# IN-SITU TRANSESTERIFICATION OF MICRO ALGAE TO FATTY ACID METHYL ESTER (FAME): PROCESS CONTROL AND OPTIMIZATION STUDY USING ASPEN PLUS SIMULATION

by

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Thesis submitted in partial fulfilment of the requirement for

Degree Bachelor of Chemical Engineering

2021

## ACKNOWLEDGEMENTS

The accomplishment of this study would not have been possible without the voluntary or involuntary participation and help of a lot of individuals. Their contributions and sincerity are much appreciated and thanked, even though their names are not included. However, a specific statement of thanks is in necessary to convey my heartfelt gratitude and indebtedness to the following individuals.

First and foremost, I want to express my gratitude to Allah SWT, the highest authority, for He is the one who is continuously guiding and blessing me as I go ahead. It is because of His never-ending grace that I am able to persevere and eventually get where I am meant to be. Then there is my wonderful family, who are always willing to provide a helping hand or lend a sympathetic ear when I need it. In the effort of finishing this project, no one has been more essential to me than you. Thank you for always being there for me and supporting me no matter what. I would not have made it this far without their unwavering love and support.

Next, I do want to express my thanks to my supervisor, Professor Ir. Dr. Zainal Ahmad, who is not only nice, but whose assistance in offering top-notch professional advice, stimulating recommendations, and positive encouragement has aided me in pursuing this project in the first place. In addition, I do want to express my gratitude to my best friends for their assistance in sharing information and expertise, as well as for continually motivating me to stay hopeful in reaching my objectives. Thank you very much

Mohamad Zainul Wafak Bin Makin

June 2022

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## ABSTRAK

Simulasi transesterifikasi in-situ alga mikro kepada asid lemak metil ester (FAME) telah dibentangkan dalam kajian ini. Simulasi dijalankan untuk mengkaji kesan masa tindak balas, suhu reaktor, nisbah refluks dan nisbah molar biojisim-metanol ke atas ketulenan dan kadar alir FAME dan Gliserol dalam operasi unit yang berbeza seperti Reaktor Tangki Kacau Berterusan (CSTR), Unit Pemulihan Metanol (MRU) dan Unit Pemulihan Gliserol (GRC). Model lengkap loji pengesteran telah digunakan untuk mensimulasikan hasil dan pengoptimuman prestasi. Simulasi telah dijalankan menggunakan Aspen Plus V10. Untuk keadaan optimum pada 64.8216 °C, ketulenan dan kadar alir untuk FAME dalam CSTR ialah 0.007118007 dan 7.8193233 kmol/jam. Di samping itu, pada keadaan nisbah refluks optimum 10.9, ketulenan FAME dalam MRU ialah 0.6630. Selain daripada itu, ketulenan gliserol dalam GRC ialah 0.8547 untuk nisbah refluks ideal 1.8999. Selain itu, teknik talaan Cohen-Coon, telah dipilih sebagai kaedah talaan pengawal dalam kajian ini. Secara keseluruhan, CSTR dan Pemisah Fasa 1 mempunyai pengawal yang lebih mantap kerana ia mempunyai ralat peratusan keluaran pengawal yang lebih rendah. Sementara itu, pengawal MRU dan GRC lebih agresif kerana ia mempunyai ralat peratusan keluaran pengawal yang tinggi.

# IN-SITU TRANSESTERIFICATION OF MICRO ALGAE TO FATTY ACID METHYL ESTER (FAME): PROCESS CONTROL AND OPTIMIZATION STUDY USING ASPEN PLUS SIMULATION


## ABSTRACT

Simulated in-situ transesterification of micro algae to fatty acid methyl ester (FAME) has been presented in this study. The simulation was carried out to study the effect of reaction time, reactor temperature, reflux ratio and the molar ratio of biomass-methanol on purity and flowrate of FAME and Glycerol in different unit operation such as Continuous Stirred Tank Reactor (CSTR), Methanol Recovery Unit (MRU) and Glycerol Recovery Column (GRC). A complete model of the esterification plant was used to simulate the result and optimization of the performance. The simulation was conducted using Aspen Plus V10. For optimal conditions at 64.8216 °C, the purity and the flowrate for FAME in CSTR are 0.007118007 and 7.8193233 kmol/hr, respectively. In addition, at optimal reflux ratio conditions of 10.9, the purity of FAME in MRU is 0.6630. Other than that, the purity of glycerol in the GRC is 0.8547 for an ideal reflux ratio of 1.8999. In addition, the Cohen-Coon tuning technique, was chosen as the controllers tuning method in this study. In overall, CSTR and Phase Separator 1 have more robust controllers since it has lower controller output percentage errors. Meanwhile, MRU and GRC controllers are more aggressive since it has high controller output percentage error.

# CHAPTER 1: INTRODUCTION

## 1.1 Introduction

This research will be focusing on the in-situ transesterification of micro algae to fatty acid methyl ester (FAME). In this case, micro algae are the biomass feedstock or reactant for the esterification process. Aspen plus simulation will be used to simulate the dynamics of the process. There are several parameters to be varied in order to observe the effect on the esterification process. The simulation will be validated using various statistical analysis method.

## 1.2 Background

Increased awareness of energy demands and requirements for minimising global climate change has resulted from the world's growing population and the significance of preventing global warming. In recent years, biofuel, a renewable and environmentally friendly fuel produced as an alternative to fossil fuels, has gained popularity. Renewable biofuels (transport fuels generated from biomass feedstock) are predicted to grow quickly over the next several decades on a worldwide scale. The first-generation biofuel, biodiesel, is made from a range of renewable lipids, including raw vegetable oils, waste vegetable oils, animal fats, and non-edible oils. Biodiesel has identical physicochemical qualities to diesel made from crude oil and may be used in diesel engines directly.

Table 1.1 Common raw materials used for biodiesel production and their oil yield(Oh et al., 2012)

| Oilseeds | Oil yield (tonne/ha/yr) |
|---|---|
| Palm Oil (Malaysia) | 3.93 |
| Rapeseed (EU) | 1.33 |
| Soybean (USA) | 0.46 |
| Sunflower (Argentina) | 0.66 |
| Jatropha (India) | 1.44 |
| Coconut (Philippines) | 0.66 |

The cost of biodiesel production is influenced by the kind and availability of feedstock, production process, additives utilised, and operating expenses. Among these numerous aspects, raw materials account for the majority of the cost of producing biodiesel. The availability (regional production and productivity), cost, and oil qualities all play a role in raw material selection (stability and cold flow properties). Countries like the United States of America (USA) and those in the European Union (EU) are self-sufficient in edible oil production and even have a surplus to export. As in Table 1.1, edible oils like soybean and rapeseed are extensively used to make biodiesel in the United States and Europe, respectively. Leading Asian nations like Malaysia and the Philippines, on the other hand, use edible oils like palm oil and coconut oil to make biodiesel. However, due to limited edible oil production, the major raw material used in India is Jatropha curcas (Jatropha), a non-edible and underutilised feedstock(Oh *et al.*, 2012).

Transesterification, the process of converting vegetable oils into compounds with more technically suitable fuel characteristics, has gained a lot of traction in recent years. Transesterification is a critical step in the biodiesel manufacturing process because it lowers the viscosity of the feedstock/vegetable oils to a level comparable to standard fossil-based diesel oil(Mumtaz *et al.*, 2017).

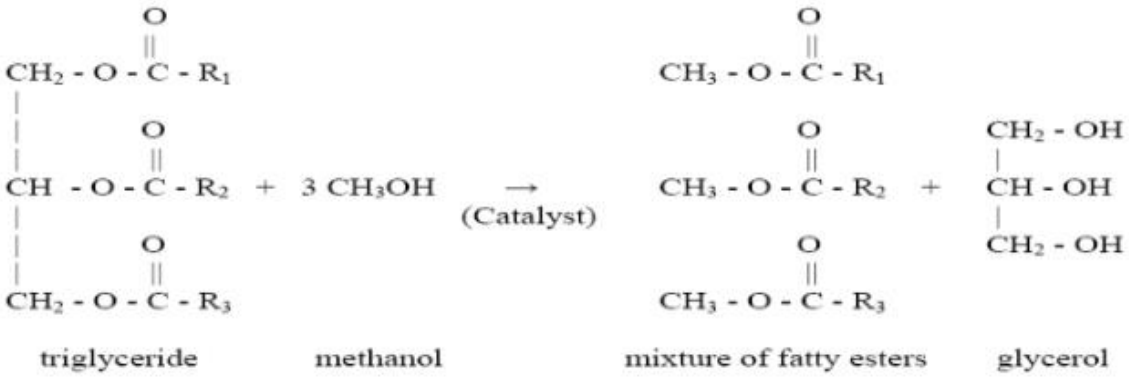


Figure 1.1 Transesterification process(Mumtaz *et al.*, 2017)

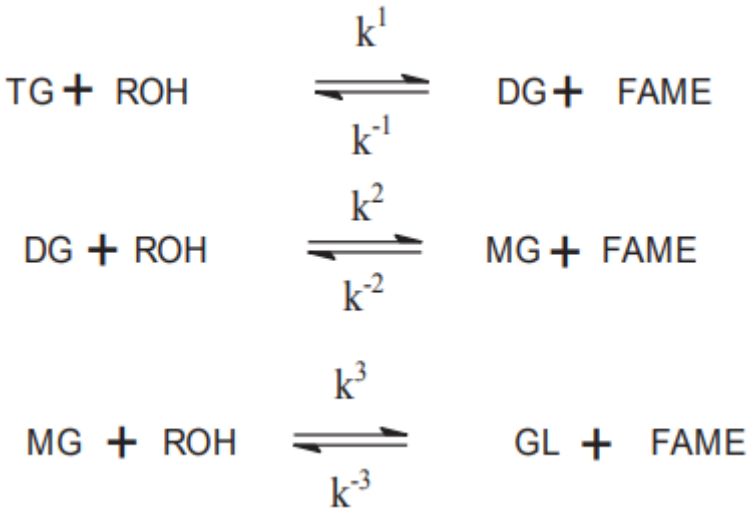


Figure 1.2 Overall transesterification process(Mumtaz *et al.*, 2017)

As shown in Figure 1.1, transesterification is a set of chemical processes in which the alkoxy moiety is exchanged, resulting in the transformation of one ester into another. Transesterification is an equilibrium reaction that describes the alcoholysis of carboxylic esters and is generally carried out in the presence of a standard catalyst (e.g., NaOH or KOH) to speed up the equilibrium adjustment and produce larger ester yields.

Vegetable oils are triglyceride molecules that vary structurally in their glycerol-bound alkyl moiety. In the presence of a suitable catalyst, transesterification of these triglyceride

molecules with short-chain alcohols produces fatty acid methyl esters and glycerol; a series of three reversible processes shows the whole transesterification process as outlined in Figure 1.2 (Mumtaz *et al.*, 2017).

Microalgae, on the other hand, are unicellular eukaryotes that can thrive in any habitat on the planet and provide many advantages such as high biomass output, rapid growth rate, and high photosynthetic efficiency. It also produces the most oil compared to other conventional terrestrial crops (10,000 litres of oil per acre of land). As a result, algal biomass is being considered as a possible fuel for biodiesel synthesis (Kelani and Ahmad, 2020). Therefore, Aspen simulations are performed to simulate the effect of parameters such as feed flow rate, and the molar ratio of alcohol to oil on the energy consumed and biodiesel yield.

Despite the benefits of microalgal oil as a biodiesel feedstock, algal biodiesel is still in the laboratory owing to technological obstacles that must be addressed before it can be made economically and sustainably. The extra methanol to oil molar ratio required by this manufacturing process makes in situ transesterification more water tolerant. The necessity to remove unreacted methanol (>94%) from product streams, on the other hand, necessitates a higher distillation heat load, which raises the operational cost (Salam, Velasquez-Orta and Harvey, 2016). However, using these crops for fuel would need a large amount of freshwater and arable land. This would result in unfair competition between the use of these resources for energy crops, agriculture, and home use. The growing cost of food oil crops has been substantially affected by their usage as energy crops in Europe and America. High prices may benefit farmers, but they may result in food shortages in many developing nations, particularly in those where food accounts for over half of their incomes. It may also contribute to global food insecurity. Food and fuel competition for land can have detrimental environmental consequences (Mitchell, 2008). Palm oil has recently been cultivated in large areas of

rainforests, notably in Malaysia, Indonesia, and Thailand, since it is in great demand for food and fuel (Salam, Velasquez-Orta and Harvey, 2016). This has resulted in deforestation, which has a negative impact on forest ecosystems. Nonedible feedstocks that need marginal land and little freshwater are ideal for supplying raw materials for large-scale biodiesel synthesis in a sustainable manner. Waste oils and microalgae, particularly marine organisms, are examples of such feedstocks. Despite the fact that microalgae have a short growing time, high lipid productivity, and can be used to capture concentrated $CO_2$, they can be grown on non-arable land using wastewater and are adaptable to harsh environments, these advantages have yet to be translated into algal biodiesel commercialization. The issue is that a variety of constraints must be solved before algae biodiesel can become a commercial reality.

## 1.3 Problem Statement

Due to its competitive combustion efficiency and low sulphur emissions, microalgae-based biodiesel production has attracted a lot of attention as a viable alternative source that may successfully meet energy requirements. Many micro-algal species can accumulate up to 50%–70% (w/w) lipid in dry biomass under ideal culture circumstances (Chisti, 2007). The absence of process technologies has hindered the commercialization of algal biodiesel. The main barriers to scaling up and developing an industrial level microalgae biodiesel process are high processing costs and a large amount of energy (Levine, Bollas and Savage, 2013). Biodiesel is a mixture of fatty acid alkyl esters made via a transesterification reaction with a strong acid or alkali as a catalyst. This traditional method has various disadvantages, including the need for a catalyst, high energy consumption, high production costs, and a complex purification and separation procedure.

Other than that, the rigid cell walls construction and tiny size of the algal cells cause extracting oil from algal biomass remains a key problem in the whole process (Johnson and Wen, 2009). Due to the reduced effectiveness of the traditional extraction method, additional physical and chemical pre-treatment methods were integrated into the extraction operation, which added to the costs without increasing the extraction efficiency significantly. These procedures use a lot of solvents like hexane, chloroform, and methanol, which are bad for your health and the environment (Cheng *et al.*, 2011).

Simple feedback control is the most frequent control method in commercial production plants. In most cases, single input single output (SISO) control loops are used, and loop interaction is frequently ignored. The operation of the transesterification reactors is fraught with challenges. The frequent overshoot of reactor temperature and fluctuation of its internal pressure is one of the issues connected with the operation of these reactors (Mjalli and Hussain, 2009). Furthermore, variance in reactor conversion measurement is a common issue. This is mostly due to the practical challenges experienced in online measuring and the usage of time-consuming and costly offline analysis. The temperature of the reactant feed has a direct impact on the reaction kinetics, reactor temperature, and pressure. In addition, incorrect mixing and temperature fluctuations in the cooling jacket are considered reactor disturbances (Mjalli *et al.*, 2009).

Understanding the complicated reaction kinetic process involved in transesterification reactor modelling is the first step. Freedman and colleagues started working on chemical kinetics for biodiesel generation in the early 1980s. Because only one overall reaction was included in their kinetic model, it was of limited use. Chemical processes and heat transfer properties in biodiesel transesterification reactors are quite complicated. Because of the strong nonlinearity in the dynamics of this reactor, a process control method that can handle the variation of operating areas is required. Nonlinearity in biodiesel transesterification reactors

can be caused by differences in reactant concentration, temperature, coolant temperature, ambient temperature, instrumentation noise, or miscalibration. (Mjalli *et al.*, 2009).

For the purpose of this research, the information on the chemical components involved in the process was obtained from the Aspen PLUS data bases. In-situ transesterification is a single-step process that combines oil extraction with triglyceride transesterification to produce biodiesel. As a result, this approach minimises the number of unit operations, such as extraction and purification, as well as the amount of equipment needed, the amount of solvent used, and the amount of energy used. Aspen Plus Dynamic will be applied as it is able to gain a solid understanding of the processes' unique dynamics and possible outcomes. It may be used to improve plant safety, operability, and productivity throughout design and operation. Aside from that, it has the potential to reduce capital and operational expenditures. The conversion percentage of micro algae to fatty acid methyl ester (FAME) also would be observed by changing some manipulated variables along with the energy consumed in this process.

## 1.4 Research Objectives

This research project aims to achieve the following objectives:

i. To carry out the sensitivity analysis for the in-situ reactor in the production of the biodiesel plant
ii. To apply optimisation analysis for the in-situ reactor in the production of the biodiesel
iii. To implement the process control strategy to control plant using Aspen Plus Dynamic

## 1.5 Scope of the Thesis

The scope of the study for this project is to solve the problem stated above in the problem statement through simulation. The simulation of the steady state in-situ transesterification process was taken from literature (Ahmed *et al.*, 2020). The process simulation used dry microalgal biomass as a sample of the feedstock in the process.

The project will be focusing on selective analysis, optimization of the purity of FAME and the dynamic of the transesterification process along with the evaluation of the conversion of micro algae. The simulation of this process is a study of the dynamic model in the system and the variable that effecting the conversion yield of micro algae and flowrate of the FAME.

The dynamic model and the process flow will be simulated using Aspen Plus V10 software which was granted access from the license provided by Universiti Sains Malaysia (USM). The software provides a user-friendly interface with pre-programmed data available where users can practice and do the simulation.

## 1.6 Sustainable Development Goals (SDGs)

The Sustainable Development Goals (SDGs), often referred to as the Global Goals, were established by the United Nations in 2015 as a global call to action to eliminate poverty, conserve natural resources, and ensure that by the year 2030, peace and prosperity would be experienced by everyone. The 17 SDGs acknowledge that development must balance social, economic, and environmental sustainability and that actions in one area will have an impact on results in others. The creativity, know-how, technology, and financial resources of all of society are necessary to achieve the SDGs in every context.

One of the sustainable development goals (SDGs) is affordable and clean energy which is related to this research. Clean energy is applied where biodiesel is a clean-burning liquid fuel

developed from renewable energy sources like vegetable oils and animal fats, which can be used to power vehicles in place of diesel. Goal 7 of the SDGs seeks to address this enormous imbalance by ensuring that everyone has access to cost-effective, dependable, and modern energy services. Enhancing energy efficiency and making investments in renewable energy is essential for increasing energy availability. That aims in line with the content and the conclusions of this research.

# CHAPTER 2: LITERATURE REVIEW

## 2.1 Introduction

In the previous chapter, to combat climate change, the world must look for alternative energy, low-carbon energy and fuel sources. Biodiesel is one of the promising options for non-renewable energy which has drawn a lot of attention recently. Biodiesel can be extracted from animal fat or plant oil. In Chapter 2, all the previous discoveries and reviews available are presented from credible scientific records and references that are related to this final year project topic. This chapter covers the overview of the biofuel which act as renewable energy, the environmental impact of the esterification process, the potential of micro algae as a feedstock for biofuel, the transesterification process and the process dynamics of the process itself.

## 2.2 Biofuel

Day by day, the world's energy consumption is growing, thereby leading to an increase in pollution which further escalates the issue of global warming. To cope with the energy needs and at the same time minimise pollution, the development of sustainable alternative energy sources has become the key priority. Many nations are focusing on utilising diverse alternatives including solar energy, geothermal, wind, hydropower, thermal or photovoltaic, and biofuels. Every alternative normally comes with its own advantages and downsides, and the growth of optimal and realistic alternatives with time is the desired answer. Among the biofuels, Second-generation biofuels (biodiesel, bioethanol, and biogas) provide key alternatives and may be generated from sustainable resources accessible, with decreased or virtually no emissions on their burning. Biodiesel may be created from non-edible oils, waste cooking oil, waste grease, or animal fats, while bioethanol and biogas can be made from agricultural waste (wheat straw, maize cobs, etc.) and other sustainable resources. The availability of these resources, costly processing, and production cost cannot satisfy the present supply and demand of energy needs

in the most effective way. Biofuel production from microalgae which falls under third-generation biofuels has now become an important research subject. Advantages like simple culture, non-competition with the food supply chain, greater lipid 6 content, and less processing are acquired based on the utilisation of microalgae which help in the overall reduction of biofuel production cost (Jacob-Lopes, 2018).

## 2.3 Environmental Impact

The removal of catalyst residues, residual TG, free glycerine, and other undesirable products soluble in biodiesel is another issue that plagues biodiesel production. To achieve environmental sustainability, proper removal and treatment of these contaminants are critical. Based on Karaosmanoglus et al (Karaosmanoğlu *et al.*, 1996) research washing with hot distilled water, washing with water and petroleum ether, and neutralisation with sulphuric acid are three biodiesel refining techniques (1:1). On the basis of biodiesel purity and refining expenses, the results reveal that washing with distilled hot water at 50℃ is the optimal refining procedure.

Separation of unreacted MeOH, washing with citric acid and water solutions, and final drying are all steps in the purification of the methyl ester (biodiesel) phase. The unreacted MeOH is recycled back to the transesterification unit before the methyl ester washing process. The soap that forms and the water that remains after the residual GLY is washed are recycled back to the GLY purifying unit. The separated wet MeOH is delivered to the rectification unit at the end of the methyl ester drying section to collect the surplus MeOH for future transesterification. Though the water consumption for the complete production line was not specified, waste water is created from the biodiesel plant as a result of the water washing refining stage. This has resulted in a cost disadvantage in biodiesel manufacturing. Enhancing

the refining process and creating circumstances that preserve excellent product quality while also ensuring environmental quality requires careful consideration.

## 2.4 Micro Algae Potential

Microalgae are photosynthesising cells that convert carbon dioxide into biofuels, meals, feeds, and high-value bioactive. The rise in the world's population and predictions of a protein shortage in the early 1950s prompted research for new alternative and unconventional protein sources. Algal biomass looked to be a viable choice for this purpose at the time(Spolaore *et al.*, 2006). Biological lipids are one sort of alternative fuel that is being considered. This fuel, known as biodiesel, is made by converting glycerolipids into methyl or ethyl esters of fatty acids, as well as glycerol as a byproduct, in a simple transesterification process.

Microalgae strains can generate up to 60% of their total cellular mass in the form of lipid. It's also worth noting that many microalgae thrive in salty water, which isn't suited for agricultural irrigation. Furthermore, many microalgal strains can withstand extensive temperature swings and strong light intensities. As a result, the southwestern United States' arid regions, which contain huge reserves of saline groundwater, might be exploited for microalgal mass cultivation. Because these places are unsuitable for conventional agriculture, competing for land uses would be limited(Roessler *et al.*, 1994).

Microalgae appear to be the only biodiesel source with the ability to replace fossil fuel. Microalgae, unlike other oil crops, develop extraordinarily quickly and many are particularly oil-rich. Within 24 hours, microalgae often quadruple their biomass. During exponential growth, biomass doubling durations can be as quick as 3.5 hours. Microalgae can have an oil concentration of up to 80% by weight of dry biomass(Metting and Pyne, 1986). Microalgae create a wide range of lipids, hydrocarbons, and other complex oils, depending on the species(Banerjee *et al.*, 2002). Although not all algal oils are suited for biodiesel production,

suitable oils are plentiful. The production of food, fodder and other items produced from crops will not be harmed by the use of microalgae to make biodiesel.

## 2.5 Transesterification

There is a two-stage process for transesterification. The solvent extraction method is used to extract triacylglycerides (TAG) from the lipid-rich microalgae in the first stage. Solvents such as chloroform, methanol, hexane, or isopropanol are commonly used for extraction, either solely or in groups. The TAG is converted into fatty acid methyl ester (FAME) in the presence of a monohydroxy alcohol (such as methanol) and a catalyst (an alkali or acid) in the second stage, with glycerol as a by-product.

Though this two-stage system is widely known and readily scalable, it has several disadvantages. It creates vast volumes of solvent-contaminated effluent causing major recycling challenges. Moreover, handling and storage of the enormous quantities of solvents becomes tiresome and might cause environmental as well as health problems. The one-pot synthesis and recovery of FAME (single-stage transesterification process) is an alternative strategy for biodiesel manufacturing from biomass in a single phase (Ghosh, Banerjee and Das, 2017).

Many commercial companies have offered comprehensive, inventive, and cutting-edge transesterification technology to continuously manufacture high-quality biodiesel at a competitive price. Building a sustainable biodiesel business will require further efforts in biodiesel research and development, as well as the development of a reliable technology to generate, refine, and reclaim the value of the end products. It is critical to evaluate both traditional and modern improvements in biodiesel production technology in order to fully comprehend the evolution of biodiesel production technology.

A batch, stirred tank reactor is the most typical device for manufacturing biodiesel through the transesterification process. Because transesterification is a reversible reaction, it is difficult to accomplish full conversion and high yield in a single step without eliminating reaction products like FAME and GLY. The usage of continuous stirred tank reactors (CSTRs) has recently become a common batch process modification. A biodiesel system with two reactors in sequence, where the glycerine is extracted from the first reactor before feeding to the second, delivers greater product purity and lower running costs than a single reactor, according to research (Kapilakarn and Peugtong, 2007). The volume of the CSTRs can be varied to provide for a longer residence time for the first CSTR, allowing it to function at a higher reactant concentration and reaction rate, and therefore accomplish a greater degree of reaction.

## 2.6 In-situ Transesterification

In situ transesterification is the procedure where the extraction and transesterification reactions are carried out concurrently. It has an advantage over conventional approaches as just a single step is needed instead of two distinct phases of extraction and reaction. This type of combination leads to intensification since it requires a minimum quantity of solvent, reduced reaction time, and simple separation of the products. The condition of biomass is significant in this strategy since a larger quantity of biodiesel is generated from dry biomass as compared to wet-dry biomass. (Jacob-Lopes, 2018)

The conventional energy-consuming process in a crop-based biodiesel plant where the crude oil is extracted from an oil-crop feedstock (e.g., soybean) using an organic solvent (usually hexane) and then fed to the reactor for transesterification. Although hexane is efficient in oil extraction and alkali-catalysed transesterification, which is beneficial in high speed and mild heating conditions (60°C), three energy-intensive operations, namely hexane vacuum

evaporation, process heating, and stirring during transesterification, are involved in these two-step process (Ayorinde *et al.*, 1988). In a normal biodiesel production process, stir-ring may not be as expensive as process heating or methanol recovery. However, no stirring or a better method of stirring should be considered as a potential process alternative cost-cutting method (Ehimen, Sun and Carrington, 2010). The reason for these costly operations is that, on the one hand, hexane is methanol-immiscible and must be removed from crude oil before adding methanol for transesterification; on the other hand, methanol is oil-immiscible and requires stirring during transesterification to homogenise the reactants. If a "super-agent" that can act like hexane while also being miscible with methanol and oil could be discovered, evaporation, heating, and even stirring would be unnecessary. Combining oil extraction with transesterification in one stage, or "in situ transesterification," with the help of a "super-agent," would simplify the process and lower operational expenses significantly (Xu and Mi, 2011).

Refined oil from vegetables or other oil seeds such as canola, rapeseed, or soymeal is required for two-step transesterification. The refined oil feedstock accounts for up to 88%of the overall cost of a two-step biodiesel manufacturer (Haas *et al.*, 2006). Controlling the water concentration of the feedstock, catalyst, or methanol is also crucial during two-step transesterification, especially for alkali-catalyzed processes. In most cases, the highest water content in oil that may be tolerated is 0.3 weight percent. Beyond this point, saponification of the oil to soap may occur, reducing biodiesel output and complicating product separation (Freedman *et al.*, 1984). Furthermore, two-step biodiesel manufacturing requires energy-intensive and time-consuming hexane extraction stages. The hexane extraction and drying phases can account for up to 90% of the total process energy (Lardon *et al.*, 2009). A reactive extraction (also known as "in situ transesterification") might be utilised instead. The biomass is fed directly into the reaction system in this procedure. The extraction, biomass pre-treatment, and degumming procedures are all eliminated.