UNIVERSITI TEKNIKAL MALAYSIA MELAKA

YLISTRUM BALLOTI (SAUCER SCALLOP) AS HETEROGENOUS CATALYST SUPPORT FOR CONVERSION OF WASTE COOKING OIL TO BIODIESEL

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BACHELOR OF MECHANICAL ENGINEERING TECHNOLOGY WITH HONOURS

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**A thesis submitted
in fulfillment of the requirements for the degree of
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APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Mechanical Engineering Technology with Honours.

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DEDICATION

To my parents, Darmawi Bin Abd Wahed and Noraini binti Abdul Aziz, as well as my siblings, friends, and teammates who have been the most supportive during the course of this journey. From the bottom of my heart, I dedicate this. I've come this far in life due of their words of encouragement and support. I'd want to thank all of the UTeM professors and personnel that assisted me in completing my studies, especially my supervisor, who always remembers and guides my research to keep it on track. Despite the miserable recollections of completing our education and study, the occasions we shared will live on in our memories.

ABSTRACT

Biodiesel is another option of fuel that is renewable and releases clean -burning fuel the same as conventional diesel. Waste Cooking Oil are one of the sources that produce biodiesel. Biodiesel is produced domestically and can be used in any diesel engine with little or no modification to the engine or fuel system. This project main goal was to produce green and clean fuel using conventional transesterification method of solid waste shell. *Ylistrum balloti* that are generally known as saucer scallop are the main catalyst for transesterification process with the help of MeOH (MeOH) as alcohol. Transesterification is the chemical process involved to converts the high Free Fatty Acid (FFA) into methyl ester and glycerol. The abandoned shell of scallops was calcined at 800 °C for 4 hours and SEM was used to characterize the catalyst. The best parameters were identified at 5 wt.% percent of catalyst loading, 9:1 molar ratio of MeOH to oil with reaction temperature of 65 °C and reaction time for 90 minutes. It was discovered that the catalyst made from scallop waste shells has strong catalytic activity and environmentally beneficial qualities, making it as one of the good optional of heterogenous catalyst. The properties of produced biodiesel were analyzed according to the ASTM D6751, EN 14212 and AOCS.

ABSTRAK

Biodiesel merupakan salah satu bahan api yang boleh diperbaharui dan menghasilkan pembakaran yang bersih sama seperti diesel konversional. Sisa minyak masak merupakan salah satu sumber yang menghasilkan biodiesel. Proses transesterifikasi merupakan proses yang dapat menukarkan sifat minyak kepada biodiesel. Biodiesel yang terhasil mampu digunakan dalam mana-mana enjin diesel tanpa mengubah mana mana komponen dalam enjin atau sistem bahan api. Matlamat utama projek ini adalah untuk menghasilkan bahan api yang jernih dan bersih menggunakan kaedah konvensional transesterifikasi sisa kulit pepejal. Secara umumnya, kulit Ylistrum balloti atau dikenali sebagai kekapis merupakan pemangkin utama proses transesterifikasi dengan bantuan metanol sebagai alkohol. Transesterifikasi ialah proses kimia yang terlibat untuk menukarkan asid lemak bebas yang tinggi kepada metil ester dan gliserol. Cengkerang kekapis yang terbengkalai akan dibakar pada suhu 800˚C selama 4 jam dan SEM digunakan untuk mengkaji sifat pemangkin. Parameter yang terbaik telah dikenal pasti pada 5%, kepekatan pemangkin , nisbah molar diantara minyak dan metanol 9:1 dengan tindak balas suhu 65˚C dan masa tindak balas selama 90 minit. Didapati bahawa pemangkin yang diperbuat daripada kulit sisa kerang mempunyai kadar pemangkinan yang berkualiti serta baik utuk alam sekitar, menjadikannya pilihan yang sesuai sebagai pemangkin heterogen. Kesemua sifat biodiesel yang dihasilkan akan dianalisis mengikut ASTM D6751, EN 14212 dan AOCS.

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In the Name of Allah, the Most Gracious, the Most Merciful

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LIST OF SYMBOLS AND ABBREVIATIONS

WCO	-	Waste Cooking Oil
FFA	-	Free Fatty Acid
CaO	-	Calcium Oxide
KOH	-	Potassium Hydroxide
˚C	-	Degeree celcius
wt%	-	Weight Percentage
CSS	-	Calcined Scallop Shell
SDG	-	Sustainable Development Goal
SEM	-	Scanning Electron Microscopy
EDS	-	Energy Dispersive Spectrometer
ASTM	-	American Society fot Testing Materials
EN	-	European Standard
AOCS	-	American Oil Chemist's Society
AV	-	Acid Value
UTeM	-	Universiti Teknikal Malaysia Melaka
B100	-	Pure Biodiesel
FAME	-	Fatty Acid Methyl Ester

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CHAPTER 1

INTRODUCTION

1.1 Background

Biodiesel is another renewable fuel that mostly being utilized due to its characteristic. It is renewable and releases clean-burning fuel comparable with conventional diesel. Waste Cooking Oil (WCO), animal fats and vegetable oil are one of the few common sources that can be produced as clean and green biodiesel. To change the properties of biodiesel from oil, transesterification is the chemical process involved to converts the high Free Fatty Acid (FFA) into methyl ester and glycerol. Biodiesel was produced mainly in United States since 90's by Rudolf Diesel and it can be used in any diesel engine with little or no modification It contributed to some countries in reducing their reliance on foreign energy sources.

WCO is an ideal feedstock for the production of biodiesel due to the issue of being abudantly disposed off in a dustbin and drainage system. Their disposal creates a number of environmental issues such as water pollution, soil contamination, air pollution, greenhouse gas emissions and also energy resource wastage. It proves that biodiesel is cost effective as it is an economical alternative resulting in significant savings.

In contrast to edible oils, WCO does not generate food against fuel concerns since it is constantly easy to find and has no environmental impact. Some of the major issues regarding WCO were it has extremely high content of FFA and water content that can resulted to overall poor performances. That can makes transesterification more challenging. Hence, author proposed that the pre-treated process was needed before transesterification process to decrease the FFA content (Monika et al.,2023).

Waste seashell were chosen as the project's raw materials because they are abundant, affordable, renewable and safe for the environment. It also continue to the creation of catalyst sourced from more environmentally susitainable origins. To create a circular pattern, seashells in particular type can be used. *Ylistrum balloti* a special type of scallop seashell. The appearances of this scallop comes in shades of pink, orange and brown. The famous name for *Ylistrum balloti* is saucer scallop and the local call it *‘kekapis’*.

There are several factors that impact biodiesel production quality. One of them are catalyst, molar ratio, reaction temperature and reaction time. Since there are a lot of parameters, identifying the right combination of parameters proves to be a challenging task. A well-planned process is required to reduce the time consumption to choose the right combination by narrow down the parameter combination.

In this study, *Ylistrum balloti* and WCO was used to improve the biodiesel performance according to the standard method. The transesterification method was applied to convert high FFA with the aid of MeOH in producing good and quality B100. Effect of involved parameters such as molar ratio of MeOH to oil, catalyst loading, reaction time and reaction temperature was also being investigated.

1.2 Problem Statement

The fast rise of the population growth has increased the demand for lacking of fossil resources. This situation is an excellent approach to promote the development of biodiesel as a new renewable fuel. It may be a concerned about the rising prices in petroleum since it was utilize every day. Futhermore, the change in atmosphere, environmental concern and also the usage of diesel engine which is poor performance than diesel engine led to the reason why the production of biodiesel is a good replacement to fuel.

Heterogeneous catalyst are widely used nowadays due to effectiveness and non-corrosion. According to Sirisomboochai et al. (2021), To produce biodiesel, catalyst are used to complete the transesterification process. Due to high activity and low in cost, Calcium Oxide (CaO) were mainly choose to be as catalyst. Since a large amount of saucer scallops or *Ylistrum balloti* are produced yearly and it can be found at the roadside of the seafood stall, it contributed to air pollution which give stink smell. To overcome this situation, *Ylistrum balloti* was applied to be one of the catalyst. A good catalytic activity for this process has been found in Calcined Scallop Shell (CSS) which also shows alkaline characteristics and an unusual porous structure.

Biodiesel is literally the best alternative to encounter with global problem. Researches from Ogunkunle and Ahmed (2019) have investigated throughly and it turn out that biodiesel was choose to be a very brilliant substances. It has a low toxicity and higher in biodegradability. Additionally, it has lower combustion emission compare to petroleum diesel in term of emission. This is called closed carbon cycle which it is not contribute to global warming.

1.3 Research Objective

This study's primary goal is to maximise the biodiesel production from used WCO and produce clean and green biodiesel to replace fuel to biodiesel. The goals are as follows in further detail:

1. To produce biodiesel from WCO using *Ylistrum balloti* as heterogeneous catalyst via conventional method.
2. To analyze the production by utilising the variables such as molar ratio of oil to alcohol, catalyst concentration, reaction time and reaction temperature.

1.4 Scope of Research

The scope of this research are as follows:

1. Identify all parameters used to produce a high yield biodiesel production.
2. Produce heterogeneous catalyst from waste *Ylistrum balloti.*
3. Catalyst calcination ranging from 800˚C to 900˚C at 10˚C/min
4. Perform catalyst characterization using SEM-EDS.
5. Conduct the physical chemical preparation, raw WCO and methyl ester.
6. Testing the fuel properties according to ASTM D6751, EN 14212 and AOCS.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Energy efficiency has been cited as a significant strategy in today's modern society. To solve concerns including rising fuel prices, fiercer competition in the market, stricter regulations, climate change and the energy crisis has been studied by the decreasing supply of fossil fuel. It is crucial for utilities to provide effective methodologies to accurately and effectively analyse the size, variability and sources that occur in the system in order to conduct strategic planning and create an energy-efficient distribution network. Corrective and preventative solutions reduction may be planned and carried out properly. As well as promptly and effectively with the use of complete and accurate information of world biodiesel.

To get a better outcome, the researchs about biodiesel have been done. The best way to improve or solve this problem is to carry out an experiment to gain biodiesel. This is important because it can create attraction and awareness about the important of biodiesel without maximizing the required cost.

2.2 Advantages and disadvantages of biodiesel

Biodiesel have many pros and cons itself. Whether negative or positive, an concern action must be examined throughly, so an improvement can be made. However, biodiesel can give a huge impact on environment. Biodiesel are preferable than regular diesel fuel since it can sustain environmental health and also human safety. To convert biodiesel into efficient, safe and cheap, a proper care are needed and this is one of biggest disadvantage that we need to study. With adequate study, there might be a development of our future of engine using biodiesel in the near future. Table 2.1 present the list of advantages and disadvantages of using biodiesel.

Table 2.1 The advantage and disadvantages of using biodiesel

Advantages	**Disadvantages**
Biodegradibility	May damage rubber house engine
Safe to handle	Energy intensive production
Better lubricity	Poor cold weather performance
Renewable resources	Lower fuel efficiency than diesel
Environmental-friendly	Different storage and stability
Reduced greenhouse gas emission	Economic viability

The benefits of utilizing biodiesel can be categorized broadly into three main areas. That is environmental impact, energy security and economic implications. Previous research has shown that, in terms of environmental effects, biodiesel derived from vegetable oil leads to a 57% decrease in greenhouse gas emissions compared to traditional fossil diesel. Specifically, biodiesel produced from cooking oil results in an even more significant which is 86% reduction in greenhouse gas emissions when compared to fossil diesel (Cabrera-Jiménez et al., 2022).

Moreover, Aljaafari et al. (2022) presented a comprehensive review of biodiesel contributes to a positive impact on harmful exhaust emissions that reducing particulate matter by 47% in comparison to fossil diesel. Biodiesel also is a renewable resource as it is plant-based that allowed for regrowth of the feedstock. When it comes to energy security, it is important to note that fossil diesel would not last forever. On the flip side, biomass is a resource that can renew and it is available in many countries. Using local sources like biomass for energy helps reduce the reliance on fossil oil.

Regarding economic consequences, the bioenergy sector provided employment for 2.8 million individuals worldwide in 2014 were investigated by Ambaye et al. (2021). This industry also offers direct support to local agriculture, serving as an additional that means to support local farmers. Additionally, research has indicated that engine lifespan is extended when using biodiesel due to its natural lubricating properties. Finally, Aljaafari et al. (2022) describes that biodiesel presents a more pleasant exhaust odor, releasing a fragrance reminiscent of fried food or a barbecue when burned. Figure 2.1 illustrates global production of biofuel by country in 2022.

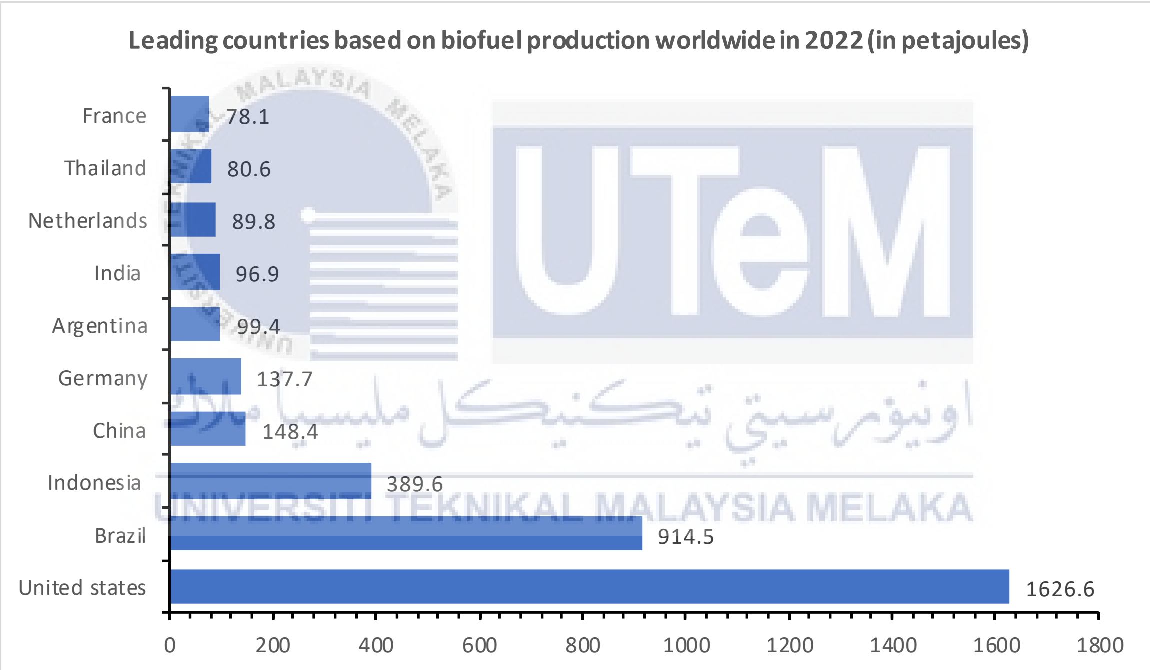

Figure 2.1 Global biofuel production by country in 2022 (Global Biofuel Production 2022 | Statista, 2023)

One appealing aspect of biodiesel is its compatibility with existing diesel engines, typically requiring minimal modifications. However, challenges arise concerning vehicle warranties and biodiesel use. Additionally, Yusoff et al. (2020) indicated that biodiesel has a nearly 10% lower energy content and distinct physical properties compared to conventional diesel oil leading to certain alterations in engine performance and emissions.

Biodiesel is considered safe as it lacks hazardous materials. Zulqarnain et al. (2021) carried out numerical study that have shown biodiesel breaks down much faster than regular diesel. In environmentally delicate areas like wetlands, marine environments and national parks. Users have chosen for biodiesel to replace harmful petroleum diesel, taking advantage of its quicker biodegradability. However , nowadays biodiesel is more expensive than regular diesel due to its production mainly relying on pure vegetable oil. Nevertheless, Monika et al. (2023) suggested that the cost of biodiesel could be lowered by utilizing more affordable raw materials such as animal fat and WCO.

2.3 Type of feedstock

Biodiesel was produced using organic and natural materials. Specific considerations must be made throughly while selecting the proper feedstock since it can reduce the cost of producing biodiesel. A sizeable portion of the total cost of biodiesel production is referable to the cost of raw ingredients. Biodiesel derived from various feedstocks exhibits notable variations in purity and composition. The initial selection of feedstock at a biodiesel plays a crucial role in determining key factors like biodiesel purity, cost, composition, and overall production.

WCO may be turned into biodiesel which eliminates the need to dispose of used cooking oil and saves waste management expenses by replacing out pricey edible vegetable oils for less expensive WCOs. Studies have found that utilising less costly raw materials such as animal fats, non-edible oils, residues from the refining of vegetable oils and WCOs can help reduce the cost of producing biodiesel (Topare et al., 2022). The main feedstock sources for the manufacturing of biodiesel depicted in Table 2.2.

Table 2.2 A major source of feed stock for biodiesel production.

Common feedstock group	Type of oil
Edible oil	Apricot, artichoke, avocado, almond seed, coconut, corn, cottonseed, cashew, babassu, false-flax, grapeseed, groundnut, hazelnut, hibiscus seed, macadamia, maize, mustard, olive, peanut and pecan
Non-edible oil	Castor oil, Copaiba oil, crude castor oil, crude rubber seed oil, Jatropha oil, Jojoba oil, Mahua oil, milk bush oil, Nagchampa oil, Neem oil, Karanja oil, Soap nut fruit seed oil, and Tobacco seed oil are a few of the oils that are listed as being bitter.
Animal fats	Tallow from beef, tallow residue, Fish oil, refined lard, chicken fat, and leftover pig carcasses
Others	Sewage sludge, brown grease, fungi, and algae oil Waste cooking oil, waste biomass oil

2.3.1 First generation

First-generation biodiesel is produced from food-based feedstocks. Edible feedstocks for this type of biodiesel include rapeseed, soybean, coconut, palm, mustard, olive oils and others. According to investigation by Ogunkunle et al. (2019), first generation feedstock has a variety of nutritive components and it is almost clean and healthful. There are no chemical steps involved in the extraction of edible oil from its sources. In the early days of the biodiesel era, producing biodiesel from edible feedstocks was common. Plants for these feedstocks were easily accessible and the conversion process was straightforward. This making the first-generation feedstocks an appealing choice.

According to Veluru et al. (2022), using these feedstocks raised concerns about increasing the cost of food products and the potential for food shortages. The adaptability to environmental conditions for biodiesel production from edible feedstock, coupled with high costs and limited planting area, presented challenges. These drawbacks prompted biodiesel producers to explore alternative fuel sources.

2.3.1.1 Waste cooking oil

A previous research from Monika et al. (2023), the used of WCO are a suitable choices for biodiesel production. It is also distinguished from other oils by its affordability, availability, convenience of collection from homes and restaurants and renewability. Additionally, it reduces the requirement for growing crops for biodiesel. Despite their relevance in the manufacturing of biodiesel, leftover cooking oils are often disposed of in dangerous ways that cause environmental contamination.

High concentrations FFAs are frequently found in used frying oil. FFAs should be handled and decreased to an adequate level since they are unwanted components that impede the transesterification reaction and limit the biodiesel production. As highlighted by Bhonsle et al. (2022), leftover cooking oil is utilized to make 89% of the biodiesel. Waste cooking oil depicted in Figure 2.2

Figure 2.2 Waste cooking oil (Rettke, 2022)

2.3.2 Second generation feedstocks

Second-generation biodiesel is produced from nonedible feedstocks including nagchampa oil, jatropha oil, neem oil, calophyllum inophyllum oil, karanja oil, r ubber seed oil and madhua indica oil. The exploration of non-edible feedstocks is driven by the limitations of first-generation feedstocks. Study by Brahma et al. (2022) found that instead being environmentally friendly and cost-effective, second-generation biodiesel addresses issues of food inequity and diminishes the demand for farmland.

Utilizing second-generation feedstocks eliminates the necessity for replanting food and agricultural land (Singh et al., 2021). However, there are some drawbacks associated with second-generation fuels. That is lower in biodiesel. The high concentration of FFAs in non-edible oil which reacts with the base catalyst to generate emulsions and soap and impede the base transesterification process and reduce biodiesel production. Figure 2.3 exhibits non edible sources used in biodiesel production

Figure 2.3 Non edible sources used in biodiesel production (Vilas Bôas & Mendes, 2022)

2.3.3 Third generation feedstocks

Based on research made by Reetu et al. (2023), The biodiesel known as third-generation biodiesel is made from microalgae. The production of biodiesel has the outstanding benefit of significantly lowering greenhouse gas emissions. It also promises faster development and productivity, less rivalry for agriculture land, more oil production, and less of an influence on the world's food supply. But issues including high production costs, solar radiation dependency, mass manufacturing complexity and oil extraction obstacles must be resolved.

Studies have found that the goal of current research efforts is to improve the extraction procedures and increase the pace at which biodiesel is produced from algal biomass (Yaashikaa et al., 2022).This biodiesel generation's sources aim to overcome issues associated with earlier generations, including concerns about feedstocks affecting the food chain and environmental conditions. Notably, third-generation biodiesel demonstrates adaptability to diverse requirements, such as economic viability and environmental considerations.

2.4 Methods to produce biodiesel

An alcohol and feedstock such as fat or oil are a compulsory when it come to a good biodiesel production. Through the feedstock quality, whether tranesterification, dilution, microemulsion and transesterification processes are employed to produce biodiesel. The most popular production method are through transesterification. This technique is effective when the oil or fat has less than 0.1% free fatty acid, less than 0.1% moisture and less than 10 ppm phosphorous (S Brahma et al., 2022). This criteria need to be studied in order to fulfill the standard that potray a good biodiesel production

Frequent feedstocks for biodiesel production are soybean, rapeseed, sunflower, cottonseed, palm seed, palm kernel oil, corn and mustard seed oil are all used. It is also possible to produce biodiesel from hog, beef, and poultry fat and grease. Palm oil and animal fat contain large amounts of free fatty acids which encourage the creation of soap. This has a harmful impact on downstream processing and lowers productivity. The main process, advantages and disadvantages of each biodiesel process was given in Table 2.3.

Table 2.3 Main process, advantages and disadvantages of biodiesel production (Suzihaque, et al.,2022)

Process	Main process	Advantages	Disadvantages
Pyrolysis	Preheating is used to break down the oil, which can happen with or without a catalyst at a specific temperature	The method is easy and free waste pollution.	Equipment that costs a lot and operates at high temperatures is required.
Transesterification	A reaction involving alcohol and carboxylic acid that has a catalyst present. A glycerol will be obtained at the end of the process by separating to alcohol and biodiesel.	High conversion at cheap cost, gentle reaction conditions, and applicability for industrial production bring biodiesel closer to petroleum diesel.	There several purification and separation procedures. It also contain low FFA and moisture in raw material
Ultrasonic heating method	Obtain the viscosity of the oil by solubilized in a specific solvent.	Pollution free and easy to conduct.	High viscosity, limited stability, incomplete combustion.
Microwave irradation method	Utilizes microwave radiation to heat and interact with materials for various purposes, including chemical synthesis, sample preparation, and material processing	Can accelerate chemical reactions compared to traditional methods, reducing reaction times and increasing overall efficiency.	expensive than traditional heating apparatus. Initial setup costs may be a consideration, especially for smaller laboratories.

2.4.1 Pyrolysis

As mentioned by Pourkarimi et al. (2019), pyrolysis represents a thermochemical method wherein biomass undergoes heating within the temperature range of 400ºC to 500ºC, all the while excluding exposure to oxygen. This results in the formation of bio-oil, exhibiting similarities to crude oil. The subsequent conversion of bio-oil into transportation fuels involves two primary processes: hydrocracking and hydrotreating.

Hydrotreating is a procedure where bio-oil reacts with hydrogen to eliminate sulfur and oxygen from the oil. The hydrotreated bio-oil necessitates a secondary reaction with hydrogen before it becomes suitable for the production of gasoline and diesel fuels, characterized by the requisite short hydrocarbon chains essential for hydrocracking.

2.4.2 Tranesterification

Alkyl esters that is commonly known as biodiesel are generated through the transesterification of oils and fatty acids. In the transesterification process, triglycerides undergo a reaction with alcohol such as methane or ethane, resulting in the production of glycerol and fatty acid esters. Methyl esters are the more preferred choice for alkyl ester production in many countries due to their lower cost compared to ethyl esters. Since the inception of biodiesel, various studies have explored transesterification methods.

Transesterification, a chemical process extensively applied in the production of biodiesel from natural oils and fats, involves the combination of three moles of alcohol (MeOH) with one mole of triglyceride. Glycerol, a byproduct of this process, is typically formed at temperatures ranging from $60^{\circ}C$ to $65^{\circ}C$ with the aid of a catalyst. The outcome of this process is the formation of alkyl ester, commonly known as biodiesel.

The initial step involves the creation of diglycerides through the reaction of MeOH with triglyceride. Subsequently, MeOH reacts with the diglycerides to produce monoglycerides. The final products of this reaction include monoglyceride, MeOH, and glycerol. Figure 2.4 below shows biodiesel production from different feedstock .

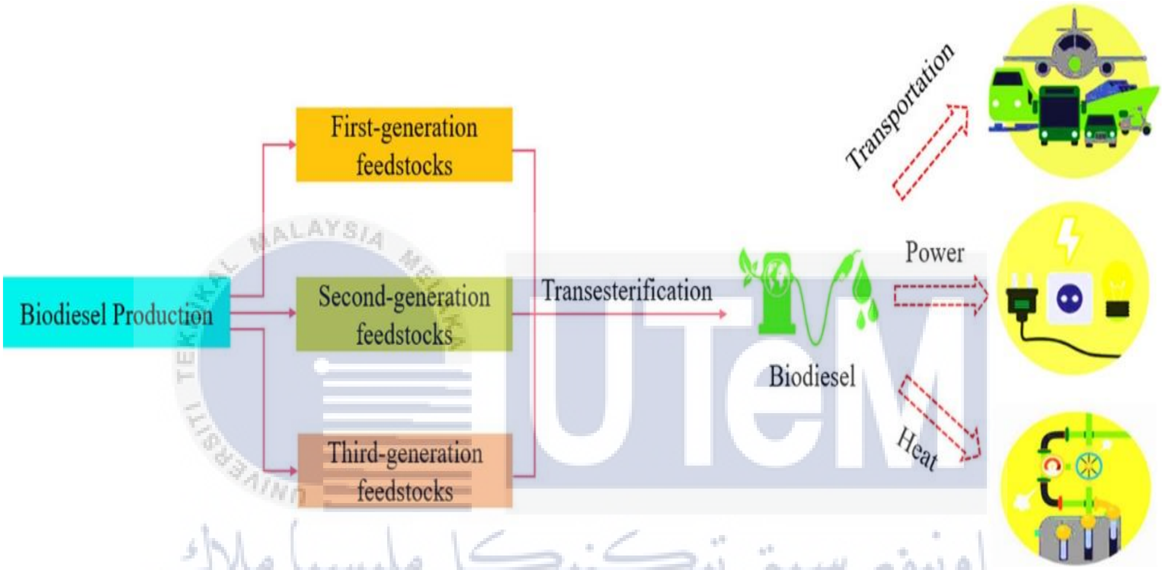

Figure 2.4 Biodiesel production from different feedstock generation (Zulqarnain et al., 2021).

2.4.3 Microwave irradation method

Microwave irradiation also known as microwave-assisted synthesis or heating, employs microwave radiation to interact with materials for various applications such as chemical synthesis, sample preparation, and material processing. Microwaves, a form of electromagnetic radiation, permeate the material, inducing molecular rotation and generating heat. This method ensures efficient and uniform heating, distinguishing it from conventional heat transfer mechanisms like conduction or convection, which may cause uneven temperature distribution (Ramamurthy & Krishnan, 2022).

Bao et al. (2023) describes that microwave irradiation finds application across diverse fields such as organic synthesis, material science, and analytical chemistry. Notably, its usage is not universal, and not all reactions are compatible with this method. The selection of the appropriate technique depends on the specific demands of the synthesis or process under consideration.

2.4.4 Ultrasonic heating method

Ultrasonic heating is not a conventional method for biodiesel production but ultrasonication is utilized as a supplementary process to enhance efficiency. According to Oliveira et al. (2021), in biodiesel production, typically achieved through transesterification, ultrasonication induces cavitation, improving mass transfer, reaction kinetics, and reducing particle size. The process creates localized high temperatures and pressures, potentially accelerating the reaction. While ultrasonication aids in certain aspects, it is not the primary heating method; traditional heat sources remain crucial. Researchers explore ways to optimize ultrasonication's role in biodiesel production, aiming to increase overall efficiency and yield..

2.5 Transesterification for biodiesel

Oil-derived triglycerides which is fats are transformed into biodiesel by a chemical process called transesterification. Because it has a far lower viscosity than petroleum diesel, biodiesel produced through the transesterification process can replace it in diesel engines. The industrial biodiesel manufacturing method utilised internationally involves transesterification of triglycerides, which make up the majority of vegetable oils and animal fats. MeOH is generally the cheapest alcohol, methyl esters are the most typical form of esters.

However, ethanol is cheaper than alcohol. Ethyl esters are the most often created esters. Bases, acids, and enzymes can catalyze the reaction. It occurred at low or high temperatures. Biodiesel is made from the methyl esters of fatty acids generated. From this reaction, glycerol is obtained as a reaction byproduct (Ulakpa et al.,2022).

According to the finding of one research by Hoogendoorn and Kasteren (2020), Glycerol is a result of transesterification, which normally occurs with the help of a catalyst at 60 °C to 65 °C. Biodiesel (alkyl ester) is the final product. A starting point is the conversion of MeOH l and triglycerides into diglycerides. Diglycerides and MeOH combine to form monoglycerides. The end results of this process are monoglyceride, MeOH and glycerol. The transesterification process were illustate in Figure 2.5.

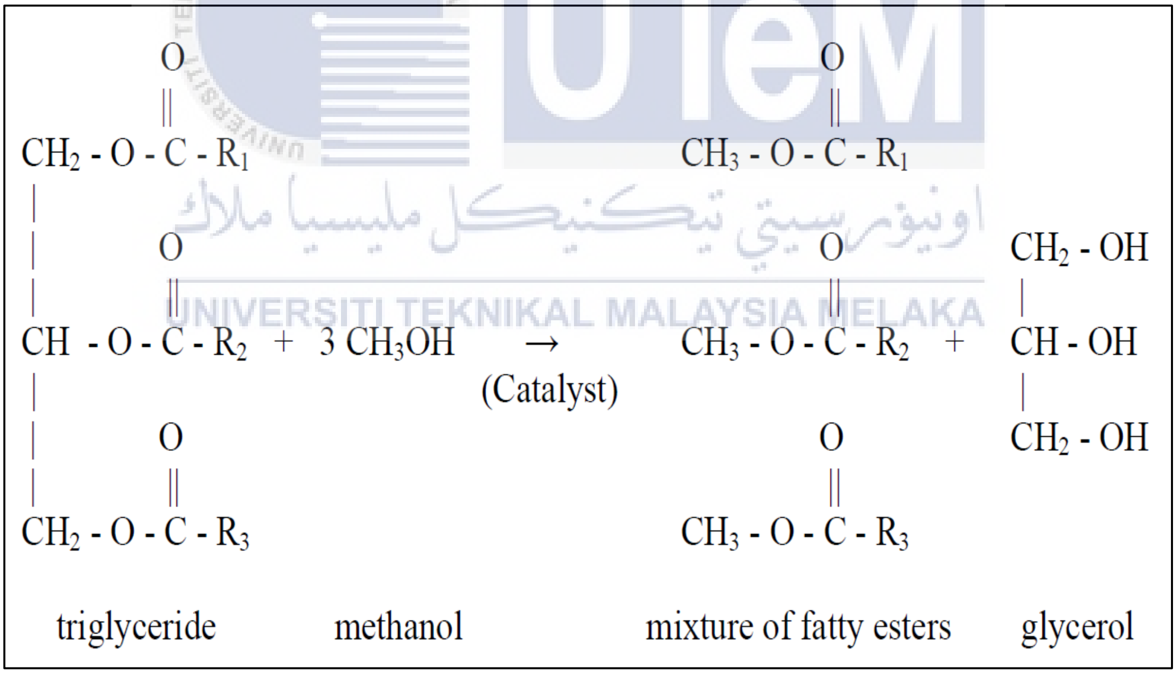

Figure 2.5 Transesterification process to biodiesel (Vignesh et al.,2019)

2.6 Variables affecting the transesterification reaction

Numerous studies have found that the key factors affecting the transesterification process include the molar ratio of oil and alcohol, the type and concentration of the catalyst, the reaction temperature, reaction time and others. These are crucial variables that require investigation since they are crucial in producing high-quality biodiesel. These indicators must all be measured in great detail. Figure 2.6 present the important parameter involved in producing a clean biodiesel.

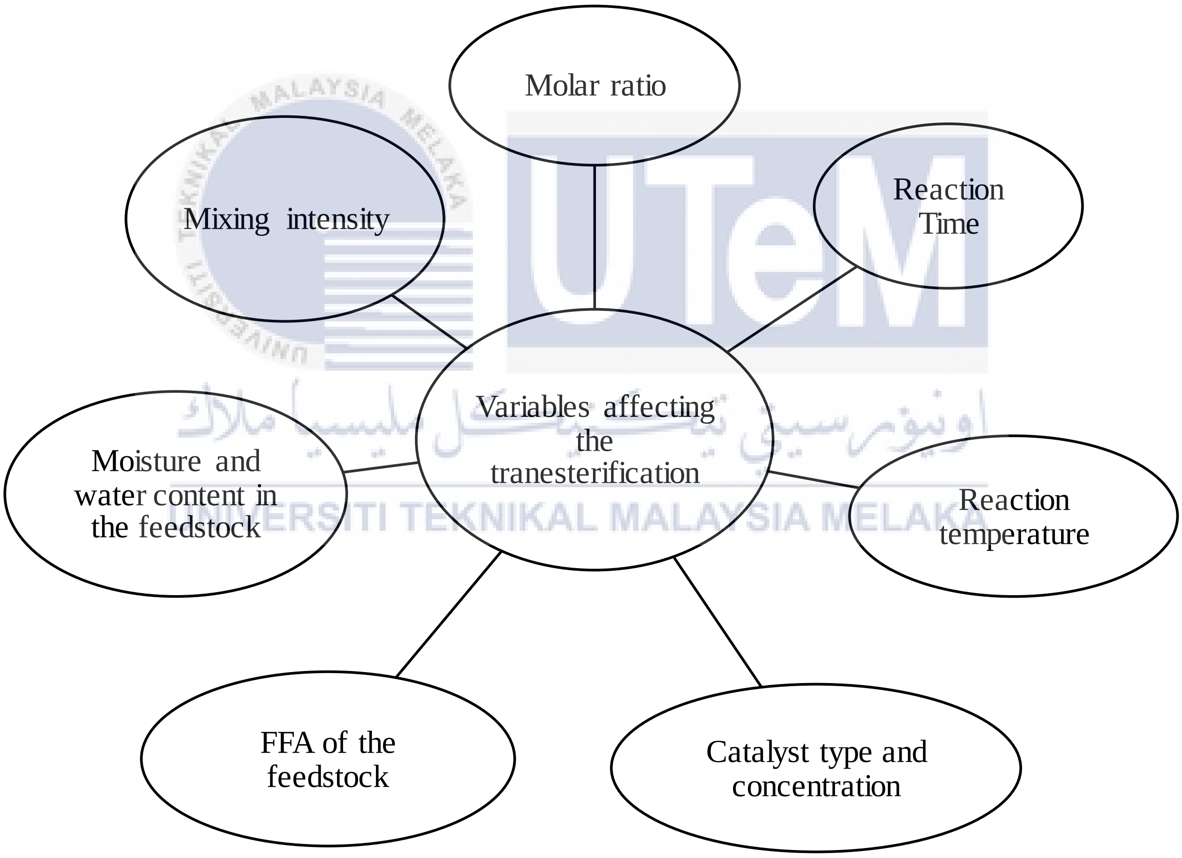

Figure 2.6 Variables that affecting the transesterification

2.6.1 Molar ratio

The most popular variable in transesterification process is the molar proportion of alcohol to oil. Suzihaque et al. (2022) proposed that some of the alcohols that may be used to make biodiesel include MeOH ethanol, isopropyl alcohol, propanol, pentanol, and butanol Research reveals that MeOH is often used in the production of biodiesel since its commercial significance is less significant than that of other longer chain alcohols.

Consequently, transesterification for biodiesel might be carried out more cheaply. Biodiesel might be produced from it since recycling it is simple. The oil to MeOH ratio during transesterification is influenced by the catalyst type. In comparison to base catalyst, an acid catalyst requires a higher oil to MeOH ratio.

2.6.2 Reaction time

The production of biodiesel typically increases as the reaction time lengthens. The feedstock, catalyst, and concentration all have a role in the optimal transesterification reaction time. At a rate that was 4000 times quicker than acid catalyst, base catalyst produced the most biodiesel. (Suzihaque, et al.,2022) One reason for the extensive usage of acid catalyst as a pre-treatment before the transesterification process is due to this.

Lack of response time will cause the alcohol and raw ingredients to be improperly mixed and distributed. Starting with a very low output of biodiesel, the process begins. The feedstock has evolved over time to include the two major triglycerides, monoglyceride and diglyceride, to facilitate the synthesis of biodiesel.

The optimal reaction time for the transesterification of spent cooking oil was 60 minute according to a research by Topare in the year of 2021. The best amount of reaction time for the transesterification process, according to the study was 5 hours. This was because the WCO had significant quantities of free fatty acids. Four hours was the best reaction time for transesterification of cooking oil, according to tests with low free fatty acid levels of WCO (Saad et al.,2020).

2.6.3 Reaction temperature

An reaction called endothermic reaction is the one that boost temperature for production of biodiesel during the process of transesterification. Generally speaking, when the reaction's temperature rises, more biodiesel will be produced, resulting in a decrease in an oil's viscosity value. This is caused by two factors: the oil's high viscosity at low temperatures and the lack of a reaction temperature, which causes the reactants to not mix well. However, increasing the reaction temperature above the optimal temperature stops the formation of biodiesel by forcing the alcohol to evaporate.

Alcohol will evaporate if a reaction temperature over the transesterification alcohol's boiling point. Shortening the time the alcohol was in contact with the oil and reducing the amount of overall biodiesel produced. According to studies, during the transesterification process, WCO performed best at a temperature of 600˚C. The optimal temperature for WCO during the transesterification process, according to research with a high free fatty acid content of WCO, was 750˚C. 650˚C was the best transesterification process temperature for WCO, according to research with low free WCO fatty acid (Monika et al.,2023).

2.6.4 Catalyst loading

Catalyst is a substance that help the whole process to boost up the reaction. With a help from a catalyst, the molecules can interact in a split of second rather than it might take times. During the chemical reaction, molecules break the chemical bond between their atom.

The different between atom also will make a new bond. This bonded atom make bond stronger to each other.

Depending on the catalyst type and concentration, transesterification occured. An acid catalyst is preferred for feedstocks that include a lot of FFA, whereas a base catalyst is suggested for feedstocks that contain less FFA. By decreasing the process's activation energy, any kind of catalyst can speed up a reaction. Catalyst concentration are one of the key factors that greatly affects the production of biodiesel. With lower catalyst concentrations, less biodiesel is typically produced. The reduced yield is due to the lower catalyst concentration which leaves less catalytic surface available for the transesterification process to take place.

Yields reached their peak and then began to decline as the catalyst concentration increased. By creating slurry, raising viscosity, and then boosting soap production, increased catalyst concentration decreased biodiesel yield. The output of biodiesel will decline as soap creation rises. Therefore, the best amount of catalyst was usually researched while producing biodiesel through transesterification. Figure 2.7 show the classification of different catalyst.

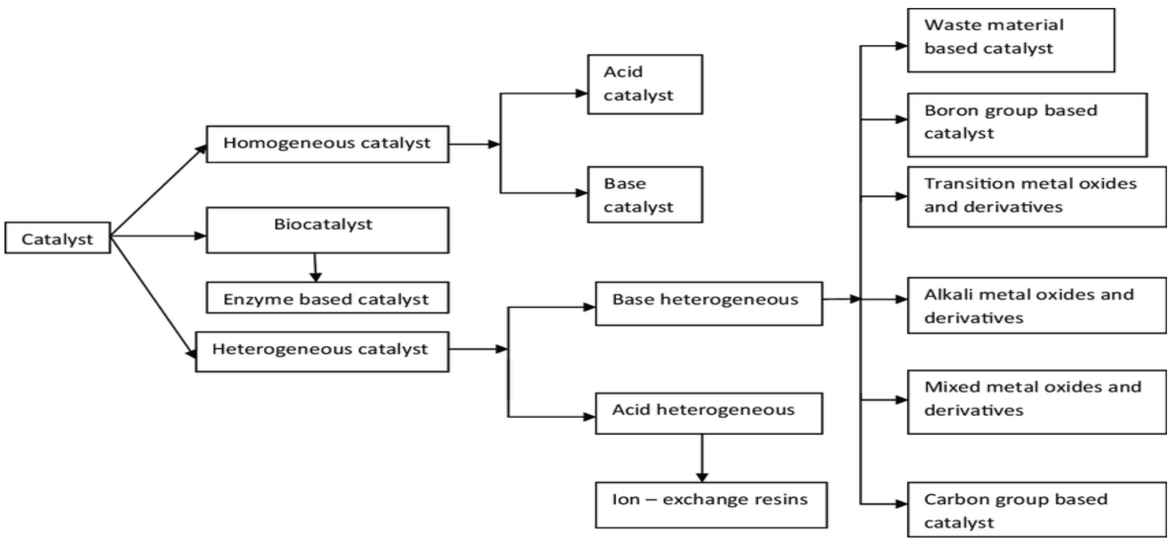

Figure 2.7 Classification of different catalyst (Moazeni et al., 2019)

Homogeneous catalysts speed up reaction rates and aid in transforming triglycerides into biodiesel and glycerol. However, employing homogeneous catalysts comes with certain disadvantages including the possibility of soap formation, the necessity for meticulous separation and washing procedures, and the need for precise control over reaction conditions (Mandari & Devarai, 2021). Scientists are actively investigating and advancing catalysts to improve the effectiveness and sustainability of biodiesel production methods.

According to Murat Kibar et al. (2023), heterogeneous catalysts present benefits such as simplified separation, reusability, and diminished concerns regarding soap formation when contrasted with homogeneous catalysts. Furthermore, they enable continuous processes and are frequently more environmentally sound. Nevertheless, the selection of a catalyst hinges on multiple factors, encompassing the particular feedstock, reaction conditions, and overall process considerations. Scientists persist in investigating and refining heterogeneous catalysts to enhance the efficiency and sustainability of biodiesel production.

From the variable for transesterification process above, an outcome for biodiesel production was studied. A different feedstock, catalyst type, reaction time, catalyst loading, reaction time and biodiesel yield was analyze. This Table 2.4 show variable catalyst for tranesterification process. A different variable might produce a different biodiesel yield. However, it can be as our references.

Table 2.4 Variable for transesterification process

Feedstock	Catalyst type	Reaction temperature (°C)	Molar ratio (MeOH:Oil)	Catalyst loading (wt.%)	Reaction time (min)	Biodiesel Yield (%)	Authors
Goat fat	Nano	70	12:1	1	180	93.7	Khalifeh, R. et al., 2020
Palm	Lime	65	15:1	6.0	120	97.0	Roschat W et al., 2016
Canola	Dolomite	60	9:1	5.0	90	97.4	Korkut et al., 2016
Palm	Commercial	60	9:1	2.0	240	84.5	Poosumas J., 2016
WCO	NaOH	55	6:1	1	70	94.0	Soji-Adekunle A R, et al.,2018
Soybean	CaO	65	15:1	3	120	97.80	Huang, J. et al.,2021
Canola	Eggshell	50	9:1	3	180	97.6	Maryam K, et al., 2020
Pongamia pinnata	KOH	150	10:1	1.0	90	92	Wilkanowicz SI, et al, 2020

2.7 Biodiesel standard

Biodiesel standards is essential to uphold product quality, guarantee compatibility with existing infrastructure, minimize environmental impact, instill consumer confidence, comply with regulations, and facilitate global trade in biodiesel products. Table 2.5 show ASTM D6751 and EN 14214 properties. These are the common testing in order to fulfill the standard. However, most of researcher will not run all the testing. The main reason is due to the cost. Event one testing required a lot of money due to the machinery cost. It also depends on the purpose of the experiment.

There were several factors that impacted biodiesel quality. Filtration, feedstock composition, production of biodiesel technology and oil extraction method are a few examples. Authorities set a high requirement for biodiesel quality. The biodiesel generated has to meet to the American Society for Testing Materials (ASTM 6751), European Standard (EN 14214), and other standards and specifications (Brahma et al.,2022). These standards provide guidelines for evaluating biodiesel fuels for their numerous physical and chemical characteristics that make them suitable for use in engines..

The EN 14214 and ASTM D6751 standards must be met by the oils used in the manufacturing of biodiesel. US and EU guidelines serve as the foundation for biodiesel rules in other nations. Renewable feedstock and diesel fuel are used to create B100 biodiesel. The ASTM D6751 standard describes biodiesel as long chain fatty acid mono-alkyl esters made from vegetable and animal fats (Hamamre et al, 2023).

They may be made with MeOH or ethanol as long as the mono-alkyl esters satisfy the requirements. According to Haster,2018, a minimum ester percentage of 96.5%, EN 14214 limits the use of biodiesel to mono-alkyl esters produced from MeOH and fatty acid methyl esters (FAME). Table 2.5 show the ASTM D6751 and EN 14214 properties.

Table 2.5 ASTM D6751 and EN 14214 properties (Sakthivel, 2018)

PROPERTY SPECIFICATION	UNITS	DIESEL ASTM D975		BIODIESEL			
				ASTM D6751		EN 14214	
		TEST METHOD	LIMITS	TEST METH0D	LIMITS	TEST METHOD	LIMITS
Flashpoint	°C	ASTM D975	60–80	ASTM D 93	130 min	EN ISO 3679	101 min
Cloud point	°C	ASTM D975	−15 to −5	ASTM D2500	−3 to −12	-	-
Pour Point	°C	ASTM D975	−35 to −15	ASTM D97	−15 to −16	-	-
Cetane number	-	ASTM D4737	46	ASTM D613	47 min	EN ISO 5165	51 min
Density at 15 °C	Kg/m3	ASTM D1298	820-860	ASTM D 1298	880	EN ISO 3675/ 12185	860–900
Kinematic viscosity at 40 °C	mm^2/s	ASTM D445	2.0 to 4.5	ASTM D445	1.9–6.0	EN ISO 3104	3.5–5.0
Iodine number	g I2/ 100 g	-	-	-	-	EN 14111	-
Acid number	mg KOH/g	-	-	ASTM D664	0.5 max	EN 14104	0.5 max
Cold filter plugging point	°C	EN 590	-8	ASTM D6371	Max +5	EN 14214	
Oxidation stability	-	ASTM D2274	25 mg/L max	-	-	EN 14112	3h min
Carbon residue	% m/m	ASTM D4530	0.2 max	ASTM D4530	0.050 max	EN ISO 10370	0.3 max
Copper corrosion	-	ASTM D130	Class 1 max	ASTM D130	No. 3 max	EN ISO 2160	Class 1
Distillation temperature	°C	ASTM D86	370 max	ASTM D1160	360	-	-
Lubricity (HFRR)	m	IP 450	0.460 mm (max.) (all diesel containing less than 500 ppm – sulfur)	ASTM D6079	520 max	-	-
Sulphated ash content	%mass	-	-	ASTM	ASTM D874 0.002 max	EN ISO 3987	0.02 max
Ash content	%mass	ASTM D482	100 max	-	-	-	-
Water and sediment	%mass	ASTM D2709	0.05 max	ASTM D2709	0.005 vol% max	EN ISO 12937	500 mg/kg
Monoglycerides	%mass	-	-	-	-	EN 14105	0.8 max
Diglycerides	%mass	-	-	-	-	EN 14105	0.2 max
Triglycerides	%mass	-	-	-	-	EN 14106	0.2 max
Free glycerine	%mass	-	-	ASTM D6584	0.02 max	EN 1405/ 14016	0.02 max
Total glycerine	%mass	-	-	ASTM D6548	0.24	EN 14105	0.25
Phosphorus	%mass	-	-	ASTM D4951	0.001 max	EN 14107	0.001 max
Sulphur (S 10 grade)	ppm	ASTM D5453	10 max	-	-	-	-
Sulphur (S 15 grade	ppm	-	-	ASTM D5453	150 max	-	-
Sulphur (S 50 grade)	ppm	ASTM D5453	50 max	-	-	-	-
Sulphur (S 500 grade)	ppm	ASTM D5453	500 max	ASTM D5453	500 max	-	-
Carbon	wt%	ASTM D975	87	ASTM PS121	77	-	-
Hydrogen	wt%	ASTM D975	13	ASTM PS121	12	-	-
Oxygen	wt%	-	-	ASTM PS121	11	-	-
Total contamination	mg/kg	-	-	ASTM D5452	24	EN 12662	24
Boiling point	°C	-	-	ASTM D7398	100-615	-	-
Saponification value	mg KOH/g	-	-	ASTM D5558 − 95	370 max	-	-

2.8 Catalyst characterization

Loy et al. (2024) showed that the examination and comprehension of the features and arrangement of catalyst material was encompassed by the term catalyst characterization. These materials play a crucial role in chemical processes. Characterizing catalyst is essential for gaining deeper understanding of the catalyst'behaviour, performance and the processes underlying catalytic reactions. In this study, the Scanning Electron Microscope (SEM), X-ray diffraction (XRD) and X-ray flurescene (XRF) are employed as methods to analyze and describe the calcined catalyst.

2.8.1 X-ray fluorences (XRF)

According to ThermoFisher Scientific (2020), XRF is an analytical method that non-destructively determines the elemental composition of a material. This technique involves exposing the sample to X-rays, prompting the emission of flurescent X-rays from the atoms in the sample. By measuring the enerfy and intensity of these emitted X-rays, one can identidy and quantity the elements present in the sample.

It is widely applied across diverse fields such as geology, environmental sciences, archeology and material science. Marguí et al. (2022) studied XRF offers a rapid and simultaneous analysis of multiple element in various types of samples. Its capability to provide both qualitative and quantitative insights inti the elemental composition maked it a valuable tool in scientific research and industrial applications.

2.8.2 X-ray diffraction (XRD)

XRD is an anvanced analytical technique utilized to unveil the crystallographic structure of material. By exposing a sample to X-rays and observing the resulting diffraction pattern, scientists can discern valuable information about the arrangement of atoms within the crystal. This method is instrumental in identifying crystal structures, determining crstallographic phases and unveiling various structural characteristics of materials (Vasile–Adrian Surdu & Romuald Győrgy, 2023).

It is widely appplied in the fields such as chemistry, physics, material science and geology. Ali et al. (2022) analyzed that XRD is particularly effective in studying crystalline material like minerals, metals and ceramics. It enables researchers to gain profound insights into the composition, crystal symmetry and other structural properties of materials. This method can enhancing understanding of their behaviour and performance.

2.8.3 Scanning element microscope (SEM)

As highlighted by Lou et al. (2023), SEM is a cutting-edge imaging technique in the realm of microscopy for its ability to provide high-resolution visualizations of material surfaces at a minute scale. Employing a focused beam o f electrons, SEM scans the surface of a sample and the resulting interactions between the electrons and atoms produce signals that are harnessed to generate intricate images.

Distinguised by its superior magnification and resolution, SEM surpasses the capabilities of traditional light microscopes. Its applications span a wide array of scientific and indutrial domains, encompassing fields such as biology, geology and nanotechnology (Bai et al., 2024). By facilitating detailed examination, composition and elemental distribution within samples, SEM serves as an invaluable tool for researches seeking comprehensive insights into microstructural intricacies of materials.

In essence. SEM stands at the forefront of microscopic exploration that enable scientist to dig into finer details of diverse materials and contributing significantly to advancement of understanding of their characteristic and functionalities.

2.9 Sustainable Development Goal for biodiesel production (SDG)

The Sustainable Development Goals (SDGs), established by the United Nations in 2015, constitute a set of 17. Global targets aiming to tackle pressing challenges and foster a more sustainable, inclusive world by 2030 (Glass & Newig, 2019). Covering areas from poverty reduction to climate action, the SDGs address interconnected issues, providing a universal call to action for governments, businesses and individuals alike.

Encompassing diverse aspects such as health, education, gender equality, and environmental sustainability. These goals serve as a collective framework. It was use to underscoring the imperative for cooperative efforts to achieve social, economic, and environmental progress on a worldwide scale. This goals benefiting both current and future generations. Figure 2.8 below exhibits the related SDG regarding to the biodiesel prodction.

Figure 2.8 Sustainable goal development for biodiesel production

Sustainable Development Goal 7, emphasizing "Affordable and Clean Energy," positively impacts biodiesel production. Biodiesel, a renewable substitute derived from biomass, effectively curbs greenhouse gas emissions and encourages sustainable farming practices. The use of locally sourced feedstocks enhances energy security and diminishes reliance on imported oil. Biodiesel's environmentally friendly combustion improves air quality and its integration into existing infrastructure facilitates a smooth shift to a more sustainable energy source (Morte et al., 2023).

Biodiesel production reinforces the attainment of Sustainable Development Goal 8, "Decent Work and Economic Growth." This sector creates job opportunities in agriculture, feedstock cultivation and biofuel processing particularly benefiting rural areas. As a renewable energy focus, biodiesel aligns with Goal 8's emphasis on job creation and fostering inclusive economic advancement (Blair et al., 2021c). The industry's commitment to innovation and local resource utilization enhances the potential for sustainable employment, promoting economic growth and offering decent work opportunities for diverse communities.

Next, Sustainable Development Goal 13, "Climate Action." Functioning as a renewable and lower-carbon substitute for traditional fossil fuels. Biodiesel plays a crucial role in mitigating climate change by reducing greenhouse gas emissions during combustion. Sourced from sustainable feedstocks, biodiesel actively advocates for a more sustainable energy future, aligning seamlessly with the goal's overarching aim of combating climate change and its adverse effects. The industry's steadfast commitment to cleaner energy sources and environmentally friendly practices propels the global transition towards a low-carbon economy (Kiehbadroudinezhad et al., 2023b).

CHAPTER 3

METHODOLOGY

3.1 Introduction

The detail process of the study were presented in this section. The method, chemical preparation and selection were explained. The process involved such as catalyst characterization, the transesterification process ande the fuel testing were identified. st characterization.

3.2 Material and equipment

In this study, samples of biodiesel are produced by utilizing *Ylistrum balloti* as the main heterogeneous catalyst with the aid of conventional transesterification method. WCO was converted to biodiesel using a few components and a few specific techniques, which are each explained in more detail below. Figure 3.1 shown the waste *Ylistrum balloti* before undergone cleaning process.

Figure 3.1 Waste *Ylistrum balloti*

3.2.1 Preparation of waste cooking oil

The raw WCO was obtained from Dietetic and Food Service Department, Hospital Melaka. WCO was preferred due to its characteristic such as readily available, relatively inexpensive and available in large quantities. The raw WCO neet to be filter off using very fine mesh filter to remove the unwanted debris, contaminant and impurity.

3.2.2 Apparatus

This experiment will be carry out at lab located at Fakulti Teknologi Kejuruteraan Mekanikal (FTKM) . Most of the chemicals that were used in this research were purchased from Polyscientific Chemicals Sdn Bhd, Melaka Filter paper with the size of 12.5 cm, a separating funnel, a thermometer, a hot plate, a retort stand, a magnetic stirrer, conical flasks and beakers were all used throughout the experiment. The list of tests and equipment used for characterization of oil characteristics may be found in Table 3.1.

Table 3.1 List of test and equipment used for oil properties characterization

No	Property	Equipment	Unit	Standards
1	Flashpoint	Pensky-martens flash point – automatic NPM 131	°C	ASTM D 93
2	Cloud point	Cloud point tester	°C	ASTM D2500
3	Pour point	Pour point tester	°C	ASTM D97
4	Density	Pycnometer	g/cm3 (15°C)	ASTM D 1298
5	Kinematic viscosity	HK 265A	mm2 /sec, (40°C)	ASTM D445
6	Iodine number	Titration	gI2/100g	EN 14111
7	Acid number	Titration	Mg KOH/g	ASTM D664
8	Water content	Volumetric KF titrator	% w/w	ASTM D2709
9	Calorific value	C2000 basic colorimeter	J/kg	ASTM D 975

3.3 Flow chart

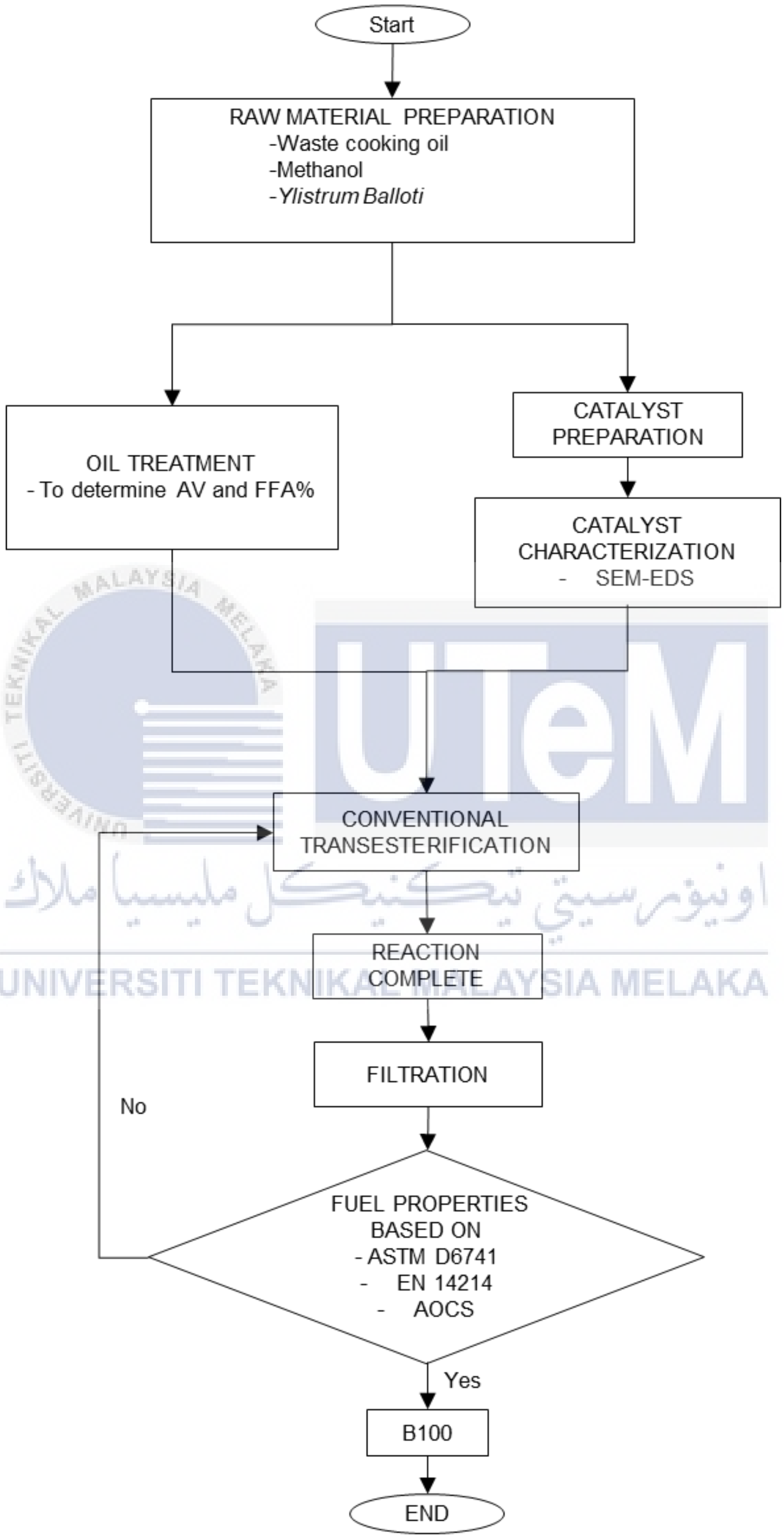

Figure 3.2 Workflow of the research activity

3.3.1 The production process

Figure 3.2 presented the overall process in producing the Free Acid Methyl Ester (FAME) from waste WCO and waste *Ylistrum balloti*. The experiment begun with the heating process of raw WCO. The oil need to be heat up untill it reach 110˚C to 120˚C and let it stay for about 30 minutes. It is to remove the presence of water in oil as the boiling point for water is 100˚C. After completed the heating process, the temperature of oil need to be between temperature of 70˚C.

Catalyst need to be prepared while the oil reach 70˚C. A weight of 0.5g CSS and 33.0 g of MeOH are measured using weighting balance and mixed it together in a beaker. Once the subtances are mixed, leave it for about 10 minutes. The solution heated up for a better reaction between the mixtures and the treated oil.

A weight of 200 ml of WCO was recorded. A prepared mixtures of MeOH and catalyst was poured into the WCO then placed it on stirring hot plate with a temperature of 60˚C to 65˚C with the reaction time of 90 minutes. The beaker was wiped frequently using tissue to remove excess water from the mixtures. After the reaction has completed, the mixture was poured into seperation funnel and leave for about 24 hours. It will seperated into three layers which is MeOH,oil and fat or glycerin.

From the funnel, only the oil will be taken. The oil was filtered with filter paper to remove any leftover impurities. Then the filtered oil heated again within 110˚C to 120˚C for 30 minutes to remove any excess water during transesterification process. To remove any excess MeOH discovered in the oil, the water vapour will be removed. Following heating process, let the oil chilled until reached 70˚C then a titration process was used to determine the oil's AV and FFA content. Figure 3.3 shows the seperation of the mixtures.

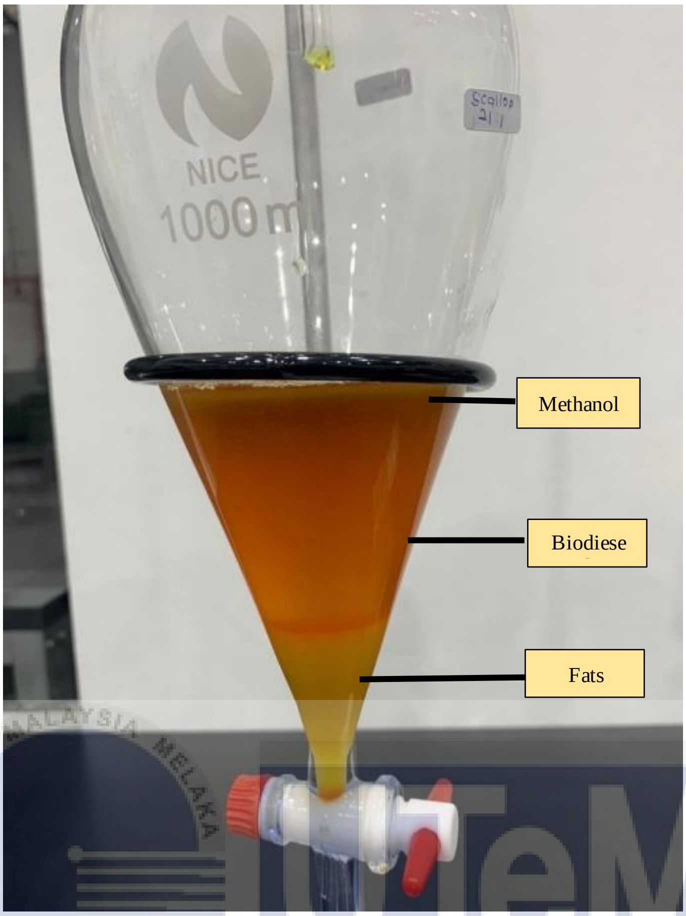

Figure 3.3 The seperation of the mixtures

3.3.2 Catalyst preparation of the waste *Ylistrum balloti*

Ylistrum balloti waste shell was collected from the seafood restaurant in Melaka. There are several seafood restaurant that have been chosen as a place to collect all the shell. After a few collection, the waste saucer scallop shell was cleaned thoroughly to remove any fats dirt or organic matter using distilled water. The cleaning process must be very detail as it may affect to the production. This process can use toothbrush or cleaning cloth to make sure there is no fats left.

After the cleaning process, the shell was air-dry to the sunlight until it completely dry. This process is important to reduce the moisture content of the catalyst. Next, the sample was crushed and sieved before proceed to the next process which is furnace. Calcination is the process of heating the shells at high temperatures to remove organic compound and convert the calcium carbonate present in the shells into calcium oxide. Then, the cleaned shell must be placed in a furnace and heat them up at temperature of 800˚C to 900˚C for about 4 hours with a heating rate of 10˚C/min.

Every calcined sample was stored in an airtight container to stop it from reacting with carbon dioxide (CO_2) and moisture in the air. After being collected, the catalyst samples for the produce were placed in an airtight glass container (desiccator) for future used. Figure 3.4 shows the process of catalyst prepareration.

Figure 3.4 Preparation of catalyst

All this produced catalyst will be characterized and a deeper understanding about characterization of the catalyst can be studied with the help of Scanning Electron Miroscopy

(SEM) to identify the element of the catalyst.. It is a method to determine the characterization of the catalyst. This method can identified the condition of the catalyst such as surface area, structure and strengthness of the structure to gain the performances.

3.3.3 Transesterification process by conventional method

This transesrification method that was conducted through this experiment is to transform a WCO to a clean biodiesel with the help of MeOH, oil and also the presence of CSS which is *Ylistrum balloti*. The goal of this transesterification method was to reduce the FFA untill archive all the standard and condition that have been set by ASTM D6751 and EN 14214. Figure 3.5 show the pre-treated oil and strring hot plate that for transesterification process.

Figure 3.5 Pre-treated oil and strring hot plate for tranesterification process.

3.4 Fuel properties testing

A testing process for biodiesel will be carried out to confirm all the important parameters used are follow the standard of by ASTM D 6751 and EN 14214 while conducting the experiment. Testing the properties of biodiesel is crucial to ensure its quality and compliance with standards

3.4.1 Free fatty acid (FFA) and Acid Value (AV)

Raw oil characterization can be characterized through the method of finding acid value and FFA. The presence of FFA can also lead to greater acid values in biodiesel. It can have negative consequences on the fuel's stability and storage life. Acidic circumstances may speed up fuel breakdown. Resulting in increased in acidity, corrosion and deposit development in fuel systems.

High amounts of FFA in the feedstock used for biodiesel synthesis can cause a number of problems. FFA can react with the alcohol used in the process to generate undesirable byproducts such as soaps during transesterification. These soaps can generate emulsions. It will block biodiesel separation from the glycerin co-product and even clog fuel filters.

The value of FFA was calculated according to the value of AV. Titration was involve to neutralize the FFA present. All the equipment and chemical reagent need to be prepare before start the experiment. A 5g of pre-treated WCO must be pour into a conical flask. A 25 ml of isopropyl propanol combine together with pre-treated WCO. About three drops of phenolphthalein must be drop into the combined solution.

The titration apparatus was set up by attach the burette to the burette stand and fill it with solution of potassium hydroxide (KOH). KOH need to be titrate slowly from burette into sample solution while swirling the flask. The KOH solution must be add until the indicator undergoes a pale pink color and record the volume of the base solution. To calculate the acid value and FFA content of the sample, Equation (3.1) and (3.2) must be use.

$$AV = \frac{56.1 \times N \times v}{m} \tag{3.1}$$

$$FFA = AV \times 0.5 \tag{3.2}$$

Where AV is the acid value obtained from the calculation. N is the normality of potassium hydroxide, V is the volume of titrate potassium hydroxide, while m is the weight of the sample oil. 56.1 is the molecular weight of potassium hydroxide.

3.4.2 Biodiesel yield

The biodiesel yield refers to the amount of biodiesel produced from a given quantity of raw materials and typically measured as a percentage. It is an important factor in assessing the efficiency and economic viability of biodiesel production processes. Biodiesel is commonly produced through transesterification. A chemical reaction involving triglycerides (fats or oils) and alcohol. The yield is influenced by factors such as the type of feedstock, the quality of the raw materials, the catalyst used, reaction conditions such as temperature, pressure and the efficiency of separation and purification steps. Biodiesel yield is calculated using the following Equation (3.3)

$$Yield\ (\%) = \frac{Weight\ of\ biodiesel\ produced}{Weight\ of\ oil\ sample} \times 100\% \tag{3.3}$$

A high biodiesel yield is desirable for economic reasons, as it maximizes the production of biodiesel from a given amount of raw materials. Researchers and industry practitioners continuously work to optimize biodiesel production processes to achieve higher yields and improve the overall sustainability of biodiesel as a renewable fuel source.

3.4.3 Density

Density displayed the weight of the sample divided by the volume and weight of water at the same temperature. One of the most crucial aspects of biodiesel was its density. It was related to the diesel engines' power and calorific value generated per volume of biodiesel. The Biodiesel with the best treatment outcome had a density of 860-900 kg/m³ according to the EN 14214. The formula for density was mass of oil divided by its volume using 50 ml of Bomex Pycnometer. This experiment was conducted in laboratory in Utem and the Bomex pyncometer was shown in Figure 3.6 below.

Figure 3.6 Bomex pyncometer

3.4.4 Flash point

The temperature at which the vapour of the biodiesel and air combination can catch fire in the presence of an open flame or spark is known as the biodiesel flashpoint. It is a crucial safety factor particularly when thinking about the handling, storing, and moving of biodiesel. The composition of the biodiesel affects the flashpoint and feedstock properties also have an impact.The value of flash point for biodiesel has minimum test limit which for ASTM D 6751 is 93˚C while for EN 14214 is 101˚C at the Oil Analysis Laboratory of Universiti Teknikal Malaysia Melaka (UTeM), the Pensky-Martens closed cup test (NORMALAB NPM 131) shown in Figure 3.7 was used to determine the flash point.

Figure 3.7 Pensky-Martens closed cup test

3.4.5 Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is a microscope variant that utilizes a focused electron beam to examine a small area of a sample at high magnification. To maintain the electron beam's focus on the sample and prevent scattering off air particles, a vacuum is applied to the area. Upon striking the sample, the electron beam induces the emission of secondary electrons from the sample, and their detection provides an image depicting the surface topography.

SEM analysis can be complemented with Energy Dispersion Spectroscopy (EDS) to identify the components within a sample. Elements in the sample emit X-rays with distinct energy signatures as they decay on the sample's surface. The EDS detector captures these X-rays and utilizes them to ascertain the elemental composition of the sample. The SEM-EDS analysis shown in Figure 3.8 was conducted at the Material Science Laboratory at the Faculty of Engineering, UTeM, with the output obtained from the JEOL (JSM-6010PLUS/LV) machine.

Figure 3.8 SEM JEOL (JSM-6010PLUS/LV) machine analysis

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

Previous studies have shown that reaction temperature, catalyst loading, molar ratio of oil to alcohol and reaction time are the main variable that affect transesterification performances. This chapter explained on catalyst calcination, characterization of raw feedstock for biodiesel production and the effect of parameters involved in this production. Next, the biodiesel chemical and physical properties are also being discussed.

The catalyst that have been collected will be placed at one place. After a few collections, the waste scallop shell must be clean thoroughly to remove any fats dirt or organic matter using distilled water. The cleaning process must be very detail as it may affect to biodiesel production. This process can use toothbrush or cleaning cloth to make sure there is no fats left.

4.2 Catalyst characterization of *Ylistrum balloti.*

In order to comprehend the characteristics of the *Ylistrum balloti*, many techniques were used. The techniques utilized to examine and comprehend the properties of the *Ylistrum balloti* are the scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS) method. For this project, the characterization of the catalyst based on SEM-EDS.

The visible effect to the raw shells having undergone calcination process in the high calcination temperature of 800°C for 4 hours was illustrated in Figure 4.1. the outer surface of the uncalcined shell was transformed from their natural colour (pink/orange/brown) to white as calcination temperature increase.

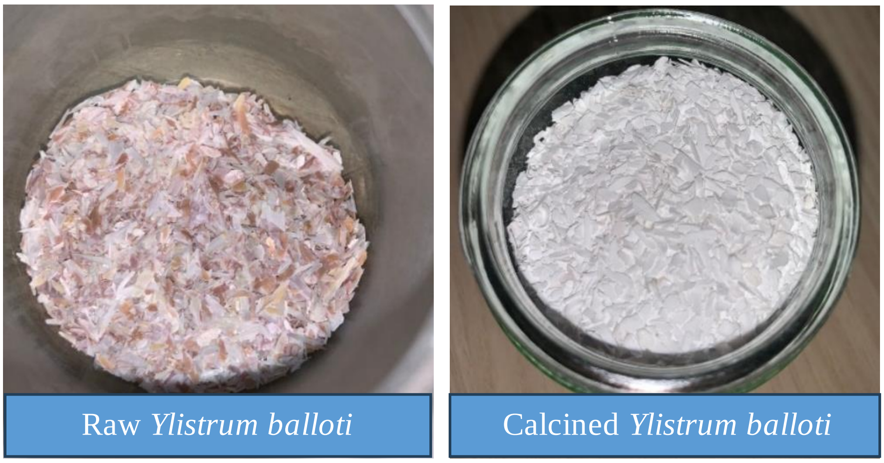

Figure 4.1 Preparation of calcium oxide catalyst derived from waste shells calcined at 800°C for 4 hours

4.2.1 Catalyst morphology

Figures 4.2 and 4.3 display the surface morphologies of both the uncalcined and calcined shell-derived catalysts, captured through a scanning electron microscope (SEM) coupled with Energy Dispersive X-ray Spectroscopy (EDS). In Figure 4.2, the uncalcined samples reveal an amorphous crystal structure. The majority of these samples exhibit uneven and irregular particles, characterized by asymmetry, rugged surfaces, and a broad size distribution. This observation is indicative of the inherent variability in the morphology of the unprocessed samples.

The utilization of SEM coupled with EDS provides a comprehensive view of the structural characteristics of both uncalcined and calcined catalysts. The subsequent Figure 4.3 likely illustrates distinctive features arising from the calcination process and further analysis may reveal insights into the transformation of the crystal structure and surface morphology during this thermal treatment.

As the calcination increased, a significant transformation occurred in the microstructures, shifting from a layered architecture to a porous structure. The resulting calcined waste shells displayed irregular shapes with some forming aggregates by bonding together. Despites this, the smaller grain size and aggregates contributed to higher specific surface areas. Given that sample were considered less porous or even non-porous. The particles size directly influenced the surface area (Li et al., 2019)

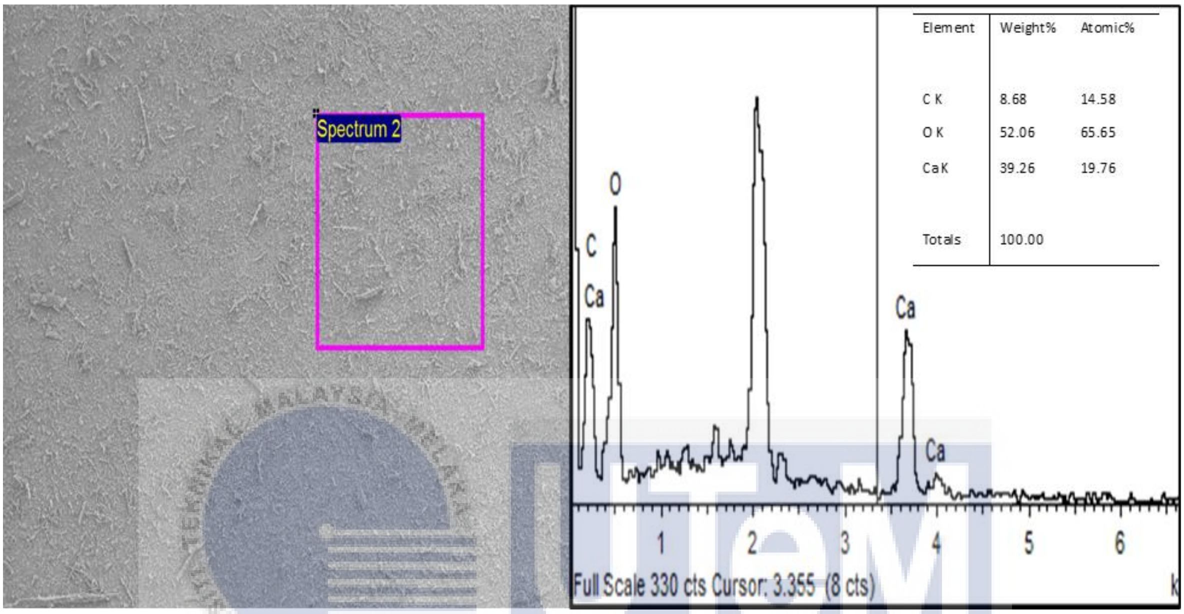

Figure 4.2 Surface morphologies uncalcined shell-derived catalysts

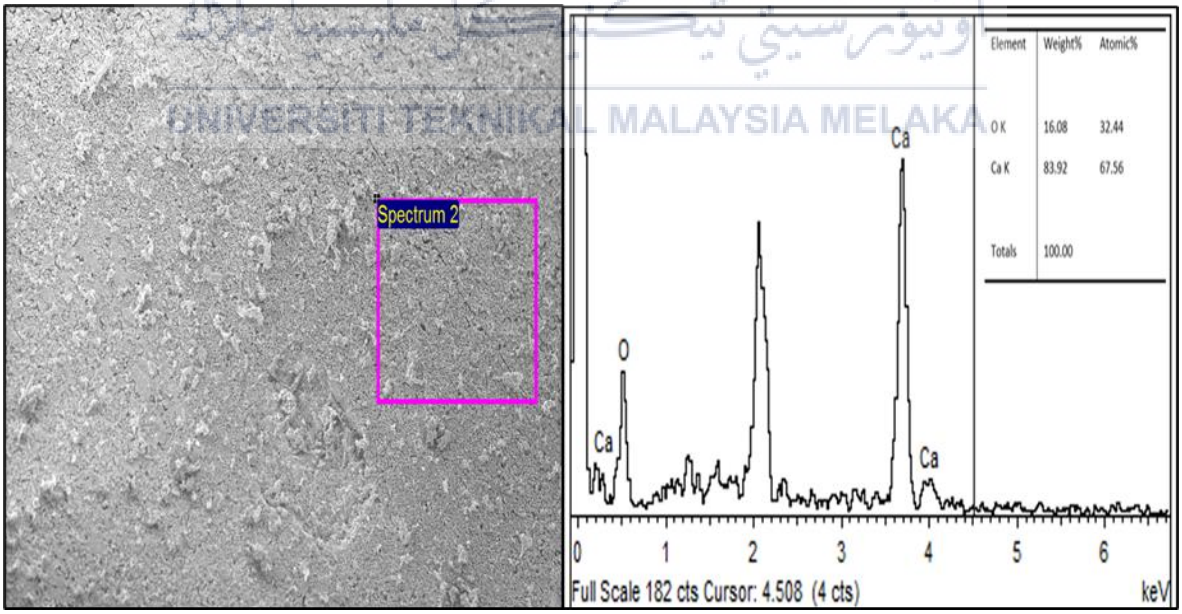

Figure 4.3 Surface morphologies of calcined shell-derived catalysts

Table 4.1 displays the weight percentage of element found in the uncalcined samples of calcium carbonate ($CaCO_3$) shells as determined from the Elemental Dispersive X-ray Spectroscopy (EDS) spectra. The data clearly indicates that the $CaCO_3$ shells are mainly consists of calcium and oxygen elements.

Table 4.1 Element weight percentage present in uncalcined samples

Element	Weight (%)	Atomics (%)
C K	8.68	14.58
O K	52.06	65.65
Ca K	39.26	19.76
Totals	100	

Table 4.2 Element weight percentage present in calcined samples

Element	Weight (%)	Atomics (%)
C K	-	-
O K	16.08	32.44
Ca K	83.92	67.56
Totals	100	

Examining Table 4.2 reveals an obvious pattern of increasing calcium element that verify the SEM data analysis. This trend strongly supports and confirms the conclusion that carbonate minerals were indeed formed during the calcination process. The calcined catalyst exhibit a composition consisting of carbon, oxygen. The consistency acrross both tables further solidies the understanding of the elemental composition and transformation of the catalyst during the calcination process.

4.3 Characteristics of Waste cooking oil

The WCO will be characterized according to the standard measure. The properties of oil that will be study is acid value, FFA, density and color. The standard measure that will be used is ASTM D664 and ASTM D6751. This standard measurement is used to determine the acidity and density of the oil. At the end of the characterization a value of the result will be obtain. Table 4.3 show the properties of cooking oil.

Table 4.3 The properties of cooking oil.

Raw WCO	Unit	Standard measure	Result
Acid value	Mg/KOH	ASTM D664	4.26
FFA	%	-	2.13
Density	Kg/m^3	ASTM D6751	894
Colour	-	-	Dark brownish-black
Iodine value	$gI_2/100$ g	ASTM D554	92.5
Saponification value	Mg KOH/g oil	ASTM D55588	195.48

4.4 Conventional transesterification method

The aim is to get an acid value as defined by ASTM D 6751 of 0.5 mg KOH/g or below. The transesterification process was carried out using a number of experimental parameters, such as catalyst concentration, MeOH to oil mole ratio, reaction time, and reaction temperature. Three different runs of transesterification were carried out, using molar ratios of 5:1, 9:1, 12:1, 15:1 and 21:1 for MeOH to oil.

4.5 Effect on variable in biodiesel production

Reaction time, reaction temperature, MeOH to oil molar ratio, and catalyst concentration are the four factors examined in this paper that are crucial to the transesterification process. Performance of the transesterification process depended on the type of catalyst used. As a result, it was tested how response variables would behave if *Ylistrum balloti* were present. The MeOH to oil molar ratio is one of the factors that has the most impact on biodiesel oil output.

4.5.1 Effect of MeOH to oil molar ratio

The most important transesterification variable in this process is alchohol to oil molar ratio. There are many kind of alcohol that can be used in order to produce biodiesel such as MeOH, proponol, pentanol, ethanol and butanol. In this project, MeOH was used as alchohol. A low or excessive amount of MeOH led to a bad production of biodiesel. The reaction will not progress to the synthesis of a product if the ratio of oil to MeOH is too low. The transesterification process will reverse towards the reactants rather than producing biodiesel. This is because transesterification is a reversible process (Suzihaque, et al.,2022).

In this experiment, five distinct MeOH-to-oil ratios (9:1, 12:1, 15:1, 17:1, and 21:1) were employed to observe their respective impacts on acid values. Table 4.4 provides a comprehensive comparison of acid values across these ratios during the reaction. The data enables a detailed assessment of how varying MeOH-to-oil ratios influence acid values. Figure 4.4 visually illustrates the relationship offering a clear representation of how changes in MeOH-to-oil ratio correspond to acid values throughout the experimental process. The reaction time, reaction temperature and catalyst concentration were fixed in this experiment. The molar ratio calculation according to different ratio were calculated in Appendix D.

Table 4.4 Transesterification process variables with different MeOH to oil molar ratio

Reaction time (min)	Reaction temperature (˚C)	Catalyst concentration (%)	MeOH to oil ratio	Acid value (mg KOH/g)
90	65	5	9:1	3.37
			12:1	8.64
			15:1	8.42
			17:1	9.42
			21:1	8.53

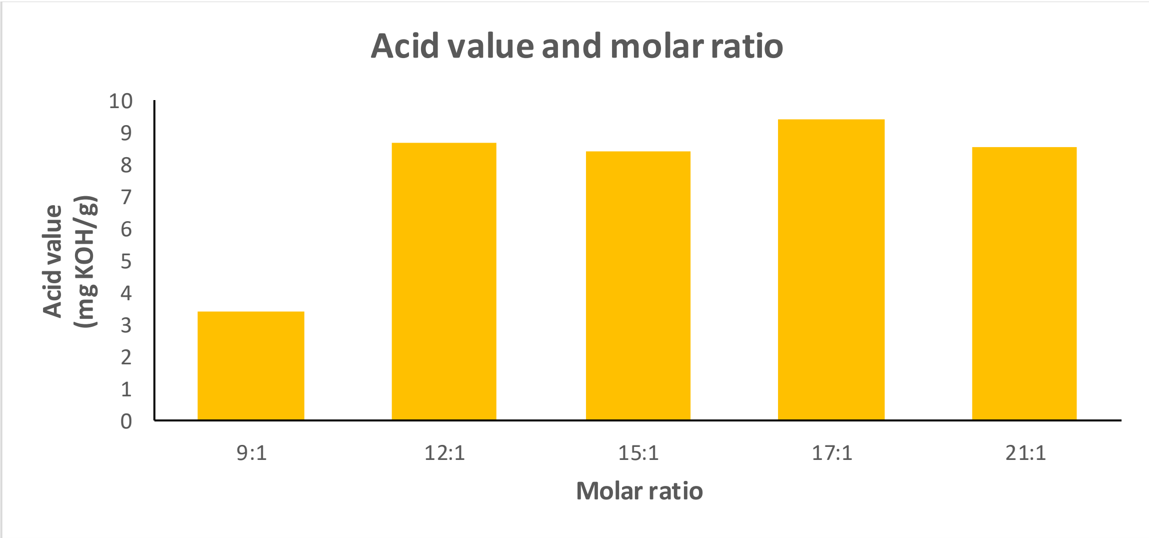

Figure 4.4 The effect of the MeOH to oil to acid values

From Figure 4.4 shown that the ratio of 12:1 to 21:1 have the highest acid value compared to the ratio of MeOH to oil ratio. As a result, using a 5% catalyst concentration with a 9:1 molar ratio and reaction temperature of 90 minutes increased the transesterification process and also has the lowest acid value. The acid value was tested and pink colour during acid value process for ratio 9:1 are the fastest to appeared to this ratio. This ratio were chosen as default for next variables which is the amount of catalyst used for transesterification process.

Nevertheless, an excessive MeOH-to-oil ratio can have adverse effects on biodiesel yield. It has been proposed that an overly abundant MeOH-to-oil ratio induces the solubility of glycerol in biodiesel and promoting the reversible conversion of biodiesel. According to Basumatary et al. (2021), the presence of an excess of MeOH can lead to a reduction in biodiesel yield and reaction rate due to the dilution of catalyst active sites.

Furthermore, surpassing the optimal MeOH-to-oil ratio may result in an increased hydrolysis of the produced biodiesel, generating soap and decreasing biodiesel yield (Betiku et al., 2019). Additionally, the separation of the biodiesel product becomes more challenging when excess MeOH is present. That is the reason why an optimal ratio between oil and alchohol need to be precised when it comes to transesterification process.

4.5.2 Effect of amount of catalyst used

In the conversion of oil to biodiesel through transesterification, the concentration of the catalyst, referred to as loading is a key factor. The catalyst's role is to offer active sites, reducing activation energy and enhancing the efficiency of the transesterification reaction (Ambat et al., 2019). With an increase in catalyst concentration during transesterification, the number of active sites rises, resulting in an accelerated reaction rate until equilibrium is achieved.

Effective management of catalyst loading plays a crucial role in biodiesel production. Striking the right balance is essential for optimal reaction kinetics during transesterification is a key stage in converting oil into biodiesel. This equilibrium significantly impacts overall yield and process efficiency. The importance of precise control over catalyst amounts to enhance the sustainability and effectiveness of biodiesel manufacturing.

The experiment involved varying catalyst concentrations to investigate their impact on the yield. The conventional method employed a reaction time of 90 minutes and 9:1 MeOH-to-oil molar ratios. The catalyst utilized in the experiment was calcined *Ylistru balloti*, and the quantities of the catalyst tested were 1%, 3%, 5%, 7% and 9%. As shown in Table 4.5, the optimal acid value (0.449 mg KOH/g) was achieved with a catalyst loading of 5% and a MeOH-to-oil molar ratio of 9:1. Acid value result with different MeOH to oil molar ratio are tabuleted in Appendix C.

Table 4.6 Transesterification process variables with different catalyst loading

MeOH to oil ratio	Reaction time (min)	Reaction temperature (°C)	Catalyst concentration (wt%)	Acid value (mg KOH/g)
9:1	65	5	1	3.104
			3	5.161
			5	0.449
			7	0.486
			9	2.353

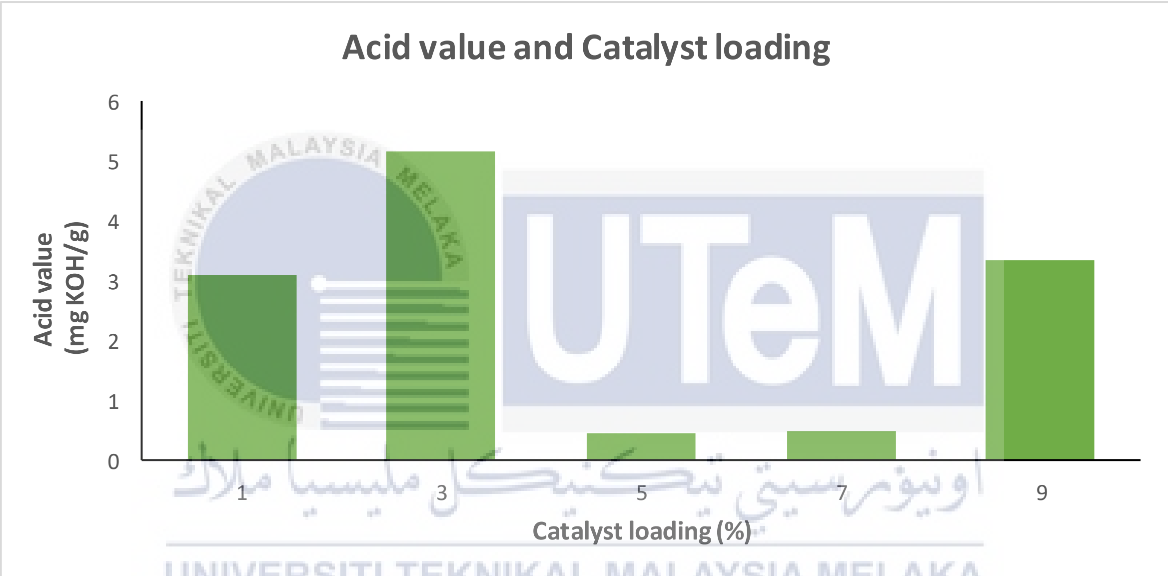

Figure 4.5 The effect of the catalyst loading and acid value

From the observation shown in Figure 4.5, 1% and 3% loading are not suitable for transesterification as it may not enough to complete the biodisel production. Insufficient catalyst amounts may result in incomplete conversion of oil to fatty acid esters, attributed to a limited number of available active sites in the reaction (Sahar et al., 2022). The catalyst concentration must be increased until reaching the optimal catalyst loading stage.

However, surpassing the equilibrium point with higher catalyst loading can lead to increased mass transfer resistance, challenging mixing of reactants and reduced product desorption in the viscous mixture. These factors impede interaction between reactants and catalyst active sites, resulting in lower biodiesel yield (Ahmad et al., 2023). This indicates that, under current reaction conditions, further increasing the catalyst loading would not lead to an improvement in biodiesel yield.

4.5.3 Effect of reaction temperature

For these experiments, the temperature and time were fixed. According to Sakthivel et al. (2020), two hours of production time and 65 °C were maintained throughout the trials in order to create high-quality biodiesel. According to the author, producing biodiesel for two hours at 65 °C will result in high-quality production since these parameters won't affect the necessary outcomes. However, in this production 90 minutes are adequate enough for *Ylistrum balloti* to show reaction with MeOH and oil to produce a lower acid value. The temperature of the production can not be too low or to high. It will lead to burning of the oil.

The production yield of biodiesel in a transesterification process is contingent upon the temperature, given the endothermic nature of the reaction. Generally, elevating the reaction temperature enhances the synthesis of biodiesel that leading to a reduction in the viscosity of the oil. This viscosity decrease is attributed to the oil's high viscosity at lower temperatures and the insufficient mixing of reactants due to the absence of an optimal reaction temperature.

However, surpassing the ideal temperature can impede biodiesel production by causing alcohol evaporation. Since the boiling point of MeOH is 64.7˚C, if reaction temperature exceeds the boiling point of the alcohol, the evaporation of alcohol occured. It will reducing the contact time between alcohol and oil and consequently lowering the overall biodiesel output. For WCO with low free fatty acid content, an optimal temperature of 65 °C was identified as ideal during the transesterification process.

4.5.4 Effect of reaction time

The experiments were conducted with fixed time. A production time of 90 minutes and were maintained throughout the experiments. These specific criteria were chosen because they are considered ideal for producing high-quality biodiesel, as suggested by Degfie et al. in 2019. The author highlighted that maintain reaction time to a 90 minutes production time ensures reliable and consistent results without compromising the desired outcome of biodiesel production.

4.6 Biodiesel yield

The yield obtained for this production was 95.05%.. There have been studies in the literature reporting that to attain a high yield production is to have a top-notch feedstock, conduct effective pretreatment, optimize the transesterification method. Appropriate catalyst preparation, mixing intensity, efficient separation method, explore catalyst recovery , stay informed about technological advancement and implement quality control measures for continual monitoring and improvement (Qadeer et al., 2021).

4.7 Fuel properties

Table 4.6 list the main properties of biodiesel production made from heterogeneous catalyst and WCO. These parameters which include density, flash point, acid value and FFA. This testing are alligned with the biodiesel standard which is ASTM D 6751. Through the entire process of transesterification, the acidity of oil was effectively reduced from 4.26 mg KOH/g to 0.449 mg KOH/g that is follow the criteria ASTM D 6751 which not exceed 0.5 mg KOH/g.

Table 4.6 Fuel properties

Property	Unit	Prepared biodiesel	ASTM D 6751	EN 14214
Acid value	Mg KOH/g	0.449	0.5 max	0.5 max
FFA	%	0.224	< 0.5	< 0.5
Flash point	˚C	220	130 min	101 min
Density	Kg/m³	900	880	860-900

4.7.1 Acid value (AV)

Milano et al. (2022) studied that the AV for biodiesel must not exceed the specifies standards to endure the product quality and performance. A higher AV indicates elevated levels of free fatty acid that can lead to increased corrosion, reduced stability and adverse effects on engine somponents. AV standards ensures the biodiesel's compatibility with engines, minimizing potential damage and maintaining optimal operation efficiency.

4.7.2 Free Fatty Acid (FFA)

Exceeding the specified standards for free fatty acids in biodiesel is desirable because high levels can affect the biodiesel's quality and performance. A studied by Maheshwari et al. (2022) examined that elevated FFA can lead to increase soap formation, corrosion and diminished stability. It can cause potential issues during storage, transportation and engine use. Adhering to the set standards is essential for ensuring the biodiesel's reliability and compatibility with engines

4.7.3 Flash point

Studies have found that exceeding the specified standards for the flash point in biodiesel is undesirable due to safety concerns. The flash point is the temperature at which a substance can ignite. Surpassing the standard could increase the risk of combustion during handling, storage, and transportation. Adhering to established flash point standards is essential to ensure the safe use and transportation of biodiesel (Algayyim et al., 2024).

4.7.4 Density

The density of biodiesel should not surpass prescribed standards to uphold its intended quality and performance were investigated by Zhang et al. (2022). Variations from these standards can affect combustion efficiency, engine compatibility and overall fuel characteristics. Complying with density standards is crucial for maintaining consistency, ensuring proper engine function, and meeting regulatory requirements in the biodiesel industry.

CHAPTER 5

CONCLUSION

5.1 Conclusion

The procedures and methods to carry out this project were clearly specified and described. The primary objective of this undertaking was to produce and analyse a high catalytic-activity catalyst. The steps and techniques needed to complete this project were outlined in detail. This project main goal was to produce and study production of biodiesel using transesterification to turn leftover *Ylistrum balloti* into biodiesel. These variables included the MeOH to oil molar ratio, reaction duration, temperature, and catalyst loading. Factors affecting the transesterification process were important for boosting the output of biodiesel.

The next goal of this project was to assess how different factors impact biodiesel production. These factors included reaction time, reaction temperature, catalyst loading, and the MeOH-to-oil molar ratio. Fine-tuning these parameters was crucial for increasing the yield of biodiesel. In order to enhance the transesterification reaction, both the catalyst concentration and the MeOH-to-oil molar ratio were increased.

The results of the transesterification process showed that optimal conditions were achieved with a 5% catalyst concentration, 9:1 MeOH to oil molar ratio, 90 minutes of reaction time and a reaction temperature of 65 °C, resulting yield biodiesel production of 95.05%. Another objective of this study involved analyzing the properties of biodiesel according to ASTM D6751 standards.

5.2 Recomendation

Several factors play a role in biodiesel manufacturing, including the reaction time, catalyst quantity, MeOH-to-oil molar ratio, and reaction temperature. Future biodiesel research could explore the energy consumption during production and the safe storage duration. In the biodiesel production discussed, *Ylisturm balloti* served as a heterogeneous solid catalyst. Another potential raw material for solid catalysts is egg shell. After undergoing a process called calcination, egg shell has the potential to act as a solid catalyst in biodiesel manufacturing.

In summary, the biodiesel production process demands significant time and energy inputs. As alternatives, one could consider using oils with lower acid numbers or increasing the quantity of oil used in biodiesel production.

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APPENDIX

Appendix A: Gantt chart PSM 1

No	Tasks	SEMESTER 1 2023/2024													
		WEEKS													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	PSM 1 Briefing														
2	Meeting with supervisor														
3	Materials research														
4	Drafting and report writing														
5	Preparation of the catalyst														
6	Correction of PSM 1 report														
7	Submission of the final report														
8	PSM 1 presentation														

Appendix A: Gantt chart PSM 2

No	Tasks	SEMESTER 2 2023/2024													
		WEEKS													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Lab preparation for upcoming experiment														
2	Industrial training briefing														
3	Meeting with supervisor														
4	Preparing catalyst														
5	Catalyst characterization														
6	Conduct experiment														
7	Fuel properties testing														
8	Preparation of thesis														
9	First submission of thesis														
10	Correction of the thesis PSM 2														
11	Submission of the thesis														
12	PSM 2 presentation														

Appendix C: Acid value result with different MeOH to oil molar ratio

MeOH to oil ratio	Weight of MeOH (g)	Volume of oil (ml)	Weight of oil (g)	Catalyst concentration (%)	Acid value
9:1	66.10	200	170.60	5	3.37
12:1	88.15	200	166.30		8.64
15:1	55.19	200	152.2		8.42
18:1	66.11	200	166.70		9.42
21:1	154.25	200	167.94		8.53

Appendix D: Molar ratio calculation

Molecular weight of Waste Cooking Oil

= 872.376(g/mol) × 1 mol

= 872.378 g

Molecular weight of MeOH

= 32.04(g/mol) × 1 mol

= 32.04 g

9:1

$= \frac{9 \times 32.03g}{1 \times 872.376}$

$= 66.10g$

12:1

$= \frac{12 \times 32.03g}{1 \times 872.376}$

$= 88.15g$

15:1

$= \frac{15 \times 32.03g}{1 \times 872.376}$

$= 110.19g$

18:1

$= \frac{18 \times 32.03g}{1 \times 872.376}$

$= 132.24g$

21:1

$= \frac{21 \times 32.03g}{1 \times 872.376}$

$= 154.25g$

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Puan

PENGKELASAN TESIS SEBAGAI TERHAD BAGI TESIS PROJEK SARJANA MUDA

Dengan segala hormatnya merujuk kepada perkara di atas.

2. Dengan ini, dimaklumkan permohonan pengkelasan tesis yang dilampirkan sebagai TERHAD untuk tempoh **LIMA** tahun dari tarikh surat ini. Butiran lanjut laporan PSM tersebut adalah seperti berikut:

Nama pelajar: NURILYANA MAISARAH BINTI DARMAWI (B092010080)
Tajuk Tesis: *YLISTRUM BALLOTI* (SAUCER SCALLOP) AS HETEROGENOUS CATALYST SUPPORT FOR CONVERSION OF WASTE COOKING OIL TO BIODIESEL

3. Hal ini adalah kerana IANYA MERUPAKAN PROJEK YANG DITAJA OLEH SYARIKAT LUAR DAN HASIL KAJIANNYA ADALAH SULIT.

Sekian, terima kasih.

"BERKHIDMAT UNTUK NEGARA"
"KOMPETENSI TERAS KEGEMILANGAN"

Saya yang menjalankan amanah,

NAMA MAHANUM BINTI MOHD ZAMBERI
Penyelia Utama/ Pensyarah Kanan
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