RECOVERY OF OIL FROM PALM OIL MILL EFFLUENT (POME) AS BIODIESEL FEEDSTOCK

AMIRANORDIANA BINTI BAHAROM

UINVERSITI SAINS MALAYSIA

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RECOVERY OF OIL FROM PALM OIL MILL EFFLUENT (POME) AS BIODIESEL FEEDSTOCK

by

AMIRANORDIANA BINTI BAHAROM

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Symbol	Description	Unit
ρ	Density	kg/m^3
μ	Kinematic viscosity	mm^2/s

LIST OF ABBREVIATION

Symbol	**Description**
BOD	Biological oxygen demand
CO_2	Carbon dioxide
COD	Chemical oxygen demand
FTIR	Fourier transform infrared
GDP	Gross domestic product
KOH	Potassium hydroxide
$MgSO_4$	Magnesium hydroxide
MPOC	Malaysia Palm Oil Council
NaOH	Sodium hydroxide
NH_3-N	Ammonical nitrogen
O&G	Oil and grease
POME	Palm oil mill effluent
SS	Suspended solids
TN	Total nitrogen
TS	Total solids

PEMULIHAN SEMULA MINYAK DARIPADA EFLUEN KILANG MINYAK KELAPA SAWIT SEBAGAI BAHAN SUAPAN BIODISEL

ABSTRAK

Permintaan tenaga bersih global di dunia semakin meningkat dengan pesat, terutamanya di antara negara maju. Terdapat kebimbangan yang semakin meningkat terhadap ketersediaan bahan api fosil yang kian berkurangan kerana ia sering digunakan untuk menghasilkan tenaga. Oleh itu, perhatian yang lebih besar telah diberikan untuk mencari sumber alternatif yang boleh diperbaharui, seperti biodiesel, yang merupakan bahan api diperbuat daripada minyak tumbuhan atau lemak haiwan. Memandangkan Malaysia merupakan salah satu pembekal minyak sawit terbesar di dunia, efluen kilang minyak kelapa sawit (POME) telah menjadi sumber utama pencemaran terhadap alam sekitar kerana kandungan minyak dan grisnya yang tinggi. Justeru, kajian ini fokus akan pemulihan minyak daripada effluen kilang minyak kelapa sawit (POME) yang akan digunakan sebagai bahan mentah dalam pengeluaran biodiesel. Minyak ini akan diekstrak dengan kaedah pengekstrakan pelarut organik, dengan mempelbagaikan jenis pelarut, nisbah pelarut kepada POME dan masa pengekstrakan untuk mendapatkan keadaan optimum untuk penhasilan minyak yang tinggi. N-heksana ialah pelarut yang paling sesuai dengan 2.6% pemulihan minyak bagi setiap 100 ml POME pada nisbah pelarut kepada POME 1:1 dan dalam masa 2 jam pengekstrakan. Tambahan pula, jumlah biodiesel yang dihasilkan daripada 10 ml minyak yang diekstrak adalah sebanyak 8.67 gram dengan kebanyakan ciri biodiesel dari POME adalah sama dengan biodiesel yang berdasarkan piawaian Malaysia MS 2800:2800.

RECOVERY OF OIL FROM PALM OIL MILL EFFLUENT (POME) AS BIODIESEL FEEDSTOCK

ABSTRACT

The global energy demand in the world has been increased tremendously, especially within developing countries. There are growing concern on the availability of fossils fuel that rapidly depleting as it keeps being used to produce energy to meet the demand. Therefore, greater attention has been placed on finding alternative renewable sources, such as biodiesel, which included producing fuel from oil made of plant or animal fat. Since Malaysia is one of the largest palm oil suppliers in the world, palm oil mill effluent (POME) has become a major source of pollution due to its high concentration of oil and grease content. Hence, this study is focusing on the recovery of oil from POME as a feedstock in the production of biodiesel. The oil is extracted using solvent extraction method, by varying types of solvent, solvent to POME ratio and extraction time to obtain the optimum condition for high yield of oil. N-hexane is the most suitable solvent with 2.6% of oil recovery for every 100 ml of POME at solvent to POME ratio of 1:1 and within 2 hours extraction time. Furthermore, the amount of biodiesel produced from 10 ml of oil extracted is about 8.67 g with most of the biodiesel characteristics in accordance with the biodiesel based on Malaysian standard MS 2800:2800.

CHAPTER 1

INTRODUCTION

The first chapter introduces an outline of the research and the significant of palm oil mill effluent (POME) treatment and its valuable by-products that can be used as a feedstock for biodiesel. In general, this chapter describes the research background of palm oil mill effluent (POME) and biodiesel, as well as the problem statement and final year project objectives.

1.2 Research Background

The global energy demand has been increasing exponentially due to the massive growth of economic and human population especially in developing countries. As fossil fuels being the most dominating sources, rapidly depleting this source to meet the energy demand will result in several environmental impacts. As shown in the Figure 1.1, the total energy consumption in Malaysia has been increasing every year since 1990. In addition, continuous usage of fossil fuels-based energy has caused a rise in global temperature, thus resulting in global warming due to the emission of greenhouse gases such as carbon dioxide (Yusoff et al.,2021a).

Hence, extensive research is done to produce an alternate energy sources which are renewable and environmentally friendly to meet the energy demand as well as secure the energy supply in the future. Biodiesel is one of the renewable energy resources that is significant as a substitute fuel for conventional engines. It also offers several advantages over fossil fuels diesel such as; 1) On a life-cycle basis, biodiesel emits 78% less CO_2 than diesel from fossil sources. 2) Biodiesel is renewable, biodegradable and more environmentally friendly. 3) It is sustainable and free from polluting compound such as sulphur and aromatic content (Mishra et al.,2018). Because of its huge potential, many countries, including Malaysia, have begun utilizing biodiesel (Atabani et al.,2012). Figure 1.1 shows the total energy consumption in Malaysia from year 1990 to 2020.

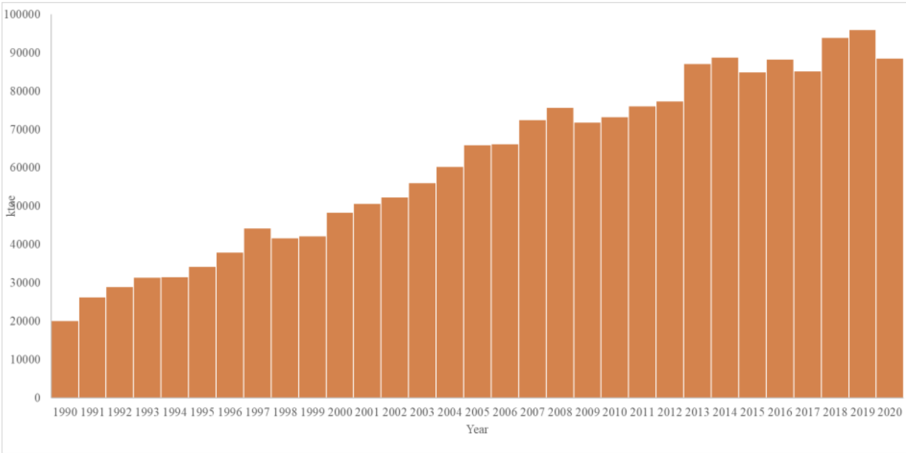

Figure 1.1: Total energy consumption in Malaysia

(source: EnerData)

Biodiesel can be synthesized from diverse feedstocks, including edible oils, non-edible oils and waste oils. However, there has been growing concerns regarding the edible oils as biodiesel feedstock because it may create a competition between the food availability and fuels production. The use of edible oils also may have an impact to the environment due to large area of lands are required for the feedstock cultivation. Hence, activities such as deforestation might occurs frequently to meet the supply demand of the feedstock (Yusoff et al.,2021a). The use of non-edible oils on the other hand can be more beneficials compared to the edible oils as it can overcome the food vs fuels problem. According to Banković-Ilić et al., (2012), another advantage of non-edible oil is that it is readily available and are abundant sources.

Palm oil mill effluent (POME) are one of sources for non-edible oil that can be potential feedstock for biodiesel production, especially in Malaysia. POME can be defined as the waste water produced from palm oil industry. Malaysia can be considered as the second largest palm oil suppliers in the world with more than 20 million tonnes of palm oil produced in 2019, and it is expected to increase the production by 2020 (Rezenia et al., 2020). With approximately 58 million tonnes of POME produced annually, the enormous quantity of POME has raised public and government concern (Ozturk et al., 2017). This is because the waste water produced from the palm oil industry has negative impact it is left untreated and discharged into the environment. This is due to the high acid content and biological oxygen demand in POME . Nevertheless, the residual oil in POME is a potential feedstock to synthesize biodiesel, Thus, many researchers are currently focusing on proper handling method of POME and suitable technique to recover the potentially valuable nutrients in POME.

Moreover, one of the limitations in the commercialization of biodiesel is the cost for the production process. According to Yusof et al., (2021a), the feedstock required in the manufacturing of biodiesel covers for about 75% of the production cost. Therefore, the utilization of low-cost oil from POME is an alternative way to minimize the production cost as well as improving the economic value in commercial industrial scale. In addition, POME has high oil and grease content that can be used for biodiesel synthesis. The challenge is how to recover the remaining oil in POME as a feedstock for the production of sustainable energy such as biodiesel. Primandari et al., (2013) state that there are several methods that can be implemented to separate the residual oil from POME, such as adsorption, chemical-biological method and solvent extraction method. In this study, solvent extraction method is chosen to recover the oil from POME due to its simplicity and ease of removing the solvent for oil characterization. Then, the oil recovered is used as raw material to produce biodiesel using transesterification reaction.

1.3 Problem Statement

Malaysia has become the second largest producer and exporter in palm oil industry with approximately 19.14 million tonnes of palm oil and 2.20 tonnes of palm kernel oil are produced annually. In addition, there are more than 500 palm oil mills that are currently operating in Malaysia producing crude palm oil (Yusoff et al.,2021a). However, the growth in palm oil sector will result in significant amounts of POME being produced, potentially posing a severe threat to the environment. Raw POME contains high concentration of oil and grease which make it dangerous if directly discharge into the environment. Hence, the residual oil in POME must be removed in order to avoid accumulation of oil in the water surface which will further cause the treatment process much more difficult (Hameed et al.,2003). Conventional biological treatment such as aerobic and anaerobic process on the other hand requires proper maintenance, and may release large amount of methane gas which are corrosive and odorous (Yusoff et al., 2021a). Oil recovery from POME through solvent extraction method can reduce the accumulation of oil in wastewater as well as having higher recovery efficiency. Moreover, the oil recovered from POME has the potential to be use as a feedstock in biodiesel production. As the global energy demand keep increasing, the utilization of oil recovered from POME as substrate in biodiesel production could reduce the dependency on fossil fuels-based diesel. Further study is conducted to determine the best organic solvent and its optimum condition for higher oil extraction efficiency.

1.4 Research Objectives

The objectives of this research are:

i. To determine the best solvent and optimum operating condition for higher yield of oil recovery from POME.
ii. To characterize the quality of oil recovered.
iii. To investigate the feasibility of using the recovered oil to produce biodiesel.

CHAPTER 2

LITERATURE REVIEW

Previous chapter discussed about the potential of palm oil mill effluent as biodiesel feedstock as well as the commercialization of biodiesel. Chapter 2 provides previous discoveries and reviews available from credible scientific researchers' records and references that are related to this final year project topic. This chapter covers the overview of the available pre-treatment method for palm oil mill effluent (POME) and its characteristics. Besides, this chapter also presents important references and information required for the production of biodiesel.

2.1 Palm Oil Mill Effluent (POME) in Malaysia

Palm oil can be derived from oil palm tree, *Elaeis guineensis,* which is originally from West Africa. The oil palm tree usually can be found growing in the wild tropical rainforest region. In 1875, palm oil was first introduced to Malaya as an ornamental plant and the first cultivation of oil palm tree began in 1917. Over the last few decades, palm oil industry has grown tremendously, becoming one of the most important agriculture-based industry in Malaysia. According to Malaysian Palm Oil Council (MPOC), in 2020, the oil palm tree plantation covered for about 18% of the total land area in Malaysia, producing 19.14 million tonnes of palm oil and 2.20 tonnes of palm kernel oil. Palm oil also have become the country's major export revenue earning at a value of RM 73.25 billion, contributing for about 3.5% of Malaysia's total gross domestic product (GDP). Figure 2.1 shows the percentage of export value of palm oil worldwide for a total of 51.042 million tonnes in year 2020 .

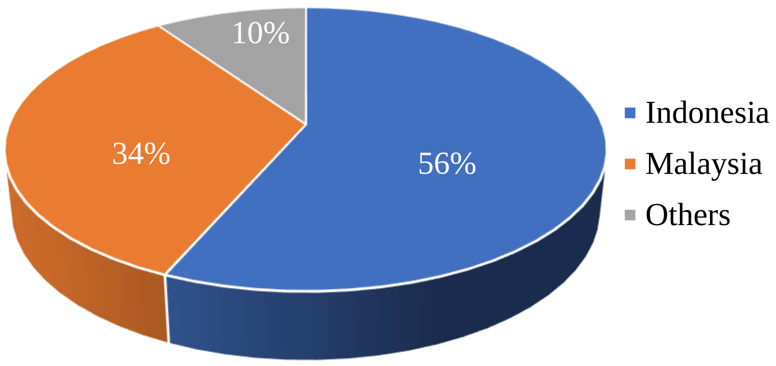

Figure 2.1: Export value of palm oil worldwide in 2020: 51.042 million tonnes (source: MPOC)

Malaysia is one of the largest producers and exporter of palm oil worldwide, accounting for 25.8% and 34.3% of the world's palm oil production and export, respectively. There are more than 500 palm oil mills that are currently operating in Malaysia due to increasing demand of palm oil. There are two type of products that can be obtained from oil palm fruit which are palm oil and palm kernel oil. Palm oil is produced from the fruit mesocarp, while palm kernel oil is extracted from the seed. Palm oil is widely been used in various industries, including production of food, biofuel and oleochemicals.

Yusoff et al.,(2021a) stated that in palm oil mill processing, only 10 wt% of palm fresh fruit bunch (FFB) is converted to palm oil, whereas the remaining 90 wt% is released as waste in the form of sludge, palm fatty acid distillate, and palm oil mill effluent (POME). According to Hameed et al.(2003), it takes around five to seven tonnes of water to produce one tonne of palm oil, with half of the consumed water ending up as POME . These waste output that keep increasing as palm oil production capacity expands could be hazardous to the environment and this problem should be addressed. As a result, one of the primary issues confronting the palm oil industry is the disposal of POME.

2.2 Palm Oil Mill Effluent (POME)

Palm oil mill effluent (POME) is a wastewater generated from palm oil mill processing. It is a colloidal suspension consisting of 95-96% of water, 0.6-0.7% of oil, and 4-5% of total solids, including suspended particles which are mostly from palm fruit mesocarp debris (Yusoff et al.,2021a). Generally, POME is produced during the manufacturing of crude palm oil from sterilization, hydrocyclone waste, and separator sludge process. The wastewater effluent from separator sludge and sterilization process has the most polluting characteristics (Poh et al., 2010). Table 2.1 shows the characteristic of POME obtained in different process of palm oil mill production.

POME also contains a large amount of oil and grease, ranging from 4000 to 6000 mg/L, which is exceptionally high when compared to the regulation threshold value of 50 mg/L for oil and grease (Primandari et al.,2013). Hence, it is vital to reduce the residual oil content in POME to the allowable limit since it can have detrimental effect on the environment, including an increase in biochemical oxygen demand (BOD) and chemical oxygen demand (COD). The oil and grease will form a film on the water body surface, reducing the oxygen content for aquatic organism (Chew et al., 2021). In Malaysia, POME was the worst source of pollution with approximately 60 million tonnes of POME produced annually (Rezania et al., 2020). With the rapid growth of the palm oil mill industry , it was necessary for the industry to treat its effluent to an acceptable level before discharging it due to its environmental effect.

Table 2.1: The characteristics of POME from sterilization, hydro cyclone and separator sludge (Poh et al., 2010)

Parameters	**Sterilization effluent (mg/L)**	**Hydrocyclone effluent (mg/L)**	**Separator sludge (mg/L)**
Biochemical Oxygen Demand (BOD)	10 000 – 25 000	-	17 000 – 35 000
Chemical Oxygen Demand (COD	30 000 – 60 000	-	40 000 – 75 000
Total Solids (TS)	40 000 – 50 000	5000 – 15 000	35 000 – 70 000
Suspended Solids (SS)	3000 – 5000	5000 – 12 000	12 000 – 18 000
Oil and Grease (O&G)	2000 - 3000	1000 - 5000	5000 – 15 000
Ammoniacal Nitrogen (NH_3 – N)	20 – 50	-	20 - 50
Total Nitrogen (TN)	350 - 600	70 – 150	500 - 900
pH	4.5 – 5.5	-	3.5 – 4.5

*All parameters are in mg/L except for pH which has no unit

Considering the risk for contamination from the palm oil sector, Malaysian government's effort to reduce the effluent from the palm oil industry has been implemented through the Environment Quality Regulations for the Palm Oil Industry in 1978. Table 2.2 summarizes the allowable discharge limit of each parameter of POME.

Table 2.2: Allowable parameters limit for disposal of POME (Yusoff et al., 2021b),(Hameed et al.,2003).

Parameters	Maximum discharged limit (mg/L)
Biochemical Oxygen Demand (BOD) at 30°C	100
Chemical Oxygen Demand (COD	1000
Total Solids (TS)	1500
Suspended Solids (SS)	400
Oil and Grease (O&G)	50
Ammoniacal Nitrogen ($NH_3 - N$)	150
Total Nitrogen (TN)	200
pH	5 - 9

2.3 Solvent Extraction

POME is made up of oils that have been lost in waste streams, with the oils often floating on top of the POME or being entrapped in the solids (Chew et al.,2021). Several technologies have been implemented in reducing the amount of oil in POME to an acceptable limit such as aerobic and anaerobic treatment. However, it is found that the residual oil in POME has potential to use as a substrate in biofuel production due to its high oil content. Studies done by Hameed et al., (2003) shows that oil can be recovered from POME through solvent extraction method.

Solvent extraction or also known as liquid-liquid extraction is the separation of a constituents from a liquid solution through contact with another liquid in which the constituents are more soluble. The constituents are transferred from one liquid to another without any changes in its chemical properties (Ahmad et al., 2003). As POME consists of organic constituents, organic solvent will be used in solvent extraction method. This is because organic component is more soluble in organic solvent making it easier to separate from inorganic component in POME. The extraction solvent used also is usually immiscible in water at the extraction condition, hence it can be easily separated from the POME and reused as solvent for another extraction process. According to Ahmad et al.,(2003), there are four basic process in solvent extraction method which are;

1. Contact between wastewater and solvent
2. Separation of extracted wastewater and solvent
3. Solvent treatment to remove extracted constituent
4. Wastewater treatment to remove residual solvent

Solvent extraction has wide range of application, especially in the chemical industries. It is generally use in industries, such as pharmaceutical, biomedical, analytical chemistry, and wastewater purification to recover pure components. The solvent used in industries has significant role in the environmental, economic performance. Hence, process industries usually used green solvents as they are more advantageous compared to organic or aqueous solvent and also due to the environmental concern (Yusoff et al., 2021b).

In the principle of separation in solvent extraction method, the separating funnel consists of two separated layers of liquids. One that is containing water with other impurities and the other, containing maximum oil and organic solvent (Lee et al., 2019). The upper layer usually will be the organic solvent as it has a lower density than water, but the opposite situation also could occur. The component equilibrium distribution between the POME and the extracting solvent, as well as the kinetics of mass transfer between the two liquid phases, will determine how effective the solvent extraction process. The extraction efficiency can be increased by increasing solvent to wastewater ratio, or utilizing multiple extraction stage (Ahmad et al., 2003).

2.4 Biodiesel

Various source of fuels, including those made from renewable feedstocks like fats and oils, have been developed as alternatives for petroleum-based fuels. Due to recent increase in petroleum prices and shortcomings of fossil fuels, the interest in biodiesel has been remarkably risen. Several types of fuels can be produced from these renewable feedstocks that contain triacylglycerol. The most prominent among these fuels is biodiesel (Knothe, G., 2010). Biodiesel is defined as mono-alkyl esters of long chain fatty acids that are commonly produced from renewable feedstocks via a process known as transesterification reaction. The reaction of involved triglycerides of vegetable oil or animal fat with alcohol, typically ethanol or methanol, in the presence of a base catalyst like potassium hydroxide or sodium hydroxide (Sarjadi et al., 2019).

In some developing countries, like Malaysia, biodiesel is one of the promising green energy in terms of its energy potential, and it continues to be the solution for climate change as well as energy sustainability, especially in its conventional forms (Bilgen et al., 2015). According to Malaysia Energy Statistics Handbook 2020 by Energy Commission, transportation sector consumed the most energy compared to other sectors such as industrial, agricultural and commercial sectors. The statistical result is illustrated in Figure 2.2.

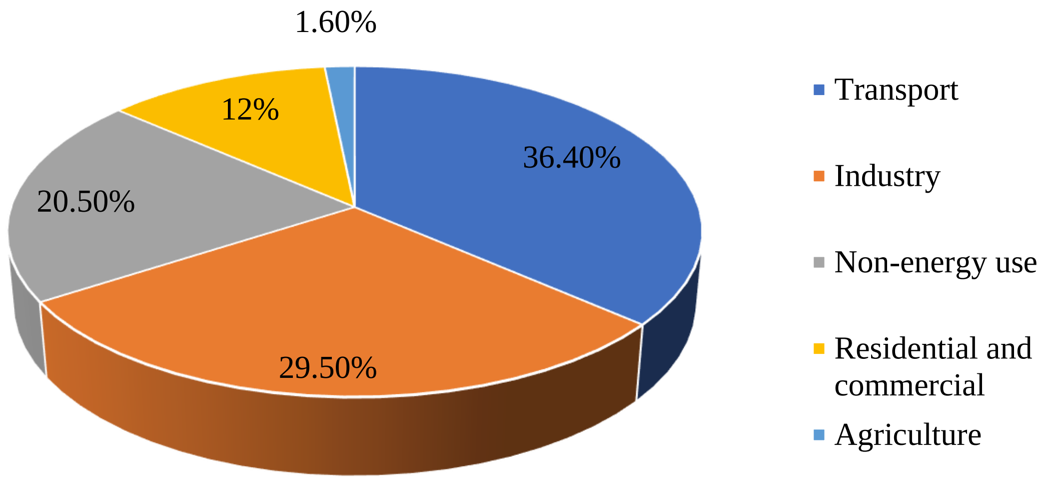

Figure 2.2: Percentage of final energy consumption by each sector in 2018 (Energy Commission, 2020)

In Malaysia, the transportation system accounted for about 36.4% of total energy use in year 2020. Despite recent increases in energy demand for Malaysia's industrial sectors, a large portion of energy consumption is still allocated to transportation sector that plays vital role in globalization and economy. The utilization of energy in transport system not only depletes non-renewable energy sources but also gives negative impacts to the environment. Statistics shows that about 13.5% of global warming is generated by transportation (Herzog et al., 2005). This phenomenon is inevitable due to the consequences of emissions of greenhouse gases from fossil fuels combustion in vehicles engine.

Since biodiesel combustion characteristics are similar as petroleum, many countries has invested in production and exportation of biodiesel, which is considered as an acceptable source of energy. Besides, biodiesel can be blended with petroleum-derived diesel at any proportion for compression ignition (CI) without any modification in the diesel engine

(Elkelawy et al.,2022). Hence, biodiesel can be a potential alternative replacing fossil fuels as it can be used in its pure form in the transportation sector. The utilization of biodiesel also can reduce the emission of carbon dioxide, CO_2 and other greenhouse gases that can lead to global warming effect.

2.5 Production of Biodiesel via Catalytic Transesterification.

Biodiesel can be derived from any vegetable oils or seed oils that are consisting of triglycerides of long chain saturated and unsaturated fatty acids through transesterification reaction. One of the primary reasons for not using vegetable oil as fuels is because of its high viscosity, typically ranging from 28-40 mm^2/s. High viscosity fuel can cause formation of deposits and injector coking due to poor atomization upon injection into the combustion chamber. Hence, transesterification of oil can reduce the oil viscosity to a range of 4-5 mm^2/s which is closer to the viscosity of petroleum-derived-diesel fuel (Knothe, G., 2010).

Transesterification is described as the important class of organic reactions in which an ester is transformed into another through interchange of the alkoxy moiety (Schuchardt et al., 1998). Transesterification is the most commonly applied technique in synthesizing biodiesel, in which alcohol and oil react with each other in the presence of catalyst. The triglyceride contain in the oil are converted into fatty acid alkyl esters, producing glycerol as the by-product which can be separated using decantation funnel. Figure 2.3 illustrates the overall chemical equation for catalytic transesterification reaction for biodiesel synthesis. Transesterification of triglycerides usually occur by heating the it with alcohol. The catalyst used in this reaction are generally categorized into homogenous and heterogeneous types.

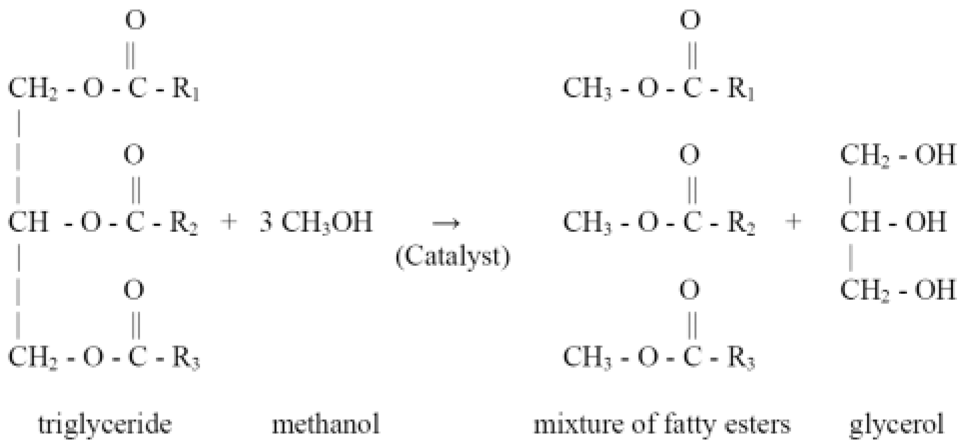

Figure 2.3: Catalytic transesterification reaction to synthesize biodiesel (Yusoff et al., 2021a)

There are two types of homogeneous catalysts in biodiesel production which are homogeneous alkali catalyst and homogeneous acid catalyst. The use of such catalyst is to enhance the productivity of the reaction. However, the utilization of homogenous catalyst can cause high amount of soap formation due to the presence of triglycerides that can contaminate the product. Therefore, synthesizing of biodiesel using homogeneous catalyst required gravity separation of glycerol and alcohol and vacuum distillation, followed by the separation of soap from the product (Yusoff et al., 2021a). Large amount wastewater are also required due to the downstream processing and purification process.

On the other hands, heterogeneous-catalysed transesterification is more advantageous compared to homogenous-homogeneous-catalysed transesterification. In transesterification reaction, heterogeneous catalyst is much easier to recover, either by using filtration or centrifugation, which making them more favourable. Moreover, there are no possibility of soap formation and the catalyst can be reused efficiently (Yusoff et al., 2021a). However, the drawback of this catalyst is that it has low catalytic activity, which will limit the mass transfer between oils and acyl acceptor.

CHAPTER 3

METHODOLOGY

This chapter presents the information on the methods applied for this final year project. It includes general research flow diagram, laboratory procedure for solvent extraction and biodiesel production, and research analysis.

3.1 Materials Preparation

Four technical grade solvents which are n-hexane, diethyl ether, petroleum ether and ethanol are used in this solvent extraction study. All of the solvents were brought and use at standard concentration. Fresh POME sample were collected from palm oil mill. The POME is stored in cold room to cool it down before being used in the experiment. For the biodiesel production study, Magnesium sulphate ($MgSO_4$) is used as the drying agent, while potassium hydroxide (KOH) is used as a catalyst in transesterification experiment. Methanol solvent is also employed in the biodiesel production study. All the organic materials were obtained in reagent grade purities and were used as received without any modification. Table 3.1 shows the list of material or reagent required and its purposes.

Table 3.1: Materials used and its purposes

Material	Purpose
N-hexane	Act as solvent for oil extraction from POME
Diethyl ether	Act as solvent for oil extraction from POME
Petroleum ether	Act as solvent for oil extraction from POME
Ethanol	Act as solvent for oil extraction from POME
Methanol	Solvent used to prepare methoxide solution
Palm Oil Mill Effluent (POME)	Sample for extraction study
Magnesium hydroxide ($MgSO_4$)	Act as drying agent to remove excess water in oil
Potassium hydroxide (KOH)	Catalyst in transesterification reaction

3.2 Research Plan

Overall, this final year project is focusing on the efficiency of oil recovery from palm oil mill effluent (POME) by solvent extraction method. Then, the recovered oil was utilized as a feedstock for biodiesel production by transesterification reaction with potassium hydroxide, KOH as a catalyst. Figure 3.1 shows the flow diagram for the research project for each of the experiment in this research. Preparation of the solution, experimental setup and sample analysis were carried out using the methodologies that was describe later.

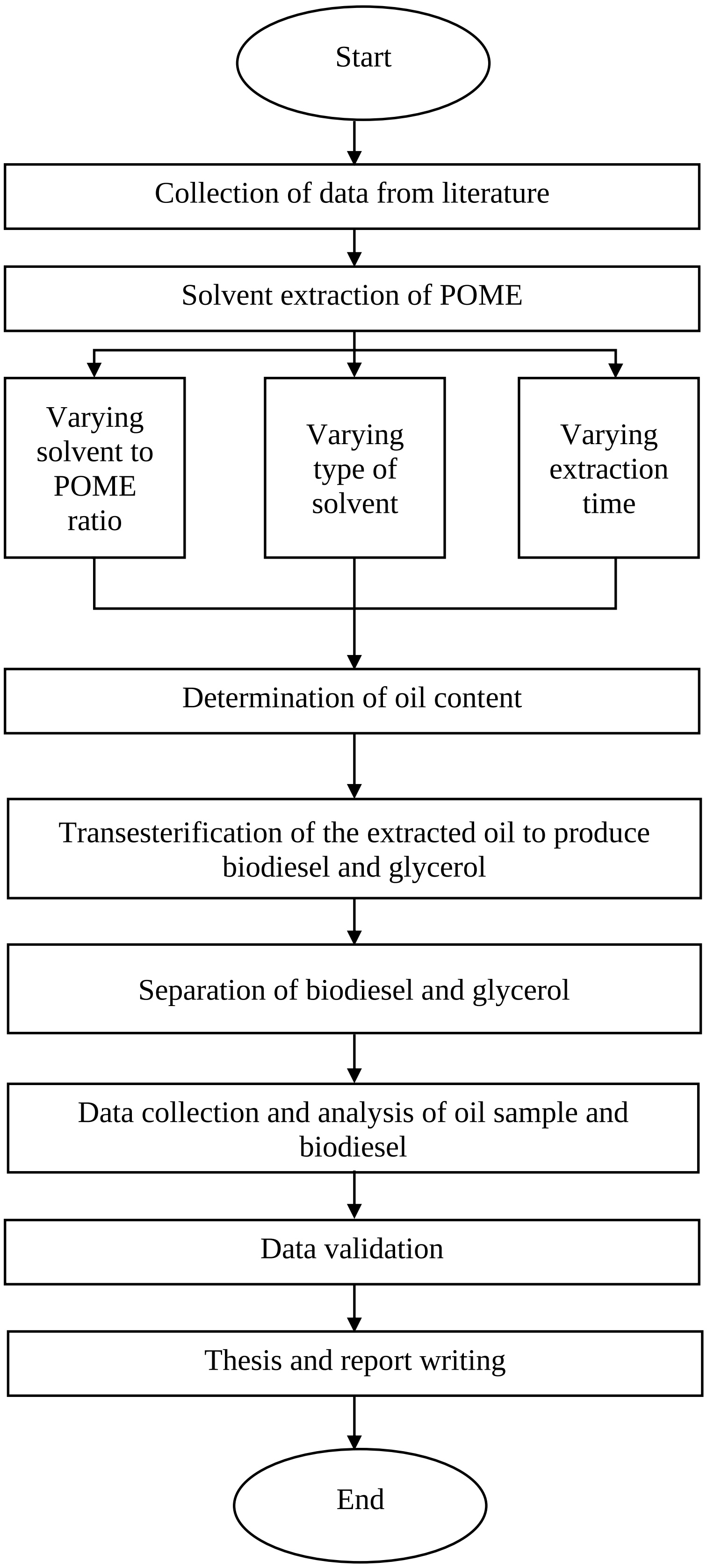

Figure 3.1: Flow diagram of the work conducted

3.3 Solvent Extraction of Palm Oil Mill Effluent (POME)

Solvent extraction or also known as liquid-liquid extraction process only required one type of solvent for the oil extraction process. 100 ml sample of POME was weighed and mixed with 50 ml of n-hexane solvent. The mixture was covered with a thin layer of aluminium wool to prevent evaporation loss of the solvent during stirring process. Then, the mixture was stirred at 200 rpm for 20 minutes. After that, the mixture was left to extract for 1 hour. The mixture was then filtered and transferred into separation funnel to allow for complete two layers separation. The upper layer consisted mixture of solvent and oil, while the lower layer is the remaining POME after extraction. Later, the contaminated solvent (upper layer) and the extracted POME (lower layer) were collected and transferred into different beakers.

3.4 Determination of oil content

The extracted POME was then transferred into another separation funnel, and the container was rinsed with 10 ml of n-hexane solvent which later was added into the separation funnel. The mixture in the separation funnel was shaken vigorously and let to settle for 5 minutes. Two complete separated layers were formed, in which the solvent layer is filtered with filter paper and collected into a clean conical flask. The extraction with 10 ml n-hexane solvent was repeated 2 times. The collected contaminated solvent was then distilled off by using rotary evaporator up to its boiling point as shown in Table 3.2. This step was required to separate the n-hexane solvent and oil. Then, the volume and weight of the extracted oil was measured. The drying process was done in drying oven at 103°C to remove the remaining moisture content in the oil. The weight of extracted oil after the drying process was measured. The percentage of extracted oil for every 100 ml of POME can be calculated as below:

$$\%Recovery\ of\ oil = \frac{Weight\ of\ oil\ extracted\ (g)}{Weight\ of\ 100\ ml\ of\ POME\ (g)} \times 100\% \qquad (3.1)$$

Similar procedures were repeated for other types of solvents, while several parameters such as extraction time, and solvent to POME ratio were varied to determine the optimum condition. Lastly, all of the oil samples obtained were sent for analysis. Figure 3.2 illustrates the experimental procedure for solvent extraction of POME.

Table 3.2: Type of solvents and its boiling point

Sample	**Solvent**	**Boiling point (°C)**
1	n-hexane	69
2	Diethyl ether	34.6
3	Petroleum ether	60
4	Ethanol	78.37

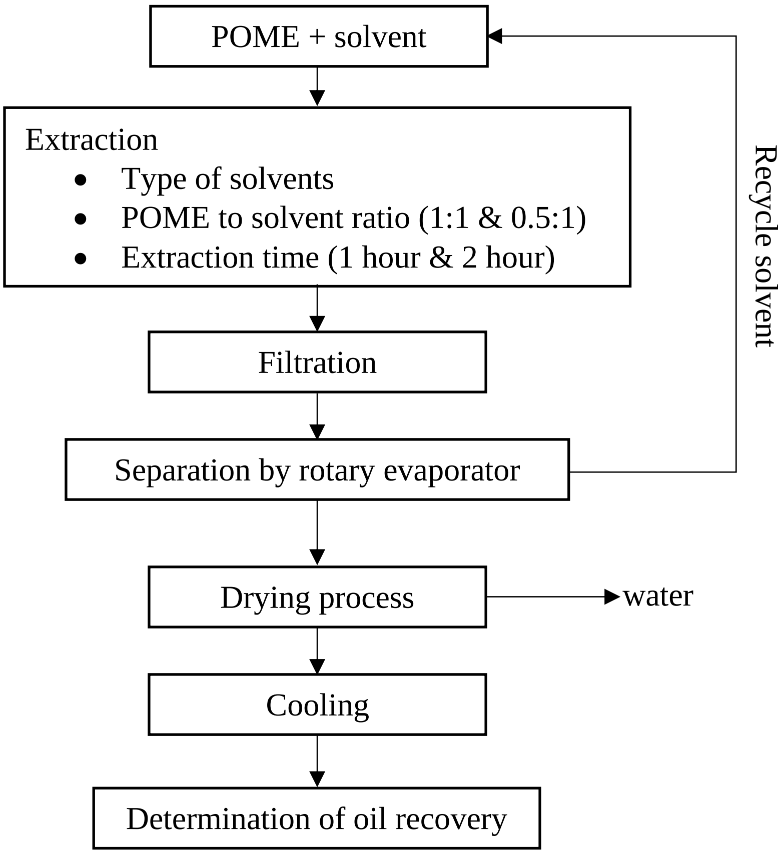

Figure 3.2: Experimental procedure for solvent extraction of POME

3.5 Production of Biodiesel from the Recovered Oil by Catalytic Transesterification Reaction

Magnesium sulphate was added to the oil to act as a drying agent removing any excess water in the oil. The oil was left for a day and the excess water is filtered out from the oil. Firstly, methoxide solution was prepared by adding 0.1 g of KOH into methanol in order to have 1% concentration of KOH for every 10 mL oil. According to research done by Ali et al. (2013), the optimum methoxide to oil ratio is 6:1 (60 mL : 10 mL) to obtain high yield of biodiesel. Then, 10 mL of oil was preheated to 60 °C and mixed with the methoxide solution. The oil and methoxide mixture were transferred into the 2- necked round-bottom glass flask for the transesterification reaction to occur. The optimum reaction time was 60 minutes with reaction temperature at 60 °C. After the transesterification process completed, the mixture was poured into the separating funnel and left to settle for 1 day forming two layers. The upper layer was the biodiesel while the lower layer was glycerol. The glycerol was separated, and the biodiesel product was evaporated with the rotary evaporator to remove excess methanol. The biodiesel was then washed with warm water for several time to remove the catalyst and by-products produced from saponification reaction. Lastly, the final product was placed in a heated oven to remove any excess water and impurities and the weight of biodiesel was measured.

3.6 Extraction of Oil using Recovered Solvent

Solvent that has been distilled off by rotary evaporator can be recovered and recycle as solvent for another extraction process. The recovered N-hexane solvent was used in this experiment. 85 ml of fresh POME was weight and mixed with 85 ml of recovered N-hexane solvent. The mixture was covered with a thin layer of aluminium wool to prevent evaporation loss of the solvent during stirring process. Then, the mixture was stirred at 200 rpm for 20 minutes. After that, the mixture was left to extract for 1 hour. The mixture was then filtered and transferred into separation funnel to allow for complete two layers separation. The upper layer consisted mixture of solvent and oil, while the lower layer is the remaining POME after extraction. Later, the contaminated solvent (upper layer) and the extracted POME (lower layer) were collected and transferred into different beakers. The collected contaminated solvent was then distilled off by using rotary evaporator up to its boiling point which is 69°C. The drying process was done in drying oven at 103°C to remove the remaining moisture content in the oil. The weight of extracted oil after the drying process was measured. Similar calculation method of percentage of oil recovery as subsection 3.4 was done. The percentage yield of oil extracted from recycled n-hexane was then compared with the percentage yield of oil extracted from fresh n-hexane solvent.

3.7 FTIR analysis of oil sample

Fourier Transform Infra-Red (FTIR) analysis test was used for all of the oil samples to characterize the oil and determine whether it is presence in the sample. The sample can be characterized using this method in order to ensure that oil was indeed present in the sample.

CHAPTER 4

RESULTS AND DISCUSSION

This chapter describes the result obtained. All the data are analysed and thoroughly discussed to meet the outlined research objectives. The result for percentage of oil recovery, effect of solvent to POME ratio, effect of extraction time and effect of different solvent on the oil extraction rate as well as the percentage of biodiesel yield were presented in this chapter.

4.1 The effect of type of solvents on the oil yield

By using four different types of solvents for solvent extraction of POME, the amount of oil yield obtained from each sample are analysed. Figure 4.1 shows the effect of different type of solvent, which are n-hexane, diethyl ether, petroleum ether and ethanol on the amount of oil recovered from POME.

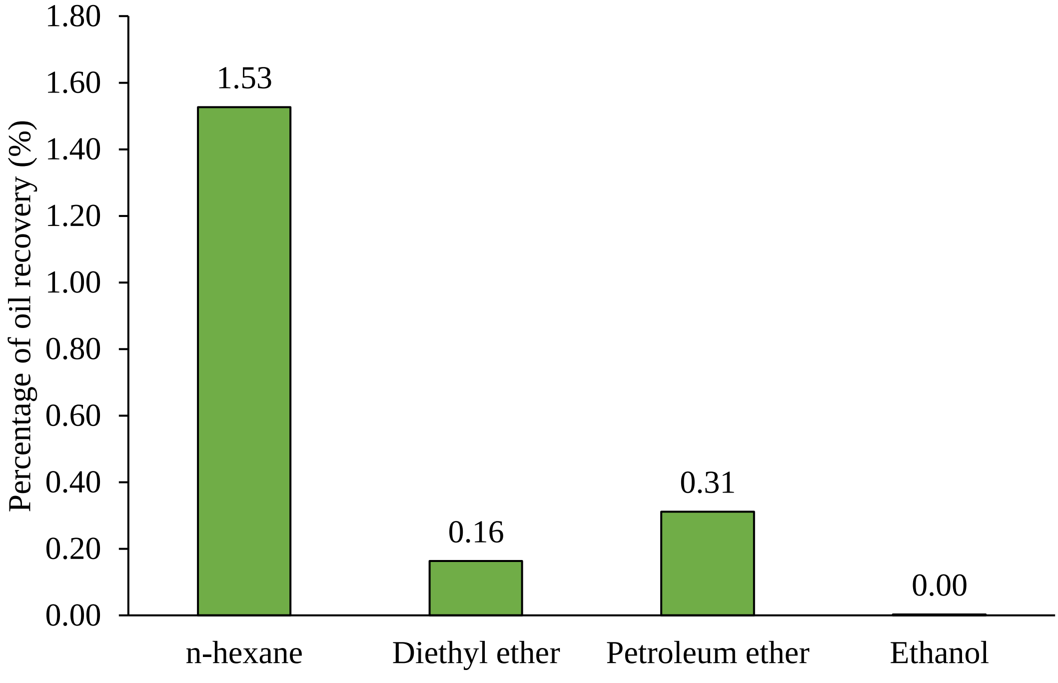

Figure 4.1: Effect of solvent types on the recovery of oil within 1 hour extraction time

In order to determine the most optimum solvent for higher yield of oil extraction, other parameters such as mixing rate, extraction time and solvent to POME ratio was kept constant.

The different in the amount of oil rendement for all of the solvents can be seen clearly from Figure 4.1, in which n-hexane gives the highest yield of oil recovery followed by petroleum ether, diethyl ether and ethanol. Solvent extraction of POME with n-hexane as the solvent gives for about 1.53% of oil recovery for every 100 ml of POME which is the highest amount compared to other solvent that only able to reach below 1% of oil yield. This is proven from the previous study conducted by Ahmad et al.,(2003) that n-hexane is the best organic solvent to extract oil from POME by solvent extraction method. Recent research done by Nuryanti et al. (2019) on solvent extraction of POME also state that n-hexane is the most suitable solvents to be used as it gives highest yield of oil recovery, which is 81.07%. at extraction time of 3 days with solvent to POME ratio at 1:1.

On the other hands, extraction of oil from POME using ethanol gives the lowest yield which is 0.003% of oil recovery. This is probably due to ethanol being a polar solvent, while n-hexane, petroleum ether and diethyl ether are non-polar solvent. According to Nuryanti et al. (2019), polar solvent tends to dissolve in water, in this case wastewater (POME). This will contribute to the difficulty in separating the oil from POME as the ethanol solvent is completely dissolved in the POME. Moreover, ethanol also is hard to recover for reuse purposes. Meanwhile, non-polar solvent such as n-hexane, diethyl ether, and petroleum ether are immiscible with water. Hence, non-polar solvents are capable of dissolving into oil without being mixed with water, thus making it easier to recover for recycle (Nuryanti et al. 2019). The use of non-polar solvent also contributes to lower the overall cost of oil extraction process, making n-hexane as the most suitable solvent for extraction process due to its higher non-polar characteristics compared to other solvents.

4.2 The effect of solvent to POME ratio on the oil yield

Another parameter that can be varied in determining the optimum condition of oil extraction is solvent to POME ratio. The solvent to POME ratio was varied between 0.5:1, 1:1, and 1.5:1. Meanwhile, other parameter such as mixing rate, and extraction time was kept constant at 200 rpm and 1 hour, respectively. Figure 4.2 shows the effect of solvent to POME ratio on the amount of oil extracted during solvent extraction process. From Figure 4.2, it can be clearly seen that the high oil yield was obtained using n-hexane solvent at a ratio of 1:1 (100 ml solvent : 100 ml POME) and at ratio of 1.5:1 (150 ml solvent : 100 ml POME)

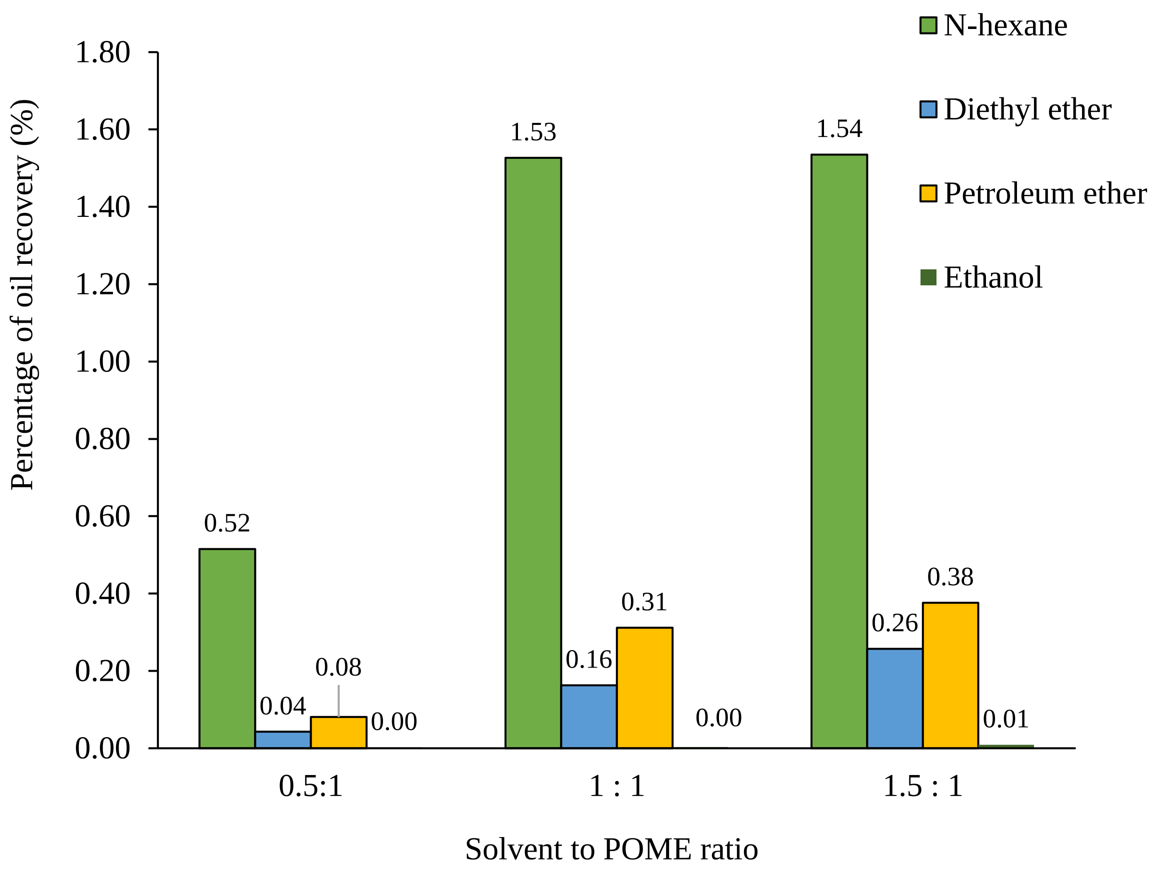

Figure 4.2: Effect of solvent to POME ratio on the recovery of oil within 1 hour extraction time