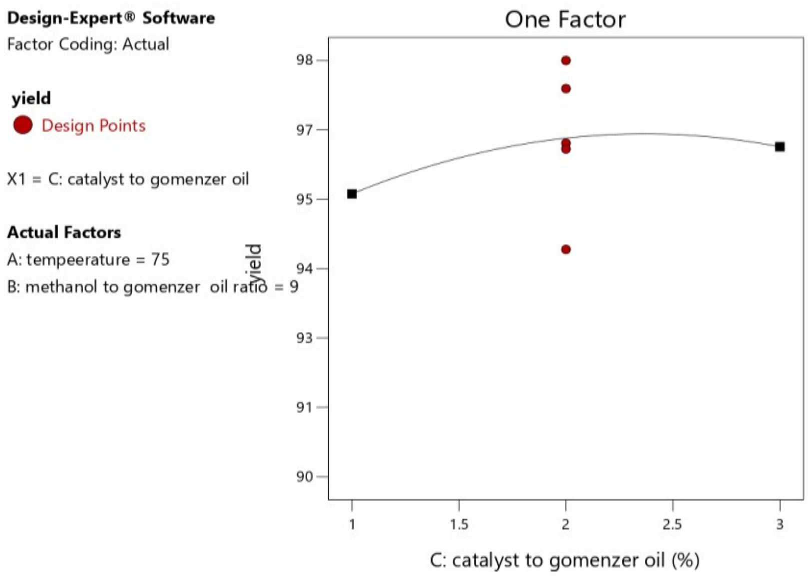


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Figure 25: The effect of catalyst loading on the yield of biodiesel

4.5 Optimization of operating process variables in transesterification reaction using RSM

The response surface methodology (RSM) is a collection of statistical and mathematical methods useful for developing, improving, and optimizing process. It also has important applications in the design, development, and invention of new products, as well as in the improvement of remaining product designs. The optimization of reaction parameters for biodiesel production from Gomenzer seed oil using transesterification reaction was summarized below:

Table 18: Goal of optimization and limits of process parameters

Name	Goal	Lower limit	Upper limit
Temperature (℃)	in range	70	80
Methanol to gomenzer oil ratio	in range	6	12
Catalyst loading (%)	in range	1	3
Yield (%)	maximize	89.99	98

The optimum possible solutions in the transesterification reaction to produce biodiesel were presented in table: 16 and Figure (26-28) below in the form of a response surface plot. The predicted optimum yield of biodiesel 98.166% was observed at 75 ℃, 2 %, and 9 of methanol to oil ratio. The yield was carefully chosen by considering the economic benefit and environmental effect.

Table 19: Optimum possible solutions

Number	Temperature	methanol to gomenzer oil ratio	Catalyst loading	Yield	Desirability	
1	**78.091**	**11.356**	**2.281**	**98.166**	**1.000**	**selected**
2	78.941	11.660	2.837	99.023	1.000	
3	79.017	11.367	2.614	98.656	1.000	
4	79.668	11.800	2.130	98.370	1.000	
5	80.000	12.000	2.000	98.260	1.000	
6	78.071	11.499	2.211	98.147	1.000	
7	78.420	11.824	2.045	98.151	1.000	
8	78.075	10.768	2.427	98.006	1.000	
9	79.400	10.875	2.657	98.306	1.000	
10	78.617	11.567	2.933	98.926	1.000	
11	77.306	11.198	2.485	98.023	1.000	
12	78.833	11.471	2.123	98.135	1.000	
13	77.863	11.486	2.447	98.322	1.000	
14	78.032	11.556	2.726	98.616	1.000	
15	77.300	11.690	2.378	98.094	1.000	
16	79.200	11.958	1.981	98.222	1.000	
17	78.147	11.310	2.222	98.090	1.000	

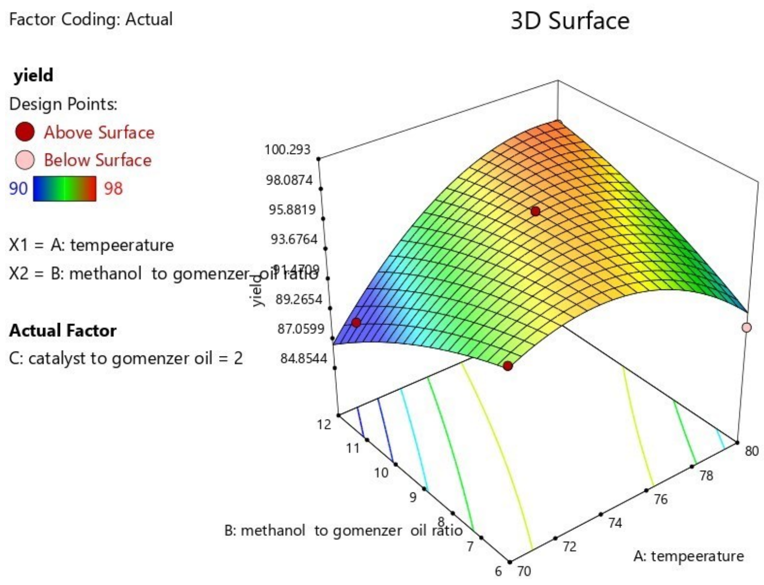


Figure 26: Response surfaces of predicted biodiesel yield at a fixed catalyst loading

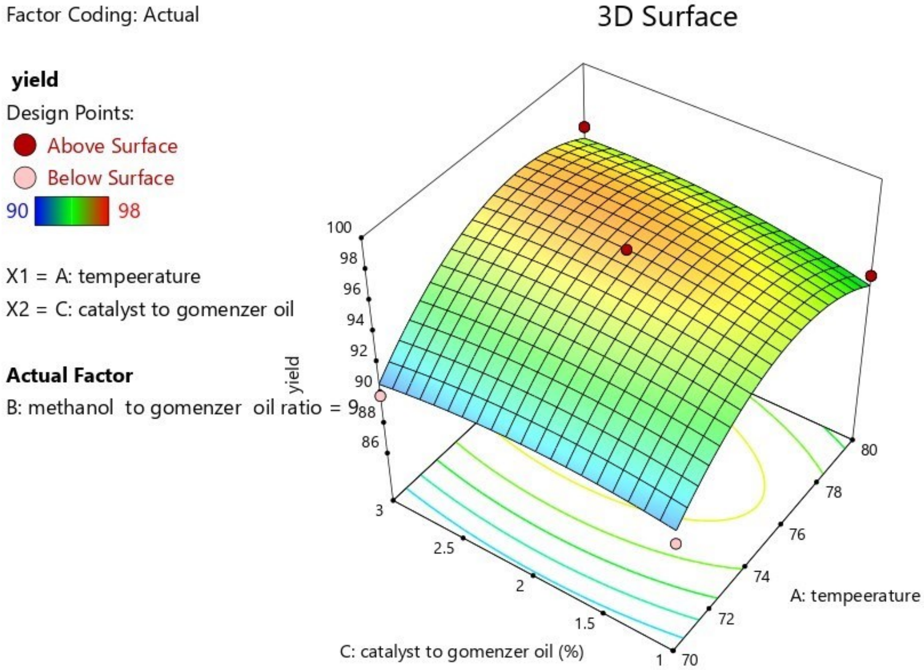


Figure 27: Response surfaces of predicted biodiesel yield at fixed methanol to oil ratio

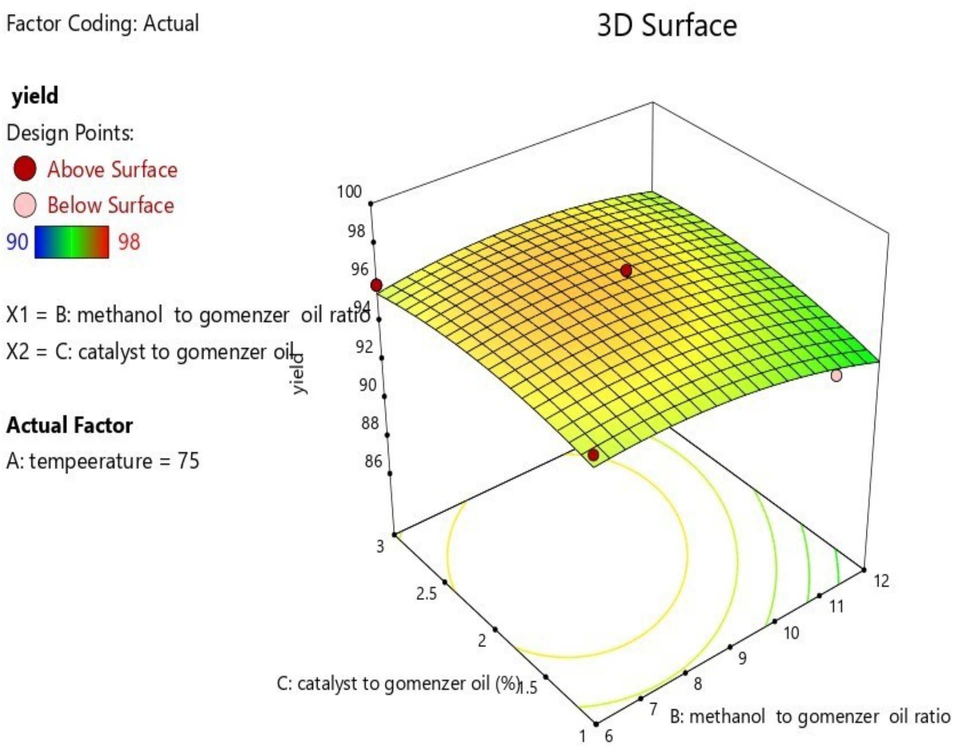


Figure 28: Response surfaces of predicted biodiesel yield at the fixed reaction temperature

4.6 Model validation

Based on the results of RSM the optimum process conditions in the transesterification reactions were 75 °C, 2 %, and a 9:1 methanol to oil ratio. To confirm the validity of the RSM model results a confirmation experiment with a triplicate set was conducted at the above-specified optimum process conditions predicted by the model. Under these conditions, the yield of biodiesel was found 97.65 %, which was close to the RSM result of 98.166%. This shows the experimental values were found to be close to the predicted values and hence the model was validated. Thus the model was useful to predict the yield of biodiesel and also to obtain optimum process parameters for the transesterification reaction of GSO.

4.7 Characterization of Biodiesel

The biodiesel obtained through the transesterification process was characterized to know the fuel properties. The characterization of the biodiesel produced from the gomenzer seed oil was conducted from the experimental analysis. The quality of biodiesel was very important for the performance and emission characteristics of a diesel engine. The characterization was performed for the following properties namely saponification value, acid value, flash point, cetane number, kinematic viscosity, and dynamic viscosity. These properties were chosen for these main reasons. One is because they are the defining properties of biodiesel and the other is because of the limitation of resources to perform other tests.

4.7.1 Acid value of biodiesel

The titration method was used to determine the acid value of biodiesel. The experimental procedures were the same as those used in the gender seed oil acid value in the equation (3.12). Therefore, the acid value of biodiesel for each experiment is given in the table below.

The Acid value was calculated as:

$$\mathrm{AV} = \frac{56 * \mathrm{N} * \mathrm{V}}{\mathrm{W}}$$

Where, V =the volume expressed in a milliliter of 0.1N solution of ethanolic KOH

W = the weight of the oil sample (the mass in grams of the test portion)

N = concentration of ethanolic KOH

56 = molar mass of KOH

Table 20: Determination of the acid number

Run	Titration volume(ml)	Color change
1	2.1	bright red
2	2.5	bright red
3	2.3	bright red
Average of this component	2.3	

The whole acidity of oil in mg KOH/ gram was considered using the following equation:

Where

V = Titration volume of potassium hydroxide (ml) = 2.3ml

N=Concentration of potassium hydroxide = 0.1M

W = the weight of oil sample (the mass in gram of the test portion) = 5 gram

Molecular weight of potassium hydroxide = 56 gram/ mol.L

$\mathrm{AV} = \frac{56 * \mathrm{N} * \mathrm{V}}{\mathrm{W}} = \frac{0.0023 * 0.1 \frac{mol}{L} 56 \frac{gram}{mol}}{5\mathrm{gram}}$

= 0.0002576

= 0.02576 %

Percentage free fatty acid (FFA) content of the oil was calculated empirically using the acid value perversely determined as follow:

$$\%\mathrm{FFA} = \frac{\mathrm{AV}}{2}$$

Where

%FFA – percentage free fatty acid content

AV= acid value of oil

$\%\mathrm{FFA} = \frac{0.0002576}{2}$ = 0.001288(mgKOH/g)

By taking the average titration volume, the acid value of biodiesel is 0.0002576(mgNaOH/g). The acid is used to determine the amount of free fatty acid content in the biodiesel. The lower acid value indicates that the quantity of free fatty acid in the biodiesel is low(Niza et al. 2011).During this result the color of change to bright red.

4.7.2 Determination of Saponification Number (SN)

The saponification value of biodiesel was determined by a titration procedure. The method of titration was the same as for gomenzer seed oil saponification determination in equation (3.13). The expression for saponification value (S.V) is given by the following equation

$\mathrm{SN} = \frac{56.1 * \mathrm{N}(\mathrm{V}_{\mathrm{B}} - \mathrm{V}_{\mathrm{S}})}{\mathrm{W}}$ ....Equation 3.13

Where, W= weight of oil taken in gram = 2 gram

N= normality of HCl solution=0.5mol/l

VS= volume of HCl solution used in the test in ml

VB= volume of HCl solution used in blank in ml

Table 21: determination of saponification value of biodiesel

The molecular mass of KOH	Actual Normality of the HCL(mol/l)	Black level titration volume(VB)ml	Titration volume(ml)	Saponification value (mgKOH/g)
56.1	0.5	28	15	182.325
56.1	0.5	28	17	154.275
56.1	0.5	28	16	168.3

The saponification value shows the amount of biodiesel that changes to soap by applying a KOH catalyst at a high temperature in the presence of water. The lower saponification value of the result is higher in the quality of the product. So from the above table, the saponification value of the biodiesel was between 154.275mgNaOH/g to 182.325mgNaOH/g.

4.7.3 Specific gravity of the biodiesel

For the determination of the specific gravity of the biodiesel, we follow the same procedure as that of the gomenzer seed-specific gravity measuring technique. After the sample was filled into a graduate cylinder at 50ml and its temperature was measured and recorded; a hydrometer was used to measure the specific gravity of the biodiesel at 15 °C specified. From this hydrometer measurement, the specific gravity of the biodiesel reads 0.8821. Therefore we compare it to ASTMD6751 for biodiesel is acceptable

4.7.4 Determination of the density of biodiesel

The specific gravity of the biodiesel produced from the gomenzer seed oil was measured by hydrometer to be 0.8821 and after multiplying the specific gravity of the oil with the water density, and then gained the density of the gomenzer biodiesel was. Therefore the density of the biodiesel was determined by the correlation of the specific gravity of biodiesel and the water density as follows.

The specific gravity of biodiesel = density of biodiesel /water density

Density of biodiesel (kg/m^3) = $0.8821*1000kg/m^3$

$= 882.1kg/m^3$

From this result, the density of the biodiesel was 882.1kg/m3, which is between the literature density (ASTM D6751 for biodiesel 870–890 kg/m3). Therefore the density of the biodiesel was acceptable.

4.7.5 Determination of Dynamic Viscosity

The dynamic viscosity of biodiesel was measured by Viscometer. 45m1 of gomenzer biodiesel was poured into a test tube and preparing stopwatch to measure the time to reach the ball from the top of the viscometer to the end of the viscometer, this was used to measure the viscosity at a temperature of 40℃.

Table 22: Experimental data during dynamic viscosity measurement

The time that taken the ball reached the bottom of the viscometer	Time(s)
T1	5
T2	4
T3	3
Average of time(s)	6

Parameters of dynamic viscosity measurement db = Density of biodiesel = 882.1kg/m^3

Mb = mass of the ball = 5 gram

Vd = volume displaced during measuring =3.45ml

K = ball constant = 6.799mpa/ (cm3/gm)

F = multiplication factor at viscosity measurement at viscometer at 70°inclination 0.952

V = kinematic viscosity (m^2/s)

μ = dynamic viscosity (N s/m^2),

ρ = density (kg/m3)

The density of ball was = db = Mb/Vd = 5/3.45 = 1.4493g/ml

$\mu = t_{av*}(d_{ball} - d_b) * K * F$

= 6*(1.4493 - 0.8821) 6.799mpa/ (cm3/gm)* 0.952

= 22.0277mpa = 22.0277*10^{-3} kg/ms

4.7.6 Kinematic Viscosity of biodiesel

The kinematic viscosity of biodiesel was determined after determining the dynamic viscosity of the gomenzer seed oil. The viscosity was a very important property related to biodiesel utilization in direct injection diesel engines. The high value of viscosity gives rise to poor fuel atomization, incomplete combustion, and deposition in the injectors. Therefore, the biodiesel viscosity must be low. The kinematic viscosity of the biodiesel was determined by the following correlation

V=kinematic viscosity

μ = dynamic viscosity of biodiesel

$V = \mu / \rho$

$= 20.945*10^{-3}$ kg/ms/802.1kg/m^3

$=2.61127*10^{-5}$ m^2/s

$= 2.6$ mm^2/s

The kinematic viscosity of the biodiesel was less than the kinematic viscosity of the Gomenzer seed oil because the biodiesel was less viscose than the gomenzer seed oil. The kinematic viscosity decreased when the transesterification temperature was increased. Therefore, the superlative temperature for the synthesis of biodiesel was 70°C .

4.7.7 Determination of Iodine Value (IV) of biodiesel

The iodine value indicates the degree of unsaturation of the biodiesel components. So the iodine value is a measure of the total unsaturated bond within the FAME product of the gomenzer seed oil. Therefore iodine absorption occurs at the double bonds in the gomenzer seed biodiesel sample and has the greater potential to polymerize in the engine and less stability. The values of iodine numbers minimize to a small extent in the Trans esterification process. The experimental results of the iodine number of biodiesel are shown in the table.23 below. The iodine values of the biodiesel determined

Experimentally titration is described in equation (3.14) above.

Where

12.69 = factor proceeding from the reason between the number of transferred electrons and the molar mass of iodine values.

Ms = mass of biodiesel sample = 2gram

N= concentration of sodium thiosulfate used = 0.1mol/liter

$IV = \frac{12.69*N(V_{B-}V_{S)}}{M}$Where, N = normality of sodium thiosuphate

Table 23: Experimental titration of biodiesel iodine number

Factor proceeding	Mass of the Sample of biodiesel	Concentration of sodium thiosulfate (c)	Volume of sodium thiosuphate used for blank (VB)ml	Volume of sodium thiosuphate used for a sample test	I.V(gI2/100g) gomenzer seed oil during experimental titration
12.69	2	0.1	143	10	84.389
12.69	2	0.1	144	12	83.754
12.69	2	0.1	140	13	80.582

The iodine number specifies the amount of iodine in g that is consumed by 100g of the biodiesel sample under the given conditions. The determination of the iodine number by titration with a solution of sodium thiosulfate was described by the. From this experimental analysis, the iodine number of the biodiesel was in the range between "80.582 to 84.389(gI2/100g)", so the maximum amount of iodine number was used to determine the higher heating value of the biodiesel.

4.7.8 Higher heating value of the biodiesel (HHV)

The heating value of biodiesel was determined by using the same procedure of the gomenzer seed oil in the above eq (3.8). The determination of the heating value (calorific value) of biodiesel was by using the empirical formula suggested by (C. E. Goering et al. 1982).The higher heating value (the energy content per unit quantity) of fatty acid methyl ester was estimated from their saponification value and iodine value using the following correlation:

HHV (energy content per unit quantity) = 49.43-[0.041(S.V) +0.015(I.V)]

= 49.43-[0.041(154.275) +0.015(84.388)]

= 41.839J/kg

Therefore the biodiesel produced from the gomenzer seed oil was a higher heating value.

4.7.9 Biodiesel Cetane number

The Cetane number was another important property of fuel; this was a measure of the combustion quality of diesel during compression ignition. The determination of Cetane number was used to know the ignition delay after injection of the fuel, of biodiesel. The Cetane number was determined with the same procedure as that of the Cetane number of gomenzer seed oil. From equation (3.11) above determine the Cetane number of the biodiesel by correlation of the saponification value and iodine value suggested by (Nguyen n.d.).

$C.N = 46.3+ (5458/S.V)-0.225(I.V)$

$= 46.3+ (5458/154.275)-0.225(84.388)$

$= 62.69$

From this result, the Cetane number of biodiesels was high; high Cetane numbers also lead to incomplete combustion and smoke if the fuel ignites too soon by not allowing enough time for the fuel to mix with air for complete combustion.

4.7.10 Measurement of flash point of biodiesel

The flash point of the biodiesel was determined using the empirical formula by (Kaisan et al. 2017)and the flash points of biodiesels were determined according to the standard procedure of the flash point measurement of flash point of biodiesel, i.e.ASTMD93. The equations between FP and HHV of biodiesel had shown Eq (3.10) above the same procedure to gomenzer seed oil. The flash point of the biodiesel is calculated from the equation below.

$HHV = 0.021FP+32.12$

$41.839J /kg = 0.021FP+32.12$

$FP = [(41.839-32.12)/0.021]$

$=46.280$

The flash point of the biodiesel was 46.28095℃, which was fitted to the ASTMD93. The high value of the flash point because do to the maximum amount of the heating value. Therefore, due to this result of the higher flash point of biodiesel; it was too safe to store biodiesel compared to Petro diesel.

Finally, the results concluded as follows in table form with a comparison to ASTM D6751 and EN 14214 standards for biodiesel fuels and ASTM D 975 for petroleum diesel fuel.

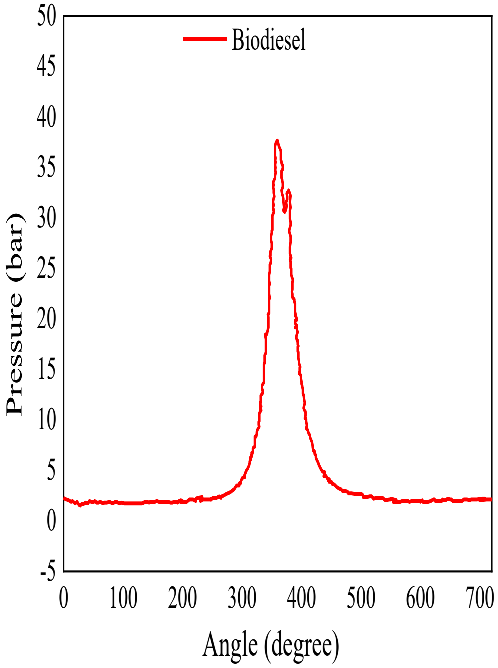
Table 24: standards for biodiesel and petroleum diesel fuel with obtained results

Property Specification	**Units**	**Diesel ASTM D975**		**Biodiesel**				**Biodiesel from gomenzer seed oil**
		Test method	**limit**	**ASTM D6751**		**EN14214**		**Obtained results**
				TESTMethod	**limit**	**TESTMethod**	**limit**	
Flash point	°C	ASTM D975	60-80	ASTM D93	130 min	ENISO 3679	101min	46.280
Cetane number		ASTM D4737	52	ASTM D613	52minimum	ENISO 5165	53 min	62.69
Density at 15°C	Kg/m^3	ASTM D1298	82-860	ASTM D1298	880	EN ISO 3675/ 12185	860-900	882.1kg/m3
Kinematic viscosity at 40°C	mm^2/s	ASTM D445	2.0 to 4.5	ASTM D445	1.9 to 6.0	EN ISO 3104	3.5 to 5.0	2.6 mm^2/s
Iodine number	gI2/100g	_	_	_		EN 14111	_	84.389(gI2/100g)
Acid number	mg KOH/g	_	_	ASTM D664	0.5 max	EN 14104	0.5v max	0.0002576
Saponification value	mg KOH/g	_	_	ASTM D558-95	370 max	_	_	182.325mg NaOH/g

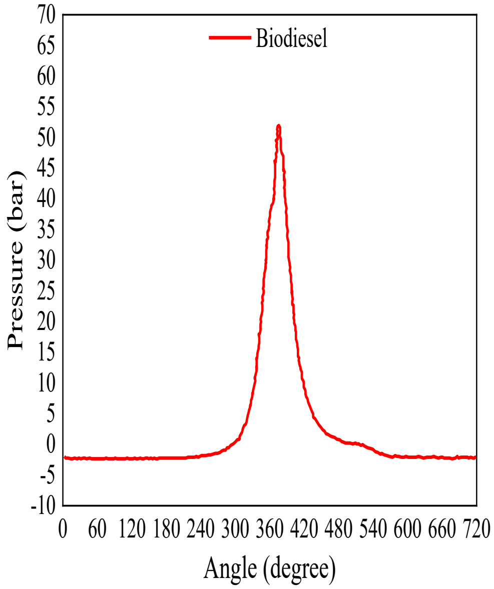
4.8 Engine Test of Biodiesel in IC Engine

4.8.1 Engine Test of Biodiesel in IC Engine

The properties of any fuel play a significant role in engine performance. The biodiesel is derived from vegetable oils via a popular process, transesterification in the presence of a catalyst and alcohol as a reactant due to the availability and cost factor methyl alcohol is commonly used and the derived biodiesel is also known as the fatty acid methyl ester. The purpose of the transesterification process is to lesser the viscosity of the oil. Combustion of Biofuel B0 in IC engine at various engine speed for blending ratio of 0% has been conducted with the results presented as follows:



a) Variation of cylinder pressure with a crank angle with n = 3081 1/min, pi = 3.2 bar

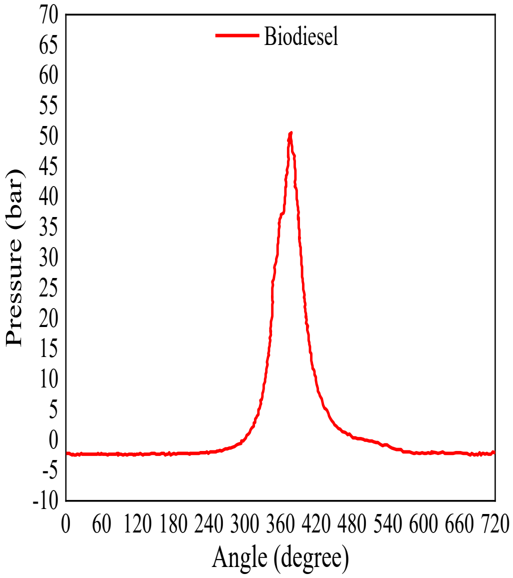


b) Variation of cylinder pressure with a crank angle with n = 1775 1/min, pi = 6.1 bar

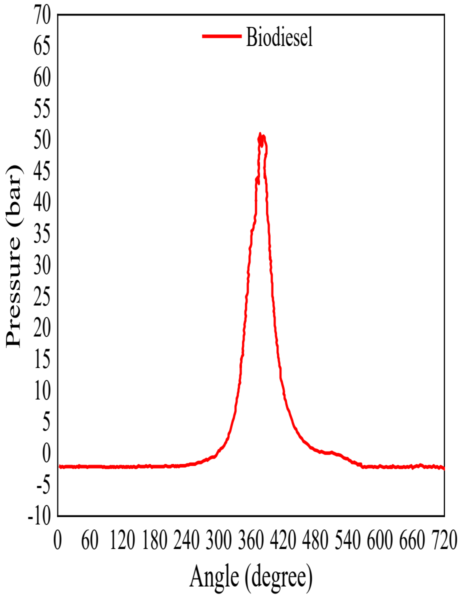
Figure 29: pressure vs crank angle at 0 % blend or pure diesel

High peak pressure and maximum rate of pressure rise correspond to a large amount of fuel burned in the premixed combustion stage.

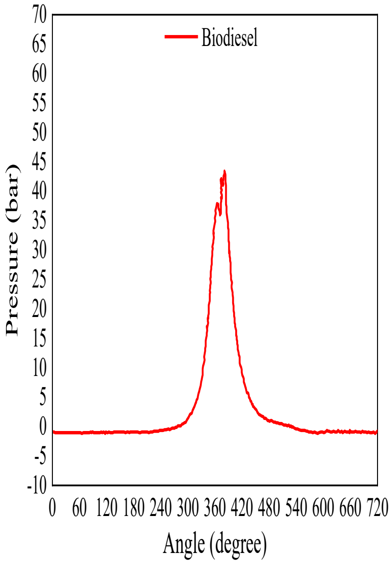
4.8.2 Engine Test of Biodiesel B5 blend in IC Engine



a) Variation of cylinder pressure with crank angle with n = 1769 1/min, pi = 5.3 bar



b) Variation of cylinder pressure with crank angle with n = 1496 1/min, pi = 5.5 bar



c) Variation of cylinder pressure with crank angle with n = 1512 1/min, pi = 4.5 bar

Figure 30: engine performance graph

Cylinder pressure is very important parameters for evaluating the engine combustion process and engine efficiency and specific fuel consumption increased with mixed biodiesel ratio. Therefore biodiesel can use as an alternative fuel for diesel engine .The diesel-biodiesel fuel can improve combustion and emission characteristics of diesel engine.

4.9 Emission Characteristics of Biodiesel in IC Engine

Emission in IC engines consists of CO_2, Hydrocarbon (HC), carbon monoxide (CO), and O_2. The emission level varies according to engine parameters, and fuel quality operating conditions of CO_2, HC, and O_2 compared to diesel fuel. The comparative result of each investigation related to the emission characteristics of biodiesel diesel fuel blends with diesel fuel is as follows:

Table 25: Performance and Emission Results for Various Test Conditions

Fuel	Emission compositions			
	CO	CO_2	HC	O_2
diesel fuel	0.236	3.76	319 rpm	16.05
diesel- biodiesel blends(B_5)	0.114	1.80	204rpm	18.75

Biodiesel emits less $C0_2$, C0, HCs than diesel fuel and generates more oxygen. Increased free oxygen results in full combustion and lower emissions. Low temperature effect on the increase in CO emissions. The increasing CO emissions are mainly due to the decrease of the temperature. The lower CO emissions can be obtained by higher oxygen content, leading to the increase in the combustion temperature. In the case of the B5 blend and pure biodiesel, lower CO emissions were observed due to the oxygen content in biodiesel (0.114 values). For pure diesel fuel powering, the CO emissions were nearly0.236 but for biodiesel fuel, this value declined to 0.114.

For the engine powered by the B5 blend, the CO2 emissions were lower compared to the pure diesel. This is a big advantage in terms of global trends to reduce the CO2 emissions. When a HC fuel is burnt, the most common product is CO2 and water. The use of the oxygenated fuels results in slightly lower CO2 emissions as the biodiesel contains less carbon in its molecules, whereas the products of biodiesel combustion contain less CO2 and more H2O. In the case of the B5 blend with 5% of the biodiesel fuel fraction, 1.80 values of CO2 emissions was recorded, but in the case of the pure diesel 3.76 values of CO2 emissions was recorded .

a) Emission analysis of pure diesel b) Emission analysis of biodiesel- diesel B_5blend

Figure 31: Emissions analysis

CHAPTER FIVE

5. CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Even though biodiesel is a good alternative over petroleum diesel in various aspects, it is always jeopardized by the high cost of feedstock and absence of economically and technically viable technology for its efficient production from any feedstock type.

In this research, biodiesel was produced using methanol alcohol with chicken eggshell calcium oxide catalyst at a constant reaction time of 3 hours and agitation speed of 400 rpm. The result shows that biodiesel production using eggshell calcium oxide was considerable potential, especially in terms of simplification of the separation process, waste reduction, and producing an environment-friendly product. Thus all decrease the production cost of biodiesel. At catalyst loading of 2% optimum methyl ester yield of 97.65 % was obtained.

Therefore, it can be concluded that eggshell CaO is an effective catalyst for the production of biodiesel from brassica carinata A.Br.seed oil through transesterification. As obtained from the experiment all the physicochemical properties of biodiesel meet the ASTM D6571 standards. During the experimental work, the effect of reaction temperature, catalyst loading, and methanol to alcohol oil ratio on the yield of biodiesel has been investigated. The analysis obtained from design expert 13.0 (Box-Behnken) for the three-factor with three levels was show that the increase in catalyst loading, reaction temperature, and methanol to oil ratio up to the optimal point increased the yield of biodiesel.

In contrast, an insufficient amount of factors leads to incomplete conversion, and further increase of these factors reduced the yield of biodiesel this could be due to catalyst leaching, alcohol loss, and formation of emulsion which made it difficulty in the separation of biodiesel from glycerol. The use of diesel-biodiesel blends leads to a decrease in CO, CO_2, HC, and O_2 compared to diesel fuel. Furthermore, from these three parameters and their interaction effect, all three parameters have been affected the yield of biodiesel even though, the effect of methanol to oil ratio and reaction temperature was higher than catalyst loading.

5.2 Recommendation

The Oil used for transesterification was extracted using hexane but, besides cost, it was taken long extraction. For this reason, extraction using mechanical pressing is recommended. In the present work, biodiesel production was conducted in the batch process system, but it should be scaled up to a continuous process system. Even though this study used low-cost eggshell catalysts once at a time, further research should be done on catalyst recycling that may be more cost-effective for biodiesel production.

The low-cost eggshell catalyst should be used in a large-scale industrial process of biodiesel production to make the process cheap and environmentally kind. A comparative study of biodiesel production using commercial CaO and chicken eggshell CaO should be done to compare the yield. In addition to this, those catalysts should be analyzed using XRD to determine the exact structure and surface area of the catalyst.

However, further work should be done on glycerol purification, emission analysis for various percentages of blends, and utilization of it for methanol production purposes. Designing of methanol pilot plant Integrating with the biodiesel to utilize the byproduct glycerol may reduce the production cost of biodiesel (by reducing the expenses for methanol).

Moreover, Ethiopia has huge potential for biodiesel resources like jatropha, castor bean, palm oil, croton macrostachyus, Moringa stenopetala, neem (margosa), sugarcane, animal fat, waste cooking oil, and Microalgeal. But the awareness creation of the stakeholder, management, interest in using biodiesel, etc. still less.so it is recommended to obtain high yield of biodiesel by using various feedstocks.

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APPENDICES

Append ix A: preparation of gomenzer seed oil

A1. Procedure and preparation

a) Drying seed in oven b) serving the seed

Appendix A2. Picture of samples and some laboratory equipment

a) Soxhlet apparatus b) GC-MS instrument

a) Purified biodiesel b) viscometer

Appendix B: Fatty acid composition of gomenzer seed oil from GC-MS analysis

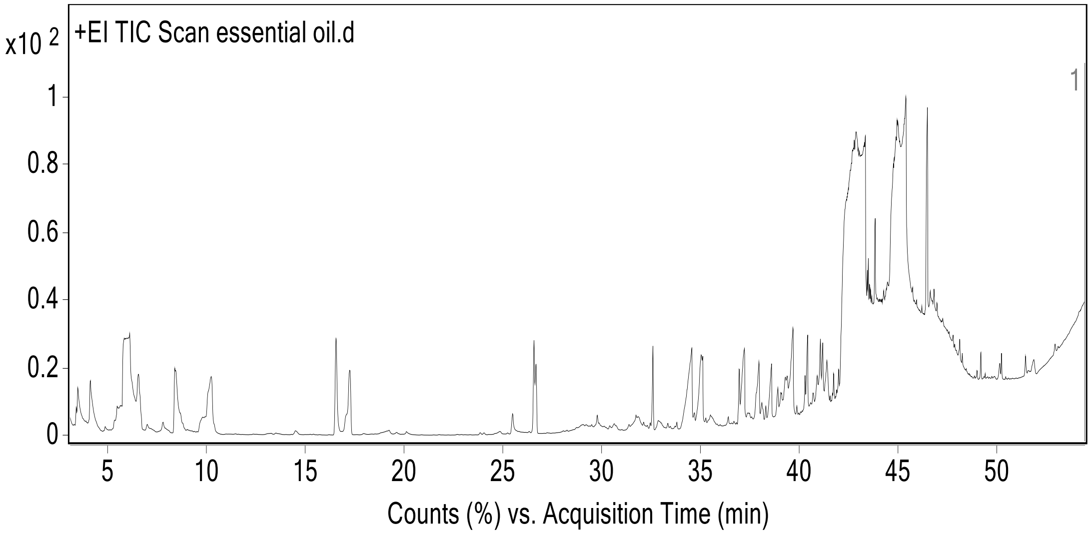
B1: Qualitative analysis report

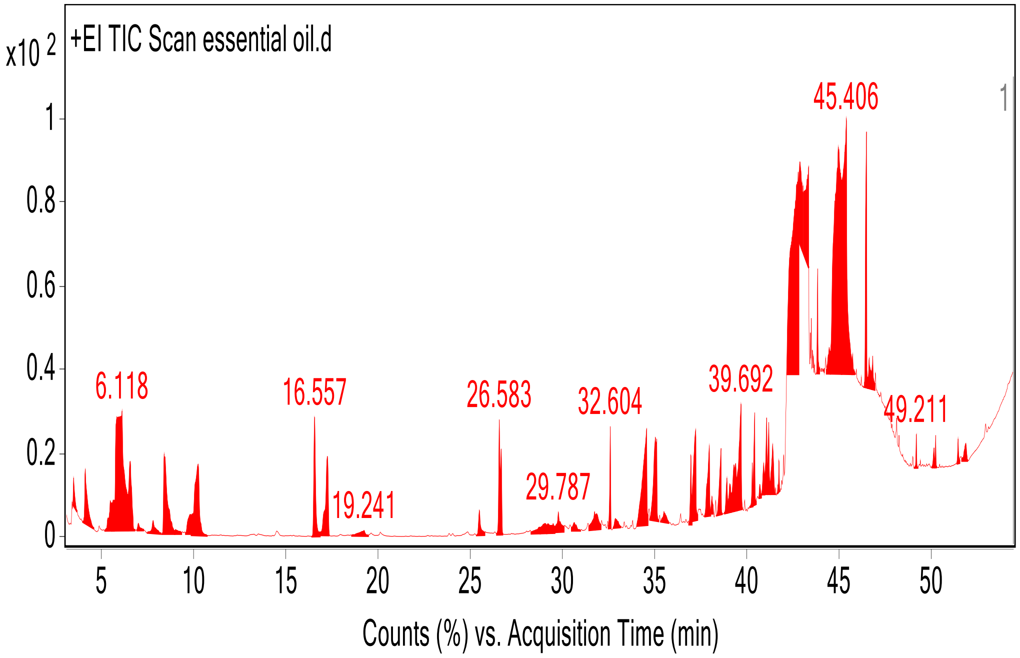
Data Filename	essential oil.D	**Sample Name**	
Sample Type		**Position**	1
Instrument Name	JU_chem	**User Name**	
Acq Method	Essencial oil (T& Y).M	**Acquired Time**	5/23/2022 9:07:49 PM (UTC+03:00)
IRM Calibration Status	Not Applicable	**DA Method**	Default-GCMS-SQ.m-7.m
Comment			

Expected Barcode		**Sample Amount**	
Dual Inj Vol	1	**TuneName**	etune.u
TunePath	D:\MassHunter\GCMS\1\5977\	**TuneDateStamp**	2022-01-29T04:02:23+03:00
MSFirmwareVersion	6.00.34	**OperatorName**	
RunCompletedFlag	True	**Acquisition Time (Local)**	5/23/2022 9:07:49 PM (UTC+03:00)
Acquisition SW Version	MassHunter GC/MS Acquisition 10.1.49 02-Nov-2020 Copyright © 1989-2020 Agilent Technologies, Inc.	**SingleQuadrupole Driver Version**	10.1.0.0
SingleQuadrupole Firmware Version	6.00.34		

Chromatograms

Fragmentor Voltage		**Collision Energy**	0	**Ionization Mode**	EI





Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	5.677	6.118	6.438	2505711.36	77399230.76	62.28
2	16.424	16.557	16.851	2499551.88	18056043.91	14.53
3	26.449	26.583	26.636	2411190.42	11327757.25	9.12
4	32.51	32.604	32.657	2132285.4	7400297.69	5.95
5	39.466	39.692	39.786	2206925.1	21188385.8	17.05
6	42.163	42.79	42.816	4233992.5	111682835.3	89.87
7	43.791	43.858	43.938	2191794.93	6507098.36	5.24
8	44.538	44.966	45.153	4753284.71	124274221.4	100
9	45.153	45.406	45.713	5362926.13	91420983.6	73.56
10	46.369	46.501	46.568	5366662.53	24931189.62	20.06

B2: main fatty acid composition of gomenzer seed oil

Common Name	Systematic name	Area	structure	Wt%	M.weight in g/mol	RT
Erucic acid	Hexanedioic acid, dioctyl ester	3382385	C22 H42 O4	0.360018669	370	46.835
Palmitic acid	n-Hexadecanoic acid	160785	C16 H32 O2	0.017113842	256	43.057
Oleic acid	9-Octadecenoic acid (Z)-	3687700	C18 H34 O2	0.392516182	282	40.921
Stearic acid	Octadecanoic acid	208253	C18 H36 O2	0.022166302	284	48.263
Linoleic acid	Octadecadienoic acid	12585617	C18 H32 O2	1.33960418	284	37.97
Linolenic acid	octadecatrienoic acid	980227	C18 H30 O2	0.104334669	284	47.275
Myristic acid	Tetradecanoic acid	21188386	C14H28 O2	2.255276834	228	39.692

Appendix C: Experimental results obtained based on the experimental design

		Factor 1	Factor 2	Factor 3	Response 1
Std	Run	A:tempeerature	B:molar to gomenzer oil ratio	C:catalyst to gomenzer oil	yield
		℃	%	%	
16	1	75	9	2	94
10	2	75	12	1	94
1	3	70	6	2	95
12	4	75	12	3	95
17	5	75	9	2	96
13	6	75	9	2	97
6	7	80	9	1	94
14	8	75	9	2	98
9	9	75	6	1	97
8	10	80	9	3	98
11	11	75	6	3	96
2	12	80	6	2	90
5	13	70	9	1	90
15	14	75	9	2	96
3	15	70	12	2	90
7	16	70	9	3	90
4	17	80	12	2	98

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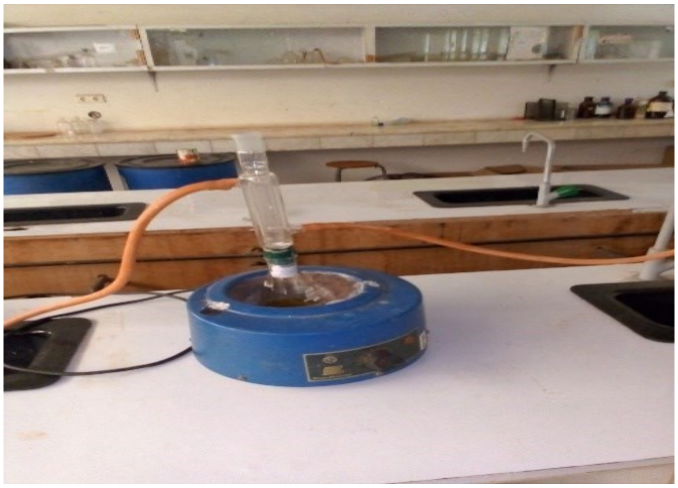
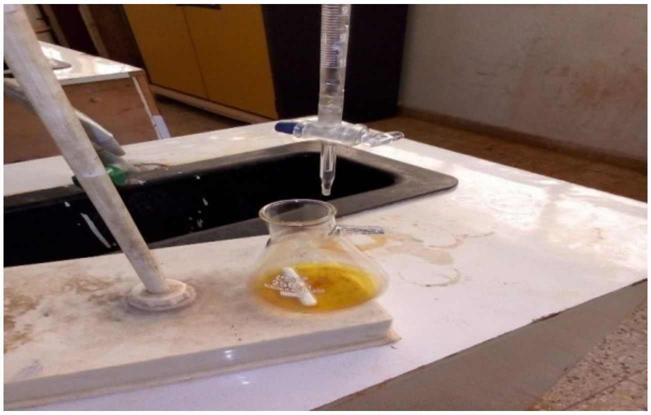
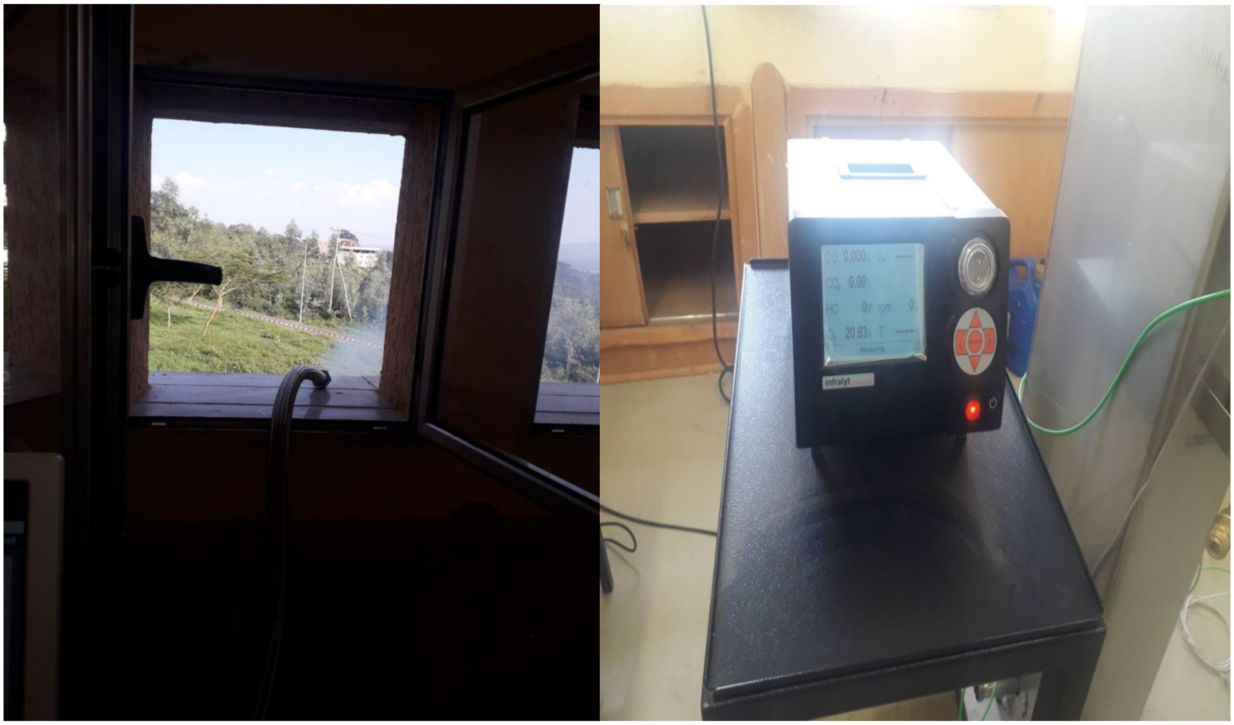
Appendix D: during characterization of gomenzer seed oil and biodiesel

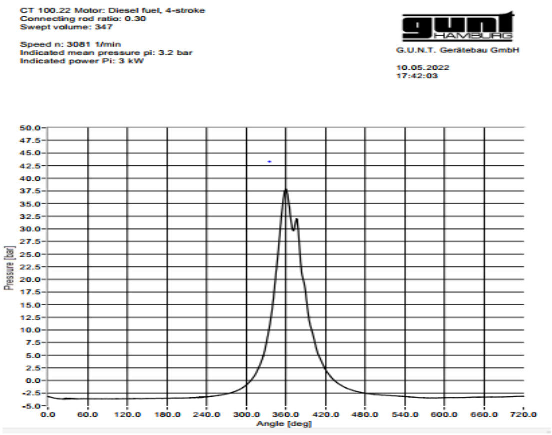




Appendix E: during engine test and emission analysis



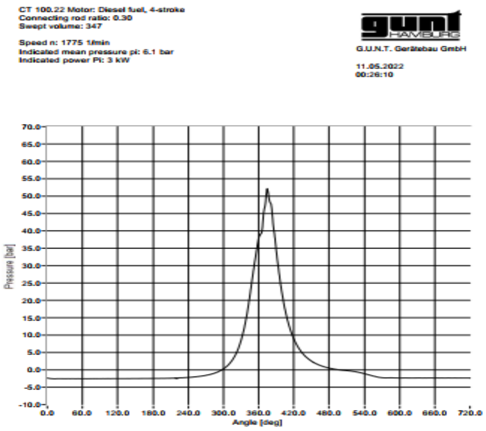
Appendix F:engine test result graph


CT 100.22 Motor: Diesel fuel, 4-stroke
Connecting rod ratio: 0.30
Swept volume: 347

Speed n: 3081 1/min
Indicated mean pressure pi: 3.2 bar
Indicated power Pi: 3 kW

G.U.N.T. Gerätebau GmbH

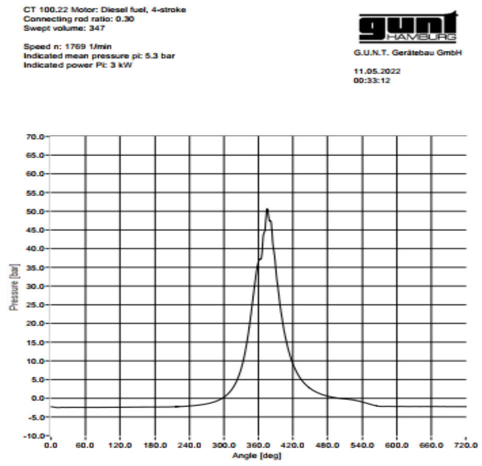
10.05.2022
17:42:03


CT 100.22 Motor: Diesel fuel, 4-stroke
Connecting rod ratio: 0.30
Swept volume: 347

Speed n: 1775 1/min
Indicated mean pressure pi: 6.1 bar
Indicated power Pi: 3 kW

G.U.N.T. Gerätebau GmbH

11.05.2022
00:26:10

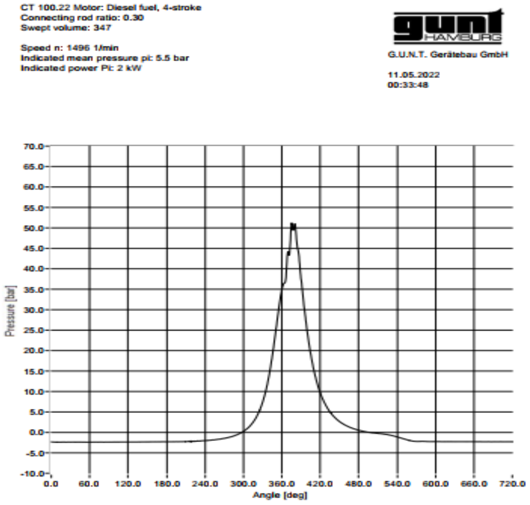


CT 100.22 Motor: Diesel fuel, 4-stroke
Connecting rod ratio: 0.30
Swept volume: 347

Speed n: 1769 1/min
Indicated mean pressure pi: 5.3 bar
Indicated power Pi: 3 kW

G.U.N.T. Gerätebau GmbH

11.05.2022
00:33:12



CT 100.22 Motor: Diesel fuel, 4-stroke
Connecting rod ratio: 0.30
Swept volume: 347

Speed n: 1496 1/min
Indicated mean pressure pi: 5.5 bar
Indicated power Pi: 2 kW

G.U.N.T. Gerätebau GmbH

11.05.2022
00:33:48