



**COMPARISON FOR RAW AND COMMERCIAL CASTOR OIL IN THE
PRODUCTION OF BIODIESEL**

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PREFACE

The study was conducted under the supervision of Dr. Peterson Thokozani Ngema, Dr. Olusegun Ayodeji Olagunju, and Prof. Suresh Ramsuroop in the Department of Chemical Engineering, Faculty of Engineering and the Built Environment of the Durban University of Technology (DUT), South Africa. The testing and analysis of data took place in the Fuel Firing Services (FFS) Refiners laboratory in Jacobs, Teakwood road, in Durban and Department of Chemical Engineering postgraduate laboratory at Durban University of Technology.

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DECLARATION

I now declare that this dissertation is a record of my original research and work completed in order to get a Master of Engineering degree in Chemical Engineering from the Durban University of Technology (DUT). This does not contain any content that has been published elsewhere or by anybody else in exchange for a degree from Durban University of Technology. I hereby declare that no copyright has been violated by the content of this dissertation, as all works mentioned by others have been properly cited in the text and included in an extensive list of references.

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DEDICATION

This dissertation is dedicated to those who supported me.

My mother Thandi Irene Mvelase: A strong super woman who stand on my behalf. I love you more such that you are everything in this world. I will forever relish and follow your values. You are a strong women even though we no father but you stayed faithful strong woman when raising us.

I dedicate the work to Phumelele, Sbusiso, Khanyisile, Ntombifuthi, Jabulile, Ntombizonke, and Syanda as well as my closest friends, my extended family, and, most importantly, the Almighty God heavenly father who guide us with unlimited guidance, my lord and savior ancestors.

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DECLARATION – PUBLICATIONS

Details of contribution to Publications that that form part and/or include research presented in this thesis (include publications in preparation, submitted, in press and published and give details of the contributions of each author to the experimental work and writing of each publication).

Publication 1: Chemical Engineering Journal (Submitted manuscript)

Journal Title: The effect of reaction temperature, catalyst concentration and alcohol ratio, the production of biodiesel from raw and purified castor oil

Authors: Sandile Brendon Masango, Peterson Thokozani Ngema, Olusegun Ayodeji Olagunju, Suresh Ramsuroop

ABSTRACT

The development of industries is associated with higher pollution levels and higher fuel costs. The research on clean energy helps to lessen the reliance on fossil fuels, the ozone layer's depletion, and the release of hazardous pollutants. The development of renewable energy sources increases energy independence and lessens the harm that fossil fuels do to the environment in the Republic of South Africa (RSA), one of the African Nations. The main the challenge is the fragility of crude oil prices, high unemployment, worries about climate change, and the requirement for the continent's developing economies to use their resources sustainably are what motivate the establishment of a successful biodiesel (Fatty Acid Methyl Ester) business. Particularly in recent years, biodiesel has evolved into one of the most popular biofuels for fuel substitution with biodiesel.

In this study, homogeneous alkaline transesterification was used to produce methyl ester biodiesel (FAME) from refined or commercial castor oil (CCO) and raw castor oil (RCO) feedstock. The obtained results were compared to those evaluated, which produce better yield by varying essential parameters, which include reaction temperature, catalyst concentration, and alcohol: oil molar ratio at a constant period of 90 minutes. The effect of potassium hydroxide (KOH) as a catalyst between raw and refined castor oil was compared. The result revealed the performance of the KOH catalyst on raw castor oil yields 98.49% FAME, which was higher than the refined castor, oil which yield was 97.9% FAME. The optimal conditions was reaction temperature 45 °C, methanol:oil mole ratio 1:9, and catalyst concentration 0.625 % w/w, were obtained from refined castor oil were applied to raw castor oil because of the same properties. It was found that the yield of raw castor oil was 98.49% which is higher to that of refined castor oil which is 97.9%. General Rate Equation, Pseudo first order model, Second order model, and Modified-Second order model are the kinetics models that were evaluated and discussed. The kinetic models behavior is that the rate of reaction r increase with the increase in temperature, at the maximum reaction temperature of the study 343.15 K, 5.1×10^{-7} (K.mL/mol.min) General Rate Equation, 5.7×10^{-8} (K.mL/mol.min) Pseudo first order, 6.9×10^{-7} (K.mL/mol.min) Second order model, and 61.4×10^{-4} (K.mL/mol.min) Modified-Second order model. Pseudo first order model and Second order model were the best fitted kinetic models then PFO shows to be best kinetic model for the study by measuring absolute error 1.4×10^{-4} and relative error 0.999 was very low. The methodology adopted for the study

include fuel quality of castor oil (raw and commercial) and produced biodiesel were tested for physicochemical properties. Physicochemical properties and ASTM standard for raw castor oil :- kinematic viscosity 1.98 (cSt) @50 °C, ASTM D445 , Ash 0.03 (g), ASTM D874, Flash point 114 °C, ASTM D93, sulphur 0.64 ppm, ASTM D5453, density 920 kg/m³, ASTM D1298/0452, water content 0.05 %, ASTM D2709, and calorific value 44121kJ/kg, ASTM D6751, while for commercial castor oil:- kinematic viscosity 2.98 (cSt) @50 °C, ASTM D445, Ash 0.05 (g), ASTM D874, Flash point 116 °C ASTM D93, sulphur 0.65 ppm, ASTM D5453, density 960 kg/m³, ASTM D1298/0452, water content 0.04 %, ASTM D2709, and calorific value 44121kJ/kg ASTM D6751.

The study is also assessed for economic feasibility to determine if it will be profitable. The process design comprises the main processing unit, reactor, separator, mixing tank, centrifugal pumps and heat exchangers. In this study only, reactor was simulated using ASPEN Plus version 11 software utilizing the generated data from the laboratory. Economic feasibility was based mostly on the reactor and scaling up to plant scale. The cost of biodiesel production per year is R 205391.34/ year, sales cost is R 2464696.08/ year which gives a profit of R 1117077.48/ year which proves economic feasibility of the study.

It was concluded that raw castor oil can be suitable used in the process of producing biodiesel since it exhibit higher yield. It was recommended that one must use the generated data to up-scale the process and design other unit in details. Asides vegetable oils, castor seeds are among the identified raw materials from which raw castor oil can be extracted for biodiesel production because it has high oil content up to 60%.

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LIST OF ABBREVIATION AND ACRONYMS

| | |
|-------------------|--------------------------------------|
| AAD | Absolute Average Diviation |
| ASTM | American Standard of Testing Method |
| AV | Acid Value |
| BP | British Petroleum |
| CaO | Calcium Oxide |
| CCO | Commercial Castor Oil |
| CO | Carbon Monoxide |
| C.V | Coefficient of Variances |
| DAG | Diacylglycerol |
| DMC | Dimethyl Carbonate |
| DUT | Durban University of Technology |
| FAAE | Fatty Acid Alkyl Ester |
| FAME | Fatty Acid Methyl Ester |
| FFA | Free Fatty Acid |
| FFS | Fuel Firing Service |
| GC-MS | Gas Chromatography Mass Spectroscopy |
| GRE | General Rate Equation |
| GHG | Greenhouse gases |
| GL | Glycerol |
| KOCH ₃ | Potassium Methoxide |
| KOH | Potassium Hydroxide |
| MgO | Magnesium Oxide |
| MeOH | Methanol |

| | |
|-----------------|----------------------------------|
| MI | Mililiter |
| MSO | Modified-Second-Order |
| NaOH | Sodium Hydroxide |
| NO _x | Nitrogen compound |
| OFAT | One Factor-At-a-Time |
| PM | Particulate Matter |
| PFO | Pseudo-First Order |
| Rpm | Revolutions per minute |
| RSA | Republic South Africa |
| SANS | South African National Standards |
| SO | Second-Order |
| US | United State |
| VOs | Volatile |

CHAPTER 1: INTRODUCTION

1.1 Background

Petroleum fuels are crucial to the development of many industries, transportation systems, agriculture, and a range of basic human needs in contemporary civilization. Due to a sharp increase in demand, gasoline supplies are fewer every day (Keera, El Sabagh and Taman 2018). Furthermore, the use of mineral fuel has generated toxic gases, which have consequently resulted in many environmental issues and problems. Biofuels thus serve as an eco-friendly alternative fuel source. Products from burning the biofuels have reduced concentrations of carbon monoxide (CO), nitrogen compounds (NO_x), sulphur compounds (SO_x), and particulate matter (PM) (Ni, Wang and Li 2020).

According to the study by British Petroleum, South Africa is among the countries causing ozone layer damage (Kan, Chen and Chen 2019). Nearly half of Africa's CO₂ emissions are attributed to it. Around 1.6% of global emissions constitute CO, and the majority of the energy used in South Africa's economy is generated by fossil fuels. To reduce the problem, the alternative fuel extraction of biodiesel needs to be aggressively implemented (Kan, Chen and Chen 2019).

The need to substitute the raw material producing diesel with raw material that will produce biodiesel. The raw material which is extracted from renewable resources such as biomass for bioenergy, is of great importance to produce biodiesel (Ayodeji 2018; Afolalu *et al.* 2021). Producing biodiesel from renewable energy remains profitable after Rudolf Diesel's test of vegetable oil as an engine fuel (Niske and Wilson, 1965). If the policies set in place are implemented, it is anticipated that both production and demand for biodiesel will rise in the upcoming years (Ayodeji 2018). The South African government intends to approve the rules requiring mandated biodiesel blends in petroleum-based fuel (Afolalu *et al.* 2021).

More than 700 different industries use castor oil, including companies that make biodiesel, plastics, cosmetics, pharmaceuticals, and lubricants (Omowanle *et al.* 2018). Castor seed oil, which is used for producing biodiesel, has a high kinematic viscosity of 226 mm²/s, in contrast to other non-edible oils, which vary from 27 to 54 mm²/s. Poor fuel injection, poor atomization, and power loss were among the issues brought on by higher kinematic viscosity in biodiesel-

powered automobiles. In this situation, it can be mixed with less viscous oil in order to optimise the biodiesel's quality attributes within permitted limits (Mallah and Sahito 2020).

Castor oil is one of the non-edible oils that is most commonly used in the process of producing biodiesel (Chidambaranathan *et al.* 2020). Originally a tree or shrub, the castor plant comes in a number of different varieties that can be cultivated. The production yield of castor oil seed worldwide is over 1.3 million tons per year, although it may be possible to obtain above 1.3 million tons per year (Belaid *et al.* 2011; Mkhize 2023). India retains a dominant market share of about 60% in the global production industry, with China and Brazil following closely behind with roughly 20% and 10% shares, respectively. Some regions of Africa and the West Indies are also producers of small quantities of castor beans or castor plants. Less than 0.15% of global oil seed commercial activity is made up of castor oil (Belaid *et al.* 2011). Castor oil seeds typically contain 40–55% oil. Castor, hence, has one of the largest potential oil yields among plants (Keera, El Sabagh and Taman 2018). Edible vegetable oil has been the main raw material used for producing biodiesel across the world (Kayode and Hart 2019).

Since free glycerol affects the quality of biodiesel fuel and diesel engines, it must be removed from biodiesel. Increased emissions of aldehyde and acrolein, issues with fuel settling, deposits at the bottom of tanks, decantation, injector fouling, and troubles with storage are some of these negative impacts (Van Gerpen 2005; Gomes, Pereira and de Barros 2010; Veljković, Banković-Ilić and Stamenković 2015). The formation of soap is another component in the production of biodiesel. A higher soap level could damage the injectors, corrode diesel engines, block filters, and weaken engines (Atadashi *et al.* 2015). Biodiesel is cleaned using wet-dry washing methods. Wet washing yields high-quality biodiesel with physiochemical properties that meet the values prescribed by the American Specification Test Method for Diesel and European Standard (ASTM D7%) and EN14214 standards. The method is time- and energy-intensive, which must be emphasized. Additionally, it can mean longer processing times, higher costs, and less biodiesel production. According to Chew and Bhatia (2008); Kwiecien, Hájek and Skopal (2009); Olkiewicz *et al.* (2014), a method is necessary to dispose off an enormous quantity of wastewater. Wastewater disposal is a drawback of wastewater generation for every 100 liters of biodiesel (Ennaceri *et al.* 2022).

Wet washing methods have been developed to purify biodiesel. Similar to the wet washing method, dry washing produces high-quality biodiesel with good physiochemical properties, but its usage has been discouraged due to the absorbents. Therefore, the chemistry of the adsorbent is still in its rudimentary stages (Atadashi *et al.* 2015). Research on the application of double separating funnels for the purification of biodiesel is the next to be taken as a result of issues with wet and dry washing methods. The least amount of water will be needed for the purifying method. The separating process for biodiesel is more environmentally friendly because less waste water is generated (Chozhavendhan *et al.* 2020).

This study compares two feedstocks to produce biodiesel through experimental work namely raw and purified or refined castor oil, in varying parameters like reaction temperature, concentration of the catalyst, and methanol to oil mole ratio, on the amount of methyl ester produced. The optimum production conditions for biodiesel fuel will be investigated.

Therefore, seeing an alternative is crucial need to curb the environmental damage and the impact on the ozone layer. Castor oil could provide an alternative for biodiesel production. Moreover, both castor oil raw and purified could be explored in the production of biodiesel.

1.2 Problem statement

The use of liquid energy products in transportation, particularly fossil fuels, is considerably escalating the global energy crisis. The continued use of fossil fuels has increased carbon dioxide and other greenhouse gases (GHG) that affect the atmosphere and cause environmental deterioration. As carbon dioxide contributes to global warming, the majority of the petroleum industry is currently conducting research on how carbon dioxide can be reduced in the atmosphere as it has an effect on global warming.

Fuels are diminishing and experiencing progressive depletion every day as demand in the RSA grows rapidly. The main concern of scientists today is the global need for convectional energy and the depletion of fossil fuels. To support human activities like industrialization, transportation, and agriculture, a steady supply of energy is essential. There is a need to produce alternate, energy because the convectional energy sources are finite.

1.3 Hypothesis

The production of biodiesel can be carried out in one processing unit through the transesterification of castor oil using glass fabricated jacketed reactor, eliminating some steps or multiple processes.

1.4 General objective

This study aims at production of biodiesel from raw and refined castor oil using homogeneous alkaline transesterification method and compared yield obtained.

- To design the reactor suitable for the production using generated data.
- To investigate the effects of parameters such as catalyst concentration, reaction temperature, and reaction time on castor oil transesterification.
- To determine the kinetic of the process.
- To characterize the biodiesel produced from castor oil.
- To investigate the quality of the produced biodiesel by comparing physiochemical properties with Petro-diesel.

1.5 Approach

In this study, biodiesel would be produced using a glass jacketed reactor. The jacketed reactor plays a dual role in one single unit: reaction and heating of the product to avoid the process's considerable energy use. The study's experiment would be conducted in the lab of chemical engineering laboratory.

There would only be one stage to this study. Castor oil is first transesterified using potassium hydroxide as a catalyst and methanol as an alcohol. At present, the one factor-at-a-time (OFAT) method is being used.

1.6 Dissertation organization

This dissertation consists of seven chapters and it is organized as follows:

CHAPTER 1: Introduction

This chapter will highlight the problem statement, explain the study's background, expound on the research title, and delineate the objectives and criteria of the study.

CHAPTER 2: Literature Review

This chapter is a review of existing research and findings on this topic. It elaborates knowledge on the problem-solving strategies and shortcoming uncounted by other researchers

CHAPTER 3: Experimental Apparatus and Methodology

This section provides details on how the design of the reactor was conducted, including all dimensions of the reactor and details on how the reactor operates in the production of biodiesel. This equipment will be tested and compared to literature studies on how it is produced.

This section presents a description of the steps used to achieve the goals and objectives of the research. There are descriptions of the data collection and analysis process.

CHAPTER 4: Results and Discussion

A comprehensive description of the experimental data and their interpretation is provided in this section. To ensure validity and reliability, the experimental data were cross-checked against the model developed and the literature.

CHAPTER 5: Cost Analysis

This section presents a detailed description of the study experimental cost of all the equipment, raw materials, utilities, and labor. In this section, scale up for pilot-scale figures from lab-scale figures.

CHAPTER 6 Reactor Design and Simulation

This section provides a detailed base on designing a reactor using generated data and simulation of the reactor

CHAPTER 7: Conclusion and Recommendations

This chapter shows if the researcher has reached the goals established at the beginning of the study and provides an overview of the research findings in relation to the established objectives. The outcomes obtained and the challenges encountered, it offers guidance and recommendations for more research.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

Biodiesel is one of the potentially useful biofuels that is easily produced from vegetable oil. An appealing substitute for regular diesel is biodiesel. Biodiesel has been a strong substitute for regular diesel for many years. (Chauhan *et al.* 2016). Further, biofuels are biodegradable, producing combustion that reduces greenhouse gas emissions. The amount of biodiesel produced worldwide increased from 3.9 billion litres in 2005 to 18.1 billion litres in 2010, and it is anticipated that this amount will surpass 33 billion litres in 2016 and reach 41.4 billion litres in 2025, representing a 25% increase above 2016 levels. Currently (2022), the production has increased by 6% (15 billion litres) compared to 2021, which is 7 million more than forecast for 2021 (Grové 2018). Keep in mind that the most countries that produce biodiesel feedstock also have functioning domestic markets, and the majority or all of their supply goes toward meeting mandated local demand (Rouhany and Montgomery 2019). Figure 2.1 shows the percentage of biodiesel produced worldwide (Rouhany and Montgomery 2019).

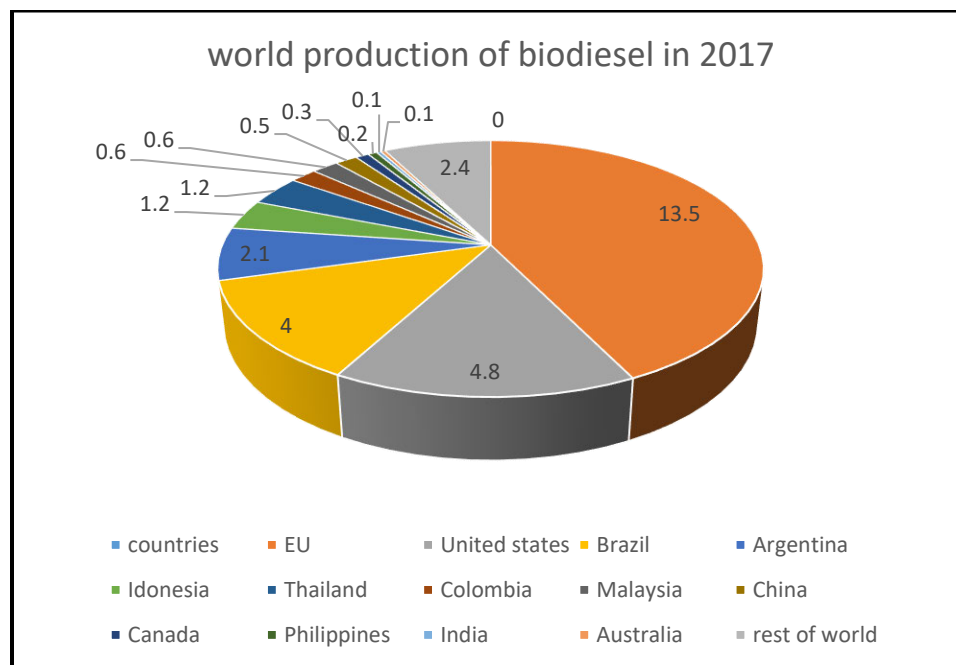


Figure 2.1: World's production of biodiesel in 2018 (Rouhany and Montgomery 2019).

This dual role as both a producer and a consumer can help to explain the limited international trade in biodiesel feed stocks (Proskurina *et al.* 2019). Argentina's exports to the United State are

anticipated to make up the majority of the small amount of biodiesel commerce during the next ten years. Many studies using lifecycle assessment (LCA) indicate that biodiesel emits 20–80% less greenhouse emissions than petro-diesel, even though its sustainability is still up for debate (Ali *et al.* 2022). The expanding market share of biodiesel and the production of second-generation biodiesel made from non-edible feed stocks will lead to a further decrease in emissions. This will negatively affect the climate by substituting biodiesel for petro-diesel as the extraction and refinement of crude oil become increasingly energy-intensive (Rouhany and Montgomery 2019). represents the percentage of feedstock around the world (Rouhany and Montgomery 2019).

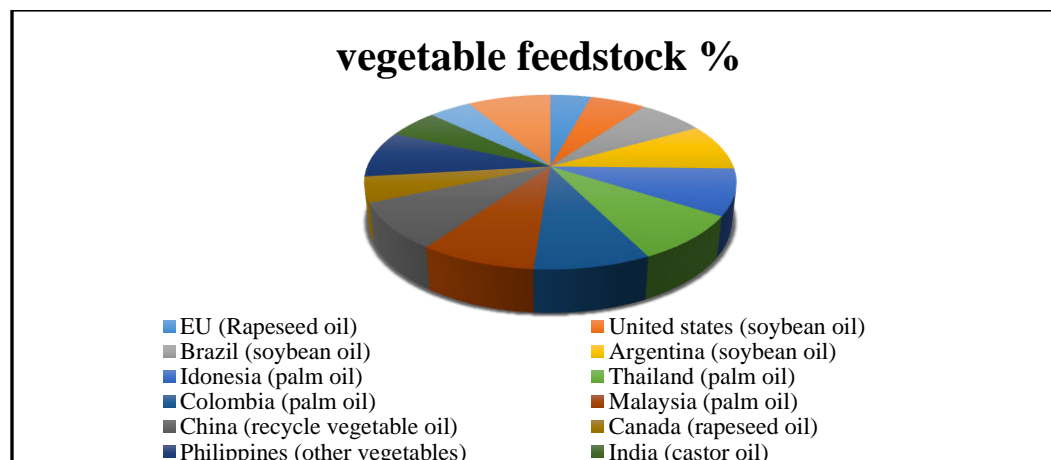


Figure 2.2: Global biodiesel feedstock in 2018 (Rouhany and Montgomery 2019)

It is now essential to find alternative renewable energy sources in order to achieve sustainable economic and environmental progress. Biodiesel has garnered a lot of interest among the numerous other substitute sources that have been found. It has many benefits, including the fact that it is clean and renewable. Furthermore, as Figure 2.2 presents, it is extensively accessible from many nations, which can significantly lower its production costs. (Ayodeji 2018).

In South Africa, biofuel has been used since sugar, ethanol, and gasoline were mixed in the 1920s (Mardhiah *et al.* 2017). The reason it became imported fossil fuels are less expensive, which rendered blending economically unviable, blending was stopped in the early 1960s. The ability of biofuels to ensure economic and energy security has reignited political and popular interest in these fuels. In response to the country's rekindled interest in the need for biofuels, the South African government launched the Biofuels Industrial Strategy in 2007.

The production of biofuels in South Africa is still in its infancy (Gupta, Malik and Bux 2017). Despite various governmental declarations and intentions over the years, and the nation has very few small-scale biofuel facilities (Gupta, Malik and Bux 2017; Zareh, Zare and Ghobadian 2017). Currently, 200 small-scale biodiesel production facilities exist, the majority of which use waste vegetable oil (WVO) as a feedstock (Topi 2020), and avoid posing a threat to agricultural land or food production (Bahadori *et al.* 2016). Mostly used for non-fuel applications (such as solvent, food and beverage, pharmaceuticals, etc.), small- and medium-scale ethanol production is exported to the EU market (Mardhiah *et al.* 2017). In 2011, it was estimated that South Africa produced only approximately 16 000 and 4770 liters of ethanol and biodiesel per day, respectively (Verdini *et al.* 2021). A few production facilities, though, have been found and granted for the manufacture of commercial biofuels in South Africa as shown (Pradhan and Mbohwa 2014).

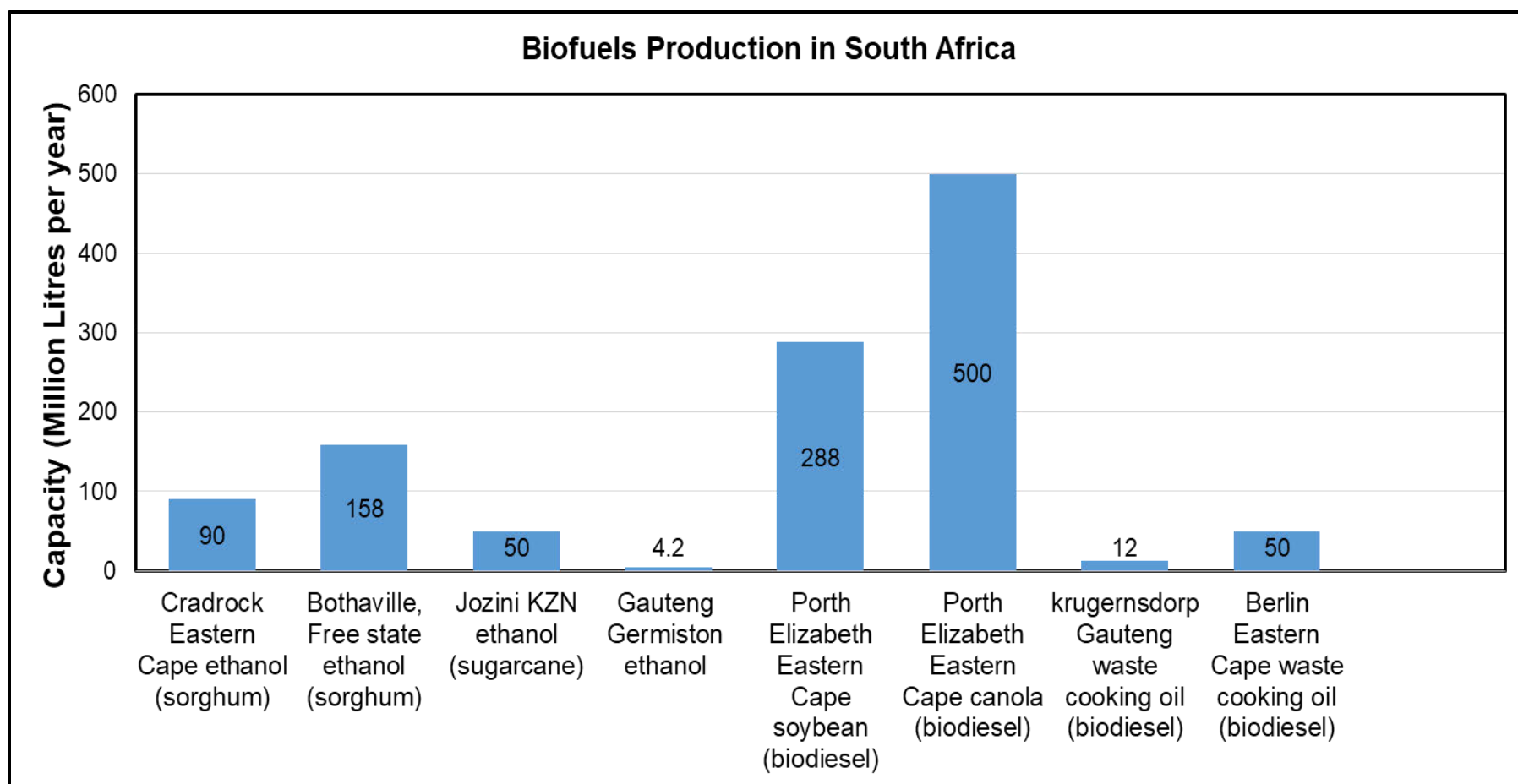


Figure 2.3 : Biofuels production data in Republic of South Africa (Pradhan and Mbohwa 2014)

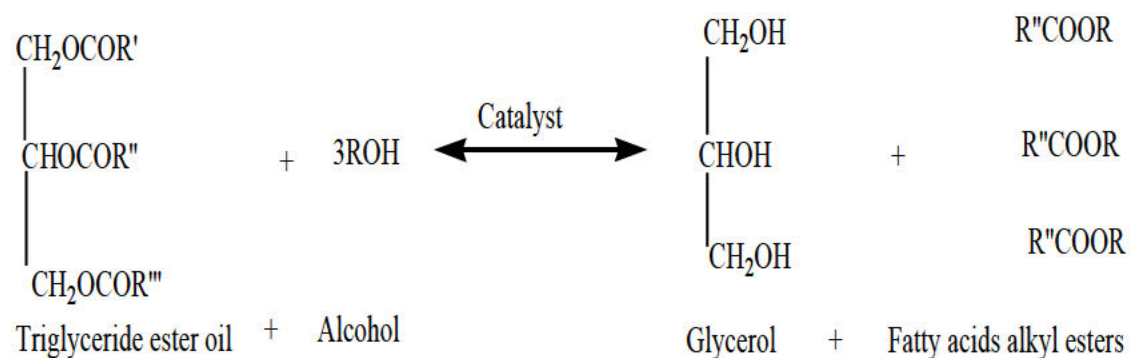
2.2 Brief history on biodiesel

Dr. Rudolph Diesel developed the diesel engine, which runs on fuels like heavy mineral oils and vegetable oils. Although the initial engine experiment proved unsuccessful, Dr. Rudolf Diesel was able to overcome this obstacle after multiple attempts by 1900. In 1911, Dr. Rudolf Diesel designed an engine that ran exclusively on peanut oil, which was displayed at the World Exhibition in Paris. "The diesel engine can be fed with vegetable oils and would help considerably in the development of agriculture in the countries that use it," said Dr. Diesel in a deep statement. The use of vegetable oil for engine fuels may appear unimportant today, but over time, these oils may become just as significant as petroleum and the coal tar products of the present day, according to Dr. Diesel in 1912. Dr. Rudolf Diesel (1900) developed the invention of diesel engine running with heavy mineral oil or vegetable oil." However, the principles of clean, renewable, locally produced fuel were established by Dr. Rudolf Diesel's innovations and agricultural theories (Boudh *et al.* 2020).Vegetables were used as an alternative to diesel during emergencies in the 1930s and 1940 (Veljković *et al.* 2021). In addition to other fossil fuels, vegetable and animal fats for the production of biodiesel have received increased attention because of the volatility of crude oil prices and environmental issues. The persistent dependence on crude oil as a primary energy source has intensified regional air pollution and amplified the effects of global warming. The release of a poisonous substance into the enclosed space of underground mines is one example of the issues brought on by carbon emissions. However, there is a chance that biodiesel will release a significant amount of air pollutants and toxins into the atmosphere (Ayodeji 2018) .

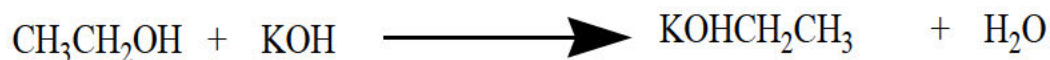
EN 14214 and ASTM D 6751 are two widely used biodiesel standards throughout the world (Oloyede *et al.* 2023). According to the South African Bureau of Standards (2006), biodiesel in South Africa is limited to fatty acid methyl ester (FAME) produced solely from vegetable oils by (The South African Bureau of Standards, 2006 SABS) SANS 1935 automotive standard. This suggests that South Africa is capable of producing crude vegetable oil from oil seeds or the biodiesel that results from them, known as FAME. (Aga, Fantaye and Jabasingh 2020).

2.3 Chemistry of biodiesel

According to its description, biodiesel is a fuel for diesel engines made by chemically converting vegetable and animal oils (Veljković *et al.* 2021). The sole reaction products illustrated in Reaction 1 are the matching fatty acid alkyl ester (FAAE) and glycerol (GL), which are formed when a low-molecular-weight alcohol interacts with triglyceride esters of oil or fat under typical conditions. In addition, alcohol (ethanol) reacts with potassium hydroxide (KOH) and sodium hydroxide (NaOH) in the presence of catalysts to form potassium ethoxide and sodium ethoxide, as shown in the reaction 2 (Galadima and Garba 2009)

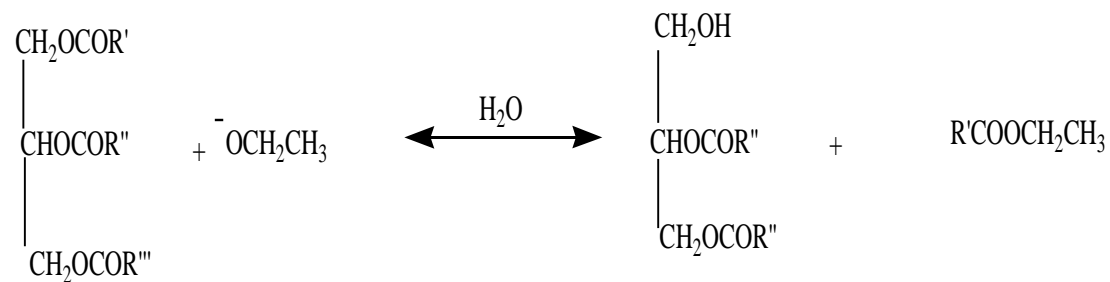


Equation 2.1: Reaction of triglyceride with alcohol to form fatty acids alkyl ester (FAAE) and glycerol (GL)

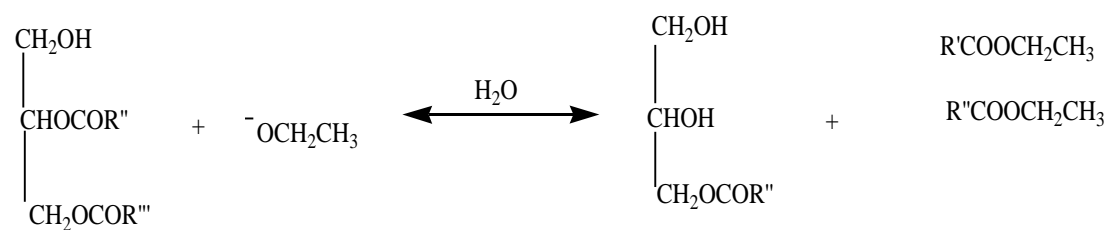


Equation 2.2: the reaction between KOH and NaOH with ethanol production potassium ethoxide and sodium ethoxide (Rocha-Meneses *et al.* 2023)

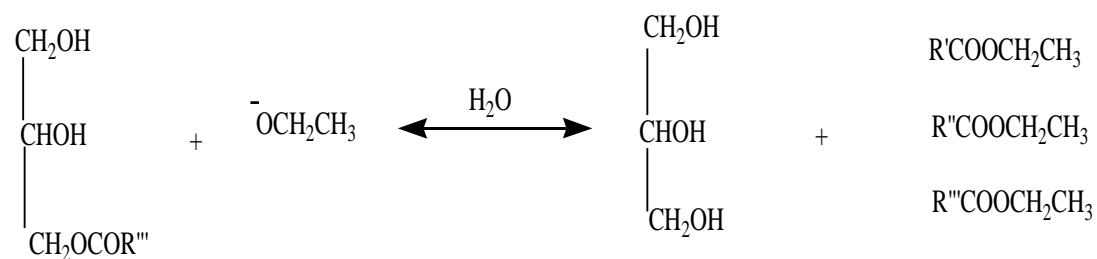
According to Rashid and Hazmi (2022), chromatographic studies are latter reaction involves monoglyceride esters (reactions 4 and 5), which in turn leads to the desired reaction products (reaction 6).



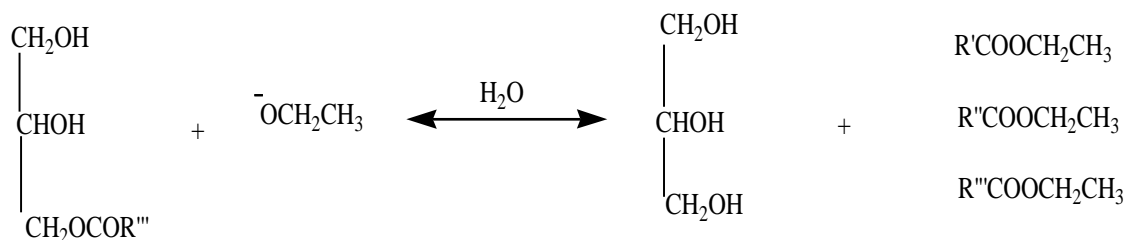
Equation 2.3:



Equation 2.4:



Equation 2.5:



Equation 2. 6:

Equation 2.3, 2.4, 2.5, 2.6 : Desired reaction product (equation 6) from sequential conversion of the triglyceride esters to diglyceride esters and monoglyceride esters (reaction 4 and 5) (Srivastava and Prasad 2000; Rashid and Hazmi 2022)

There is plenty of experimental data demonstrating that liquid KOH and NaOH catalysts promote quicker reaction because the catalyst particles are nearly equally available to the reactant molecules. Although the aforementioned liquid catalysts enhance the production process, they are also linked to separation issues, raising the cost of producing industrial biodiesel. Therefore, switching to a heterogeneous catalyst may have benefits including simple separation, non-corrosiveness, reusability, recycling, and reduced production expenses. Certain research has revealed that KOH pellets exhibit potential properties for extending the catalyst's life. (Keera, El Sabagh and Taman 2018). Alternatively, CaO, together with activated carbon, was efficient in biodiesel production according to Ayodeji (2018) and successfully produced 96.9 % biodiesel (FAME).

Keera, El Sabagh and Taman (2018) mentioned that after 10 cycles of repeated administrations, KOH remained active in the studies, while K_2CO_3 did not. Similar to this, adding suitable support materials, such as activated carbon, could increase the activity of a pure catalyst. Santoso, Sukarianingsih and Sari (2018) reported 92% conversion over a calcium oxide/magnesium oxide (CaO/MgO) catalyst system at 64.50 °C during the methanolysis of rapeseed oil. This is less than the 95.635% conversion that the study found when aluminium oxide (Al_2O_3) is used as a substitute for magnesium oxide (MgO). So, we can say that at a loading of 5 weight percent CaO and with

very little help from the acidic alumina support, calcium oxide (CaO) was spread out very well over Al_2O_3 . At the conclusion of the reaction, NaOH yields two different fatty acid methyl ester (FAME) and glycerol layers, in contrast to the homogenous technique. The extra-solid CaO/ Al_2O_3 layer was produced using a heterogeneous system that took into consideration physical loss or district-induced non-catalyst degradation. (Galadima and Garba 2009; Mohamed, Gülten and Mustafa 2023).

2.4 Use of biodiesel

Without being blended with conventional petrol or diesel, biodiesel can be used as pure diesel. It is safe and environmentally friendly in its pure state. This type of diesel has no associated exhaust pollutants, such as CO_2 . On the other hand, pure biodiesel does not negatively impact engine performance; nonetheless, its properties may cause engine deposits to clog the filter and deteriorate the fuel line. (Amran, Bello and Ruslan 2022).

An alternative way to overcome such problems encountered with pure biodiesel is to blend it with petro-diesel. Blending with petro-diesel helps overcome problems encountered with pure biodiesel. The blending model is usually denoted or presented with blending percentages like B10, B20, and B30. The blending model implies the percentage of biodiesel content mixed with 10%, 20%, 30%, etc. biodiesel, depending on the preferred blending. The advantages of blending are that it reduces engine problems, emissions, and costs. The ideal blend model, known as B20 in contemporary scientific investigations, is 20 biodiesel and 80 petro-diesel, according to several studies. The existing laws in South Africa limit the amount of biodiesel blend to 5% (Altarazi *et al.* 2022). Biodiesel is an effective additive that effectively increases lubrication and toxicity. The tiny amount of biodiesel utilized is sufficient to convert the product's low lubricity into useful fuel. An alternative fuel with low sulphur and aromatic content, a liquid nature, portability, fast availability, renewability, and a higher combustion efficiency.

2.4.1 Advantages of biodiesel

- It is renewable in nature.
- It has a better lubricating efficiency compared to petro-diesel.
- Modification of engine is not required.
- Environmentally friendly and reduced CO_2 emission.
- Less cost of production and derived from vegetables.

- Biodiesel is plant based considering carbon neutral.
- It an alternative fuel source for transportation industry.

2.4.2 Disadvantages of biodiesel

- High viscosity leads to filter blockage.
- Compared to petro-diesel, it has a greater cold-filtration plugging point temperature.
- Volumetric energy density is low.

2.5 Castor oil: Its composition and physicochemical properties

One of the most promising sources of feedstock for biodiesel production is castor oil, which has its own advantages. However, castor oil lacks sulphur, even if it is viscous compared to other vegetable oils. A greater cetane number in castor oil suggests a higher quality of ignition. Moreover, its higher oxygen content encourages more thorough combustion. (Aguado-Deblas *et al.* 2020). The composition of castor oil is shown in oil figure 2.1, shows that castor oil has better lubrication than other oils with comparable carbon chain lengths of fatty acids(Keera, El Sabagh and Taman 2018). Among all fats and oils, castor oil is distinct:

- A renewable resource, biodegradable, and nontoxic.
- The source of a hydroxylated fatty acid with eighteen carbons and one double bond.
- Ninety% of the fatty acid composition is composed of ricinoleic acid (12-hydroxyleic acid).
- Naturally occurring materials, the consistency and uniformity of the product are comparatively high.

Table 2.1: Castor oil composition (Pinzi et al. 2018)

| Fatty acid oil composition | Percentage |
|----------------------------|------------|
| Ricinoleic acid | 89.5 |
| Linoleic acid | 4.2 |
| Oleic acid | 3.0 |
| Stearic acid | 1.0- |
| Palmitic acid | 1.0 |
| Dihydroxystearic acid | 0.7 |
| Linolenic acid | 0.3 |
| Eicosanic acid | 0.3 |

Castor oil performs better as a lubricity enhancer to the hydroxylated fatty acids of ricinoleic acid than other typical vegetable oil esters (Pinzi *et al.* 2018). Castor biodiesel is the ideal fuel for cold weather since it has a very low precipitation and pour point in comparison to other biofuels. Castor oil has a precipitation point and pour point of 23 °C and 45 °C, respectively (Mallah and Sahito 2020). Castor oil stands out for having a high amount of ricinoleic acid. No other vegetable contains as much fatty acid as castor oil. Castor oil is not good for consumption of human beings can cause healthy implications, but this makes good for production of biodiesel. Castor oil transesterification utilizing various homogenous and heterogeneous catalyst types has drawn the attention of numerous researchers (Ismail *et al.* 2014; Keera, El Sabagh and Taman 2018); Pinzi *et al.* (2018); (Sánchez *et al.* 2019; Osorio-González *et al.* 2020).

The broad range of reactions that castor oil offers the electrochemical industry and the distinctive chemicals that can be derived from it make its chemical structure extremely interesting. Because these derivatives come from renewable resources and are biodegradable and environmentally friendly, they are far superior to petrochemicals (Altalhi 2022). Concerns have been raised recently regarding the use of castor oil as a feedstock in the production of biodiesel. The molecular mass of castor oil is 298 g/m, the melting point is only 5 degrees Celsius, and the solidification point is between 12 and 18 degrees Celsius. Because of its low solidification point, it is useful for industrial applications. However, the most important aspect is that it has the highest and most stable viscosity of any vegetable oil (Borowicz *et al.* 2023)

2.6 Biodiesel feedstock

When producing biodiesel, it is essential to take into account the environmental and financial advantages that come with the production of biodiesel as well as its utilization as a biofuel. Because of the high cost of feedstock, the production of biodiesel is expensive. It is a significant benefit to produce biodiesel in the same regions where feedstock is readily available (Osorio-González *et al.* 2020). This practice helps to reduce the costs associated with transportation.

Raw vegetable oils and cooking oils serve as the primary feedstock for the production of biodiesel. Additionally, various animal greases are also utilized in the process. A wide variety of vegetable oils, including rapeseed, soybean, cotton, peanut, corn, olive, sesame, safflower, sunflower, and castor oil, can be utilized in the production of biodiesel (Athar and Zaidi 2020). Trophic oily crops that contain high-quality oil that can be transesterified include *R. communis* (castor seeds), *Raphanus sativus* (radish), *Cyperus esculentus* (tiger nut), *Simmondsia chinensis* (jojoba), *Jatropha curcas* (physic nut), *Gratissima persea* (avocado), *Lupinus albus* (white lupin), the palm species *Acrocomia aculeate*, *Maruritia flexuosa*, *Elaeis loeifera*, *Syagrus coronate*, *Attalea Speciose*, and *Caryocar brasiliense* (peui) (Osorio-González *et al.* 2020). Vegetable oils extracted from energy and non-energy crops, such as soybeans, rapeseed, castor oil, sunflower, coconut, and palm oil, are the most common type of feedstock. These oils are grown all over the world. Some of the raw materials that are utilized in the production of biodiesel are presented in Table 2.1 (Osorio-González *et al.* 2020).

Table 2.1: Examples of oil feedstock used in biodiesel production

| Feedstock source (Oil content %) | Characteristics | Advantages | Disadvantages | Reference |
|-------------------------------------|--|---|---|---|
| Jatropha | <p>After extraction (fresh), the liquid is colourless and pale yellow after standing for some time</p> <p>Fresh has a pale light colour and a dark storage liquid at room temperature.</p> <p>Liquid at room temperature. Light yellow colour, and slightly pungent</p> <p>It depends on the cooking process; it can vary from yellow to dark brown and be liquid at room temperature.</p> <p>Purified as clear and vaguely yellowish-brown color liquid at room temperature</p> <p>Purified as a clear and vaguely yellowish-brown liquid at room temperature</p> <p>Semi-soil at room temperature, reddish or clear colour, depending on extraction source (pulp or kernel).</p> | Produced biodiesel keeps its quality when stored. Non-edible. Low viscosity, good oxidation stability, and high octane number | The generation of hazardous solid waste. High acidity. | (Datta and Mandal 2014; Chuah <i>et al.</i> 2021) |
| Soybean | Fresh has a pale light colour and a dark storage liquid at room temperature. | The yield of 98% biodiesel in purified oils High thermal | Cost-effective in production. Biodiesel production over the | (Chuah <i>et al.</i> 2021) |

| | | | | |
|------------------|--|---|--|---|
| | | stability.(Chuah et al. 2021) Low viscosity | long term is unsustainable. Edible. High acid value | |
| Castor bean | Liquid at room temperature. Light yellow colour, and slightly pungent | Transesterification can be perfumed at room temperature. Soluble in alcohol. Non-edible. Low acid value. High flash point | Generation of toxic solid waste. High viscosity. Decrease fuel atomization. | (Chuah <i>et al.</i> 2021) |
| Used cooking oil | It depends on the cooking process; it can vary from yellow to dark brown and be liquid at room temperature. | Environmental friendly. Cheap feedstock. Non-edible. High thermal stability | High ratio of acid esterification. High ratio of saponification. High acid value | (Pinzi <i>et al.</i> 2018; Veljković <i>et al.</i> 2021) |
| Sunflower | Purified as clear and vaguely yellowish-brown color liquid at room temperature Purified as a clear and vaguely yellowish-brown liquid at room temperature | Low viscosity | Used to produce food and fiber. Biodiesel production in long term is unstable. Edible. High acid value | (Pinzi <i>et al.</i> 2014; Veljković <i>et al.</i> 2021) |
| Palm | Semi-soil at room temperature, reddish or clear colour, depending on extraction source (pulp or kernel). | 96% yield of biodiesel in purified oils. Cheap feedstock. Good oxidation stability acceptable ratio of saponification High flash point. Low cloud point | High cloud point. Production of biodiesel may not be sustainable in the long term. edible | (Pinzi <i>et al.</i> 2014; Lam, Jamalluddin and Lee 2019) |

Crop adaptability to local conditions (precipitation, soil latitude, temperature, etc.) is preferred in oily raw materials used to produce biodiesel. The cultivation of high oil content, suitable composition, high infrastructure adaptability, and easy access to supplies for agriculture like pesticides, fertilizers, and water is the advantage for biofuel feedstock. The possibility of obtaining crops from marginal lands and commercializing the various agricultural co-products produced, meets the requirements of social sustainability (Aguirre *et al.* 2014). *Ricinus communis* possesses most of the desirable qualities to make a valuable feedstock for the production of biodiesel; however, its high oil viscosity may limit its application. Free fatty acids make up only a small portion of triglycerides. Table 2.2 displays the most common acids found in vegetable oils, which are linoleic, oleic, and palmitic acids (Athar and Zaidi 2020). Researchers have previously noted that the fatty acid content of castor oil is less variable than that of other vegetable oils when compared to other species. *Ricinus communis* may also contain trace amounts of stearic, linoleic, oleic, and linolenic acids, depending on where it comes from (Athar and Zaidi 2020).

Table 2.2: Main fatty acids in vegetables oil used as feedstock in biodiesel production (Singh, Srivastava and Prakash 2023).

| Oil | Fatty acid composition (wt%) | | | | | | | | Reference |
|--------------|------------------------------|-------|-------|----------------|-------|-------|-------|--------|---|
| | C14:0 | C16:0 | C18:0 | C18:1 cis-9 | C18:1 | C18:2 | C18:3 | Others | |
| Carbon atoms | | | | | | | | | (Mutlu and Meier 2010; Sanghamitra, Oramas and Prasad 2014) |
| Castor | - | 1.1 | 1.0 | 3.3 | 87.7 | 4.7 | 0.7 | 1.5 | (Sanghamitra, Oramas and Prasad 2014; Adeyemi, Olutona and Akintayo 2023) |

| | | | | | | | | | |
|-----------|------|-------|------|-------|---|-------|------|------|--|
| Jatropha | - | 12.80 | 6.20 | 39.94 | - | 45.40 | - | <1.0 | (Rajkumari and Rokhum 2020) |
| Soybean | - | 11.46 | 3.08 | 23.30 | - | 53.32 | 0.31 | 8.53 | (Rial <i>et al.</i> 2020) |
| Sunflower | 0.08 | 8.03 | 3.26 | 29.27 | - | 59.32 | - | 0.04 | (Veljković <i>et al.</i> 2021) |
| Pal | 0 | 46.8 | 3.80 | 37.60 | - | 10.50 | - | 1.3 | (Lam, Jamalluddin and Lee 2019; Fathurrahman <i>et al.</i> 2023) |
| Canola | - | 3.90 | 1.10 | 64.40 | - | 20.4 | 3.96 | 0.6 | (Kai <i>et al.</i> 2014; Rahmani and Goli 2023) |

Different vegetable oils are the promising source for the feedstock used to produce biodiesel. Nonetheless, the vast majority of them are restricted to food-related uses and can only be utilized for food-related purposes. Therefore, castor oil offers a number of benefits over edible oils in the production of biofuel. Because of its non-edible oil and main fatty acid (ricinoleic acid), which are composed of hydroxyl groups that are more soluble in alcohol, it offers a considerable benefit to obtain methyl esters at low temperatures alcohol (Athar and Zaidi 2020; Osorio-González *et al.* 2020).

2.7 Biodiesel production process

There have been significant attempts made to develop and enhance the properties of vegetable oil to reach as close to the properties of biodiesel fuels as possible. The most prevalent problems associated with crude vegetable oils are their low volatility, high viscosity, and polyunsaturated characteristics. Table 2.4 presents four different types methods for production of biodiesel namely, pyrolysis, micro-emulsion, esterification, and transesterification which can be used to overcome these difficulties (Nayab *et al.* 2022).

Table 2.3: Different type of biodiesel production process

| Production process | Description | Advantages | Disadvantages | References |
|--------------------|---|---|---|--|
| Pyrolysis | <ul style="list-style-type: none"> • This is a process thermal decomposition of organic matters without oxygen and catalyst. Pyrolysis materials are vegetable oils, animal fats, natural acids or methyl esters of fatty acids. • Causes simple pollution | <ul style="list-style-type: none"> • Pyrolysis of vegetable oil can yield a substance with a high cetane number. • Low viscosity product. • Yield a high cetane number product. • Yield acceptable amount of sulfur, water and sediment content product. • Yield acceptable copper corrosion values. | <ul style="list-style-type: none"> • Yield carbon residues. • Yield unacceptable pour point. • High production cost • Biofuel has no oxygenated value. • Complex equipment requirement | (Noor, Noor and Mamat 2018; Irawan and Hasan 2021) |
| Micro emulsion | <ul style="list-style-type: none"> • It is a spontaneous generated colloidal equilibrium dispersion of an optically isotropic fluid microstructure with dimension in the range of 1-150 nm. Several researchers have studied micro emulsion utilizing solvents such methanol, ethanol, ethanol, hexanol, butanol and 1-butanol. • It has demonstrated that the short term performance of aqueous ethanol micro emulsion in soybean oil. | <ul style="list-style-type: none"> • It has a pyrolyzed distillate of soy bean oil, which high had a cetane number. • High viscosity • Lower nitrogen oxide emissions | <ul style="list-style-type: none"> • No by products or waste formation • Lower nitrogen oxide emissions • Clear single phase, and thermodynamically stable colloidal equilibrium. | (Syafiuddin <i>et al.</i> 2020) |

| | | | | |
|--------------------|---|---|---|----------------------------------|
| Esterification | <ul style="list-style-type: none"> • It is the process of oil conversion into biodiesel esters using the base catalyzed transesterification method. • The process is employed to neutralize the high free fatty acid value. | | <ul style="list-style-type: none"> • It can produce biodiesel even in a high free acid raw material. • It can use acid or base catalyst for the process. | (Jiyane, Tumba and Musonge 2018) |
| Transesterificaion | <ul style="list-style-type: none"> • Transesterification, also known as alcoholysis, is the process of replacing one alcohol with another form of an ester. • Transesterification process uses acid-base catalysis to produce biodiesel from vegetables and animal fats. • Transesterification can be carried out into 2 catalytic method homogeneous and heterogeneous. | <ul style="list-style-type: none"> • Transesterification of high free fatty acid yield by-product and water. • Transesterification using catalytic homogeneous method generate lots of waste water in washing the product. • Transesterification using catalytic homogenous catalyst makes it impossible to separate catalyst and product. | <ul style="list-style-type: none"> • The most common method for production of biodiesel. • Unreacted feedstock can be recycled. • It can use acid or base catalyst for the process. • Reduce triglyceride viscosity. • Enhance engine performance. • Produces product with less CO₂ emission environmental friendly. • The by-product can (i.e., glycerol) can be converted into useful products. | (Mansir <i>et al.</i> 2018) |

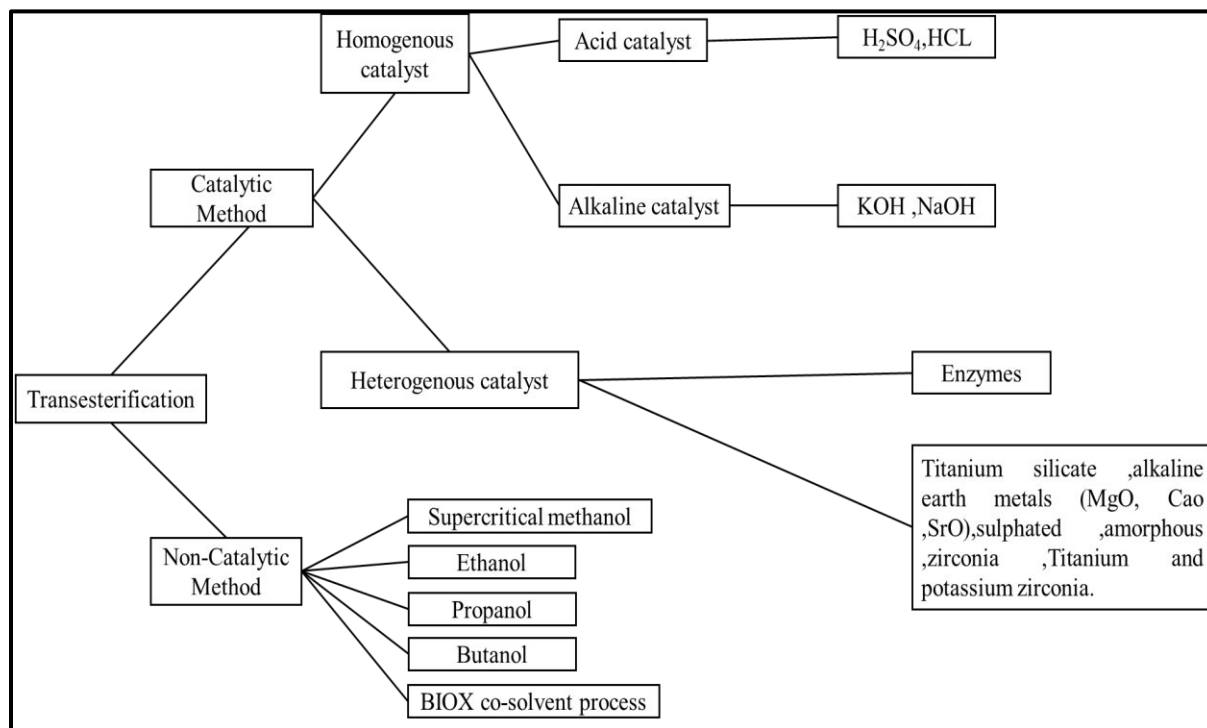


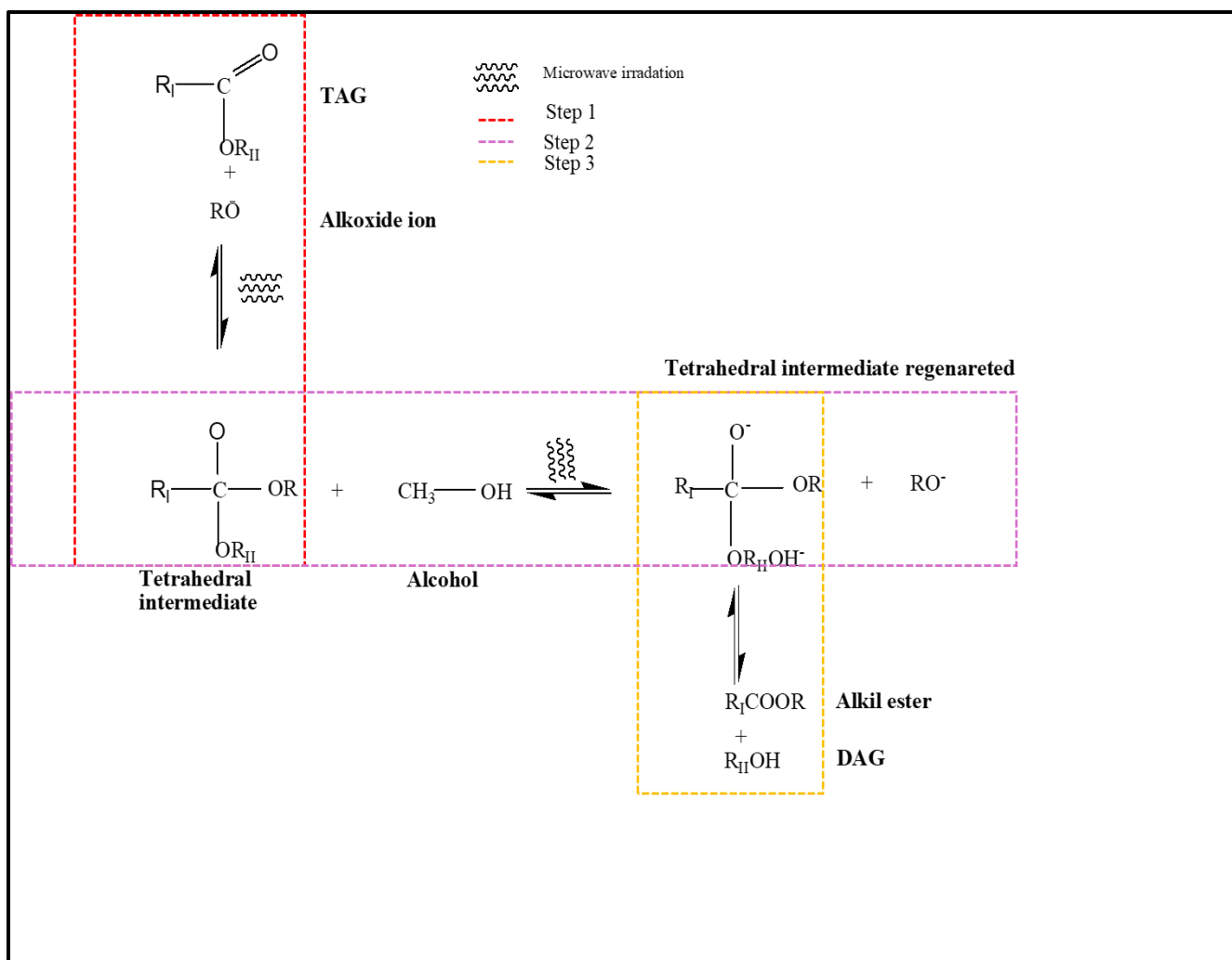
Figure 2.4: Transesterification process classification (Ayodeji 2018)

The transesterification process classification is shown in Figure 2.4 above (Ayodeji 2018). The transesterification process is the reaction of triglycerides, a type of fat or oil, with alcohol and a catalyst to produce ester and glycerol. Three long-chain fatty acids are joined to a glycerin molecule, which serves as the base of a triglyceride. The properties of the oil or fat are determined by the type of fatty acids that are bound to the glycerin. The properties of biodiesel can be influenced by the makeup of fatty acids (Deshmukh, Kumar and Bala 2019).

2.8 Homogeneous catalyzed transesterification

An alkaline or acidic solution is employed as a catalyst in this process. Typically, alkaline catalysts, like potassium methoxide and KOH, are utilized to produce biodiesel because they are less expensive and more efficient. Alkaline catalysts have the ability to produce rapid, high reaction yields (above 97%) of time (30 minutes–2 hours) at moderate temperatures (25–70°C) (Osorio-González *et al.* 2020). Raw castor oil obtained from Brazilian seeded castor is extracted via mechanical and chemical extraction. The transesterification is carried out using a simple and high-catalytic method, which is a homogenous alkaline transesterification reaction. Previous studies

reviewed the reaction conditions. The parameters were as follows: methanol to oil molar ratio of 6:1 and 1% (w/w), temperature range of (55–65°C), duration of (2–8 hours), and product yield range of 43.3–74.1% w/w biodiesel (Dias *et al.* 2013). Two methods can be used to carry out transesterification both homogeneous and heterogeneous catalytic). During the process, an electrophilic alkoxide ion attacks the electrophilic carbonyl group of a triglyceride molecule. Equation 6 presents how triglycerides break down when exposed to a microwave and an alkaline catalyst. A tetrahedral intermediate is seen in the first stage when the carbonyl group of triacylglycerol molecules is joined to the alkoxide ion. In the final stage, the tetrahedral intermediate reorganizes to form the molecules fatty acid alkyl esters (FAAE) and diacylglycerol (DAG) by reacting with alcohol to produce the new alkoxide ion. The mechanism takes place both during and after the breakdown of each fatty acid. At the end, three fatty acid ester groups are formed, which are fatty acids (FAEs) and glycerol (Osorio-González *et al.* 2020).



Equation 6: Transesterification reaction sequence of oil using alkaline catalysis and microwaves descending from (triglyceride), Diglyceride R_{II} long chain hydrocarbon which are called fatty acids chains up to glycerol R_I : R alkyl group of alcohol (Osorio-González *et al.* 2020).

Table 2.5 lists the different alkaline transesterification methods used to create biodiesel. It was found in a study using rapeseed oil that the basic transesterification could produce yields of up to 95% in less than two minutes. Longer reaction times lead to saponification. This means that in order to prevent the formation of soap, which could result in large losses, the transesterification reaction needs to be stopped at the reactor's exit point (Osorio-González *et al.* 2020). Therefore, the protonation of the carbonyl group in the triglyceride molecule and the reaction between alcohol and the protonated group to form a tetrahedral intermediate are the most crucial steps in acid catalysis. Acid catalysis requires the catalysis of hydrochloric acid and sulfonic acid. Given that

during the transesterification of vegetable oils with a high concentration of free fatty acids (FFA), soap does not form .Long reaction times and a lot of alcohol are necessary for acid catalysis (Gardy *et al.* 2019; Osorio-González *et al.* 2020) . Table 2.5 shows the Alkaline transesterification process used in the production of biodiesel from castor oil (Osorio-González *et al.* 2020).

Table 2.4: Biodiesel production from castor oil using alkaline transesterification

| Method | Conditions | Biodiesel features | Yield | Reference |
|------------------------------|--|---|--------------|---------------------------------|
| Alkaline transesterification | 1:12 oil:methanol potassium hydroxide 1.25% wt 60 °C ,1 h | High density and flash point. Low sufur content yellow color | 94.9% | (Elango <i>et al.</i> 2019) |
| Alkaline transesterification | 1:5.4 oil:methanol potassium hydroxide 1.25% wt 35.5 °C, 1 h | | 97.8% | (Meneghetti <i>et al.</i> 2006) |
| Alkaline transesterification | 1.8:24 oil:methanol potassium Hydroxide 1.45% wt 64 °C 40 minutes | Low acid value | 93.2% | (Dias <i>et al.</i> 2013) |
| Alkaline transesterification | 0.29:1 oil:ethanol potassium hydroxide 1% wt 62.5 ^{2.5} °C, 2 h.:26 minutes | High density and flash point | 85.5% | (Bateni <i>et al.</i> 2019) |
| Alkaline transesterification | 1:6 oil:ethanol potassium hydroxide 1% wt | Low acid value high flash point | 43.3 – 74.1% | (Dias <i>et al.</i> 2013) |

| | | | | |
|--|----------------------------|--|--|--|
| | 55 -65 °C, 2 h.:26 minutes | | | |
|--|----------------------------|--|--|--|

2.9 Heterogeneous catalyzed transesterification

Heterogeneous catalysis is an option for making biodiesel without generating aqueous waste. In this process, the catalyst can be retrieved, renewed, and used again. It is possible to produce large amounts of high-quality biodiesel continuously or in batches without the need for extra purification steps. One major benefit of this catalysis is that it allows simultaneous triglyceride transesterification and free fatty acid esterification, which saves time and energy (Mulyatun *et al.* 2022). Amberlyst and nafion solid catalysts are among the commercially accessible catalysts for heterogeneous esterification. Various solid acid carbon modified metal oxides, heteropolyacids, metal-incorporated porous oxides, zeolites, ion exchange resins, inorganic-oxides solid acids, and supported noble metal oxides synthesis is to serve as catalysts in the conversion of low-quality vegetable oils into biodiesel. Heterogeneous catalysts have reportedly been utilized to produce biodiesel from a variety of vegetable oils (Du *et al.* 2018). For instance, heterogeneous catalysts, such as potassium oxide (K_2O) and potassium aluminate ($K_2Al_2O_4$), magnesium aluminate ($MgO-Al_2O_3$), have been used (Sun *et al.* 2014). Recently, potassium carbonate (K_2CO_3), lithium zinc oxide, hydrotalcite calcined by magnesium aluminide (Mg-Al), Bionanocatalyst (CaO-MoO₃-SBA-15), and potassium were used. The method used for producing biodiesel from canola oil was developed using sodium methoxide (CH_3ONa) as a catalyst crystallized from dimethyl carbonate (DMC). This procedure aids in achieving conversions above 95%, with the exception of yielding glycerol as a by-product; nevertheless, the catalyst's efficiency decreases with each reaction. Certain materials derived from plants have also been suggested for use as catalysts. For instance, (Li *et al.* 2014) used a solid acid catalyst (RHC-SO₃H) made of activated carbon from rice husk (RHC) to conduct experiments with concentrated sulfuric acid. With used cooking oil waste, this catalyst demonstrated excellent catalytic performance and stability during the biodiesel production process. One of the primary drawbacks of heterogeneous catalysts is low reaction rates, which are primarily brought on by the use of a lot of catalyst and circumstances that compromise the catalyst's stability (del Valle, Núñez and Aravena 2014). The main parameters that influence chemical transesterification are temperature, alcohol-triglyceride ratio, reaction time, catalyst,

moisture content, and the quantity of free fatty acids (Osorio-González *et al.* 2020). The purpose of this study is to look into the effects of catalyst, alcohol-oil molar ratio, reaction temperature, and reaction time. The effect of different parameters during the production of biodiesel is displayed in (Mulyatun *et al.* 2022).

Table 2.5: Effect of main parameters on transesterification

| Parameter | Effect | Reference |
|----------------------------------|---|-----------------------------------|
| Time | Longer reactions are said to accelerate the process of converting fatty acids into methyl esters. Longer reaction times consequently require a lot more energy. | (Aboelazayem <i>et al.</i> 2018) |
| Temperature | Temperature has an impact on the reaction rate, depending on the catalyst. The optimal temperature is between 25 °C or 60 °C and 120 °C. Three moles of fatty acids alkyl esters and one mole of glycerol can be produced via the stoichiometric ratio of 3:1 alcohol to oil ratio. | (Keera, El Sabagh and Taman 2018) |
| Alcohol triglyceride molar ratio | Even though alcohol gets in the way when glycerol is separated from biodiesel, an excess of alcohol is needed for the most effective transesterification reaction to promote product equilibrium. | (Batani <i>et al.</i> 2019) |
| Catalyst | Compared to acid catalyst an alkaline catalyst accelerated the rate of reaction. When vegetable oil has a high free fatty acid content, acid transesterification is recommended. | (Sánchez <i>et al.</i> 2019) |
| Moisture content | Triglycerides and alcohol both need to be anhydrous for alkaline catalysis since water causes saponification. | (Elango <i>et al.</i> 2019) |
| Content of free fatty acids | To avoid the formation of soap during the alkaline catalysis, the quantity of free fatty acids should be as low as 0.5% w/w of oil. | (Zeng <i>et al.</i> 2014) |

2.10 Biological transesterification

Cooking oils and other feed stocks which are high in free fatty acids require an acid-catalyzed esterification process prior to alkaline transesterification. Equipment corrosion will make the production of biodiesel more economical. To counteract the effect, more chemicals will be needed in addition to the biodiesel. As a result, pollution in the environment also rises. Biological catalysis offers a useful remedy for these problems. Biological agents can operate in milder conditions, emit

fewer pollutants, and promote the separation of glycerol when used as catalysts in the biodiesel production process (Yan *et al.* 2014). Biological catalysis can generally make use of free or immobilized enzymes as well as microbial cells. Given that immobilized or free enzymes are less widely accepted than whole cell systems. These are the biocatalysts for biological transesterification that are currently most commonly used and acknowledged. An example of this type of transesterification for the synthesis of biodiesel using castor oil as a raw material is the study carried out by (Kumar *et al.* 2020). To achieve a 78% biodiesel yield, the ideal parameters were a 6:1 alcohol:molar oil ratio and 10% of the immobilized enzyme for 24 hours at 50 °C. Furthermore, it was found that the immobilized enzyme has a 12-cycle shelf life, and that after 6 cycles, 70% of the biodiesel is produced. Using biological catalysis (free and immobilized enzymes) and castor oil as a feedstock, Andrade *et al.* (2019) carried out a simulation of the entire biodiesel production process. The observed results demonstrate that flow increased and the amount of enzyme was minimized when free enzymes were used. In the enzyme transesterification reaction, the flow raises the profit. The reactor's ideal parameters in this process were a reaction temperature of 36.4 °C, a methanol:oil ratio of 9:0, and 0.3% w/w of castor oil to produce a 93% biodiesel yield. In contrast, the best conditions for achieving 99.9% yield when employing immobilized enzyme were 50 °C of reaction temperature for 4 hours with a methanol:oil ratio of 17.6% w/w kg of castor oil. The immobilized enzyme produced a larger yield of biodiesel, but the overall expense of the procedure was higher than when the transesterification reaction was carried out with a free enzyme. The biocatalyst used to produce biodiesel is shown in Osorio-González *et al.* (2020). As was previously mentioned, a transesterification reaction needed to happen at a higher temperature and take longer to complete. This resulted from the transesterification reaction's higher methanol:oil ratio and higher reaction temperature and duration. Other biological processes have also been reported in a similar manner (Milano *et al.* 2018).

Table 2.6: Biocatalysis used biodiesel production.

| Biocatalyst | Advantages | Disadvantages | Reference |
|---------------------|--|---|------------------------------|
| Whole cell | Simple preparation. There is no need for purification or immobilisation. | Simple deactivation. Low rate of reusability and recovery | (Amini <i>et al.</i> 2017) |
| Free enzymes | Environmentally benign, strong catalytic activity, excellent selectivity, and low energy consumption | Unstable. Challenging to reuse. Costly | (Andrade <i>et al.</i> 2019) |
| Immobilized enzymes | Stability and reusability | Costly. low rate of reaction During the recovery process, extra steps reduced enzyme activity | (Kumar <i>et al.</i> 2020) |

Biodiesel was produced from *Jatropha curcas* oil by Chuah *et al.* (2021) using a lipase-stabilized Pickering emulsion combined with mesoporous organosilica. With the help of this catalytic system, biodiesel could be produced at a yield of 95% when oleic was used and 87.1% when *jatropha curcas* was. A yield of above could be sustained for 15 reaction cycles (Yan *et al.* 2014). To improve the process of converting used cooking oil into methyl esters of fatty acids the recombinant yeast is utilized with functional intracellular lipase production from *Thermonyces lanuginosus* (Yan *et al.* 2014). According to reports, the modified strain's enzyme helped with oil conversion, leading to an 82% transesterification efficiency (Osorio-González *et al.* 2020).

2.11 Transesterification using ultrasound

Sound waves that have a frequency higher than what is detectable by humans are referred to as "ultrasound". The frequency range of ultrasound waves is 20 kHz to 100 kHz. Molecular spaces may compress and stretch when high-frequency sound travels through the reaction system, leading to vibration and cavitation. By supplying cavitation energy to start reaction and only a part of the mechanical energy (motor-mixer) to mix the reaction media. Raising the temperature is

specifically impacted by ultrasonic cavitation. Bubbles formed by cavitation eventually burst and separate the two liquid phases. The emulsification process is further enhanced by the passage of ultrasonic jets from one liquid to another. The yield of biodiesel and reaction rate are both increased by this phenomenon (Osorio-González *et al.* 2020). Because transesterification reactors are straightforward and safe, applying ultrasound to the process is appealing. Experiments with different feedstocks have demonstrated the advantages of ultrasonically assisted transesterification, including higher reaction rates and yields of biodiesel and lower alcohol:oil molar ratios and operating temperatures. The study conducted by (Sabzimaleki *et al.* 2015) provides an illustration of the production of biodiesel using castor oil. They optimized a number of transesterification reactions with ultrasound assistance to create biodiesel from castor oil. In ideal circumstances, they were able to achieve an 87% reaction yield, a 64% wave ultrasonic amplitude, 0.73% of an ultrasonic cycle, and an oil:methanol ratio of 1:8.15. Reaction time is the most crucial factor in the transesterification reaction to produce a high yield of little biodiesel (Crudo *et al.* 2016). The product's properties don't change when both the esterification and transesterification processes are carried out in the same reactor. Alcohol's oil solubility may rise as a result. When extracting biodiesel, combine ultrasonics with co-solvents to maximize yield. The most popular co-solvents are dichloromethane, petroleum ether, benzene, hexane, tetrahydrofuran, and chloroform (Suganya, Kasirajan and Renganathan 2014).

2.12 Heterogeneous catalyst

Modern researchers have considered a possible replacement for the homogeneous catalyst because it is both environmentally friendly and corrosive. Triglycerides, alcohol, and solid catalyst make up the three-phase system composition of the reaction mixture, which restricts the availability of active sites for the catalytic reaction and hence lowers the reaction rate. Consequently, a mass transfer resistance issue is typically encountered by the heterogeneous catalytic reaction (Abdullah *et al.* 2017).

Although the mass transfer limitation is lessened by the catalyst support, the catalyst lifecycle is shortened by alcohol's easy corrosion of the supported catalyst's active species (Mota *et al.* 2022). Enzymes are used as a catalyst in the biocatalytic transesterification process, which yields biodiesel that looks promising. The product's ease of separation without adverse reactions accounts for this (Keera, El Sabagh and Taman 2018). As a result of its unfavorable reaction and prolonged

reaction duration in contrast to alkaline catalysts, the biodiesel produced by this method is currently not commercially feasible. According to reports, enzymatic transesterification takes a full day to produce a 90.5% biodiesel yield (Pasha *et al.* 2021). The cost of the enzymes makes this process economical. Because deactivation of feed impurities could reduce the efficiency of most enzymes, they require very specific reaction conditions (Pasha *et al.* 2021).

2.13 Supercritical alcohol transesterification

A novel approach to producing biodiesel devoid of catalysts. Under supercritical conditions, the mixture of triglyceride and alcohol could become homogenous, thereby overcoming the mass transfer limitation (Bashir *et al.* 2022). However, this non-catalytic method's main drawback is how much energy it uses. The high reaction temperature's increased production costs make large-scale industrial application unfeasible (Hajjari *et al.* 2017). Catalytic and non-catalytic reactions are also at risk from the supercritical process. The downstream transesterification process will also yield a mixture of product, biodiesel, and glycerol in addition to unreacted reactants and catalyst (Ayodeji 2018). Ineffective biodiesel separation and purification may lead to severe diesel engine issues like filter plugging, carbon deposits, engine knocking, filter plug chocking, oil ring sticking, thickening and gelling of lubricating oil, and excessive engine wear (Ismail *et al.* 2014). To create high-purity biodiesel, the transesterification process's downstream will go through a number of complementary separation phases, including glycerol separation, catalyst neutralization, and biodiesel purification. There are several downstream processes, which take time and money. Research indicates that between 60 and 80 % of the total cost of a transesterification process plant was accounted for by the current downstream processing alone. Moreover, the yield of biodiesel may be lost as a result of several separations and purifications (Suthar, Dwivedi and Joshipura 2019).

2.14 Operating conditions for transesterification reactor

Temperature range is 25 °C – 180 °C for the production of biodiesel the temperature should be kept below 180 °C to accommodate alcohol reactants in a reaction. Alcohol has a boiling point 60 °C while the reactor should not exceed 180 °C to ensure the reactor does not get explode or damaged.

Pressure of the reactor must standard condition to avoid the build –up pressure within the reactor since it is processing oil which is an expanding liquid. Operating at 1 atm will eliminate problems that will result exploding the equipment.

Reactant flow rate can depend on the type of a process for example if is continuous process flow rate can have an impact on the process also in the reactor and its operating conditions. The batch process depends mostly on time, the flow rate cannot have an impact on the process and also on the reactor.

The three operating conditions are the main condition which can have an impact on reactor and also in the reaction.

2.15 Process intensification technology in biodiesel production

Biodiesel production using conventional methods has various challenges, which include, the purification of the products, which leads to the generation of waste water (Suthar, Dwivedi and Joshipura 2019); insufficient mass transfer (Bashir *et al.* 2022); equilibrium thermodynamic conditions (Cao *et al.* 2008) elevated energy needs (Najjar and Abu-Shamleh 2020). Utilizing process intensification technologies can help overcome these limitations. As indicated in Table 2.7, the application of technologies utilizing novel reactors, separation procedures, or coupled reactions is necessary to increase the reaction rate and decrease the residence time. Thus, the residence time will be shortened by this technology. Technology needs to create special reactor types for this kind of processing in order to enhance mixing and mass heat transfer between the oil and methanol in the production of biodiesel (Mohiddin *et al.* 2021). Novel reactors, been developed and applied to transesterification process which include oscillatory flow reactors, cavitation reactors, microwave reactors, and microchannel reactors which be applied to transesterification reaction to produce biodiesel (Mohiddin *et al.* 2021).

2.15.1 Transesterification reactors

Table 2.8 presents the novel reactors and transesterification reactors (Tabatabaei *et al.* 2019). The two types of reactors either improve the mixing of the oil and methanol or the heat and mass transfer between the two liquid phases to intensify the transesterification of vegetable oil to produce biodiesel. All these types of reactors, novel and transesterification reactors are energy

intensive and require a lot of time for transesterification. High pressure drop on transesterification reactors, while novel reactors consume lots of water for product purification which is a disadvantage for these two types of reactors. In order to overcome the restriction brought about by the transesterification-induced chemical equilibrium, a jacketed reactor, a separating funnel, and water bath to maintain temperature will be used to conduct the transesterification process in this study.

Table 2.7: Transesterification and novel reactors.

| Types or reactor | Description | Advantages | Disadvantages | Reference |
|------------------------|--|---|--|---------------------------------|
| Microwave reactor | <ul style="list-style-type: none"> • A microwave reactor consist of a microwave source that supplies electromagnetic energy to a chemical reactor that in enclosed in an applicator (a metallic cavity) through either coaxial cables (for lower power) or a waveguide (for higher power) • A microwave reactor is able to achieve similar biodiesel conversion with a shorter reaction time and in a more efficient manner. | <ul style="list-style-type: none"> • simple control • Time and less downstream processing | <ul style="list-style-type: none"> • .The difficulty of managing power and temperature. • Partially expanded, resulting in low production. | (Tabatabaei <i>et al.</i> 2019) |
| Fluidizes bed reactors | <ul style="list-style-type: none"> • This is the type of reactor that can carry out a variety of multiphase chemical reactions. | <ul style="list-style-type: none"> • Uniform temperature distribution • High mixing intensity | <p>High capital and operating costs</p> <p>Requirement of expensive regeneration equipment</p> | (Tabatabaei <i>et al.</i> 2019) |

| | | | | |
|--|---|--|--|--|
| | <ul style="list-style-type: none"> • This is the type of reactor that can carry out a variety of multiphase chemical reactions. • The fluid passes through the solid at a high enough speed that it causes to behave differently. • The fluid is forced through the distributor up through the solid material. • In solid phase, various flow regimes can be observed in the reactor. | <ul style="list-style-type: none"> • Continuous or batch process • lower possibility of membrane plugging • High heat and mass transfers • Convenient replacement or regeneration of catalysts • The simplest chemical reactors • Easy to clean. • Efficient use of reactor volume • Low maintenance • Low capital and construction space • Side-stream additive injection devices and other | <p>Large pressure drop</p> <p>High catalyst attrition and reactor wall erosion</p> <p>Only free-flow catalysts can be applied.</p> | |
|--|---|--|--|--|

| | | | | |
|---------------------------|---|---|--|----------------------------|
| | | mixers to improve mixing. | | |
| Tubular/plug flow reactor | <ul style="list-style-type: none"> • This is the tubular reactor, into which the reactants and reagents enter through pipes at constant velocity. • Reactants are mixed while flowing towards the outlet. • This unit can only produce biodiesel in a batch process. | <ul style="list-style-type: none"> • The simplest chemical reactors • The simplest chemical reactors • The simplest chemical reactors • Easy to clean • Low maintenance • Low capital and construction space • Uniform product quality • Compatible with side side-streams additive injection device and other mixers to improve mixing | <p>Requirement for a large length-to-diameter ratio</p> <p>Operative at stationary conditions</p> <p>Inappropriate for slow reactions, i.e., requiring slow mixing and/or large hold-ups</p> | (Awogbemi and Kallon 2022) |

| | | | | |
|--------------------|--|---|---|---|
| Packed bed reactor | <ul style="list-style-type: none"> • . This unit is constructed with random packing or structured packing. • In the column, liquids tend to wet the surface of the packing, and vapours pass across the wet surface where mass transfer takes place. • The unit can be randomly filled with small objects, such as activated carbon. • The maximum conversion rate yield of approximately 75% biodiesel was observed at 12 h at 40 °C. | <ul style="list-style-type: none"> • High heat and mass transfers. • Regeneration of catalysts. | <p>High capital and operating costs.</p> <p>High cost of internal equipment to replace.</p> <p>High pressure drop</p> | (Wang <i>et al.</i> 2019; Mingxin <i>et al.</i> 2022) |
| Oscillatory flow | <ul style="list-style-type: none"> • Is a vertical or horizontal continuous reactor consisting of one or several tubes each with an equal-distance baffle at one or both ends of the tubes. Bellows diaphragms or pistons are used to generate oscillatory flow and | <ul style="list-style-type: none"> • Improves resident time. • Improve heat and mass transfer between liquids | <ul style="list-style-type: none"> • Consumption of energy. • Reaction requires a lot of time. • High cost of maintenance. | Tabatabaei <i>et al.</i> , 2019 |

| | | | | |
|-----------------------|---|---|--|--|
| | <p>periodic changes in flow direction.</p> <ul style="list-style-type: none"> • Reversal cycles are repeated due to interaction with opposing vortices. • Improves heat and mass transfer under low shear and practical global mixing conditions. • Helical baffles and wire wool configurations are used to promote interphase dispersion in two-phase liquid-liquid systems (methanol and oil) in transesterification. | | | |
| Microchannel reactors | <ul style="list-style-type: none"> • This reactor has a large volume or surface. • The reactor exhibits a short diffusion ratio and efficient heat and mass transfer dissipation. • The low production throughput of microchannel reactors prohibits the addition and application of | <ul style="list-style-type: none"> • Simple control and time. • Less downstream processing. • Shorter reaction time and high biodiesel production. | <ul style="list-style-type: none"> • High energy consumption. • Difficult to manage power and temperature results. • Low yield in biodiesel production. | (Gopi, Thangarasu and Ramanathan 2022) |

| | | | | |
|--|---|--|--|--|
| | parallel reactors to amplify the production of biodiesel. | | | |
|--|---|--|--|--|

CHAPTER 3: EXPERIMENTAL APPARATUS AND METHODOLOGY

This chapter provides the full description of all equipment used to perform experiments and experimental procedure. The main the equipment was the glass jacketed reactor. It was customer with certain diameter and volume that suit experimental. All other equipment are discussed later in this chapter.

3.1 Glass jacketed reactor

Figure 3.1 shows a jacketed reactor consists vertical shell with a standard tori spherical dish on both ends. The temperature of reactants is controlled by circulating heating or cooling media fluid through the bath. Ideally suitable for heating below 250 °C. Commonly used for heat and mass transfer, including hot water, steam, and hot oil. The jacketed reactor is a device that combines reactions and increases or cools the temperature in a single unit (Jiyane, Tumba and Musonge 2018). Three primary characteristics are used to characterize jacketed performances. The first is how quickly the jacket temperature may be changed. The consistency and stability of the jacket's temperature. When taken as a whole, these significantly affect batch time and total operating expenses (Johnson *et al.* 2021). It is the foundation of important safety and quality factors that could ultimately determine whether the response is successful or fails. In the transesterification reaction, alcohol and VOs are the two reactants. At first, the mass transfer is limited because they cannot mix properly (Sabzimaleki *et al.* 2015). Because of the poor mass transfer of triglycerides from the oil phase to the oil-methanol interface, which causes a slower reaction rate, oil is only weakly soluble in methanol. Consequently, in order to create an emulsion by increasing the region of contact between the two immiscible phases, vigorous mixing is needed (Bhangu, Gupta and Ashokkumar 2017). The two phases are typically mixed with a magnetic stirrer, and the temperature maintained in the jacketed reactant helps to improve the mixing of the two reactants.

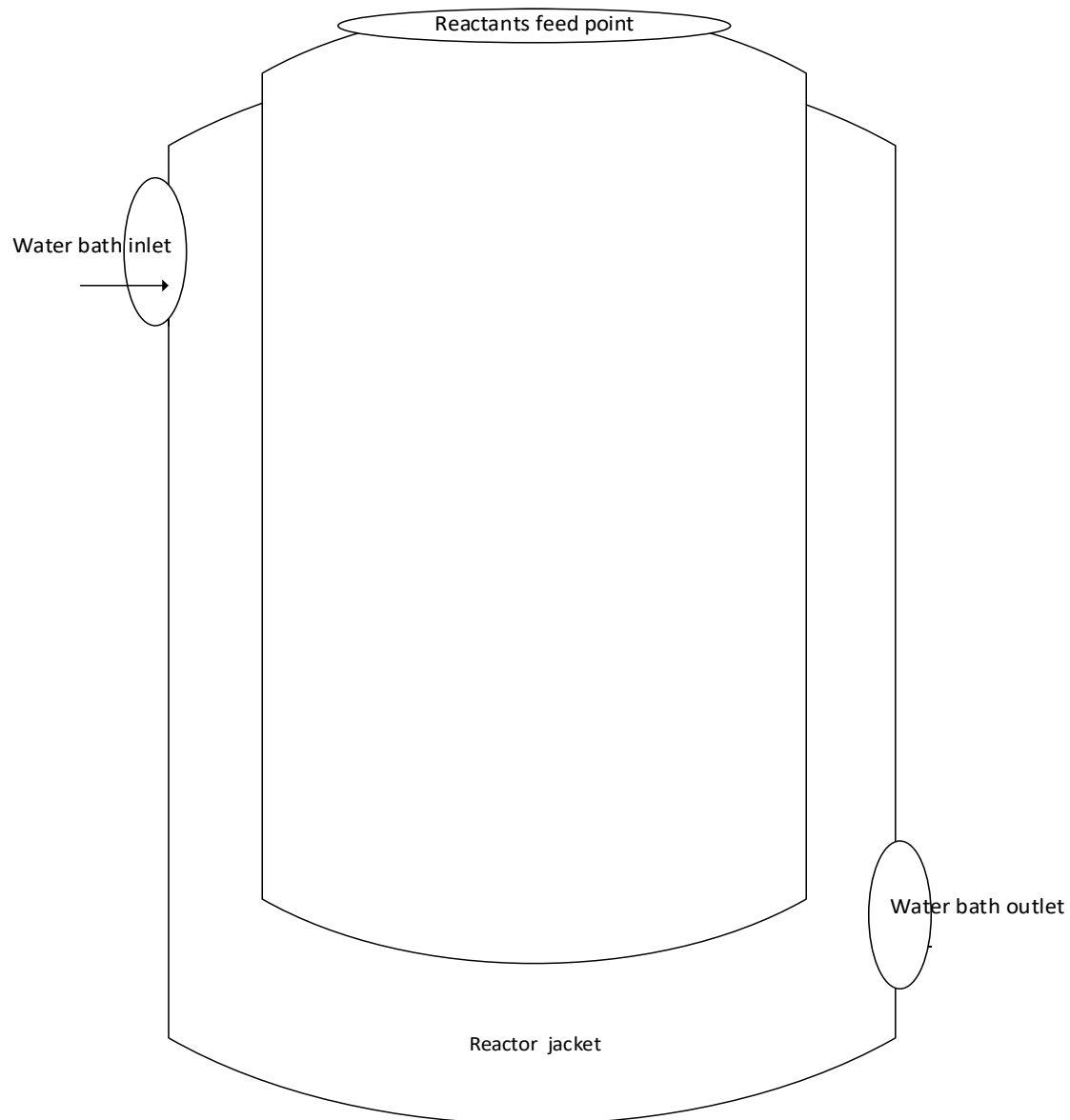


Figure 3.1: Glass jacketed reactor

3.1.1 Features for jacketed reactor

- Double wall for temperature controlling and monitoring

- Outer diameter and inside diameter (8.5 cm and 6 cm) respectively and height is 23 cm
- Feed reactant inlet diameter 3cm
- Inlet and outlet water bath holes diameter 0.5cm both are similar
- Volume of a reactor is 500 ml

The jacketed reactor design's ability to produce biodiesel from a variety of feed sources, including canola oil, sunflower oil, and leftover cooking oil, demonstrated the suitability of the concept. For the production of biodiesel from castor oil, the jacketed reactor is a highly appropriate solution. The reactor's ability to produce biodiesel from a variety of feed sources, including canola oil, sunflower oil, and waste cooking oil, demonstrated the suitability of the concept.

3.2 CHEMICALS AND REAGENTS

- Commercial 84% purity, purchased from LabCare suppliers
- raw castor oil 48% purity, produced by Mkhize (2023) at Durban University of Technology Chemical Engineering laboratory
- Methanol 99.9% purity, purchased from LabCare suppliers
- Potassium hydroxide pellets 99.99% purity, purchased from LabCare suppliers
- Phenolphthalein indicator 98 % purity, purchased from LabCare suppliers
- Ethanol 99.9% purity, purchased from LabCare suppliers
- Sodium hydroxide 98% purity, purchased from LabCare suppliers

3.3 METHODOLOGY

Vegetable oils are chemically processed into biodiesel through a process called transesterification. This method, which is described in the literature review, produces biodiesel and glycerol by reacting vegetable oil with alcohol that has shorter chains, like methanol or ethanol, in the presence of a catalyst (Kayode and Hart 2019). In this research, both raw and purified castor oil were used. Since castor oil is inedible and does not jeopardise food security, it was employed. (Sabzimaleki *et al.* 2015). A less expensive, non-edible feedstock is castor oil. It is one of the most promising oil feedstocks in South Africa for the production of biofuel. We measured the oil's acid value and water content. The affordability, effectiveness, and availability of methanol (99.8%) made it the preferred solvent. The homogeneous alkaline catalyst KOH was selected for this investigation due to its environmental friendliness, ease of separation from the product, and recyclable nature. This

kind of catalyst doesn't need to be further purified in order to be used continuously.. All the experiments were conducted in Chemical Engineering post graduate laboratory at Durban University of Technology (DUT). Analytical equipment used in characterization of finished biodiesel (FAME) were the FFS Refiners laboratory. Methyl ester (FAME) characterization was done in the FFS Refiners research and development laboratory in Durban Teakwood branch.

Jacketed reaction technology was chosen for the reaction and ability to perform heat mass transfer depending on how reactive they are in a single process stream. This process of performance, reaction, heat, and mass transfer took place within a single unit.

3.3.1 Oil characterization procedure

The mass of 0.6g of NaOH was measured and 0.015 moles was manipulated to calculate the mass of NaOH. The calculated amount of NaOH with 0.1N concentration, was dissolved in 150 ml of water, the solution with reagents were prepared in 500ml beaker. A known mass of (± 5.00 g) of castor oil was transferred in 250ml beaker then placed in hot plate to warm the temperature of oil and decrease castor oil viscosity. The 50 ml of 99.9% Ethanol was transferred into a 150 ml conical flask then oil was dissolved in the solution and allowed to cool off for couple of minutes, moreover few drops of phenolphthalein indicator were added. 0.1 N NaOH solution was transferred into a burette. This was titrated against a NaOH solution in a burette with continuous stirring. This equation provides the *castor oil's* acid value..:

$$\text{Acid Value}(AV) = \frac{40 \times N \times V}{m} \quad \text{Equation 3.1}$$

Where the constant 40.0 is the molar mass of NaOH, N is the normality of the standardized NaOH solution, V is the volume of the NaOH solution in (ml) and m (g) is a mass of the castor oil. Acid Value for purified castor oil was calculated 1.03 and the raw castor oil was found to be 0.4 which is within the limit of required AV for direct transesterification in both types of castor oil. The extraction of raw castor from castor seeds using various types green solvents in the previous study of Mkhize (2023) could be the method which was decreasing acid value to castor oil, which could be the reason why acid value for raw castor oil is lower than that purified castor oil. To ensure the accuracy of the feedstock characterization relative error was calculated.

$$AE = AV_{lit} - AV_{Cal} \quad \text{Equation 3.2}$$

AV_{lit} :- Acid value in literature

AV_{cal} :- Acid value calculated

3.3.2 Materials and experimental procedures

The homogeneous alkaline catalyst was used in the study adapted from method applied in the work of producing castor biodiesel (Keera, El Sabagh and Taman 2018). Mixture of potassium hydroxide and purified castor oil was also applied in the study (Keera, El Sabagh and Taman 2018). These were purchased from associated chemical enterprises, South Africa. The raw castor oil was produced by Mkhize (2023) from castor seeds in the same laboratory. Raw castor was used in this study to produced biodiesel. In this study, the parameters that were investigated to find optimum conditions are presented in Table 3.1.

For determination of the biodiesel on the Anton Paar was used to determine the density. The density meter DMA 4100 M is built with refractometer inside, for the measurements reflected on the screen of the refractometer.

Table 3.1: Range of parameters investigated.

| Parameters | Value Range | Units |
|------------------------|---------------|--------------|
| Catalyst concentration | 0.625 – 3.125 | % wt KOH |
| Alcohol ratio | 3:1 – 9:1 | Methanol:oil |
| Reaction Temperature | 45 – 70 | °C |

The design expert software was used to determine the number of experiments based on the parameters listed in Table 3.1.

3.3.3 Mixing reagents

Mixing reagents was carried in an orbital shaker with a 100 ml glass beaker filled with a known mass of methanol and KOH pellets as a catalyst mixture. The speed and time were estimated

according to the amount of KOH required in a batch. The reagent was initially in different phase. KOH pellets as a catalyst was required to dissolve in alcohol methanol and form a single phase.

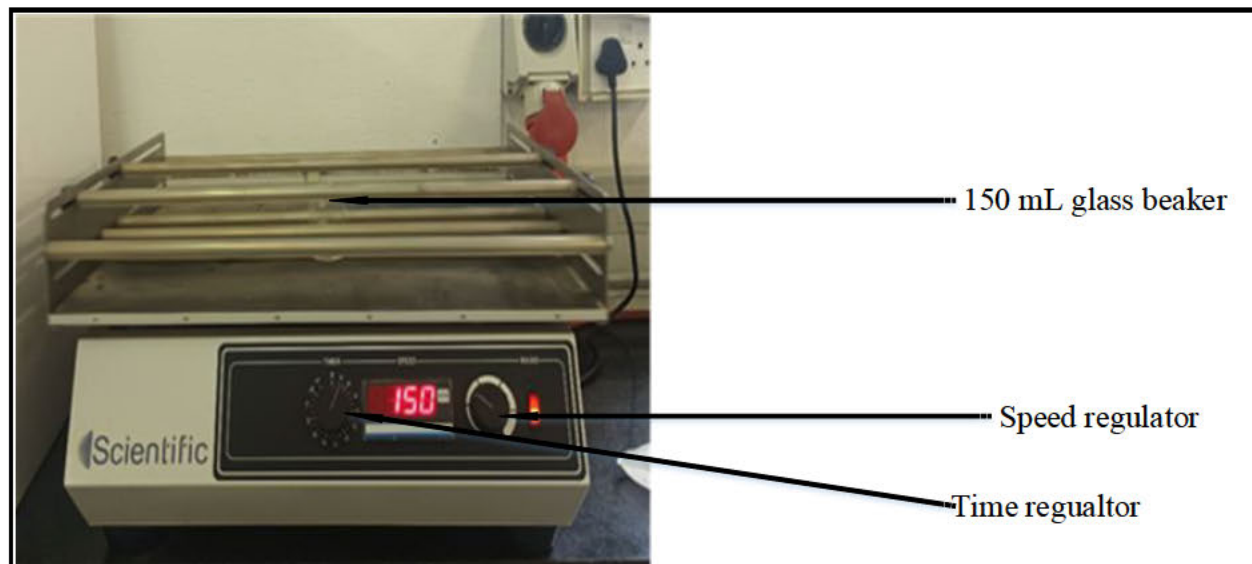


Figure 3.2: Orbital shaker

Methanol and KOH pellets solution was placed in an orbital shaker in a speed of (150- 200 rpm) revolution per minute for time of (20 minutes) for KOH to dissolve in methanol and form a homogeneous potassium methoxide.

3.3.4 Weigh balance

The amount of reagent required were in the weighed using the weigh balance to find the required amount. The weigh balance is set to give the scale reading in grams. The weigh balance is more accurate because it consists of 4 digits. The weigh balance was calibrated to weigh the maximum amount 100 grams. A 50 grams of castor oil was measured in weigh balance.

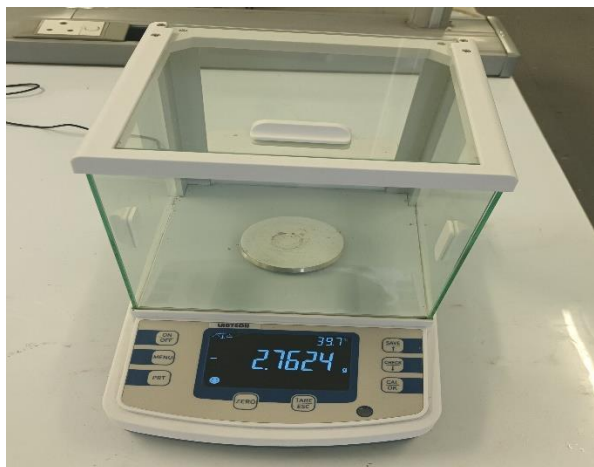


Figure 3.3: Weigh balance

3.3.5 Heating oil

Castor oil was at a room temperature which makes it more viscous and denser. To speed-up the reaction there are reactants such as castor oil which need to be heated at a temperature of 45°C – 60°C . For the reaction and mixing to take place the viscosity of oil must be decrease with heat, and the magnetic stir to move within oil.

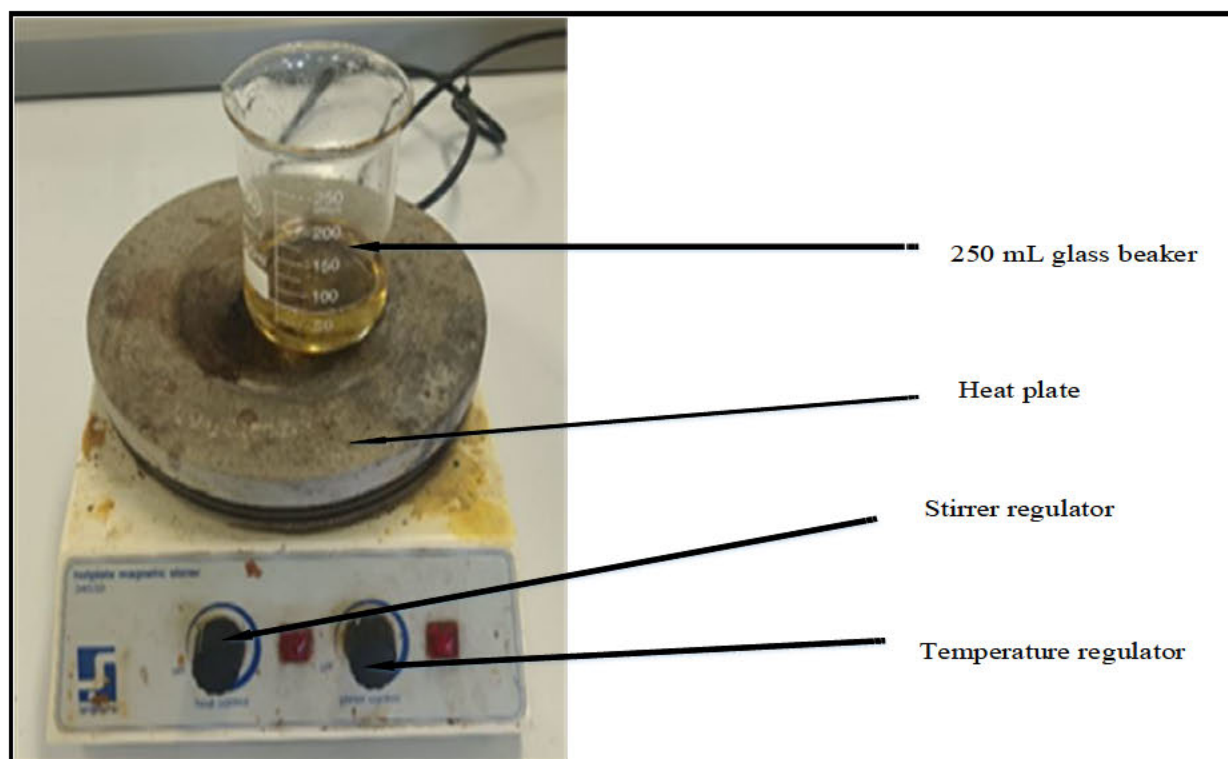


Figure 3.4: Heating plate

3.3.6 Transesterification of purified and raw castor oil

A weighed sample of (± 50 g) of oil placed in a 250 ml glass beaker to heated up to ($45 - 60^{\circ}\text{C}$) in a heat plate. A weighed sample of alcohol according to the ratio of methanol:oil, and weighed sample of KOH catalyst according to oil percentage, were placed into the orbital shaker to dissolve the KOH catalyst. A 500-millilitre jacketed reactor containing a known mass of castor oil was filled with water from a hot water bath that was equipped with an immersion temperature regulator-circulator to get the oil to the reaction temperature.

Potassium methoxide (KOCH_3) solution prepared in 100ml beaker was poured into the reactor containing purified castor oil and agitated, using a magnetic stirrer stirring at 580 rpm. A graham's condenser, connected to the jacketed beaker, was used to capture methoxide MeOH escaping from the reaction vessel as shown in Figure 3.5 and 3.6. Water bath was used to supply the required temperature to the reactor equipped with a glass and the graham's condenser on top for a cooling purpose. Water bath water supply the system with hot water at a controlled temperature. The graham's condenser was used for a condensate of reactant to return into a solution. The condenser

was left there was no evaporation occurring with the system e.g. at temperature below 45°C and above.

To manage pressure build-up the tap was 100% open for cold water to cool off the system. The reaction was performed at a vacuum pressure. The evaporation was slowly taking place because the drop was formed in 19.48 minutes (min) and second drop was formed in 28.40 minutes, third drop as formed at 39.00 minutes the fourth and fifth droplets formed at 40.50 and 60.00 minutes, respectively. This shows that evaporation rate was slowly there was no possible explosion because of a buildup pressure within the reactor that would have result in an explosion. Contrary after the first droplet, the next droplet were formed at interval of (9-10) minutes as it is mentioned. The behavior of evaporation rate at 62.5°C was the same as the one at 65°C. Tap water was used to supply a graham condenser as cooling water for in case of volatile reactants such as alcohol at particular temperature, to evaporate and be condensed within the system. The reaction was performed at a vacuum pressure to trap reactants to return to the system not to escape the system to maintain equilibrium system.

After 90 minutes of reaction time, the mixture was transferred into 250 ml conical flask to empty the reactor as shown in Figure 3.7, then poured to a separating funnel. The product in separating funnel after settling for couple of hours it then forms two layers which is biodiesel layer, and glycerol layer as shown in Figure 3.8 the bottom glycerol layer was decanted. The remaining biodiesel was cleansed with (20%v) of deionized water added and the mixture was smoothly shaken for 5 min. The mixture was then left to settle in the separating funnel for few hours. Figure 3.9 shows three distinct layers in a separating funnel. The transesterification products free of glycerol after settling for hours the washing of the sample is applied. The layers that are formed when washing product are: the bottom whitish layer consist mainly soap, bottom layer consist of water and top clear yellow layer methyl ester biodiesel (FAME) layer with a high concentration of fatty acid methyl esters biodiesel (FAME) as shown in Figure 3.10, after several wash the final washed product was weighed in the weigh balance to find the conversion percentage of methyl ester from castor oil. The washing procedure of castor oil is applied to both types of castor oil.

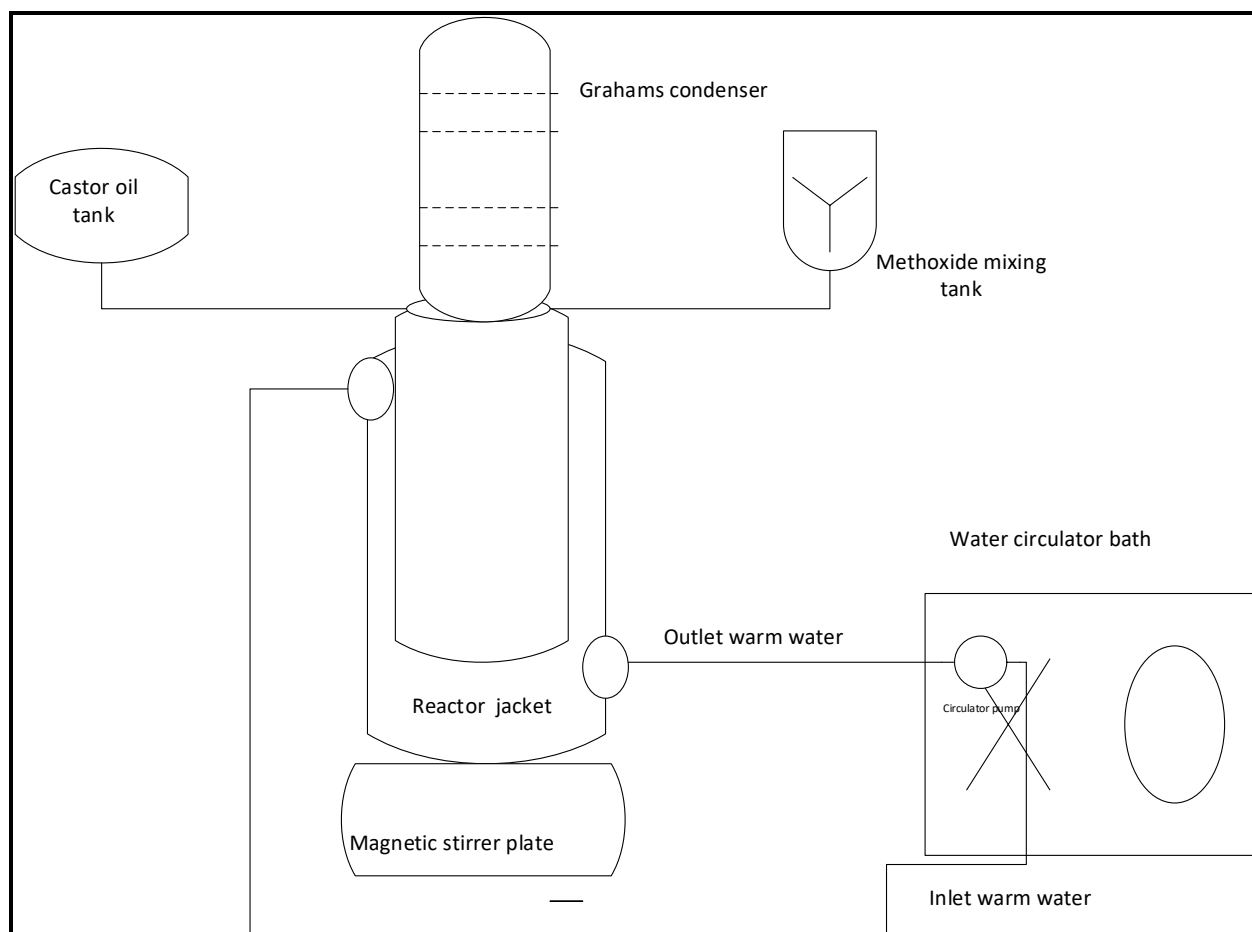


Figure 3.5: schematic diagram of experimental set-up for laboratory scale transesterification

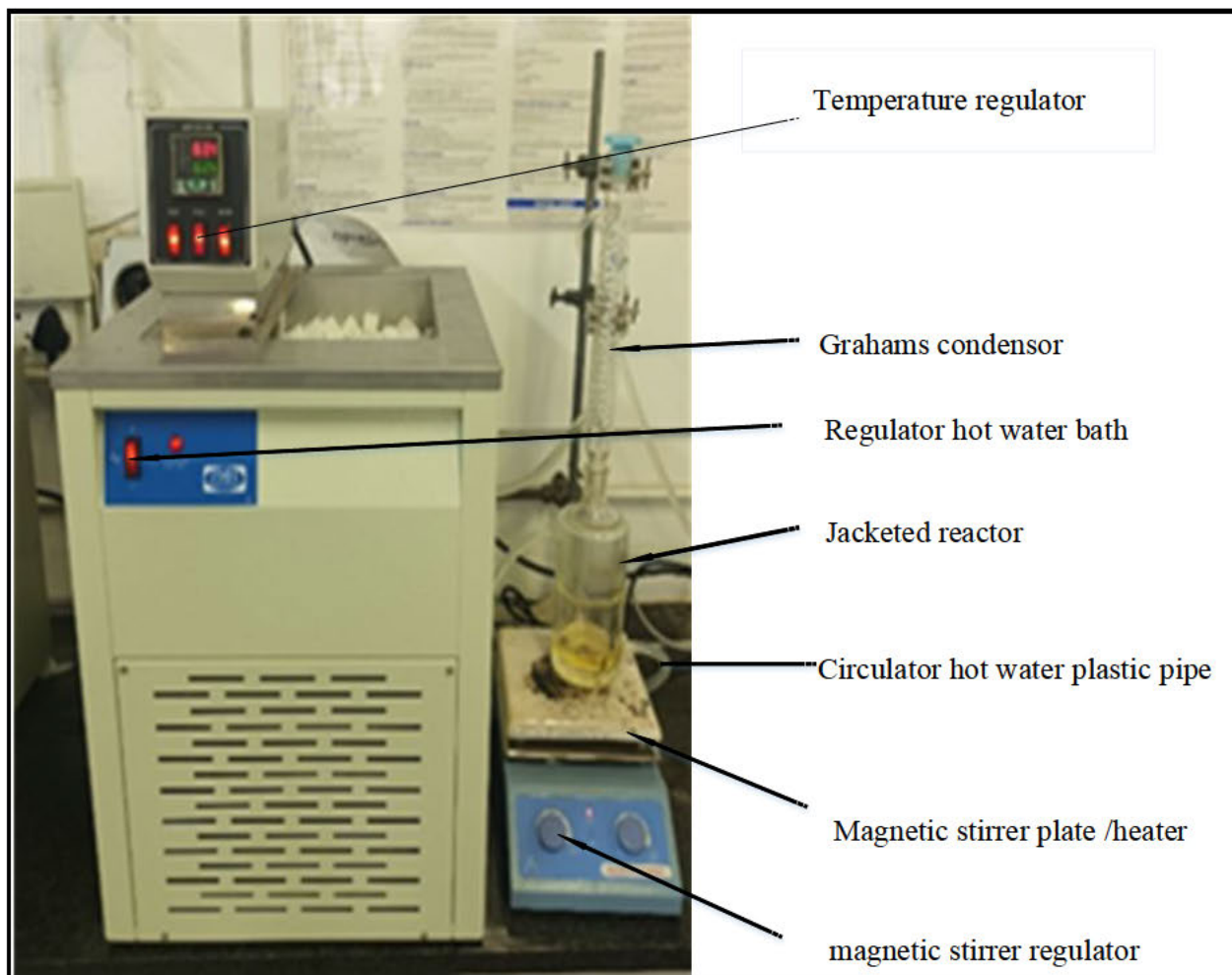


Figure 3.6: Transesterification Equipment set-up

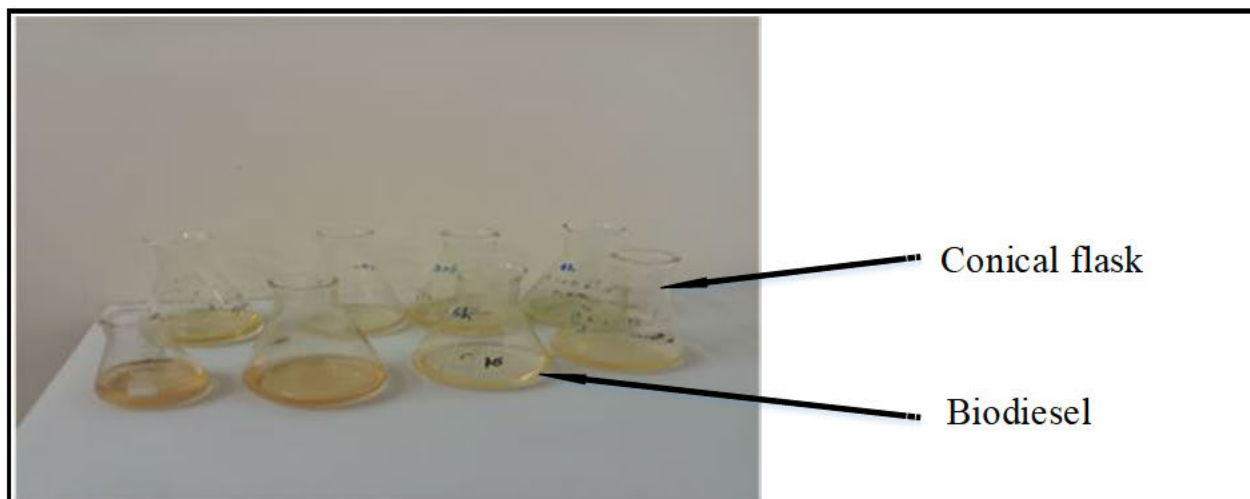


Figure 3.7: Product samples from transesterification reaction

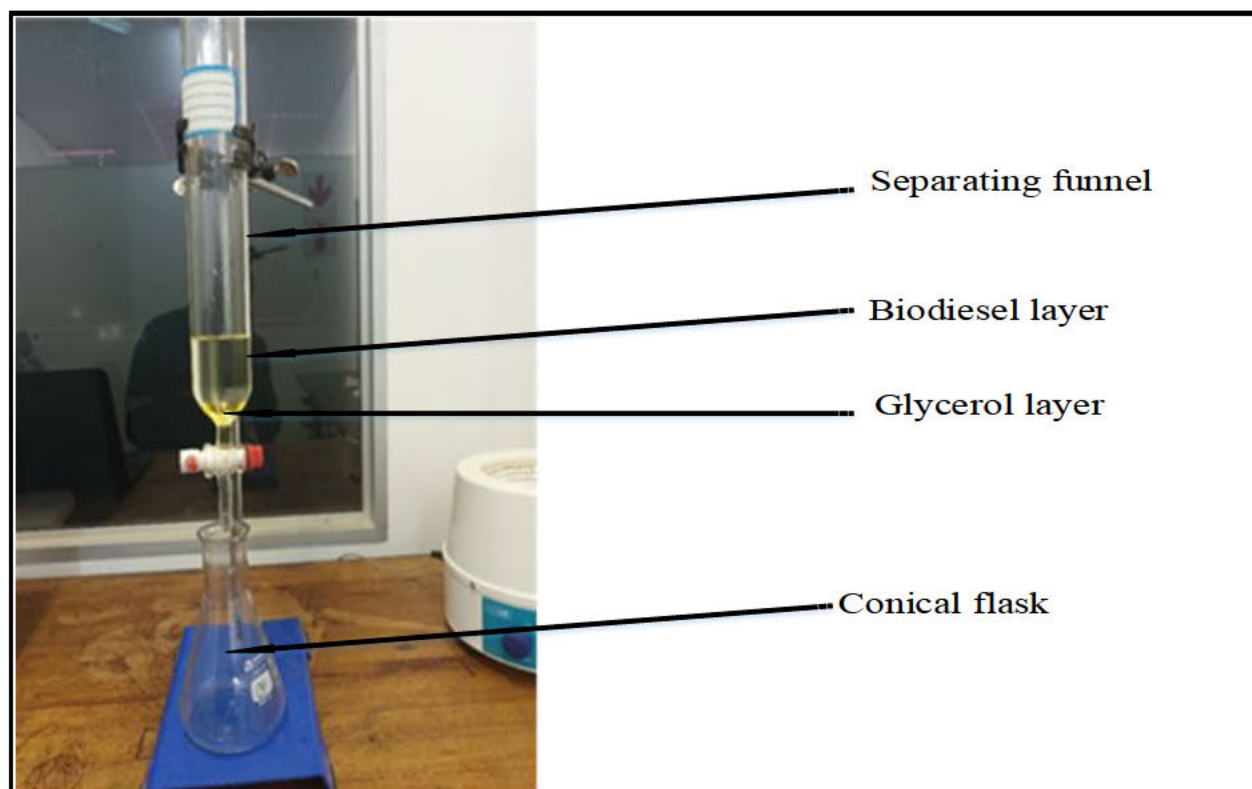


Figure 3.8: Reaction product mixture with glycerol and FAME

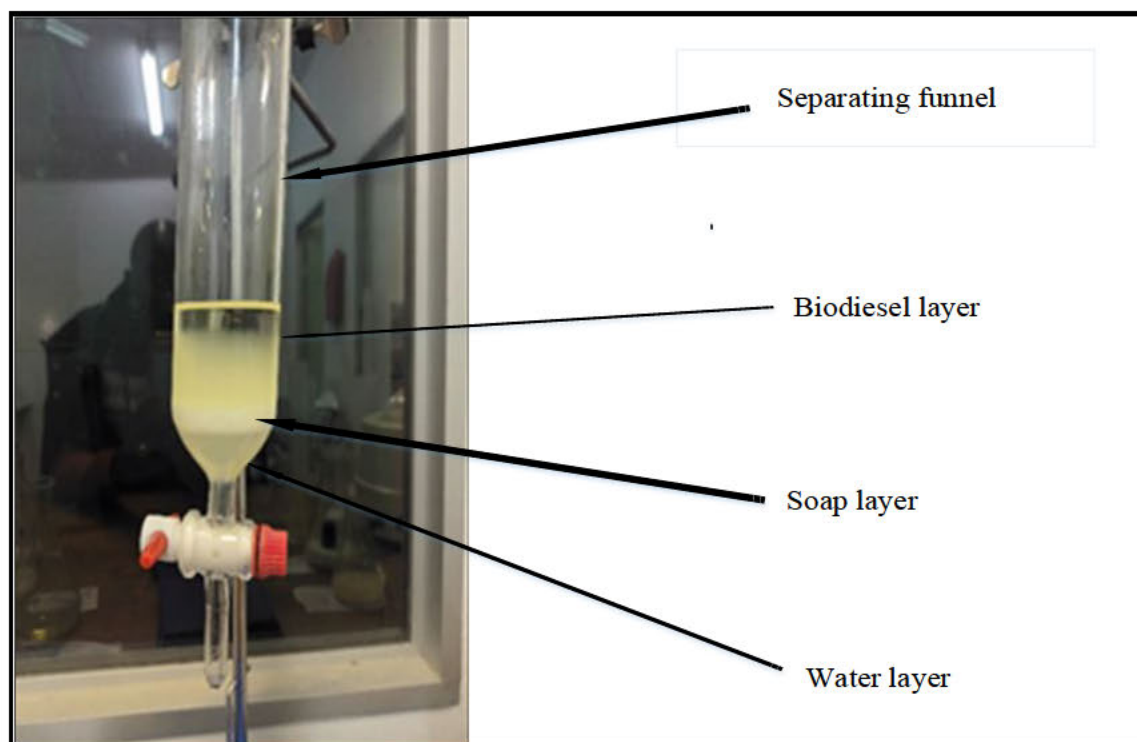


Figure 3.9: Transesterification product after 1st wash

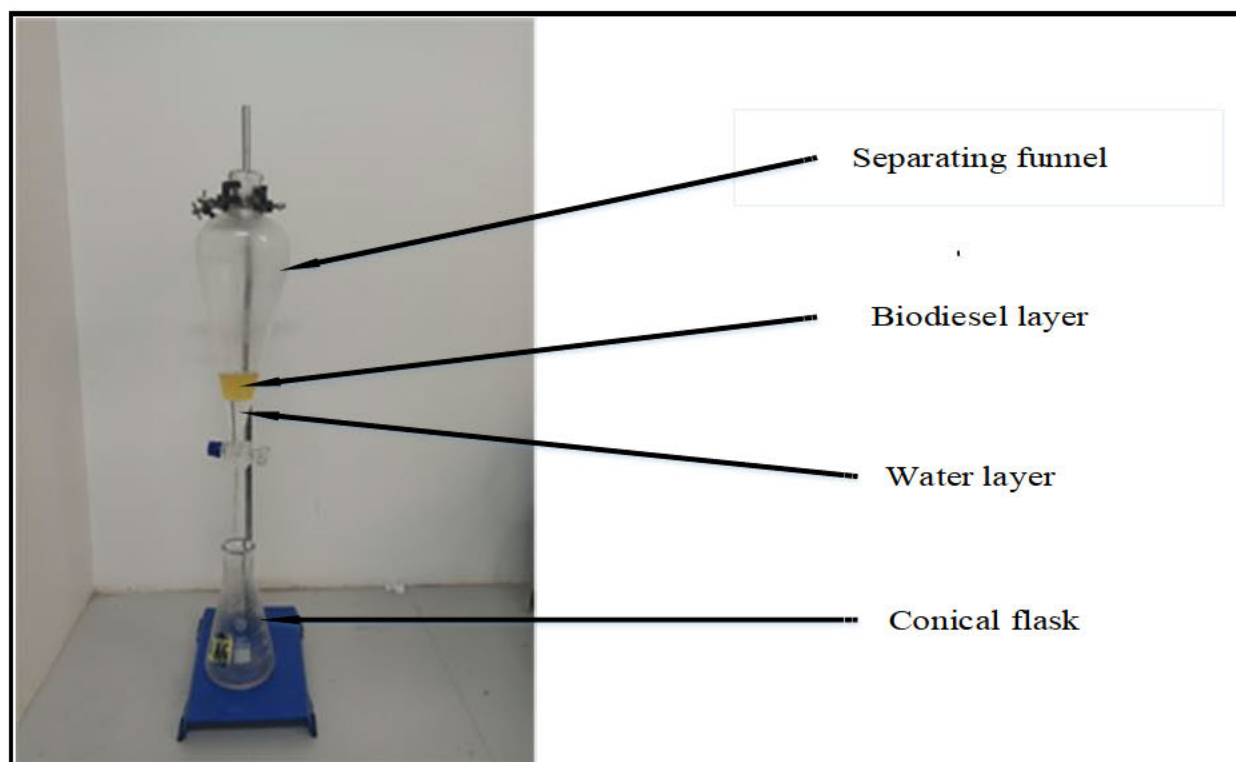


Figure 3.10: Methyl ester (FAME) sample after final wash

In final washing of the product biodiesel there were two layers formed, clear water in bottom layer, and biodiesel layer at the top. In final there was clear water without soap which is white layer that usual appear between biodiesel and water layers. This means the final product is pure, no soap formation.

The final (FAME) biodiesel product was calculated as the ratio of mass of biodiesel (FAME) divide by mass raw material castor oil (M) feed in the transesterification reaction.

$Y = \text{percentage (FAME) yield (\%)}$

$$Y = \frac{m}{M} \times 100\% \quad \text{Equation 3.3}$$

There were 22 experimental runs were conducted in the transesterification reaction using purified castor oil to find optimum conditions. The optimum conditions were investigated firstly, so that they can be applied when processing raw castor oil.

3.3.7 Methyl ester biodiesel (FAME) characterization

For auto ignition analysis of biodiesel, the Pensky-Martens closed-up tester shown in Figure 3.11 was used to determine the flash point of biodiesel in the range of 60°C–190°C, which is operated automatically. The Pesky-Martens consist of an electronic temperature measuring device, which is a thermocouple. It has been determined that electric igniters, bottled gas flames, and natural gas flames are suitable sources of ignition.

Tests must be carried out in a space or compartment that is free of drafts. Assemble the automated apparatus for use according to the manufacturer's guidelines for setting up, inspecting, and using the device.

Before beginning the test, it was ensured that every part of the test cup and all of its accessories have been thoroughly cleaned and dried. This will ensure that any solvent used to clean the apparatus has been removed. To guarantee that every specimen is removed from the test cup, the test cup is dried, and then it is covered with toluene or acetone solvent. Follow the manufacturer's directions for adjusting the flash point detection system.

The sample container was filled with 75 ml of heavy fuel oil that was up to the mark of the test cup. The test cup with the oil must be at 18°C–320°C below the expected flash point. Turn on the electric igniter and modify its power. At a temperature of 500°C, the oil ignited, which corresponded to the reported FFS refiner's operating procedure. The clean procedure was followed, and then biodiesel samples were injected to be tested.

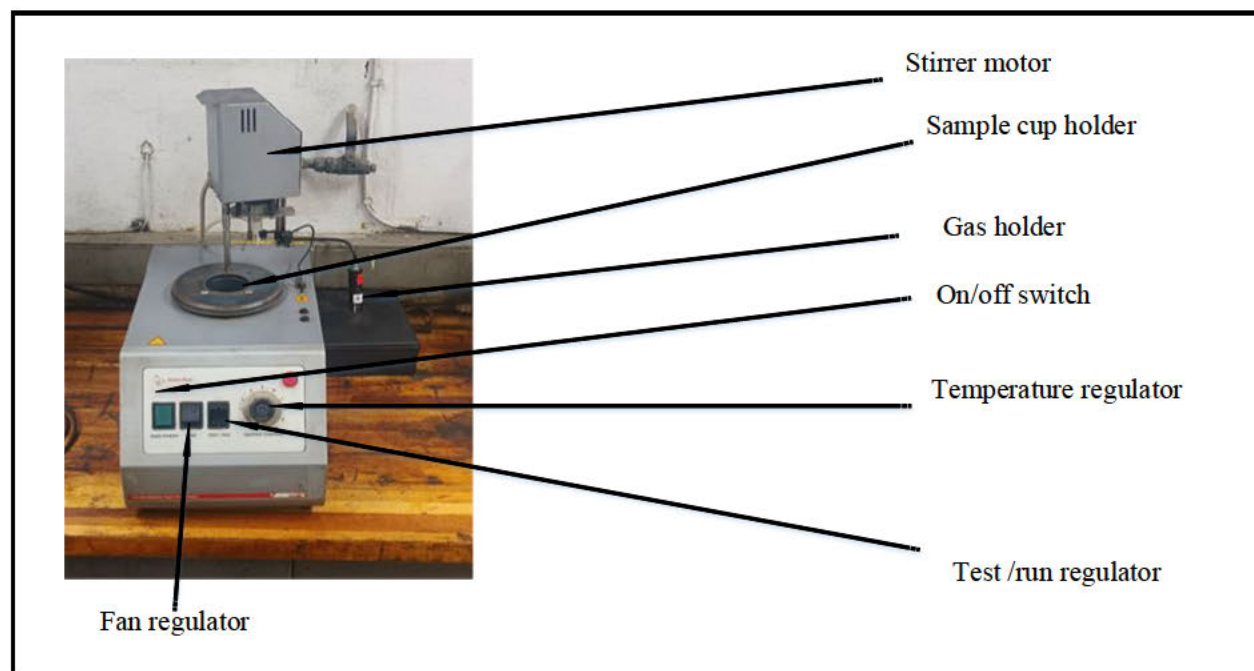


Figure 3.11: Pensky- Mortens

3.3.8 Viscometer bath

The type of viscometer commonly used for viscosity determination on petroleum products. This test setup shows how to use a calibrated glass capillary viscometer to time how long it takes for a certain amount of liquid to flow under gravity. This is done to find out the kinematic viscosity of clear and cloudy liquid petroleum products (Wolak, Zajac and Slowik 2021). This test covers a range of kinematic viscosities at all temperatures, from 0.02 mm²/s (cSt) to 300000 mm² /s (cSt). The temperature-controlled bath is calibrated with thermometers with a range from 0°C to 100°C, depending on the temperature required. A stopwatch is a timing device capable of taking readings. The viscometer-controlled bath uses transparent liquid for measurement of sufficient depth of flow time. The narrow, dry, and clean calibrated capillary (viscometer) was chosen for the type of fluid that was inserted into the bath. The oil was stirred thoroughly with a glass rod for

5 minutes, heated up to 40 °C, shook vigorously for 1 minute to ensure mixing, and charged into a viscometer. The viscometer was closed with a stopper and then immersed in a bath for 20–30 minutes to reach the required test temperature. In order for the advancing ring of contact to travel from the timing mark to the second, the stopper was removed to allow the oil to flow freely while looking at the stop watch. Then time was recorded in seconds for calculation.

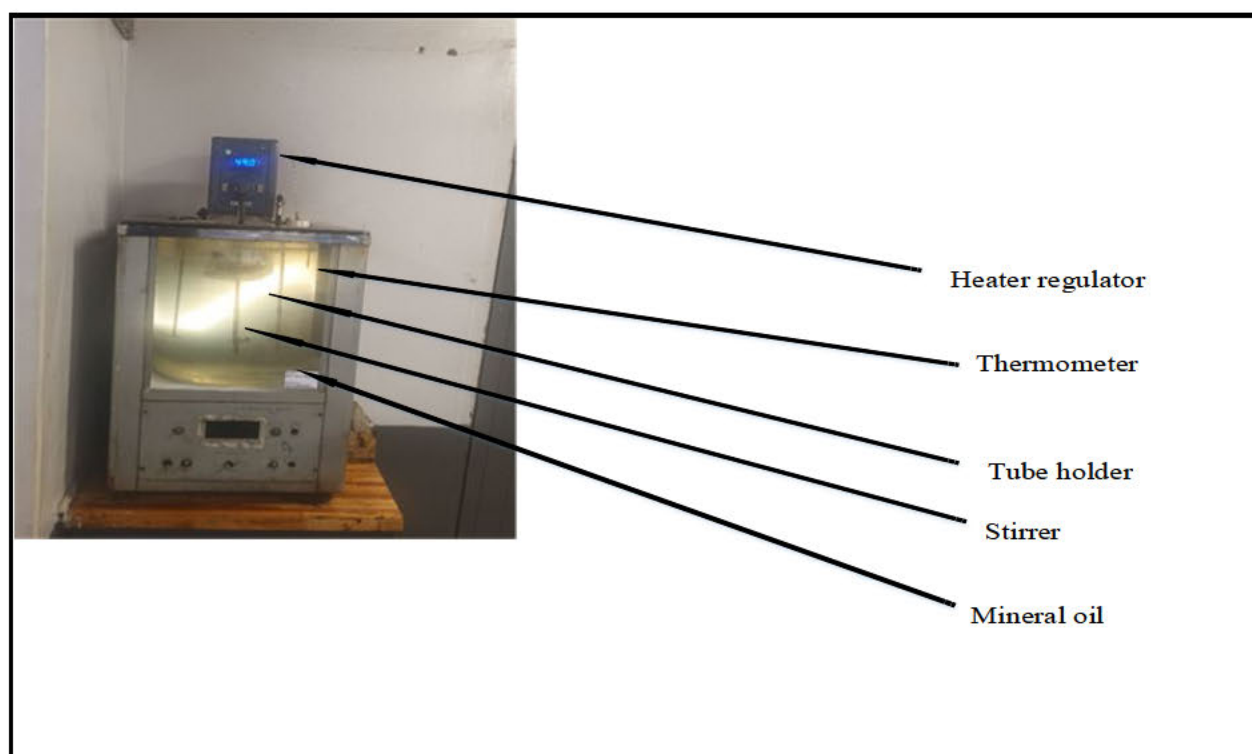


Figure 3.12: Viscometer bath

3.3.9 X-3500Ray Fluorescence for sulphur

Determination of sulphur in methyl ester (FAME) products that are in a liquid phase at ambient conditions. The Lab X-3500X-ray fluorescence was used to measure sulphur. The X-ray beam and the peak intensity were the measurements of sulphur. The X-ray beam consists of an X-ray fluorescence spectrometer, sample cups, and X-ray transparent films (Mylar L70) (Hughes *et al.* 2018). The transparent film, which is in the instrument sample holder cup, was cleaned to ensure no oil and grease. The oil was charged into the cup, and it was measured to a minimum as marked in the cup. The sample was placed into a holder cup for tray analysis, which was tray 1 in the X-ray Fluorescence, and closed, then it was chosen on the display screen. Select the sulphur range

on the screen. Once the analysis is complete, the results will be displayed on the screen, and the sample will be removed.

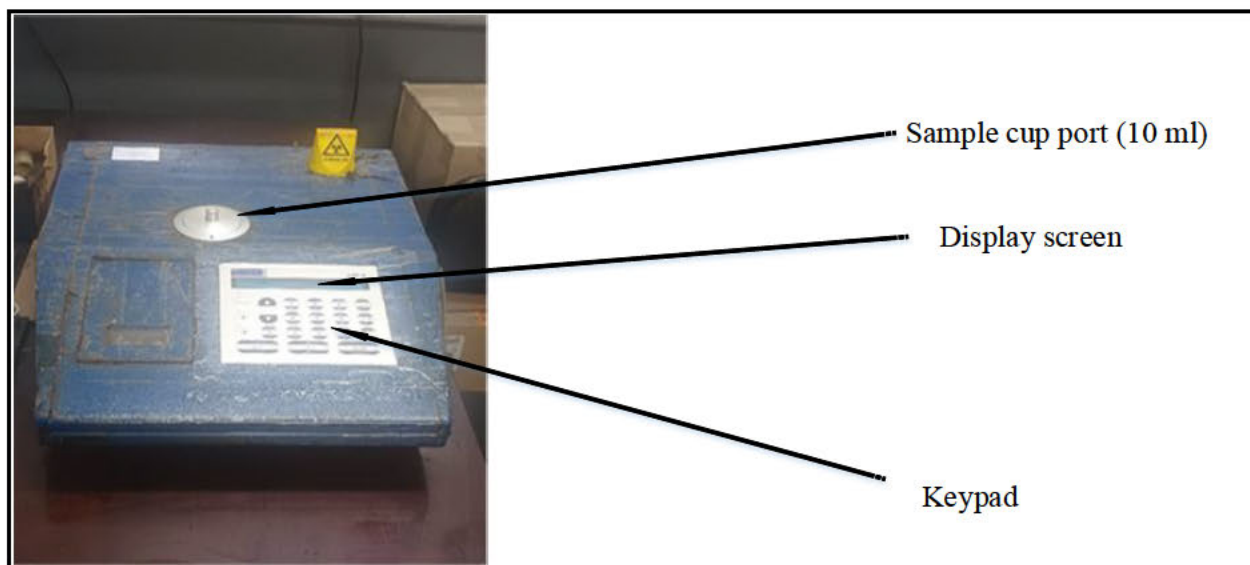


Figure 3.13: X-3500Ray Fluorescence

3.3.10 Karl fischer titrator DL31

Karl Fischer titration in Figure 18 was used to test water content present in the biodiesel sample. Are they within the limits that are acceptable for biodiesel? The determination of water content in a fuel oil sample The Karl Fischer can only be efficient in samples with a lower water content ranging from (0.005–30% v/v). Once the titration is complete, the result will be displayed on the screen (water mass %).

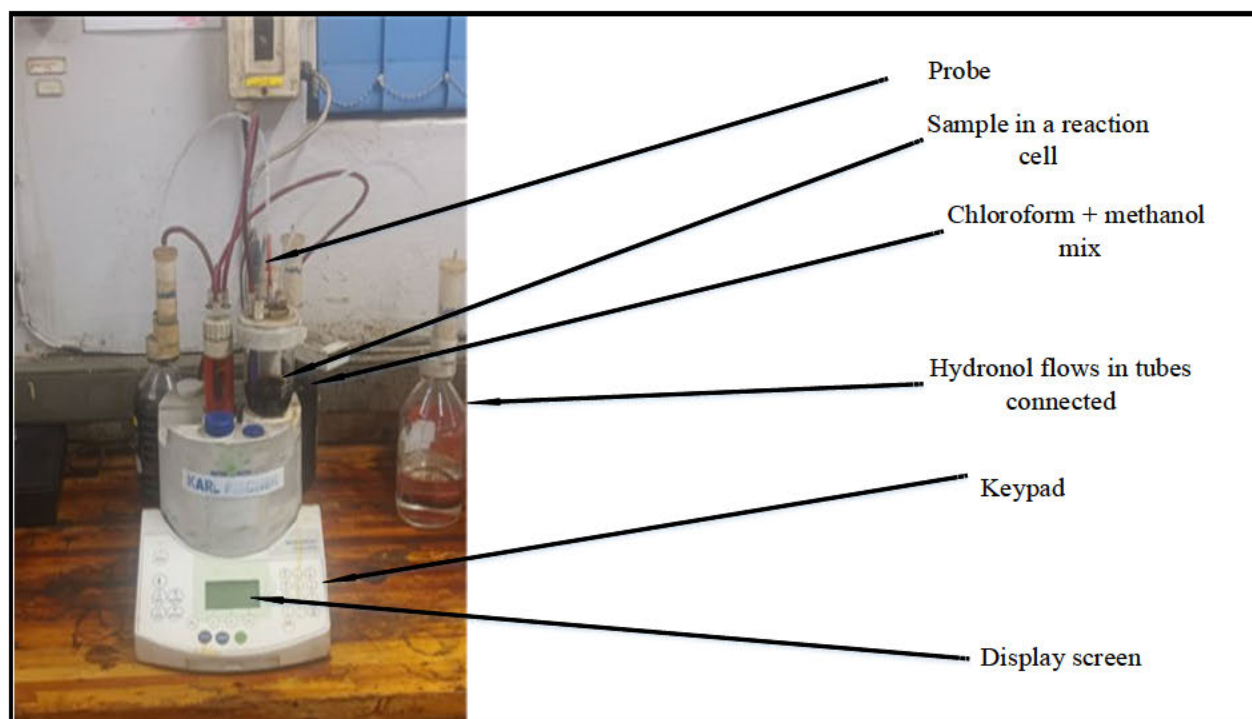


Figure 3.14: Karl Fischer

3.3.11 Furnace

The electric muffle furnace operates at a temperature of 775°C to 800°C, which is preferable (Roushdy 2022). The sample was charged into the crucible at approximately 90 minutes minimum to 150 minutes maximum capacity, whereas 150 ml of the maximum amount of sample, which is 1255 g, was weighed in the analytical balance. The sample was charged into the furnace to heat up to 700°C for 1 hour, then allowed to cool to ambient temperature in a desiccator for 1 hour. The ash was weighed after it had cooled. Then it was calculated as follows:

$$\text{ash mass} = \frac{\text{total weight} - \text{crucible}}{\text{sample weight}} \times 100$$

Equation 3.4

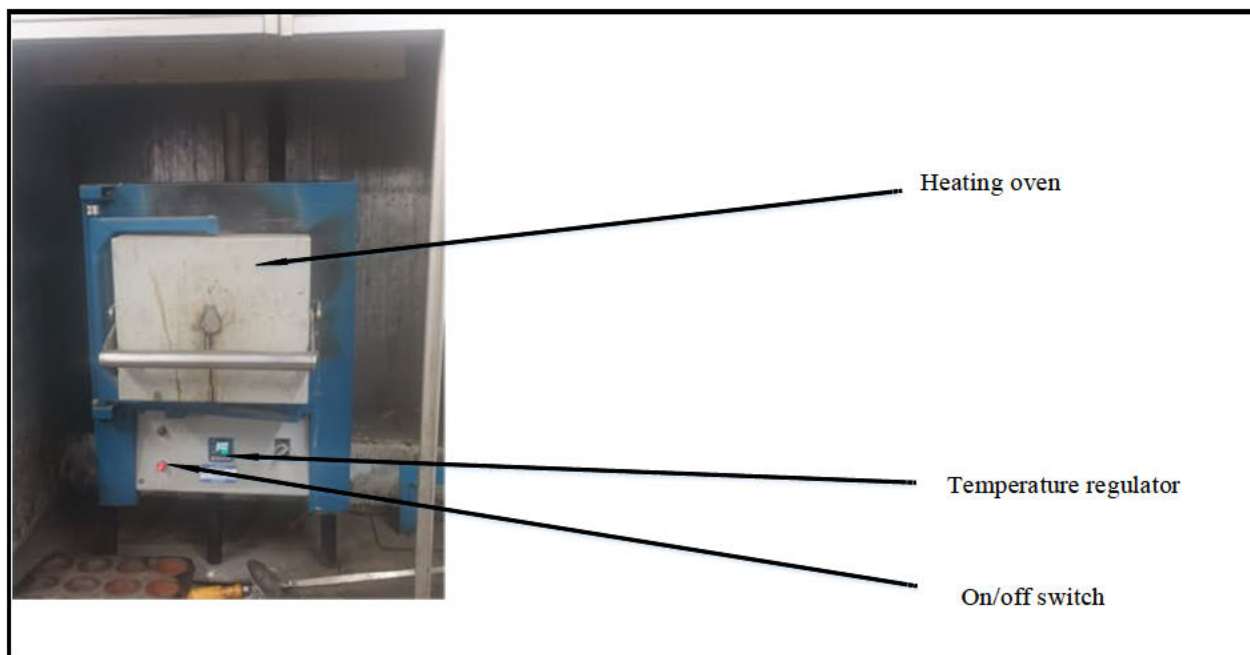


Figure 3.15: Electric furnace

3.3.12 Refractive index and density

The DMA 4100 M, which has a measurement range of 1.26 to 1.72 nD and a temperature range of 4°C to 125°C, is shown in Figure 3.16. It has an accuracy of 0.0002 nD. With precision, a high accuracy of up to 0.03°C is possible. Peltier temperature control inside. The ideal bench is temperature stabilized and sealed to shield it from outside influences. Distilled water was used to initially remove any filth or residue that had been left inside the pipe system before calibrating the DMA 4100 M. Before starting to calibrate, the built-in fan was opened to blow air across the path to dry it out. The next step was to add 15 mL of toluene to a syringe which was injected into the density meter's inlet, and the measurement for the distilled toluene at room temperature was 866.9 kg/m³, which is in accordance with the information provided by Verma and Pramanik (2023).



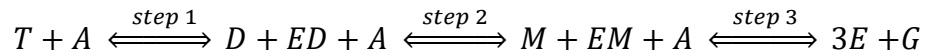
Figure 3.16: Anton Paar DMA 4100 M refractometer with density meter

3.4 Kinetic models of transesterification reaction

The transesterification reaction's kinetics have been studied theoretically. One of the objectives is to do biodiesel kinetics, therefore, this section present the procedural. The process of making biodiesel involves mixing triglycerides and free fatty acids (FFA) from vegetable oils and animal fats with alcohol while an enzyme, an acid, or an alkaline catalyst is present (Feyzi *et al.* 2017; Jayaraman *et al.* 2020; Li *et al.* 2020). According to stoichiometry, 1 mol of triglycerides and 3 mol of alcohol react to yield 1 mol of glycerin and 3 mol of fatty acid methyl ester (biodiesel). The kinetics of transesterification reactions can be modelled using many kinds of simple empirical kinetic models. Pseudo-First Order (PFO) (Ramezani, Rowshanzamir and Eikani 2010; Roschat 2019), Second-Order (SO) (Feyzi *et al.* 2017), and Pseudo-Second-Order (Darnoko and Cheryan 2000; Gao *et al.* 2019) models are the most fundamental and commonly utilized empirical kinetics modes of this reaction in the literature . These kinetic models were all put forth using empirical data. Additionally, because incomplete triglyceride conversions occur during the reaction period, kinetics models integrating reversible Second-Order are commonly utilized. (Phoopisutthisak, Prasertsit and Tongurai 2019). It is observed that the reaction kinetics initially follows the pseudo-

first-order kinetics model, then transitions to the pseudo-second-order model, particularly in the later phases of the reaction (Csernica and Hsu 2013). Furthermore, it has been noted that transesterification reaction kinetics depart from the Second-Order-Model, particularly in the reaction's later phases. Additionally, when the reaction is spontaneous ($\Delta_r G \ll 0$ conditions or at short initial times of reaction), a modified version of the Second-Order model (MSO) is derived (Csernica and Hsu 2013; Ezzati, Ranjbar and Soltanabadi 2021). The glycerol effect is the cause of this deviation, according to Ezzati, Ranjbar and Soltanabadi (2021) studied, this deviation is caused by the glycerin byproduct, which lowers the solubility of triglyceride in methanol and reduces mass transfer of triglyceride into methanol, thereby inhibiting the rate of the transesterification reaction by limiting the triglycerides from diffusing into the methanol phase from the oil phase (Ezzati, Ranjbar and Soltanabadi 2021).

3.4.1 General Rate Equation (GRE)



The symbols A, T, D, M, G, and E stand for alcohol, triglycerides, diglycerides, monoglycerides, glycerine, and methyl ester, respectively. The transesterification reaction system initially consists of two phases since methanol and oil are immiscible. In this case, mass transfer controls the reaction's kinetics in the early stages. During the beginning of the reaction and for the duration of the reaction time range, the chemical reaction stage regulates the action stage. This holds true for the early phases of the reaction as well as the transesterification reaction rate (Ezzati, Ranjbar and Soltanabadi 2021). When the impeller speed at the transesterification reaction is at least 600 rpm, it has been found that the mass transfer at the first step of mass transfer control is tiny (Ezzati, Ranjbar and Soltanabadi 2021). As a result, it appears that transesterification happens in a single phase. According to reports, the reaction of triglyceride and methanol transesterification is slower than that of diglyceride and monoglyceride with methanol. Because of this, the rate may be ascertained in step 1, and the rate equation is predicated on the assumption. According to Step 1, the rate equation can be represented as follows:

$$r = k_1[T][A] - k_{-1}[D][E] \quad \text{Equation 3.5}$$

Where the concentrations of triglyceride, alcohol, diglyceride, and methyl ester are, in order, [T], [A], [D], and [E]. Additionally, step 1's rate constants are k_1 and k_{-1} . It is assumed that the concentrations of the reaction's intermediates, monoglyceride and diglyceride, are near their equilibrium concentrations.

$$r = k_1[T][A] \left[1 - \frac{Q}{K} \right] \quad \text{Equation 3.6}$$

$$K = k_1 k_2 k_3$$

The overall reaction Q, reaction quotient. Where the concentrations of triglyceride, alcohol, in order, [T], [A]. Additionally, step 1's rate constants are k_1 and K is the overall reaction,

$$Q = \frac{[G][E]^3}{[T][A]^3} \quad \text{Equation 3.7}$$

Where the concentrations of triglyceride, alcohol, diglyceride, methanol and glycerol are, in order, [T], [A],[E] and [G]. The equilibrium constant of the overall reaction K.

$$K = k_1 k_2 k_3$$

3.4.2 Pseudo- first-order model (PFO)

Assumptions

- High initial concentration of alcohol.
- Value of Q is approximately zero

$$r = k_{PFO}[T]$$

Where the concentration of triglyceride is [T]

Where k_{PFO} is the PFO is rate constant and is defined as:

$$k_{PFO} = k_1[A]_0 \quad \text{Equation 3.8}$$

Initial concentration of alcohol is $[A]_0$ and reaction rate k_1

3.4.3 The Second-order model (SO)

Assumptions

- High initial concentration of alcohol.
- Value of Q is close to zero

$$r = k_{so}[T][A]$$

Equation 3.9

Concentration of triglyceride and alcohol are in order [T] and [A]

Where k_{so} is the SO rate constant and is defined as:

$$k_{so} = k_1$$

3.4.4 Modified-Second-order model (MSO)

$$r = k_1[T][A]e^{(-\frac{Q}{K})}$$

Equation 3.10

k_1 is a rate constant, Q is reaction quotient, K is the overall rate constant, and the concentration of triglyceride and alcohol are in order [T] and [A] respectively

Since the transesterification reaction is known to significantly deviate from the SO kinetics model, this equation has been presented to investigate the inhibitory influence of glycerol on the reaction. For both the initial phase and the end of the reaction, GRE and MSO kinetic modelling work effectively (Ezzati, Ranjbar and Soltanabadi 2021).

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Introduction

The presentation of results for the study, homogeneous transesterification process, which produces biodiesel (methyl ester) from purified or commercial castor oil (CCO) and raw castor oil (RCO). Parameters such as reaction temperature, catalyst concentration, and alcohol:oil ratio during a 90-minute contact period were manipulated for the production of biodiesel. One of the goals achieved in the study was to investigate how these factors affect the amount of biodiesel produced.

4.2 Purity and acid value

Prior the experiments, the purity of all chemicals used in this study were checked to ensure that there is no contamination.

The acid value of castor oil depends on the density. The effect of acid value is to decrease or increase the biodiesel yield. It determines the formation of byproduct which is soap. Soap is easily formed on products when the acid value is greater than 4. Table 4.1 presents the comparison of acid values of raw and commercial castor oil with literature and the absolute relative error for accuracy.

Table 4.1: Comparison of acid value (AV) of raw and CCO with literature

| | AV of RCO literature | AV of RCO measured | Reference | AV of CCO literature | AV of CCO measured | Reference |
|-------------------------|----------------------|--------------------|--------------------------------------|----------------------|--------------------|----------------------------------|
| Acid Value | 0.25 | 0.4 | (Osorio-González <i>et al.</i> 2020) | 2 | 1.03 | (Akpan, Jimoh and Mohammed 2006) |
| Absolute Relative error | 0.15 | | | 0.97 | | |

$$AR = |AV_{lit} - AV_{meas}|$$

Equation 4.1

In the presentation of the results obtained in this study, the one factor at a time (OFAT) experimental method was used. The OFAT experimental method is used because it does not require substantial statistical expertise to comprehend the results collected. The experimental method is the method that is often used for experimental design. Biodiesel was produced in a batch process using two types of castor oil: raw and purified or commercial castor oil (CCO). A jacketed reactor performed dual activities: mass transfer and keeping the reaction temperature constant.

4.3 Parameters considered for investigation

The number of parameters, some are dependent variables and others independent variables. These parameters that have an impact on the generation of biodiesel. The following parameters listed below were investigated in the study using the homogenous transesterification experimental method in Chapter 3 to find the optimum conditions for the conversion of castor oil to FAME on a laboratory scale. The parameters are:

- Reaction temperature
- Catalyst concentration
- Methanol:oil molar ratio

The CCO was carried out experiment at the specified range of parameter in Table 3.1 in Chapter 3. The repetition of experiments were done to ensure the accuracy and consistency of results. The CCO was used to find optimum conditions. The optimum conditions were applied to raw castor oil (RCO), which means RCO was measured at optimum condition. The RCO used to perform experiments was produced in the same laboratory by Mkhize, (2023) and Mkhize et al. (2023). RCO was produced from castor seeds or castor beans. Therefore, the product of RCO was converted further to produced biodiesel in this study.

4.4 Effect of process parameters in biodiesel production

The yield of biodiesel is greatly influenced by the heat and mass transfer of the reactants, alcohol, and castor oil. The mass transfer of transesterification reaction is a concern due the fact that raw materials which is methanol and vegetable oil are immiscible. The many physical and chemical characteristics of the raw materials have a significant impact on the final product of biodiesel.

According to this study, the production of biodiesel is greatly influenced by reaction temperature, the methanol-to-oil ratio, and catalyst concentration. Some of crucial process parameters were discussed and considered to produce biodiesel more sustainably and economically in the previous section (Kargbo, Harris and Phan 2021).

4.4.1 Effect of reaction temperature

Temperature has an impact on how biodiesel is produced. Depending on the raw material's chemical and physical properties. The transesterification process can be performed at a different temperatures (Ahmad *et al.* 2020). According to several studies, the recommended range of temperature is 50–70°C, but at the temperature of 70°C, there is a risk of alcohol (methanol) is above boiling point and it can evaporate.

Measurements in this work were carried out at the reaction temperature range 45 to 70°C. The range was selected based on Keera, El Sabagh and Taman (2018) reaction temperature range (30 – 60°C), which indicates that even at lower temperatures there will be a production of biodiesel from feedstock castor oil. The yield has been calculated. Figure 4.1 shows the effect of reaction temperature, the increase in temperature decrease the biodiesel yield due to that as the reaction temperature increases methanol reaches the boiling point, as the results it begins to evaporate which favors the reverse reaction. The optimum reaction temperature was found using commercial castor oil (CCO). It was found to be at reaction temperature of 45°C and yield was 97.9% biodiesel at optimum temperature. The measurements were repeated three times and average value was recorded. The raw castor, which was produced in the same laboratory by Mkhize (2023) was measured at optimum. The yield obtained was 98.49 % biodiesel using the same optimum conditions obtained on commercial castor oil. The biodiesel yield from raw castor oil is higher than that of commercial castor oil, which shows that the acid value has the impact. The acid value for commercial castor oil is greater than that of raw castor oil which can be the caused by extraction process of raw castor oil.

The optimal reaction temperature is frequently close to the alcohol boiling point, according to Keera, El Sabagh and Taman (2018). According to Tabatabaei *et al.* (2019) at the very least, it is conceivable that no economically viable reaction could be carried out below 32°C. However,

temperature is another essential factor that will impact the production of biodiesel. As illustrated in Figure 4.1, an experiment was conducted in the reaction temperature range 45 to 70°C.

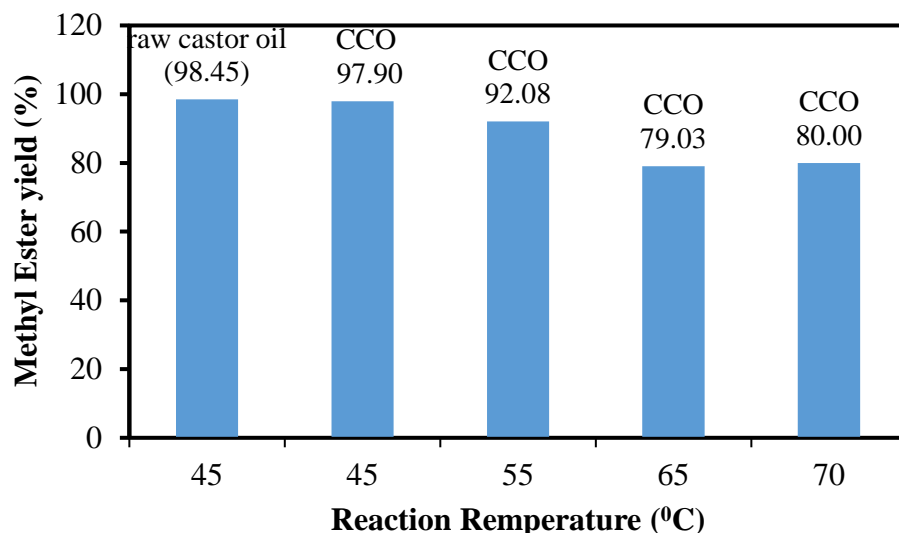
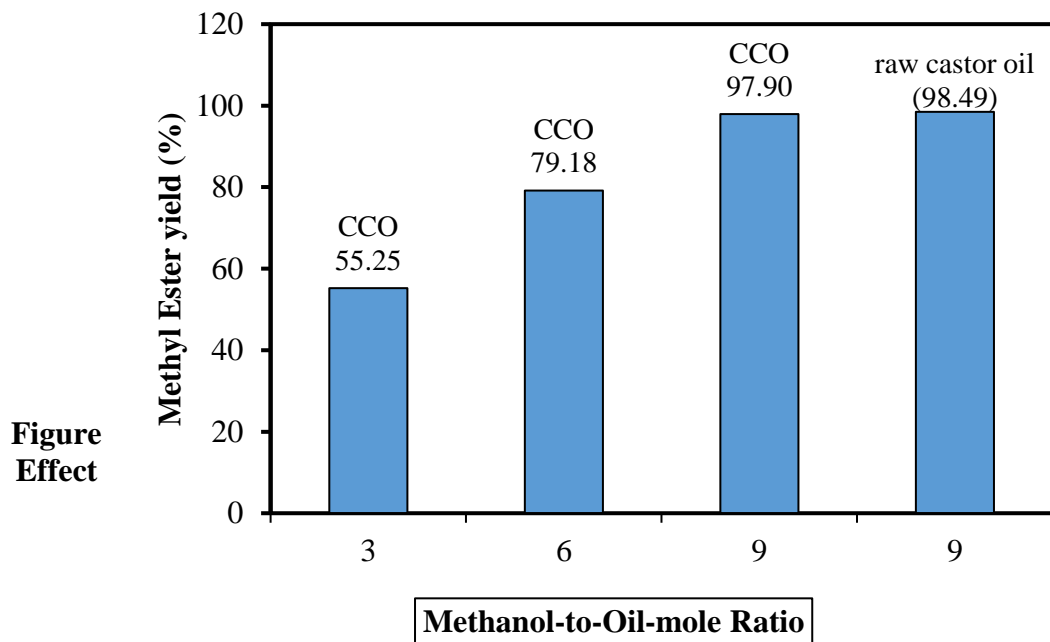


Figure 4.1: Effect of reaction temperature on methyl ester yield

4.4.2 Effect of methanol to oil molar ratio

The molar ratio of alcohol to oil is an essential parameter that influences the conversion efficiency and production of biodiesel (Musa 2016). Since the transesterification reaction is reversible and the stoichiometric molar ratio of alcohol to oil is 3:1, higher molar ratios are required to enhance miscibility and the interaction between alcohol molecules and triglycerides. In fact, the molar ratio needs to be greater than the stoichiometric ratio in order to shift the reaction towards completion (Musa 2016). Moreover, excess methanol is required during the transesterification of triglycerides into biodiesel in order to break the glycerin-fatty acid bonds (Wilson *et al.* 2019). Greater alkyl ester conversion occurs in a shorter time at higher alcohol-to-oil molar ratios (Musa 2016). For the purposes of the present study, the transesterification reaction was conducted using the stoichiometric ratio, requiring for 9 mol of methanol to produce 1 mol of glycerol and 9 mol of methyl ester. Because the reaction is reversible, more methanol is needed to get it to move in the direction of the products. As seen in Figure 4.2, the methanol:oil ratio was varied during the process, ranging from 3:1 to 9:1. In every experiment, the reaction time was ninety minutes. The catalyst and reaction temperature remained at 45°C and 0.5% wt. KOH, respectively. The methyl

ester biodiesel yield for molar ratio 3:1 is 55.5 wt%; however, the amount of methanol concentration did not complete the phase separation. When the methanol ratio was raised to 9:1, it was easy to separate the products of the transesterification reaction, and 97.9 % methyl ester biodiesel (FAME) was produced. When decreasing the methanol:oil ratio to 3:1 or 6:1, the yield decreases from 79.18% and 55.25%, respectively, this is due to the conglomeration of methanol. The optimum conditions were obtained at the ratio of 9:1 and yield of 97.9%. The same ratio was applied to raw castor oil, the methyl ester biodiesel (FAME) yield increases to 98.49% and 98.19%, respectively. Reaction towards completion: Musa (2016) states that the molar ratio should be greater than the stoichiometric ratio.



methanol:oil mole ratio on methyl ester yield

4.3.3 Effect of catalyst concentration

In this study, it is noted that the catalyst concentration plays a significant role in the yield of methyl esters in a range of (0.625–3.125 wt%) while maintaining the temperature, methanol:oil molar ratios of 45°C and 9:1, respectively. The reaction time was constant for 90 minutes throughout the experiment. It has been found that in the castor oil reaction, a lower catalyst concentration of 0.625 wt% KOH is sufficient to produce a high yield of methyl ester biodiesel (FAME). When the catalyst concentration is 3.125 wt% KOH, the methyl ester yield is 69.6 wt%. Hence, the increase in catalyst concentration decreases the yield and favors the byproduct, which is a high yield of soap and water. The results show that the high concentration of an alkaline catalyst favors the saponification reaction. On the contrary, 0.625 wt% catalyst concentration was considered to be the optimum catalyst concentration for the study. As mentioned in the study of Keera, El Sabagh and Taman (2018), increasing the concentration of catalyst decreases the biodiesel yield as presented on Figure 4.3.

According to Keera, El Sabagh and Taman (2018) methyl esters were treated with different amounts of catalysts (0.5-2 wt%) and reaction times (60 minutes), but the temperature and molar ratio of methanol to oil were kept constant at 60 °C and 9:1. A 65% biodiesel yield was obtained, showing that a lower catalyst concentration of 0.5 wt% KOH is insufficient for the entire reaction. They also noticed that the biodiesel production improved to 95% when they increased the catalyst concentration to 1 wt%. Therefore, it is found that the yield of biodiesel decreases when the catalyst concentration increases. The reason for this behaviour is the high concentration of alkaline catalyst, which promotes the saponification reaction and results in the generation of water molecules and soaps as byproducts (Maheshwari *et al.* 2022). Furthermore, The optimal catalyst concentration is considered to be 1 wt% (Maheshwari *et al.* 2022).

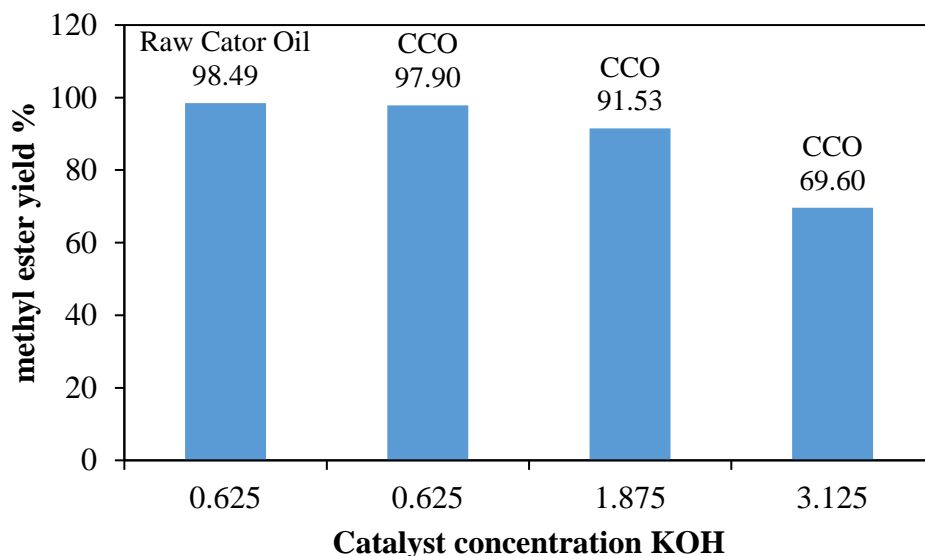


Figure 4.3: Effect of catalyst concentration of methyl ester yield

4.4 Methyl ester (FAME) characterization

One of the objectives is met, which is to characterize the product (methyl ester) to ensure that is biodiesel. The product was compared with the standard. The American Society for Testing and Materials (ASTM) specified test procedures to evaluate the physical and chemical properties of the produced biodiesel. Table 4.2 listed the outcomes of characterizations, the absolute average deviation was calculated for each property and measured in reference to previous recent studies for verification. The following equation was used for absolute average deviation.

$$AAD = \left| \frac{P_{lit} - P_{meas}}{P_{lit}} \right| \quad \text{Equation 4.2}$$

AAD absolute average deviation. Where P_{lit} stand for the parameter/ variable in the literature and P_{meas} is the parameter/ variable measured in study.

Table 4.2: Methyl ester (FAME) characterization

| property | Test method | South African National Standard (SANS) specification limits | Refine castor oil results | Raw castor oil results | Absolute average deviation (AAD) Raw castor oil | Absolute average deviation (AAD) purified castor oil | References |
|----------------------------------|-------------|---|---------------------------|------------------------|---|--|---|
| Kinematic viscosity (cSt) @ 50°C | ASTM D445 | 1.9-6 | 2.98 | 1.98 | 0.003 | 0.03 | (Attia, Nour and Nada 2018) |
| Ash (g) | ASTM D93 | - | - 0.005 | 0.003 | - | - | - |
| Flash point °C | ASTM D93 | 130 max | 116 | 114 | 0.70 | 0.40 | (Chidambaranathan <i>et al.</i> 2020) |
| Sulphur (ppm) | ASTM D5453 | 10 max | 0.65 | 0.64 | 0.36 | 0.35 | (Ayodeji 2018; Chidambaranathan <i>et al.</i> 2020) |

| | | | | | | | |
|---|--------------------|-----------|-------|-------|-------|-------|---|
| Density kg/m ³ at 25°C | ASTM D1298/4052 | 860-900 | 960 | 920 | 0.01 | 0.032 | (Attia, Nour and Nada <u>2018</u>) |
| Water content % vol. at 25°C | ASTM D2709 | 0.005 max | 0.04 | 0.05 | 0.00 | 0.20 | (Attia, Nour and Nada <u>2018</u> ; Chidambaranathan <i>et al.</i> <u>2020</u>) |
| Calorific Value kJ/kg | ASTM D6751 | 44121 | 44121 | 44121 | 0.003 | 0.003 | (Etim <i>et al.</i> <u>2022</u>) |

The outcomes indicate that the methyl ester biodiesel (FAME) produced using a jacketed reactor meets biodiesel specifications and ASTM standards. There are many characteristics of biodiesel, but the ones that directly affect the engine's performance, such as viscosity, flash point, density, calorific value, etc. are the most crucial. All of these characteristics help the engine last longer, provide better lubrication, and enable complete combustion so that the engine can generate more energy (Jain and Sharma 2010).

4.4.3 Calorific Value

When it comes to the performance of biodiesel in engines, calorific value is one of the most crucial properties. The quality of the heat energy generated by burning a unit of fuel is what is meant to be understood. Because the calorific value rises with increasing chain length and decreases with increasing unsaturation, it is essential for estimating fuel consumption. The consumption of fuel decreases with increasing calorific value. Since the calorific value of methyl ester biodiesel (FAME), 44212 KJ/kg, is virtually the same as the calorific value of conventional diesel. 44000 KJ/kg (Kargbo, Harris and Phan 2021) this confirms that the product produced was biodiesel, and the percentage difference is 0.275% which emphasis the accuracy of the product.

4.5 Kinetic of the reaction results

PSO and SO mechanism with assumptions kinetic parameters were obtained using the PSO and SO transesterification homogenously catalyzed alkaline catalyst. Four kinetic models were considered for homogenous catalyzed transesterification.to determine the kinetic of the product. To evaluate the accuracy of GRE results for transesterification reaction of methanol to oil molar ratio 9:1 were used to evaluate the accuracy of the transesterification reaction. The relative error and absolute error was not calculated for GRE and PSO. The models are said to be suitable for the entire reaction.

The kinetic results obtained for SO and PSO kinetic models exhibit a minor difference when calculating reaction rate. The difference of the two kinetic models is that SO kinetic model is suitable at short initial time only while, PSO is suitable for the entire reaction. Since there minor difference between the two kinetic models reaction rate values. Therefore they are suitable for

different conditions it wouldn't make sense to calculate absolute error and absolute error for both models. The four transesterification reaction kinetic models considered for the study beside the different conditions suitable for to consider the reaction rate in all 4 of them. GRE reaction rate at the temperature of 318.15 K is high followed by PSO, while SO and MSO are almost the same. The difference begins as the temperature increases from 328.15 K -343.15 K the reaction rate for GRE is smaller than all, whereas reaction rate for MSO and SO greater than all followed by PFO. Temperature increases with the decline GRE reaction rate and also increases with the increase in reaction rate for PFO, SO and MSO. The reaction rate for PFO, SO and MSO increase from $(2.8 \times 10^{-8} - 5.7 \times 10^{-8} \text{ K, ml/mol, min})$, $(3.4 \times 10^{-8} - 6.9 \times 10^{-7} \text{ K, ml/mol, min})$ and $(3.4 \times 10^{-8} - 6.9 \times 10^{-7} \text{ K, ml/mol, min})$ respectively, as temperature increases from (318.15 -343.15 K). This means the three kinetic models are suitable for the study. Comparison of the three since MSO and SO have almost same values PFO is the most accurate for the study accounting absolute error and relative error.

Table 4.3: Kinetic modelling results

| Tempera ture ^K | GRE r (K,ml/mol, min) | MSO r (K,ml/m ol,min) | PFO r (K,ml/mol ,min) | Absolute Error (PFO) | Relative Error (PFO) | SO r (K,ml/mol. min) | Relative Error (SO) | Absolute Error (SO) | Refere nce (PFO) | Referen ce (SO) |
|------------------------------|-----------------------------|-----------------------------|-----------------------------|----------------------------|----------------------------|----------------------------|---------------------------|---------------------------|------------------------------------|---|
| 318.15 | 8.5×10^{-08} | 3.4×10^{-08} | 2.8×10^{-08} | 1.2×10^{-05} | 0.997732 134 | 1.2×10^{-05} | 0.999999 783 | 0.158858 142 | (Galva n <i>et al.</i> 2020) | (Chaud hary <i>et</i> <i>al.</i> 2015) |
| 328.15 | 1.3×10^{-07} | 5.6×10^{-08} | 4.5×10^{-08} | $3. \times 10^{-04}$ | 0.999859 571 | 3.2×10^{-05} | 0.999999 798 | 0.275761 445 | (Galva n <i>et al.</i> 2020) | (Chaud hary <i>et</i> <i>al.</i> 2015) |
| 338.15 | 1.6×10^{-07} | 6.9×10^{-08} | 5.7×10^{-08} | 3.1×10^{-05} | 0.998180 01 | 3.1×10^{-04} | 0.999999 786 | 0.325048 198 | (Galva n <i>et al.</i> 2020) | (Chaud hary <i>et</i> <i>al.</i> 2015) |

| | | | | | | | | | | |
|--------|-----------------------|-----------------------|---------------|-----------------------|-----------------|-----------------------|----------------|-----------------|------------------------------------|---|
| 343.15 | 5.1×10^{-07} | 6.9×10^{-07} | 5.510^{-08} | 1.4×10^{-04} | 0.999960 565 | 1.4×10^{-04} | 0.999306 03 | 0.001000 852 | (Galva n <i>et al.</i> 2020) | (Chaud hary <i>et</i> <i>al.</i> 2015) |
|--------|-----------------------|-----------------------|---------------|-----------------------|-----------------|-----------------------|----------------|-----------------|------------------------------------|---|

The correctness of the PFO and SO models in the section will be examined. The results of the impact of the catalyst amount, temperature, and methanol-to-oil molar ratio on the transesterification reaction rate is shown by the kinetic of the product using 4 types of kinetic modelling in the study. The findings indicated that as the temperature rises, it also increases the reaction rate. The transesterification is composed of three sequential reversible steps, with the intermediate product being diglycerides and monoglycerides, and each step yielding one mol of fatty acid methyl ester (FAME). As a byproduct, 1 mol of glycerine is also produced. Given that the SO or PSO kinetics models were developed at short initial time of the reaction, it was predicted that the reaction kinetics would follow these models at short initial reaction times. To assess the accuracy of the SO and PFO kinetics models, the reaction rate has been calculated using the generated equations using the triglyceride concentration that was acquired from the calculation. To assess how well SO and PFO kinetics models are at modelling reaction kinetics at shorter initial reaction times, relative error and absolute error must be considered are defined as:

$$RE = \frac{Rate_{lit} - Rate_{cal}}{Rate_{lit}} \quad \text{Equation 4.3}$$

Where $Rate_{lit}$ is the reaction rate in the literature and $Rate_{cal}$ is the reaction rate calculate

$$AE = Rate_{lit} - Rate_{cal} \quad \text{Equation 4.4}$$

Where $Rate_{lit}$ and $Rate_{cal}$ are reaction rate in literature and calculated reaction rates respectively. Relative and absolute errors for calculation of reaction rate are presented in table 4.3. As observed by Ezzati, Ranjbar and Soltanabadi (2021) According to his study, the two models' relative and absolute errors are small at the beginning of the reaction. Relative and absolute errors increase as time goes on. Therefore the errors get smaller as the system approaches the initial reaction timing. Their error analysis shows that the SO and PFO kinetics models are not applicable for the whole reaction time. This has been evaluated using both simulated and calculated data (Ezzati, Ranjbar and Soltanabadi 2021). The accuracy of the models is low at short initial reaction phases. Initially, in the reaction, the two kinetic models behave in very similar ways. This results from an initial high alcohol concentration that starts to deviate as the reaction proceeds.

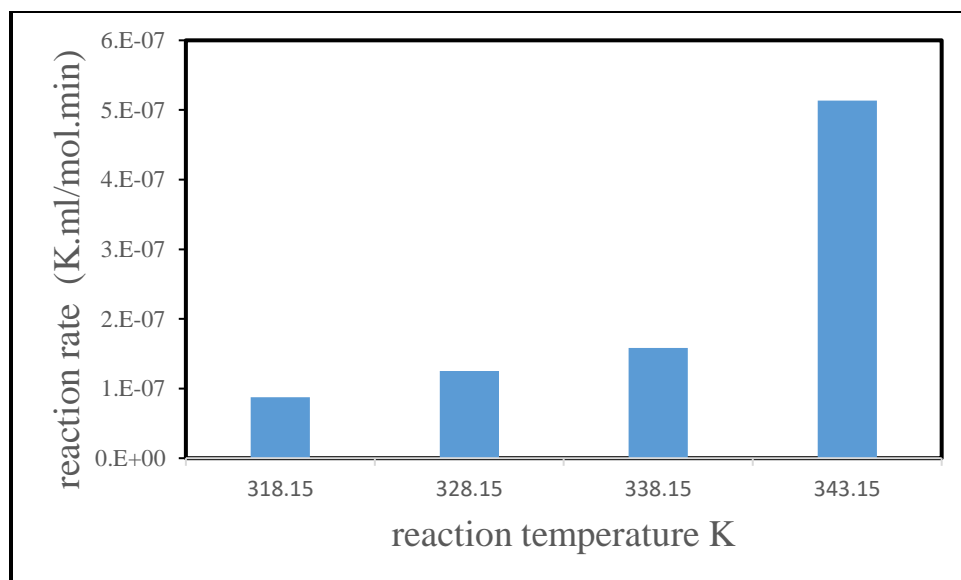


Figure 4.5: General Rate Equation kinetic model

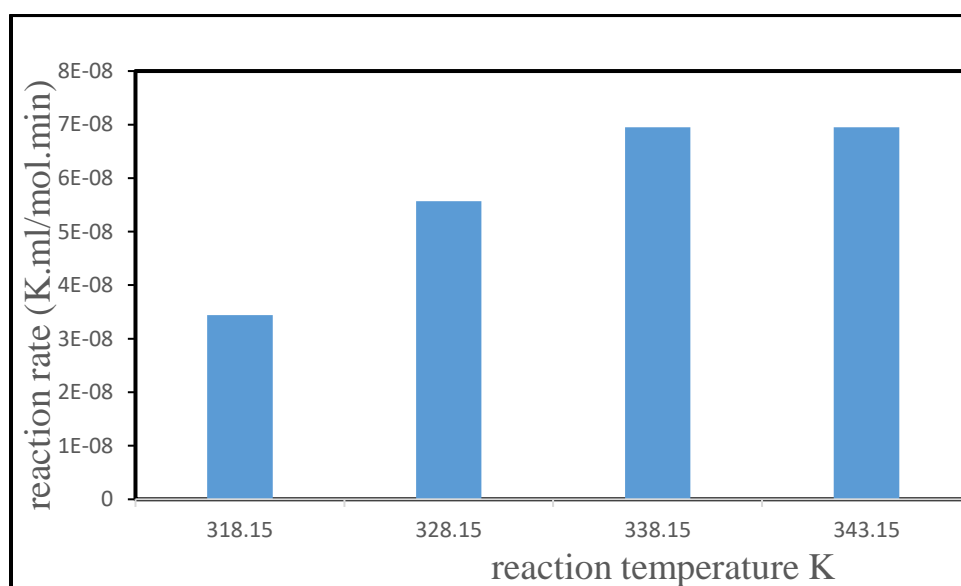


Figure 4.6: Modified Second order kinetic model

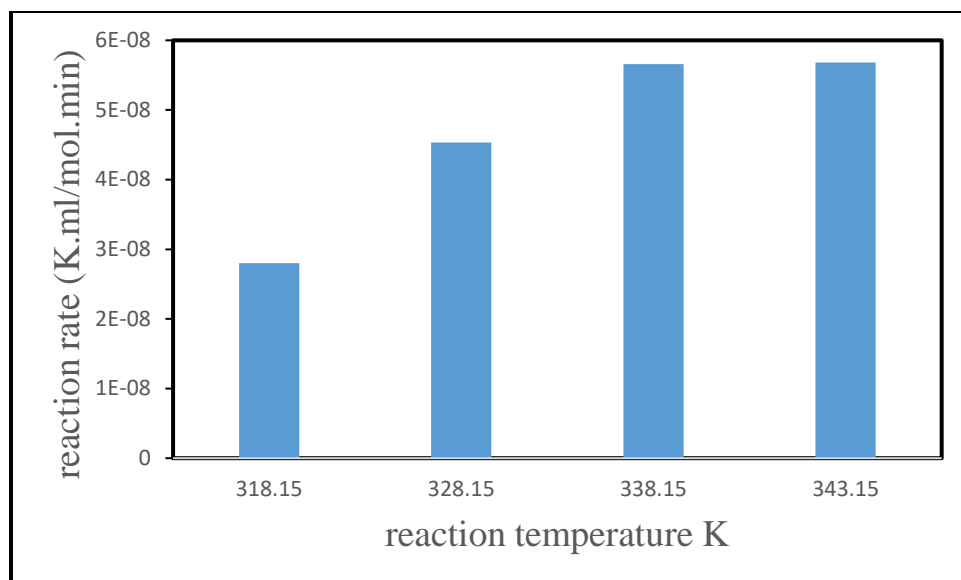


Figure 4.7: Pseudo First order kinetic model

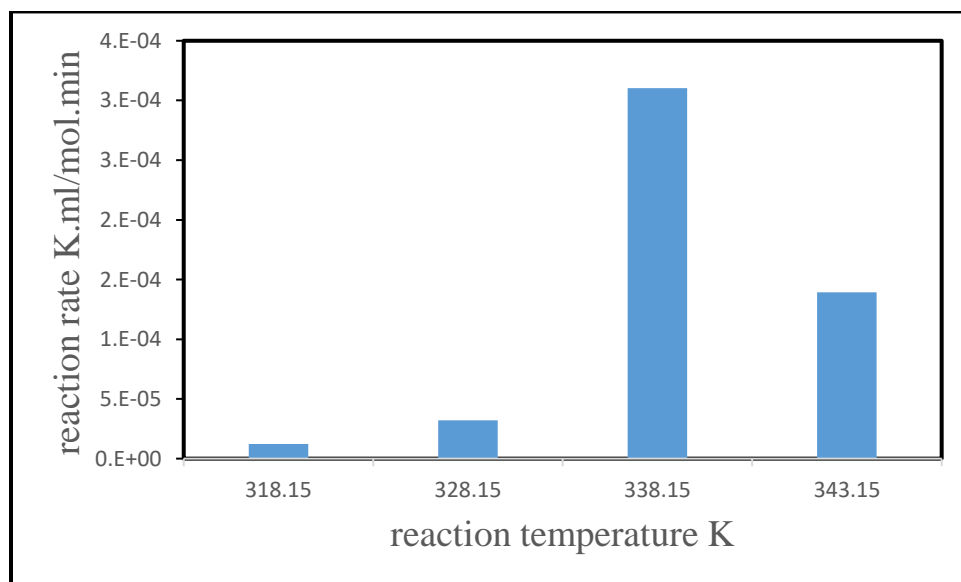


Figure 4.8: Second order kinetic model

The kinetic models GRE, MSO, PFO and SO in figure (4.5 - 4.8) exhibit the behavior of rate of reaction against reaction temperature.

According to Ezzati, Ranjbar and Soltanabadi (2021) the rate constants, kinetic parameters of reactions based on GRE, MSO, SO, and PFO kinetic models were obtained by simulation and compared to calculated values. The results obtained at a reaction temperature of 338 K, 120-minute reaction time, a catalyst concentration of 1 wt% KOH catalyst, and methanol:oil molar ratio of 6:1 show that the accuracy of these models followed the order $GRE > MSO > SO > PFO$ at the entire time of reaction. The accuracy at a shorter initial time shows that $SO > PFO$, which means the SO model was more accurate compared to PFO, according to Ezzati, Ranjbar and Soltanabadi (2021). The results of the Ezzati, Ranjbar and Soltanabadi (2021) study were presented graphically. The current study results shows that the accuracy of $PFO > SO$. The molar ratio for both studies has an impact; for this study, the molar ratio was 9:1, methanol:oil and for Ezzati, Ranjbar and Soltanabadi (2021) is 6:1 methanol:oil. The reaction temperature can also have an impact. The studies are in agreement that PFO and SO are more accuracy at the initial phase of reaction. GRE and MSO are accurate for the whole range of time.

CHAPTER 5: DESIGN OF THE REACTOR

5.1 Introduction

Based on the American Society of Agriculture Engineers (ASAE), the whole process of making biodiesel includes degumming the crude oil, refining it, washing the transesterification ester, refining the glycerol, and recovering the methanol. The transesterification portion is the most important one among these since it deals with any variables and the quality of production. (Rakseethong 2012). The first part of this chapter is to simulate the reactor using generated data.

5.2 Description of the process

The transesterification of castor oil was done in batch mode in a jacketed reactor with a raw tank for feedstock castor oil, which is pumped to a heat exchanger to increase the temperature of castor oil, which also lowers the viscosity so that castor oil can flow easily prior to the reactor. Methoxide mixing tank: mixing methanol and catalyst (KOH) to form a homogenous solution by dissolving solid KOH into liquid methanol to form methoxide. Methoxide is pumped into the reactor after the castor is fed in. The reaction within the reactor is stirred with the agitator/impeller the reaction time is 90 minutes at a temperature of 45°C. The reaction product is discharged into the settling tank to settle for a couple of hours, then two layers will be formed. Decant the bottom glycerol layer by opening the bottom valve of the oil waste storage tank and finish all the glycerol in the tank. The biodiesel left in the settling tank was discharged into the biodiesel storage tank to be tested. If the quality of biodiesel in the biodiesel storage tank is bad, it is transferred into a settling tank to be washed with distilled water. The washing water is sent to the waste water treatment plant, while a mixture of water, catalyst, and soup is recycled with a pump into the mixing tank.

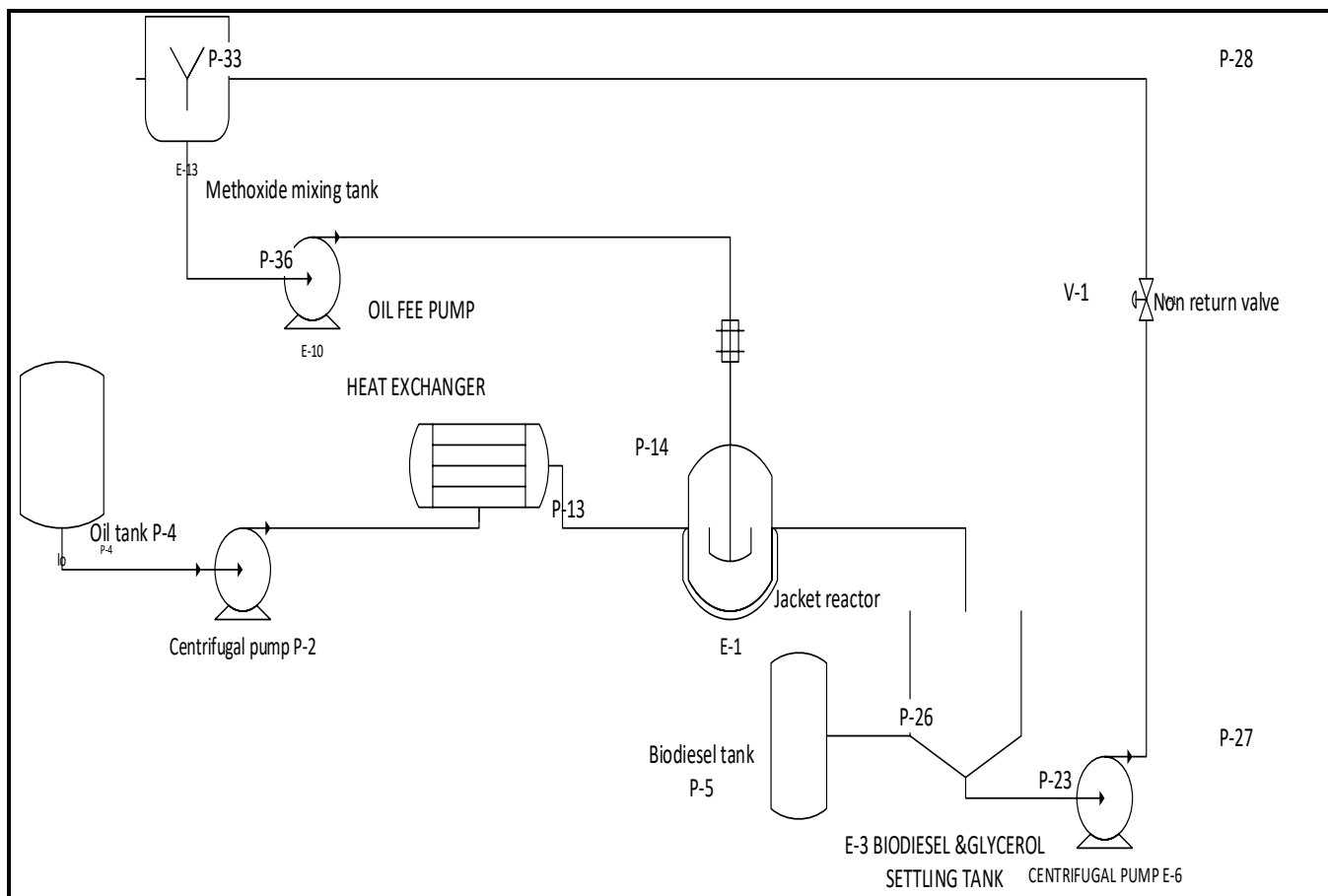


Figure 5.1: Process flow diagram for transesterification reaction process

5.3 Reactor Design

In this study, one of the objectives is to design a reactor. The type of reactor was designed for the transesterification reaction process for the production of biodiesel from castor oil. The two-phase reactants are immiscible; the mass transfer is slow to diffuse from methanol to triglycerides, which leads to an insufficient conversion of triglycerides into biodiesel. A jacketed batch reactor with stirrer is used to provide enough contact between reactants at any given temperature range (25 – 70°C). The process was conducted at 45°C for a contact time of 90 minutes. The reactor was chosen to intimately contact castor oil and methanol, with agitation to distribute and well-mix triglycerides and methanol moles. Contrary to safe operation with respect to data handling within reactions. In this study there is only one equipment that was designed which is a reactor.

One is recommended to complete the detailed design of all equipment in Figure 5.1 for the complete process. Consideration of the waste water management, the waste water produced from the process will be sent waste water effluent plant, to ensure that the process complies with environmental regulations.

Sample calculation is present on Appendix C table C1.1

Discussion of a flow sheet table

A flow sheet table is a table of the steps in progress from the first unit until the last unit. The processing unit and stream for the interpretation of the process flow diagram make it easier for anyone to understand the process. The purpose of this step, P-33, is to mix methanol and KOH catalysts to dissolve catalysts and form a homogenous phase before pumping P-36 as MeOH into E-1.

The esterification process is used for high-free fatty acid neutralisation in a feedstock. Sulphuric acid is mixed with feed stock and contains high-free fatty acids (Maina 2021). According to the molar ratio of H_2SO_4 to triglyceride, it is mixed in P-33, heated up to 60°C and pumped into P-66 when the fatty acid content has decreased to an acceptable value for transesterification.

In P-14, MeOH is flowing at a flow rate of 5 kg/hr to E-1 to be mixed with vegetable oil in E-1 to begin the reaction.

In the P-13 shell and tube heat exchanger, heat up the feedstock vegetable oil to the required temperature (45–70°C) for reaction and lower the viscosity of the feedstock to flow easily into E-1 and ease the reaction. The shell and tube heat exchangers were chosen based on the petroleum product to be handled and the viscous material on the shell side, while steam or service material flowed on the tube side (Silaipillayarputhur and Khurshid 2019).

Reactor vessel E-1 was designed for batch process accounting, residence time requirements, and data handling. By taking into account all of the necessary time, including reaction time, feeding time, and reactor washing time, experiments were able to determine the residence times. The vessel was specified to have an aspect ratio of 1:9 (Brassard, Godbout and Raghavan 2017). The design of the reactor was validated with calculation from table 5.2.

Settling tank Stream p-26 for settling the product mixture from E-1 for a couple of hours. The settled product is separated efficiently. Glycerol settled at the bottom and decanted, while biodiesel was on top.

Stream P-23 recycles a mixture of water and the remaining catalyst after washing the product at a flow rate of 1.5 kg/hr so that it cannot mess up the fresh mixture product on stream P-33. The recycled stream P-23 with catalyst can be further used up; its life-cycle is approximately 10 times; it can be reused many times.

Pump P-27 with a recycling mixture of the remaining catalyst and water is pumped at a flow rate of 1.5 kg/hr to recycle the valuable material to be reused in P-33. The centrifugal pumps were chosen to handle the type of material used; their motors are sized to accommodate viscous material (Paternost, Bannwart and Estevam 2015).

5.4 Biodiesel production simulation

ASPEN Plus version 11 was used for the simulation. The components used for the simulation were available from the ASPEN PLUS databanks. The property method used for the simulation is Braun K-10 (BK10) which is classified as the property method for petroleum applications. ASPEN PLUS mixer was used to mix two different streams, namely Potassium hydroxide and methanol. The mixed stream and the castor oil triglyceride were fed into the heater in order to increase the temperature to the reaction temperature of 45°C. ASPEN simulation is presented in Appendix C table C1.1.

The heated mixture is fed into the reactor, where transesterification reaction takes place. The reactor was simulated using the stoichiometric reactor. The stoichiometric reactor (RStoic) is based on the known fractional conversion. The RStoic model specifications are summarized in **Table 5.1**. The reaction assumed for the model is based on the transesterification of Ricinoleic acid Triglyceride. This was selected because Ricinoleic acid is a major component of castor oil. The conversion used in the model is based on the experimental results.

Table 5.1: Simulation chemical reaction

| Chemical reaction Conversion | Conversion |
|---|------------|
| Ricinoleic acid Triglyceride + 3 Methanol \rightarrow Biodiesel + Glycerol $C_{57}H_{104}O_9 + 3CH_3OH \xrightarrow{KOH} 3C_{19}H_{36}O_3 + C_3H_8O_3$ | 73.7% |

During process construction the applicable unit processes were selected, and the input stream were defined together with the operating conditions for the process units which ultimately generate the outputs of the process unit. The experimental results for the current study were used for the unit process conditions when constructing simulation.

Simulation of transesterification process discussion

Process simulation can provide general mass and energy balances, which are essential for the economic analysis of biodiesel production. A typical process simulation approach includes unit and operation input, chemical and kinetic model definition, property estimation drawing of the flow sheet, and component definition. The simulation results exhibit different biodiesel yield result compared to experimental biodiesel yield using same optimum condition conditions obtained from experiment. The simulation biodiesel yield is 72.7 % whereas experimental biodiesel yield was 97.9 % for purified castor oil and 98.45% for raw castor oil. The simulation results presented on appendix C, table C1.2.

The features of the design was calculated based on design of batch reactor. The energy balance and mass balance are done on simulation.

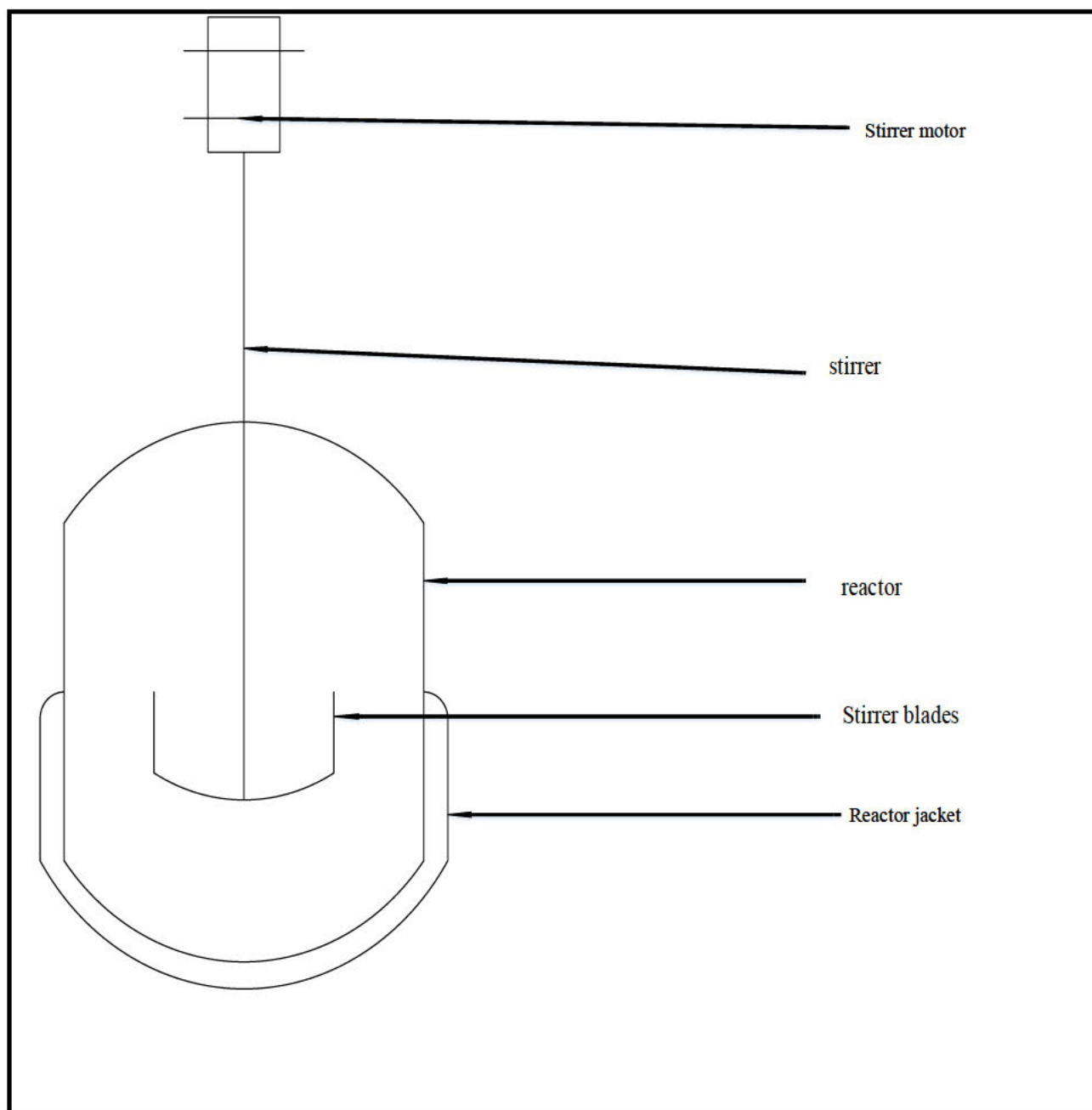


Figure 5.2: Designed batch reactor

To find volume the volume of the reactor

Assumption made

- The feed rate of castor oil is 500 L per batch.
- The feed rate of methanol is 165.27 L per batch.

- The feed rate of KOH catalyst 33.05224 (% w/w) of castor oil
- The Molar mass for triglyceride is 877g/mol
- The Molar mass for methanol is 32.04g/mol
- The reaction temperature is 45°C and the residence time is $T_R = 90$ minutes.
- Feeding time $T_F = 5$ minutes.
- The time of discharging and cleaning the reactor $T_C = 10$ minutes.
- The thermal conductivity of fluid
- The total needed for the process is
- The system is a constant pressure batch process and a constant batch process and a constant volume reaction.
- The jacket is used as an insulation for the reactor.

The reactor external and internal appearance

- Vertical cylindrical tank with torispherical top and bottom is chosen because of data handling can cause corrosion(Sinnott and Towler 2019)
- The number of baffles is usually 4(Sinnott and Towler 2019) (Unit Operations of Chemical Engineering, McCabe et al.,(2001), 6th Edition, pg 241)
- Impeller's rotational speed, $N = 200$ rpm (Marinos-Kouris and Maroulis 2020)
- The corrosion allowance is 2.0 mm.(Sinnott 2005)
- A 20% factor of safety for batch reactor. (Peters and Timmerhaus 2018)
- The capacity of reactor is 80% of volume , measured at top tangent line to allow vapor space (*rules of thumb for chemical engineering*)(Hall 2017) Design Requirements
 1. Batch time
 2. Reactor volume
 3. Reactor diameter
 4. Reactor Height
 5. Head and bottom thickness
 6. Power requirement
 7. Reactor insulation
 8. Impeller design
 - Diameter

- Length width
- Elevation

Calculations

1. Total batch time

Batch time is a preparation time of reactor and reaction time (Davis and Occelli 2009)

$$T_B = T_C + T_R + T_F \quad \text{Equation 5.1}$$

$$T_B = 105 \text{ minutes}$$

2. Reactor Volume

Using the up-scaled values for feed of transesterification process

$$V = V_R + V_M + V_C \quad \text{Equation 5.2}$$

$$V = 665.2612 \text{ liter}$$

Applying 20% safety factor,

$$V = 837.9761 \text{ liter}$$

For 80% filling capacity

$$\text{The reactor capacity/volume } V = 997.8918 \text{ l}$$

The reactor diameter

$$V = V_{\text{cylindrical tank}}$$

1 liter of castor oil is equivalent to 0.956 kg

$$V = \frac{\pi D^2 h}{4} \quad \text{Equation 5.3}$$

Since $H = D$ then,

$$V = \frac{\pi D^3}{4} \quad \text{Equation 5.4}$$

$$D = 0.462888 \text{ m}$$

Use $D = 0.5 \text{ m}$

The reactor height

$$H_R = D = 0.5 \text{ m}$$

Height of head,

$$H_H = 10\% \text{ of reactor height}$$

$$H_H = 0.05 \text{ m}$$

Height of bottom

$$H_B = 10\% \text{ of reactor height}$$

$$H_B = 0.05 \text{ m}$$

$$H_T = H_B + H_H + H_R$$

$$H_T = 0.6$$

Reactor area

$$A = 2.181284034 \text{ m}^2$$

Shell thickness

$$t = \frac{Pr}{SE - 0.6p} + C \quad \text{Equation 5.5}$$

(Peters and Timmerhaus 2018)

Where

t- Maximum wall thickness, inside

P- maximum allowable working pressure, psi

r- inside radius with corrosion allowance

S- maximum allowable tensile strength, psi

E- joint efficiency

C- corrosion allowance inside

$$S = S_u + F_m + F_a + F_r + F_s \quad \text{Equation 5.6}$$

S_u = (Peters and Timmerhaus 2018)(Process Equipment Design, Hesse and Rushton pg 81)

F_m = (Peters and Timmerhaus 2018)(Process Equipment Design, Hesse and Rushton pg 84)

$F_a = F_r$ since radiographing and stress relieving is not required

$F_s =$ (Peters and Timmerhaus 2018)(Process Equipment, Hesse and Rushton *pg* 84)

$S=15,000$ psi

Calculation of internal pressure

$$P = H\rho g + P_{atm} \quad \text{Equation 5.7}$$

(Peters and Timmerhaus 2018)(Process equipment Design by Hesse & Rushton, pg. 81)

P = maximum allowable working pressure

H = reactor height

ρ = fluid density

g = gravitational acceleration force

$P = 5168.722$ Pascals

For allowable pressure (Holloway, Nwaoha and Onyewuenyi 2012)

33% safe pressure allowance

$P = 6874.401$ Pa

Pressure of the reactor during the reaction the reaction **$P = 6874.401$ Pa**

Impeller design

$$\frac{D_a}{D_r} = 0.5 \quad \text{Equation 5.8}$$

D_a = impeller/stirrer diameter (m)

D_r = reactor diameter (m)

Impeller diameter **$D = 0.25$ m**

Length of impeller

Impeller length is 60 – 80 % of the reactor diameter (Bertrand) (Geankoplis, 2003, PG 141)

$$L = 0.70D_r \quad \text{Equation 5.9}$$

Length **L = 0.35m**

Width of impeller

For pitched-blade turbine with blades at 45°

$$\frac{W}{D_a} = \frac{1}{8} \quad \text{Equation 5.10}$$

(Principles of transport process and Unit Operation by Geankoplis, 3rd edition pg 144)

W = width of the impeller (m)

D_a = impeller diameter (m)

W = 0.03125m

Consider **W = 0.03m**

Elevation of impeller

$$\frac{C}{D_r} = \frac{1}{3} \quad \text{Equation 5.11}$$

C = elevation of impeller

C = 0.166667 m

Consider **0.2 m**

Impeller rotation

Reynolds number

$$Re = \frac{D^2 N \rho}{\mu} \quad \text{Equation 5.12}$$

N_{Re} = 37094.23053

N_{Re} = Reynolds number

D = Impeller diameter

ρ = density of fluid

μ = Kinematic viscosity

Tank Insulation

$$Q = \Delta H \times T_R \quad \text{Equation 5.13}$$

$$Q = 1703.877 \text{ Btu/hr}$$

The reaction temperature for the batch stirred reactor is 45°C and the temperature for the surrounding is assumed to be ambient temperature 25°C

$$\Delta T = T_R - T_a \text{ } ^\circ\text{C} \quad \text{Equation 5.14}$$

$$\Delta T = 20^\circ\text{C}$$

$$\frac{Q}{A} = k \frac{\Delta T}{x} \quad \text{Equation 5.15}$$

$$Q = (2\pi r H) k \frac{\Delta T}{x}$$

$$x = 0.218185 \text{ ft} = 0.0665 \text{ m}$$

$$Q = UA\Delta T \quad \text{Equation 5.16}$$

$$\Delta T = T_{\text{CCO}} - T_{\text{MOH}}$$

Overall heat transfer

$$U = 9.018124199 \text{ W/m}^2\text{K}$$

T_{CCO} = temperature for castor oil

T_{MOH} = temperature for methanol

Power Requirement

The approximately mixer power for stirred-tank reactors for reaction with heat transfer is 3.25 hp/1000 gal (Christwardana *et al.* 2020)

$$P = V \times \frac{264.1720 \text{ gal}}{1 \text{ m}^3} \times \frac{3.25 \text{ hp}}{100 \text{ gal}} \quad \text{Equation 5.17}$$

$$P = 9.363805 \text{ hp} = 6982.59 \text{ Watt}$$

Consider $P = 6983 \text{ Watt}$

Table 5.2: Design specification of a reactor

| Identification | |
|------------------------------------|---|
| Materials handled | Castor oil and methanol |
| Function | Transesterification reaction |
| Residence time | 90 minutes |
| Reaction temperature | 90°C |
| Reactor insulation | Calcium silicate |
| Materials of construction | Stainless Steel Type302 |
| Design data | |
| Parameters | Proposed design |
| Reactor Capacity/ volume | 997.8918 L |
| Reactor Diameter | 0.5m |
| Reactor Area | 2.181284034 m ² |
| Reactor Height | 0.6 m |
| Type of impeller | Four bladed pitched turbine |
| Impeller Diameter | 0.5 m |
| Impeller elevation | 0.5 m |
| U Overall heat transfer in liquids | 1.588 Btu/ft ² °F = 9.018124199 W/m ² K |
| Heat transfer within reaction Q | 772.8658 Btu/hr |
| Reynolds Number | 37094.23053 |

| | |
|----------------------------------|-----------|
| Power required by impeller motor | 6982.59 W |
|----------------------------------|-----------|

The design of the reactor was based on suitable generated data. The results obtained from experimental lab work were then simulated to ensure the convection yield of biodiesel. The experiments were conducted to give guidance on how much was needed to produce a high yield of biodiesel (Mohiddin *et al.* 2021). Designing the reactor requires a certain amount of capacity to accommodate reactants. Simulated results from experimental work also provided operating conditions like reaction temperature, reaction concentration, and pressure to accommodate reactor conditions (Aboelazayem, Gadalla and Saha 2018). The temperature of the reactor will increase the reaction rate and mass transfer rate of reactants since mass transfer is a critical factor for transesterification reactions (Tran, Chang and Lee 2017). The temperature used for the distillation is high because there are various reactors required for the purification of the product (Keller 2014). The energy consumption for the reactor producing biodiesel is less compared to the series of distillations required for the production of convectional diesel (Kianimanesh, Abbaspour-Aghdam and Derakhshan 2017). The reactor for convectional diesel requires a smaller capacity, more than 1000 litres, because the process of making diesel uses crude oil as its raw material (Silva and de Andrade 2021). The disadvantage of conventional diesel production is that it produces many by-products before producing the final product. The production of convectional diesel in a reactor is expensive compared to the production of biodiesel in a batch reactor.

The design volume for the reactor assumption was estimated to (500 L – 1000 L). The physical features of the reactor design are on calculation in appendix C table C1.2. The calculations were in agreement with the assumptions most of the design requirements are calculated based on data handle and generated data

CHAPTER 6: COST ANALYSIS

6.1 Introduction

The castor plant can grow in different climatic conditions. Compared to other oil-producing plants like rapeseed and jatropha, this plant's cultivation costs are cheaper. Additionally, due to their high level of poisonousness, the use of castor seeds and seed cake as food for humans and animals is prohibited (Carrino *et al.* 2020).

Different technological and financial barriers exist when producing biofuel from lignocellulosic feedstock and energy crops. The idea of a biorefinery is to address the process drawbacks of the current method and make lignocellulose-derived bioenergy competitive with other sources of energy. Facilities that create biofuels, energy, and biochemicals from renewable feedstock are referred to as biorefineries. Recently, there has been increase in the interest in biorefineries that use energy crops as feedstock. This is expected to play a substantial part in tackling climate change and lessening reliance on fossil fuels in ways that are financially viable (Kurian *et al.* 2013; Sandin *et al.* 2015).

The usage compared against the production of energy is a critical element in the development of biorefineries. The energy produced by the biorefinery in the form of heat, electricity, and liquid or gaseous fuels ought to be greater than the energy used in various process units (Rahimi and Shafiei 2019). The energy analysis of these processes is therefore crucial. Technical and economic research are required to forecast if the biorefinery would be feasible. The technical and economic studies are techniques that demonstrate future plans and possibilities for the development of biorefineries as well as the strengths and limitations of each process (Rahimi, Shafiei and Karimi 2020).

Energy efficiency is defined as the process's energy input to output ratio (Shafiei *et al.* 2013). The energy outputs include both generated heat and the energy present in biodiesel. The energy inputs include energy present in fuel, steam, electricity, and raw materials (Shafiei *et al.* 2013).

Raw materials

These are essential materials required manufacture the product:

- castor oil
- raw castor oil

- Methanol
- KOH

Miscellaneous materials (plant supplies)

These are materials that are required for the plant to operate:

- Personnel Protective equipment PPE
- cleaning materials,
- instrument charts
- and accessories

Utilities

These are plant essential services required for good operation.

Operating cost

This is man power needed to operate the plant: these include the plant operation process hours.

Assumptions made:

- plant will operate 12 hours/ day
- 80% efficiency/ overall yield
- Pump efficiency 80%
- one year life project
- Plant producing 100 000 (kg/y)
- Plant operate 300 days/ year
- Settling is not included as labour hours
- Motor size is equal to all

$$\text{Raw material requirement at 80\% yield} = \frac{\text{plant producing}}{\text{production yield}}$$

Equation 6. 1

Calculations of raw materials is represented by equation no 6.1. Labour rates were observed from the (FFS refiners) payroll they use a rate of R100 /hr calculate the labour cost represented by equation no 6.2.

$$\text{labour cost} = \text{labour rate} \times \text{hours worked} \quad \text{Equation 6.2}$$

The total cost of the raw materials and biodiesel is represented in eqn. no 6.3

Material cost is determined by availability prices which depends on location for the material sourced and the quantity requested.

$$\text{production cost} = (\text{labour rate} \times \text{time worked}) + \text{feedstock cost} \quad \text{Equation 6.3}$$

$$\begin{aligned} \text{electricity cost} = \\ \text{electricity cost} \times \text{no. of motors} \times \text{size of motor} \times \text{working hours} \end{aligned} \quad \text{Equation 6.4}$$

$$\text{water cost} = \text{cost of water} \times \text{quantity} \times \text{working hours} \quad \text{Equation 6.5}$$

Table 6.1: Detail flow sheet of a batch transesterification and esterification process using castor oil

| Stream name | MeOH methoxide | H₂SO₄ | Castor oil P- 13 | Methoxide P-14 | Product P-26 | Byproduct P-23 | Feed stock P-25 | Ra tank P-4 | Methoxide P-36 | Catalyst P-27 | |
|------------------------------------|---------------------------|------------------------------------|---------------------------------|---------------------------|-------------------------|---------------------------|--------------------------------|----------------------------|---------------------------|--------------------------|---|
| Temperature (°C) | 25 | 25 | 50 | 62.5, 65 | 62.5. 65 | 60 | 62,5 65 | 25 | 25 | 60 | |
| Mass flow (kg/hr) | 5 | 0.5 | 10 | | 7.5 | 1.5 | 12.5 | 7 | 5 | 1.5 | |
| Methanol | 1 | - | - | - | - | - | 1 | - | 1 | 1 | |
| H₂SO₄ | - | - | - | 1 | - | - | - | - | - | - | |
| KOH | - | - | - | 1 | 1 | - | - | - | - | - | |
| Castor oil | - | - | 1 | - | - | - | - | 1 | - | - | |
| Glycerol | - | - | - | - | 1 | - | - | - | - | - | |
| Water | - | - | - | - | 0.005 | - | - | - | - | - | |
| Biodiesel | - | - | - | - | 1 | | 1 | - | - | - | 1 |

| | | |
|--------------------------|--|--|
| Steam pressure (700 kPa) | | |
|--------------------------|--|--|

Table 6.2: Equipment costs fixed capital costs and total capital investment.

| Type equipment | | |
|------------------|------|--------------|
| Reactor jacketed | E-1 | R 3607.04244 |
| Heat exchanger | P-13 | R 102812.56 |
| Pumps | E-2 | R 159468.7 |
| | E-10 | R 159468.7 |
| | E-6 | R 159468.7 |
| Total cost R | | 584825.6424 |

$$\text{total revenue} = \text{proice} \times \text{quality sold}$$

Equation 6.6

$$= P \times Q$$

$$\text{Unit Variable} = \sum \text{units cost}$$

Equation 6.7

$$\text{Variable cost} = \text{units Variable Cost} \times Q$$

Equation 6.8

$$\text{Pump Cost} = \frac{\text{motor size} \times \text{working hours}}{\text{pump efficiency}} \times \text{unit per hour} \quad \text{Equation 6.9}$$

$$\text{Total cost} = \text{utility cost} + \text{variable cost} \quad \text{Equation 6.10}$$

$$\text{Profit} = \text{total avenue} - \text{total cost} \quad \text{Equation 6.11}$$

$$\text{Contribution margin} = \text{total avenue} - \text{variable cost} \quad \text{Equation 6.12}$$

$$\% \text{contribution margin} = \frac{\text{contribution margin}}{\text{total avenue}} \times 100 \quad \text{Equation 6.13}$$

6.2 Preliminary economic study

According to a preliminary economic assessment, the projected capital for a batch with a 500–1000 L capacity would be R 766400. Depending on the desired design, DUT Supply Glass is now selling the jacketed reactor for R5000 per 500 ml. A 300-day production cycle sales prediction was generated for scale-up yields, taking into account the cost of biodiesel. The assumption of a 12-hour production cycle was based on reactor's capacity and the biodiesel yield obtained. Before deducting any operating costs, the sales were projected as follows for the scale-up production yield of 684 l/day at a biodiesel cost and a converted amount of biodiesel per litre: for the amount of R12.00 per litre of biodiesel, the annual sales were estimated to be R2464696.08/year. These estimates are based on biodiesel yields at laboratory scale. The earnings anticipated from these sales are R 1117077.48/year, taking into account the cost of the equipment where the jacketed reactor is included. For the estimation computation, see Appendix C. Unscaled biodiesel yield projections included predicted annual sales as well as consideration of production utilising jacketed reactors and the same characteristics as scale-up yield. The estimated yearly sales were R 2464696.08 for the price of R12.00 per litre of biodiesel and R684.64 per day of biodiesel. According to scaling factors from Peter and Timmerhaus (1991), the predicted or projected annual sales are estimated based on an exaggerated estimate of the actual sales.

6.3 Discussion

Commercial castor oil R 625.24 R/l which is cheap compared to raw castor oil to be extracted from seed with a green solvent to be used in Soxhlet extractor or any mechanical energy.

Extraction on its own requires energy, the energy will increase the cost for the production and exceed the estimated yearly biodiesel production cost. Commercial castor oil will have cheaper biodiesel production cost compared to raw castor oil and its extraction from seeds. The advantage of raw castor oil is the biodiesel yield compared to commercial castor because of acid value present in two feed stock. The plant production will cost R 205391.34 per year including all the cost, while the sales of the product manufactured or produced will cost R 2464696.08 per year or annual. The sales of the product biodiesel for the study minus the production cost of biodiesel will give a profit of R 1117077.48 per year. The business with this figures of the profit is the successful business. The study cost analysis results are good the production of biodiesel is recommended considering the cost analysis made in this study.

CHAPTER 7: CONCLUSION AND RECOMMENDATION

Since non-renewable resources are depleted, there is an increasing need for alternate energy sources worldwide. Numerous renewable energy sources are being investigated and evaluated as a result. Transesterification of vegetable oils yields biodiesel, which is gaining prominence as a renewable fuel.

The physical and chemical properties characterization system was conducted in order to validate the procedures and consistency of the biodiesel data that had already been measured in previous studies and since the absolute average deviation less than 2 it confirms the validation of the results obtained (Jain and Sharma 2010).

The characterization of castor oil favors direct transesterification since the acid value is below 4, which is the limit of the acid value. Study's objective was to produce biodiesel using a homogeneous alkaline catalyst in a jacketed reactor from vegetable oils.

In this study, 97.9% methyl ester yield was obtained using purified castor oil and 98.49% methyl ester yield from raw castor oil, although the optimum conditions are not the same as in the previous studies.

Based on the information gathered and the outcomes of this study, castor oil both raw and purified has been found to be an effective feedstock for the biodiesel production process in the Republic of South Africa and elsewhere. However, as competitive crops with superior biodiesel yields will always be preferred. High yield in raw castor oil shows to have superior over purified castor oil. The lower quantity of raw castor oil perhaps limited the study because scarce availability which remains a matter for concern. However commercial castor oil R 625.24 R/l which is cheap compared to raw castor oil to be extracted from seed with a green solvent to be used in Soxhlet extractor or any mechanical energy, however the extraction process on its own cost more than price commercial castor oil per litre. Biodiesel was estimated to cost R684.64 per litre to produce and have annual sales of R2464696.08. These sales figures were scale-up values and ranges; as the operational cost was taken into consideration, they were overestimated; however, they show that the production of biodiesel has the potential to be profitable.

It has been stated that the transesterification of castor oil has been studied experimentally in small lab reactors. Operating parameters were 9:1 molar ratio of methanol to oil reaction temperature

from 45°C to 70°C at an interval of 10°C, catalysts 0.625% of KOH, % v/v of castor oil and 550 RPM for 90 minutes. PFO model has low relative error and absolute error compared to the SO model and as a result it is more accurate for the study. PFO and SO kinetics modelling are suitable for transesterification reaction at short initial time of the reaction, not for whole range, while GRE and MSO kinetic modelling are suitable for whole range of reaction. PFO and SO kinetic model as the reaction continues the reaction rates starts to deviates.

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APPENDICES

APPENDIX A : Sample of calculations acid value and mole ratios

- **Acid Value**

NaOH solution was prepared at laboratory, preliminary calculation were perform the amount of NaOH weight, required for the solution since NaOH were available in solid phase

Amount of NaOH need to prepare

$$c = \frac{n}{v}$$

$$n = c \times v = 0.1 \times \frac{150}{1000} ml = 0.015 moles$$

$$n = \frac{m}{Mr}$$

$$m = n \times Mr = 39.997 \times 0.015 = 0.6 g$$

- **Assumptions:**

Molar mass of ethanol = 46.08 g/mol

Molar mass of NaOH = 39.997g/mol

Molar mass of MeOH = 32 g/mol

Molar mass of triglyceride = 877g/mol

Where: n - Number of moles

 v- Volume

 c- Concentration

 Mr- Molar mass

 m- Mass

0.1 N of NaOH pellets was weighed to 0.6g mass and dissolved into 250ml glass beaker having 150ml water and stirred vigorously until NaOH dissolve completely. The 0.1N solution of NaOH was used in a burette stand for titration in determining the acid value for castor oil.

- **Acid Value of Raw castor oil**

5 g oil raw castor oil needed. NaOH solution is prepared in a 250ml glass beaker. 50 ml of 99.9% Ethanol was transferred into a 150 ml conical flask then oil was dissolved in the solution. Therefore the solution is titrated with NaOH in a burette until the color change to sweet pink is noticed.

$$Acid\ Value(AV) = \frac{40 \times N \times V}{m}$$

$$(AV) = \frac{40 \times 0.1 \times V}{5.03}$$

Where the constant 40.0 is the molar mass of NaOH, N is the normality of the standardized NaOH solution, V is the volume of the NaOH solution and m (g), of the castor oil. Acid Value for purified castor oil was found 1.03 which is within the limit of required AV for direct transesterification. NB the titration was repeated three times for assurance.

- **Transesterification process**

Mole ratio 1:3

Methanol to oil

Mass of castor oil is fed into the reactor = 40g

$$\frac{M\ oil}{Mr\ triglyceride} \times \frac{X\ methanol}{3(Mr\ MeOH)}$$

$$= \frac{40g}{877g/mol} \times \frac{X\ methanol}{3(32.04\ g/mol)} = 4.38g\ methanol$$

Mole ratio 6:1

$$\frac{M\ oil}{Mr\ triglyceride} \times \frac{X\ methanol}{6(Mr\ MeOH)}$$

$$= \frac{40g}{877g/mol} \times \frac{X \text{ methanol}}{6(32.04 g/mol)} = 8.77g \text{ methanol}$$

Mole ratio 9:1

$$\frac{M \text{ oil}}{Mr \text{ triglyceride}} \times \frac{X \text{ methanol}}{9(Mr \text{ MeOH})}$$

$$= \frac{40g}{877g/mol} \times \frac{X \text{ methanol}}{9(32.04 g/mol)} = 13.15g \text{ methanol}$$

- **Catalyst concentration**

Basis: 40g of oil

$$\text{Mass of catalyst} = \frac{\text{mass of catalyst}}{\text{mass of oil}} \times 100$$

$$= \frac{0.25}{40} \times 100 = 0.625 \text{ wt\% KOH}$$

APPENDIX B : Reactor specifications

Design of a jacketed reactor on pilot scale

Table B1.1 : jacketed reactor design

| Design of a jacketed reactor | Units |
|------------------------------|-----------------|
| Pressure | 30-70 kPa |
| Material | Stainless steel |
| Capacity | 500 -1000 L |
| Usage application | Industrial |
| Temperature | 25 -250 °C |

APPENDIX C: Reactor design calculations and simulation report

Table C1.1: Simulation report

| Material | | | | | | | |
|------------------------|--------------|---------------|-----------------|--------------|--------------|--------------|-----------------|
| Stream Name | Units | TRYGLY | METHANOL | KOH | S1 | S2 | BIODIESL |
| Description | | | | | | | |
| From | | | | | MIXER | HEATER | REACTOR |
| To | | HEATER | MIXER | MIXER | HEATER | REACTOR | |
| Stream Class | | CONVEN | CONVEN | CONVEN | CONVEN | CONVEN | CONVEN |
| Maximum Relative Error | | | | | | | |
| Cost Flow | \$/hr | | | | | | |
| MIXED Substream | | | | | | | |
| Phase | | Liquid Phase | Liquid Phase | Liquid Phase | Liquid Phase | Liquid Phase | Liquid Phase |
| Temperature | C | 25 | 25 | 25 | 49.04700869 | 45 | 45 |
| Pressure | bar | 1.01325 | 1.01325 | 1.01325 | 1.01325 | 1.01325 | 1.01325 |
| Molar Fraction | Vapor | 0 | 0 | 0 | 0 | 0 | 0 |
| Molar Fraction | Liquid | 1 | 1 | 1 | 1 | 1 | 1 |
| Molar Fraction | Solid | 0 | 0 | 0 | 0 | 0 | 0 |
| Mass Fraction | Vapor | 0 | 0 | 0 | 0 | 0 | 0 |
| Mass Fraction | Liquid | 1 | 1 | 1 | 1 | 1 | 1 |
| Mass Fraction | Solid | 0 | 0 | 0 | 0 | 0 | 0 |
| Molar Enthalpy | cal/mol | - | - | - | - | - | - |
| | | 559096.7472 | -57797.48648 | 129024.2636 | 58562.52426 | 104762.2563 | 119260.0707 |
| Mass Enthalpy | cal/gm | - | - | - | - | - | - |
| | | 598.9590535 | -1803.794953 | -2299.66655 | 1813.046289 | 897.9431747 | 1022.207523 |

| | | | | | | | |
|----------------|-----------|-------------|--------------|-------------|-------------|-------------|-------------|
| Molar Entropy | cal/mol-K | - | - | - | - | - | - |
| | | 1514.719277 | -57.38156709 | -27.0918964 | 55.24493076 | 189.3204339 | 261.8516704 |
| Mass Entropy | cal/gm-K | - | - | - | - | - | - |
| | | 1.622715262 | -1.790814573 | 0.482872959 | 1.710336396 | 1.622712202 | 2.244395344 |
| Molar Density | mol/cc | 0.00759146 | 0.024746172 | 0.033235779 | 0.023981296 | 0.020235324 | 0.007572965 |
| Mass Density | gm/cc | 7.086228311 | 0.792920797 | 1.864714662 | 0.774610807 | 2.360837813 | 0.883531278 |
| Enthalpy Flow | cal/sec | - | - | - | - | - | - |
| | | 6.655100594 | -6.588862119 | 0.159699066 | 6.748561185 | 13.31949042 | 15.16274492 |
| Average MW | | 933.44736 | 32.04216 | 56.10564 | 32.30062279 | 116.6691381 | 116.6691381 |
| Mole Flows | kmol/hr | 4.28519E-05 | 0.000410397 | 4.45588E-06 | 0.000414853 | 0.000457705 | 0.000457705 |
| KOH | kmol/hr | 0 | 0 | 4.45588E-06 | 4.45588E-06 | 4.45588E-06 | 4.45588E-06 |
| METHANOL | kmol/hr | 0 | 0.000410397 | 0 | 0.000410397 | 0.000410397 | 0.000284412 |
| BIODIESL | kmol/hr | 0 | 0 | 0 | 0 | 0 | 0.000125985 |
| GLYCEROL | kmol/hr | 0 | 0 | 0 | 0 | 0 | 4.19949E-05 |
| TRYGLYC | kmol/hr | 4.28519E-05 | 0 | 0 | 0 | 4.28519E-05 | 8.57038E-07 |
| Mole Fractions | | | | | | | |
| KOH | | 0 | 0 | 1 | 0.010740873 | 0.009735275 | 0.009735275 |
| METHANOL | | 0 | | 0 | 0.989259127 | 0.896641221 | 0.621388119 |
| BIODIESL | | 0 | 0 | 0 | 0 | 0 | 0.275253102 |
| GLYCEROL | | 0 | 0 | 0 | 0 | 0 | 0.091751034 |
| TRYGLYC | | 1 | 0 | 0 | 0 | 0.093623504 | 0.00187247 |
| Mass Flows | kg/hr | 0.04 | 0.01315 | 0.00025 | 0.0134 | 0.0534 | 0.0534 |
| KOH | kg/hr | 0 | 0 | 0.00025 | 0.00025 | 0.00025 | 0.00025 |
| METHANOL | kg/hr | 0 | 0.01315 | 0 | 0.01315 | 0.01315 | 0.009113181 |
| BIODIESL | kg/hr | 0 | 0 | 0 | 0 | 0 | 0.039369313 |
| GLYCEROL | kg/hr | 0 | 0 | 0 | 0 | 0 | 0.003867506 |
| TRYGLYC | kg/hr | 0.04 | 0 | 0 | 0 | 0.04 | 0.0008 |
| Mass Fractions | | | | | | | |
| KOH | | 0 | 0 | 1 | 0.018656716 | 0.004681648 | 0.004681648 |
| METHANOL | | 0 | 1 | 0 | 0.981343284 | 0.246254682 | 0.170658821 |
| BIODIESL | | 0 | 0 | 0 | 0 | 0 | 0.737253057 |

| | | | | | | | |
|----------------|-----------|-------------|--------------|-------------|-------------|-------------|-------------|
| GLYCEROL | | 0 | 0 | 0 | 0 | 0 | 0.072425201 |
| TRYGLYC | | 1 | 0 | 0 | 0 | 0.74906367 | 0.014981273 |
| Volume Flow | l/min | 9.40792E-05 | 0.000276404 | 2.23448E-06 | 0.000288317 | 0.000376985 | 0.001007321 |
| Liquid Phase | | | | | | | |
| Molar Enthalpy | cal/mol | - | - | - | - | - | - |
| | | 559096.7472 | -57797.48648 | 129024.2636 | 58562.52422 | 104762.2563 | 119260.0707 |
| Mass Enthalpy | cal/gm | - | - | - | - | - | - |
| | | 598.9590535 | -1803.794953 | -2299.66655 | 1813.046287 | 897.9431747 | 1022.207523 |
| Molar Entropy | cal/mol-K | - | - | - | - | - | - |
| | | 1514.719277 | -57.38156709 | -27.0918964 | 55.24493076 | 189.3204339 | 261.8516704 |
| Mass Entropy | cal/gm-K | - | - | - | - | - | - |
| | | 1.622715262 | -1.790814573 | 0.482872959 | 1.710336396 | 1.622712202 | 2.244395344 |
| Molar Density | mol/cc | 0.00759146 | 0.024746172 | 0.033235779 | 0.023981296 | 0.020235324 | 0.007572965 |
| Mass Density | gm/cc | 7.086228311 | 0.792920797 | 1.864714662 | 0.774610807 | 2.360837813 | 0.883531278 |
| Enthalpy Flow | cal/sec | - | - | - | - | - | - |
| | | 6.655100594 | -6.588862119 | 0.159699066 | 6.748561181 | 13.31949042 | 15.16274492 |
| Average MW | | 933.44736 | 32.04216 | 56.10564 | 32.30062279 | 116.6691381 | 116.6691381 |
| Mole Flows | kmol/hr | 4.28519E-05 | 0.000410397 | 4.45588E-06 | 0.000414853 | 0.000457705 | 0.000457705 |
| KOH | kmol/hr | 0 | 0 | 4.45588E-06 | 4.45588E-06 | 4.45588E-06 | 4.45588E-06 |
| METHANOL | kmol/hr | 0 | 0.000410397 | 0 | 0.000410397 | 0.000410397 | 0.000284412 |
| BIODIESL | kmol/hr | 0 | 0 | 0 | 0 | 0 | 0.000125985 |
| GLYCEROL | kmol/hr | 0 | 0 | 0 | 0 | 0 | 4.19949E-05 |
| TRYGLYC | kmol/hr | 4.28519E-05 | 0 | 0 | 0 | 4.28519E-05 | 8.57038E-07 |
| Mole Fractions | | | | | | | |
| KOH | | 0 | 0 | 1 | 0.010740873 | 0.009735275 | 0.009735275 |
| METHANOL | | 0 | 1 | 0 | 0.989259127 | 0.896641221 | 0.621388119 |
| BIODIESL | | 0 | 0 | 0 | 0 | 0 | 0.275253102 |
| GLYCEROL | | 0 | 0 | 0 | 0 | 0 | 0.091751034 |
| TRYGLYC | | 1 | 0 | 0 | 0 | 0.093623504 | 0.00187247 |
| Mass Flows | kg/hr | 0.04 | 0.01315 | 0.00025 | 0.0134 | 0.0534 | 0.0534 |
| KOH | kg/hr | 0 | 0 | 0.00025 | 0.00025 | 0.00025 | 0.00025 |

| | | | | | | | |
|----------------|-------|-------------|-------------|-------------|-------------|-------------|-------------|
| METHANOL | kg/hr | 0 | 0.01315 | 0 | 0.01315 | 0.01315 | 0.009113181 |
| BIODIESL | kg/hr | 0 | 0 | 0 | 0 | 0 | 0.039369313 |
| GLYCEROL | kg/hr | 0 | 0 | 0 | 0 | 0 | 0.003867506 |
| TRYGLYC | kg/hr | 0.04 | 0 | 0 | 0 | 0.04 | 0.0008 |
| Mass Fractions | | | | | | | |
| KOH | | 0 | 0 | 1 | 0.018656716 | 0.004681648 | 0.004681648 |
| METHANOL | | 0 | 1 | 0 | 0.981343284 | 0.246254682 | 0.170658821 |
| BIODIESL | | 0 | 0 | 0 | 0 | 0 | 0.737253057 |
| GLYCEROL | | 0 | 0 | 0 | 0 | 0 | 0.072425201 |
| TRYGLYC | | 1 | 0 | 0 | 0 | 0.74906367 | 0.014981273 |
| Volume Flow | l/min | 9.40792E-05 | 0.000276404 | 2.23448E-06 | 0.000288317 | 0.000376985 | 0.001007321 |

Table C1.2 : Reactor design calculations

| Parameters/properties | value | units | quantity | units | quantity | units |
|------------------------------|-------------|----------------------|-------------|----------------|----------|-------|
| castor oil | 500 | L | 480500 | g | 480.5 | Kg |
| molar mass triglyceride | 877 | g/mol | | | | |
| molar mass methanol | 32.04 | g/mol | | | | |
| required methanol | 157989.7149 | G | 157.989715 | kg | | |
| required methanol reaction | 165.2612081 | L | | | | |
| reactor capacity | 665.2612081 | L | 0.72709468 | m ³ | | |
| KOH | 33.05224162 | %(w/w of Castor oil) | | | | |
| reactor capacity for safety | 798.3134497 | L | | | | |
| reactor capacity for filling | 997.8918121 | L | | | | |
| 1 liter of castor oil | 0.956 | Kg | | | | |
| reactor volume | 953.9845724 | Kg | | | | |
| 1 cubic meter | 874.7 | Kg | | | | |
| reactor capacity | 1.090642017 | m ³ | | | | |
| PI | 3.14155926 | | | | | |
| Diameter | 0.462887769 | M | 0.5 | m | 1.64042 | ft |
| Area | 2.181284034 | m ² | 7.156443911 | [[ft]] ^2 | | |
| height of head | 0.05 | M | | | | |
| height of bottom | 0.05 | M | | | | |
| total height of reactor | 0.6 | m. | | | | |
| S_u | 60,000 | Psi | | | | |
| F_m | 1 | | | | | |
| F_a | 1 | | | | | |
| F_r | 1 | | | | | |
| F_s | 0.25 | | | | | |
| S | 15,000 | psi | 2.17557 | Pa | | |

| | | | | | | |
|---------------------------|-------------|------------------------|-------------|--------------------|--|--|
| Patm | 101.325 | Pa | | | | |
| H | 0.6 | M | 2.4 | | | |
| G | 9.81 | m/s ² | 603.4242 | | | |
| P | 878.1383418 | kg/m ³ | 9.81 | | | |
| P | 5168.72228 | Pa | 14308.3444 | | | |
| P safe allowance | 6874.400632 | Pa | 0.99704932 | psi | | |
| | | | | | | |
| impeller design | | | | | | |
| properties | value | Units | | | | |
| Da/Dr | 0.5 | M | | | | |
| Da | 0.25 | M | | | | |
| Length L | 0.35 | M | | | | |
| W | 0.03125 | M | | | | |
| impeller elevation | 0.166666667 | M | 0.2 | m | | |
| mass enthalpy triglycerol | -598.959054 | cal/gm | | | | |
| mass enthalpy methanol | -1803.79495 | cal/gm | | | | |
| mass enthalpy glycerol | -2299.66655 | cal/gm | | | | |
| mass enthalpy biodiesel | -1022.20752 | cal/gm | | | | |
| ΔH | 644.0547935 | cal/gm | 1159.29863 | btu/lb. | | |
| Q | 772.8657522 | Btu/hr | | | | |
| U overall heat transfer | 1.588173256 | Btu//ft ² F | 9.018124199 | W/m ² K | | |
| reaction temperature | 45 | °C | | | | |
| ambient temperature | 25 | °C | | | | |
| ΔT | 20 | °C | 68 | °F | | |
| R | 0.25 | M | 0.82021 | ft | | |
| Π | 3.14159 | | | | | |

| | | | | | | |
|------------------------|-------------|-------------------|------------|---|--|--|
| X | 0.218185499 | Ft | | m | | |
| power of stirrer motor | 9.363805196 | Hp | 6982.58953 | w | | |
| N rotation number | 200 | Rpm | | | | |
| fluid density ρ | 8843.264557 | kg/m ³ | | | | |
| μ | 2.98 | CSt | 0.00000298 | | | |
| N_{Re} | 37094.23053 | | | | | |

APPENDIX D : Cost vialation calculation

Table D1.1 : equipment cost

| type | cost \$ | cost in Rands |
|------------------|---------|---------------|
| jacketed reactor | 40000 | 766400 |
| pumps X3 | 8323 | 478406.04 |
| heat Exchangers | 5366 | 102812.56 |
| total cost | | 1347618.6 |

| | |
|---|---------------------------------|
| cost of water (R/L) | 0.043 |
| steam consumption time | 4 |
| quantity (m3) | 100 |
| cost of PPE ® | 1000 |
| number of people | 5 |
| cost of electricity unit (R/kW.h) | 3.062 |
| number of pump motors | 4 |
| raw materials | cost of materiel (R/l) |
| castor oil raw castor oil | 625.24 |
| KOH (refined castor oil) | 1200 |
| | 0 |
| methanol | 1600 |
| Total raw materials cost | 3425.24 |
| | |
| labour rate (R/h) | 100 |
| | |
| Hours worked(equipment working hours) | 11 |
| operation days/year | 300 |
| working hours | 12 |
| expected yield product | 110% |
| raw material requirement (t/y) | 187572 |
| biodiesel | cost R/L |
| 1 litre | 12 |
| biodiesel yield | 110% |
| 1 day production | 684.64 |

| batch production | cost of water (R/m3) | size of motor (kW) | cost of a day (R/h) |
|------------------|----------------------|--------------------|---------------------|
| | 45 | 15 | 409.0909091 |

| cost steam (R/h) | total avenue (R/m3) | total cost (R/m3) | profit | controbuton margins | %contribution margin |
|------------------|---------------------|-------------------|--------|---------------------|----------------------|
| 136.24 | 12000 | 6140.088118 | | 11816.28 | 98.469 |

| utilities | cost of utilities (R) |
|--------------------------------|-------------------------|
| electricity | 2020.92 |
| steam | 284.8372093 |
| water | 409.0909091 |
| | |
| total cost of utilities | 2714.848118 |

| | |
|------------------------------|--------------------|
| equipment cost | 1347618.6 |
| labour cost | 1100 |
| production cost | 18240.08812 |
| profit | |
| plant production /yr) | 205391.34 |
| quantity sold Q (m3) | 1 |
| unity cost (R/h) | 183.72 |
| variable cost | 183.72 |
| biodiesel sale annual | 2464696.08 |
| profit | 1117077.48 |