Addis Ababa University

Addis Ababa Institute of Technology

School of Chemical and Bio Engineering

Production, Characterization and optimization of Food-grade Lubricant from the Rapeseed

BY

Yemariamnesh Mamuye

Advisor

Dr.Solomon Kiros

A thesis submitted to the School of Chemical and Bio Engineering as part of the partial fulfillment of the requirement for the degree of Master of Science (chemical and Bio engineering in the process engineering stream)

ADDIS ABABA UNIVERSITY
ADDIS ABABA, ETHIOPIA
September 2023

Addis Ababa University

Addis Ababa Institute of Technology

School of Chemical and Bio Engineering

A thesis submitted to the School of Chemical and Bio Engineering as part of the partial fulfillment of the requirement for the degree of Master of Science (chemical and Bio engineering in the process engineering stream)

Signed by the Examining Committee:

Dr.Solomon kiros		
Advisor	Signature	Date
Dr.Nurelegne T.		
Internal Examiner	Signature	Date
Dr.Shimelis S.		
External Examiner	Signature	Date
School Chairperson	Signature	Date

Acknowledgments

First of all, I would like to thank Almighty GOD for giving me health,strength and wisdom to accomplish this thesis through His protection and strength, and always helps me in all situations and challenges .

I would like to express a special thanks to my advisor Dr.Solomon Kiros for his continuous and appreciable guidance, giving advice, sharing knowledge, skin, experience and suggestions at every stage of the thesis. I would also like to express my sincere gratitude to the School of Graduate Studies, Addis Ababa University, which is acknowledged for providing financial support to carry out this work.

I especially thank to Ato HintsaSilassie Seifu, who helped me succeed in my laboratory work and give me useful ideas, advice and support whatever I needed, I would like to be my deepest gratitude to my mother and Dr. Adane Desta, for whose guidance, encouragement, wisdom and motivation to my achievement. Finally, I would like to thank the Addis Ababa chemistry laboratory staff member who helps in research.

Abstract

In food processing manufacturing line, Cross-contamination is a major concern for any equipment used in the handling, packaging or processing of ingredients used in the food or beverage industry. In this research work, the production, purification and characterization of food grade lubricants from rapeseed are performed by double transesterification conversion methods. Experiments on the production and purification of food grade lubricants were performed according to standard scientific procedures. The rapeseed oil was extracted in the Soxhlet apparatus using normal hexane, and the relevant physicochemical properties (viscosity, density,acid value, free fatty acid value, PH and saponification number)were measured and analyzed in accordance with the ASTM and EN standard of oil quality. In addition, Rapeseed biodiesel was produced in the first transesterification conversion process using the solvent ethanol and NaOH used as catalyst and the same quality parameters as oil was done rapeseed biodiesel. Finally, food grade lubricants were produced from rapeseed biodiesel, ethylene glycol solvent and KOH were used as catalyst. Molar ratio, temperature and catalyst concentration were among the main variables of the experimental process studied. The box-Behnken design was applied in the manufacturing process in the design software to achieve the lubricating product interaction and individuality. The maximum product 94% was obtained at a temperature of 165ºC, the molar ratio was 3:1 and a 1% catalyst concentration by weight was consumed. The properties(density,viscosity,acid value, pour point, cloud point and flash point) of the manufactured food grade lubricants have been tested and conformed to ASTM quality specifications of food grade lubricant.

Keywords: First Trasesterfication, Second Transesterification, Rapeseed oil,Rapeseed Biodiesel, Food grade lubricant.

Table content

List of figures

List of table

List of Abbreviations

ASTM	American Standard for Testing Materials
AV	Acid Value
EN	European Standard
FAEE	Fatty Acid Ethyl Ester
FAME	Fatty Acid Methyl Ester
FFA	Free Fatty Acid
FGL	Food grade lubricant
FTIR	Fourier Transform infrared
IR	Infrared spectroscopy
KBr	Potassium bromide
RB	Rapeseed bio lubricant
RO	Rapeseed oil
RSM	Random Surface Method
USDA	United state department of Agriculture

1. Introduction

1.1. Background of the Study

Emphasizing health and safety stands as a paramount concern within the domains of food, beverage, and pharmaceutical manufacturing [Martin, 2023], rigorous controls are instituted in the food processing sector to mitigate the risks of contamination, encompassing both biological agents like pathogens and spoilage organisms, as well as chemical agents such as toxins, carcinogens, or mutagens. To effectively manage these hazards, control measures must extend across various aspects, including the production process line [Debbie, 2021]. Among the cross contamination causes are the leakage of lubricants. Lubricants assume a crucial role within all machinery and engines, serving to diminish friction and, consequently, reduce energy consumption. Depending on the design of equipment and its operational parameters, a well-formulated lubricant can significantly contribute to prolonging the lifespan of machinery and promoting energy efficiency. Presently, the global production of lubricants totals approximately 41 million tons (MMT), with the market exhibiting modest growth at a rate of 2% annually (Don et al., 2016). Ethiopia is one of the country imported lubricants to use in all industries including food and beverage industries.

Machine lubrication processes and procedures are an important area of focus for food safety and hygiene inspectors because leaks can pose an immediate risk of product contamination. Since it is the responsibility of the food manufacturer to control any risks in the production facility, identifying potential hazards and analyzing the corresponding risks are important in achieving reductions measurable risk [ELGI 2016]. The growing concern about food-grade lubricants raises the question of whether food safety regulations and standards apply to food-grade lubricants and how food manufacturers meet operational requirements without introducing new chemicals that pose a risk [Debbie, 2021]. Food grade lubricants must perform the same technical functions as any other lubricant: provides protection against wear, friction, corrosion and oxidation, heat dissipation and energy transfer, are compatible with rubber and other sealing materials, and offers sealing performance in some case [Martin Williamson, 2023]. To address this important issue, the United States Department of Agriculture (USDA) designated the original food-grade categories of H1, H2, and H3.

Most importantly, about 95% of total lubricant production across the world is petroleum-based, generally denoted as mineral oil. These oils are composed of a complex mixture of paraffinic (linear/branch), olefinic, naphthenic, and aromatic hydrocarbons of 20 to 50 carbon atoms. These formulations are non-renewable and toxic [Willing, 2001]. However, petroleum-based lubricants exhibit satisfactory performance but they are not environmental benign due to their eco-toxicity and non-biodegradability [Donnet and Erdermir, 2004]. To this side, such lubricants are harmful to humans and the environment as a consequence of their low biodegradability and high toxicity, some are even considered to be carcinogenic. Lubricant discharge was also reported, causing strong contamination in air, soil, and drinking water. Considering the annual volume of lubricants and their environmental effects, several countries have introduced strict regulations to mitigate the effects of the disposal of these lubricants [Nagendramma and Kaul, 2012].

In order to satisfy the environmental regulations, the scientific community is developing new lubricants with greater biodegradability and less toxicity. In this sense, the lubricants obtained from bio-based sources known as bio-lubricants have emerged as potential alternatives to replace traditional mineral oils extracted from petroleum. Bio-lubricant interpreted as a lubricant obtained from natural raw materials both vegetable and animal oils, renewable and non-toxic to humans and other living things, as well as environmentally friendly. Vegetable oil used for the production bio-lubricant can be obtained from plant seeds, such as vegetable oil that can be consumed or which cannot be consume. Some of the vegetable oil can be used as bio-lubricant are castor, Karanja, neem, rice bran, rapeseed, linseed, mahua, palm oil, sunflower oil, coconut, soybean, olive and canola [Salimon *et al*,2010]. They can also be made from synthetic esters and petroleum oils that satisfy established biodegradability and toxicity criteria. In addition, bio lubricants exhibit better physico-chemical properties such as high flash point, high boiling point, high lubricity, high biodegradable, high viscosity index, low volatility and less toxic, compared with petroleum-based lubricant [Meier et al,2007].

Rapeseed oil (RO), also known oilseed rape, which belongs to the family of Brassicaceae (mustard or cabbage family) is a bright-yellow flowering, cultivated mainly for its oil-rich seed, which naturally contains huge amounts of erucic acid [Al-shehbaz *et al*, 2006]. These rapeseeds have content over 40%, in which oleic acid, linoleic acid are dominant fatty acids. It is one of the most frequently consumed vegetable oils, and it is one of the most valuable edible fats, mainly due to the high content (approx. 90%) of 18-carbon unsaturated acids. Rapeseed is the thirdlargest source of vegetable oil and the second-largest source of protein meal in the world [WU Y. *et al*, 2019].

Rapeseed, can currently be used for energy purposes in Mediterranean areas and can also be considered suitable for difficult and polluted areas, as it is a good candidate because its decontamination properties.It is also found that crops have better agro-ecological adaptation and productivity. This is better because producers can grow crops without facing production costs and can indirectly develop an environmentally friendly business. Ethiopian mustard is currently a promising new energy crop for most countries with Mediterranean, arid and semi-arid climates, because high levels of production require fewer inputs and manufacturers are also used to produce and distribute goods for other uses as well as create employment opportunities.

In addition, canola oil, produced from rapeseed varieties high in erucic acid, is often used in vegetable oil blends (sunflower, soybean, corn, etc.) to improve the composition. Fatty acid fraction of vegetable oils. Besides the above benefits, canola oil has a unique composition rich in many bioactive compounds and a proven thermal stability compared to other vegetable oils, suggesting another advantage: the potential its immense application is for the production of food-grade lubricants.

1.2. Statement of the Problem

In order to prevent toxic lubricant from coming in to contact with food, finding an alternative lubricant source for producing valuable products are needed more than ever. The issues of cross contamination are the major concern for any equipment used in handling or processing of material used mainly in the food or beverage-based industries, to use nonfood lubricant in machinery, have a potential to cause long term chronic health effects, including cancer and reproductive problems. which has stimulated to look for alternative food grade lubricant sources that are keeping equipment running and food product safe. A more convenient way is to use food grade lubricants, to reduce the risk of incidental contact with product. On the other hand, Ethiopia, endowed with varied agro-ecological zone and diversified natural resources, have been known as homeland and domestication of several crop plant. Rapeseed has been cultivated in large quantities in Ethiopia for several years without proper or planed way of cultivation. In contrary, the country spends about birr 10 billion annually to import lubricant products for domestic consumption [Bereket and Tilahun, 2017]. A convenient way to lower the cost of such lubricants is to use the locally available cheaper feedstock (raw materials like, rapeseed) as a potential source of food grade lubricant, rather than extensively importing from abroad. This can be used to improve the cost of food grade lubricant which will lower the price as well as safety of food.The selection of such source (i.e. rapeseed oil) is based on a number of factors, including accessibility, quality, cost and availability. The primary reason for choosing rapeseed oil for this study is its suitability as edible oil, which doesn't pose a threat to food security, and its ready availability in its raw or unrefined form. Consequently, the primary objective of this research will be to investigate the creation of potential food-grade lubricants through production techniques and the development of affordable and food-safe lubricants derived from rapeseed. Through systematic experimentation, we seek to unlock the potential of rapeseed oil as a source for food-safe lubricants.

1.3. Objectives of the Study

1.3.1 General Objective

The general objective of this study was production, optimization and characterization of food grade lubricants from rapeseed.

1.3.2 Specific Objectives

- ✓ To Extract rapeseed oil from rapeseed via solvent extraction method
- ✓ Synthesis and characterization of ethyl ester extracted from rapeseed oil
- ✓ Production and Physicochemical characterization of extracted food grade lubricants
- ✓ Investigate the optimum parameter for the production of food grade lubricant
- ✓ Compare the produced food grade lubricant against standard lubricant.

1.4 Research question

- How to extract rapeseed oil from rapeseed?
- How to synthesis and characterize the produced ethyl ester from rapeseed oil?
- What are the physiochemical properties to characterize the food grade lubricant?
- What are the optimum parameters for the produced food grade lubricant?
- Why to compare the produced food grade lubricant against standard lubricant?

1.5. Significance of the Study

This study is going to be more of an experimental study of chemical extraction method, optimization and characterization of food grade lubricants from rapeseed sources, in which our country is importing from different countries of the world in large amount. In one hand, for the country like Ethiopia in which 83% of its people follow agriculture-based way of life, such type of project is going to enhance the society economic status, as farmers who are going to prepare a farm of this plant will be raw material suppliers for the industries that are engaged in the production of rapeseed-based lubricants. In the second hand, food-grade lubricants are designed to enhance and protect the key assets against ailments such as rust, corrosion and friction, and it is vitally important that the risk of food contamination can be minimized, while such affordable and food grade lubricants are utilized in the country. It also improves the economic sector of the country as the country is becoming a major dependent on foreign currency in the fuel and/or lubricant imports, which will in turn decrease cost of importing. Therefore, as explained above this work will possess a significance area on the society, environment and country. Furthermore, the study will also serve as a reference for future researchers on the subject of extraction food grade lubricants using Ethiopian rapeseed resources.

2. Literature Review

2.1. Overview of food grade lubricant production process

The food industry has been a driving force behind the advancement of lubricants designed for use in food-grade applications. These specialized lubricants are carefully crafted to mitigate the potential risks linked to the presence of unavoidable contaminants in food and beverages, in accordance with clear-cut regulations governing food-grade lubricants in various nations. The introduction of a fresh global standard for maintaining food hygiene will encourage the broader adoption and utilization of such products on a global scale. This standard emphasizes the identification and comparison of lubricants that are not only food-grade but also eco-friendly and biodegradable [Russell's 2007].

A food-grade lubricant, whether in the form of industrial grease, oil, silicone, or other lubricating substances, is defined by its non-hazardous nature when it accidentally comes into contact with products intended for consumption by animals or humans. To qualify as safe, it must adhere to predetermined concentration limits and adhere to the same standards as regular lubricants, providing protection against typical wear and tear, excessive friction, corrosion, and oxidation. These lubricants also excel in dissipating heat for energy transfer, ensuring compatibility with materials like rubber, and creating effective seals when necessary [E. Tulsa in 2021]. The food industry, with its stringent food safety regulations in both developed and developing nations, holds a significant market share and is expected to maintain its dominance. The Food Grade Lubricant Market is under scrutiny in various regions, including North America, Europe, Asia Pacific, Latin America, Middle East and Africa. The report primarily focuses on assessing the growth potential, limitations, and opportunities within the Global Food Grade Lubricants Market. As of 2020, the global food grade lubricants market was valued at USD 230.4 million and is expected to reach USD 406.6 million by 2030, registering a Compound Annual Growth Rate (CAGR) of 5.9% from 2021 to 2030. Mineral oil dominated the market share in 2020 and is anticipated to continue its leadership throughout the forecast period [Allied Market Research 2023].

2.2. Raw Materials for the Production of Food Grade Lubricants

Food-grade bio-lubricants, often referred to as bio-lubricants, can be produced using various vegetable oils. While there are over 350 different types of oilseeds, palm, soybean, sunflower, coconut, safflower, rapeseed, cottonseed, and peanut oils are recognized as promising candidates for alternative bio-lubricants. These vegetable oils fall into two categories: edible oils and non-edible oils.Table 2.1, gives information on the oil content of specific edible nuts and seeds that have the potential to serve as bio-lubricants [Nadia Salih and Jumat Salimon 2022].

Table 2.1 Oil contents of some Edible and Non-Edible seeds (H.M. Mobarak et al 2014)

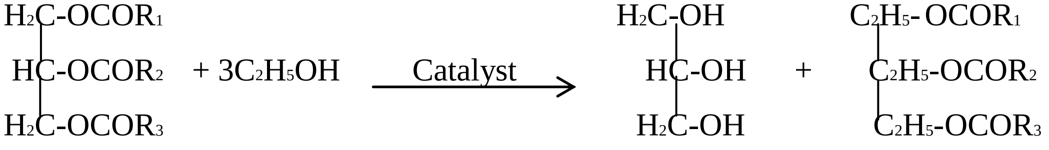
Edible Species	Oil content (% by volume)	Non Edible species	Oil content (% by volume)
Rapeseed	38-46	Castor	45-60
Palm	30-60	Jatropha	40-60
Peanut	45-55	Neem	30-50
Olive	45-70	Karanja	30-50
Corn	45-50	Mahua	35-50
Coconut	63-65	Linseed	35-45

While cooking oils are indeed suitable contenders for bio-lubricants, they continue to be extensively utilized for culinary purposes, particularly in developing nations. Conversely, non-edible oils are presently employed in the chemical industry for manufacturing items like soaps, detergents, cosmetics, and more, with their current usage being relatively limited. This suggests significant untapped potential for non-vegetable oils that could find valuable applications in lubrication. Consequently, these non-edible oils hold promise as potential candidates for future lubricants [R. Uppar *et al*. in 2022]. High viscosity index vegetable oil-based lubricants excel in maintaining their lubricating film integrity even under high-temperature conditions. The flash point, which determine the lubricant's volatility and flame-retardant characteristics, play vital roles in its performance. Generally, bio-lubricants are recognized for their high biodegradability and reduced toxicity to both humans and the environment [Jitendra Kumar Chandrakar and Amit Suhane in 2014].

2.3 Food grade Lubricant Manufacturing Chemistry

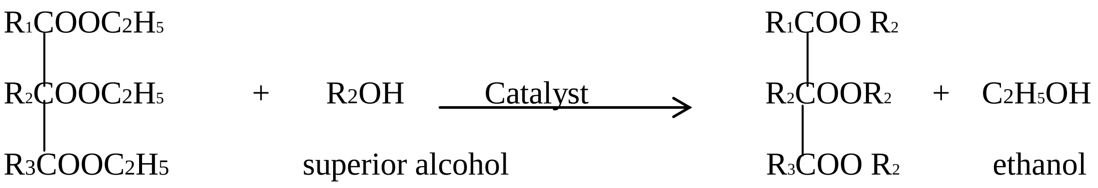
Food-grade bio-based lubricants typically involve modifying the ester components within triacylglycerides through a process called ester conversion. This conversion often entails cross-esterification, where the glycerol components of triacylglycerides are substituted with longer chains and/or branched alcohols. Triacylglycerides are naturally present in vegetable oils and animal fats, commonly referred to as lipids. This transformation takes place in the presence of a suitable catalyst, which can be either an acid or a base. During this process, the fatty ester is released, and concurrently, the OH group in glycerol is regenerated. The synthesis of food-grade lubricants through transesterification (TER) can be catalyzed either chemically or enzymatically. Equations (2.1 and 2.2) serve as a representation of a two-step esterification process catalyzed by a base for the creation of food-grade lubricants. This overall reaction unfolds in two stages, with the first step involving the transesterification conversion between triglycerides and ethanol, yielding FAEE, As an interim procedure, fatty acid ethyl esters (FAEE) are generated from vegetable oils. In this process, the triglycerides present in vegetable oils like canola oil undergo a reaction with three moles of ethanol, facilitated by a catalyst (either potassium hydroxide or sodium hydroxide), yielding three moles of FAEE and glycerol. This reaction results in the formation of two distinct phases: a denser glycerol phase and a lighter ethyl ester phase [Charles L. Peterson *et al.*, 1996]. It's important to note that this reaction is reversible. as depicted in Equation 2.1 below.

The second transesterification is the production of food-grade bio-lubricants,using various alcohols, including 2-ethyl-1-hexanol, 1-heptanol, ethylene glycol, and others are employed alongside FAEE (Fatty Acid Ethyl Ester) obtained in the earlier stages of the process. These components, together with the catalyst used in the second ester conversion, participate in the reaction. the reaction between FAEE and superior alcohol to produce Food-grade Lubricant as depicted below.



$$\begin{array}{l} H_2C\text{-}OCOR_1 \\ HC\text{-}OCOR_2 \\ H_2C\text{-}OCOR_3 \end{array} + 3C_2H_5OH \xrightarrow{\text{Catalyst}} \begin{array}{l} H_2C\text{-}OH \\ HC\text{-}OH \\ H_2C\text{-}OH \end{array} + \begin{array}{l} C_2H_5\text{-}OCOR_1 \\ C_2H_5\text{-}OCOR_2 \\ C_2H_5\text{-}OCOR_3 \end{array} \qquad (2.1)$$

Triglyceride + ethanol Glycerol + ethyl Ester/Biodiesel



$$\begin{array}{l} R_1COOC_2H_5 \\ R_2COOC_2H_5 \\ R_3COOC_2H_5 \end{array} + \underset{\text{superior alcohol}}{R_2OH} \xrightarrow{\text{Catalyst}} \begin{array}{l} R_1COO\,R_2 \\ R_2COOR_2 \\ R_3COO\,R_2 \end{array} + \underset{\text{ethanol}}{C_2H_5OH} \qquad (2.2)$$

Ethyl Ester/Biodiesel Food-grade Lubricant

Equations 2.2 Double transesterification for food-grade Lubricant production. R_1,R_2. & R_3 = alkyl group of fatty acids. R2 =Alkyl group of the superior alcohol [José Maria *et al* 2021]

2.4 Rapeseed Plant

Rapeseed, scientifically known as *Brassica napus,* belongs to the mustard family and is recognizable by its vibrant yellow flowers. In the Amharic language, it is referred to as 'Gomenzer.' Traditionally, rapeseed has been used in Ethiopia for various purposes, including as a lubricant for clay pans used in baking the traditional Ethiopian cuisine called "Injera." Additionally, it has been employed to address stomach ailments, produce special beverages, and treat various health issues.

In Ethiopia, significant mustard production occurs in regions such as Arsi, Bale, Gonder, Gojam, Wello, Shewa Sidamo, and Wellega. Over the past five years, these regions have yielded approximately 550,000 to 750,000 quintals of mustard on an area ranging from 30,000 to 45,000 hectares, as reported by CSA (Central Statistical Agency) for the years 2011/12 to 2015/16.

Furthermore, the leaves of the Ethiopian mustard plant are notably rich in vitamins C and K, beta-carotene, and antioxidants with potential anti-cancer properties, as indicated by research conducted [Takele Mitiku Abdeta in 2022].

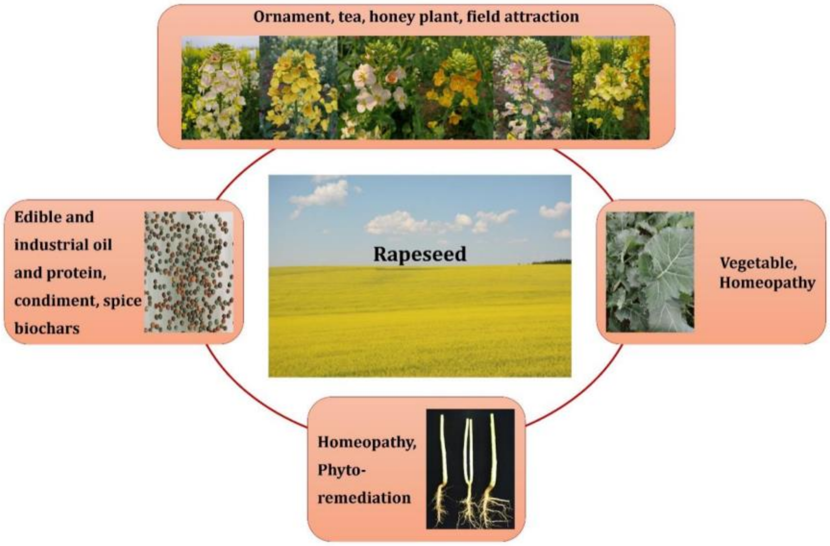


Figure 2.1. Global utilization of rapeseed [Nadia Raboanatahiry *et al*,2021]

Most vegetable oils are obtained from beans or seeds, which generally furnish two valuable commodities oil and a protein-rich meal. Rapeseed oil is one of the vegetable oils comprises of many economically important species widely used as sources of oil and food, and as ornamental plants [Gulden et al,2008]. The youngest species, *Brassicaceae napus*, is commonly used as an oil crop and has several common names-rapeseed, oilseed rape, and colza. it is an annual species, in which the winter, semi–winter, and spring types differ in their cold and drought tolerances; consequently, the growing conditions are also different across the world. Most importantly, it is wildly grown in most parts of Ethiopia.

2.5 Multiple Usages of Rapeseed Oil

Every aspect of the rapeseed plant, including the vegetable, flowers, seeds, leaves, stems, and roots, finds utility in a range of applications such as food, remedies, cosmetics, and industrial uses. Among these, the seeds hold particular importance as sources of both oil and protein. Rapeseed seeds exhibit variations in oil and protein content depending on the cultivar, alongside the presence of other components like glucosinolates, phenols, phytic acid, cellulose, and sugars. Renowned for its high-quality vegetable oil production, rapeseed competes with other crops and stands as the world's second-most produced oil crop, following soybean [Statista, 2008]. The fatty acid profile and other constituents found in rapeseed oil potentially contribute to its positive impact on human health, making it suitable for individuals with various health conditions or for preventive purposes. Rapeseed oil primarily serves as a liquid oil for frying, salad dressings, cooking, baking, and the formulation of shortenings. The choice of vegetable oil for these common applications depends on factors such as flavor, nutritional value, texture, stability, cost, and availability, with a focus on minimizing trans fats.

Additionally, rapeseed oil can be blended with other oils to enhance its physicochemical properties. Research has indicated that an 80:20 blend of rapeseed and olive oil, supplemented with 20% palm oil, represents a superior oil combination compared to blends with a higher proportion of olive oil. This blend exhibits a favorable fatty acid profile with low levels of free fatty acids, high oleic acid content, as well as low peroxide levels and a high iodine value, suggesting suitability for deep frying and prolonged storage [Roiaini, M. *et al.*, 2015]. In a broader context, beyond culinary uses, rapeseed oil and its derivatives find diverse applications in the production of bio-lubricants, greases, paints, varnishes, lacquers, protective coatings, hydraulic fluids, soaps, cosmetics, printing inks, oilcloth, and serve as a raw material in the manufacturing of various chemicals.

2.6. Extraction of oil from oilseed

The procedure of separating triglycerides from oilseeds is known as extraction, which employs various chemical, biochemical, and mechanical methods to maximize oil yield while maintaining consistent product quality. Extracting valuable bioactive compounds, important in industries like food and pharmaceuticals, often necessitates the extensive use of solvents such as water, n-hexane, ethanol, chloroform, methanol, and petroleum ether. Oilseed processing and oil extraction aim to obtain top-quality oils with minimal unwanted components, achieve high extraction yields, and produce valuable meal byproducts. There are multiple methods for extracting oil from oilseeds, with solvent extraction and mechanical extraction using a screw press being two common approaches. In the developed countries today, the term "oil mechanics" isn't widely used due to its association with lower oil recovery rates (Nurhan Dunford, 2016).

2.6.1 Solvent extraction

Solvent extraction is a chemical method employed to extract oils from vegetables, oilseeds, and nuts using solvents. It is considered one of the most efficient techniques for vegetable oil extraction, resulting in minimal oil residue in the meal or byproduct. Commonly utilized solvents in this process include hexane, diethyl ether, petroleum ether, and ethanol [A.K. Yusuf, 2018]. In the industrial processing of edible oils, a solvent extraction step is often included, either following or preceding pressing. Hexane-based processes have been commercially employed for a substantial duration. These processes can yield over 95% oil recovery with a solvent recovery rate of over 95%, equivalent to a mechanical extrusion-based oil yield of 60-70%. However, smaller processors tend to prefer screw presses due to their lower initial capital costs. In modern oilseed processing facilities, solvent hexane extraction is the prevailing method, with plant capacities ranging from 100 to 9,000 tons per day [Nurhan Dunford, 2016].

Soxhlet-based solvent extraction is the primary technique for extracting vegetable oils from oilseed raw materials. While the Soxhlet process is commonly used on a laboratory scale [Abdelaziz *et al.*, 2014], large-scale extraction necessitates commercial solvent extraction methods. The key advantage of the Soxhlet process lies in its solvent recycling during extraction. However, drawbacks of this method include high solvent consumption, time-intensive operation, significant energy usage, and sample dilution due to large solvent volumes. There is growing interest in enzyme-assisted supercritical fluid extraction, water-based extraction, and water extraction processes for producing specialty and gourmet oils [Nurhan Dunford, 2016].

2.6.2 Mechanical press

Mechanical pressing is the predominant method for continuously processing oilseeds without the use of solvents. Pressing constitutes the central stage of the oil extraction process, where the majority of the oil is extracted from the seeds. It is the most cost-effective step since it doesn't necessitate heat input or organic solvents; it merely requires mechanical energy input. The pressing process can be described as a compression stage that facilitates the flow of liquid out of the porous material [R. Savoire *et al.*, 2012]. In essence, it involves applying pressure, typically using a hydraulic press or screw, to expel the oil from the oil-containing material. This method enhances oil efficiency by subjecting the oil-rich material to increased mechanical stress.

Historically, pressing has been used for centuries to extract oils from oil-containing materials, and it was only post-World War II that solvent extraction began replacing this method. Currently, over 98% of global oil production relies on solvent extraction. However, for specialty oils like extra virgin olive oil or extra virgin canola oil, as well as preliminary pressing before solvent extraction in some cases, this traditional technique still finds application in rural settings. The primary objective of the pressing process is to separate the oil phase from the solid grain phase [Matthäus B, 2012]. Mechanical expression results in high-quality oil but with relatively lower efficiency. Typically, it is employed by smaller-capacity factories for specialty product production.

2.7 Properties of Rapeseed Oil

Oil extracted from rapeseed, which is sometimes referred to as canola oil when derived from specific low-erucic acid varieties, possesses several common properties as shown in table 2.2 below [Encinar *et al*., 2010]. It's important to note that while these properties are common in rapeseed oil, there can be variations depending on the specific variety and processing methods used.

Table 2.2 Rapeseed oil properties [Encinar et al.2010]

Property	Rapeseed oil
Density @15.6 °C [g/ml]	0.9186
Kinematic viscosity@ 40°C[mm2/s]	33.07
Flash point[°C]	246
Acid value[mg KOH/g oil]	1.02
Saponification value[mg KOH/ g oil]	170.4
Iodine value	108
Cloud point [°C]	-
Pour point [°C]	-19

2.8 Biodiesel production process

Biodiesel is gaining growing significance as an alternative to traditional diesel fuel, primarily due to the diminishing reserves of petroleum and the environmental impacts associated with emissions from petroleum-powered engines. Biodiesel, derived from renewable sources, is composed of simple fatty acid alkyl esters. To establish itself as a viable fuel option, biodiesel needs to be economically competitive with conventional petroleum-based diesel fuel. One approach to lower the production costs of biodiesel involves utilizing more affordable feedstocks, which can include inedible oils, edible oils, animal fats, used cooking oils, and by-products from refining processes [Berchmans and S. Hirata, 2008].

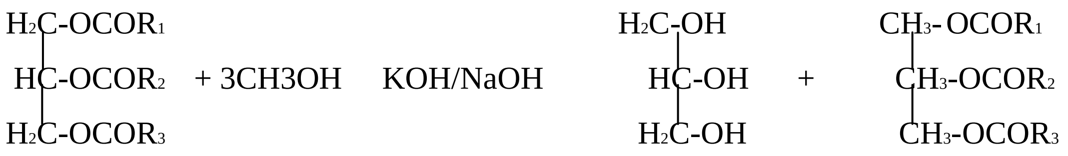
Biodiesel is manufactured through a transesterification reaction involving vegetable oil or animal fat and alcohol [S. D. Romano and PA Sorichetti, 2011]. In chemical terms, transesterification entails the conversion of a triglyceride molecule or complex fatty acid. This process neutralizes free fatty acids, removes glycerol, and results in the formation of an alcohol ester. It is achieved by blending methanol (commonly known as wood alcohol) or ethanol with an alkaline solution, typically sodium hydroxide or potassium hydroxide. Transesterification can take different forms: basic, acid, or enzymatic, with the basic method being the most commonly employed across production scales:

I. **Basic Transesterification:** This method is used most often at all scales of production.The base-catalyzed cross-esterification of vegetable oils proceeds faster than the acid-catalyzed reaction. For this reason and the fact that alkaline catalysts are less corrosive than acidic compounds, using low temperatures (60°C) and low pressures, high conversions (98%) are achieved with minimal side reaction.and low reaction time. Industrial processes often favor basic catalysts, such as alkali metal alkoxides and hydroxides, but their water-free requirement makes them unsuitable for typical industrial processes where avoidance is not possible. completely water.The need for high energy and post-reaction treatment to remove the catalyst from the product is a problem using basic catalysts [P. M. EJIKEME et al 2010].

II. **Acid Transesterification:** Less common in industrial production, it is sometimes used as the initial step with highly acidic raw materials.

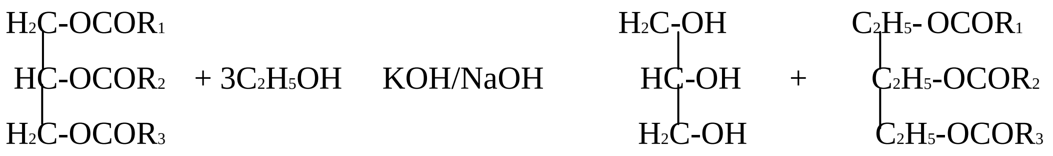
III. **Enzymatic Transesterification:** This method is used less frequently, with lipase as the typical enzyme involved.

Various alcohols have been investigated for biodiesel production, but methanol and ethanol are the most commonly used alcohols due to their low cost and availability [Abarnaebenezer Selvakumari 2019].When methanol is employed as the alcohol in ester conversion, the resulting reaction product is a blend of methyl esters. Similarly, if ethanol is used, the outcome is a mixture of ethyl esters. In both cases, glycerol will be the co-product of the reaction. This is shown schematically in Equations 2.3 and 2.4.



$$\begin{array}{l} H_2C\text{-}OCOR_1 \\ HC\text{-}OCOR_2 \\ H_2C\text{-}OCOR_3 \end{array} + \text{3CH3OH} \quad \text{KOH/NaOH} \quad \begin{array}{l} H_2C\text{-}OH \\ HC\text{-}OH \\ H_2C\text{-}OH \end{array} + \begin{array}{l} CH_3\text{-}OCOR_1 \\ CH_3\text{-}OCOR_2 \\ CH_3\text{-}OCOR_3 \end{array} \qquad (2.3)$$

Triglyceride + Methanol Glycerol + Methyl Ester/Biodiesel



$$\begin{array}{l} H_2C\text{-}OCOR_1 \\ HC\text{-}OCOR_2 \\ H_2C\text{-}OCOR_3 \end{array} + 3C_2H_5OH \quad \text{KOH/NaOH} \quad \begin{array}{l} H_2C\text{-}OH \\ HC\text{-}OH \\ H_2C\text{-}OH \end{array} + \begin{array}{l} C_2H_5\text{-}OCOR_1 \\ C_2H_5\text{-}OCOR_2 \\ C_2H_5\text{-}OCOR_3 \end{array} \qquad (2.4)$$

Triglyceride + ethanol Glycerol + ethyl Ester/Biodiesel

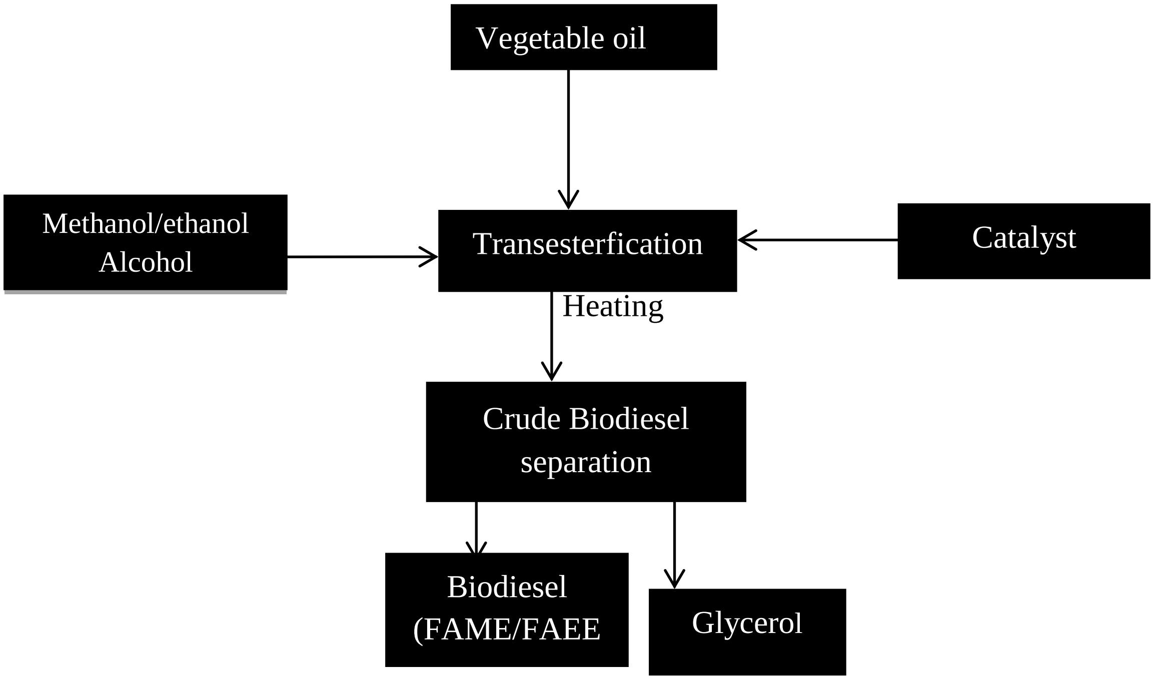


Figure 2.2 Scheme of biodiesel production process from vegetable oil [Md.R.Nahian *et al* 2016]

2.9 Manufacturing Process of Food Grade Lubricating Oil

Bio-lubricants intended for food-grade use are exclusively crafted from biobased materials or natural vegetable oils. This practice contributes to both a safer food supply and a more sustainable future for the industry,[N. Salih and J. Salimon in 2022]. To enhance their thermal and oxidation stability, making them suitable for a broad spectrum of operating conditions while remaining biodegradable, numerous chemical reactions have been developed for the production of lubricants. Among these, the most commonly employed chemical pathways to derive bio-lubricants from vegetable oils are epoxidation and transesterification, as outlined [J.M. Encinar *et al.* in 2020].Epoxidation involves the introduction of oxygen atoms into the molecular structure of the oil, creating epoxy groups that enhance the lubricant's stability and performance. Transesterification, on the other hand, entails the transformation of vegetable oil into esters through chemical reactions with alcohols. Transesterification is the general term used to describe the important type of organic reaction in which one ester is converted to another by exchanging an alkoxy moiety. When the initial ester reacts with alcohol, the conversion of the ester is called alcohol degradation [Ulf Schuchardta *et al.*, 1998].Both of these methods play a pivotal role in the advancement of sustainable and biodegradable lubricants, contributing to a greener and more responsible future for the lubricant industry.

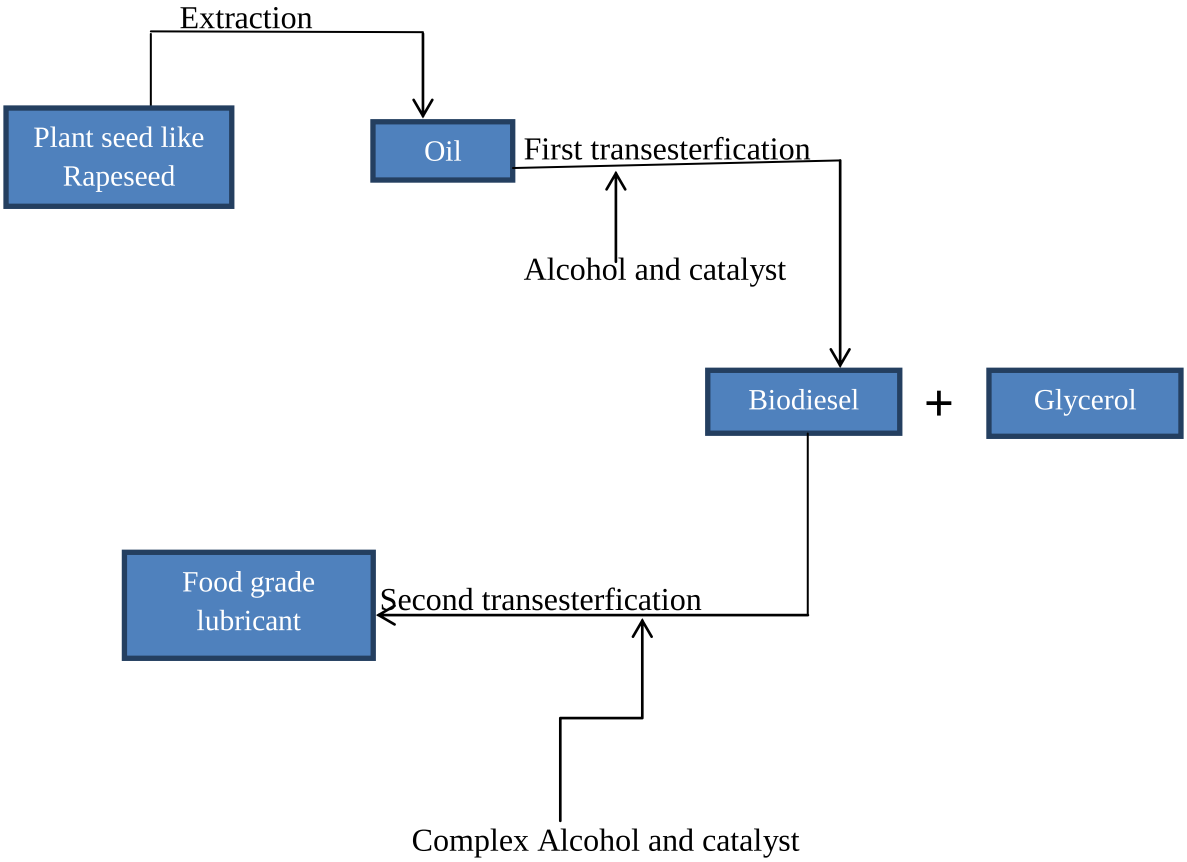


Figure 2.3 double Transesterification of food grade lubricant production pathway.

In general, the production of food-grade lubricants encompasses several key process steps. It begins with the selection of biobased or natural vegetable oils known for their food-grade suitability and biodegradability. These oils may undergo chemical modifications, to enhance their thermal and oxidative stability, and has the following process steps.

2.9.1 Alcohol and Catalyst Mixture

To create food-grade lubricants from complex alcohols, a crucial step involves combining them with a catalyst before introducing biodiesel into the mixture. This mixture is then stirred thoroughly until the catalyst achieves complete dissolution within the alcohol. This carefully orchestrated process ensures the effective transformation of raw materials into high-quality lubricants suitable for food-related applications, with the catalyst playing a pivotal role in facilitating the desired chemical reactions and enhancing the lubricant's performance and stability.

2.9.2 Chemical Reaction

The chemical reaction takes place when the biodiesel is mixed with the complex alcohol–catalyst mix as described in the previous paragraph. This requires certain conditions of time, temperature and stirring. Since alcohols and biodiesel do not mix at room temperature, the chemical reaction is usually carried out at a higher temperature and under continuous stirring, to increase the mass transfer between the phases. The alcohol and catalyst mix are then charged into a closed reaction vessel where the biodiesel is added. The reaction system is totally closed to the atmosphere to prevent the loss of alcohol. The temperature of reaction mixture is kept just near the boiling point of the alcohol to speed up the reaction. Excess alcohol is normally used to ensure total conversion of the biodiesel to its food grade lubricant.

2.9.3. Catalyst Removal

Upon the completion of the reaction, the catalyst is separated from the mixture using a centrifuge. This separation is achieved by adjusting the stirring speed and duration, causing the catalyst to settle like solid particles, which can then be filtered to effectively separate it from the food-grade lubricant. This meticulous process ensures the catalyst's removal, leaving behind a purified and high-quality food-grade lubricant ready for use in various applications, including within the food industry.

2.10. Parameter Affecting Transesterification Reaction

The transesterification process is subject to several operational parameters that impact its outcome. These parameters include the reaction temperature, the molar ratio of alcohol to oil, the type and quantity of catalyst used, the duration of the reaction, the rate of stirring, and the presence of free fatty acids [Admasu E., 2011].

2.10.1. Amount of Catalyst

The alkaline catalyst system is very sensitive to both water and free fatty acids. These circumstances lead to catalyst consumption and lead to in product purification. The addition of too many catalysts will create emulsions that increase viscosity and lead to gel formation. They reduce the apparent performance of the ester. As a result, further increase in catalyst concentration does not increase conversion and leads to additional costs because it must eventually be removed from the reaction medium. If the oil has a high content of free fatty acids and more water, acid-catalyzed cross-esterification is appropriate. Thus, in the double transesterification reaction, sodium methoxide, lipase, potassium hydroxide, and others can be used at concentrations of 0.5 to 1.5% w/w to oil. For refined oils, an amount of about 1% wt catalyst can be required for successful conversion [S. D. Romano and P. A. Sorichetti 2011] .

2.10.2. Alcohol to Oil Molar Ratio

One of the most important variables affecting ester yield is the alcohol/oil molar ratio. Cross-esterification is an equilibrium controlled reaction where an excess of alcohol is required to drive the reaction forward. By further increasing the alcohol/oil molar ratio, the conversion efficiency remains the same. In addition, less alcohol consumption may result in reduced product [Admasu E., 2011].

.

2.10.3. Reaction Time and Temperature

The reaction rate is significantly influenced by the temperature at which the process is conducted. Elevated temperatures expedite the time required to achieve maximum conversion. Transesterification can be carried out within a temperature range spanning from room temperature up to the boiling point of the alcohol used, eliminating the need for pressurized reactors. However, when the reaction temperature surpasses the boiling point of the alcohol, it leads to alcohol evaporation, generating numerous bubbles that can impede the reaction progress. The accomplishment of base-catalyzed cross-esterification is contingent upon the reaction duration, which is typically maintained near the boiling point of the alcohol under atmospheric pressure. Excessive reaction time doesn't enhance conversion but rather encourages a reverse reaction, causing ester hydrolysis and diminishing product yield [G.Anastopoulos et al,2009].

2.10.4. Stirring Rate

As the reaction can only take place at the interface between the liquid and the oil, and because alcohol cannot completely mix with the oil, the conversion of esters is inherently a relatively slow process. Consequently, robust mixing is essential to enhance the contact area between these two immiscible phases. The level of agitation, especially in the context of alcohol degradation, plays a crucial role. Facilitating the mass transfer of triglycerides from the oil phase to the oil-alcohol interface is a significant factor in controlling the rate of the alcohol esterification reaction due to the heterogeneous nature of the reaction mixture. Limited mass transfer between these phases in the initial stages of the reaction results in a sluggish reaction rate, primarily dictated by mass transfer. Rapid mixing serves to expedite the transesterification reaction, meaning that altering the intensity of mixing can directly impact the kinetics of ester conversion (Ester Degradation Reaction)[B.Freedman et al,1984].

2.11. Food grade lubricant Properties and Specification Standards

2.11.1. Viscosity

Viscosity stands as a pivotal characteristic of any oil, representing its capacity to resist flowing. In the context of food-grade bio-lubricants, viscosity assumes paramount importance as it directly signifies the lubricant's resistance to flow. Viscosity is inherently influenced by factors such as temperature, pressure, and the thickness of the lubricating film. High viscosity corresponds to heightened flow resistance, resulting in a thicker lubricating film, while low viscosity implies lower flow resistance and a thinner film. Typically, the evaluation of viscosity follows the kinematic viscosity method outlined in ASTM D445, with measurements conducted at both 40 and 100°C [N. Salih and J. Salimon, 2022].

2.11.2 Viscosity Index

The Viscosity Index (VI) of a food-grade bio-lubricant serves as an indicator of how its kinematic viscosity changes in response to temperature fluctuations. Viscosity and temperature have an inverse relationship, meaning that machinery operating across a broad temperature range requires a lubricant with a higher VI. A higher VI implies minimal viscosity alterations with temperature shifts, whereas a lower VI signifies significant viscosity changes. Consequently, a higher VI enhances the stability of the lubricating oil product in varying temperature conditions. Bio-lubricants derived from vegetable oils typically exhibit higher VIs compared to mineral oil, ensuring their effectiveness even at elevated temperatures by preserving the oil film's thickness, thus rendering them suitable for a wide temperature range [H.M. Mobarak *et al.*, 2014].

A desirable bio-lubricant should possess a moderate Viscosity Index (VI), which essentially serves as a quality indicator. In the automotive industry, VIs are commonly used to characterize lubricants. The kinematic viscosity of the bio-lubricant in several studies were assessed using a physical model rheometer, the MCR 301 from Anton Paar Instruments (Germany). Viscosity and VI can be determined following the ASTM D 2270-04 method [S. Samidin et al, 2021].

2.11.3 Cloud Point

The cloud point (CP) for a food-grade lubricant refers to the temperature at which the initial indication of wax formation becomes observable, typically marked by the onset of cloudiness. The development of wax crystals can potentially lead to issues like filter blockages and the accumulation of deposits on equipment surfaces, including heat exchangers. Furthermore, this process tends to elevate the viscosity of food-grade lubricants. To prevent complications like filter clogging, it is essential to maintain temperatures above the turbidity point [Agrawal, A.J *et al.*, 2017].

2.11.4 Pour Point

At lower temperatures, the viscosity of a food-grade lubricant significantly increases, resulting in a notable resistance to flow. The pour point denotes the lowest temperature at which the lubricant can naturally flow or be poured under gravity, signifying the temperature at which motion ceases, not when solidification occurs. The pour point (PP) holds particular significance for equipment operating in cold environments or handling chilled liquids. High-viscosity oils may cease to flow at low temperatures due to wax formation, resulting in a pour point that exceeds the cloud point [F.J. Owuna *et al.*, 2020]. Vegetable oil-based bio-lubricants, in contrast, exhibit a lower pour point compared to mineral oils, delivering excellent lubrication performance during cold starts. The PP is determined following the ASTM D97 standard, a widely used method for assessing liquid flow properties at low temperatures. The sample is preheated and then gradually cooled at a specified rate, with flow characteristics evaluated within 3°C temperature intervals. To enhance precision, PP measurements are often conducted with a 1°C resolution, exceeding the specified 3°C increments [Muhammad Arif Dandan, 2018].

2.11.5 Flash Point and Fire Point

The flash point (FP) of a food-grade lubricant represents the lowest temperature at which the evaporating oil can ignite when exposed to an external ignition source, while the fire point designates the temperature at which the combustion of the lubricant generates sustained heat for at least five minutes after the removal of the ignition source. Both the flash point and fire point serve as critical indicators of the volatility and fire resistance of food-grade lubricants [F.J. Owuna *et al.*, 2020]. These values can be determined in accordance with the ASTM D92 method. It's essential that food-grade lubricants are not formulated using raw materials with flash points below 38°C (100°F) due to safety considerations. As a result, the flammability of the raw materials chosen for food-grade lubricant production hinges on their respective flash points, underlining their importance [N. Salih and J. Salimon, 2022].

2.12 Food Grade Lubricant Categories

In the United States and around the world, the National Sanitation Foundation (NSF) oversees a plan that mirrors the USDA program to properly evaluate lubricant use on machinery that comes into contact with food products. Facilities seeking to comply with applicable U.S. law must check all labels to ensure that the lubricant they use meets applicable standards and NSF classifications. This system assures consumers that food and pharmaceutical manufacturers are using food-grade lubricants that are safe for human consumption. [Ken Thayer 2018
H1: Lubricants are intended for applications where there is a risk of incidental or accidental food contact under normal conditions of use. This does not mean that lubricants or greases are safe in direct food contact or safe to consume. H1 lubricants offer beneficial antirust properties, they never contain heavy metals such as lead, arsenic, mercury, mutagens, teratogens or carcinogens. Any contact must be limited to a trace, not exceeding 10 parts per million or 0.001%.

H2: Lubricants are used in applications where there is no risk of food contact. Suitable for machine parts and equipment. Since there is virtually no risk of contamination, these lubricants are subject to fewer restrictions than H1 lubricants. For example, the oil used for forklift maintenance might be an H2 lubricant. In addition to rust prevention, H2 lubricants must be free of antimony, arsenic, cadmium, carcinogens, lead, mercury, mineral acids, mutagens, selenium, teratogens and heavy metals. [E.Tulsa 2023]

H3: This category of lubricants is food grade lubricants used in food processing environments where there is incidental contact of food with the lubricated surface. H3 lubricants are often based on edible oils, and are safe for human consumption such as corn oil, sunflower oil, soybean oil, or cottonseed oil, and inherently biodegradable, as well as meet FDA regulations. Moreover, H3 lubrications are often used to clean and prevent rust on equipment such as hooks, conveyor belts, and trolleys.

3. MATERIAS AND METHODS

3.1. Materials and Equipment

Raw materials: For this study, the basic raw material, i.e. rapeseed which was required for the production of food-grade lubricants was purchased from local market in shola market area where as the commercial lubricant was obtained from local industries.

Chemicals: Lab grade chemicals such as Hexane, Distilled water, Ethanol, Sodium Hydroxide, potassium Hydroxide, HCl, ethylene glycol and phenolphthalein were some of them obtained from School of Chemical and Bioengineering, Chemical laboratory where as other purchased from local laboratory reagent suppliers.

Equipment's: the lists of the main equipment's employed in this work are presented in table 3.1.

Table 3.1 List of equipment's used during the study.

Materials/Equipment's	Model of equipment	Purpose
Oven	-	Used to dry the samples
Coffee grinder	M.M2105	Used to grind the samples
Soxhlet	-	For extraction of oil
White fabric	-	To put the sample in to the Soxhlet
Rotary Evaporator	RVO400	separates low b.pt solvents from the sample
Density bottle	-	To measure density
Vibro viscometer	Sv-10	To measure viscosity
Thermometer	-	To measures temperature
Bunsen burner	-	To measure flash point
Digital balances	65.474T	To weigh the samples
Graduated cylinder	_	Volume measurement tool
Separating funnel	_	For separation of liquid samples
PH meter	3505 PH meter	PH Measuring tool
Beakers	APPROX VOL	To store liquid samples
Different size flasks	GG-17	to store biodiesel and lubricant samples
Filter paper	_	For separation of solid impurities
Water bath	TXF 200	heat transfer source at low temp.
Oil bath	RVO400	Heat transfer sources at high temp.
Refrigerator	_	For cooling purposes

3.2 Experimental Framework of the study

The framework of the experimental work was described in figure 3.1

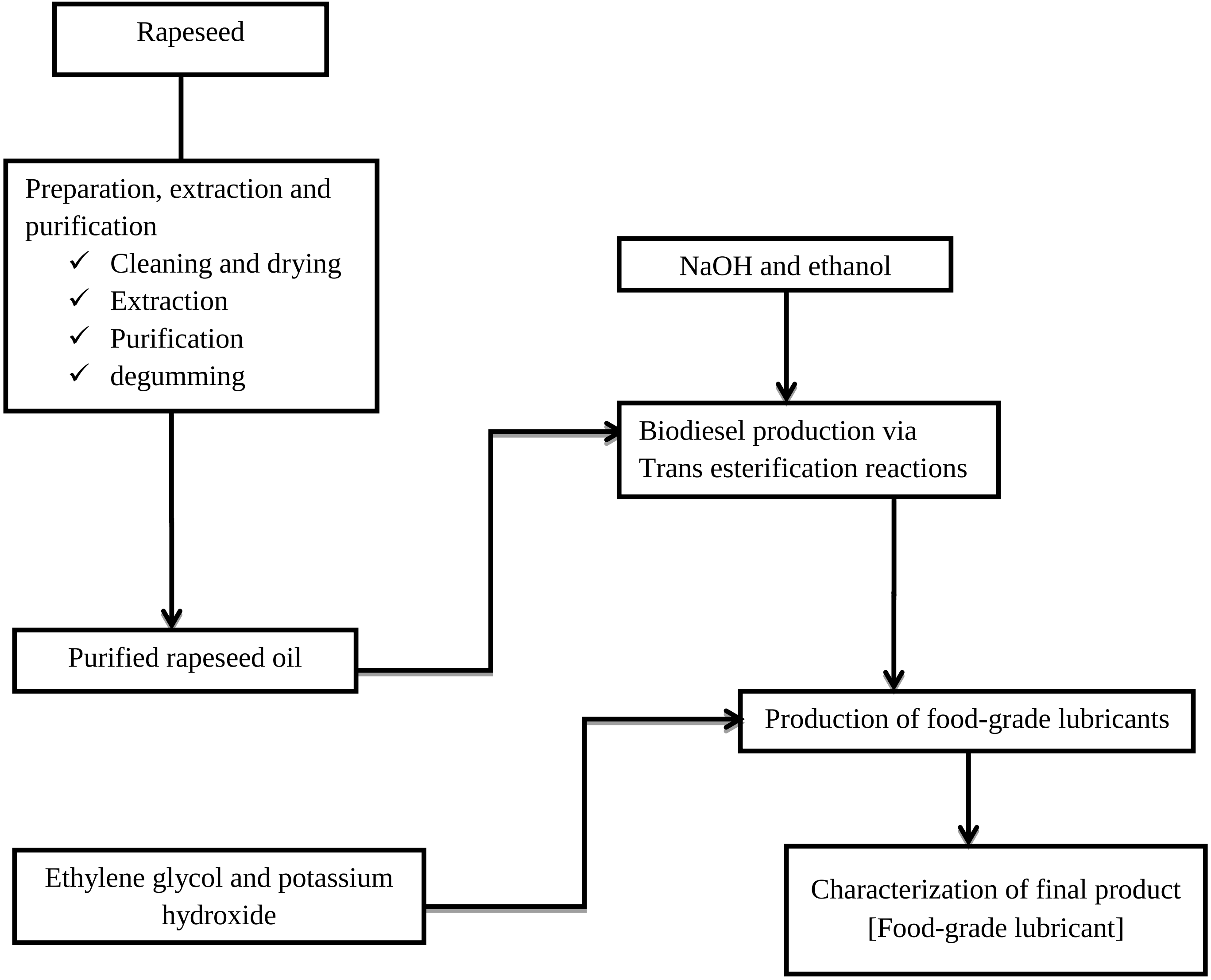


Figure 3.1 Experimental frame work of research

3.3. Experimental Methods

3.3.1 Rapeseed collection

952 g of rapeseed was collected from the market and sorted from impurities. The seeds were dried to the appropriate humidity in an oven at 40°C/min for storage[S Sensoz et al 2000], then placed in a dry place to avoid mold or rotten.

3.3.2. Rapeseed Size Reduction

Impurities from the rapeseed were removed, and heated to stabilize them before solvent extraction. The dried seed were subjected to size reduction using a coffee grinder with a 0.5 mm sieve size. The sample was sieved using a vibrator with mesh size arranged. This makes it possible to study the influence of particle size on oil yield and quantity. The range of sieve size appropriate for seed extraction is 0.224-1.8 mm [s sensoz et al 2000].

3.3. 3.Raw Rapeseed Moisture Content Determination

The moisture content determination was conducted using an oven, with all measurements carried out on the same day. The procedure involved several steps: firstly, the working temperature was set to 100°C. Secondly, the operational time was adjusted accordingly. Subsequently, 26 grams of rapeseed sample were meticulously weighed and placed inside the oven. The weight was then measured at one-hour intervals over duration of four hours. This process was repeated until a consistent weight was achieved, allowing for the determination of the percentage moisture content of the seeds [Douglas Schaufler 2023].

3.3.4. Rapeseed Essential Oil Extraction Procedure

Two round-bottom flasks were utilized, one containing 300 mL of normal hexane, and the other with 400 mL of normal hexane. Samples weighing 60g and 100g were enclosed in white cloth sacs and positioned at the center of the extractor. The Soxhlet apparatus was then heated to a temperature range of 75°C to 80°C and allowed to operate for duration of three hours. This procedure was duplicated with the same sample quantities placed in the white cloth sacs. The weight of the extracted oil was measured at regular intervals during each run. Upon completion of the extraction process [J.Redfern 2014], the resultant mixture comprising oil and hexane was obtained. Subsequently, the oil was subjected to heating to facilitate the solvent's evaporation from the oil using an evaporator.


a) b)

Figure 3.2: (a) Picture of Soxhlet extraction apparatus and (b) rapeseed oil in a beaker

3.3.5 Oil yield and Physicochemical Characterization of the extracted oil

3.3.5.1 Determination of Percentage of Oil Extracted

The yield of oil extracts was expressed as a percentage of the weight of oil obtained from extraction relative to the weight of rapeseeds used for extraction [A.K. Yusuf, 2018].

$$\text{Yield of oil extraction} = \frac{\text{mass of oil extracted}}{\text{mass of rapeseed used}} \times 100\% \text{--} (3.1)$$

3.3.5.2 PH Determination

To determine the pH value of a sample, a 3505-pH meter instrument was employed. Initially, a 30 ml sample of the oil was prepared. The pH electrode was calibrated using a buffer solution before immersing it into the oil sample. Subsequently, the pH reading was taken and duly recorded.

3.3.5.3. Determination of Kinematic Viscosity of Oil

The kinematic viscosity of the oil was determined using a Vibro Viscometer. Initially, a 35 mL sample of the oil was heated to 60°C. This sample was then placed into the Vibro Viscometer's sample holder, and the Viscometer's sensor was submerged in the 35 mL of oil. The dynamic viscosity of the oil was subsequently displayed on the Vibro Viscometer's screen at a temperature of 40°C, measured in millipascal-seconds (mpas) [Bilal et al., 2013]. To calculate the kinematic viscosity, we used the formula where it's equal to the dynamic viscosity divided by the density of the oil. Therefore, the kinematic viscosity was determined as follows:

$$\text{Kinematic viscosity of the oil} = \frac{\mu}{\rho} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.2$$

Where, μ = Dynamic Viscosity and ρ = Density of oil

3.3.5.4 Density Determination

The density of the extracted oil was established using a density bottle through a two-step measurement process. Initially, the empty density bottle was weighed and recorded. Then, the density bottle was filled with the oil up to a predefined mark of 25 mL, and its weight was measured again. Both sets of measurements were recorded. To determine the density of the oil, the following calculation was performed:

$$\text{Density} = \frac{M2-M1}{25} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.3$$

Where, M1=weight of empty bottle; M2=weight of oil filled bottle

3.3.5.5. Determination of Acid Value

To determine the acid value (AV) and assess the percentage of free fatty acid (%FFA) in the oil, it was began by accurately weighing 5.0 grams of the oil into a thoroughly dried conical flask. Next, 25 mL of absolute ethanol alcohol into the flask was introduced, followed by heating the mixture with agitation in a water bath for 10 minutes, then allow it to cool before adding 2-3 drops of phenolphthalein. Initiate titration by gradually adding 0.1 N KOH solution until a pink color appears, signifying the endpoint, and record the volume used. To calculate the AV, expressible in milligrams of potassium hydroxide (KOH) per gram of oil, use the formula: AV (mg KOH/g) = (Volume of 0.1 N KOH used) x 0.1 N x 56.1 (molar mass of KOH) divided by the weight of the oil in grams. Additionally, compute %FFA by applying the equation: %FFA = (AV in mg KOH/g) divided by 1,000 x 100. This comprehensive method allows for the precise determination of the oil's AV and %FFA, following the protocol described by Bilal *et al*. in 2013.

$$\text{Acid value} = \frac{56.1 \ast V \ast N}{W}\text{--}3.4$$

Where: V is the volume expressed in milliliter of 0.1N solution of KOH

W = weight in gram of the oil

N = normality of KOH

Free fatty acid is calculated as[Chika Muhammad *et al 2018*]:

$$\text{Free fatty acid} = \underline{\text{Acid value}}\text{--}3.5$$

3.3.5.6. Determination of Saponification Value

To determine the saponification value, it was started by weighing approximately 2 grams of rapeseed oil into a 250 mL conical flask. Then, 25 mL of a 0.5 N alcoholic potassium hydroxide solution was added. Attached with a reflux condenser and heated the flask contents on a boiling water bath for 1 hour, it was shacked intermittently. While the solution is still hot, introduce 3 drops of phenolphthalein indicator and titrate the excess potassium hydroxide solution using 0.5 N hydrochloric acid until the color disappears, noting the volume of hydrochloric acid used as V_a. Repeat the same procedure without the oil sample to determine V_b (volume of hydrochloric acid at the endpoint). To calculate the saponification number, the following formula was applied [Chika Muhammad et al. in 2018]:

$$\text{Saponification Number} = \frac{56.1 * (Vb-Va) * N}{W} \text{--3.6}$$

Where: W= weight of oil taken in gram; N= normality of HCL solution

Va= volume of HCL solution used in the test in milliliter.

Vb= volume of HCL solution used in blank (without oil sample) in milliliter.

3.3.5.7. Ester Value

The ester value is defined as the mg of KOH required to react with glycerin (glycerol/or glycerin) after saponify one gram of fat. It is calculated from the saponification value:

Ester Value (EV) = Saponification Value (SV) – Acid Value (AV)…………………….…3.7

3.3.6 Transesterification of Rapeseed Ethyl Ester (REE) from Rapeseed Oil

The first step involved in the transesterification process aimed at transforming extracted rapeseed oil into biodiesel entailed the reaction of the oil with ethanol, facilitated by the presence of NaOH as a catalyst. This reaction took place with a molar ratio of 15:1 (ethanol to oil), at a controlled temperature of 50ºC, over a duration of one hour, while stirring at a speed of 700 rpm. The catalyst was employed in an amount equivalent to 1% of the total weight of the reactants, resulting in the production of ethyl ester (biodiesel) and glycerol as the key products.

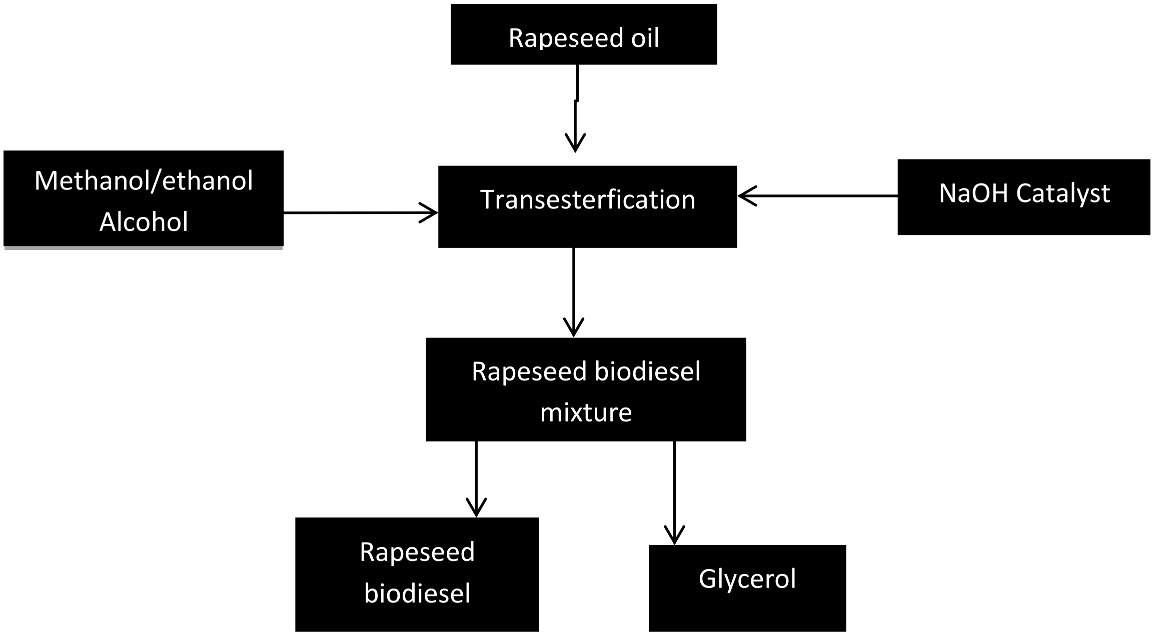


Figure 3.3 Biodiesel production process from rapeseed oil though transesterfication

In the initial phase of the experiment, the NaOH catalyst was combined with ethanol and stirred thoroughly until achieving complete homogeneity. Subsequently, 40 ml of rapeseed oil was accurately measured using a graduated cylinder and then heated to 50ºC using a reflux setup with a stirrer. Once the desired temperature was reached, the NaOH-ethanol mixture was introduced into the continuously stirred and heated oil. Following the completion of the reaction, 4 ml of pure glycerol was introduced due to the formation of a strong emulsion during ethanolysis. Notably, gravity separation did not suffice to isolate the glycerol. Subsequently, the reaction mixture was allowed to stand until a clear separation occurred, which took approximately 24 hours, with the lighter biodiesel (rapeseed ethyl ester) layer rising above the denser glycerol layer. The final step involved removing the glycerol layer to isolate the biodiesel layer.

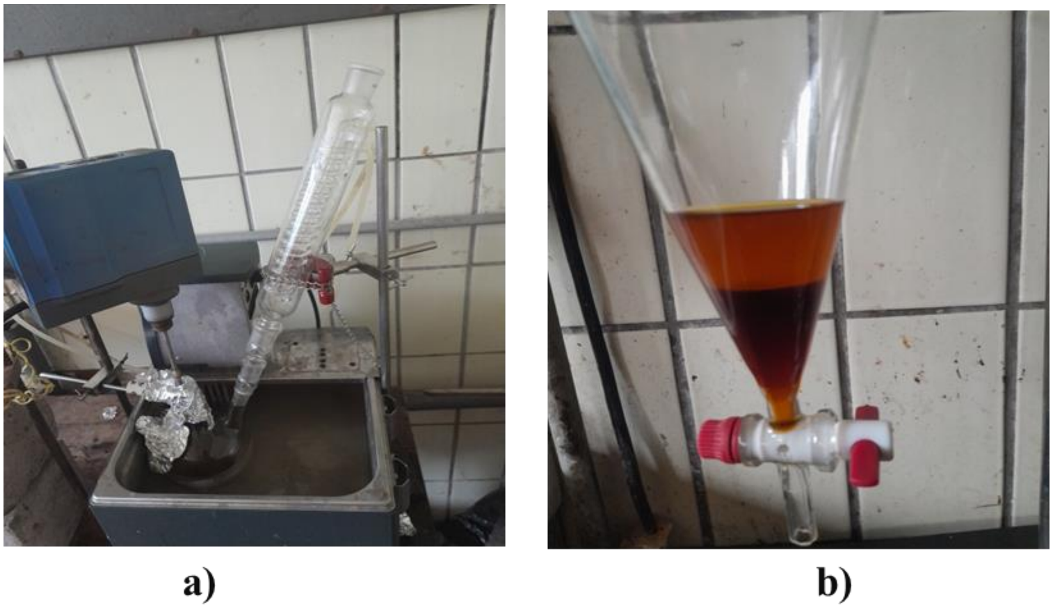

a) b)

Figure 3.4 (a) experimental setup for transesterification reaction, (b) produced biodiesel

3.3.6.1 Physico-Chemical Characterization of Rapeseed Ethyl Ester (Biodiesel)

The physico-chemical analysis of biodiesel followed a procedure similar to that used for measuring rapeseed oil properties. Parameters such as density, viscosity, acid value, free fatty acid value, and saponification number were measured in a consistent manner.

3.3.7 Transesterification of Rapeseed Ethyl Ester (REE) to Rapeseed Food Grade Lubricant

The production of food-grade lubricant followed a similar procedure to biodiesel production, with the exception of using a different alcohol (ethylene glycol) and KOH as the catalyst. The process involved transesterifying rapeseed ethyl ester with ethylene glycol, maintaining constant reaction time and stirring speed. Various mole ratios of Ethylene Glycol (EG) to rapeseed ethyl ester (biodiesel) were tested, ranging from 2:1 to 4:1, and the reactions were conducted at temperatures between 140°C and 160°C, with catalyst concentrations ranging from 0.5% to 1.5% by weight, all for a duration of one hour. After cooling the reaction mixture to room temperature in an ice bath, the resulting combination was filtered to separate waste from the liquid mixture, yielding the food-grade lubricant. These runs were conducted under atmospheric pressure.

3.3.7.1 Physico-Chemical Characterization of Food Grade Lubricant

3.3.7.1.1. Determination of Pour Point

The pour point (PP) is defined as the lowest temperature at which a liquid can flow or pour, as explained by Nadia Salih and Jumat Salimon in 2022. To ascertain the pour point of the food-grade lubricant, a procedure was followed wherein 30 ml of the lubricant oil was placed in a beaker, sealed with aluminum foil, and a thermometer was inserted into it. Subsequently, the beaker was positioned in a refrigerator. The sample's temperature was periodically checked at five-minute intervals by tilting it horizontally. The pour point was identified as the temperature at which the surface of the lubricant remained stable without sagging for five seconds.

3.3.7.1.2. Determination of Cloud point

Cloud point is the temperature at which the first crystal wax of food grade lubricant is formed when cooled. To determine cloud point of lubricant, 30 ml of lubricant was measured and poured in to beaker. Then, the beaker was sealed by roller (aluminum foil) and thermometer was inserted in to the sample through small hole. The sample was put on refrigerator and cooled for minutes. The temperature of the food grade lubricant when the first cloud crystal wax was observed was measured.

3.3.7.1.3. Determination of Flash point

The flashpoint of a food-grade lubricant, as defined by Nadia Salih and Jumat Salimon in 2022, is the lowest temperature at which the lubricant oil can catch fire when exposed to an external ignition source. To determine this flashpoint, a procedure was followed where 40 ml of the food-grade lubricant was poured into an open metal cup and placed on a heat source. A thermometer was secured to a retort stand and inserted into the cup. Using a test flame applicator, a flame was passed over the lubricant surface in a continuous, smooth motion, and the temperature at which the sample ignited was noted as the flashpoint, with the aid of the thermometer.

3.3.7.1.4. Determination of Viscosity, Density, Acid Value (AV) and Free Acid Value (FFA)

The procedure employed for characterizing oil properties, including viscosity, density, acid value (AV), and free fatty acid value (FFA), closely resembled the method used for the food-grade lubricant analysis.

3.4. Experimental Design for Food Grade Lubricants Production

The experiment was devised utilizing Design Expert software version 13, which employed the Box-Behnken experimental model to enhance the extraction of food-grade lubricants from rapeseed. It's a common practice in experiments to aim for optimization of results, and this entails designing input parameters to achieve the best possible outcomes. Optimization can involve minimizing or maximizing specific parameters. Beyond standard methodologies, the Response Surface Technique stands out as the most effective means to attain optimal results. Response Surface Methodology (RSM) is a collection of mathematical and statistical tools particularly valuable for modeling and analyzing scenarios where multiple variables and objectives influence the desired outcome. The primary objective is to optimize this response (as highlighted by Montgomery in 2005) while establishing a more robust relationship between the optimal input material parameters and the optimal output and yield parameters.

The performance of the food-grade lubricant serves as the metric for assessing the response. To explore the joint impacts of catalyst concentration, the molar ratio of ethylene glycol to biodiesel in rapeseed, and reaction temperature on the quantity of food-grade lubricant produced at three different grades, we implemented a three-variable, three-level experimental design, encompassing 17 test runs.

Table 3.2: Independent variables and level of main factors that affect transesterification reaction

Variables	Unit	Level		
		-1	0	+1
EG to biodiesel ratio	mole/mole	1:1	2:1	3:1
Catalyst	Wt.%	0.5	1	1.5
Reaction temperature	°C	145	155	165

Typically, the determination of the number of design points for Box-Behnken Designs (BBDs) relies on the formula 2K(K-1) + NC, where K represents the number of factors, and NC signifies the number of center points. This approach is valuable for assessing the suitability of multiple variables, as it helps unveil potential interactions between these variables. It's favored for its well-structured design and its capacity to yield favorable and reliable outcomes.

3.5. Functional group determination

The IR spectrum was captured using a Perkin Elmer Spectrum-100 FTIR spectroscope. A purified sample weighing approximately 2 mg was made into KBr pellets and used for the recording. The resulting IR spectrum was calibrated across the wavenumber range of 4000 to 400 cm^{-1}. The FTIR instrument comprises key components including the radiation source emitting light directed to the sample via the interferometer. This device separates source radiation into varied wavelengths. A slit chooses wavelengths passing through the sample. A beam splitter divides the incident beam; half goes to the sample, half to the reference. The sample's chemical properties absorb light. A detector compares energy passing through the sample with the reference. The resulting electrical signal is sent to a recorder via the interferometer, translating energy into frequency/wavelength data. This data forms a fingerprint displayed on a computer monitor, originating as numerical data. This spectroscopic instrument can be found within the Chemistry Department of Addis Ababa University. The biolubricant sample was combined with KBr particles to enable infrared analysis. The resulting mixture was then compressed to achieve a thickness of approximately 1mm, which is ideal for FTIR analysis. The identification of functional groups was accomplished using established IR absorption characteristics from fundamental spectroscopy textbooks.

4. RESULTS AND DISCUSSION

4.1. Physico-Chemical Characterization of Extracted Oil

The physicochemical characterization of extracted oil is shown in table 4.1

Table 4.1 Quality parameter of extracted oil

Quality parameter	Unit	Experimental result
Rapeseed oil yield	%	43.125
PH	_	7.2
Density	g/ml	0.925
Kinematic viscosity	Cst	32.528
Saponification value	mg KOH/g oil	164.56
Acid value	mg KOH/g oil	0.935
FFA	%	0.465
Moisture content	%	6.4

Moisture content

The moisture content of the rapeseed was determined to be 6.4%. This indicates the amount water present in rapeseed. This finding aligns closely with the prior research by Nevendon Voca *et al.*, in 2022, where the critical moisture content range was identified as being between 7% to 10%. It's important to note that excessively high moisture content can lead to a reduction in storage life, as it may promote rapid losses due to mold growth. Conversely, very low moisture content, below 4%, may also be detrimental to the seeds, causing extreme desiccation or resulting in hard seededness.

Percentage of Extracted Oil

The highest oil yield, reaching 43.125%, was achieved when the extraction process was conducted at a temperature of 80°C for duration of 3 hours, utilizing n-hexane as the solvent. This oil yield closely aligns with the figures reported in the literature by H.M. Mobarek *et al.* in 2014, which indicated a range of 38% to 46% oil yield**.**

Kinematic Viscosity

The kinematic viscosity of the rapeseed oil was 30.088 mpas obtained from vibro viscometer on screen displayed. The kinematic viscosity of the oil is determined according to equation (3.2) and the result was 32.528mm2/s, refers to how easy the oil pours at a specified temperature . This result was consistent with the previous report of Encinar (2010) of 33.07 mm2/s. Oil viscosity affects fuel oil storage and handling. High viscosity oils are difficult to pump and run in CI engines. In this study, the results showed that rapeseed oil was low viscosity and can be used in CI engines.

Saponification Value

The average saponification value obtained for rapeseed oil was 164.56, as shown table 4.1, is indication of the nature of fatty acid constituent of fat present in rapeseed oil. A high saponification number indicates a lower molecular weight proportion of fatty acids, which in turn signifies the oil's greater potential for soap production. The result aligns with previous findings in the literature [Encinar (2010)].

Acid Value

In the case of rapeseed oil, the determined acid value was 0.935; this indicates the amount of free acid present in the oil and serves as a crucial indicator of the quality of vegetable oil. it can be calculated using equation 3.4 and the corresponding results were given in table 4.1. The result that aligns with what has been reported in the literature. It's noteworthy that the maximum acceptable acid value for alkaline transesterification of oil is reported as 1.02 according to Encinar *et al.*, in 2010. A higher acid value is indicative of the degradation of oil fats, and it can be seen as a sign that the oil is free from hydrolytic rancidity, emphasizing its overall quality.

4.2 Physico-Chemical Characterization Of Rapeseed Ethyl Ester (Biodiesel)

Table 4.2: Physico-chemical properties of rapeseed oil biodiesel

Property	Unit	Experimental Result
Density at 20ºC	g/ml	0.87
Kinematic viscosity at 40ºC	mm^2/s	5.4
Acid Value	mgKOH/g oil	0.4488
Free Fatty Acid	%	0.22
Pour point	ºC	-6
Flash point	ºC	156
Cloud point	ºC	-7

Kinematic viscosity

The kinetic value of biodiesel was 5.4mm2/s.as shown in Table 4.2 above.this value corresponds to the ASTM D445 standard range of 3.5 to 5.9 mm2/s.Low viscosity fuel cannot properly lubricate the fuel injection pump and causes leaks and wear. On the other hand, fuels with higher viscosity form large droplets during fuel injection and cause poor combustion, exhaust smoke and emissions.

Acid number

The acid number of biodiesel was 0.4 mg KOH/g. this value corresponds to the maximum international standard range of 0.5 set by the EN14214 standard [Sh.A.Biktashev et al 2011].Acid number is a measure of the number of carboxylic groups such as free fatty acids. Higher acid numbers lead to corrosion of fuel pumps systems and internal combustion engines. Lower acid numbers contain fewer free fatty acids.

Flash point

The flash point of biodiesel was achieved at 156°C.The flash point of a fuel is the factor that determines its ability to ignite, store and transport the fuel.This result is consistent with the ASTM D93 standard range of 140°C to 160°[Oguz et al 2016].

4.3 Physico-Chemical Characterization of Food Grade Lubricant

The physicochemical properties of food grade lubricant shown in table 4.3 below

Table 4.3 Physico-chemical properties of food grade lubricant

Property	Unit	Experimental Result
Density at 20ºC	g/ml	0.875
Kinematic viscosity at 40ºC	mm^2/s	13.714
Acid Value	mgKOH/g oil	0.336
Free Fatty Acid Composition	%	0.168
Cloud point	ºC	-1
Flash Point	ºC	216
Pour point	ºC	-9

Pour point

The freezing point of food grade lubricants is -9°C as shown above. Table 4.3 describes the lowest temperature at which food-grade lubricants flow. The results meet the scope of ASTM D-97, as agreed by previous correspondent J.M Encinar 2020.High pour point values become semi-solid at temperatures that cause machine freezes during processing operations.Therefore, lower pour points are considered more desirable.

Cloud point

The cloud point of food grade lubricants is -1 oC, indicating the suitability of food grad lubricants when cold, as shown in Table 4.3.This result is consistent with previous work at 0 oC.

Flash Point

The flash point of food-grade lubricants is achieved at 216°C, indicating the safety risk of the lubricant in terms of fire and explosion.This result is consistent with the previous report [Encinar 2020] with ASTM D92.Where the flash point of 222°C.

4.4 FTIR Analysis of the Rapseed Biolubricant

The Fourier transform infrared spectroscopy (FTIR) technique was used to analyze the functional groups such as alcohol, alkane, alkynes, alkenes, carbonyl groups and others present in the sample which here is in the biolubricant derived from rapeseed oil. The response of the functional groups was analyzed by observing the transmission of infrared radiations and comparing it with known standards (from table of characteristics IR absorption) in order to identify the type and the nature of functional groups present in the samples. The provided figure below demonstrates that the second transesterification of rapeseed oil methyl esters (FAME) into biolubricants can be verified by the emergence of a peak representing the ester carbonyl (C=O) functional group at 1740.20 cm^{-1}.

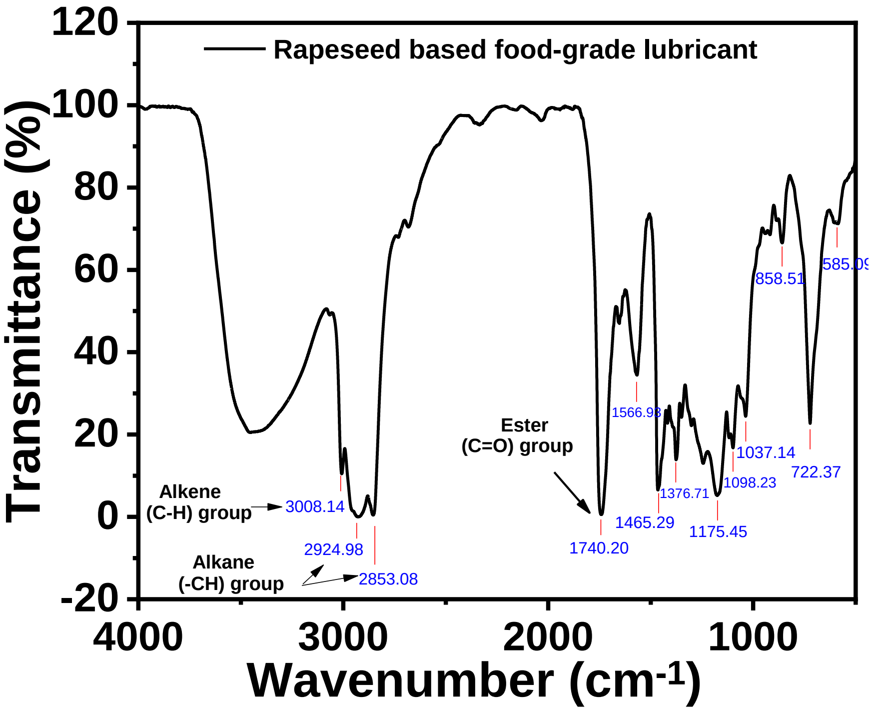


Figure 4.1 FTIR spectrum of food-grade rapeseed-based lubricant.

All carbonyl compounds absorb in the region 1820-1630 cm^{-1} due to the stretching vibration of the C=O bond. This distinctive carbonyl band is particularly useful for diagnostic purposes because it has a characteristic high intensity and few other functional groups absorb in this region as well. Additionally, the spectrum displays distinct peaks within the range of 2800 to 3010 cm^{-1}, at 1465.29 cm^{-1}, and in the fingerprint region (400-1400 cm^{-1}). These peaks correspond to simple alkanes as characterized by absorptions due to C–H stretching and bending or C–C stretching and bending bands, and potentially denote symmetric CH_3 deformation vibration or –CH bending, as well as the potential presence of alkene groups (as can be compared from table of characteristics IR absorption elsewhere). Consequently, the findings suggest that the transesterification process effectively modifies the oil, transforming it into a more valuable biolubricant.

4.5 Yield of Food Grade Lubricant from Rapeseeds

The results for yield (percentage) of rapeseed food-grade lubricants are presented in table 4.6. These findings reveal variations in lubricant performance, suggesting that different experimental conditions have an impact on lubricant characteristics. Consequently, the experimental parameters significantly influence the lubricant yield. The highest recorded lubricating efficiency, reaching 94%, was achieved under the conditions of 1% catalyst concentration, a 3:1 ratio of ethylene glycol to rapeseed biodiesel, and a temperature of 165°C.

Table 4.4 Experimental design layout using Box-Behnken design.

Run	Factor 1 A:Temprature ºC	Factor 2 B:Molar Ratio mole/mole	Factor 3 C:Catalst Concentration Wt.%	Response 1 Yield %
1	165	2	1.5	92
2	145	2	1.5	88
3	155	2	1	86.5
4	155	2	1	87
5	155	1	1.5	75.5
6	165	3	1	94
7	155	2	1	87.6
8	155	2	1	87.8
9	165	2	0.5	84
10	145	2	0.5	93
11	145	1	1	89.5
12	155	2	1	87.2
13	145	3	1	91
14	155	3	0.5	76

15	165	1	1	83
16	155	3	1.5	72.5
17	155	1	0.5	65

4.6 Statistical Analysis of Experimental Results

4.6.1 Analysis of Variance (ANOVA) for Quadratic Model

Table 4.5 Analysis of variance for the food grade lubricant

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	1009.86	9	112.21	134.02	<0.0001	Significant
A-Temprature	9.03	1	9.03	10.79	0.0134	
B-Molar Ratio	52.53	1	52.53	62.75	<0.0001	
C-Catalst Concentration	12.50	1	12.50	14.93	0.0062	
AB	22.56	1	22.56	26.95	0.0013	
AC	42.25	1	42.25	50.46	0.0002	
BC	49.00	1	49.00	58.53	0.0001	
A^2	386.23	1	386.23	461.32	<0.0001	
B^2	231.97	1	231.97	277.08	<0.0001	
C^2	239.85	1	239.85	286.49	<0.0001	
Residual	5.86	7	0.8372			
Lack of Fit	4.81	3	1.60	6.12	0.0563	not significant
Pure Error	1.05	4	0.2620			
Cor Total	1015.72	16				

Factor coding is **Coded**.

Sum of squares is **Type III - Partial**

The **Model F-value** of 134.02 suggests that the model is indeed significant. The probability of obtaining an F-value as large as this one due to random noise is very low, at approximately 0.01%. This indicates that the model's performance is unlikely to be a result of random chance and is likely capturing meaningful relationships in the data.

P-values less than 0.0500 generally indicate that model terms are statistically significant. In this particular case, terms A, B, C, AB, AC, BC, A^2, B^2, and C^2 all have p-values less than 0.0500, signifying that they are significant model terms. Conversely, when p-values are greater than 0.1000, it suggests that the corresponding model terms are not statistically significant. If the model contains many insignificant model terms (excluding those necessary for hierarchy or other theoretical reasons), it might be beneficial to consider model reduction techniques. Reducing the number of non-significant terms can lead to a more parsimonious and interpretable model without losing important information, potentially improving the model's performance and generalizability.

The **Lack of Fit F-value** of 6.12 suggests that there is approximately a 5.63% chance that such a Lack of Fit F-value could occur due to random noise. A low probability of less than 10% for Lack of Fit indicates that there may be some issues with the model's fit to the data. In an ideal scenario, we would want the model to fit the data well, and a higher Lack of Fit F-value indicates that the model might not be adequately capturing the underlying relationships in the data. This result should be investigated further to improve the model's performance.

- **Fit Statistics:**

Table 4.6: Model adequacy measures

Std. Dev.	0.9150		R^2	0.9942
Mean	84.68		Adjusted R^2	0.9868
C.V. %	1.08		Predicted R^2	0.9226
			Adeq Precision	40.3445

The **Predicted R^2** of 0.9226 being in reasonable agreement with the Adjusted R^2 of 0.9868 suggests that the difference between these two metrics is less than 0.2. This close alignment between the Predicted R^2 and the Adjusted R^2 indicates that the model is performing well in terms of explaining the variation in the data.

An **Adequacy Precision** ratio greater than 4 is considered desirable as it signifies a strong signal-to-noise ratio. In this case, with a ratio of 40.345, a very strong signal, indicating that the model can effectively navigate the design space. This high signal-to-noise ratio suggests that the model's predictions are reliable and can be confidently used for making decisions and optimizations within the design space.

Table 4.7: Coefficients in terms of coded factors

Factor	Coefficient Estimate	Df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	87.22	1	0.4092	86.25	88.19	
A-Temprature	-1.06	1	0.3235	-1.83	-0.2975	1.0000
B-Molar Ratio	2.56	1	0.3235	1.80	3.33	1.0000
C-Catalst Concentration	1.25	1	0.3235	0.4850	2.01	1.0000
AB	2.37	1	0.4575	1.29	3.46	1.0000
AC	3.25	1	0.4575	2.17	4.33	1.0000
BC	-3.50	1	0.4575	-4.58	-2.42	1.0000
A^2	9.58	1	0.4459	8.52	10.63	1.01
B^2	-7.42	1	0.4459	-8.48	-6.37	1.01
C^2	-7.55	1	0.4459	-8.60	-6.49	1.01

The coefficient estimate tells us how much we can expect the response to change for each unit change in a factor's value, assuming that all the other factors remain constant. In orthogonal designs, the intercept represents the average response across all experimental runs. The coefficients then represent adjustments to this average based on the specific settings of the factors. When factors in a design are orthogonal, the Variance Inflation Factors (VIFs) are equal to 1. VIFs greater than 1 indicate that there is some degree of multicollinearity, meaning that the factors are correlated with each other to some extent. The higher the VIF, the more severe the correlation among the factors. As a general guideline, VIFs less than 10 are considered tolerable, suggesting that the multicollinearity is not extreme and should not unduly affect the model's reliability.

4.6.2 Development of Regression Model Equations

- **Final equation in terms of coded factors**

$Yield$ =87.22 -1.0625 * A + 2.5625 * B + 1.25 * C + 2.375 * AB + 3.25 * AC -3.5 * BC + 9.5775 * A^2 -7.4225 * B^2 -7.5475 * C^2

Where, A = Temperature, B = Molar Ratio, C= Catalyst Concentration

The equation expressed in terms of coded factors allows us to predict the response for specific combinations of factor levels. Typically, in this representation, the high levels of factors are coded as +1, and the low levels are coded as -1. This coded equation serves as a valuable tool for assessing the relative influence of different factors by examining their coefficients. In summary, the provided equation relates the response (in this case, the yield of Food Grade Lubricant) to the variables involved in the transesterification process, and it does so use a coded factor framework.

- **Final equation in terms of actual factors**

$Yield$ =2,497.55-30.9215 * A + 2.44 * B -23.87 * C + 0.2375 * AB + 0.65 * AC -7 * BC + 0.095775 * A^2 -7.4225 * B^2 -30.19 * C^2

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

4.6.3 Model Fit Test Diagnostics

A standard probability plot is a graphical technique employed to assess whether the errors observed during a test analysis follow a normal distribution throughout the entire experiment. In the provided Figure 4.2, a normal probability distribution is displayed, where data points are roughly aligned along straight lines. This alignment suggests that the quadratic model meets the assumption of ANOVA, indicating that the distribution of errors is nearly normal.

Design-Expert® Software

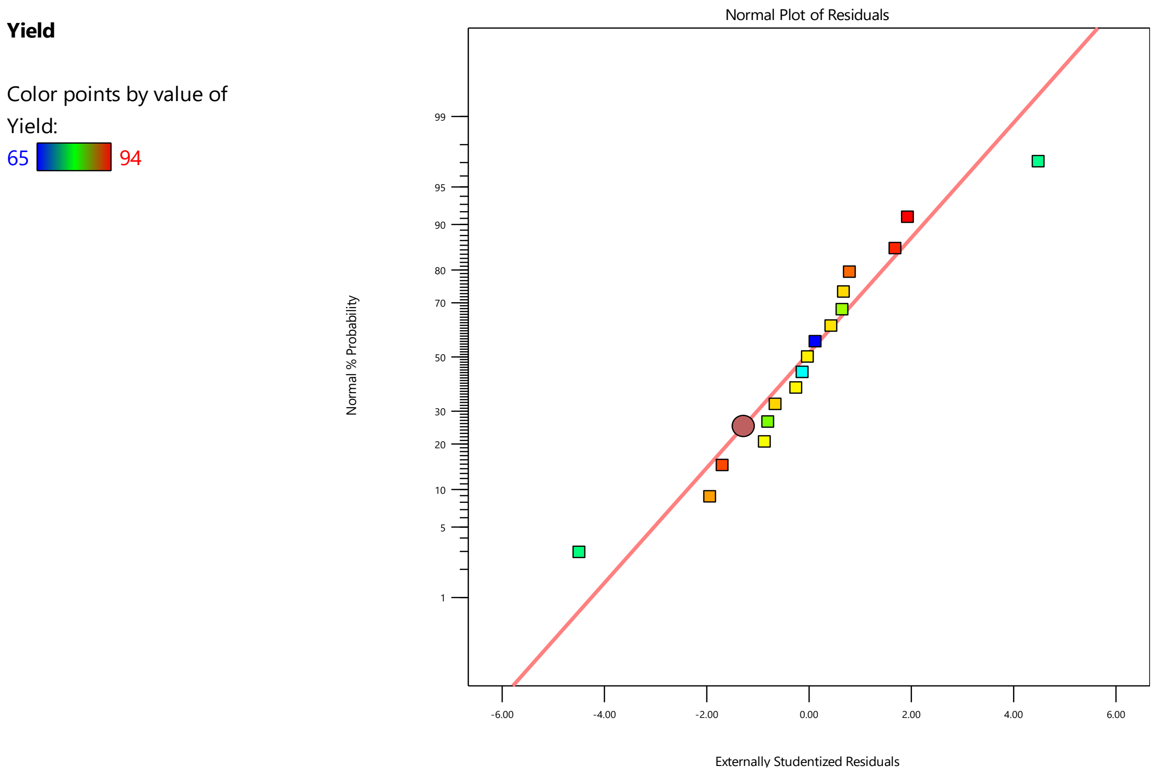


Figure 4.2 Normal probability plot of residuals

The correlation between the actual experimental data and the predicted value can be used to determine the fit of the model. A model is perfect if the slope of the line is in units (all data points lie along the line). Corresponds to zero error. The graph of the predicted slope versus the actual slope is shown in Figure (4.3). It can be observed from the graph that all the data points are close to the perfect fit line. Therefore, the regression model equation provided an accurate description of the experimental data.

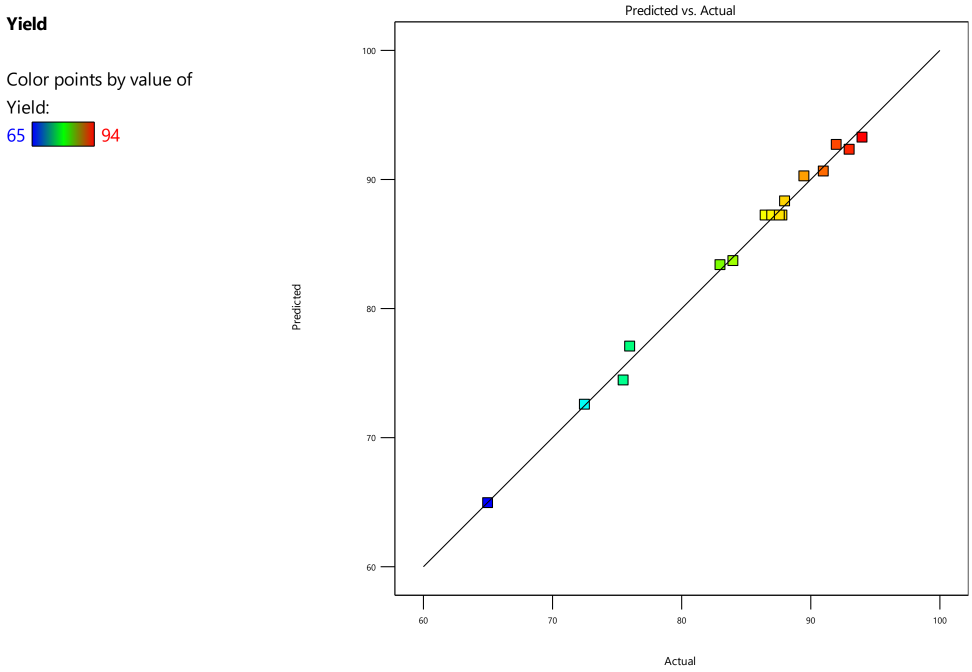


Figure 4.3 Predicted versus actual yield

Design-Expert® Software

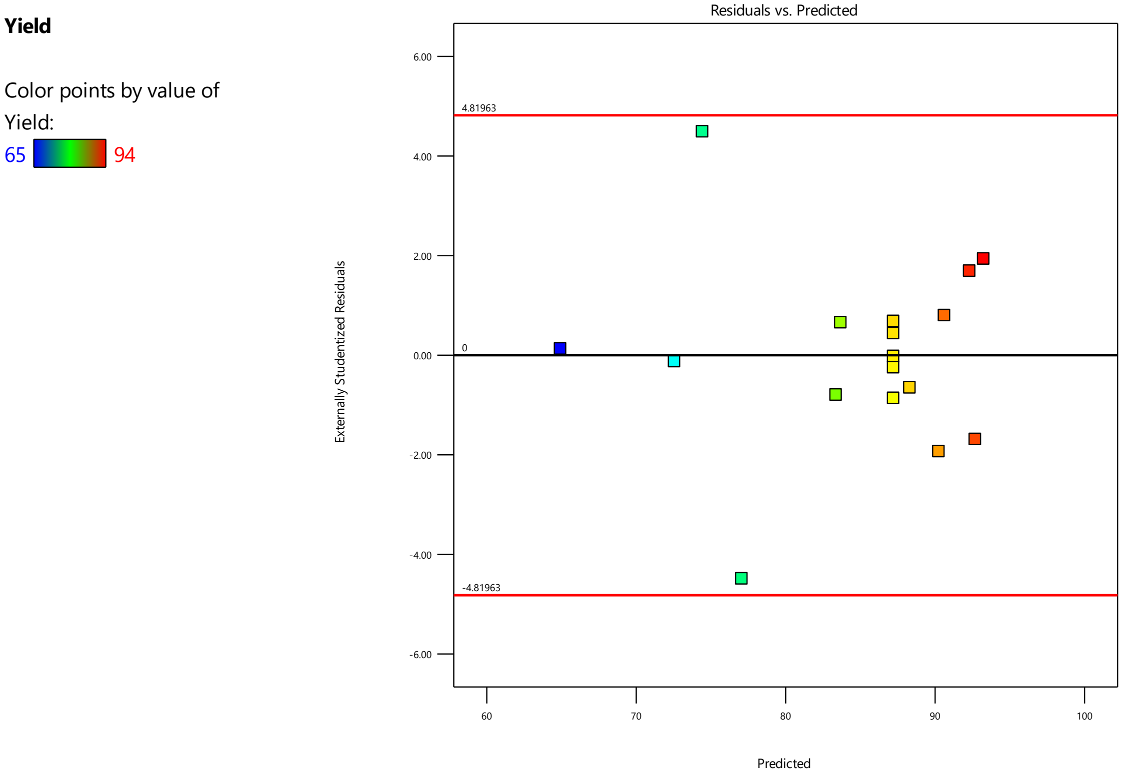


Figure 4.4 Residual versus predicted value plot

Another graphical method for assessing the validity of a model involves plotting residuals against predicted values. A model, such as the process description model in this context, is deemed perfect when the residuals exhibit an unstructured pattern. Histograms are also utilized to examine various aspects like nonlinearities, unequal variances, and the presence of outliers. As demonstrated in the figure above, the histogram of residuals displays an unstructured pattern. This finding indicates that there is no necessity to make specific adjustments or downplay individual errors in the model, as it appears to adequately capture the underlying patterns in the data.

4.7 Effect of Individual Variable Factors on Performance of Food Grade Lubricants

The results obtained from the analysis of variance (ANOVA) reveal that the performance of food grade lubricants is indeed influenced by the input variables, which include the ethylene glycol/biodiesel ratio, temperature, and catalyst concentration. This investigation was conducted within defined operating conditions. Furthermore, the model also indicates the significance of interaction effects, signifying that variations in these process variables have a notable impact on the production of food grade lubricants.

4.7.1 Ethylene Glycol to biodiesel ratio

The molar ratio of ethylene glycol to biodiesel is one of the main factors affecting the transesterification conversion reaction. At constant temperature and catalyst, the performance of food grade lubricant is shown in the figure below. This indicates that the performance of food grade lubricants increases as the ethylene glycol/biodiesel ratio increases to the optimum value. But the reduction of ethylene glycol leads to a decrease in the transesterification conversion coating efficiency and leads to a lower product yield. Maximum food-grade lubrication efficiency (94%) was obtained with an ethylene glycol and biodiesel ratio of 3:1. so the ratio was taken as the optimal value of the molar ratio. On the other hand, increase the molar ratio of ethylene glycol to biodiesel above the optimal value (3:1) Excess ethylene glycol, reduces the performance of food grade lubricants.

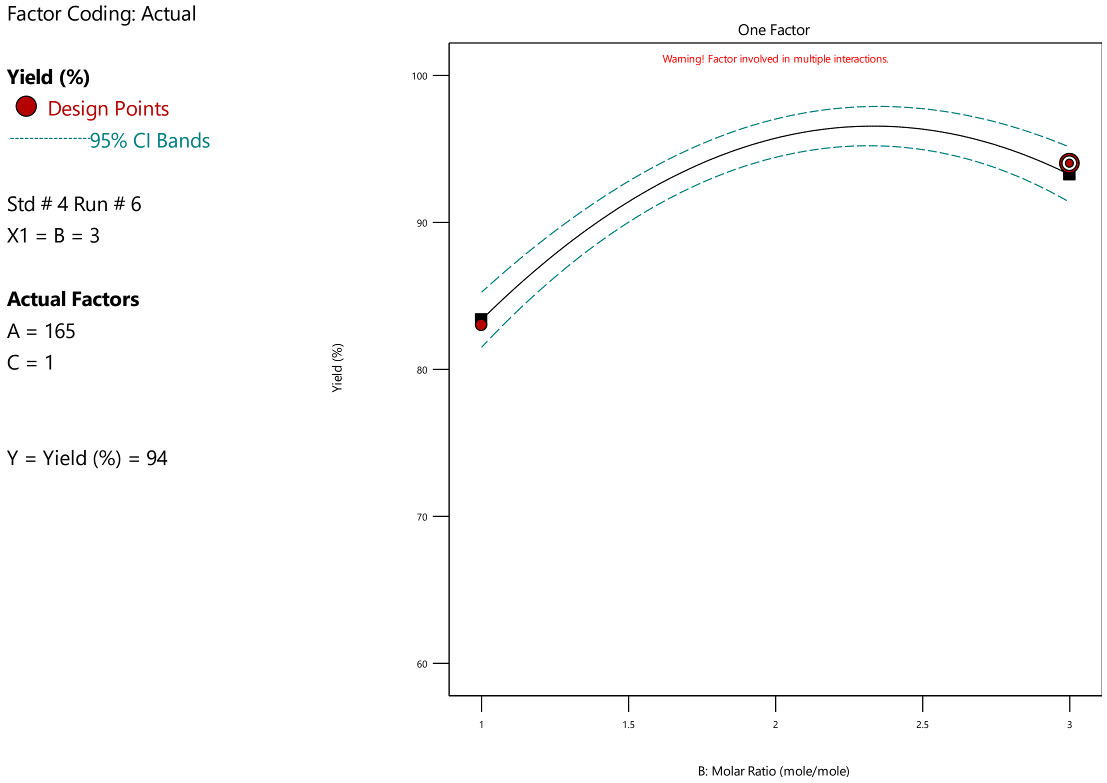


Figure 4.5 Effect ethylene glycol to biodiesel ratio on yield of food grade lubricants

4.7.2 Effect of Temperature

Regarding the effect of temperature on the yield of food grade lubricants (Figure 4.6) at constant of ethylene glycol to biodiesel ratio and catalyst concentration, as the temperature was increased, the reaction proceeds at a faster rate. Achieving a high conversion. This shows that the effect of the reaction temperature on the yield of transesterification reaction. The reaction rate and efficiency increase as the temperature was increased from 145 ºC to 165 ºC. For the final yield of the food grade lubricant, the temperature at which the highest efficiency (94%) was selected at 165 ºC and the yield begins to decrease slightly when the temperature approaches the boiling point of ethylene glycol.

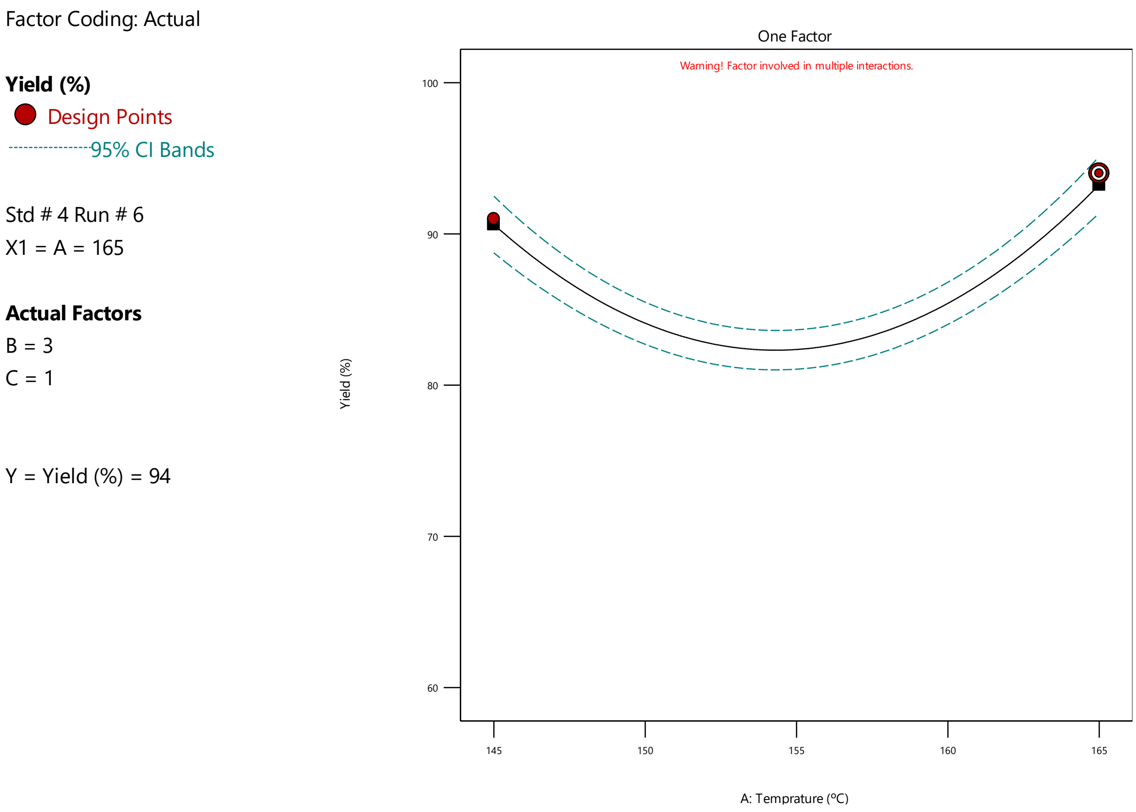


Figure 4.6 Effect of temperature on yield of food grade lubricant

4.7.3 Catalyst Concentration

Figure 4.7 illustrates the impact of catalyst concentration on the performance of food grade lubricants, while keeping temperature and the ethylene glycol/biodiesel ratio constant. As anticipated, the addition of more catalyst led to a higher reaction rate, resulting in the attainment of equilibrium in a shorter reaction time. This positive effect of catalyst concentration on the reaction rate response is evident. Specifically, when using 0.5% catalyst (by weight), the final conversion was less than 1%, and the yield remained at 1% weight. However, when 1% weight catalyst was used, the efficiency substantially improved to 94%. The efficiency demonstrated a linear increase with the growing amount of catalyst, but it started to decline with further increases in catalyst concentration. This suggests the presence of an optimal catalyst concentration beyond which no further enhancement in conversion was observed. Consequently, an intermediate catalyst concentration of 1% weight was selected.

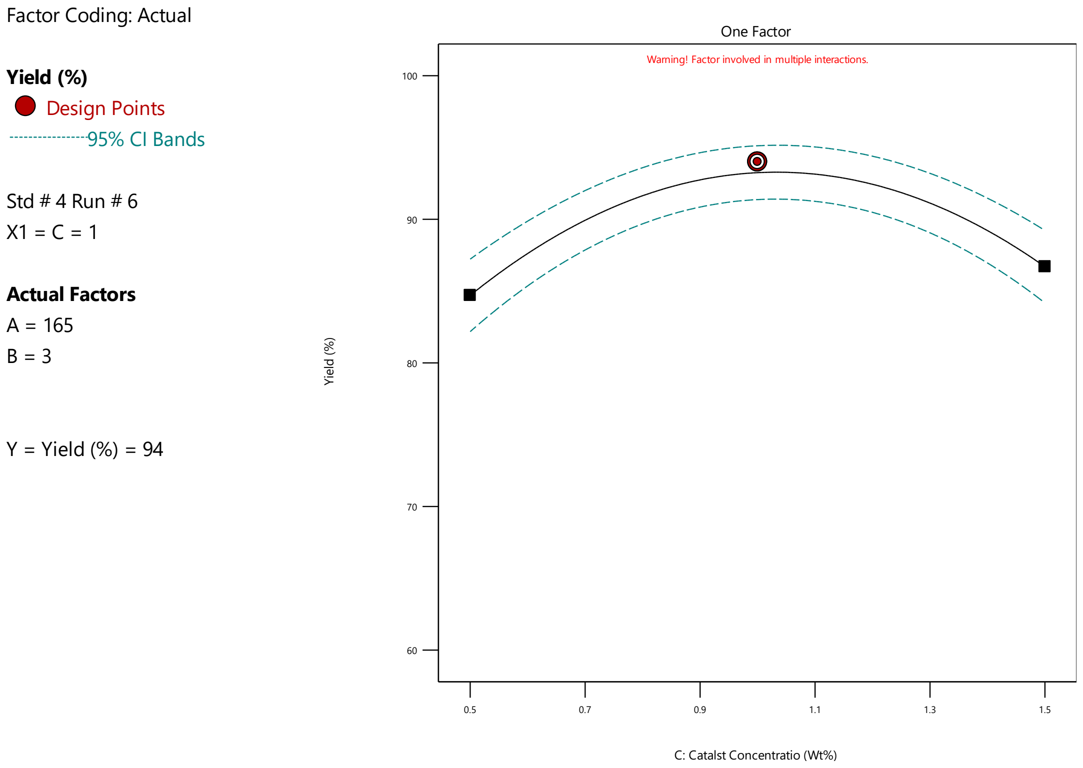


Figure 4.7 Effect of catalyst concentration

The above effects of temperature, catalyst concentration and ethylene glycol to biodiesel ratio on yield of of lubricant agreed with the previous report of J. M. Encinar *et al.*, 2020.

4.8 Interaction Effect of Process Variables on Food Grade Lubricant Yield on 3D Surface

The ANOVA reaction surface plot provides insights into the interaction between the ethylene glycol (EG) to biodiesel ratio, temperature, and catalyst concentration on Food Grade Lubricant (FGL) efficiency. When we observe the effect of increasing temperature from 145 ºC to 165 ºC, we can see a corresponding rise in efficiency, going from 65% to 94%. Additionally, as the ethylene glycol to biodiesel ratio increased from 1:1 to 3:1, FGL productivity showed an increase as well, ultimately reaching the peak efficiency of 94%. This graphical representation helps illustrate how changes in temperature and the EG to biodiesel ratio influence FGL efficiency. Figure 4.8 shows a very significant 3D plot of the interaction effect of the experimental variable on FGL productivity.

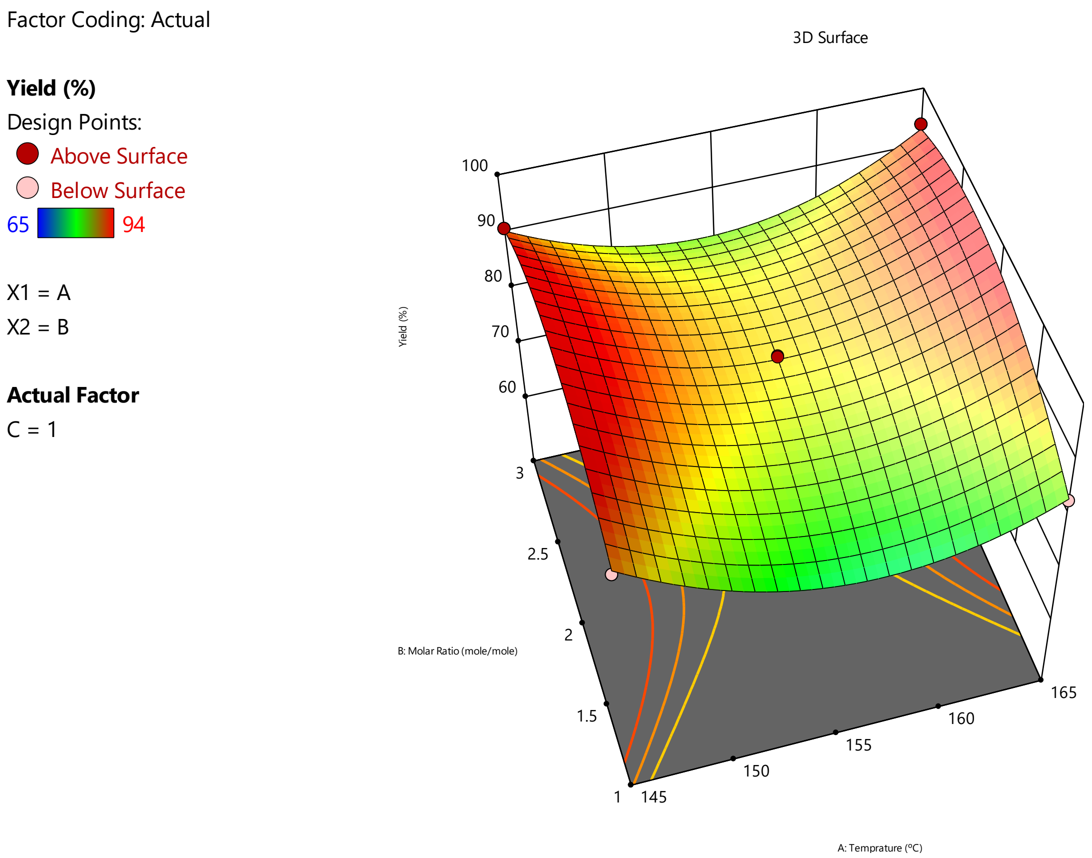


Figure 4.8 Response surface plot of effect of EG to biodiesel ratio and temperature at constant catalyst concentration.

The effect of the interaction between temperature and catalyst concentration at constant molar ratio on the performance of food grade lubricants was also analyzed by response surface (3D). As shown in figure 4.9 below, process variables affect the performance of food grade lubricants at higher values. This was because the increased catalyst concentration, the efficiency also increases up to medium catalytic value of 1%, and the high reaction rate at higher temperatures constituting a higher efficiency. By increasing the amount of catalyst from 0.5 to 1% with an increase in temperature from 145 ºC to 165 ºC, the efficiency was close to the optimum value. Furthermore, the performance decreases with excessive catalyst use and temperatures exceeded 165 ºC.

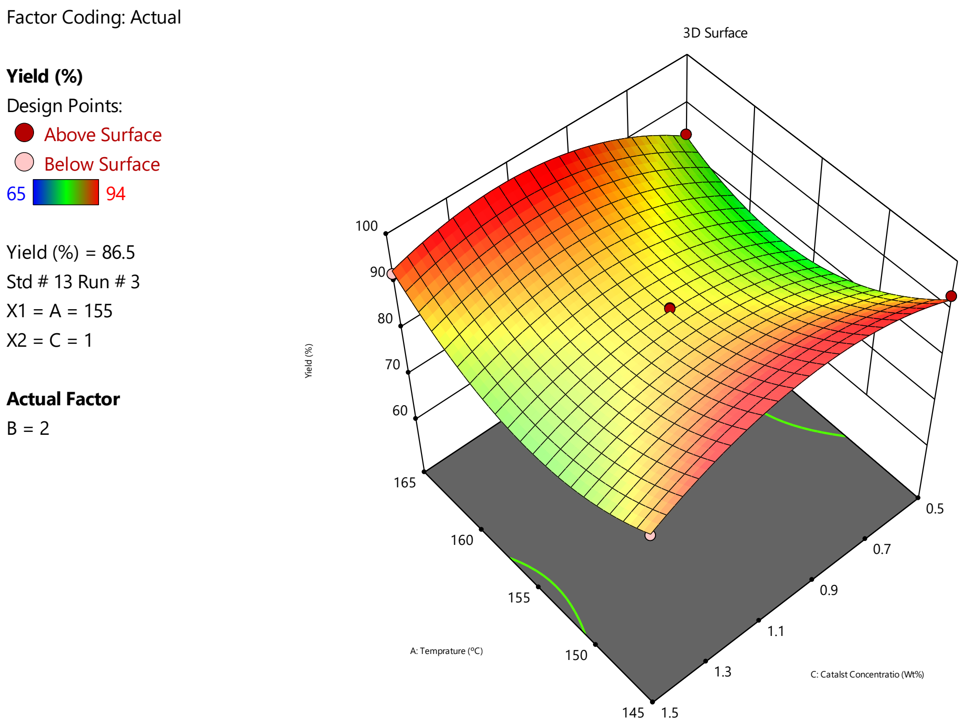


Figure 4.9 Response surface plot of effect of temperature and catalyst concentration at constant molar ratio.

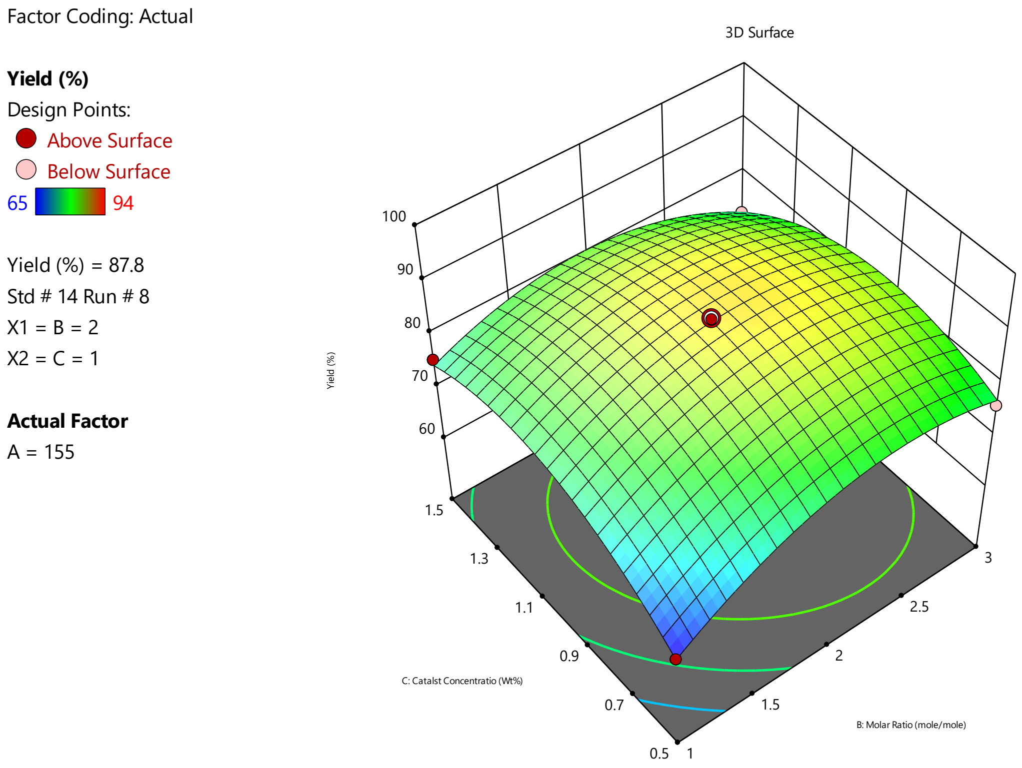


Figure 4.10 Effect of catalyst concentration and molar ratio at constant temperature

As can be seen in figure 4.10, we can observe the influence of catalyst concentration and molar ratio on the reaction surface, while maintaining a constant temperature. As the ethylene glycol (EG) to biodiesel ratio shifts from 1:1 to 3:1, there is a notable acceleration in the performance of the food grade lubricant with the increasing catalyst concentration. This performance increase is quite significant, and it reaches its highest point at a specific optimal value. However, beyond this optimal value, both above and below, the product performance starts to decline.

4.9 Optimization of Process Variables of Transesterification Reaction Using RSM

The aforementioned findings highlight the significant influence of three variables in the second transesterification process, as well as their interactions, on the performance of food grade lubricants. To optimize these variables within their specified ranges for maximum product conversion, we utilized Design Expert 13. The selected optimal configuration, as determined by the model, resulted in a food grade lubricant yield of 96%. This optimal configuration includes an ethylene glycol/biodiesel ratio of approximately 2.3, a temperature of around 165 ºC, and a catalyst concentration of approximately 0.9 wt. %, all of which fall within a desirability value of 1. These optimization results closely align with the outputs from the ANOVA analysis. The ANOVA results affirm that the transesterification process is indeed influenced by temperature, catalyst concentration, and the molar ratio of biodiesel to EG.

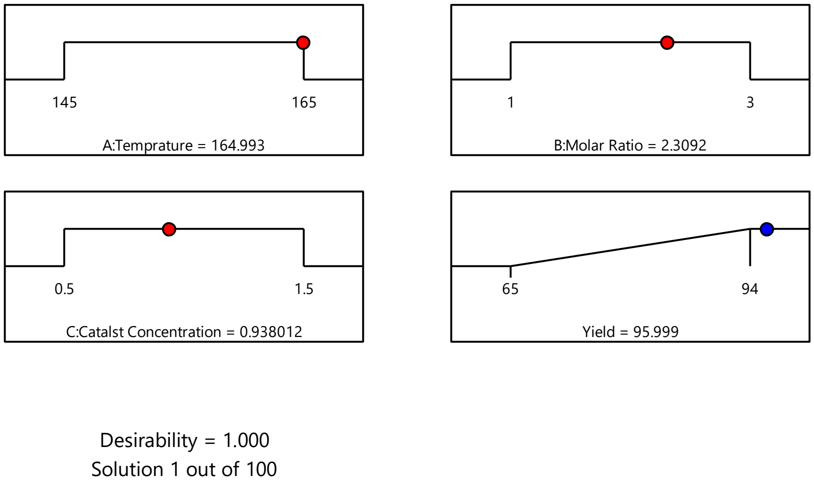


Figure 4.11 Optimization of transesterification reaction

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In this research work, we explored into the production and analysis of food-grade lubricants derived from rapeseed through a double transesterification conversion process. The pivotal factors impacting the effectiveness of this conversion reaction on lubrication performance include the catalyst concentration, the molar ratio of ethylene glycol to rapeseed biodiesel, and the temperature. To conduct our study, we employed the Behnken Box design method for statistical purposes and employed the response surface method for optimizing experimental parameters. The findings obtained from our experiments were assessed by examining various physicochemical properties.

Interestingly, this study yielded an impressive maximum efficiency of 94.0%, which was achieved under specific optimal conditions: a temperature of 165 ºC, a catalyst concentration of 1% by weight, and a molar ratio of ethylene glycol to rapeseed biodiesel at 3:1. These conditions represent the midpoint for temperature and catalyst concentration in relation to the optimal conditions that enhance the performance of food-grade lubricants. Further deviations in these parameters were found to diminish the performance of the food-grade lubricant, and concurrently, the yield of the process decreased. The response surface optimization method underscored the influence of all experimental parameters on lubrication performance.

It's noteworthy that the physicochemical properties of rapeseed oil, rapeseed biodiesel, and the resulting food-grade lubricants conform to the specifications outlined in ASTM and EN standards. In conclusion, our study indicates that rapeseed can serve as a viable alternative raw material for producing food-grade lubricants without compromising food safety. Rapeseed boasts a favorable oil content and can be harvested throughout the year, making it a promising candidate for sustainable lubricant production.

5.2 Recommendations

In Ethiopia, various industries rely on imported lubricants, while non-food-grade lubricants are commonly used in food processing facilities. To enhance the nation's industrial landscape and reap the associated benefits, the following improvements are recommended:

- **Exploration of Environmentally Friendly, Non-Toxic Lubricants:** It is essential to explore the production of food-grade lubricants using locally available, environmentally friendly, and non-toxic sources, such as rapeseed oil.
- **Transition to Food-Grade Lubricants in the Food Industry:** Instead of traditional lubricants, the food industry should adopt the use of food-grade lubricants. This change will ensure compliance with food safety standards and promote better hygiene in food processing.
- **Diversified Research on Food-Grade Lubricants:** Expanding research efforts to develop food-grade lubricants from various vegetable oils is crucial. This will offer a wider range of options and potentially improve the performance and sustainability of these lubricants.
- **Investigation into Agitation and Reaction Time Effects:** It is imperative to conduct research on how agitation and reaction time impact the performance of food-grade lubricants. Understanding these factors can lead to optimized lubrication processes in the food industry.

By implementing these recommendations, we can advance its industrial practices, promote local resource utilization, and enhance food safety standards in the food processing sector.

Reference

A.K. Yusuf,A Review of Methods Used for Seed Oil Extraction International Journal of Science and Research (IJSR),2018 7(12),233-238

Abdelaziz, A.I.M., Elamin, I.H.M., Gasmelseed, G.A. and Abdalla, B.K. (2014). Extraction, refining and characterization of Sudanese castor seed oil. Journal of Chemical Engineering, 2(1):1-4

Admasu Esubalew,2011, Biodiesel Production from Jatropha Oil Using Ethanol with Alkali Catalyst,17-19

Allied Market Research 2023, Food Grade Lubricants Market, Global Opportunity Analysis and Industry Forecast, 2021–2030, P 250

Al-Shehbaz, I.A.; Beilstein, M.A.; Kellogg, E.A. Systematics and phylogeny of the Brassicaceae (Cruciferae): An overview. Plant Syst. Evol. 2006, 259, 89–120.

Aluyor, E. O., Obahiagbon, K. O., Ori-jesu, M., Biodegradation of vegetable oils: A review. Sci. Res. Essay, 2009, 4, 543– 548.

B. Freedman, E.H. Pryde, T.L. Mounts. Variables affecting the yield of fatty esters from transesterified vegetable oils, Journal of American Oil Chemists Society 1984; 61(10):pp.1638–1643.

Bereket Tesfaye and Tilahu Tefera, (2017). Extraction of essential oil from Neem seed by using Soxhlet extraction methods. International Journal of Advanced Engineering, Management and Science, 2017, 3, 2454-1311.

Bertrand Matthäus (2012). Oil Technology, Federal Research Institute for Nutrition and Food, (Vol. 2)

Bilal S1, Mohammed-Dabo I. A1, Nuhu M1, Kasim, S. A2, Almustapha I. H1 and Yamusa Y. A2 Production of biolubricant from Jatropha curcas seed oil Journal of Chemical Engineering and Materials Science 2013 4(6), 72-79

Charles L. Peterson, Daryl L. Reece,Josephc. Thompson,Sidney M. Beck And Craigchases, Ethyl Ester Of Rapeseed Used As A Biodiesel Fuel-A Case Study, Biomass and Bioenergy 1996. 10(5/6), Pp. 331-336

Chika Muhammad, Zaliha Usman, Francis Agada Biodiesel Production from Ceiba pentandra Seed Oil Using CaO Derived from Snail Shell as Catalyst, Petroleum Science and Engineering 2018; 2(1): 7-16

Cmolik, J.; Pokorny, J.; Reblova, Z.; Svoboda, Z. Tocopherol retention in physically refined rapeseed oil as a function of deodorization temperature. Eur. J. Lipid Sci. Technol. 2008, 110, 754–759.

Debbie Sniderman Food grade lubricants and their regulation February 24, 2021

Donnet, C.; Erdemir, A. Historical developments and new trends in tribological and solid lubricant coatings. Surface and Coatings Technology, 2004, 180–181

Douglas Schufler,cleaning and storage of oil seed,Mechanical seed cleaning and holding Agriculture hand book,2023,354.

E. Tulsa 2023 Food Grade Lubricants,lubrication engineers

ELGI (2016) food grade lubricants supplement, New Food,19(1), www.newfoodmagazine.co

Encinar, J.M.; Nogales-Delgado, S.; Pinilla, A. Biolubricant Production through Double Transesterification: Reactor Design for the Implementation of a Biorefinery Based on Rapeseed. Processes 2021, 9, 1224. https:// doi.org/10.3390/pr9071224

F.J. Owuna, M.U. Dabai, M.A. Sokoto , S.M. Dangoggo, B.U. Bagudo, U.A. Birnin-Yauri a, L.G. Hassan, I. Sada, A.L. Abubakar , M.S. Jibrin ,Chemical modification of vegetable oils for the production of biolubricants using trimethylolpropane: A review, Egyptian Journal of Petroleum,2020,29(1),75-82

G. Anastopoulos, Y. Zannikou, S. Stournas, S. Kalligeros. Transesterification of vegetable oils with ethanol and characterization of the key fuel properties of ethyl esters, Energies 2009; 2:pp. 362-376.

G. Baskar, I. Abarnaebenezer Selvakumari, Advances in bio-oil extraction from nonedible oil seeds and algal biomass, Advances in Eco-Fuels for a Sustainable Environment, 2019

Gulden, R.H.;Warwick, S.I.; Thomas, A.G. The biology of Canadian weeds. Brassica napus L. and B. rapa L. Can. J. Plant Sci. 2008, 88, 951–996.

H.M. Mobarak, E. Niza Mohamad, H.H. Masjuki, M.A. Kalam, K.A.H. Al Mahmud, M. Habibullah, A.M. Ashrafu.The prospects of biolubricants as alternatives in automotive applications, Renewable and Sustainable Energy Reviews 33 (2014) 34–43

Hanny Johanes Berchmans and Shizuko Hirata, Biodiesel production from crude Jatropha curcas L. seed oil with a high content of free fatty acids, Bioresource Technology 99 (2008) 1716–1721

James Redfern,Malcoln Knninmonth,Dariel Burdass and Joanna Veran,Using soxlet Ethanol Extraction to produce and test plant material(essential oil) for their anti macribial properties,J Microbial Bio Educ,2014,15(1),45-46

Jitendra Kumar Chandrakar, Amit Suhane, The Prospects of Vegetable based Oils as Metal Working Fluids in Manufacturing Application –A Review International Journal of Engineering Research & Technology (IJERT) 2014, 3 (5)

José María Encinar,Sergio Nogales and Juan Félix González, Biodiesel and biolubricant production from different vegetable oils through transesterification, Engineering Reports. 2020; 2: e12190.

Juan Antonio Cecilia, Daniel Ballesteros Plata, Rosana Maria Alves Saboya , Francisco Murilo Tavares de Luna, Celio L. Cavalcante, Jr and Enrique Rodrígue Castellón, An Overview of the Biolubricant Production Process: Challenges and Future Perspectives process 2020 8,27

Ken Thayer The challenges of food-grade lubricants, food and beverage article november 19, 2018

Lv, M.;Wu,W. An advanced aqueous method of extracting rapeseed oil with high quality. J. Food Process Eng. 2018, 42, e12957.

Martin Williamson, Food-Grade Lubricants Explained, machinery lubrication Feb 2023

Matthew C. Menkiti1, Ocholi Ocheje Chinedu M. Agu Production of environmentally adapted lubricant basestock from jatropha curcas specie seed oil Int J Ind Chem (2017) 8:133–144

Meier, M. A. R., Metzger, J. O., Schubert, U. S., Plant oil renewable resources as green alternatives in polymer science. Chem. Soc. Rev. 2007, 36, 1788– 1802.

Momar Talla Dieng, Takumi Iwanaga, Yokoyama Christie Yurie and Shuichi Torii, Production and Characterization of Biodiesel from Rapeseed Oil through Optimization of Transesterification Reaction Conditions, Journal of Energy and Power Engineering 13 (2019) 380-391

Muhammad Arif Dandan, Wan Mohamad Aiman Wan Yahaya , Syahrullail Samion1, Mohamad Nor Musa, A Comprehensive Review on Palm Oil and the Challenges using Vegetable Oil as Lubricant Base-Stock Journal of Advanced Research in Fluid Mechanics and Thermal Sciences 2018, 52 (2) 182-197

Nadia Raboanatahiry, Huaixin Li, Longjiang Yu, Maoteng Li. Rapeseed (Brassica napus):Processing, Utilization, and Genetic Improvement. Agronomy, 2021, 11, 177622.

Nadia Salih and Jumat Salimon, A Review on New Trends, Challenges and Prospects of Ecofriendly Friendly Green Food-Grade Biolubricants, 2022,12(1),1185 - 1207

Nagendramma P, Kaul S. Development of eco-friendly/biodegradable lubricants: an overview. Renew Sustain Energy; 2012, 16:764 – 74.

Nde DB, Foncha AC, Optimization Methods for the extraction of vegetable oils:A Review Processes.2020;8(2):209.https://doi.org/10.3390/pr8020209

Nurhan Dunford,(2016), Oil and Oilseed Processing II, Ferguson College of AgAg Research FAPC-159

P. M. Ejikeme, I. D. Anyaogu , C. L. Ejikeme , N. P. Nwafor, C. A. C. Egbuonu, K. Ukogu And J. A. Ibemesi, Catalysis In Biodiesel Production By Transesterification Processes-An Insight, E-Journal Of Chemistry 2010, 7(4), 1120-1132

Rajeew Kumar(2009), BIODIESEL PRODUCTION PROCESS, G.B.Pant University of Agriculture & Technology, Pantnagar-Uttrakhand

Rajendra Uppar, P. Dinesha and Shiva Kumal (2022), A critical review on vegetable oil-based bio-lubricants: preparation, characterization, and challenges review, Environment, Development and Sustainability, https://doi.org/10.1007/s10668-022-02669-w

Raphaëlle Savoire ,Jean-Louis Lanoisellé & Eugène Vorobiev,(2012), Mechanical Continuous Oil Expression from Oilseeds: A Review, Food and Bioprocess Technology An International Journal,5(5)

Roiaini, M.; Ardiannie, T.; Norhayati, H. Physicochemical properties of canola oil, olive oil and palm olein blends. Int. Food Res. J. 2015, 22, 1228–1234.

Russell Publishing Ltd. Food grade lubricants – are they environmentally friendly and biodegradable? 2007,p 23

S Sensoz,D Angin and S Yorgun, Influence of particle size on the pyrolysis of rapeseed (Brassica napus L);Fuel properties of Bio oil, Biomass and Energy.2000,19(4)271-279

S. D. Romano and P. A. Sorichetti, Dielectric Spectroscopy in Biodiesel Production and Characterization, Green Energy and Technology, 2011,vol 8,p103

S. D. Romano and P. A. Sorichetti, Dielectric Spectroscopy in Biodiesel Production and Characterization, Green Energy and Technology,2011,VIII,103

Salimon, Jumat; Salih, Nadia; Yousif, Emad "Biolubricants: raw materials, chemical modifications and environmental benefits". European Journal of Lipid Science and Technology, 2010, 112 (5): 519–530.

Salma Samidin, Nadia Salih and Jumat Salimon Synthesis and Characterization of Trimethylolpropane Based Esters as Green Biolubricant Basestock, Biointerface research in applied chemistry Article,,2021, 11(5), 13638 – 13651

STATISTA. Worldwide Oilseed Production since 2008. Available online: https://www.statista.com/statistics/267271/worldwideoilseed-production-since-2008.

Takele Mitiku Abdeta, Genetic variability and heritability in Ethiopian mustard (Brassica carinata A.Braun), International Research Journal of Plant Science (2022), 13(1)

Ulf Schuchardta, Ricardo Serchelia, and Rogério Matheus Vargas, Transesterification of Vegetable Oils, J. Braz. Chem. Soc, 1998,9(1), 199-210

Willing A. Lubricants based on renewable resource: an environmentally compatible alternative to mineral oil products. Chemosphere, 2001, 43:89–98.

Wu, Y.; Zhou, R.S.; Wang, Z.G.; Wang, B.; Yang, Y.J.; Ju, X.R.; He, R. The effect of refining process on the physicochemical properties and micronutrients of rapeseed oils. PLoS ONE, 2019, 14, e0212879.

Appendices

Appendix A-1 Determination of Moisture Content

Time (h)	0	1	2	3	4
Weight (g)	26	24.801	24.436	24.345	24.342

Moisture content of seed $= \frac{1.658 \text{ gram}}{26 \text{ gram}} * 100 = 6.4\%$

Appendix; A-2 Determination of Density of Rapeseed Oil

Mass of Empty bottom flask =19.8763g

Mass of oil filed=43.0018g

Volume of flask=25ml

Density $= \frac{\text{mass of oil filled - mass of empty bottom flask}}{\text{Volume of flask}}$

$= \frac{43.0018g - 19.8763g}{25} = 0.925g/ml$

Appendix; A-3 Determination of Kinematic Viscosity of Oil

Kinematic viscosity of oil $= \frac{\text{dynamic viscosity}}{\text{Density of oil}}$

=30.088/0.925=32.528cst

Appendix; A-4 Determination of Saponification Value

Table 4.2 Saponification value of rapeseed oil

Run	Volume of HCl solution used in blank	Volume of HCl solution used in the test	Weight of oil taken for test (gram)	SV
1	20	8.2	2	165.495
2	20	8.6	2	159.885
3	20	8	2	168.3
Average				164.56

Appendix A-5 Determination of Acid Value

Table 4.3 Acid value of rapeseed oil

Run	Volume of KOH solution used in the titration	Weight of oil taken for test (gram)	AV
1	1	5	1.122
2	0.7	5	0.7854
3	0.8	5	0.8976
Average			0.935

Appidix B-1 Feed Material Requirement for Biodiesel Production

To determine the necessary quantities of ethanol and catalyst for each run, we calculated them based on the given process parameters, starting with a fixed amount of 40 ml of purified rapeseed oil. The calculation for the amount of ethanol needed at a molar ratio of 15:1, ethanol to oil, followed this formula:

$$\frac{\text{Density of solvent*volume of solvent}}{\text{molecular weight of solvent}} = \text{molar ratio of solvent [density of oil*volume of oil/molecular weight of oil]}$$

Substituting all requirement:

$$\frac{0.789\ \text{gm/ml*Ve}}{46\text{gm/mol}} = 15\ [0.918\text{gm/ml*40ml/869.1gm/mol}]$$

Solving for Ve =37.06ml ml of ethanol

Where; Ve = volume of ethanol

The amount of catalyst required for the experiment was calculated as;

Mass of oil =density of oil * Volume of oil

Mass of oil = 0.918 gm/ml *40 ml =36.72 gm

Mass of catalyst = 1% * mass of oil =1%*36.72gm= 0.37 gm of catalyst were used

Similarly, the amount of ethanol and catalyst is calculated for all experiments.

Appendix B-2; Experimental process conditions for the trasesterfication conversion reaction

Run	Temprature ºC	Molar Ratio mole/mole Rapeseed biodiesel/ Ethylene glycol	Catalst Concentration Wt%	Agitation speed, rpm	Reaction time,min
1	165	2	1.5	700	60
2	145	2	1.5	700	60
3	155	2	1	700	60
4	155	2	1	700	60
5	155	1	1.5	700	60
6	165	3	1	700	60
7	155	2	1	700	60
8	155	2	1	700	60
9	165	2	0.5	700	60
10	145	2	0.5	700	60
11	145	1	1	700	60
12	155	2	1	700	60
13	145	3	1	700	60
14	155	3	0.5	700	60
15	165	1	1	700	60
16	155	3	1.5	700	60
17	155	1	0.5	700	60

Appendix C: Feed stock and experimental set up picture

Rapeseed.

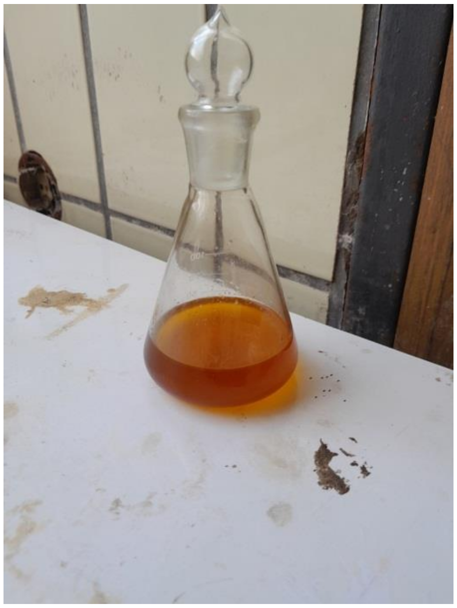
Oil extraction

separation of oil from n-hexane(Vibro evaporator)

Appendix D: Characterization method and product

Density Determination.

Acid value determination



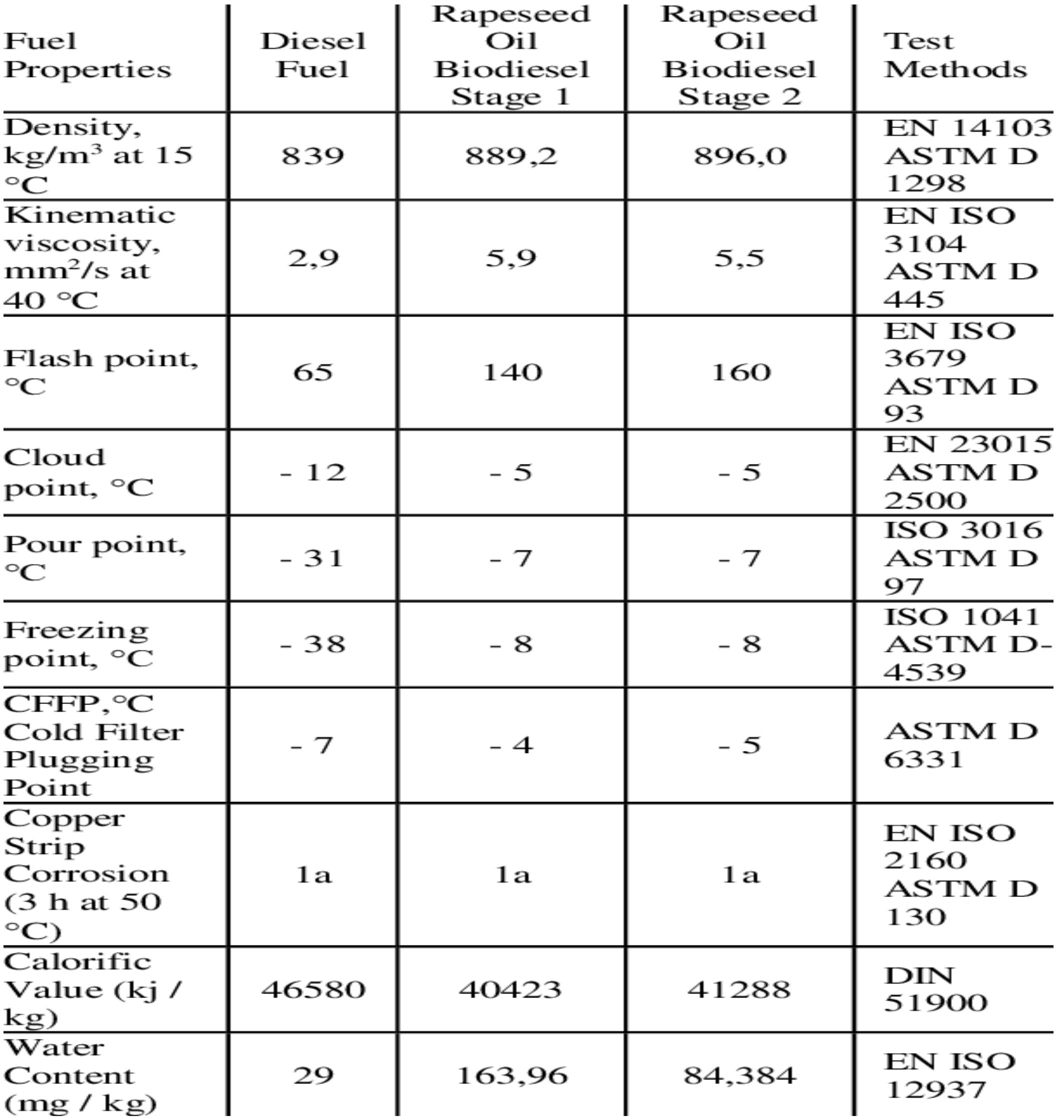
Product of rapeseed biodiesel.

Product of food-grade lubricants

Appendix E optimization experiment of food grade lubricant

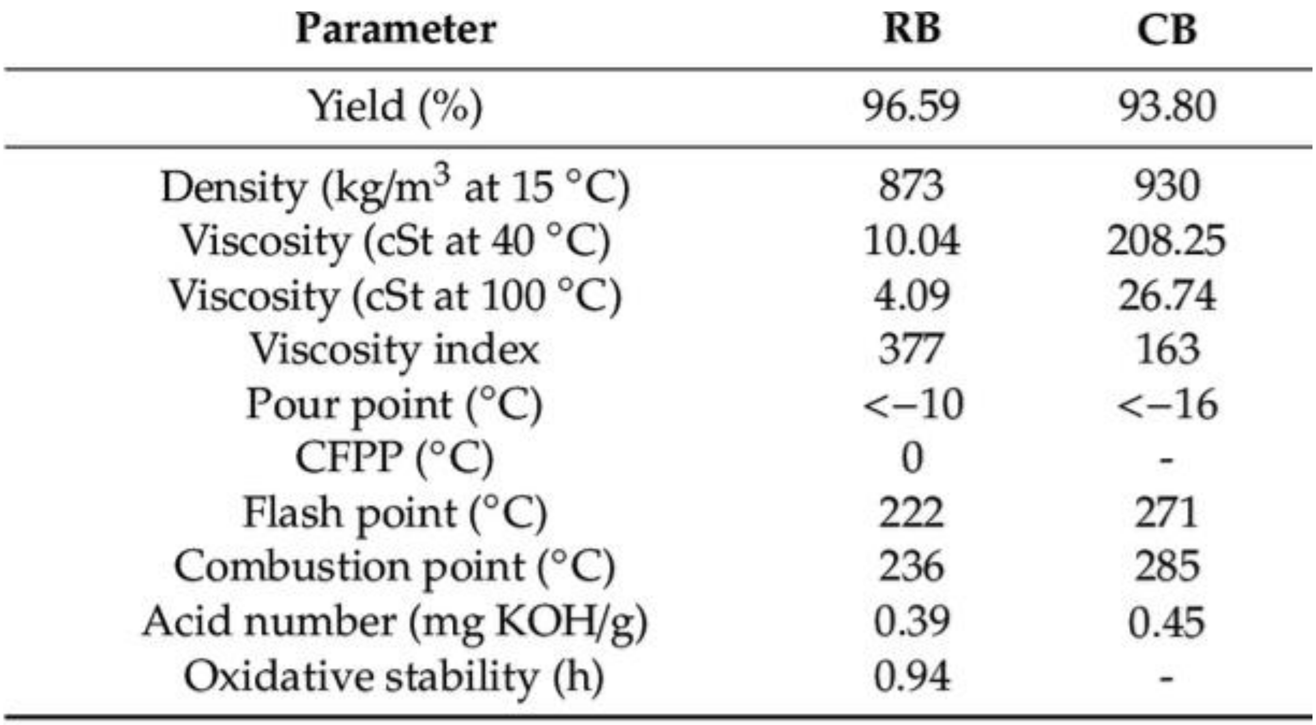
Trial	Temprature	Catalyst concentration	Molar ratio of EG/Biodiesel	yield
1	165	1	2.3	95.9
2	165	1	2.3	95.6
3	165	1	2.3	95.8

Appendix F Rapeseed Biodiesel standard



Fuel Properties	Diesel Fuel	Rapeseed Oil Biodiesel Stage 1	Rapeseed Oil Biodiesel Stage 2	Test Methods
Density, kg/m^3 at 15 °C	839	889,2	896,0	EN 14103 ASTM D 1298
Kinematic viscosity, mm^2/s at 40 °C	2,9	5,9	5,5	EN ISO 3104 ASTM D 445
Flash point, °C	65	140	160	EN ISO 3679 ASTM D 93
Cloud point, °C	- 12	- 5	- 5	EN 23015 ASTM D 2500
Pour point, °C	- 31	- 7	- 7	ISO 3016 ASTM D 97
Freezing point, °C	- 38	- 8	- 8	ISO 1041 ASTM D-4539
CFFP,°C Cold Filter Plugging Point	- 7	- 4	- 5	ASTM D 6331
Copper Strip Corrosion (3 h at 50 °C)	1a	1a	1a	EN ISO 2160 ASTM D 130
Calorific Value (kj / kg)	46580	40423	41288	DIN 51900
Water Content (mg / kg)	29	163,96	84,384	EN ISO 12937

Appendix G; Rapeseed lubricant standard [J.M.Encinar 2020]



Parameter	RB	CB
Yield (%)	96.59	93.80
Density (kg/m^3 at 15 °C)	873	930
Viscosity (cSt at 40 °C)	10.04	208.25
Viscosity (cSt at 100 °C)	4.09	26.74
Viscosity index	377	163
Pour point (°C)	<−10	<−16
CFPP (°C)	0	-
Flash point (°C)	222	271
Combustion point (°C)	236	285
Acid number (mg KOH/g)	0.39	0.45
Oxidative stability (h)	0.94	-

RB=Rapeseed bio lubricant

CB= Castor bio lubricant