UNIVERSITI TEKNIKAL MALAYSIA MELAKA

CATALYST DEVELOPMENT FROM WASTE SHELLS FOR CLEAN BIODIESEL PRODUCTION

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Faculty of Mechanical Technology and Engineering

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This report is submitted to the Faculty of Mechanical Technology and Engineering in partial fulfilment of the requirements for graduation in degree of Bachelor of Mechanical Engineering Technology with Honours

Faculty of Mechanical Technology And Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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I declare that this report entitled " Catalyst Development From Waste Shells For Clean Biodiesel Production " is my own work except for each piece that I have explained the source.

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This report entitled " Catalyst Development From Waste Shells For Clean Biodiesel Production " has been submitted and reviewed as to meet the conditions and requirements of project writing for the award of the Bachelor of Mechanical Engineering Technology with Honours.

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Supervisor Name : DR. MAHANUM BINTI MOHD ZAMBERI

Date : 25/01/2024

DEDICATION

Through this project, I wholeheartedly and sincerely dedicated to my parents, Umping Anak Angah and Enja Anak Lulong, my supervisor, Dr Mahanum Binti Mohd Zamberi, and my friends, Muhammad Hazim Bin Hussin, Yeoh Kean Weng and Nurilyana Maisarah Binti Darmawi for the encouragement and guidance they gave me throughout this project. Indeed, without them, it might be difficult for me to complete this project.

ABSTRACT

Biodiesel as renewable and alternative fuel is growing at a rapid rate because of the variety of benefits that it offers, including being a fuel derived from a renewable source, being non-toxic, and environmental friendly. These qualities make it the most suitable fuel to replace petroleum diesel, which produces dangerous emissions that are harmful to both human beings and the environment. The aim of this study is to produce heterogeneous or solid catalysts from waste razor clam and saucer scallop shell that contains calcium carbonate ($CaCO_3$) which can be decomposed into calcium oxide (CaO) at high temperature through calcination process, which involved heating the material to a temperature of 800°C, 900℃ and 1000°C and used as catalyst for the production of biodiesel. The scanning electron microscope (SEM) combined with energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) methods were employed to observe their characteristic. The shells of the saucer scallop and the razor clam are the waste shells that are utilized. The synthesis of this biodiesel made use of the transesterification technique, in which waste cooking oil (WCO) was combined with alcohol and catalyst before being heated at a specific temperature and for a specified duration. Due to its lower cost compared to ethanol, methanol was selected as the preferred alcohol for this research. This project had the potential to contribute to pollution prevention by collecting waste for its purpose. Upon the completion of this research, the catalysts was successfully developed from waste razor clam shell and saucer scallop shell in which the catalyst characterization done shows that both of this shells contain calcium carbonate ($CaCO_3$) before calcined and calcium oxide (CaO) after calcined. The biodiesel produced was meet the requirements of both ASTM D6751 and EN 14214.

ABSTRAK

Biodiesel sebagai bahan api boleh diperbaharui dan alternatif telah berkembang dengan pesat kerana pelbagai faedah yang ditawarkannya, termasuk bahan api yang diperoleh daripada sumber boleh diperbaharui, tidak toksik dan mesra alam. Kualiti ini menjadikannya bahan api yang paling sesuai untuk menggantikan diesel petroleum, yang menghasilkan pelepasan gas berbahaya kepada manusia dan alam sekitar. Matlamat kajian ini adalah untuk menghasilkan pemangkin heterogen atau pepejal daripada sisa cengkerang pepahat dan kerang piring yang mengandungi kalsium karbonat ($CaCO_3$) yang boleh diuraikan menjadi kalsium oksida (CaO) pada suhu tinggi melalui proses pengkalsinan, yang melibatkan pemanasan bahan untuk suhu 800℃, 900℃ dan 1000℃ dan digunakan sebagai pemangkin untuk penghasilan biodiesel. Mikroskop elektron pengimbasan (SEM) digabungkan dengan kaedah spektroskopi sinar-X (EDS) penyebaran tenaga dan pembelauan sinar-X (XRD) telah digunakan untuk memerhatikan ciri-cirinya. Cengkerang pepahat dan kerang piring adalah kerang sisa yang digunakan. Sintesis biodiesel ini menggunakan teknik transesterifikasi, di mana sisa minyak masak (WCO) digabungkan dengan alkohol dan pemangkin sebelum dipanaskan pada suhu dan tempoh tertentu. Oleh kerana kosnya yang lebih rendah berbanding etanol, metanol telah dipilih sebagai alkohol pilihan untuk penyelidikan ini. Projek ini berpotensi menyumbang kepada pencegahan pencemaran dengan mengumpul sisa untuk tujuannya. Setelah selesai penyelidikan ini, pemangkin telah berjaya dihasilkan daripada sisa cengkerang pepahat dan kerang piring di mana pencirian pemangkin yang dilakukan menunjukkan kedua-dua cengkerang ini mengandungi kalsium karbonat ($CaCO_3$) sebelum dikalsin dan kalsium oksida (CaO) selepas dikalsin. Biodiesel yang dihasilkan memenuhi keperluan ASTM D6751 dan EN 14214.

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CHAPTER 1

INTRODUCTION

1.1 Background

In accordance with research by Jackson et al. (2018), the world's population is growing, and economies are developing quickly, which is driving up energy demand globally. This leads to a decrease in the amount of fossil fuels consumed and a rise in the number of health problems affecting people globally, report Gundupalli and Bhattacharyya (2019). For the reason it is renewable, non-toxic, and ecologically friendly, biodiesel which also known as fatty acid methyl esters (FAME) are widely acknowledged because of it feasible substitutes for fuels generated from fossil fuels (Caban et al., 2013; Verma et al., 2016). One of the processes that may happen in the presence of an appropriate catalyst is alcoholization, also known as transesterification. Catalysts are often classified as either homogeneous or heterogeneous. Both the quantity and quality of biodiesel may be impacted by the type and quantity of catalyst utilized.

Calcium oxide (CaO) is a promising catalyst, and various studies have been conducted on CaO-catalyzed transesterification (Hamid et al., 2023; Haryono et al., 2023; Sun et al., 2023). The composition of shells varies depending on the kind of shell but often includes carbon, hydrogen, oxygen, calcium, strontium, and magnesium. Seashells, which are primarily composed of $CaCO_3$ (95% to 99.9%) with a small amount of organic matrix, can be decomposed to CaO at high temperatures (700°C to 1000°C) and used as catalysts for biodiesel production, as reported by Alsaiari et al., (2023). Thus, piles of shells may be discovered in many locations on Earth, causing environmental damage via the decomposition of residual tissues attached to shells and contributing to scent and visual pollution as reported by Topić Popović et al., (2023).

The objective of this research is to develop and characterize the heterogenous catalyst from waste shells of saucer scallop (*Ylistrum Balloti*) shell and razor clam (*Ensis Arcuatus*) shell for the production of clean biodiesel by using waste cooking oil as a raw material. The overall goal of this research is to convert waste into wealth by using these waste mollusk shell as a raw material for the production of biodiesel as well as to develop catalysts for this particular purpose. Indirectly, this raised the idea that waste can be turned into wealth. Both catalysts and biodiesel were developed from waste in an effort to make this concept a reality. This has the potential to transform the way people think about waste, which was previously discarded without knowledge of its potential alternative uses that could turn it into a valuable resource if employed for other purposes.

1.2 Problem statement

According to the Food and Agriculture Organisation of the United Nations (2016), every year, a massive amount of waste shells is produced as by-products in the aquaculture and seafood sectors worldwide. Shells take a long time to degrade, resulting in land waste. It will also deplete renewable natural resources and produce major environmental and ecological issues (Yang et al., 2005). Murphy et al., (2017) state that discarded shells are thrown at sea or disposed of in landfills while Morris et al., (2019) discovered that stacking and burying waste shells changes soil's composition, structure, and physical and chemical properties, reducing its ability to retain fertilizer and water, infecting aquatic organisms, polluting soil, and water, and affecting human health. This problem has caused worldwide to confront the contamination caused by inappropriate waste shell disposal, which endangers the environment and humanity.

In addition, an investigation into the problems caused by using petrodiesel, a fossil fuel categorized as non-renewable since it does not improve the environment (Abdullah et al., 2019; Maher and Bressler, 2007). A research by Silverman et al., (2012) found that this problem has resulted in petrodiesel generating hazardous pollutants such as carbon monoxide (CO), hydrocarbons (HC), particulate matter (PM), sulphur dioxide (SO_2), and nitrogen oxides (NO_x), all of which are damaging to people. These pollutants are harmful to people. The combustion of petrodiesel may also emit carbon dioxide (CO_2), which can increase global temperatures until it causes global warming. Additionally, the combustion of petrodiesel produces relatively large quantities of greenhouse gases (GHG), which can severely influence the environment and human beings.

In light of this issue, the concept of investigating green and sustainable alternatives to conventional technology, such as conventional diesel (also known as biodiesel) has emerged. The goal of this study is to develop and characterize a catalyst from waste shells, specifically those of the saucer scallop (*Ylistrum Balloti*) and the razor clam (*Ensis Arcuatus*). This initiative aimed to develop catalysts that could be used in the transesterification process of waste cooking oil into clean biodiesel. This project reduced improper waste disposal and educated people on how to transform waste into a valuable resource. The project had the potential to contribute to pollution prevention by collecting waste for its purposes.

1.3 Research objectives

The main aim of this research is to reduce the generation of wastes such as razor clam shell, saucer scallop and waste cooking oil (WCO) by making it as a catalyst and raw material in biodiesel production. The objectives of the project are as follows:

1. To develop and characterize the heterogenous catalyst from waste shells of saucer scallop (*Ylistrum Balloti*) and the razor clam (*Ensis Arcuatus*) for biodiesel production.
2. To perform catalyst characterization using scanning electron microscopy combined with energy dispersive X-ray spectroscopy (SEM-EDS), and X-ray diffraction (XRD).

1.4 Scope of study

The scope of this research are as follows:

1. Selecting and preparing the waste mollusk shells from saucer scallop and razor clam as heterogenous catalyst in biodiesel production.
2. To perform cleaning, drying, heating, crushing, grinding, and calcination process of the raw catalyst.
3. Perform the catalyst characterization method such as scanning electron microscopy (SEM), and X-ray diffraction (XRD).

1.5 Significant of research

This research holds significant importance for several reasons. Firstly, it focuses on identifying the characteristics of waste shells, particularly from saucer scallops and razor clams, to determine if they can be used as catalysts. Secondly, the research aims to develop catalysts from saucer scallop and razor clam shells for clean biodiesel production. Biodiesel is an environmentally friendly alternative to conventional fuels, and this research seeks to contribute to its cleaner production. Additionally, the findings of this research can serve as a valuable reference for future researchers and students. By documenting the characteristics of waste shells, the development of catalysts, and their applications in biodiesel production, this research provides a useful resource for those interested in waste utilization, catalyst development, and sustainable energy.

In summary, this research's significance lies in its potential to identify the characteristics of waste shells, develop catalysts for clean biodiesel production, explore their use as valuable resources, and serve as a reference for future researchers and students. It contributes to waste management, sustainable energy production, and knowledge dissemination, with the aim of promoting more efficient and environmentally friendly practices.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of Biodiesel

Biodiesel is one of the renewable alternative sources for internal combustion diesel engines where it burns with less emissions of particulates, soot, and carbon dioxide. This is because it is extracted from animal fats and vegetable oils that can be eaten or not. The transesterification method is used to convert vegetable oil or animal fat oil into biodiesel fuel.

Biodiesel fuel contains long-chain mono-alkyl esters and it is a renewable, biodegradable, agriculturally oriented, non-toxic, environmentally friendly, and readily available fuel compared to diesel (petroleum or fossil). In general, it is produced from either conventional or non-conventional sources. Biodiesel raw materials are categorized into three groups such as first generation raw materials, second generation raw materials, and third generation raw materials.

"First generation" biodiesel fuel is produced from edible oil raw materials consisting of sunflower oil, peanut oil, coconut oil, palm oil while the term "second generation" biodiesel fuel is produced from non-edible vegetable oil raw materials consisting of mahua oil, castor oil, pongamia oil, and etc (Vijay Kumar et al., 2017). According to Sakthivel et al., (2017), "Third generation" biodiesel raw materials are created from various sources outside used cooking oil, including microalgae, fish oil, animal fat, pyrolysis oil, and others used in biodiesel production. The selection of raw materials to produce biodiesel depends on the cost of raw materials and how easily it can be found in certain country.

2.2 History of biodiesel

The first biodiesel was produced in 1853 by scientists E. Duffy and J. Patrick, who began transesterifying vegetable oil. At the same time, German scientist Rudolf Diesel developed the first diesel engine, which had a flywheel at the base and a 10 feet long cylinder iron. Rudolf Diesel originally used peanut oil as fuel in Germany to run his engine. Henry Ford created the Model T automobile between 1903 and 1926, and it was entirely built with biodiesel as its fuel. Despite the widespread use of fossil fuels, cooking oil was used as fuel in a number of nations during the 1920, 1930, and World War II. These nations include Belgium, France, China, Italy, Germany, Brazil, the United Kingdom, Portugal, Japan, and Argentina, where it has been reported that they can currently use vegetable oil as diesel fuel.

In 1937, G. Chavanne received a patent for "a procedure for the conversion of vegetable oil for its use as engine fuel" (Belgian Patent 422,877). The transesterification of vegetable oil using methanol and ethanol is described in full in this patent. This is the first proof that biodiesel is being produced. In 1977, Expedito Parente, a scientist from Brazil, created biodiesel by transesterifying ethanol. He refiled a patent for the same method, this time using short linear alcohols in place of glycerol to separate fatty acids from glycerol. This procedure falls under the category of "standard identity and quality." Sunflower oil that had been transesterified and refined to diesel fuel requirements was first used in South Africa in 1779. In 1983, the procedure for creating high-quality fuel and engine-tested biodiesel was finished and made public on a global scale.

The first biodiesel pilot plant (with a capacity of 30,000 tonnes of mustard seeds per year) was developed and licenced in 1989 by the Austrian business Gaskoks. The Austrian Biofuels Institute classified 21 nations as having developed commercial biodiesel programmes as of 1998. Right now, most common market service stations in Europe sell pure biodiesel. Numerous organisations are interested in continuous fuel storage. Regarding the topic of the energy crisis. As a result of concerns over global warming, biodiesel is now being utilised all over the world. The capacity of the world's population to generate renewable feedstocks such as vegetable oils and fats will determine whether or not the price of biodiesel will be able to compete with the price of petroleum in the future.

2.3 Global Biodiesel

Biodiesel fuel is utilized worldwide due to its environmental friendliness and rising recognition as a clean and renewable energy source, which has driven the biodiesel sector, which drives the global biodiesel market. This matter has raised traditional fossil fuel customers' environmental consciousness. Biodiesel is becoming more popular for commercial vehicles since it will eventually replace fossil fuels in automotive applications. Thus, biodiesel is expected to lead the worldwide market and combine renewable energy sources. Due to their capacity to make biodiesel for non-modified engines, several nations have reduced their dependency on foreign oil sources.

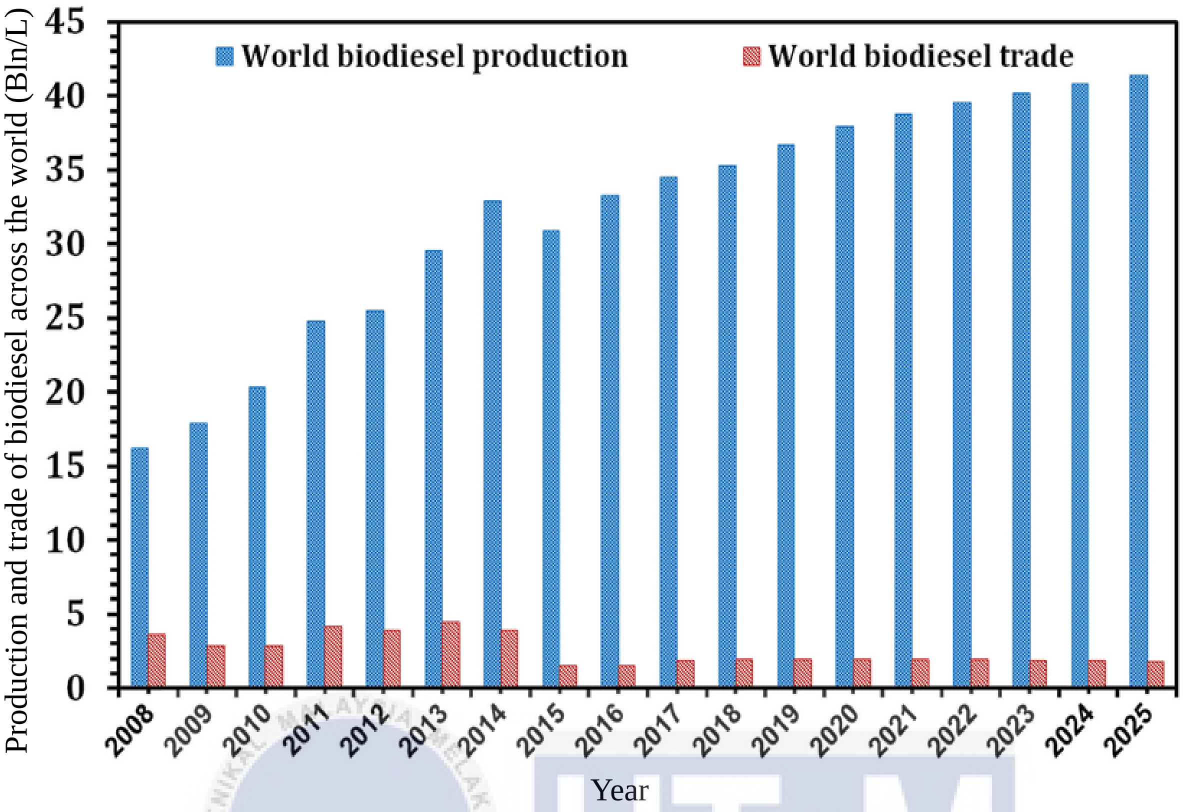


Figure 2.1: Production of biodiesel and its trade on a worldwide scale
(Outlook of Organisation for Economic Cooperation and Development (OECD) agriculture statistics, 2016)

In 2023 to 2025, world biodiesel production is expected to continue to increase while the world biodiesel trade will remain the same for the 3 year period as shown in Figure 2.1 with support from governments in many countries for the use of environmentally friendly alternative sources such as biodiesel. It is supported by the ability of biodiesel as the only fuel that can be used across many different energy segments such as electricity, transport, heat, and transport. Global biodiesel production increased from 15,000 barrels per day in 2000 to 289,000 barrels per day in 2008 to meet demand (Atabani, 2012). According to the Outlook of OECD agriculture statistics (2016), global biodiesel production will reach 37.9 billion litres in 2020. Nonetheless, it is anticipated to reach 41.4 billion litres by 2025.

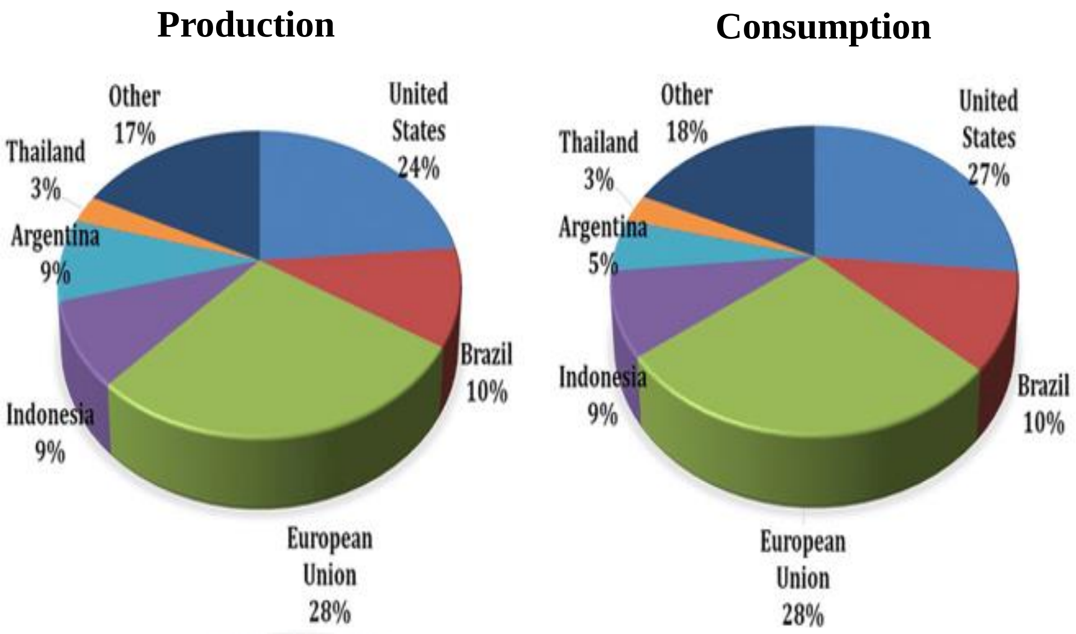


Figure 2.2: Global biodiesel production and consumption distributions by region in 2025 (Outlook of Organisation for Economic Cooperation and Development (OECD) agriculture statistics, 2016)

The European Union (EU) is made up of 27 countries which is Austria, Belgium, Bulgaria, Croatia, Republic of Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, and Sweden. Figure 2.2 shows that the EU is the country that makes and uses the most biodiesel. This is followed by the United States (U.S), other countries, Brazil, Indonesia, Argentina, and Thailand. In general, the global industries are likely to be led by governments that have made biodiesel mandatory (Table 2.1) after looking at the pros and cons of biodiesel compared to other fuels. Also, by 2025, the EU and the U.S are likely to be the countries that make and use the most biofuel.

In Table 2.1, Argentina, and Columbia used B10, a blend of 10% biodiesel and 90% petroleum diesel, while Brazil has used B8 (8% biodiesel, 92% petroleum diesel) since 2017, B9 (9% biodiesel, 91% petroleum diesel) in 2018, and B10 in 2019. Indonesia produces the most biodiesel and palm oil in the world. Indonesia has implemented B20 (20% biodiesel, 80% petroleum diesel) since early 2016 and B30 (30% biodiesel, 70% petroleum diesel) by 2020 for transportation and industries. Malaysia launched B7 (7% biodiesel, 93% petroleum diesel) in 2016 and B15 (15% biodiesel, 85% petroleum diesel) in 2021, lagging behind other nations. However, Thailand utilized B7 in 2016. Later that year, Thailand aggressively reduced palm oil use in biodiesel from B7 to B5 (5% biodiesel, 95% petroleum diesel) due to decreased palm oil output. Thailand upgraded from B5 to B10 in 2018.

Table 2.1: Current issued biodiesel mandates in some countries
(Malaysia, Economic Planning Unit (E.P.U.) Eleventh Malaysia Plan, 2016–2020. 2015)

Country Current and Future Biodiesel Mandate	Country Current and Future Biodiesel Mandate
Argentina	B10
Brazil	B8 (2017), B9 (2018) and B10 (2019)
Colombia	B10
Indonesia	B20 (2016), B30 (2020)
Malaysia	B7 (2016) and B15 (2021)
Thailand	B5 (2016) and B10 (2018)

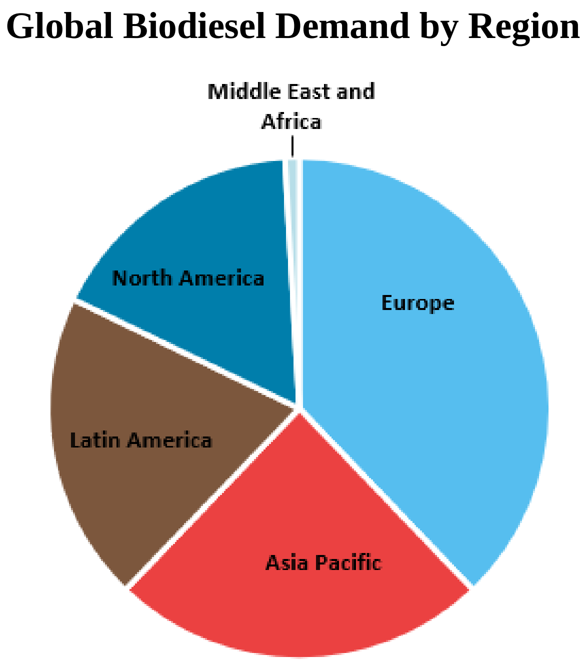


Figure 2.3: Global demand of biodiesel by region
(Source: https://mcgroup.co.uk/researches/biodiesel, 2023)

According to Figure 2.3, the demand for biodiesel is the largest in Europe since it may contribute to efforts to prevent global warming, enhance European energy independence, and boost agriculture in Europe. Asia Pacific is the second most demanding area for biodiesel, followed by Latin America and North America. The Middle East and Africa have lower demand for biodiesel. According to the monitoring of the development of the various sectors of renewable energies in the European Union (EurObserv'ER) 2020, 99.7% of biodiesel has been to comply with the European Union's Renewable Energy Directive in Europe, where the economy in that continent receives the majority of its support (Joint Research Centre (JRC), 2016).

Due to the rules for closing businesses have been relaxed and there has been a surprising increase in work and production in industries, biodiesel is in high demand in the Middle East and Africa. This matter is anticipated to stimulate further expansion of the region's biodiesel industry during the Coronavirus disease (COVID-19) outbreak. In 2020, the demand for biodiesel in the Middle East and Africa was 4.25 million tonnes, and it is expected to rise to 8.072 million tonnes by 2030, with a healthy compound annual growth rate (CAGR) of 5.97%. The global market was valued at United State dollar (USD) 90.4 billion in 2020, and it is projected to reach USD 187.6 billion by the end of 2031, expanding at a CAGR of 6.9% from 2021 to 2031. At that time, using biodiesel as a transportation fuel drives the majority of biodiesel's global market share.

Table 2.2: Leading biodiesel producers, major used feed stocks, and price per litre in 2021 (Bello et al., 2022)

Rank	Region/countries	Predominant feedstock	Biodiesel blend	The market Price in USD/L	% Production share
1.	European Union	Rapeseed oil/waste oils	-	1.39	36
2.	USA	Soybean/other oils	B100	1.4742	19
3	Brazil	Soybean	B100	0.59	12
4.	Indonesia	Palm oil	B20	1.09	10
5.	Argentina	Soybean	-	-	7
6.	Thailand	Palm oil	B20	0.84	4
7.	Malaysia	Palm oil	B10	0.49	3.34
8.	China	Waste oil	-	-	3
9.	Columbia	Palm oil	-	-	1.5
10.	Canada	Waste oils	B100	1.046	1.4

Table 2.2 shows the foremost biodiesel producing nations or regions, the most commonly used feedstocks, and the price per litre of biodiesel in 2021. In the European Union, rapeseed or waste oil is the primary feedstock, and the market price of B100 biodiesel is 1.39 USD per litre. The United States of America uses soybean or other oils as the primary feedstock, and the market price of B100 biodiesel is 1.4742 USD per litre. Both Brazil and Indonesia used soybeans and palm oil as feedstocks for biodiesel production, and the market price for B100 biodiesel is 0.59 USD per litre and B20 biodiesel costs 1.09 USD per litre. The market pricing for B10 biodiesel in Malaysia is 0.49 USD per litre, and palm oil is the primary feedstock used in biodiesel production.

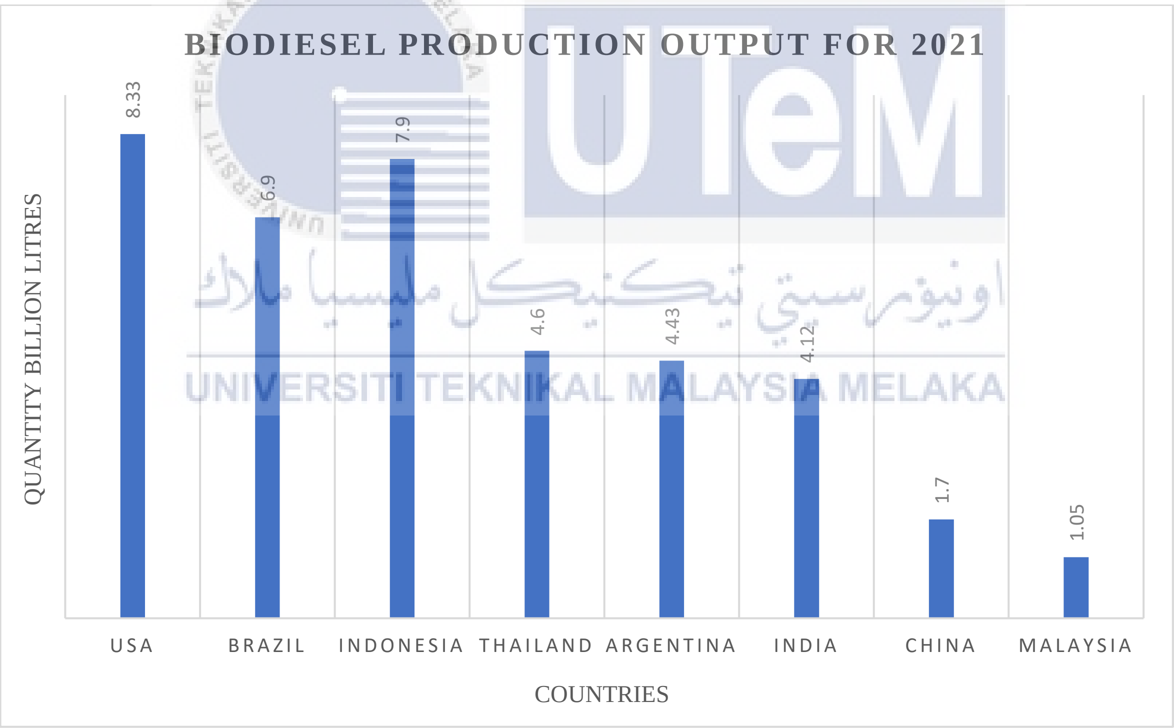


Figure 2.4: Biodiesel production output of some countries in 2021
(Bello et al., 2022)

In 2021, the United States and Indonesia were the world's largest biodiesel producers as shown in Figure 2.4. During that year, each of these countries produced biodiesel to approximately 8.33 and 7.9 billion litres, which means that the United States is estimated to reach a production level of over 1 billion gallons of biodiesel by 2025. In 2021, the biodiesel supply available for export from the United States can be more than 4.3 million barrels. By that year, the United States will have become a nation with one of the largest bioenergy capacities in the world, with a total of 13,574 MW (megawatts). The United States continues to be the industry leader in biodiesel, even in this day and age.

2.4 The advantages and disadvantages of Biodiesel

Biodiesel is known to replace fossil fuels that have the potential to replace diesel fuel as a prominent alternative fuel in the transportation industry (Fattah et al., 2013). It is comparable to diesel due to its character as pure and green energy derived from renewable energy sources such as animal and plant-based products and microalgae, as well as the presence of suitable catalysts. In addition, Fattah et al., (2013) state that biodiesel's properties can potentially reduce engine emissions of greenhouse gases, unburned hydrocarbons, particulate matter, poly-aromatics, and sulphur. According to Leong et al., (2013) and Silitonga et al., (2018), there is a need to produce new energy that is less harmful to the environment in light of the environmental concerns associated with increased use and reliance on fossil fuels. This has led to the setting of Sustainable Development Goal, SDG-7 (Affordable and Clean Energy) by The United Nations so that by 2023, everyone will have access to affordable, safe, and renewable energy with the aim of reducing the adverse effects of fossil fuel use.

Although biodiesel is known to have many advantages over other fuels, it will also have disadvantages. Biodiesel is preferred as an alternative fuel because it remains cleaner after combustion than other fuels. This matter is due to its low rate of contaminant production and its regenerative nature. According to Coronado et al., (2009), biodiesel can reduce emissions of pollutants such as carbon dioxide, hydrocarbons, sulphur dioxide, and particulate matter by an average of 40% and by as much as 78.45%. Biodiesel's drawback is that it has a higher viscosity than diesel, which can cause engine damage (Hoekman et al., 2012). This matter can lead to a decrease in atomization quality (Mohanad et al., 2017), which reduces the rate of heat emission (Atabani et al., 2012). The additional advantages and disadvantages of biodiesel listed in Table 2.3 are as follows.

Table 2.3: Advantages and disadvantages of biodiesel (Singh et al., 2020; Suresh et al., 2018; Safaque 2017)

Advantages	**Disadvantages**
• Low emissions of Greenhouse Gaseous and pollutants • Low toxicity • Non-flammable and non-explosive • High biodegradation rate • Renewable fuel • Safer than petrodiesel • Reduces the power consumption • Reduce noises • Carbon neutral and it does not create carbon dioxide • Easy to use • Biodiesel reduces the use of foreign oils	• High feedstocks cost • Requires land for growing feedstocks • Low crop yield for some feedstocks • High viscosity • Emission of Nitrogen Oxide was slightly higher than petrodiesel • The price of biodiesel is quite high, and availability is less • Requires energy to produce biodiesel • can harm rubber houses in some engines • Biodiesel fuel distribution infrastructure needs improvement

2.5 Transesterification process for biodiesel

Transesterification occurs when lipids react with alcohol to form biodiesel. Triglycerides (TGs) are the primary constituents of lipids (oil or fats). Triglycerides are three fatty acid esters and one glycerol esters. Thus, lipid alcoholysis yields monoalkyl esters, sometimes known as biodiesel, and glycerol as a by-product. The transesterification technique is also commonly used to produce biodiesel from fats (triglycerides). According to Leoneti et al., (2012) and Baskar et al., (2016), a transesterification process with TGs and alcohol produces biodiesel or mono-alkyl esters of long-chain fatty acids, such as fatty acid methyl ester (FAME).

Alcohols, such as methanol and ethanol, may be catalysed by the alkoxy group of an ester to convert TGs to FAME and glycerol in this process. Then, in alkaline, acidic, or enzymatic catalysts, a lipid interacts with a mono-alcohol to create FAME and glycerol (Cavonius et al., 2014) as shown in Figure 2.4. Hidayat et al., (2020); Habibullah et al. and Fattah et al., (2014) state that the transesterification process changes the alkoxy portion of one ester to another. This is analogous to hydrolysis. However, instead of water, alcohol is employed in this process, giving rise to the name alcoholysis (Marin-Suarez et al., 2019).

Thangaraj et al., (2019) in previous research state that transesterification is a series of reversible actions that convert triglycerides to products. In the first phase, triglycerides react with alcohol to form diglycerides, which are subsequently converted to monoglycerides and glycerol, yielding one alkyl ester at each stage. Reaction duration, temperature, pressure, kind of catalyst and concentration, type of feedstock oil, alcohol to oil molar ratio, and mixing are all parameters that influence biodiesel production in transesterification processes. Various feedstock oils having primary or secondary alcohols containing 1 to 8 carbon atoms have been widely employed for biodiesel manufacturing. Raw ingredients account for 60% to 80%

of the cost of producing biodiesel. As a result, employing low-cost waste cooking oils, animal fats, and non-edible oils may significantly lower total biodiesel costs (Nguyen et al., 2018 and Binhayeeding et al., 2018). Figure 2.5 depicts a schematic representation of the transesterification process for producing biodiesel.

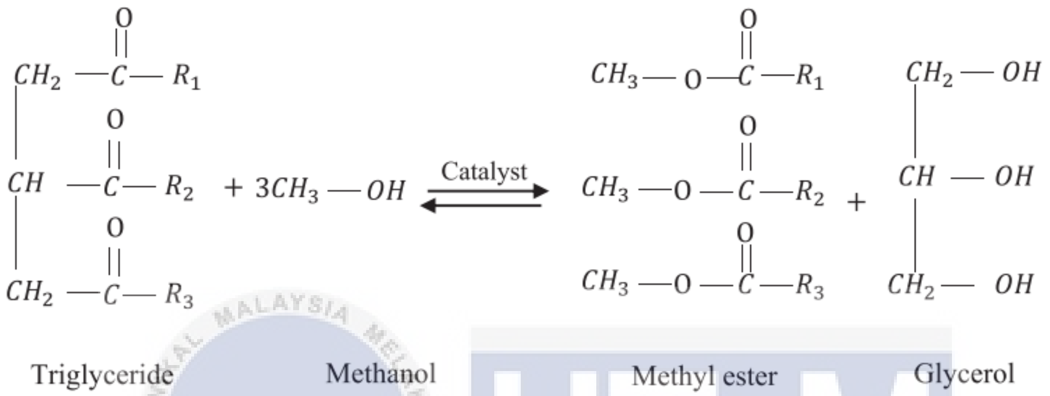


Figure 2.5: Chemical reaction of transesterification process for biodiesel production (Razzak et al., 2013)

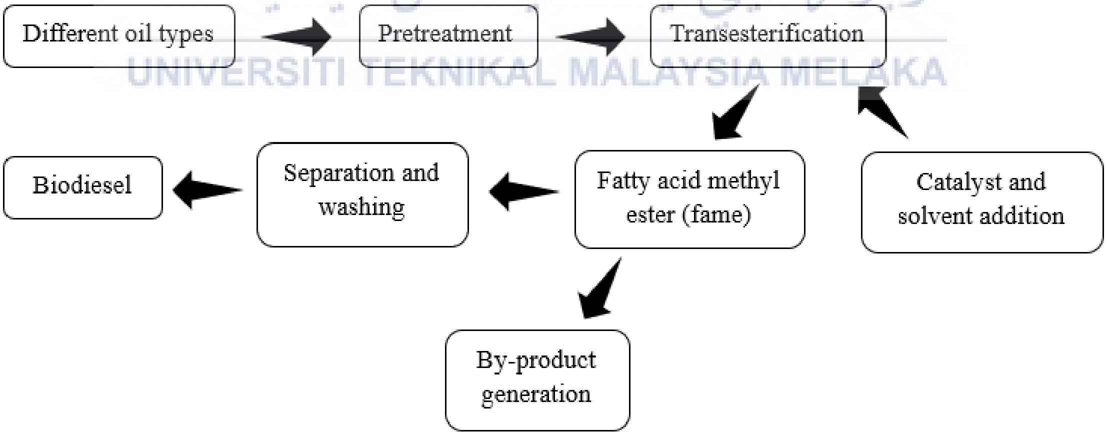


Figure 2.6 : Transesterification process of biodiesel production (Rezania et al., 2019)

2.6 Types of feedstock

The first stage in the manufacturing of biodiesel is the selection of raw materials since the purity, cost, composition, and volume of biodiesel produced are dependent on the raw materials used and the area while taking accessibility and economic aspects into account, as indicated in Table 2.4. Biodiesel is classified depending on the availability and kind of raw materials utilized, including whether it is edible, non-edible, or waste-based (Demirbas, 2009). According to Mahdavi et al., (2015), the choice of raw materials used in the manufacture of biodiesel results in varied purity and composition depending on the kind of raw materials utilized.

Table 2.4: Feedstock used for biodiesel production by countries (Changmai et al., 2020)

Country	Types of feedstock
India	Jatropha/Pongamia Pinnata (Karanja)/Soybean/Rapeseed/Sunflower
Argentina	Soybeans
Brazil	Soybeans/Palm Oil/Castor/Cotton Oil
France	Rapeseed/Sunflower
Peru	Palm Oil/Jatropha
Germany	Rapeseed
Spain	Linseed Oil/Sunflower
Italy	Rapeseed/Sunflower
Turkey	Sunflower/Rapeseed
Greece	Cottonseed
Sweden	Rapeseed
Norway	Animal Fats
China	Jatropha/Waste Cooking Oil/Rapeseed Oil
Indonesia	Palm Oil/Jatropha/Coconut
Malaysia	Palm Oil
Philippines	Coconut/Jatropha Oil
Bangladesh	Rubber Seed/Pongamia Pinnata Oil
Pakistan	Jatropha Oil
Thailand	Palm Oil/Jatropha/Coconut Oil
Iran	Pal Oil/Jatropha/Castor/Algae Oil
Singapore	Palm Oil
Ghana	Palm Oil
Zimbabwe	Jatropha Oil
Kenya	Castor Oil
Mali	Jatropha Oil
United Kingdom	Rapeseed/Waste Cooking Oil
Ireland	Frying Oil/Animal Fat
Canada	Rapeseed/Animal Fat/Soybean Oil
Mexico	Animal Fat/Waste Oil
United State Of America	Soybeans/Waste Oil/Peanut
Cuba	Jatropha Curcas/Moringa/Neem Oil
Australia	Jatropha/Pongamia/Waste Cooking Oil/Animal Tallow
New Zealand	Waste Cooking Oil/Tallow

2.6.1 Edible feedstock

In the early phases of biodiesel production, the consumable feedstock is widely utilized due to its availability as crops and the relatively simple conversion process. According to Aransiola et al., (2014) , this has led to an increase in food prices, which poses a threat to supply constraints. Producing biodiesel from edible feedstock presents obstacles such as climate adaptability, high costs, and limited growing space. Due to these limitations, users were compelled to seek additional alternatives for biodiesel production (Tariq et al., 2012). The research by Mahdavi et al., (2015) state that rapeseed oil, soybean oil, coconut oil, maize oil, palm oil, mustard oil, olive oil, and rice oil are all examples of first-generation biodiesels. The numerous sources of first-generation feedstock are listed in Table 2.5 (a) and the advantages and disadvantages of the first, second, third, and fourth generations of the feedstock are discussed in Table 2.5 (b).

Table 2.5 (a) : Various feedstock used in biodiesel production (Selvaraj et al. 2016 and Busic et al., 2018)

	First - generation	**Second - generation**	**Third - generation**	**Fourth - generation**
Sources	**Edible oil:** Soybean, coconut, sunflower, Palestine, corn, rice bran, olive, sesame seed, palm, pistachio, rapeseed, peanut oil, safflower oil	**Non-edible oil:** Neem, Jatropha, Moringa, Alga, Orange, Linseed, Mahua, Karanja/ Pongamia jojoba, Kusum, Cottonseed, Rubber seed, Deccan hemp, Sea mango, Waste cooking oil, Animal source	Oleaginous microbes microalgae bacterium, yeast, and fungi	Genetically optimized feedstocks algae to biofuels.
Benefits	Renewable energy sources, little environmental impact, simple production, and transformation into biofuel	Renewable sources, sustainability, land utilisation efficiency, and non-competition with food crops	Renewable sources, environmentally friendly, high lipid production, do not compete with land utilization and food, high growth rate tendencies	Renewable sources, capture large amounts of carbon with genomically synthesized microbes, high yield and lipid containing algae, economical running production
Limitations	Net energy losses, compete with food crops (food energy crime), raising cost of food due to food competition land scarcity	High cost, inefficiency, unsustainability, land and water use competition	Insufficient biomass production to commercialization, higher initial investment cost for production	Advanced synthetic biology, high initial investment

Table 2.5 (b): Advantages and disadvantages of different type of the feedstock (Bello et al., 2022)

	First generation (Edible)	**Second generation (Non-edible)**	**Third generation (Microalgae)**	**Fourth generation (Sludge waste)**
Advantages	• Low production costs • High biodiesel output • Accessibility and affordability • High oil content after extraction	• Mostly accessible • Superior in every way, less harmful, and increases engine performance • Free, low-sulphur and low-aromatic content • Produced biodiesel complies with ASTM standards	• Increased productivity per square foot and safer storage • Less harmful and biodegradable • Compared to other oil crops, have a far higher capability for oil production • Ability to flourish in ponds of water on non-arable soil, salty water, and wastewater that are unfavourable for oil crops • Reduce Green House Gaseous emissions to lessen pollution	• Free, straightforward conversion • Serve the dual aim of turning trash into a product with value-added • Had neutral lipid and a greater calorific value
Disadvantages	• The rivalry between food and fuel • There has been a decrease in the supply of edible oil • Over-exploration causes ecological destruction and forest loss • Only a little percentage of regular diesel was added	• Significant amounts of potentially soap-forming free fatty acids (FFA) • Problems with collections • Esters are susceptible to hydrolysis when exposed to high moisture levels • Problematic feedstock structure	• Microalgae as a feedstock for fuels is not commercially feasible due to the complexity of the harvesting and lipid extraction method • Calls for a stringent purification procedure, which adds effort, time, and money to the process • Due to the predominance of unsaturated fatty acids, there is a high risk of oxidative destruction	• Susceptible to rapid microbial breakdown\ • Greater viscosity increases the likelihood of deposits accumulating during storage • Have a foul odour, include elements of metal, and are poisonous
References	Putra et al., 2020	Munir et al., 2021	Nan et al., 2015	Marchetti,, 2007

2.6.1.1 Soybean *(Glycine Max)*

Ghazali et al., (2015) report that the fatty acids found in soybean oil are linoleic acid (50% to 60%), oleic acid (20% to 30%), palmitic acid (6% to 10%), and linolenic acid (5% to 11%). According to Singh et al., (2019), the physical parameters of soybean oil include a density of 916 kg/m^3 (at 15°C), a viscosity of 31.83 mm^2/s (at 40°C), and a heating value of 39.6 MJ/kg. Because of its nitrogen-fixing abilities, soybeans thrive in both subtropical and tropical climates. Nitrogen in the soil is replenished by growing soybeans. Soybean grows with a net positive fossil energy balance because it requires less fertiliser.

2.6.1.2 Coconut *(Cocos Nosifera)*

According to Karmakar et al., (2010) and Kumar et al., (2010), coconut trees may reach a height of 15 to 18 m and are utilised as a raw material in the production of biodiesel. Coconut oil is primarily made up of three fatty acids which is lauric (45%), palmitic (8%), and myristic (17%). It is a triglyceride with a low percentage of polyunsaturated fatty acids (6%), a high proportion of polyunsaturated fatty acids (2%), and a high amount of saturated fatty acids (86%). Except for the monounsaturated fatty acid oleic acid and the sole polyunsaturated fatty acid linoleic acid, it is composed entirely of saturated fatty acids. Quite a bit of biodiesel may be made from coconut oil. According to Singh et al., (2019), its physical properties include a density of 914 kg/m^3, a viscosity of 27 mm^2/s at 40°C, and a heating value of 37 806 MJ/kg. Research by Ahmed et al., (2014) state that using a minimum mix of 1% coconut biodiesel is thought to enhance mileage by 1 to 2 km owing to better oxidation, and lower emissions by 60%.

2.6.1.3 Palm oil *(Arecaceae)*

The selection of palm oil as a feedstock in the production of biodiesel is because of the advantages lie in its low price and high oil output per hectare in comparison to other edible oils. Palm trees may grow to be anywhere between 10 and 15 m tall. Palm oil has a density of 897 kg/m^3, a viscosity of 40.65 mm^2 /s at 40°C, and a heating value of 39.867 MJ/kg (Singh et al., 2019). It is rich in both monounsaturated and saturated fatty acids, but especially in the medium-chain kind. According to Li Yebo et al., (2016), it contains stearic acid (3% to 6%), linoleic acid (9% to 12%), palmitic acid (39% to 48%) and oleic acid (36% to 44%). It is widely known that Malaysia and Indonesia are the recent decade's two largest palm oil producers before its becoming more popular in Europe since palm oil contains a lot of saturated fatty acids.

2.6.1.4 Sunflower *(Helianthus annuus)*

Sunflower is the world's fifth most-grown oilseed crop. Its tree may reach a height of 3 meters. Sunflower seed oil production yields more per hectare than soybean and rapeseed. Sunflower oil has a density of 918 kg/m^3 (at 15°C), a viscosity of 34.01 mm^2/s (at 40°C), and a heating value of 39.56 MJ/kg (Singh et al., 2019). It has a high concentration of linoleic acid (30% to 70%), which makes it unsuitable for use as a biodiesel feedstock. It also has a high concentration of oleic acid (15% to 40%), palmitic acid (5% to 8%), and stearic acid (2% to 6%). However, according to Li Yebo et al., (2016) pure sunflower is not suited as a fuel primarily because of its high iodine number and limited oxidation stability.

2.6.2 Non-edible feedstock

Since first-generation feedstocks have limitations, scientists are interested in exploring alternatives. The primary benefits of second-generation biodiesel are that it is ecologically benign, has reduced production costs, removed food inequality, and needed less space for cultivation, as stated by Mahdavi et al., (2015); Aransiola et al., (2014); and Tariq et al., (2012). Second-generation biodiesel has several advantages over its first-generation counterparts, such as requiring less agricultural land and reducing or eliminating the need to reroute food-plant relays. Still, it also has some drawbacks, such as necessitating the use of a larger quantity of alcohol (Tariq et al., 2012). Due to the need to cultivate non-edible crops on arable land, there is a direct influence on the economics of society and food production when the non-edible feedstock is grown in marginal zones. To fight the social and economic issues connected with non-edible oil, researchers are concentrating on developing alternative solutions that are financially feasible and readily accessible to a larger audience.

2.6.2.1 Jatropha *(Jatropha curcas)*

Plants that produce oilseeds like *Jatropha* are often grown in semiarid or marginal environments. The Jatropha plant is a member of the Euphorbiaceae family, and it may grow to a height of between 5 and 7 m at its maximum (Kibazohi et al., 2011 and Openshaw et al., 2000). *Jatropha* plant needs estimated rain 100 to 150 cm each year. The oil content of Jatropha seeds ranges anywhere between 20% and 60%. According to Singh et al., (2019), the heating value of *Jatropha* oil is 38.96 MJ/kg, while its density is 916 kg/m^3 (at 15°C), and its viscosity is 37.28 mm^2/s (at 40°C). Reksowardojo et al., (2013) state that some of the components of *Jatropha* are saturated, such as palmitic acid (13.6% to 15.1%) and stearic acid (7.1% to 7.4%), but the majority of the components of *Jatropha* are unsaturated, such as oleic acid (34.3% to 44.7%) and linoleic acid (31.4% to 43.2%).

2.6.2.2 Rubber seed *(Hevea brasiliensis)*

Rubber seed is a member of the *Euphorbiaceae* family of plants which its tree may grow to be very big, reaching heights of up to 34 metres (Ahmad et al., 2014). It is vital for the development of the rubber plant to take place in an area that does not experience frost. According to Al-Widyan et al., (2010) and Gill et al., (2011), the brown oil content of copra, also known as the kernel of rubber seed, ranges from 40% to 50% by weight, while the oil content of the fruit's seeds ranges from 50% to 60%. In previous research by Singh et al. 2019, rubber seed oil has a density of 917 kg/m^3 (when measured at 15℃), a viscosity of 42.54 mm^2/s (when measured at 40℃), and a heating value of 38.64 MJ/kg. Ghazali et al. (2015) state that there is a significant concentration of unsaturated fatty acids in rubber seed oil, including linolenic acid (16.3%), linoleic acid (39.6%), and oleic acid (24.6%).

2.6.2.3 Jojoba *(Simmondsia chinensis)*

Jojoba can only be found in the *Simmondsiaceae* plant. The liquid wax ester and the oil extracted from the seeds of the jojoba plant are the two most valuable items that may be derived from this plant. The plant may grow to a maximum height of up to 1 to 2 metres. The leaves of the jojoba plant have an oval form, and their width ranges from 1.5 to 3 cm while their length ranges from 2 to 4 cm and their colour is a greyish green as state by Selim et al., (2003) and Huzayyin et al., (2004). Jojoba oil has a density of 868 kg/m^3 (when the temperature is 15℃), a viscosity of 24.89 mm^2/s (when the temperature is 40℃ and a heating value of 46.47 MJ/kg (Singh et al., 2019) and the oil content of jojoba seeds ranges from around 40% to 50% (Al-Widyan et al., 2010; Shehata et al., 2011 and Kumar et al., 2011). The linoleic acid found in jojoba seeds ranges from approximately 25.2% to 34.4%, while the oleic acid found in jojoba seeds ranges from approximately 43.5% to 66%.

2.6.2.4 Karanja

Karanja is classified as a member of the *Leguminosae* family. The karanja plant may grow to a maximum height of up to 15 to 25 metres. After three to four years after planting, the flowers begin to bloom, and the fruit is ready for harvest between four and seven years later. A single Karanja plant may produce anything from 9 to 90 kg worth of seeds. Demirbas (1998); Moser (2009); Karmee and Chadha (2005) state that there is a significant degree of variation in the proportion of oil found in karanja seed (25% to 40%). According to Singh et al., (2019), the heating value of karanja oil is 35.992 MJ/kg, while its density is 933 kg/m^3 (at 15°C), and its viscosity is 39.9 mm^2/s (at 40°C). Karanja oil contains stearic acid that ranges from 2.4% to 8.9%, linoleic acid that ranges from 10.8% to 18.3%, and oleic acid that ranges from 44.5% to 71.3% (Srivastava et al., 2008; Agarwal et al., and Bajpai et al., 2009).

In general, the raw materials used in the manufacture of biodiesel may be broken down into four generations: the first generation, which includes edible sources; the second generation, which includes non-edible sources; the third generation, which includes micro-algae; and the fourth generation, which includes sludge waste.

2.7 Types of catalyst

The function of a catalyst is to increase the conversion rate in a specific reaction. The presence of a catalyst accelerates the reaction, increasing the yield of the product. The quantity and kind of catalyst employed affect the reaction rate and conversion efficiency. Various catalysts are used in the biodiesel transesterification process. These are grouped into four primary categories which is homogeneous catalysts, heterogeneous catalysts, biocatalysts, and nanocatalysts (Shan et al., 2018; Akubude et al., 2019), which may be further subdivided into distinct subcategories as shown in Figure 2.6.

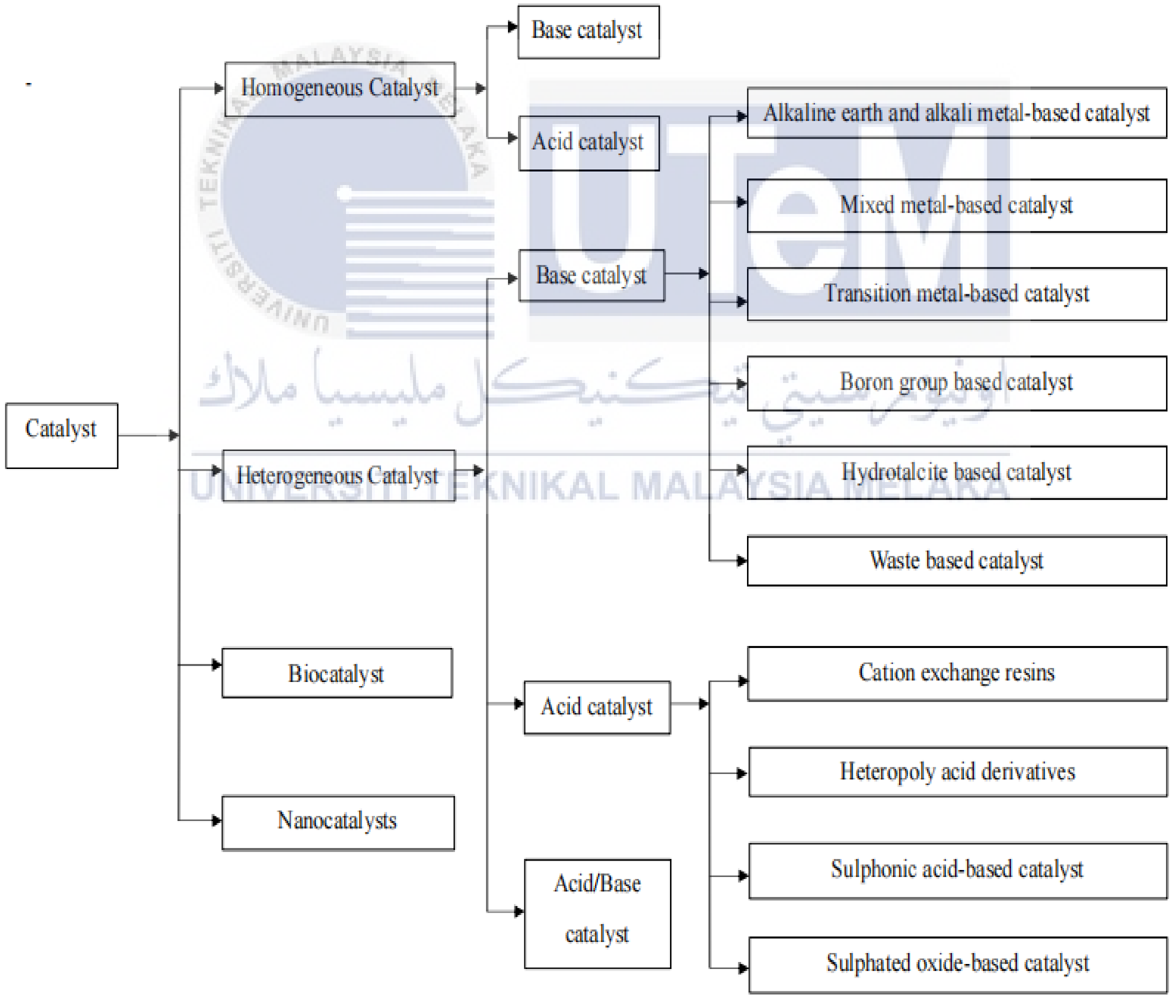


Figure 2.7: Catalyst used in biodiesel production (Fattah et al., 2020)

Generally, homogeneous, and heterogeneous catalysis often used to catalyse transesterification or alcoholysis. Homogeneously catalysed reactions are often quicker and need less loading than heterogeneously catalysed ones. On the other hand, Heterogeneous catalysts are in a distinct phase from the reaction system, allowing for catalyst removal at various phases. These may be reused without requiring extensive cleaning. Furthermore, since there are fewer dissolved ions than in homogeneous catalysis, high-purity glycerine may be produced, enabling further usage in industrial processes. According to Orphardt (2003), the primary distinction between a homogeneous and heterogeneous catalyst is that a heterogeneous catalyst may be mechanically separated from other reactants or the end product by filtering or centrifugation, while a homogeneous catalyst cannot. Table 2.7 shows the advantages and disadvantages of common type of transesterification catalyst in biodiesel production.

2.7.1. Homogenous catalyst

Homogeneous catalysts work well in liquid environments, often dissolved in a solvent along with all reactants. In a recent study by Ganesan et al. (2021), these catalysts efficiently converted vegetable oils with low water and free fatty acid (FFA) content (below 2%), showing high reactivity, cost-effectiveness, and good yields. It was preferred for biodiesel synthesis due to its ease of use and quick reaction times. However, challenges include dealing with the produced by-products, separating the catalyst, and managing excess water where it can be harsh on equipment, forming soap when reacting with FFAs, requiring additional steps like water washing and purification, which increase production costs (Mansir et al., 2021). The homogenous catalyst are categorized into acid and base types, as listed in Table 2.8, showcasing their use in biodiesel production from various raw materials.

Table 2.6: Advantages and disadvantages of common type of transesterification catalyst in biodiesel production (Jayakumar et al., 2021)

Catalyst	Type	Advantages	Disadvantages	References
Homogenous	Acid	– Tolerance to water and FFA – Simultaneous transesterification and esterification	– Reduced catalytic activity – Catalyst neutralization needed – Extra step for catalyst removal	Ghadge and Raheman, 2006; Su, 2013)
	Base	– High conversion rate – Low-cost catalyst – Widespread availability	– Soap formation with FFA and water – Extra step for catalyst removal – Wastewater production	Meher et al., 2006; Huang et al., 2010
Heterogeneous	Acid	– Tolerant to water and FFA – Simultaneous transesterification and esterification – Catalyst recovery – Catalyst recycling and regeneration	– Catalyst leaching loss – High temperature requirement – High alcohol/oil ratio – Extended reaction time	Kulkarni and Dalai, 2006; Lam et al., 2010; Behera et al., 2020
	Base	– Faster reaction rate – Easy catalyst recovery – Catalyst recycling and regeneration – High biodiesel yield	– Catalyst leaching loss – Susceptible to soap and FFA in oil	(Lam et al., 2010; Naeem et al., 2021; Nath et al., 2019, 2020)
	Ion exchange Resin	– By-product removal – High fatty acid conversion – Easy product separation – Dual catalysis (acidic and basic)	– Resin deactivation by FFA adsorption – Sluggish reaction rate	(Deboni et al., 2018; Shibasaki-Kitakawa et al., 2013; Siddique et al., 2021)
Biocatalyst	Enzyme	– Biodiesel from FFA – Water insensitivity in fat/oil – High biodiesel yield – Moderate reaction temperature – Environmentally friendly process – Potential for recovery, reuse, and recycle	– High biodiesel yield – Moderate reaction temperature – Environmentally friendly process – Potential for recovery, reuse, and recycle – Variable reaction rates – Catalyst cost impact – Enzymatic activity inhibition	(Nomanbhay and Ong, 2017)
	Free	– Efficient catalytic activity – High selectivity – Low energy consumption – Simultaneous esterification and transesterification – Superior activity to conventional acid catalysts	– Methanol- induced lipase inactivation – Lipase stability – Recovery and reusability	(Saranya and Ramachandra, 2020; Thangaraj et al., 2019; Zhou et al., 2021)
	Immobilized	– Enhanced lipase stability and reusability – Operational flexibility – Heightened thermal and chemical stability – Downstream operation elimination – Dependence on immobilization support	– High mass transfer resistance – Lower reaction rate – Moderate surface-to-volume ratio – Separation via centrifugation and filtration – Costly fabrication	(Tacias-Pascacio et al., 2017; Thangaraj et al., 2019; Xie and Huang, 2020)

Table 2.7: Homogenous catalyst of biodiesel production from various feedstock (Fattah et al., 2020)

Feedstock	Catalyst	Reaction parameter	References
		Oil: alcohol ratio/Catalyst amount/Temperature/Reaction time	
Madhuca longifolia oil	H_2SO_4, KOH	Ester.: 1: 0.35 v/v/ 0.01 v/v/ 60°C/ 30 min Trans.: 1:4 v/v/ 7 g/L/ 60°C/ 30 min	Saravanan et al., 2020
Elaeagnus angustifolia L seed oil	Potassium methoxide	9: 1 M/ 1 wt.%/ 60°C/ 1 h	Kamran et al., 2020
Rice bran oil	KOH	10:1 M/ 0.005 v/v/ 50°C/ 2 h	Goga et al., 2019
Sunflower oil	KOH	6:1 M/ 1 wt.%/ 60°C/ 3 h	Dueso et al., 2018
Castor oil	KOH	5.4:1/ 0.73 wt.%/ 64°C/ 2.5 h	Aboelazayem et al., 2018
Millettia pinnata oil	H_2SO_4, CaO	Ester.: 12:1 M/ 1% v/v / 60°C/ 3 h Trans.: 6:1 M/ 1 wt.%/ 60°C/ 2 h	Ruhul et al., 2017
Jatropha curcas oil	H_2SO_4, NaOH	Ester.: 5:1 v/v/ 0.008 v/v/ 50°C/ 2 h Trans.: 5:1 v/v/ 8 g/L/ 50°C/ 2 h	Dubey and Gupta, 2017
Rice bran oil	NaOH	6:1 M/ 2 wt.%/ 60◦C / 2 h	Wakil et al., 2016
Calophyllum inophyllum oil	H_2SO_4, sodium methoxide	Ester.: 30:1 M/ 10 wt.% / 75°C/ 2 h Trans.: 7.5:1 M/ 1 wt.%/ 55°C/ 1.5 h	Jahirul et al., 2015
Aphanamixis polystachya oil	HCl, KOH	Ester.: 24:1 M/ 1% v/v/ 60°C/ 3 h Trans.: 6:1 M/ 1 wt.%/ 60°C/ 2 h	Palash et al., 2015
Palm oil	KOH	25% v/v/ 1 wt.%/ 60°C/ 2 h	Fattah et al., 2014
Calophyllum inophyllum oil	H_2SO_4, KOH	Ester.: 12:1 M/ 1.5% v/v/ 60°C/ 3 h Trans.: 6:1 M/ 1 wt.%/ 60°C/ 2 h	Fattah et al., 2014
Ceiba pentandra oil	H_2SO_4, NaOH	Ester.: 8:1 M/ 1% v/v/ 60°C/ 2 h Trans.: 8:1 M/ 1 wt.%/ 50°C/ 1 h	Silitonga et al., 2013

2.7.1.1 Base or alkaline homogenous catalyst

Homogeneous base catalysts have received significant attention in transesterifying vegetable oil to FAME. This matter is due to the high reaction rate of the process catalysed by this catalyst and the fact that it is inexpensive and conveniently available. By the base, it is roughly 4000 times quicker than transesterification by acid (Williams, 2015). If the FFA and water content are above 0.5% and 0.06 wt% (percentage by weight) respectively, this process has limited feedstock flexibility owing to undesirable side reactions such as soap manufacturing. According to Aderibigbe et al., (2021), homogenous alkali-triggered transesterification provides many benefits, including a quick reaction time, lower alcohol consumption, and less corrosive catalysts.

In addition, when extra-pure virgin oils with FFA content and acid values less than 0.5% and 1 mg KOH/g are utilized, homogeneous alkali catalysts improve yield and purity. Otherwise, soap formation occurs due to the high FFA, reducing output and making product separation difficult (Lam et al., 2010). Another significant disadvantage of this approach is that the catalyst used cannot be recovered and hence cannot be reused. Consequently, it must be neutralized as an aqueous salt waste stream before being disposed of. Furthermore, Selvakumar and Sivashanmugam (2017) state that a large amount of wastewater is generated during the purification of biodiesel, which presents environmental issues.

2.7.1.2 Acidic homogeneous catalyst

Acidic catalyst is neutral to FFA and improves transesterification or esterification of vegetable oils or fats with high FFA (≥2 wt%). Ganesan et al. (2021) state that homogenous acid catalysts respond slower than base catalysis but are unaffected by free fatty acids and water. Alcohol is needed in large quantities for the acid-catalysed transesterification process, which yields FAME at low temperatures and pressure (Wang et al., 2021). According to Kulkarni and Dalai (2006), acid catalysts esterify waste cooking oil (WCO) and animal fats before transesterification with a base catalyst to reduce FFA levels. Homogeneous alkali catalysts are limited by soap generation and the usage of high-quality extra-pure food oils. Homogeneous acid catalysts can help to overcome these concerns. Table 2.10 shows the results of some studied on acidic homogeneous catalyst.

Table 2.8: Results of some studied on acidic homogeneous catalyst (Changmai et al., 2020)

Catalyst	**Feedstock**	**Methanol to oil molar ratio**	**Catalyst loading (wt%)**	**Temperature (℃)**	**Reaction time (min)**	**Yield (%)**	**References**
H_2SO_4	Chicken/mutton tallow	30 : 1	1.25/2.5	50/60	1440	99.01 ± 0.71/93.21 ± 5.07 25	Bhatti et al. 2008
H_2SO_4	WCO	20 : 1	4	95	600	90	Wang et al. 2006
H_2SO_4	Used frying oil	3.6 : 1	0.1	65	40	79.3	Nye et al. 1983
H_2SO_4	Soybean oil	6 : 1	3	60	2880	98	Canakci and Gerpen (1999)
H_2SO_4	*Zanthoxylum bungeanum*	24 : 1	2	60	80	98	Zhang and Jiang (2008)
H_2SO_4	Tobacco seed oil	18 : 1	1	60	25	91	Veljkovic et al. 2006
$C_2HF_3O_2$	Soybean oil	20 : 1	2	120	300	98.4	Miao et al. 2009

2.7.2 Heterogeneous catalyst

A heterogeneous catalyst, also known as a solid catalyst, minimizes chemical and time and acts at distinct stages in the liquid reaction mixture. Heterogeneous solid catalysts provide continuous biodiesel generation in fixed bed reactors, enabling industrial output (Malani et al., 2018). According to Onukwuli et al., (2020), heterogeneous catalysts can overcome the obstacles of the homogeneous catalysed process, such as energy-inefficient separation of the catalyst, removal of by-product, corrosivity, and its reaction with FFA to form soap. Research by Nadeem et al., (2021) state that most heterogeneous catalysts for transesterification are made from waste materials and may be reused, lowering manufacturing cost.

Heterogeneous catalysts are reusable and cost-effective (Refaat 2010) which can help to reduce biodiesel production costs (Endalew et al., 2012). These catalysts endure high FFA and moisture. Dehkordi and Ghasemi (2012) found that a reusable heterogeneous catalyst is non-corrosive, unlike homogeneous catalysts, it resists water and FFA (Uprety et al., 2016). The solid–liquid two-phase reaction limits heterogeneous catalyst mass transfer. Thitsartarn et al., (2011) state that heterogeneous catalysts have higher reaction parameters for biodiesel production. Heterogeneous catalysts yielded less biodiesel due to fewer active catalytic sites (Mansir et al., 2018). Heterogeneous or solid catalysts divided into basic or alkaline and acidic catalyst.

2.7.2.1 Base or alkaline heterogeneous catalysts

Most of the time, alkaline earth metal oxides, mixed metal oxides, and supported alkali metals are the heterogeneous base catalysts used to make FAME that are studied (Veillette et al., 2017). Some benefits of heterogeneous base catalysts include a relatively fast reaction rate, mild conditions for the reaction, less energy use, the ability to easily remove the catalyst from the reaction mixture, a high chance of refilling and renewing the catalyst, and less corrosion. Some major problems include pollution of the catalyst when it is exposed to outdoor air and its sensitiveness to FFA content in the oil because of its basic property.

Rocha et al., (2019) reported that soap is made when the FFA content is more than 2%, and contamination can happen if the active sites of the catalyst are leached away. Solid alkali catalysts are more likely to speed up a chemical reaction than solid acid catalysts. To solve the problems that came up when making biodiesel with homogeneous alkali catalysts, a lot of study was done. Talha and Sulaiman (2016) state that heterogeneous alkali catalysts are mostly alkaline oxides and alkaline earth metals. Oxides, hydrotalcite, metallic salts, anion exchange resins, and zeolites are supported over a large surface area of the catalysts. Alkaline earth metal oxides are widely used as solid alkali catalysts because they are inexpensive and have high basic strengths.

In addition, the alkaline earth metal oxides, such as beryllium oxide (BeO), magnesium oxide (MgO), calcium oxide (CaO), strontium oxide (SrO), barium oxide (BaO), and radium oxide (RaO), are used successfully as catalysts. Uzoh et al., (2014) say that CaO is a great heterogeneous catalyst because it is strong, basic, and completely soluble in alcohol. CaO and BaO are usually stronger than MgO. However, BaO is poisonous and dissolves quickly in ethanol or methanol according to Du et al., (2019). CaO is thought to be a good heterogeneous catalyst because it is selective, works better, is easy to get, does not dissolve as easily in methanol, and needs mild reaction conditions. It is easy to get and inexpensive. The CaO heterogenous catalyst can be produced using calcination method where the raw catalyst such as razor claim and saucer scallop shell that contain $CaCO_3$ is calcined at the temperature of 700℃ to 1000℃ to decompose the $CaCO_3$ into CaO.

2.7.2.2 Acidic heterogeneous catalysts

Acids catalyse esterification and transesterification without soap production (Serio et al., 2008). Thus, unlike base catalysts, acid catalysts may produce biodiesel from low-quality oil with high FFA and water concentration. Alkaline catalysts generate methoxide anion from methanol better in transesterification. Heterogeneous acid catalysts may esterify and transesterified biodiesel. Heterogeneous acid catalysts avoid vessel corrosion and toxicity. According to Mansir et al., (2017), these catalysts can produce biodiesel from low-quality, inexpensive feedstocks without acid pre-treatment. Nata et al., (2017) state that heterogeneous catalysts make continuous biodiesel synthesis using inexpensive animal fat, WCOs, and other waste-based oils more economically feasible.

Metal oxides, mixed oxides, sulphated metal oxides, sulfonated carbon materials, cation exchange resins, heteropoly acids, and zeolites are used in biodiesel manufacturing Carvalho et al., (2017). Heterogeneous acid catalysts with large pores, strong acid sites, and hydrophobic properties are excellent as reported by (Lam et al., (2010). Heterogeneous acid catalysts react slower than heterogeneous alkali catalysts but provide promising results under mild circumstances. These catalysts need greater temperatures, catalyst loading, and methanol-to-oil ratios than heterogeneous alkali catalysts.

2.8 Catalyst characterization

The study and understanding of the characteristics, structure, and composition of catalyst materials is referred to as the process of catalyst characterization. These catalyst materials are utilised in chemical processes. This characterisation is vital for acquiring insights into the behaviour, performance, and processes involved in catalytic reactions in order to acquire a better understanding of the catalyst. In this research, the method used to characterize the calcined catalyst is scanning element microscope (SEM), X-ray diffraction (XRD) and X-ray fluorescence (XRF).

2.8.1 Scanning element microscope (SEM)

The SEM magnifies specimen size, shape, composition, crystallography, and other physical and chemical characteristics where it can show electron beam reflections from material contact. SEM can examine catalyst surface appearance, particle size, and structure and is a flexible method for studying catalyst microstructure morphology. The study of morphology, chemical content, surface, and internal microstructure of catalysts helps produce materials with higher selectivity, conversion rate, and lifespan. and lifetime. A tightly focused electron beam raftered over the sample's surface produces a broad variety of signals that disclose the sample's shape and chemical makeup.

SEM microscopes have a greater depth of field, which enables a considerable portion of the material to be in focus at once, giving them a three-dimensional look essential for understanding catalyst surface structures. SEM is widely used to characterise nanostructures' dimensions, size, forms, particle aggregation, aspect ratio, and porosity. According to Garlisi and Palmisano (2017), Levchuk et al., and Cojocariu (2016), SEM is used to analyse catalytic thin film porosity, thickness, homogeneity, and composition.

2.8.2 X-ray diffraction (XRD)

X-ray diffraction, often known as XRD, is a technique that may be used to evaluate the crystallinity of the catalyst and perform qualitative detection of the components that are present. One of the most often used methods is called X-ray diffraction (XRD), which stands for "x-ray diffraction." X-rays having wavelengths in the range of, have enough energy to pass through materials, and are well suited to investigate the composition and organisation of the solid's interior. XRD may be used to determine the bulk phases present as well as estimate particle sizes.

A target that is subjected to the impact of high-energy electrons is the component that makes up an X-ray source. The scattering of X-ray photons in an elastic manner by atoms arranged in a periodic lattice constitutes X-ray diffraction. The determination of the size and shape of supported particles may be accomplished using electron microscopy in a relatively simple manner. It is also able to provide information about the particles' make-up, for instance, by analysing the manner in which electrons are diffracted or by detecting the X-rays that are created when electrons contact with matter. Both of these methods are examples of how this information may be obtained.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This research project delved into the innovative use of waste shells as a basis for developing a catalyst dedicated to biodiesel production. Specifically, the saucer scallop shell (*Ylistrum Balloti*) and razor clam shell (*Ensis Arcuatus*) were chosen among various waste shell types to serve as the fundamental raw materials for catalyst production and these waste shells was obtained from the food stall (Pasri Seafood Restaurant) in Melaka. The selection criteria were rooted in the recognition that these waste shells inherently contained calcium oxide, a key element with catalytic potential. The catalyst development process encompassed a series of methodical procedures to extract and enhance the catalytic properties of the calcium oxide present in the waste shells. These procedures involved various stages, including but not limited to calcination at different temperature ranges.

The meticulous application of these processes ensured the success of the catalyst synthesis. Subsequently, the developed catalyst was employed in the biodiesel production process, and the resulting biodiesel met the critical parameters stipulated by industry standards such as ASTM D6751 and EN 14214. This not only underscores the capacity to repurpose waste materials for valuable applications but also signifies a significant stride toward sustainable and standardized biodiesel production, contributing to broader efforts in environmentally responsible energy practices.

3.2 Catalyst development flowchart

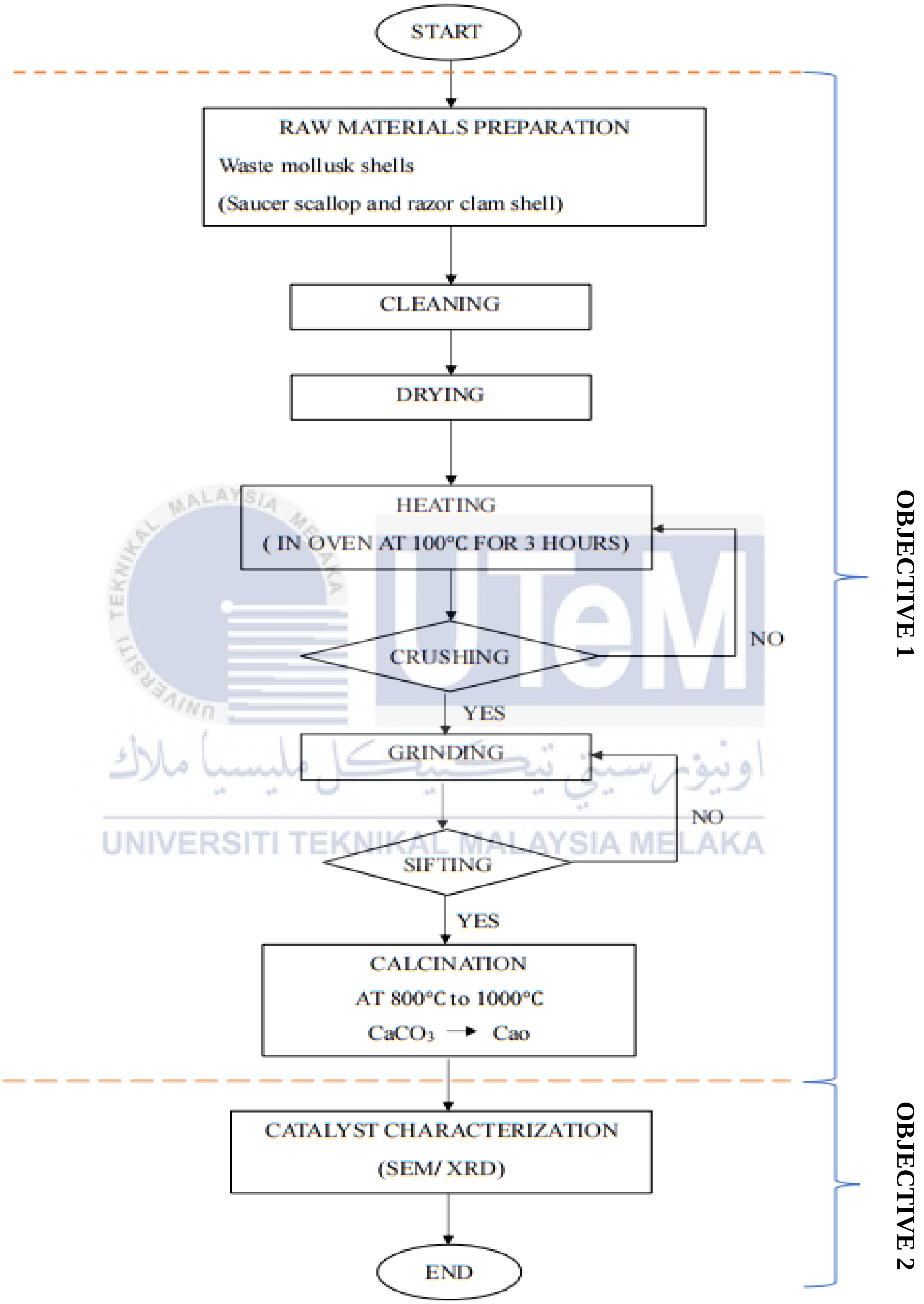


Figure 3.1: Catalyst development flow chart

Figure 3.1 shows the flow chart for biodiesel production starting with the preparation of raw materials, namely waste cooking oil, catalyst from waste shells and alcohol. In this research, the waste cooking oil used is from the same source while for alcohol, methanol is used because it is cheaper compared to ethanol. Next for the catalyst, the waste shells used are the saucer scallop (*Ylistrum Balloti*) shell and the razor clam (*Ensis Arcuatus*) shell. The next process is catalyst preparation starting with cleaning, drying, heating (oven at 100℃ for 3 hours), crushing, grinding, calcination at 800℃, 900℃ and 1000℃ for 24 hours at 10 ℃/min heating rate and catalyst characterization using scanning electron microscope to identify the optimal temperature where the catalyst contains a higher amount of calcium (Ca) among these temperature. The remaining catalyst then calcined at optimal temperature for the use of biodiesel production.

3.3 Equipment

In the execution of this research, the equipment employed for catalyst development comprised the resources available at the University Technical Malaysia Melaka lab, including the laboratory ceramic mortar, laboratory test sieve, and laboratory glass jar which is found in the machine maintenance laboratory. The calcination furnace (or heat treatment furnace) used is found in the material science laboratory, Faculty of Mechanical Technology and Engineering (FTKM), the scanning electron microscope (SEM) is found in the material science laboratory, FTKM (technology campus) while X-ray diffraction (XRD) is available at the neuroscience laboratory, Faculty of Industrial and Manufacturing Technology and Engineering (FTKIP). . Additionally, household appliances such as an oven, knife, brush, tray, and blender were utilized. Table 3.1 documented the processes and equipment utilized in the catalyst production, presenting a comprehensive list of the tools employed in various stages of the research. This inclusive approach demonstrated the adaptability of the research methodology to the available resources, combining both specialized laboratory equipment and common household tools to facilitate successful catalyst development.

Table 3.1: Lists of process and equipment used in catalyst production

NO	PROCESS	EQUIPMENT
1	Cleaning	Knife, toothbrush
2	Drying	Tray
3	Heating	Oven
4	Crushing	Laboratory ceramic mortar
5	Grinding	Blender Brand: Philip Time: 1 Second
6	Sifting	Laboratory test sieve Brand: Endecotts Size: 2mm, 500 Micron
7	Calcination	Heat Treatment Furnace Brand: Nabertherm Model: N 41/H Temperature: 800°C, 900°C, 1000℃
8	Catalyst Characterization	Scanning Electron Microscope (SEM) Brand: JEOL Model: JEOL JSM-6010PLUS/LV X-ray Diffraction (XRD) Brand: Rigaku Model: MiniFlex

3.4 Catalyst development

Catalyst development refers to the process of producing catalysts from waste shells, specifically the shell of the saucer scallop (*Ylistrum Balloti*) and the shell of the razor clam

(*Ensis Arcuatus*), both of which contain calcium carbonate ($CaCO_3$) from the raw material, is referred to as catalyst development. This process continues until these waste shells are successfully decomposed through the calcination method at a temperature ranging from 800℃, 900℃ and 1000℃ for 24 hours at 10 ℃/min heating rate into calcium oxide (CaO). The process of developing a catalyst takes quite a bit of time since it involves several different procedures throughout its production. These processes include catalyst selection and catalyst preparation, both of which will be elaborated upon in sections to come.

3.4.1 Catalyst selection

The selection of the catalyst is based on the type of catalyst to be used, either homogeneous or heterogeneous. In this research, a heterogeneous catalyst, which is a solid catalyst, is used, and waste shells will be the source of the raw material. The shells of the saucer scallop (*Ylistrum Balloti*) and the razor clam (*Ensis Arcuatus*) was obtained from a food stall (Pasri Seafood Restaurant) in Melaka. This shells that were used as catalysts in this research contained calcium carbonate ($CaCO_3$), which was decomposed into calcium oxide (CaO) through the calcination method at temperatures ranging from 800℃, 900℃ and 1000℃ for 24 hours at 10 ℃/min heating rate. Following this, these catalysts undergo characterization through the utilization of a scanning electron microscope (SEM) together with energy dispersive X-ray spectroscopy (EDS) , enabling the identification of morphology structure and elements present in the shells both before and after the calcination process and X-ray diffraction (XRD) to detect the composition that present in the raw and calcined catalyst.

3.4.2 Catalyst preparation

Razor clam shell

The razor clam shell was cleaned with a knife and brush to remove any contaminants

The razor clam shell was dried under the sun

The razor clam shell was heated at 100°C for 3 hours in the oven

The razor clam shell was crushed in a laboratory ceramic mortar before being blended

The razor clam shell was ground using a blender for a second

The ground razor clam shell was sieved using a laboratory test sieve with a 2 mm size

Razor clam shell with 2 mm in size

The razor clam shell was calcined at temperatures of 800°C, 900°C, and 1000°C

The razor clam shell, calcined at 800°C, 900°C, and 1000°C, was characterized with SEM to identify the optimal temperature

The razor clam shell was calcined at 900°C, the chosen optimal temperature

Calcined razor clam shell

Figure 3.2: Catalyst preparation flow process of razor clam shell

The flow diagram for preparing the catalyst from the razor clam shell is shown in Figure 3.2. In general, both the inside and outside of the razor clam shell contained a significant amount of dirt, including mud and fat adhering to the surface. This dirt had to be carefully scraped off from the skin using a knife, and the inside of the shell needed to be brushed to remove any fat residues. Cleaning the razor clam shell was a time-consuming process due to the delicate and sharp nature of the shell, making it challenging to clean. After washing, the razor clam shell was left to sun-dry until it was completely dry and free of any lingering odors.

Subsequently, the razor clam shell was heated in an oven at 100°C for 3 hours before being crushed using a laboratory ceramic mortar, ground, sieved with 2 mm laboratory test sieve, and subjected to the calcination method at temperatures between 800℃, 900℃ and 1000℃ for 24 hours at 10 ℃/min heating rate to decompose the calcium carbonate ($CaCO_3$) into calcium oxide (CaO). After being calcined at 800℃, 900℃, and 1000℃, these 2 mm in sized calcined samples underwent characterization using a combination of scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS) known as SEM-EDS to identify the elements contained in the catalyst and determine which temperature had a higher amount of calcium (Ca) for the selection of an optimal temperature.

Next, the raw and calcined catalyst also undergo a characterization using X-ray diffraction method to identify the elements that present in the catalyst. The remaining raw catalyst of the razor clam shell was subsequently calcined at the optimal temperature for use in the production of biodiesel. The preparation flow process of the catalyst using saucer scallop shell was performed the same way with razor shell as shown in Figure 3.2.

CHAPTER 4

RESULT AND DISCUSION

4.1 Introduction

The envisioned result of this research project was the production of a catalyst containing calcium carbonate in its raw state. The calcined catalyst was expected to consist of calcium oxide, signifying the successful decomposition of calcium carbonate ($CaCO_3$) into calcium oxide (CaO). Furthermore, for its application in biodiesel production, it was anticipated to demonstrate exceptional efficiency in generating biodiesel that adheres to the standards of both ASTM D6751 and EN 14214. The resulting solid catalyst underwent examination using characterisation methods, a scanning electron microscope (SEM) combined with energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD). The morphological structure and elements contain of the raw and calcined shell was examined using the SEM-EDS and XRD.

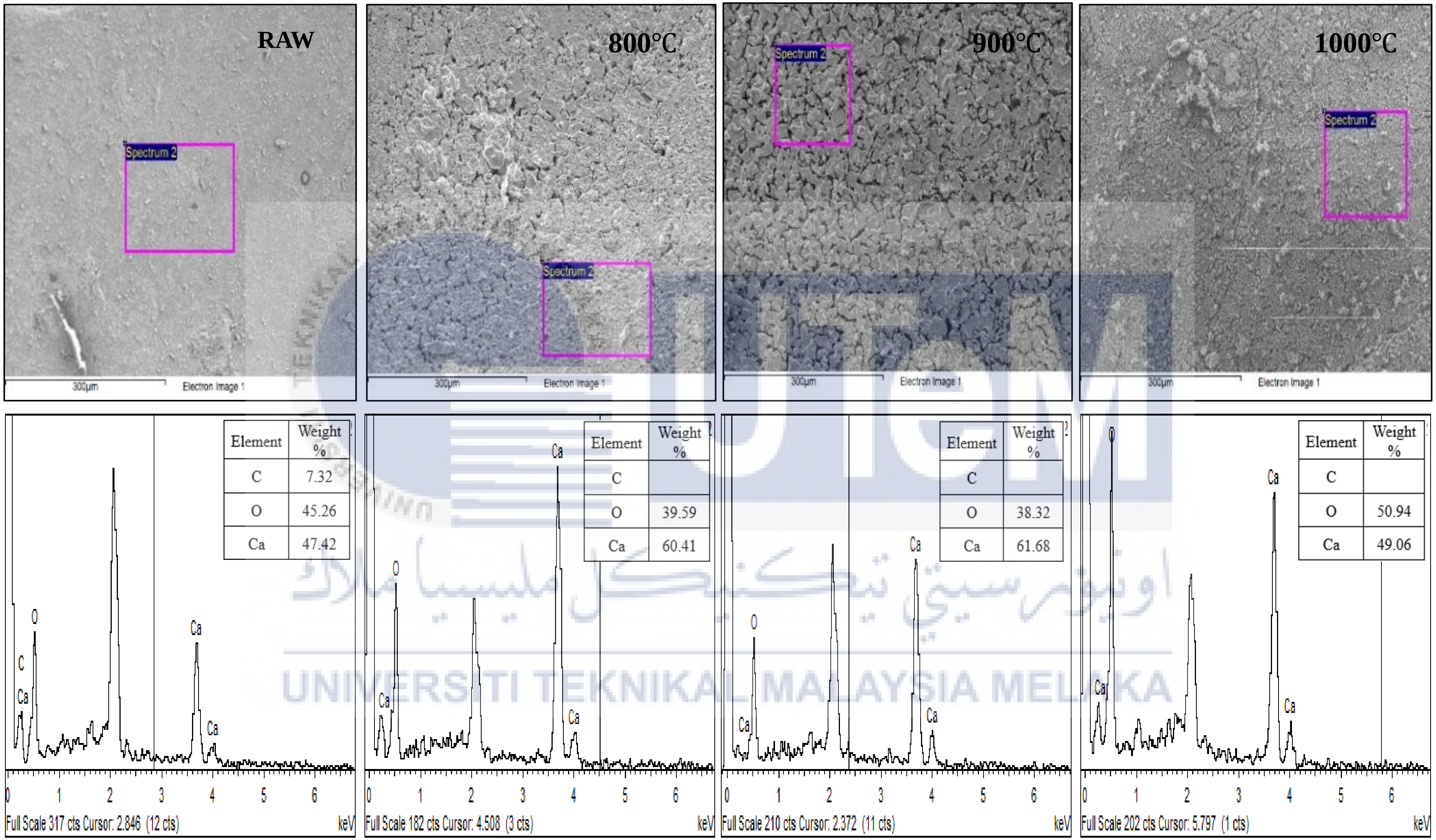
4.2 Catalyst Characterization

Catalyst characterization involves studying the properties, structure, and composition of catalyst materials. In this research, the catalyst characterization method used is SEM coupled with energy dispersive X-ray spectroscopy (EDS), known as SEM-EDS, and XRD. These methods were used to examine the morphological structure and elemental composition of both raw and calcined razor clam shell and saucer scallop shell.

4.2.1 Scanning Electron Microscope (SEM) Analysis

Scanning electron microscope (SEM) is one of the catalyst characterization methods used for characterizing the catalyst. Generally, SEM is used to examine the morphological structure of the catalyst and in order to detect the elements content of the catalyst, it was combined with energy dispersive X-ray spectroscopy (EDS) to perform the SEM-EDS used to examine the elements content of the catalyst. Figure 4.1 and Figure 4.2 shows the SEM-EDS analysis results of the raw and calcined razor clam shell and saucer scallop shell at the temperature of 800℃, 900℃, and 1000℃ for 24 hours at 10 ℃/min heating rate.

During characterizing the razor and saucer scallop shell using scanning electron microscope (SEM) which was combined with energy dispersive X-ray spectroscopy to perform SEM-EDS with the purpose to examine the elements content of the raw and calcined shell, the purple colour square shape shown in Figure 4.1 and Figure 4.2 indicates the spectrum in area inspection that is used where the sample was examined among the area of the spectrum with the resolution of ×200 where the morphological structure of the samples is in it most clear resolution. The resulting elements contents of the shell from this SEM-EDS analysis is not representing the whole area of the sample, it just represent the elements that exist among the area of the spectrum. The type of spectrum that have been chosen either by point or area is based on which type of spectrum have the highest calcium value reading.



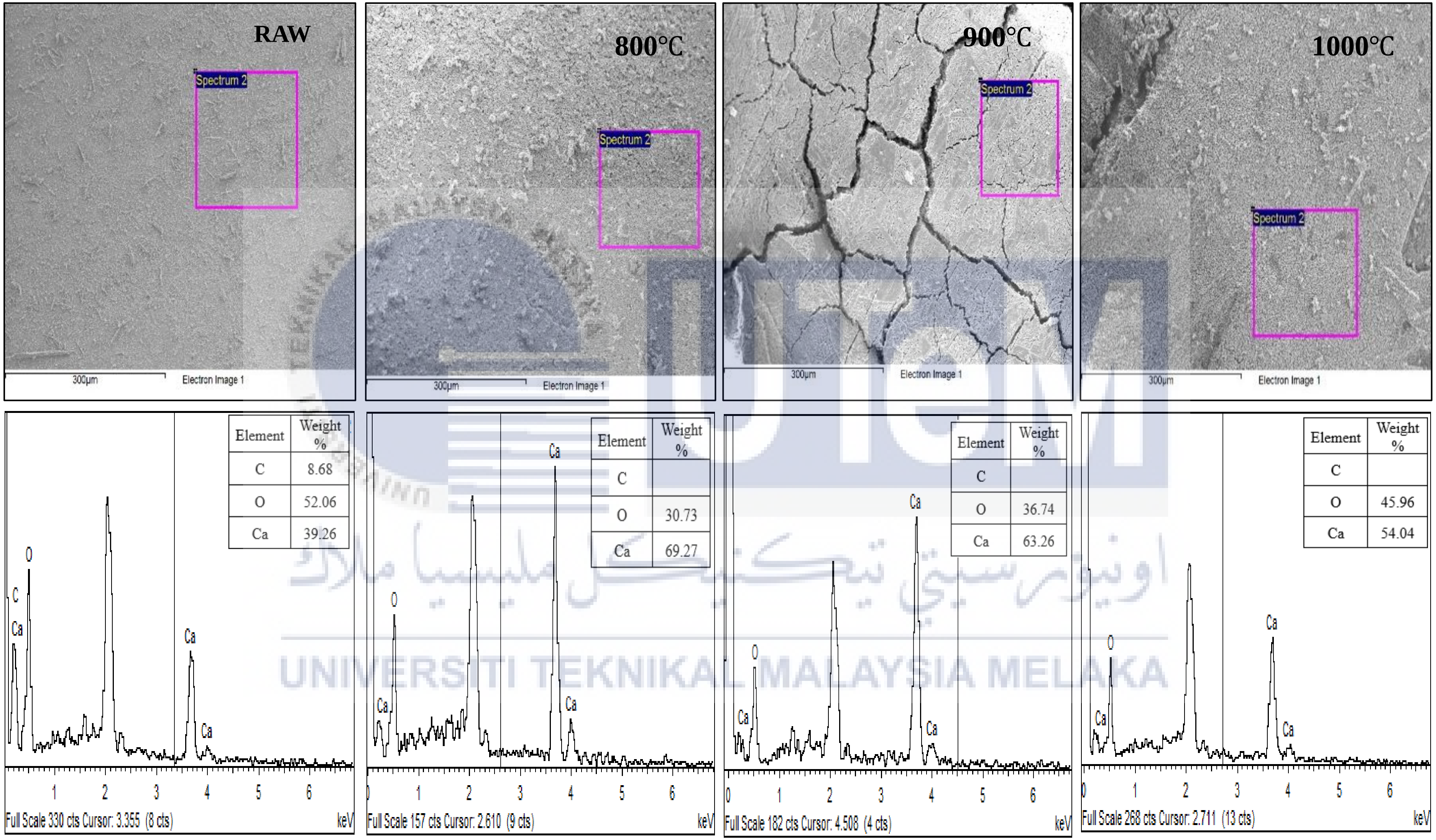
Element	Weight %
C	7.32
O	45.26
Ca	47.42

Element	Weight %
C	
O	39.59
Ca	60.41

Element	Weight %
C	
O	38.32
Ca	61.68

Element	Weight %
C	
O	50.94
Ca	49.06

Figure 4.1: SEM-EDS of razor clam shell



Element	Weight %
C	8.68
O	52.06
Ca	39.26

Element	Weight %
C	
O	30.73
Ca	69.27

Element	Weight %
C	
O	36.74
Ca	63.26

Element	Weight %
C	
O	45.96
Ca	54.04

Figure 4.2: SEM-EDS of saucer scallop shell

In this research, both 2 mm in size catalyst made from razor clam shell and saucer scallop shell was calcined at three different temperature which is 800℃, 900℃, and 1000℃ to find which temperature that the catalyst have a highest amount of calcium (Ca). It is known that the purpose of the calcination process is to decompose the calcium carbonate ($CaCO_3$) contain in the shell into calcium oxide (CaO) at high temperature.

By referring to Table 4.1, as expected, it is shown that both raw razor and saucer scallop shell that is 2 mm in size containing calcium carbonate. After being heat at a high temperature which is at 800℃, 900℃, and 1000℃, both of this 2 mm shell contain calcium oxide means that the calcium carbonate is successfully decomposed into calcium oxide throughout the calcination process. According to Zul et al., (2021), greater element content of calcium (Ca) suggests enhanced catalytic performance and effectiveness of the catalyst and this is evident when commercial calcium oxide has a higher elemental content of calcium than oxygen. Based on this SEM-EDS result, due to the highest element content of the calcium, it shown that 900℃ is the optimal temperature for calcined razor clam shell and for saucer scallop shell, the optimal temperature is 800℃. The different in optimal temperature of razor clam shell and saucer scallop shell can vary due to differences in their chemical composition and structure even though both of this shells is composed mainly of calcium carbonate ($CaCO_3$).

Table 4.1: Energy dispersive X-ray spectroscopy (EDS) results of element of commercial calcium oxide (CaO), raw and calcined razor and saucer scallop shell at 800℃, 900℃, and 1000℃

EDS Element Percentage (%)	**Samples**								
	Commercial CaO	Raw Razor	Calcined Razor (800℃)	Calcined Razor (900℃)	Calcined Razor (1000℃)	Raw Scallop	Calcined Scallop (800℃)	Calcined Scallop (900℃)	Calcined Scallop (1000℃)
Ca	62.41	47.42	60.41	61.68	49.06	39.26	69.27	63.26	54.04
O	37.59	45.26	49.59	38.32	50.94	52.06	30.73	36.74	45.96
C	N/A	7.32	N/A	N/A	N/A	8.68	N/A	N/A	N/A

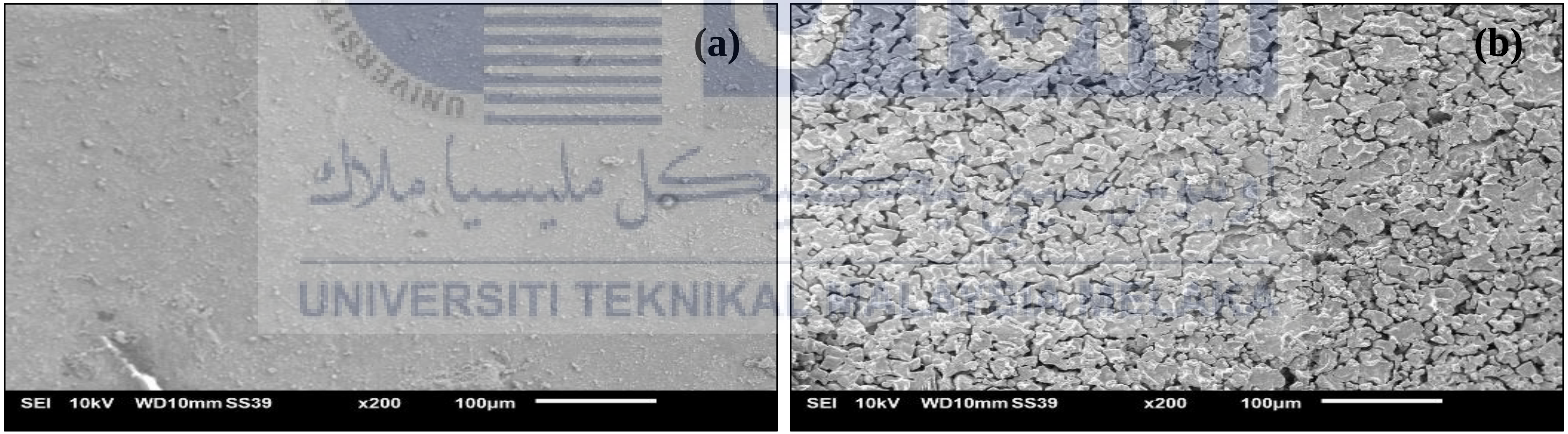


Figure 4.3: SEM morphological structure of (a) uncalcined razor clam shell and (b) calcined razor clam shell at 900℃

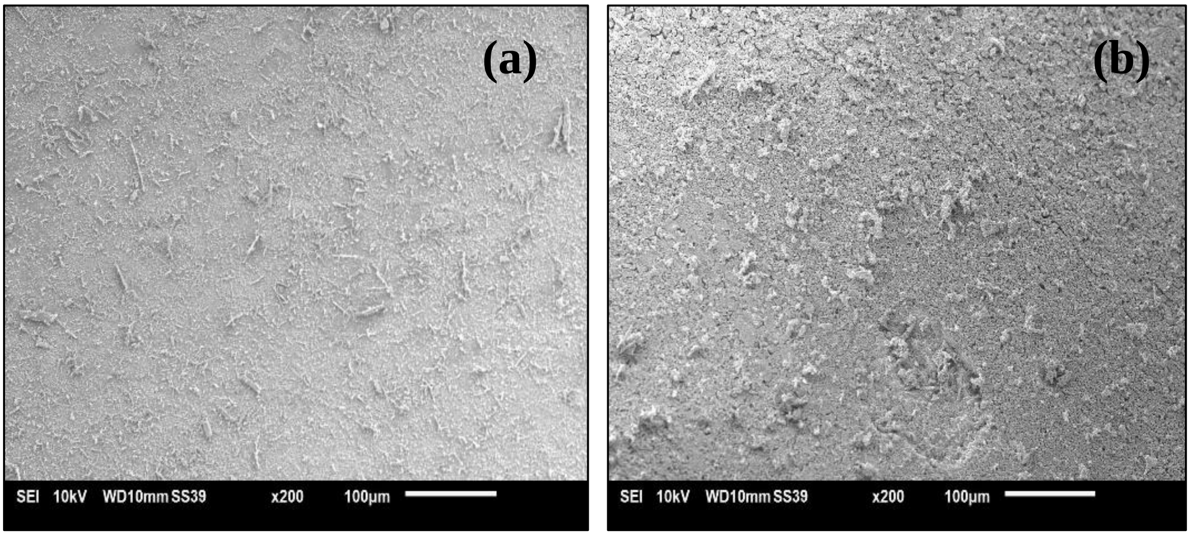
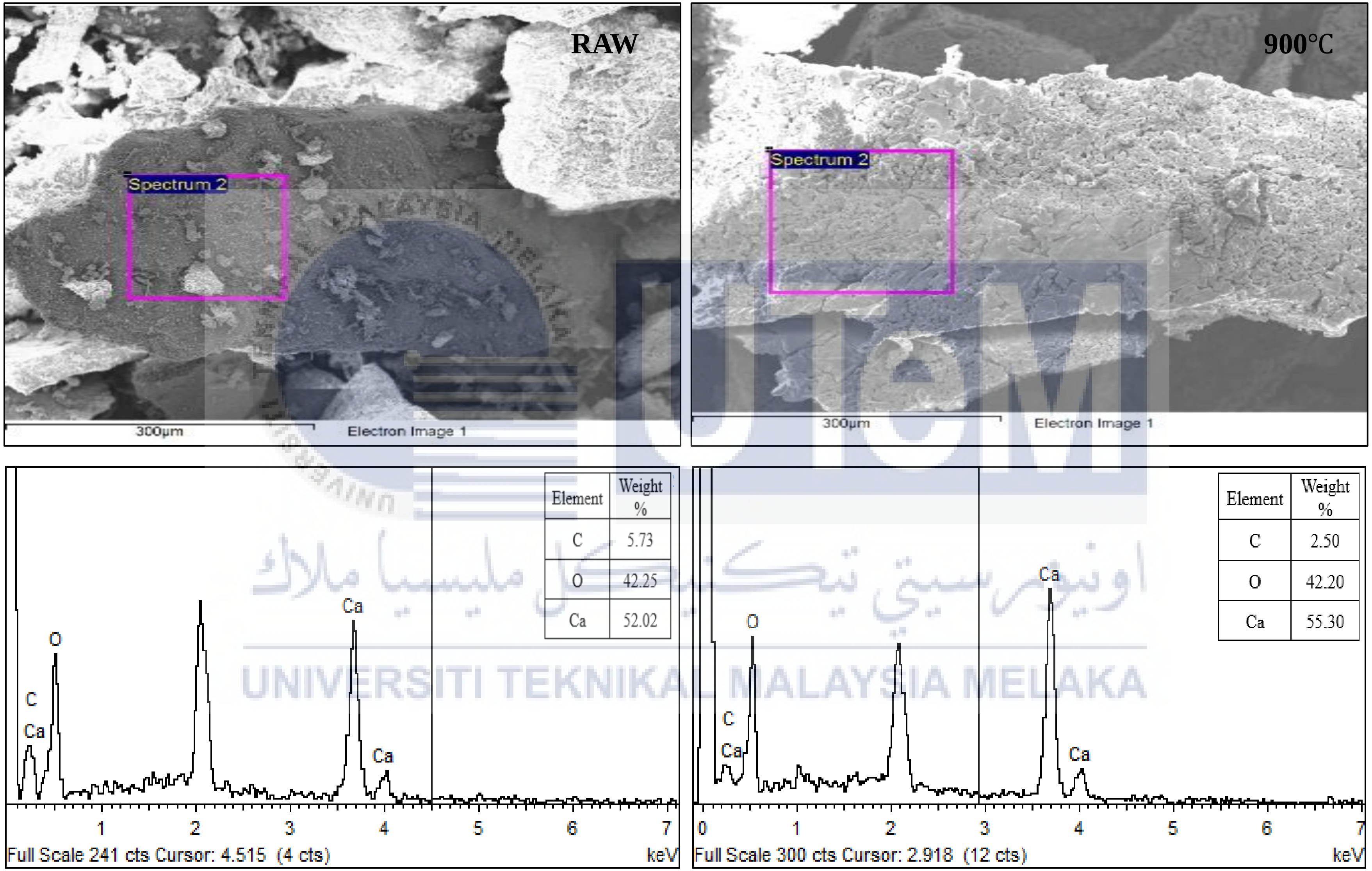


Figure 4.4: SEM morphological structure of (a) uncalcined saucer scallop shell and (b) calcined saucer scallop shell at 800℃

The morphological structure of uncalcined razor and saucer scallop shells in Figure 4.3(a) and Figure 4.3 (a) shows that before these shells undergo the calcination process, it seems to exhibit unclear and irregular shape or size. After these shells is calcine at 900℃ and 800℃ as shown in Figure 4.3 (b) and Figure 4.3 (b), the irregular shape of the particles seems to gather and form a macropores structure that create voids and spaces within the particles. This may be due to the decomposition of the calcium carbonate ($CaCO_3$) into calcium oxide (CaO) which leading to the release of carbon dioxide gas, water, and organic matter (Jamilludin et al., 2023).

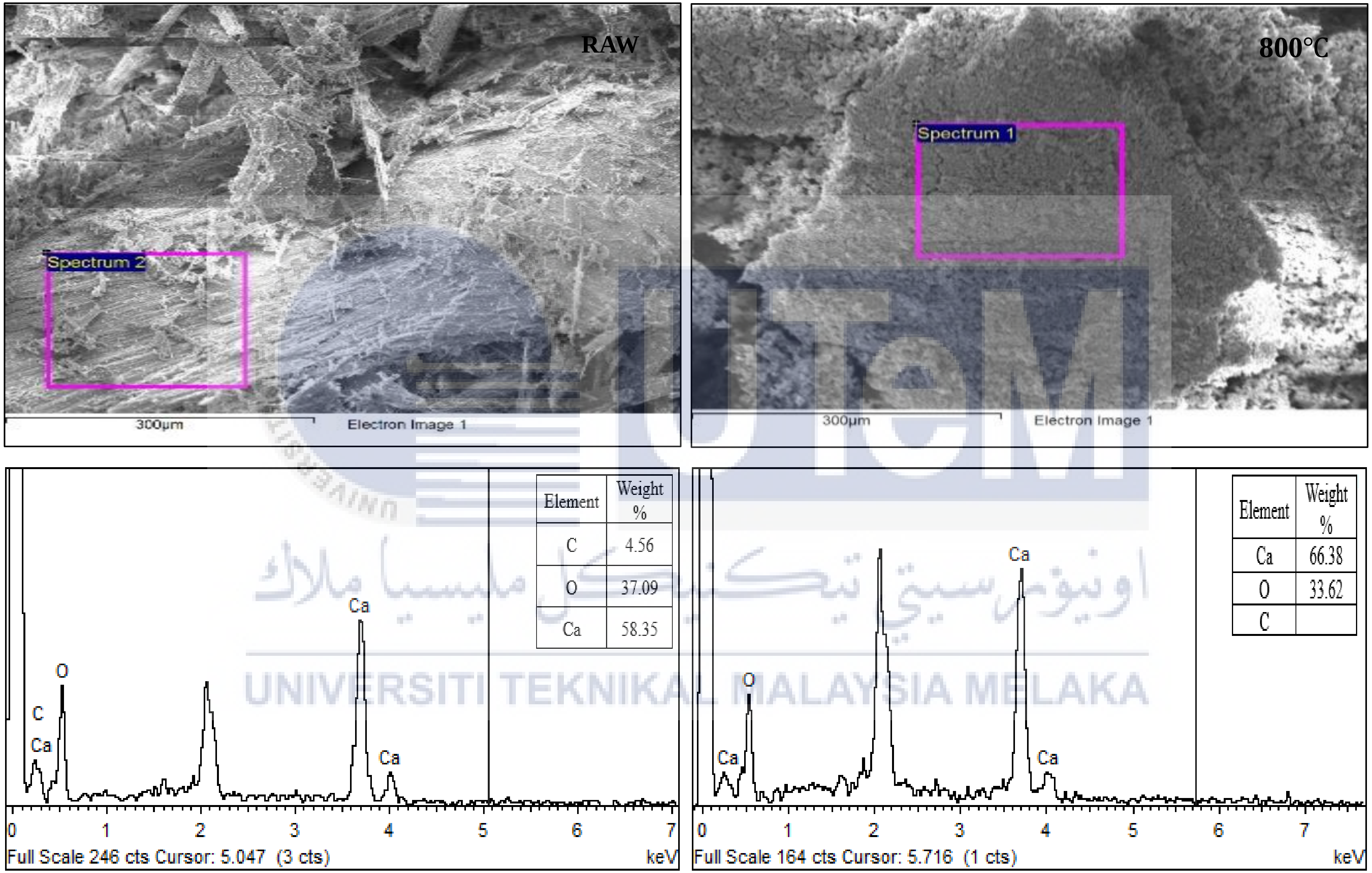
In this research, other than the 2 mm in size of the catalyst that is developed, there are also a 500 micron in size catalyst was also developed for the purpose of making a comparison between these two size of the catalyst made from both razor and saucer scallop shell which size is most suitable used for biodiesel production. The 500 micron size of the shell was calcined once after the optimal temperature have been decided which is 900℃ and 800℃. Figure 4.5 and Figure 4.6 represents the SEM-EDS analysis of 500 micron razor and saucer scallop shell.



Element	Weight %
C	5.73
O	42.25
Ca	52.02

Element	Weight %
C	2.50
O	42.20
Ca	55.30

Figure 4.5: SEM-EDS of 500 micron razor clam shell



Element	Weight %
C	4.56
O	37.09
Ca	58.35

Element	Weight %
Ca	66.38
O	33.62
C	

Figure 4.6: SEM-EDS of 500 micron saucer scallop shell

Table 4.2: Comparison between the SEM-EDS results of 2 mm and 500 micron of raw and calcined razor and saucer scallop shell

Catalyst	Optimal Temperature	Catalyst Size	Elements Percentage (%)		
			Ca	O	C
Razor clam shell	900℃	2 mm	61.68	38.32	N/A
		500 micron	55.30	42.20	2.50
Saucer scallop shell	800℃	2 mm	69.27	30.73	N/A
		500 micron	66.38	33.62	N/A

Refer to Table 4.2, it can be seen that the elements weight of calcium (Ca) in the 2 mm calcined razor and saucer scallop shells is higher than the 500 micron. It was detected that the element such as carbon is existed in the 500 micron calcined razor clam shell during the calcination process. This carbon may be existed due to the condition of the furnace that was used for heating a metal before it used to calcine the 500 micron of the razor and saucer scallop shells.

Based on the SEM-EDS result of the 2 mm and 500 micron calcined razor and saucer scallop shells, it is confirmed that 2 mm is the most suitable size compared to 500 micron if refer to the calcium content of the calcined shells. Other than that, it is known that the catalyst which is 500 micron in size or in powder form can cause the filtration paper easily to clog. This is because it is already dissolved with the mixture of the waste cooking oil and alcohol even it is not completely poured into the filtration paper during the filtration process of transferring this mixture into the separating funnel before transferring and filter it for the second time into the conical flask.

The 2 mm calcined razor and saucer scallop shells, if compared to the 500 micron or powder form of this catalyst, it is not easily clog because during the biodiesel production process, this catalyst is not dissolved. Therefore, when transferring and filtrated this mixture into the separating funnel, the 2 mm catalyst is not poured into the filtration paper, only the mixture of this reaction is poured into the filtration paper. This is the reason why 2 mm of the razor clam shell is used in the production of the biodiesel. In addition to this matter, this 2 mm catalyst can be reuse for the purpose of the next biodiesel production.

4.2.2 X-Ray Diffraction (XRD) Analysis

Basically, the X-ray diffraction (XRD) is a technique that may be used to evaluate the crystallinity of the catalyst and perform qualitative detection of the components that are present. In this research, XRD analysis is used to determine the phase presents in the raw and calcined razor clam shell at 900℃ and saucer scallop shell and 800℃ to detect the presents of the calcium carbonate ($CaCO_3$) and calcium oxide (CaO) based on the peaks that obtained from the XRD analysis pattern. In the research reported by Najah et al., (2020), the data of XRD for calcium carbonate ($CaCO_3$) and calcium oxide (CaO) from Joint Committee of Powder Diffraction Standard (JCPDS) is shown in Table 4.3 together with the XRD data of raw and calcine razor clam shell and saucer scallop shell.

Table 4.3: XRD data of calcium carbonate ($CaCO_3$) and calcium oxide (CaO) from JCPDS, raw and calcine razor clam shell and saucer scallop shell

Samples	**Compound**	2θ				
JCPDS	$CaCO_3$	29.4°	39.4°	43.2°	47.4°	48.5°
	CaO	32.2°	37.3°	53.8°	64.1°	67.3°
Raw Razor	$CaCO_3$	29.4°	39.4°	43.0°	47.5°	48.4°
	CaO	N/A	N/A	N/A	N/A	N/A
Calcined Razor	$CaCO_3$	N/A	N/A	N/A	N/A	N/A
	CaO	32.2°	37.4°	53.9°	64.1°	67.4°
Raw Scallop	$CaCO_3$	29.4°	39.5°	43.2°	47.5°	48.5°
	CaO	N/A	N/A	N/A	N/A	N/A
Calcined Scallop	$CaCO_3$	N/A	N/A	N/A	N/A	N/A
	CaO	32.2°	37.3°	53.8°	64.1°	67.4°

In X-ray diffraction (XRD), the intensity at 2θ are the standard and widely accepted notation for describing diffraction angles in X-ray diffraction studies as it helps identify the crystal lattice spacing and provides information about the crystal structure of the material being studied. The XRD pattern of raw and calcined razor clam shell at 900℃ was plotted using an Origin 2024 software as shown in Figure 4.7. As stated by JCPDS in Table 4.3, the peaks of calcium carbonate ($CaCO_3$) should be present at 2θ range 29.4°, 39.4°, 43.2°, 47.4° and 48.5°. Referring to Table 4.3 and Figure 4.7, the peaks of calcium carbonate ($CaCO_3$) in razor clam shell before calcined was detected at 2θ range 29.4°, 39.4°, 43.0°, 47.5° and 48.4° and this result parallel with what is recorded by JCPDS. This results confirmed that raw razor clam shell contain calcium carbonate ($CaCO_3$) before calcination process.

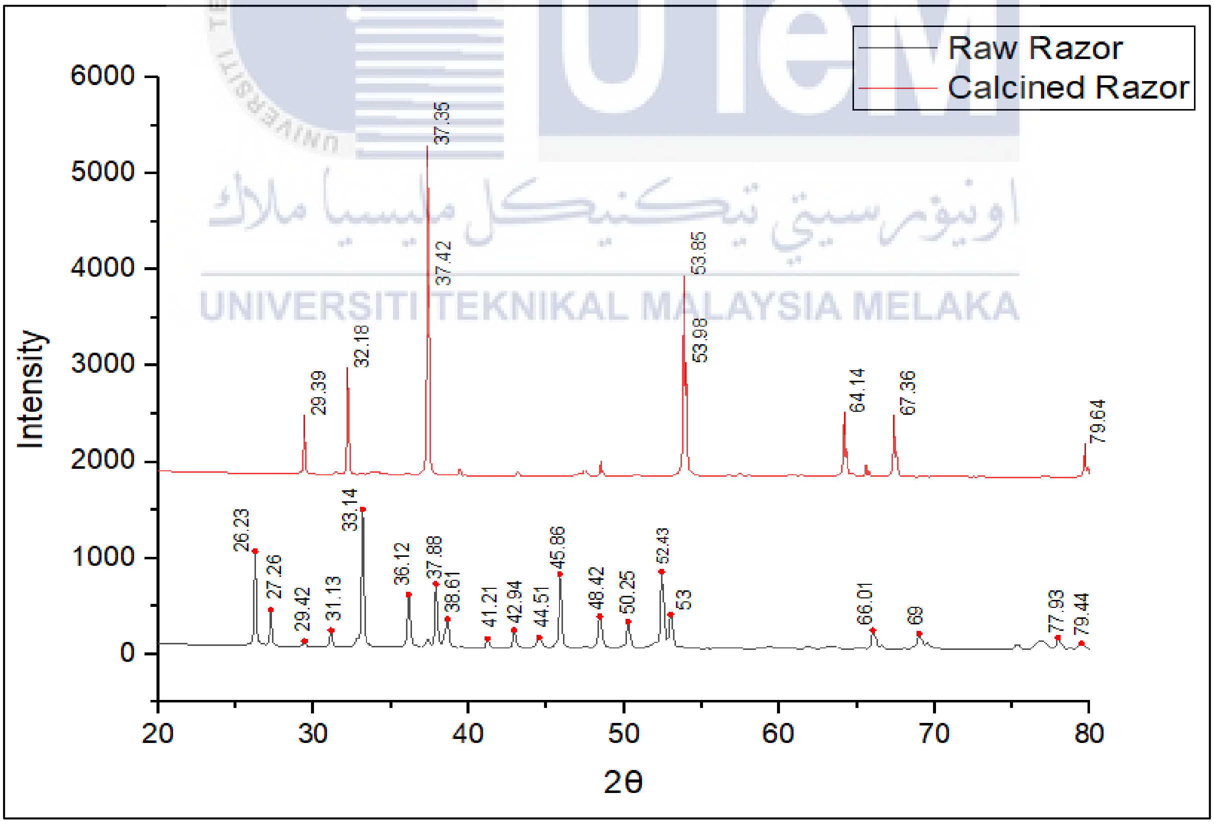


Figure 4.7: XRD pattern of raw and calcined razor clam shell at 900℃

According to the research done by Hazri et al., (2020) and Lesbani et al., (2016), Joint Committee of Powder Diffraction Standard (JCPDS) state that the calcium oxide (CaO) peaks must be present at the intensities 2θ range between 32.2°, 37.3°, 53.8°, 64.1°, and 67.3°. The XRD pattern of calcined razor clam shell at 900℃ in Figure 4.7 shows that the peaks of calcium oxide was detected in the calcined razor clam shell at 2θ range 32.2°, 37.4°, 53.9°, 64.1°, and 67.4°. This XRD pattern on calcined razor clam shell are similar with the research done by Aitlaalim et al., (2020) where the calcium oxide (CaO) peaks was appeared at 2θ range 32.26, 37.44, and 53.9◦. These result confirm that $CaCO_3$ was decomposed into CaO after the razor clam shell calcined at 900℃.

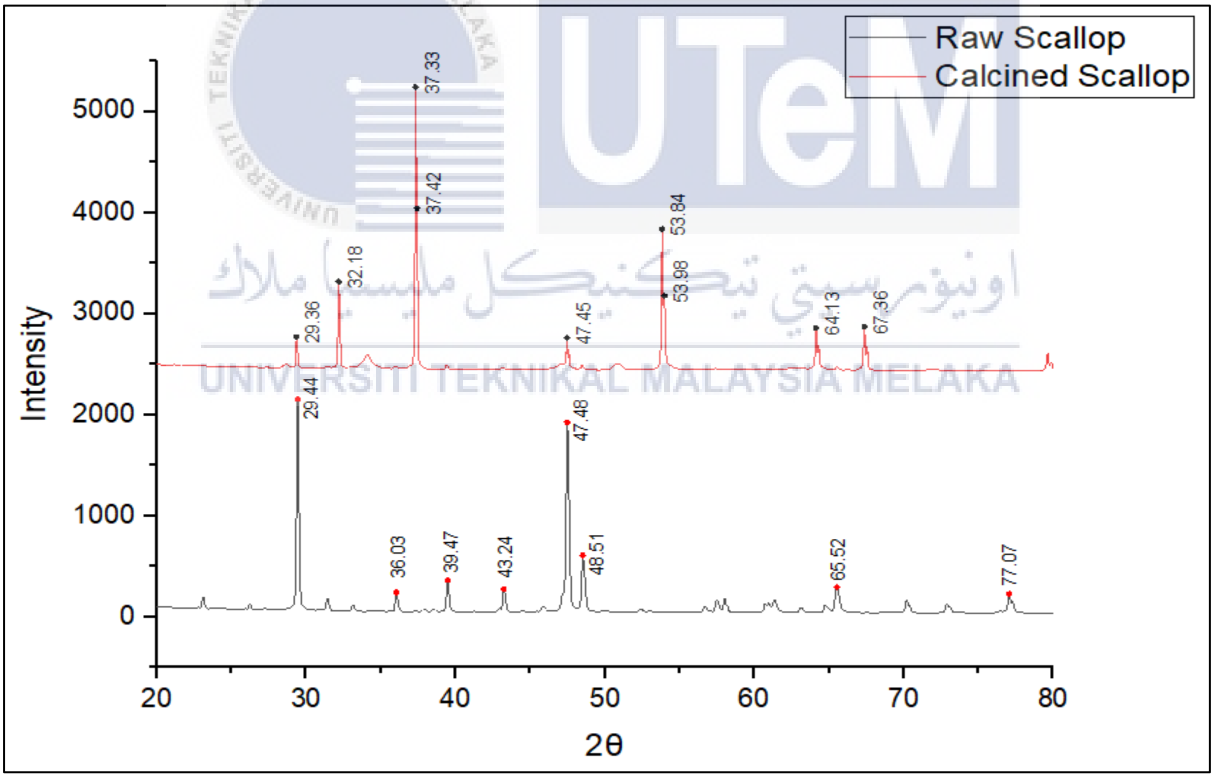


Figure 4.8: XRD pattern of raw and calcined saucer scallop shell at 800℃

Figure 4.8 shows the XRD pattern of raw and calcined saucer scallop shell at 800℃ that was plotted using an Origin 2024 software. The JCPDS stated that the peaks of calcium carbonate should be present at the intensity of at 2θ range 29.4°, 39.4°, 43.2°, 47.4° and 48.5°. In research conducted by Izza et al., (2020), it was reported that the main peaks of calcium carbonate ($CaCO_3$) for scallop shell is at the intensities of 2θ range 24°, 29°, 32°, 39°, 43°, 47° and 48° and based on the JCPDS (85-1108), this resulting peaks indicates the calcium carbonate ($CaCO_3$) for scallop shell. If refer to Table 4.3 and Figure 4.8, the presence of $CaCO_3$ in uncalcined saucer scallop shell was detected at 2θ range 29.4°, 39.5°, 43.2° ,47.5° and 48.5°. The obtained results align with the findings reported in the JCPDS standard and the study conducted by Izza et al. (2020), confirming the presence of calcium carbonate ($CaCO_3$) in the uncalcined saucer scallop shell before the calcination process.

According to the research done by Chaihad et al., (2020), the calcined scallop shell have a specific peaks at the intensities of 2θ range 32.64°, 37.69°, 54.26°, 64.55° and 67.74° in which this peaks represent the characteristic of the calcium oxide (CaO). Izza et al., (2020) reported that after the saucer scallop shell was calcined at 800℃, the new peaks that represented the calcium oxide (CaO) was obtained at the intensities of 2θ range 34°, 51°, 54°, 63° and 64°. Based on JCPDS (48-1467), this resulting peaks indicates the calcium oxide (CaO) for scallop shell. The XRD data and pattern in Table 4.3 and Figure 4.8 shows that calcined saucer scallop shell at 800℃ contain calcium oxide (CaO) where the peaks of calcium oxide was detected at 2θ range 32.2°, 37.3°, 53.8°, 64.1°, and 67.4°. It is confirmed that $CaCO_3$ was decomposed into CaO after the saucer scallop shell calcined at 800℃.

4.2.3 Fuel Properties

The prepared biodiesel as shown in Table 4.4 was obtained from the biodiesel production where calcined razor clam shell at 900℃ that prepared in this research was used for the production of the biodiesel. The property of the prepared biodiesel was compared to the requirement of biodiesel that have been set by ASTM D6751 and EN 14214. It is notice that the prepared biodiesel properties was test based on the acid value, flashpoint, and density.

Table 4.4: Properties of prepared biodiesel compared to ASTM D6751 and EN 14214

Property	Unit	Prepared Biodiesel	ASTM D6751	EN 14214
Acid value	mgKOH/g	0.486	less than 0.5	0.5max
Flashpoint	℃	222	greater than 93	greater than 120
Density	g/ml	881	NA	860 to 900

Refer to Table 4.4, it shows that acid value of the prepared biodiesel can be consider less than 0.5 mgKOH/g where ASTM D6751 and EN 14214 standard for biodiesel acid value is less than 0.5 mgKOH/g. The flashpoint of the prepared biodiesel is 222℃ which is higher than the requirement for biodiesel flashpoint set by ASTM D6751 and EN 14214 standard which must be higher than 93℃ and 120℃. The prepared biodiesel density is met the requirement set by the EN 14214 standard in which it density is between the range 860 g/ml to 900 g/ml. Based on this results, it is confirmed that the prepared biodiesel meets the requirements of ASTM D6751 and EN 14214 standard.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In summary, this study successfully aimed to produce a heterogeneous catalyst containing calcium oxide (CaO) derived from waste saucer scallop (*Ylistrum Balloti*) and razor clam (*Ensis Arcuatus*) shells. The calcination process effectively decomposed calcium carbonate ($CaCO_3$) in the shells into calcium oxide (CaO) at temperatures of 800°C, 900°C, and 1000°C. The resulting catalysts were characterized using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD) to determine their morphology, elemental composition, and optimal calcination temperature for higher calcium content.

The waste shells underwent a series of preparation steps, including cleaning, drying, and grinding, before being calcined at different temperatures. SEM-EDS analysis revealed the elemental composition of the raw and calcined shells, while XRD confirmed the transformation of $CaCO_3$ into CaO. The study demonstrated that 900℃ was the optimal calcination temperature for razor clam shells, while 800℃ was optimal for saucer scallop shells, based on the highest calcium content. The XRD patterns confirmed the successful decomposition of $CaCO_3$ into CaO in both types of shells.

The envisioned outcome of the research was the production of a catalyst containing calcium oxide for biodiesel production, meeting the standards of ASTM D6751 and EN 14214. The resulting solid catalyst was characterized using SEM-EDS and XRD, confirming its suitability for biodiesel production. The research successfully demonstrated the potential of using waste materials for sustainable and environmentally friendly biodiesel production. The prepared biodiesel met the stringent requirements of ASTM D6751 and EN 14214 standards, further emphasizing the success of the developed catalysts in the production of clean biodiesel.

5.2 Recommendations

This comprehensive study successfully demonstrates the feasibility of saucer scallop and razor clam, as a raw material for the production of biodiesel and the development of a calcium oxide (CaO) catalyst. The research has achieved its objectives, meeting the stringent standards of both ASTM D6751 and EN 14214 for the produced biodiesel. The recommendation for further improvement and future research directions includes:

1. The shells should be subjected to calcination in a specialized heat treatment furnace exclusively dedicated to non-metallic samples, ensuring the exclusion of unwanted elements that might impact the composition of the shell.
2. The calcined shells should be subsequently submitted for characterization through the X-ray fluorescence (XRF) method, facilitating a comprehensive analysis and extraction of detailed data regarding the elemental composition within the shells.
3. Heat the saucer scallop shell more than 3 hours before crushing it since that this shell are tough to crush, and the prolonged heat exposure makes them more fragile, making the crushing process easier.
4. Thermogravimetric analysis (TGA) should be incorporated in catalyst development to optimize calcination, estimate loading, study support interactions, ensure cluster consistency, and use kinetic analysis for enhanced synthesis and stability evaluation.
5. Brunauer-Emmett-Teller (BET) analysis should be integrated as a standard procedure in catalyst development to understand surface area and porosity, crucial factors

influencing catalytic activity, for informed decisions and enhanced performance optimization.

6. It is recommended to use a combination of sieve analysis, digital callipers, and image analysis software for accurate verification of crushed razor shell particles at 2 mm, ensuring reliable size confirmation and precise quality control in production, while adherence to standardized procedures, instrument calibration, and multiple measurements enhances accuracy.

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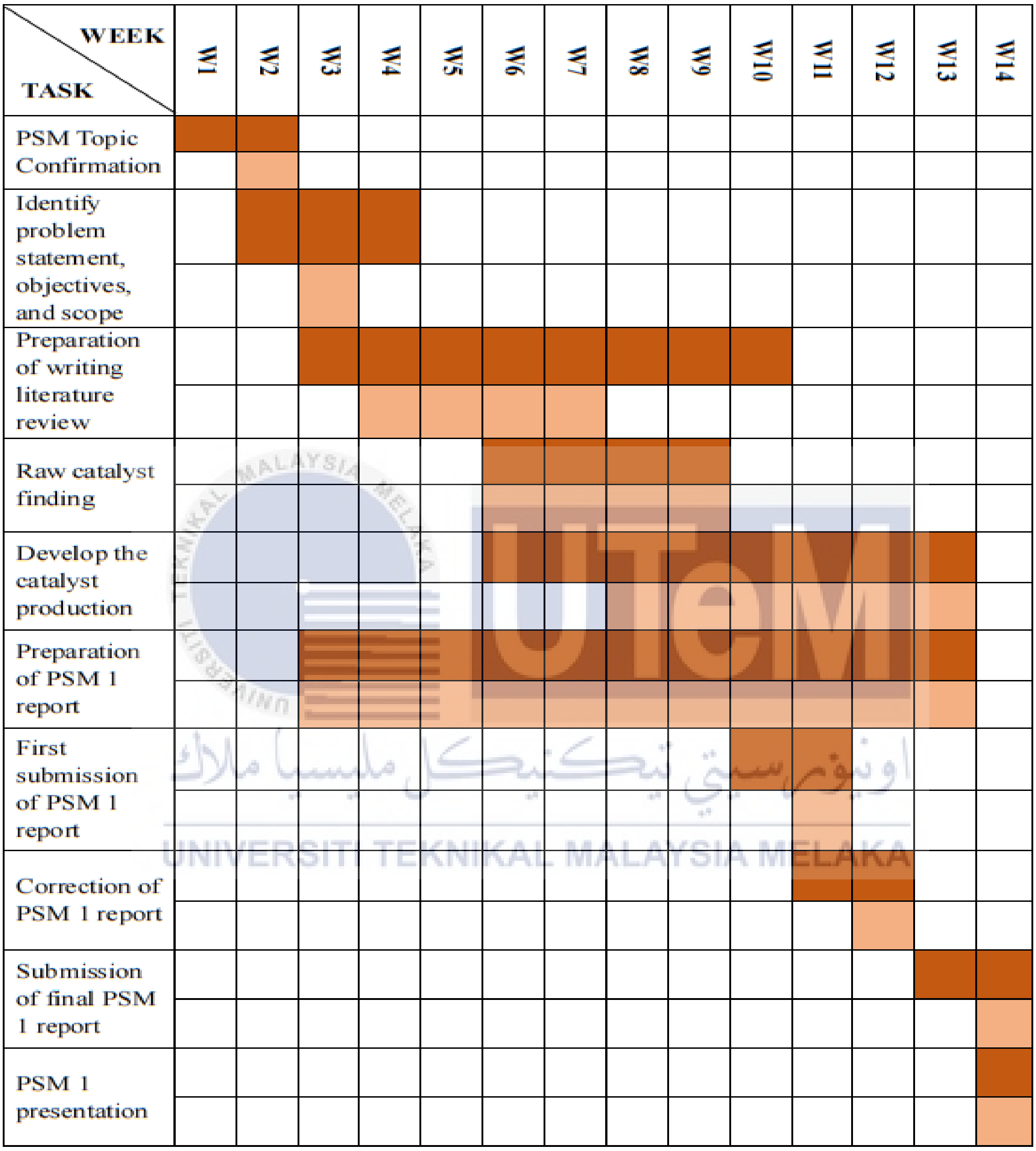
APPENDIX

Appendix 1: Gantt chart PSM 1



WEEK TASK	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14
PSM Topic Confirmation	■	■												
		▨												
Identify problem statement, objectives, and scope		■	■	■										
			▨											
Preparation of writing literature review			■	■	■	■	■	■	■	■				
				▨	▨	▨	▨							
Raw catalyst finding						■	■	■	■					
						▨	▨	▨	▨					
Develop the catalyst production						■	■	■	■	■	■	■	■	
								▨	▨	▨	▨	▨	▨	
Preparation of PSM 1 report			■	■	■	■	■	■	■	■	■	■	■	
			▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	
First submission of PSM 1 report										■	■			
											▨			
Correction of PSM 1 report											■	■		
												▨		
Submission of final PSM 1 report													■	■
														▨
PSM 1 presentation														■
														▨

NOTE:

■ Plan

▨ Actual

Appendix 2: Gantt chart PSM 2

WEEK TASK	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14	W15
Raw Material preparation	🟫	🟫	🟫	🟫											
	🟧	🟧	🟧												
Catalyst preparation				🟫	🟫	🟫	🟫	🟫							
				🟧	🟧	🟧									
Catalyst characterization						🟫	🟫	🟫	🟫	🟫					
							🟧	🟧	🟧	🟧					
Results and data discussion						🟫	🟫	🟫	🟫	🟫	🟫	🟫			
							🟧	🟧	🟧	🟧	🟧	🟧			
Preparation of PSM 2 report			🟫	🟫	🟫	🟫	🟫	🟫	🟫	🟫	🟫	🟫	🟫		
			🟧	🟧	🟧	🟧	🟧	🟧	🟧	🟧	🟧	🟧	🟧		
First submission of PSM 2 report											🟫	🟫			
												🟧			
Correction of PSM 2 report													🟫	🟫	🟫
													🟧		
Submission of final PSM 2 report														🟫	🟫
														🟧	🟧
PSM 2 presentation															🟫
															🟧

NOTE:

🟫 Plan

🟧 Actual