SYNTHESIS OF BIODIESEL FROM *Cocos nucifera* COPRA OIL, AN AGRICULTURAL RESIDUE

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FACULTY OF SCIENCE
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KUALA LUMPUR

2018

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DISSERTATION SUBMITTED IN FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE

INSTITUTE OF BIOLOGICAL SCIENCES
FACULTY OF SCIENCE
UNIVERSITY OF MALAYA
KUALA LUMPUR

2018

UNIVERSITY OF MALAYA

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Name of Degree: Master of Science

Title of Project Paper/Research Report/Dissertation/Thesis ("this Work"):

SYNTHESIS OF BIODIESEL FROM *Cocos nucifera* COPRA OIL, AN AGRICULTURAL RESIDUE

Field of Study:

Environmental Science and Management

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SYNTHESIS OF BIODIESEL FROM *Cocos nucifera* COPRA OIL, AN AGRICULTURAL RESIDUE

ABSTRACT

The residue, especially from the agricultural industry has the potential of being used as a value-added product such as biofuel, pharmaceutical compound and compost fertilizer. In addition, utilization of agricultural residue could reduce the amount of waste being sent to the landfill and contributes toward environmental sustainability. Therefore, this study will emphasize on the potential of matured *Cocos nucifera* copra residue as an alternative bioresource for second generation of biodiesel. The study was initiated by determining the optimum oil yield that can be extracted from copra residue by using three different techniques; Soxhlet extraction (SXE), ultrasonic-assisted extraction (UAE), and microwave-assisted extraction (MAE). In addition, the volume (mL) of hexane as solvent and extraction time (min (minute) / hour (hrs)) are also recorded. The results depicted that the copra residue oil (CRO) extracted using SXE (48 hrs/ 400 mL) produced the highest yield, compared to UAE (30 min/ 300 mL) and MAE (15 min/ 100 mL) which are 81.39%, 75.80%, and 62.97%, respectively. Then, the experiments were continued by analysing the free fatty acids (FFA) by using gas chromatography-mass spectrometer (GCMS), as the FFA content exceeding 5% would cause saponification during the transesterification process, which will affect the conversion of CRO to biodiesel. The results of the analysis showed UAE contained the lowest FFA concentration (0.18 - 0.27%) compared to SXE and UAE. Finally, transesterification is performed using methanol and potassium hydroxide (KOH) as a catalyst for converting CRO to biodiesel. The transesterification processes were designed using adaptive neuro-fuzzy inference system (ANFIS) and response surface methodology (RSM) to optimize biodiesel yields by comparing different methods (conventional and in-situ) and techniques (stirring hot plate-assisted (HT), ultrasonic-assisted (UT), and microwave-assisted (MT)). The

transesterification results indicated that the highest biodiesel yield was synthesised using conventional method with HT technique of transesterification (3:1, methanol to oil ratio (v/w) / 1% catalyst to oil weight (w/w)) showing 96.85% biodiesel yield. Overall, it is observed that SXE method is more efficient in extracting oil from copra residue. On the other hand, CRO extracted using MAE and UAE are more suitable to be used for biodiesel synthesis since the content of FFA are lower compared to the one extracted using SXE. In addition, conventional method with HT technique of transesterification could be a better option for biodiesel synthesis from CRO.

Keywords: Copra residue oil, biodiesel, free fatty acid, extraction, transesterification

SINTESIS BIODIESEL DARIPADA MINYAK KOPRA *Cocos nucifera*, SISA PERTANIAN

ABSTRAK

Sisa khususnya daripada industri pertanian mempunyai potensi digunakan semula sebagai produk nilai tambah seperti biobahan api, bahan farmaseutikal dan baja kompos. Tambahan pula, langkah penggunaan semula sisa pertanian mampu mengurangkan penghantaran sisa ke tapak pelupusan sampah dan menjamin kelestarian alam sekitar. Oleh itu, kajian ini akan mengfokuskan potensi sisa kopra *Cocos nucifera* yang matang sebagai sumber alternatif biodiesel generasi kedua. Kajian ini dimulakan dengan mengkaji had maksimum minyak yang boleh diekstrak daripada sisa kopra dengan menggunakan tiga teknik berbeza; Soxhlet (SXE), ultrasonik (UAE) dan gelombang mikro (MAE), serta isipadu (mL) bahan pelarut iaitu heksana dan masa ekstrak (minit (min)/ jam (j)) turut dicatat. Keputusan menunjukkan minyak sisa kopra (CRO) yang diekstrak menggunakan teknik SXE (48 j/ 400 mL) adalah yang tertinggi berbanding UAE (30 min/ 300 mL) serta MAE (15 min/ 100 mL) yang mana masing-masing 81.39%, 75.80% dan 62.97%. Kemudian, eksperimen diteruskan dengan menganalisis kandungan asid lemak bebas (FFA) menggunakan kromatografi gas-spektrometer jisim (GCMS), kerana kandungan FFA yang melebihi 5% akan menyebabkan berlakunya saponifikasi ketika proses transesterifikasi dilakukan, iaitu penukaran CRO kepada biodiesel. Keputusan analisis menunjukkan UAE mengandungi konsentrasi FFA terendah (0.18 – 0.27%) berbanding SXE yang mengandungi kepekatan FFA tertinggi dan MAE menunjukkan peratus kandungan. Akhir sekali, transesterifikasi dilakukan dengan menggunakan metanol dan kalium hidroksida (KOH) sebagai pemangkin untuk menukarkan CRO kepada biodiesel. Proses transesterifikasi ini menggunakan *adaptive neuro-fuzzy inference system* (ANFIS) dan *response surface methodology* (RSM) untuk mereka bentuk eksperimen bagi mengoptimumkan hasil biodiesel dengan

membandingkan dua kaedah (konvesional dan in-situ) dan tiga teknik (plat panas (HT), ultrasonik (UT) dan gelombang mikro (MT)) yang berbeza. Keputusan transesterifikasi menunjukkan hasil biodiesel yang tertinggi apabila kaedah konvensional dengan teknik HT (3: 1, metanol nisbah kepada minyak (v / w) / 1% pemangkin kepada berat minyak (w / w)) digunakan berbanding kaedah dan teknik-teknik yang lain, iaitu sebanyak 96.85%. Keseluruhannya, dapat diperhatikan bahawa teknik SXE adalah lebih cekap dalam mengekstrak CRO. Sebaliknya, CRO yang diekstrak dari teknik MAE dan UAE lebih sesuai untuk digunakan untuk sintesis biodiesel memandangkan kandungan FFA dalamnya lebih rendah berbanding SXE. Seterusnya, transesterifikasi yang menggunakan kaedah konvensional dengan teknik HT boleh menjadi pilihan yang lebih baik untuk sintesis biodiesel daripada CRO.

Kata kunci: Minyak sisa kopra, biodiesel, asid lemak bebas, pengekstrakan, transesterifikasi

ACKNOWLEDGEMENTS

All praises to Allah S.W.T and his faithful messenger, our prophet Muhammad PBUH as with all HIS blessings, I have successfully completed this study. Upon completion of this study, I would like to express my gratitude to many parties.

First and foremost, the most of appreciation is dedicated to my supervisor, Dr. Zul Ilham Zulkiflee Lubes as he acted as the biggest contributor to this study. He also had given me so much guidance, advice, care, attention, and also support in financial during the study.

Next, my gratitude is expressed to the ones who are always there in times of hardship or happiness of my life: my parents and family members. Their restless support and encouragement instigates the strength to finish this journey.

I owe my profound gratitude to the members of Biomass Energy Technology laboratory especially Atiqurrahman and Rabiatul Adawiyyah for their continuous assistance. Also, I want to acknowledge the members of Institute of Biological Sciences (ISB), Environmental Science and Management Programme (SPAS), Regional Centre of Expertise Central Semenanjung (RCE), University of Malaya Community and Sustainability Centre (UMCares), Tun Ahmad Zaidi Residential College (TAZRC) and all individuals who involved either directly or indirectly, for making my life more enjoyable in this university.

Last but not least, the author would also like to record his appreciation to Mybrain15 scholarship (MyMaster) from the Ministry of Higher Education (MOHE) and University of Malaya for awarding the postgraduate research grant (PG289-2016A). Indeed, only Allah can repay all your kindness.

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^{o}C	:	Degree celcius
ρ	:	Density
v	:	Volume
V	:	Kinematic viscosity
w	:	Weight
g	:	Gram
GWh	:	Giga watt hour
ha	:	Hectare
hrs	:	Hours
Ktoe	:	Kilo tons oil energy
L	:	Liter
min	:	Minute
mL	:	Milliliter
Mtoe	:	Million tons oil energy
MW	:	Mega watts
TWh	:	Terra watts hour
Adeq. precision	:	Adequate precision
Adj. MS	:	Adjusted mean of square
Adj. R^2	:	Adjusted R^2
Adj. SS	:	Adjusted sum of square
ANFIS	:	Adaptive neuro-fuzzy inference system
ANOVA	:	Analysis of variance
B5	:	5% biodiesel + 95% diesel
C. V.	:	Correlation of variance

CFPP	:	Cold filter plugging point
CN	:	Cetane number
Cor. total	:	Corrected total
CP	:	Cloud point
CRB	:	Copra residue biodiesel
CRO	:	Copra residue oil
df	:	Degree of freedom
e	:	Error
FA	:	Fatty acid
FAME	:	Fatty acid methyl ester
FFA	:	Free fatty acid
FIS	:	Fuzzy inference system
GC	:	Gas chromatography
GCFID	:	Gas chromatography-flame ionization detector
GCMS	:	Gas chromatography-mass spectrometer
GHG	:	Greenhouse gases
HHV	:	Higher heating value
HT	:	Stirring hot-plate-assisted transesterification
HTC	:	Stirring hot-plate-assisted conventional transesterification
HTI	:	Stirring hot-plate-assisted *in situ* transesterification
KOH	:	Potassium hydroxide
MAE	:	Microwave-assisted extraction
MS	:	Mean of square
MT	:	Microwave-assisted transesterification
MTC	:	Microwave-assisted conventional transesterification
MTI	:	Microwave-assisted *in situ* transesterification

Pred. R^2	:	Predicted R^2
RMSE	:	Root mean square deviation
RSM	:	Response surface methodology
SD	:	Sustainable development
SDGs	:	Sustainable development goals
SEM	:	Standard error of mean
SMEs	:	Small and medium enterprises
SS	:	Sum of square
SSE	:	Sum of square error
Std	:	Standard deviation
SXE	:	Soxhlet extraction
UAE	:	Ultrasonic-assisted extraction
UT	:	Ultrasonic-assisted transesterification
UTC	:	Ultrasonic-assisted conventional transesterification
UTI	:	Ultrasonic-assisted *in situ* transesterification

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CHAPTER 1: INTRODUCTION

1.1 Background

Nowadays, the production of energy is constrained, which could be seen through the decline in fossil fuel reserve and fluctuating price each year (Feria *et al*., 2011). Without any doubt, the utilization of fossil fuel also contributes to emissions of greenhouse gasses (GHG), the main contributor towards global warming (How *et al*., 2012). Therefore, finding the sources of new energy becomes more important than before. By 2030, Malaysians' demand for energy will rapidly increase to almost 100 million tons of oil equivalent and there is an urgent need to explore and increase the utilization of renewable energy such as biodiesel (Shuit *et al*., 2009). Biodiesel is a type of fuel that is green and environmental-friendly in comparison to petroleum diesel fuels and it is a type of renewable energy that can be regenerated in a short period of time. Furthermore, biodiesel is believed to be one of the keys to accomplish the Sustainable Development Goals (SDGs) related to goal 7, goal 12, goal 13, and goal 15 (Nussbaum, 2011). Thus, the selection of suitable feedstock for biodiesel production is important to make sure that it is really sustainable and natural biomass such as *Cocos nucifera* (coconut) can be considered as a potential feedstock for biodiesel (Cheng & Zhu, 2009).

Cocos nucifera or coconut is derived from the Aracacea family, the subfamily of cocoidea. The coconut oil can be categorized in two types: virgin and refined. The virgin oil is produced from fresh fruit (ripe) while refined oil is extracted from dry coconut named copra (DebMandal & Mandal, 2011). *Cocos nucifera* oil, which is extracted from copra can be used for many purposes such as cooking oil, hair oil, and lamp oil (Solangi & Iqbal, 2011). The coconut oil yield contains high triacylglycerol composition. The lauric acid makes up the most it, followed by myristic acid, and linoleic acid (Pontoh, 2016; Kumar & Krishna, 2015). These components are essential in biodiesel production.

However, biodiesel is commonly produced from vegetable oil such as rapeseed, corn, soybeans and palm oil and mostly these resources are largely used in food production for human; thus, it is not suitable to cope with the high demand for a better fuel source (Karmakar *et al*., 2010). Therefore, it has become a challenge for scientists to find new sources that can be utilized as a feedstock without conflicting food security. Hence, the involvement of agricultural waste or by-products in the biodiesel utilization becomes essential (Ashnani *et al*., 2014).

Agricultural waste is a type of waste that is produced from agricultural activities. According to the United States Environmental Protection Agency (USEPA), agricultural waste is the by-products generated by the rearing of animals and the production and harvest of crops or trees (Ashnani *et al*., 2014). One of the problems faced by the local authority who handles waste management is the increase of agricultural waste from time to time (Foo & Hameed, 2010). This problem might be caused by the increase in demand of food due to urbanization. The problem can be overcome by having a proper waste management system such as converting waste to energy to decrease agricultural waste that is being disposed to the landfill (Ashnani *et al*., 2014). Hence, this study will emphasize on the potential of agricultural waste, *Cocos nucifera* (coconut) copra to be synthesized as renewable energy sources, biodiesel.

1.2 Problem Statement

In this era of globalization, the high demand for progress has made the population to be highly dependent on energy to continue with their daily lives. As defined by the laws of physics, energy can neither be created nor destroyed; only converted into other forms but the world is still on the verge of an energy crisis. The world consumption for energy gradually increases since 1990 with the values reaching 12,806 Mtoe in 2012 (Ashnani *et al*., 2014). This high consumption rate in turn increases the depletion rate of the raw

material used for energy generation such as fossil fuels and coal (Bonassa *et al*., 2017. Because of this, more and more nations are redirecting their focus towards renewable energy sources to supplement their growing energy needs. Renewable sources are diverse and are easily found across the globe such as biomass, which can be used as feedstock for biodiesel (Demirbas *et al*., 2009; Karmarkar *et al*., 2010). Biodiesel is a sustainable and eco-friendly substitute for fossil diesel.

The agricultural industry without any doubt has been one of the major contributors to economic growth in the world (Kamal-Chaoui & Robert, 2009). Each year, about 998 million tons of agricultural waste generated globally. Meanwhile, in Malaysia, 12 million tons are disposed into landfills annually. In 2009, about 15% of the waste generation in Asia was contributed by agro waste, by which Malaysia generated about 0.122 (kg/cap/day) and it was expected to reach 0.210 (kg/cap/day) by 2025 (Agamuthu, 2009).

The agricultural waste generation might consist of various types of waste but mostly, this waste is composed of organic matter, which is highly potential to being converted into value added products such as biodiesel (Agamuthu & Fauziah, 2011).

Cocos nucifera (coconut), which is consumed by Malaysians at approximately 17.3 kilograms per year between 2011 until 2015, has a high potential to be utilized as renewable energy sources, biodiesel (Malaysia Department of Statistic, 2016).

1.3 Research Objectives

The main aim of this study is to evaluate the potential of copra residue in production of oil and to determine the possibility of using copra residue oil for biodiesel production.

i) To enhance the copra residue oil yield using different techniques of extraction.

ii) To enhance copra residue biodiesel yield using different methods and techniques.

iii) To optimize the copra residue biodiesel yield using adaptive neuro-fuzzy inference system (ANFIS) and Response Surface Methodology (RSM).

1.4 Scope of Work

This study will focus on *Cocos nucifera* (coconut) copra oil extraction enhancement by using different techniques and parameters. Then, the study will identify whether the oil could be potentially utilized as biodiesel by doing transesterification experiment. Next, free fatty acid analysis will be completed to find which techniques extracted the lowest FFA compound to avoid problems arising such as saponification reaction during conversion of oil into biodiesel. Furthermore, optimization of biodiesel yield production will be done by employing not only different methods and techniques, but also using artificial intelligence software: ANFIS and RSM. Last but not least, the biodiesel will undergo gas-chromatography analysis to identify fatty acid methyl ester (FAME) since the composition will determine the quality of the produced biodiesel.

1.5 Dissertation Outline

The dissertation is organized into five chapters: introduction, literature review, research methodology, results and discussion, and conclusion with future study recommendation.

The first chapter is a general introduction that gives a brief explanation regarding this study, which will emphasize on the recap of study literature, objectives, problem statement, and dissertation structure.

Next, chapter 2 focuses on a literature review of the past studies related such as environmental sustainability, waste generation, renewable energy, coconut production, and so on.

The third chapter will cover materials used and study the methodology for oil extraction using different techniques namely Soxhlet extraction, ultrasonic-assisted extraction, and microwave-assisted extraction. This chapter will also discuss the findings on biodiesel conversion and optimization from copra residue oil by employing transesterification operation.

Furthermore, the fourth chapter will emphasize on the conversion process of copra residue oil into biodiesel by conducting transesterification reaction. This chapter will include the enhancement of biodiesel yield by employing different methods (conventional and in-situ) and techniques namely stirring hot-plate-assisted, ultrasonic-assisted, and microwave-assisted. Next, it will discuss the potential of employment of ANFIS and RSM in optimizing the biodiesel yield.

Last but not least, the fifth chapter will summarize the findings of the study and a few recommendations for future research will be proposed in the chapter.

CHAPTER 2: LITERATURE REVIEW

2.1 Sustainable Development

2.1.1 Background

Nowadays, people are able to get information faster and this trend foster fast changes in community. This lead to environmental issue that could affect the ecological, economical, and social without any proper solution (Nussbaum, 2011). Hence, there is a need of a solution in solving the problems such as adopting sustainable development concept to protect the environment.

Sustainability can be translated as a process of maintaining productivity either natural or man-made products by substituting greater resources or similar value without disturbing or affecting the natural system (Shaker, 2015). Next, the concept of development is designed to restore and conserve the environment that is adversely affected by ecological exploration by humans and at the same time caused by socio-political thinking that cares about community development (Sachs, 2015). In addition, there is a debate among scholars on the environment polluted by the existence of the industry, saying that this problem is a unique feature of the industrial process (Kahle & Gurel-Atel, 2014; Blewitt, 2015). Nonetheless, it is undeniable that humans contribute to the destruction of the environment by exploring the widespread ecosystems, chemicals from industries that are emitted directly to the environment, and lifestyle that ignores cleanliness and environmental health (Nussbaum, 2011).

Sustainable development is referred to as a matter of solving problems faced by human beings in relation to social, political, and economical fields. Sustainable science refers to studies related to sustainable development and environmental science (Blewitt, 2015). According to Brundtland Report, sustainable development can be defined as "development that meets the needs of the present without compromising the ability of

future generations to meet their own needs" (Magee *et al*., 2013). This definition, do not explicitly explain about either the development or environment, and is used as a general term. Through this definition, many parties focus on sustainable development that involves the development of intergeneration. Furthermore, sustainable development focuses on the responsibility of the current generation in restoring, maintaining, and enhancing natural resources for the next generation (Blewitt, 2015).

Sustainability can be translated as a process of maintaining productivity either natural or man-made products, by substituting for greater resources or similar value without disturbing or affecting the natural system (Zelenika & Pearce, 2014). Sustainable development is also referred to as the task of solving the problems faced by human beings in relation to social, political, and economical fields (Blewitt, 2015). Sustainable science refers to studies related to sustainable development and environmental science (Nussbaum, 2011). Furthermore, sustainable development focuses on the responsibility of the current generation in restoring, maintaining, and enhancing natural resources for the next generation.

2.1.2 Sustainable Development Goals (SDGs)

The Sustainable Development Goals (SDGs) are a collection of 17 goals that have been endorsed by the United Nation (United Nation, 2015). Those expansive targets would be interrelated; however, each needs its own focus to attain. The SDGs as illustrated in Figure 2.1 cover a wide run of social and financial advancement issues such as poverty, education, climate change, environment, and so on (Griggs *et al*., 2013). The SDGs, which are known as Plan 2030, was created to supplant the Millenium Development Goals (MDGs) that finished in 2015 (Anger, 2010; Sachs, 2012). In fact, unlike MDGs, the SDGs do not differentiate between developed and developing nations but apply to all nations.

Figure 2.1: The list of 17 Sustainable Development Goals (SDGs) (Source: United Nation, 2015)

(a) ***Goal 1: No Poverty***

Since 1990, poverty has been cut by more than half but more than 1 in 5 people live their life with less than $1.25 per day. That target may not be satisfactory for human subsistence, in any case. It may be essential to raise income figure to as high as $5 per day (Fan & Polman, 2014). Poverty is more than the need of wage or assets. Individuals live in poverty on the off chance that they need fundamental services such as healthcare and education. They also encounter starvation, social segregation, and prohibition from choice making forms. Sexual orientation imbalance plays an expansive part in propagating poverty and its dangers. Accomplishing goal 1 is hampered by developing disparity, progressively delicate statehood, and the impacts of climate change (Le Blanc, 2015).

(b) ***Goal 2: Zero Hunger***

Globally, about 1 in 9 individuals are underfed, which is the endless larger part of those individuals who live in the developing countries (Fan & Polman, 2014). Agriculture is one of the biggest employers in the world and the major source of salary for destitute

country family units, giving jobs for 40% of the worldwide populace. Women make up to almost 43% of the agrarian labor constrain in creating nations and over 50% in parts of Asia and Africa but be that as it may, women claim as it was only 20% of the land owned by them (Keestra *et al*., 2016). Goal 2 targets that by 2030, we ought to end starvation and all shapes of ailing health. This would be done by multiplying rural efficiency and livelihoods of small-scale nourishment producer and guaranteeing feasible nourishment generation frameworks and continuously improve land and soil quality. Other targets bargain with keeping up seeds genetic diversity, anticipating exchange limitation, and twists in world rural markets to restrain extreme nourishment cost instability (Nilsson *et al*., 2016).

(c) Goal 3: Good Health and Well-Being

Goal 3 is to accomplish widespread health scope to incorporate fundamental medicines and vaccines. Critical strides have been made in expanding life anticipation and decreasing a few of the common reasons related with child and maternal mortality. Furthermore, advanced study has been made on clean water access and sanitation, diminishing jungle fever, tuberculosis, polio, and the spread of HIV/AIDS but as half of women in the developing countries have gotten the essential health care, the need for family planning is expanding exponentially as the populace increases. While needs are being tended to steadily, more than 225 million women were neglected for contraception (Boerma *et al*., 2015). By 2030, this goal proposes to settle down the preventable death of infants and children below 5 years old and scourges such as tuberculosis, intestinal sickness, and water-borne maladies by 2030 (Liu *et al*., 2016). In addition, health and well-being should be considered to incorporate the target related to the anticipation and treatment of substance manhandle, traffics incidents' deaths and injuries, also from hazardous chemicals, greenhouse gases emission, water pollution, and soil contamination (Schmidt *et al*., 2015; Tangcharoensathien *et al*., 2015).

(d) ***Goal 4: Quality Education***

Education, although easily and widely accessible, has been achieved today especially in primary schools. Access to education is not limited to men only; it is even open to women (Hajer *et al*., 2015). Something to realize is that this vast access is not a guarantee of the quality of education. Currently, it is estimated that over 60% of women from the world's total youth (103 million) are still lacking of knowledge such as reading skills (Griggs *et al*., 2014). Hence, the primary objective for goal 4 is to promote the balance between men and women, especially in obtaining free education and most importantly, quality.

(e) ***Goal 5: Gender Equality***

Women in terms of their involvement in various sectors such as health, education, and politics can help a country generate sustainable economic, societal, and humanitarian status. In 2014, it was recorded that a total of 143 countries pledged to secure the balance of engagement between men and women in their constitution (Nillson *et al*., 2016). Among the issues that still persecute women is exploitation as sexual tools, forced marriages, and public views that degrade them. To achieve this goal, there is a need for legislation to protect women. It should also be remembered that the involvement of women is an agent of a change rather than the recipient of the change (Sachs, 2012).

(f) ***Goal 6: Clean Water and Sanitation***

In 2017, the record shows that 4.5 billion people in the world still have not managed safe sanitation systems. Goal 6 is aimed at giving the impetus to the importance of clean water use and environmental sanitation in everyday life. To that end, the parties involved have made many things as an indicator for sanitation and sanitation such as toilets in schools and offices (Hák *et al*., 2016). This goal will also emphasize on the cleanliness

of the water especially for drinking and reducing the release of dirty water or sewage openly.

(g) Goal 7: Affordable and Clean Energy

SDGs targeted that at the end of its implementation period, access to affordable and sustainable energy use can be achieved entirely. It aims to increase the production and use of renewable energy internationally (Lu *et al*., 2015). To achieve this, the need for holistic cooperation from all countries to facilitate access to these goals is easy. If this goal is achieved, an economic spike and development will occur not only progressively but also sustainably.

(h) Goal 8: Decent Work and Economic Growth

Goal 8 estimates that at least 7% of the change in Gross Domestic Product (GDP) annually is to increase economic productivity in less developed countries. Thus, the existence of agents such as innovation, entrepreneurship, and growth of small and medium-sized enterprises (SMEs) is essential for the success of this productivity. The target is divided into two periods of 2020 and 2030 (Griggs *et al*., 2013). By 2020, targeted youth unemployment can be reduced by implementing a global strategy in creating employment opportunities for youth. Meanwhile, for the year 2030, it is targeted at providing sustainable tourism-related policies and to open new job opportunities. Furthermore, the strengthening of domestic financial institutions and increased trade assistance for developing countries is considered and referred to as a means of achieving sustainable economic growth (Grigss *et al*., 2014; Kellogg, 2017).

(i) Goal 9: Industry, Innovation and Infrastructure

According to sources, manufacturing-related industries are a major source of household income worldwide. Nevertheless, less developed countries recorded a relatively low per-capita ($100) per capita rating compared to developed countries like

Europe and North America, which recorded a revenue value of $4,621. For the record, the product manufacturing industry contributes the highest rate (80%) to the total manufacturing output and 10% in the less developed countries in the industrial economy. In terms of infrastructure, this goal expects many facilities such as cellular mobile signals to be improved especially in the remote areas or less developed countries (Lu *et al*., 2015; Kellog, 2017).

(j) Goal 10: Reduced Inequalities

Goal 10 targets to reduce the cost of exporting goods from less developed countries. In 2015, 65% of products exported from less developed countries are tax-free compared to 2005 (41%) (Griggs *et al*., 2013; Hajer *et al*., 2015). In the meantime, in case of transfers, the targeted transfers are only 3% of the charge charged to migrant workers who send money to their respective countries. However, a 6% transfer charge is charged by the company involved and 11% is imposed by commercial banks. Although there are services that charge between 2% to 4%, the service is not much (Nilsson, 2016).

(k) Goal 11: Sustainable Cities and Communities

By 2030, this goal is aimed at the wider access to safety and affordable housing. To achieve this target, the percentage of individuals living in the slums or informal settlement is used to measure. For the record, the percentage decreased from 39% (2000) to 30% (2014) (Griggs *et al*., 2013). Furthermore, some rural movements into urban areas have accelerated the process towards achieving this goal when more good alternative housing is provided (Lu *et al*., 2015).

(l) Goal 12: Responsible Consumption and Production

This goal will encourage the usage of eco-friendly products and at the same time ensuring that waste generation is reduced. The goal targets to increase participation in

recycling material and waste by 2030. In addition, companies should implement green practices, hence publishing their sustainable practicing reports (United Nation, 2015).

(m) Goal 13: Climate Change

On December 2015, the climate change issue was identified and discussed by the UN during Climate Change conference in Paris. The report summarized that in order to tackle climate change, it is not impossible if the SDGs are being complied with. In addition, the climate issue is linked to a few factors such as poverty, gender equality, and energy. Hence, the UN proposed the public sector to instigate some initiatives as to reduce negative impacts on the environment (Lu *et al*., 2015).

(n) Goal 14: Life Below Water

Seas cover 71% of the earth's surface and contain more than 200,000 species that contribute as world's biggest sources of protein. Hence, about 30% of marine habitats have been annihilated and 30% of the world's marine life is over-exploited. The sea contamination becomes more stunning as 15 tons of plastic are discharged into the seas directly each minute (Griggs *et al*., 2013). A few nations, including Kenya and different communities around the world have prohibited the use of plastic for retail buys. Progress in the seas improvement contributes to poverty diminishment of low-income families and sound of nourishment (Anger, 2010). The targets incorporate with avoiding and decreasing marine contamination and destruction, ensuring marine and coastal environments, and manageable fishing activity.

(o) Goal 15: Life on Land

The main purpose of this goal is to protect biodiversity which included forest, desert, and mountain eco-systems from further destruction. Accomplishing a "land degradation-neutral world" can be achieved by recovering corrupted forests and land lost due to drought and surge. This goal calls for more consideration to avoiding invasive species

and more assurance of endangered wildlife. Hence, "The Mountain Green Cover Index" will be used to monitor the restoring activity of biodiversity towards achieving the goal (Hák *et al*., 2016).

(p) ***Goal 16: Peace, Justice and Strong Institutions***

The goal targets to diminish savage wrongdoing, sex trafficking, forced labour, and child abuse. The UN has recognized that more women were victimized in 2017. However, casualties involving women had declined with 84% (2004) to 71% (2014). One of the biggest targets is to see sex trafficking, forced labour, and child abuse end during the implementation of the goal though accomplishing the goal might be challenging because of the dependency on reported crimes only (Kellog, 2017).

(q) ***Goal 17: Partnerships for the Goals***

The final goal (17) was established due to a problem that might arise during the implementation of the past 16 Goals. Hence, this goal was included to guarantee that nations and organizations cooperate instead of compete. Creating large stakeholder organizations to share information, expertise, innovation, and economy is seen as basic to by and large successful of the SDGs (United Nation, 2015; Le Blanc, 2015).

2.1.3 Sustainable Development in Malaysia

2.1.3.1 Adoption in Malaysia

(a) ***Ninth Malaysian Plan (9MP)***

Malaysia through its Ninth Malaysian Plan (9MP) has exposed the blueprint of government agenda for 5 years (2006-2010). This comprehensive plan will explain the distribution of budget for various sectors (Saadatian *et al*., 2012). Furthermore, Malaysia always takes the sustainable development through the 9MP seriously and it was proven that Malaysia was ranked the 38th among 146 nations and the 2nd in Asia for its effort in enforcing sustainable development. Next, Malaysia was indexed at the 9th place among

133 countries based on the endeavours taken to diminish environmental impact on human well-being and environmental assurance imperativeness (Foo, 2013). In Malaysia, there are a lot programs that have been planned by the government for environmental sustainability, but as for other countries, there are challenges in conserving the environment especially in the financial development. Hence, Malaysia recognized the sustainable development concept and implied the concept within policies, vision, mission, and plans. Moreover, Malaysia has adapted the Agenda 21 as part of the important factor for improving sustainable development implementation (Saadatian *et al*., 2009).

(b) ***Malaysia National Vision Policy (NVP)***

Malaysia's National Vision Policy (NVP), which was proposed by the government from the year 2001 to 2010, has implemented the sustainable development concept. The policies related to sustainable development are encouraging more equitable society, sustaining economic development, and pursuing environmentally protection. However, there are weaknesses in the implementation even though Malaysia has done a lot of plans relating to the sustainable development, where it is lack of comprehensive engagement and insufficient indicators for the sustainable development (Saadatian *et al*., 2012).

2.1.3.2 Malaysia Sustainable Assessment Approaches

The importance of assessing the sustainable development has been recognized by scholars and policy planners. Hence, some frameworks and mediums were created to conduct such assessment.

(a) ***Malaysia Quality of Life Index (MQLI)***

MQLI was developed by the Economic Planning Unit (EPU) under Department of Prime Minister in 1999 as a tool to assess not only the Malaysians' life quality but also sustainable development approaches. Then, it was updated in 2004. MQLI assessed the sustainable development using 14 practices, such as the quality of air, deforestation, water

cleanliness, financial, working life, transportation and communication, well-being, education, housing, environment, family, social involvement, public condition, and culture with leisure (Hassan, 2017).

(b) ***Malaysia Urban Quality of Life (MUQL)***

Next assessment, MUQL was created in 2002 by the same department as MQL. MUQL as MQLI focused on the same approaches but particularly on Malaysians who live in the urban area. The assessment was added with extra rubrics such as urban service, solid waste generation, and river quality. This assessment implied four themes, such as air, water, land, and environment itself including the inland and offshore (Saadatian *et al*., 2009; Saadatian *et al*., 2012).

(c) ***Malaysia Urban Indicator Network (MURNINet)***

MURNINet focuses on urban development towards sustainable development and this approach was developed by Federal Municipality Council. This assessment contains 11 rubrics related to sustainable development such as infrastructure, transport, environmental management, affordable housing, and so on (Foo, 2013; Hassan, 2017).

(d) ***Green Building Index (GBI)***

Green Building Index (GBI) was created to approach the assessment implied on building development. The approach targets to encourage the developers, architects, and engineers in embedding sustainable activity during the building development process. The GBI main focus is on energy saving, recycling, climate friendly, and to protect the ecosystem either at local or global levels. GBI consists of six rubrics such as energy and water efficiency, indoor quality, sustainable planning, and so on (Abidin, 2010).

2.2 Waste Generation

2.2.1 Background

In Malaysia, Municipal Solid Waste (MSW) is about 98% of total wastes which are primarily household, industrial, and commercial wastes. These wastes can be utilized to produce landfill gas (LFG) which comprises methane (CH_4), carbon dioxide (CO_2), and greenhouse gases (GHG). These gases are very efficient for power generation and can be utilized in transportation industries as well as in food industries. Currently, in Malaysia, there exist over 261 landfill sites (Ahmad *et al*., 2014; Sadeghi *et al*., 2015).

From 2001 to 2005, MSW generation increased from 16,200 to 19,100 tons in Malaysia. In other words, this increment can be expressed as an average of 0.8 kg per day for each person. This trend reflects that by 2020, the MSW generation will be increased to 30,000 tons approximately (Agamuthu *et al*., 2009; Begum *et al*., 2009; Manaf *et al*., 2009; Badgie *et al*., 2014). In minimizing waste in Malaysia, it is imperative to make necessary steps to achieve the UN Agenda 21 which is obviously related to the control of the waste management strategies. Solid waste management mainly refers to the discipline that co-ordinates and organize the disposal practice of solid wastes (Oh *et al*., 2010; Laohalidanond *et al*., 2015). Although the economic development in Malaysia is in drastic measures, solid waste management in the country is relatively poor (Moh & Abd Manaf, 2014).

In 2006, generated solid wastes in Malaysia were estimated as 7.34 million tons. According to previous study, Kuala Lumpur, with the population of 1.604 million, became the main contributor in generating solid wastes from 2620 to 3070 tons within five years from 1995 to 2000 (Sahimaa *et al*., 2015). The report also reflects that about 4000 tons of solid wastes were produced per day in the year 2000 (Kamaruddin *et al*., 2016). Increasing population, rapid urbanization, and quick industrialization process are

the main reasons for increasing solid wastes every year. Air Hitam Sanitary Landfill (AHSL) is located in Puchong for the generation of Landfill Gas (LFG) power with a capacity of 2.096 MW (Agamuthu & Fauziah, 2011; Noor *et al*., 2013; Samah *et al*., 2013; Xiang *et al*., 2014). In November 2003, construction of the project was completed and was accredited in April 2004. Area of the landfill is 58 ha that receives about 3000 tons of garbage per day from the Klang Valley. Within the distance of 30 meters from the site, a TNB substation was established which is capable to feed 2 MW of the generated power to the public grid (Xiang *et al*., 2014). On the other hand, the biggest municipal solid waste combustor system of the country was established in the State of Terengganu with a capacity of 100 tons per day and the system is adequate to supply 1.5 MW to the public grid (Suthar & Singh, 2015).

Kyoto Protocol was adopted in December 1997 at the third session of a Conference (COP 3) held in Kyoto, Japan. The main objective of the conference was to reduce the emissions of six GHGs for industrialized countries. These six types of GHG with different Global Warming Potential (GWP) are: carbon dioxide (CO_2) with GWP as 1, methane (CH_4) with GWP as 21, sulphur hexafluoride (SF_6) with GWP as 23900, nitrous oxide (N_2O) with GWP as 310, hydrofluorocarbon (HFC) with GWP as 11700, and perfluorocarbon (PFC) with GWP as 9200. The GWP is commonly expressed as the equivalent of CO_2 (Al-Jarallah & Aleisa, 2014; Sevigné-Itoiz *et al*., 2015).

The target of 1st commitment period was set by the Kyoto Protocol for 5 years starting from 2008 to 2012 with three market mechanisms towards the reduction of GHG emission. The three market mechanisms are Clean Development Mechanism (CDM), Emissions Trading (ET), and Joint Implementation (JI) under Article 12, 17, and 6 respectively. The legal documents of operational rules (Marrakesh Accords) were adopted in November 2001 at seventh session of the conference (COP7). The conference

was entitled as the United Nations Convention on Climate Change in Marrakech, Morocco, November 2001. On February 16, 2005, the Kyoto Protocol came into effect (Edjabou *et al*., 2015; Sevigné-Itoiz *et al*., 2015).

2.2.2 Agricultural

Total land area of Malaysia is 32.90 million ha where 61% of the land area is under natural forest (20.1 million ha) and 14.9% of total lands are under agriculture area (4.89 million ha). Rubber (39.67%), oil palm (34.56%), rice (12.68%), cocoa (6.75%), and coconut (6.34%) are the main agricultural crops in Malaysia (Ashnani *et al*., 2014). To reduce the carbon footprint (CO_2) and adopt sustainable agricultural practices to conserve tropical rainforests and wildlife, Malaysia is devoted to sustainable development (Mekhilef *et al*., 2011). Though, production of processing residuals in a large quantity with no economic value is one of the noticeable characteristics of forestry and agricultural sectors, the energy in solid wastes particular biomass can be deduced by direct combustion process or can be converted into more worthful and useable form of energy (Shuit *et al*., 2009).

2.2.2.1 Agricultural Industry

Malaysia's small scale industry known as Small and Medium Enterprises (SMEs) is one of the contributors for Malaysia's economic development. There are 907,605 SMEs in total as recorded by the year 2015. SMEs comprise of agriculture (1.13%), mining and quarrying (0.10%), manufacturing (5.26%), and services sectors (89.20%). In agriculture sectors, crops contribute about 67.82% compared to livestock, forestry and fisheries, with 14.01%, 6.71%, and 11.45%, respectively. In fact, it is believed that manufacturing and services sectors are also involved either directly or indirectly in using agriculture as trading products such as banana chip, fruit juices, desiccated coconut (desiccated copra), and so on (Malaysia Department of Statistic, 2017).

2.2.2.2 Agricultural Waste

Agricultural waste is produced through various farming activities which include dairy farming, horticulture, seed growing, livestock breeding, grazing land, market gardens, nursery plots, and woodlands. Agricultural waste can be both organic and non-natural wastes (Shafie *et al*., 2012). Most companies or organizations involved in the agricultural industry prefer to use the low-cost waste management methods such as land disposal and relocation. Poor waste management system leads to increment of waste from time to time especially agricultural waste and this increment will eventually give an impact to the environment (Saeed *et al*., 2008).

The increment of agricultural waste that is not properly managed can have some impacts on the environment in terms of pollution such as water pollution and air pollution. The pollution that occurs can risk human health and lead to serious and vital diseases. According to Conserve Energy Future (CEF), pollution that comes from agricultural waste is the primary source of pollution in water and lakes, as the chemicals and toxins go into the groundwater upon settling at the bottom of the water body. The accumulation of these substances leads to diseases such as blue baby syndrome and neurological ailments (Demirbas *et al*., 2009).

Recycling, reprocessing, and utilization of wastes in a positive manner can enhance the access to beneficial use of the waste compared to the traditional methods of waste disposal (Guerrero *et al*., 2018). One of the approaches that can be used in order to reduce agricultural waste production that will be sent to the landfill is by converting them to products that have a beneficial use such as biodiesel (Mekhilef *et al*., 2011; Satari & Karimi, 2018). Biodiesel can be produced through transesterification process by converting oil that has been extracted from the residue of leftover of feedstock such as copra residue of *Cocos nucifera* to usable biodiesel.

2.2.2.3 Utilization of Agricultural Waste

Generating energy from agricultural wastes are very effective because of the sustainability of bio-resources, environmental concerns, and economic reflection. Malaysia is well-known as one of the foremost agricultural countries in the world. Besides the two major agricultural resources, which are: oil palm and rubber, Malaysia has other valuable resources, such as cocoa, rice, and coconut (Ashnani *et al*., 2014). The total area of oil palm and rubber agricultural land combine over 330,000 square kilometers. So, generating energy from by-products and residuals of 362 palm-oil mills become the major target for the Government of Malaysia. As the first palm oil producer in the world, Malaysia is processing about 71.3 million tons of fresh fruit bunches per year which results in about 19 million tons of crop residuals per year consisting empty fruit bunches, fibers, and shells (Shuit *et al*., 2009; Mekhilef *et al*., 2011).

Empty fruit bunch (EFB) and Mesocarp fiber (MF) are the important resources of the oil palm biomass which is produced about 15.8 and 9.6 Million tons per year respectively and can be processed under heat and pressure to extract Molded Oil Palm (MOP). This MOP is distinctive bio-based material and exceedingly useful for furniture, electronics, buildings, packaging, and automobile products. The scientific name of Oil palm is *Elaeis guineensis*. It is the most significant species in *Elaeis* genus that belongs to Palmae family. Though the primary application of palm oil is in the food industry, it presents as a new source of raw material for biodiesel fuel. Based on the report of Malaysian Palm Oil Board, cultivating area of oil palm has increased from 4,304,914 ha to 4,487,957 ha within one year from 2007 to 2008. During the same year, total export of oil palm also increased from 19,574,242 tons to 21,750,074 tons. Other agricultural industries also produce biomass sources, such as cassava, rice husks, corns, corn straws, paddy straws, cocoa, coconut, and sugarcane residuals.

Kedah and Selangor are concerned for paddy cultivation and produced about 2128 million tons of rice in 2006. Paddy straw and rice husk are produced only for 1 to 3 times in a year during harvesting seasons which is about 1.5% of the country's energy consumption in 1996. Besides that, lands for coconut cultivation are decreased from 350,000 ha to 249,000 ha during the years from 1975 to 1995. Fronds and debris are the forms of coconut wastes which are produced by the processing and consumption of coconut fruits. During the replantation of the coconut trees, some wastes are also generated. It is also found that the land area of cocoa cultivation also decreased from 452,000 ha to 235,000 ha during the years from 1991 to 1996. Though cocoa wastes are generated from cocoa leaves and woody biomass during replanting, pruning of the cocoa plants is the main process in generating the cocoa wastes. Through the pruning process, cocoa plants are trimmed to keep the heights within 3 to 4 meters, however, the process is rejected nowadays because of detrimental effects on the plants. From the cocoa wastes, 17 million barrels of oil energy was produced in 1996 (Mekhilef *et al*., 2011; Shafie *et al*., 2012; Ashnani *et al*., 2014).

Another agricultural resource is sugarcane which is cultivated exclusively throughout the dry season, especially in the northern states of Malaysia. the area of total cultivation was increased from 18,600 ha to 20,300 ha during the years from 1976 to 1980. The increasing trend was not continued and declined to about 18,000 ha in 1997. The productions of sugarcane cultivation are granulated sugar, molasses, bagasse, dry leaves, and cane tops. Though, granulated sugar is the main product of the sugarcane cultivation, about 30% of the sugarcane cultivation produces bagasse (Goh *et al*., 2010).

2.3 Coconut (*Cocos nucifera*)

2.3.1 Background

Coconut oil is derived from kernel Cocos nucifera, a tropical plant that is actually edible oil, but is largely consumed for both edible and non-edible purposes. Cocos nucifera exists in three types: “typica” (giant variety), “nana” (dwarf variety), and hybrid variety (Fonseca *et al*., 2014). Hybrid variety produced by pollination develops certain qualities such as high oil yield, larger nut size, and pest resistance (Arlee *et al*., 2013). The giant varieties usually grow slower and will take about 6-10 years to bear fruit after planting compared to dwarf varieties which grow faster and only take about 4-5 years to grow fruit. In qualities, the giant Cocos nucifera produces better oil, copra, and fiber compared to the dwarf one. Giant variety conducts cross-pollination, while dwarf is more to self-pollination since its male and female phases are overlapping (DebMandal & Mandal, 2011). The coconut tree is abundantly planted worldwide, to which in Malaysia, about 51,126ha were planted and 427,976.4 tons were produced in 2015 (Malaysia Department of Agriculture, 2015).

Coconut tree is widely known as a multipurpose plant since every part of it can be utilized for many things; for that reason, it is known as the tree of life. For instance, the husk from the fruit can be used as brushes, mats twine, rope, and also for caulking boats. Its juice is not only for drink but can also be used for medicinal purpose. Next, its oil is usually used as cooking oil, hair oil, and lamp oil (Solangi & Iqbal, 2011).

The coconut copra usually will produce about 70% of oil. Coconut oil, like other vegetable oil and animal fats, are also triglyceride (Alamu *et al*., 2010). The Cocos nucifera oil mainly contains more than 91% of fatty acid with medium chain fatty acid being the major part. The most abundant fatty acids that can be found in the oil are lauric

acid and myristic acid, followed by linoleic acid (Pontoh, 2016; Kumar & Krishna, 2015). These components are indeed essential for utilization of biodiesel.

2.3.2 Utilization of Coconut

For years, coconuts have been served for many purposes especially for human benefits. The coconut contains a lot of good effects such as on food, human well-being, environmental sustainability, transportation, and so on. It was reported that the coconut has highly nutritional values that can be utilized for humans. For instance, the coconut water contains high vitamins, folic acid, sugar, and free of amino acids. Further, its kernel is mostly used for food substance (Solangi & Iqbal, 2011).

In terms of medicinal values, coconut with its high content of fatty acid composition has benefitted human being. For example, the medium chain saturated fatty acids (MCFA) in range of C8 to C18 will be synthesized into energy when it is consumed as food and most importantly, it will not involve transporting cholesterol. The coconut may consist of other positive effects such as antidote, antioxidant, antithrombotic, anticholecystitic, anticancer, antiviral, and many more benefits derived from the consumption of coconut (DebMandal & Mandal, 2011).

Furthermore, coconut can be used to sustain environment. In the previous study, the physicochemical content of coconut was utilized as bio-repellent to reduce insect nuisance. Further, in the Philippines, coconut is utilized as biodiesel. It is reported that coconut oil biodiesel is far better compared to petroleum diesel and it only needs small amount of coconut oil biodiesel to reduce vehicle gas emission (DebMandal & Mandal, 2011; Kumar & Krishna, 2015).

2.3.3 Production in Malaysia

In Malaysia, 653,362.3 tons of coconut were supplied annually between 2011 and 2015 contributed by both imports and production. Coconuts are utilized for exports, seed, processing (food and feed products from primary products), food (direct consumption), and waste (damaged and loss during handling and transportation). Figure 2.2 shows the average utilization of coconuts by Malaysians between year 2011 and 2015; it was depicted that more than 86% coconuts were consumed as food, exports (6.72%), seed (0.05%), processing (4.35%), and waste (2.33%) annually. In addition, during coconut processing as food products, especially for the desiccated product, there would be copra residue left unattended and regarded as waste. In fact, most of them would end up in a landfill (Malaysia Department of Statistic, 2016).

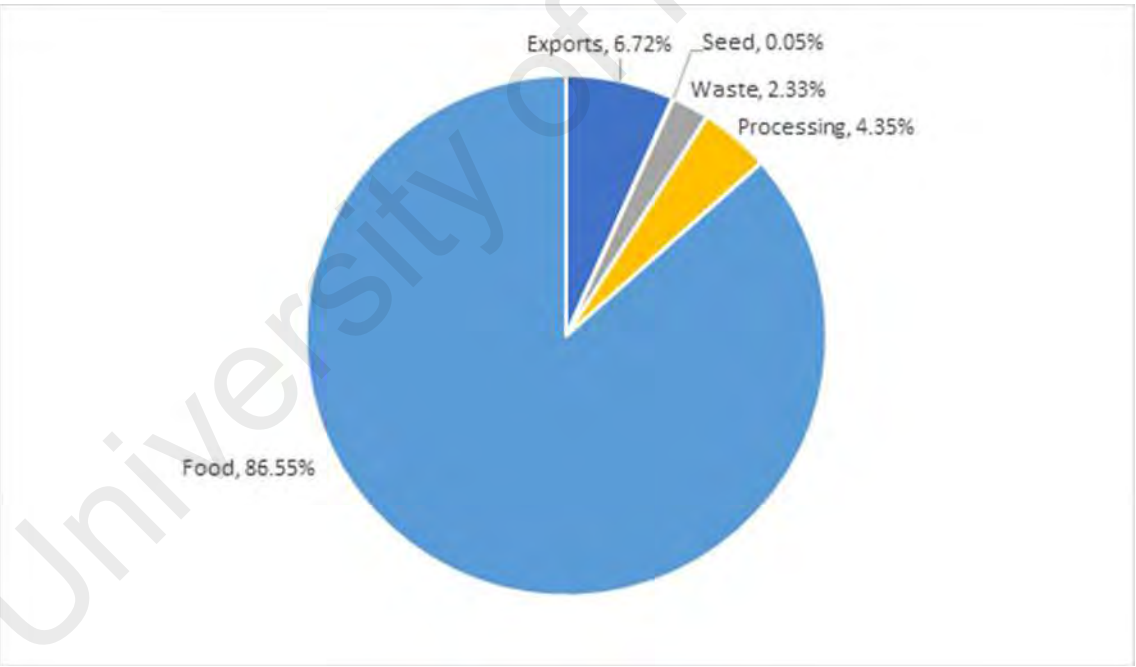


Figure 2.2: Average of coconut utilization in Malaysia between year 2011 to 2015

2.4 Biofuel as Renewable Energy

2.4.1 Background

The high dependency on perishable energy sources such as coal and fossil fuels will be a country's downfall once they are fully depleted. Because of this, more and more nations are redirecting their focus towards renewable energy sources to supplement their growing energy needs and to reduce the amount of debt they are in (Zhou & Thomson, 2009; Sorda *et al*., 2010). Renewable energy by definition refers to energy sources that are self-replenishing over time, making them able to provide the raw material required to generate and meet the energy requirements of humans. Renewable sources are diverse and are easily found across the globe; only requiring the expertise to convert them into a usable form. Sources such as solar, wind, and biomass are considered renewable as they are present in abundance and are replenished on a regular basis (Zhou & Thomson, 2009).

Biofuels are biomass materials directly used as solid fuel or converted into liquid or gaseous fuels that can be stored so that the harnessed energy can be released through combustion when needed. This chemical reaction permits to release the binding energy that holds electrons to a nucleus in the organic molecules in order to produce work and heat. In a narrower sense, biofuels may be only perceived as liquid or gas transportation fuels derived from biomass (Bonassa *et al*., 2017). Many different biomass raw materials can be used to produce biofuels including energy crops, agricultural residues, or forest products for example. Biofuels are energy carriers that store the energy derived from biomass, commonly produced from plants, animals and micro-organisms, and organic wastes (Zhou & Thomson, 2009). Biofuels may be solid, liquid, or gaseous and include all kinds of biomass and derived products used for energetic purposes. Biofuels are renewable energy sources; meaning that fresh supplies can be regrown. They are a possible substitute product for fossil fuels. Compared with the latter product, there are some advantages in subscribing to biofuels. Advantages and benefits of biofuels,

however, depend on the categorization of the specific biofuel, type of feedstock used, and the technology applied to produce them (Bonassa *et al*., 2017).

There are two global liquid transportation biofuels that might replace gasoline and diesel fuel, which are ethanol and biodiesel. Transport is one of the main energy consuming sectors. It is assumed that biodiesel is used as a petroleum diesel replacement and ethanol is used as a gasoline replacement respectively (Tye *et al*., 2011; Baumert *et al*., 2018).

Biofuels for transportation are primarily driven by government policies, world's ethanol production for transport fuel tripled between 2000 and 2007 from 17 billion to more than 52 billion litres, while biodiesel expanded eleven-fold from less than 1 billion to almost 11 billion litres. These fuels together provided 1.8% of the world's transport fuel by energy value (36 Mtoe out of a total of 2007 Mtoe). In Europe, there has been a continuing increase in the use of biofuels in road transport over the past decade from 0.1% in 1997 to 2.6% in 2007 (Sorda *et al*., 2010; Ashnani *et al*., 2014).

There are a variety of biofuels potentially available, but the main biofuels being considered globally are biodiesel and bioethanol. Bioethanol can be produced from a number of crops including sugarcane, corn (maze), wheat, and sugar beet (Tye *et al*., 2011). Biodiesel is the fuel that can be produced from straight vegetable oils, edible and non-edible, recycled waste vegetable oils, and animal fat (Baumert *et al*., 2018). The main producing countries for transport biofuels are the USA, Brazil, and the EU. Production in the United States is mostly ethanol from corn, in Brazil is ethanol from sugar cane, and in the European Union is mostly biodiesel from rapeseed (Sorda *et al*., 2010; Ashnani *et al*., 2014).

2.4.2 Biofuel Generations

First, second, and third generation biofuels are the three types which are addressed for transport based on the current or future availability, where the second and third generation biofuels are named as advanced biofuels (Zhou & Thomson, 2009).

First-generation biofuels refer to the traditional or conventional chains (Baumert *et al*., 2018). On the other hand, second-generation biofuels are not available in the market as its processing is very complicated and expensive (Tye *et al*., 2011). Third-generation biofuel is based on simple microscopic organisms that live in water and grow hydroponically which makes this type of biofuel very promising and different from the other two (Yang *et al.*, 2018). Depending on the feedstock type, efficiency of energy of a biofuel chain has to be evaluated based on two aspects, one: the net energy yield per area unit, and two: the cost of energy for transformation processes (Zhou & Thomson, 2009). Energy yield per hectare for plant biomass depends on a function of several things, such as the type of plant, soil properties, climate, and crop management. Photosynthesis is more efficient for C_4 type plants and this type of plants are energy cost-effective in humid tropical regions with unlimited water, for example, sugarcane in Brazil. In contrast, maize in the US demands extensive energy inputs (Ashnani *et al*., 2014).

(a) ***First Generation***

First-generation liquid biofuel requires a comparatively simple process for its production and usually generated from sugars, grains, or seeds. Biodiesel is another renowned first-generation biofuel which is produced from straight vegetable oils of oleaginous plants through the process of transesterification or cracking. In the process of transesterification, alkaline, acid or enzymatic catalyzers, and ethanol or methanol are used. As a byproduct, fatty acid (biodiesel) and glycerin are generated in this process (Baumert *et al*., 2018). Basically, a small fraction of plant biomass is utilized in the

biodiesel generation and left a huge fraction as residual. First-generation fuels are widely in use and produced commercially in a significant quantity in various countries with about 50 billion liters per year (Ullah *et al*., 2017). However, due to the conflict with the food supply, the viability and production of the first-generation biofuel are in question. Only a little fraction of total plant biomass will decrease the efficiency of land usages. Moreover, there involves a high production cost for the first-generation biofuels comparing with the production of food. The cost of certain crops and food products are upraised because of the rapid growth of global biofuel production from grain, sugar, and oilseed. As a result, non-edible biomass is in the interest to use for the production of biofuels. Again, biodiesel production process is not acceptable as a cost-effective emission reduction technology. Therefore, it is suggested to determine more effective and efficient alternatives for the reduction of GHG by focusing on renewable and conventional technologies (Azizi *et al*., 2017).

(b) ***Second Generation***

Second-generation biofuels are generally produced from agricultural lignocellulosic biomass which is non-edible residuals of food crop production or non-edible whole plant biomass (for example grasses or trees which are specifically cultivated for the production of energy) (Tye *et al*., 2011; Guerrero *et al*., 2018). The production process follows two fundamentally different approaches, such as biological and thermochemical processing. In terms of the production, the main advantage of second-generation biofuel over the first-generation biofuel is the usages of non-edible feedstocks that limits the direct usages of food in fuel production. The feedstock can be farmed specifically for energy purposes that enable higher production per unit land area, thus for the production of biofuels, a greater amount of aboveground plant materials can be used. Comparing with the first-generation biofuel, the above process will increase land usages efficient way. It is agreed that the primary characteristics of feedstocks show potential for significant energy and

environmental benefits of the production of second-generation biofuels. From the literature study, it can be identified that most sophisticated processing equipment are necessary for the production of second-generation biofuel. It is also necessary to invest more for per unit of production and provide larger-scale facilities in confining capital cost scale economies. Potential energy and higher economic outcome of second-generation biofuels can be achieved through extensive research and development on the feedstock production and conversion technologies (Li *et al*., 2017; Ullah *et al*., 2017; Guerrero *et al*., 2018; Lemões *et al*., 2018).

(c) ***Third Generation***

To develop the latest biofuel generation process, researchers are now focusing on microscopic organisms rather than past agricultural substrates and waste vegetable oils. Recent scientific research and technological knowledge present that third-generation biofuel is the viable alternative energy resource without having any major drawbacks comparing with the first and second-generation biofuels. The third-generation biofuel is generated from microbes and microalgae which do not need soil and land to grow (Azizi *et al*., 2017; Yang *et al*., 2018). Many of them flourish in salty, brackish, plain dirty-wastewater, or agricultural run-off. Moreover, microalgae are more productive than terrestrial fuel crops. Proteins, carbohydrates, lipids, and nucleic acids are available in all microalgae with different proportions. With the varying types of algae, the percentages of components also vary. Some algae types are comprised of fatty acids up to 40% of their overall mass. The most significant characteristic of algal oil is its yield which affects the biodiesel production. Based on some estimation results, the yield of oil from algae is more than 200 times compared with the yield from the best plant or vegetable oils per acre. Most importantly, microalgae are the types of photosynthesizing organisms which growth rate is very high and can complete the full growing cycle in few days. In each year, approximately 46 tons of oil per hectare can be produced from diatom algae. Though

different amounts of oil can be produced from a different type of algae species, it is possible to produce oil up to 50% of the weight of some types of algae (Demirbas, 2011; Shirvani *et al*., 2011; Sakhtivel *et al*., 2017; González-González *et al*., 2018).

2.4.3 Biofuel Production

Presently five types of biofuels are produced by using oil palm biomass, which are: bio-ethanol, bio-methanol, bio-briquettes, hydrogen gas, and pyrolysis oil. The production of the fermentation process of grains is the bio-ethanol fuel Grains contain sugar, such as EFB, and the process is very similar to brewing technique (Mukherjee & Sovacool, 2014). Fuels used in flexible-fuel-vehicles contains 85% of bio-ethanol where the bio-ethanol works as fuel addictive that normally cut down vehicle's carbon monoxide and other smog-causing emissions. Nowadays, fuels as a mixture of gasoline and 85% ethanol are available in Brazil, US, and European market (Timilsina & Shrestha, 2011).

For application in spark ignition engines, bio-methanol is most suitable because of its high octane rating. As the alternative fuel, the demand of bio-ethanol and bio-methanol in Malaysia is still not high because petrol is much familiar in this country. As a result, production of bio-ethanol and bio-methanol are not upgraded and technologies to commercialize the product are undeveloped even though there is a huge opportunity for the product to be commercialized (Ong *et al*., 2011).

To convert biomass into a uniform and solid fuel, briquetting process is an alternative method through which empty fruit bunches (EFB) and palm kernel expeller (PKE) are used into briquettes at high temperature and pressure by using screw extrusion technique. Several advantages are encountered with this method, which includes: cost efficiency, availability through all year round, high calorific value, longer burning duration, and environment-friendly (Chiew & Shimada, 2013).

Hydrogen is known as synthetic fuel. Different kinds of energy sources; for example, fossil fuels, nuclear energy, and biomass; are utilized to extract hydrogen. Higher engine efficiency is the main advantage of using hydrogen as fuel. Hydrogen used in Malaysia is primarily derived from electrolysis in the oleo-chemical processing industry, metal cutting, and welding works or steam reformation of natural gas (NG) in the oil, gas, and petrochemical industries. However, it is still at early stage of research for the development of improved technologies in producing hydrogen as synthesis fuel (Kalamaras & Efstathiou, 2013).

From dried biomass, a kind of tar known as Pyrolysis oil can be extracted. But, this technique is still in the preliminary stage to use as a substitute for petroleum production (Mukherjee & Sovacool, 2014).

2.4.3.1 Worldwide

Combustible renewables are mostly consumed directly about 1.8% by the transport sector, about 17.6% by industries, and about 80.6% by other sectors mainly the households. Production of heat and electricity dominated industries are using bioenergy for the application of state-of-the-art biomass combustion for generation of power. Two types of industrial sectors are the paper and pulp sector and cane-based sugar industry (Zhou & Thomson, 2009; Sorad *et al*., 2010). On the other hand, during the years from 2001 to 2006, global ethanol fuel production increased more than doubled while biodiesel expanded nearly about six times. Currently, US and Brazil are dominating the world in the production of ethanol. In 2007, the production reached to a record of 52 billion liters. From 14% of USA national maize production in 2006, near to half of the world's fuel (ethanol), demand was produced. At the same time, more than two-fifths of the world's demand was produced in Brazil from sugar cane and supported about 21% of its transport fuel consumption. The rest of the production comes from other countries, such as Spain,

France, Sweden, and Germany. Chinese ethanol from corn, wheat, and sugarcane is mainly used for industrial purposes (Ashnani *et al*., 2014).

In 2006, about 73% of all biodiesel production were supplied worldwide by Europe where rapeseed and sunflower seeds were the main raw materials of the production. Germany also was in the leading position as produced about 40% of the demand and followed by USA, France, and Italy by generating most of the rest. Europe continued to produce about 60% of global biodiesel production which was measured as 6.2 billion liters in 2007. Meanwhile, production of biodiesel has increased in every producing country, for example, the production became doubled in the USA and most of the producing countries in Asia or Oceania. Moreover, the production became more than triple in Brazil (Zhou & Thomson, 2009; Sorda *et al*., 2010; Ashnani *et al*., 2014).

In the present time, biofuels still do not represent a significant share in worldwide liquid fuel supply, in spite of the growth in biofuels consumption and comparatively slower growth of oil consumption, which can be measured as about 0.9% by volume and 0.6% by transport distance traveled. In Europe, essential biofuels are produced domestically and consumed. In Sweden, since 2004, all petrol was blended with 5% ethanol which was mainly originated from Brazil and Southern-Europe. Only 10% of biofuels produced in the world are sold internationally which indicates that the international trade in biodiesel is still low. However, trades are increasing noticeably as some regions of the world may produce large biomass feedstock (Yang *et al*., 2018).

2.4.3.2 Malaysia

The pyrolysis oil from EFB is rich in carbon, a crude oil substitute with potential raw materials for fuel and chemical production in petroleum fuels. Most recently, Genting Ceke Ltd. and Biomass Technologies (BTG) completed the first pyrolysis plant in Malaysia as a revolution in utilizing oil palm biomass in Malaysia as a source of pyrolysis

oil. In 2007, Malaysia's energy demand for transport reached to 180274 GJ per day (Chiew & Shimada, 2013).

The Malaysian government has gone through the transport sector's biodiesel to ensure a sustainable energy supply in the future. In 1982, Malaysian Palm Oil Commission (MPOB) used palm oil to produce biodiesel. In a powered vehicle, there are two ways to use palm oil as a fuel: direct use of vegetable oil on a diesel engine and convert the oil to methyl esters. By 2007, a total of 92 projects were approved with a capacity of 500,000 tons per year. However, in 2006, only 14 plants with the capacity of 47,790 tones were built, followed by 95,010 tons in 2007, and six plants were still in operation by 2008. Malaysia produced 500,000 tons of biofuels annually in 2008. The National Biofuels Policy (2005) launched a biodiesel fuel blend (B5) at the end of 2009. Presently, about 5% of cars in Malaysia use biofuels (Sumathi *et al*., 2008).

Kunak, Sabah has a bio-energy project with a capacity of 14 MW, of which 10 MW are sold to Sabah Electricity Sdn. Bhd. (SESB). The fuel produced is mainly from empty fruit bundles (EFB), oil palm residues in the form of shells and fibers. It is estimated that about 40,000 to 50,000 tons of carbon-di-oxide (CO_2) will be produced each year (Mohamed & Lee, 2006).

2.4.4 Malaysian Demand for Energy

In 2003, total energy demand in Malaysia was 33.9 Mtoe. Energy demand is expected to grow at a rate of 5.4% per annum, reaching 971 TWh (83.5 Mtoe) by 2020. This increase in consumption is primarily driven by industrialization, which is in great demand in manufacturing and transportation. In 2008, consumption of electricity in the residential sector increased by 4.4% over 2007 to reach 19,388 GWh (1,608 Ktoe) and the industrial electricity consumption was 41,689 GWh (3,687 Ktoe). The closing demand of commercial energy in 2008 was 44,901 Ktoe, with the highest share of the industrial

sector as 42.6%, followed by the transportation sector as 36.5%, the residential and commercial sectors as 13.8%, the non-energy sector as 6.4%, and the agricultural sector as 0.6% (Ashnani *et al*., 2014).

In the meantime, the Asia-Pacific Economic Cooperation (APEC) predicted that by 2030, the final demand of energy in 2005 will increase to 6,248 Mtoe, up to 40% at an annualized rate of 1.3% (Sorda *et al*., 2010). As a developing country, Malaysia tends to grow at a faster rate of 3.3% during 2005-2015 and a slight increase by 2030 at a rate of 3.4%. According to the 2009 Asia-Pacific Energy Research Centre (APERC) analysis results, the most energy-intensive industries in 2005 and 2015 are the industrial sectors, however, as the standard of living raises the demand of vehicle ownership, by 2030 demand from the transportation sector will be the highest (Zhou & Thomson, 2009). While Malaysia is undergoing rapid economic growth and rising energy demand, more alternative energy sources are required to meet the domestic demands. Although, Malaysia is the third largest oil-storage country in the Asia Pacific region and had 4 trillion cubic feet of oil reserved in 2006, it was realized that it cannot rely entirely on oil resources based on the production level in 2005 because only 15 years remain to be left in terms of the reservation of oil while natural gas reserves are estimated as only for 29 years (Mekhilef *et al*., 2011).

2.4.5 Carbon Emission in Malaysia

Increasing the emissions of carbon is one of the main reasons for Malaysia to turn towards the solution of renewable energy. At the Copenhagen Climate Change Summit in December 2009 in Copenhagen, the Malaysian Prime Minister conditionally agreed to commit to reduce carbon emissions to 40% of the emissions intensity of gross domestic product (GDP) by 2020 by protecting forest areas. Greenhouse gases (GHGs) are associated with carbon emissions, thus causes a threat to humankind. A scientists group

formed by United Nations was named as International Panel on Climate Change (IPCC) who agreed that the carbon dioxide (CO_2) gas is mostly responsible for global warming. Though, there are other different greenhouse gases (GHGs); such as methane, nitrous oxide, and chlorofluorocarbons; which can trap more heat compared to CO_2, attention to those gases were much inferior than CO_2. Therefore, the impact of GHGs is recognized as equal to the amount of CO_2. As a result, APERC models only the emissions of CO_2 because fuel combustion generates more than 90% of the CO_2 equivalent of energy-related GHG emissions, which in turn account for approximately the equivalent of CO_2 emissions as two-thirds of the total GHGs. Since 1990, six billion metric tons of CO_2 equivalent has been released worldwide per year with the increment of more than 20%. In 2006, Malaysia was ranked as third in Southeast Asia with a carbon footprint of 187 million tons, after Indonesia and Thailand. In the APEC region, fuel combustion is estimated to increase by 40% from 16.6 billion tons in 2005 to 23.2 billion tons in 2030, almost half of which will be originated from the electricity and heat sectors. From this measurement, Malaysia is considered as the second largest contributor for CO_2 emissions after Taipei and Taiwan during the years from 2005 to 2015, but it is anticipated to be slightly higher than Taipei by 2030 (Mekhilef *et al*., 2011; Ong *et al*., 2011; Ashnani *et al*., 2014).

2.5 Biodiesel

2.5.1 Background

Biodiesel is a renewable, eco-friendly substitute for diesel that is usually produced from plant materials such as those from the agricultural waste or even from a specific breed of plant that meets the requirement for the production of the liquid. Plants such as soybean, cottonseed, rapeseed, oil palm, wheat, and corn are some of the more common plants used to produce biodiesel but the fact that humans use these plants as a source of

food makes it hard to fully utilise these crops for biodiesel production (Abdullah *et al*., 2009; Lim & Teong, 2010).

Biodiesel can be defined as a clean burning renewable fuel made using natural vegetable oils and fats. Usually, it is made through a chemical process that converts oils and fats of natural origin into fatty acid methyl esters (FAME). Production of biodiesel intends to replace the petroleum diesel fuel or also can be blended with petroleum diesel fuel to reduce the carbon emission by vehicle exhaust (Demirbas, 2008; Sharma & Singh, 2009).

In fact, the old engine introduced by Rudolf Diesel was already designed to accept the change of fuel usage such as from petroleum diesel into biodiesel without any further modification. Furthermore, handling the biodiesel is safer compared to the petroleum diesel since it contains lower toxicity level. In addition, this kind of energy surely can be categorized as biodegradable material (Demirbas, 2008).

2.5.2 Synthesis of Biodiesel

From a chemical point of view, oils from different sources have different fatty acid compositions. The fatty acids vary in their carbon chain length and in the number of unsaturated bonds they contain. Fats and oils are primarily water-insoluble, hydrophobic substances in the plant and animal kingdom that are made up of one mole of glycerol and chemically, the oil/fats consist of 90–98% triglycerides and a small amount of mono and diglycerides. Triglycerides are esters of three fatty acids and one glycerol. These contain substantial amount of oxygen in their structures. When three fatty acids are identical, the product is simple triglycerides; when they are dissimilar, the product is mixed triglycerides fatty acids, which are fully saturated with hydrogen and have no double bonds. Those with one missing hydrogen molecule have one double bond between carbon atoms and are called monosaturated and those with more than one missing hydrogen have

more than one double bond and are called polyunsaturated. Fully saturated triglycerides lead to excessive carbon deposits in engines (Demirbas, 2008; Lin *et al*., 2011). The fatty acids are different in relation to the chain length, degree of unsaturation, or presence of other chemical functions. Chemically, biodiesel is referred to as the mono-alkyl esters of long-chain-fatty acids derived from renewable lipid sources. Biodiesel is the name for a variety of ester-based oxygenated fuel from renewable biological sources. It can be used in compression ignition engines with little or no modifications. Biodiesel is made in a chemical process called transesterification, where organically derived oils (vegetable oils, animal fats, and recycled restaurant greases) are combined with alcohol (usually methanol) and is chemically altered to form fatty esters such as methyl ester (Figure 2.3). Chemically, most biodiesel consists of alkyl (usually methyl) esters instead of the alkanes and aromatic hydrocarbons of petroleum derived diesel. Oil, ester, and diesel have different numbers of carbon and hydrogen compound. Diesel has no oxygen compound. It is a good quality of fuel. On the other hand, in the case of vegetable oils, oxidation resistance is markedly affected by the fatty acid composition. The large size of vegetable oil molecules (typically three or more times larger than hydrocarbon fuel molecules) and the presence of oxygen in the molecules suggest that some fuel properties of vegetable oil would differ markedly from those of hydrocarbon fuels (Leung *et al*., 2010; Sani *et al*., 2013).

Plants that are part for instance seed, leaves, barks, and root contained a lot of valuable potential that can be drawn out especially for renewable energy feedstock purpose. The only way to get those benefits is by extracting the crudes oil that contains triglyceride and fatty acids from the plants. These compounds store valuable asset of energy that can be shared to help maintain the survival of environment. There are a few methods that can be considered to use as the way to extract the compound from the plants part, which are

soxhlet extraction (SXE), solvent extraction (SE), ultrasonic extraction (UE), microwave extraction, and so on (Kumar *et al*., 2010; Guldhe *et al*., 2014; Vinatoru *et al*., 2017).

The extraction is commonly used to isolate the compound from natural products. The resultant of the extract is usually homogeneous liquid mixture, thus separation is necessary to choose the needed product from the extracted sample (Demirbas, 2008).

Transesterification is the process of conversion of glyceride to biodiesel or fatty acid methyl ester (FAME). The quality of biodiesel depends on total free fatty acid (FFA) concentration before the esterification happens. In some cases, the quality of the sample reduces gradually due to the wrong way of handling and unsuitable storage condition. It was said that inappropriate handling might cause the content of the water to increase. In addition, exposing the oil to open air and sunlight directly for a long period would affect the free fatty acid concentration (Mofijur *et al*., 2012; Duda *et al*., 2018).

As mentioned before, the moisture condition of the sample and free fatty acid concentration is believed to have an impact during the transesterification of the sample with alcohol. If the free fatty acid concentration is more than 5%, it will cause saponification reaction to occur and the separation process becomes hard to handle and will result in low yield of fatty acid methyl ester product.

There are two types of transesterification process, which are one-step or alkali-catalysed transesterification and two-step or acid-catalysed transesterification. Usually, the one-step transesterification is only used to convert the free fatty acid that contains concentration below than 5% while the two-step transesterification of the oil is recommended for free fatty acid that has concentration of more than 5%; however, the acid-catalysed transesterification is slower than the alkali-catalysed transesterification (Hakimi *et al*., 2017).

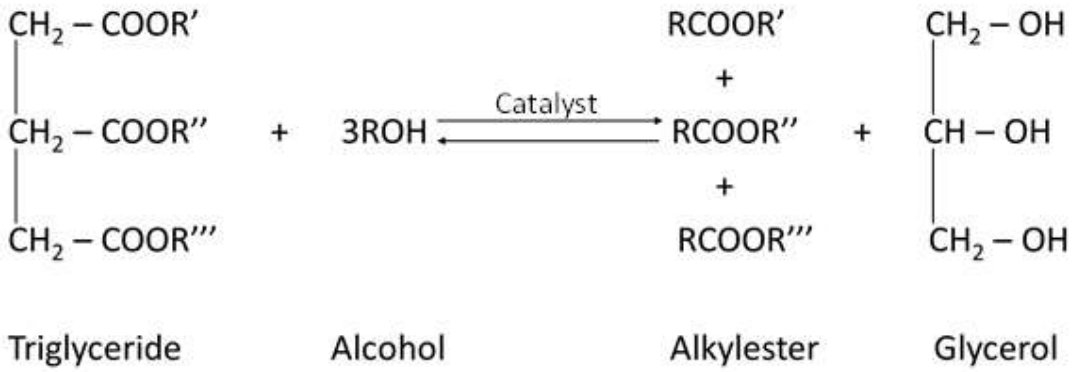


Figure 2.3: Transesterification reaction

2.5.3 Biodiesel Feedstock

Biomass has drawn a lot of attention; not only as it is valuable for economy development, but it also gives a big opportunity in the sustainability of renewable energy source. In fact, the biodiesel from biomass gives a hand in controlling the climatic changes by emitting low or almost zero carbon. Currently, biomass energy is used at 10–15% around the world. In fact, Asian countries such as Malaysia are on their way to utilizing the biomass for renewable energy such as biodiesel (Zhou & Thomson, 2009; Lim & Teong, 2010; Mofijur *et al*., 2012).

Biodiesel is one of the renewable energies that are mainly produced from plant oils or animal fats. In fact, biodiesel shows high possibility to serve as a petroleum diesel to substitute compression ignition engine. In 2003, it was recorded that the world's total biodiesel production was around 1.8 billion litres. In 2010, the demand for biodiesel was estimated to increase from 3 million tons in 2005 to 10 million tons in the European Nations. The feedstock utilized for the biodiesel production in these counties are soybean oil (28%), palm oil (22%), animal fats (20%), coconut oil (11%), while rapeseed, sunflower, and olive oils show the same utilization value of 5% (Sharma & Singh, 2009; Lim & Teong, 2010; Ashnani *et al*., 2014)

In 2006, when Envo Diesel was introduced in Malaysia, it proved how seriously Malaysia is concentrating on producing biodiesel as a renewable energy. For further information, Envo Diesel was derived from blend fuel that constitutes 5% palm oil biodiesel and 95% petroleum diesel. Malaysia does not need to import foreign feedstock to produce its own biodiesel since its land is rich in palm oil plantations. In addition, biodiesel developers can control cost and quality using raw materials from own plantations and this will enable the developers to control the cost and quality of the biodiesel when they use their own raw materials efficiently (Yusuf *et al*., 2011; Oh *et al*., 2017).

2.5.4 Benefits of Biodiesel

It is believed that biodiesel contributes a lot of benefits to earth such as zero carbon production, renewable energy, helps to protect the environment, makes the air quality better, and serves great safety and opportunity not only to humans, but also the entire earth (Demirbas, 2009).

2.5.4.1 The Secure Key for Energy and Balance

In the United States, the petroleum is imported at about a third of which the two-third of it is for transportation purposes. The United States petroleum's source fully depends on foreign trades, which can affect gasoline and diesel production if the trade becomes deficit. Not to mention, its price that fluctuates over the years without any warning is not only a big deal in the United States but also all over the world including Malaysia. Thus, the production of renewable energy such as biodiesel gives a new hope for the world's future and the stability of economy (Demirbas, 2009; Atabani *et al*., 2012).

2.5.4.2 Medium for Improve the Air Quality

For many years, we have been facing the same issue regarding air quality especially pollution caused by the emission of carbon especially from vehicles. Biodiesel seems to

give a great promise in reducing the greenhouse gases impact because the emission of carbon from the use of the biodiesel is nearly zero. In addition, biodiesel gives a good opportunity to humans to preserve their health by breathing in better air since it is able to control the air quality (Lujan *et al*., 2009; Duda *et al*., 2018).

2.5.4.3 Safety Issues

Undoubtedly, biodiesel serves safety for the user and environment since it is less combustible compared to the fossil diesel fuel. In fact, biodiesel has higher flashpoint (130ºC) compared to the fossil diesel fuel (52ºC). Indeed, it is safe for the user to handle or store; it is even safe to use as a replacement for the fossil diesel fuel to run vehicles (Jayeed *et al*., 2009).

2.5.4.4 Advantages of Biodiesel

Biodiesel is a non-toxic substance and could degrade faster than petroleum diesel, which is contributed by the presence of oxygen. In fact, biodiesel can degrade efficiently in water with 85% to 88%. Furthermore, the mixture of biodiesel and petroleum enhances the engine efficiently. The biodiesel resources are mostly obtained from crops benefitting the environment, which potentially reduce acid rain and greenhouse effect caused by pollution emission. Besides, globally, biodiesel is known as ‘‘carbon neutral’’ as it utilizes plants that act as carbon dioxide absorber from the environment (Demirbas, 2009; Leung *et al*., 2010).

In addition, lower vapor pressure and higher flash point characteristic present in biodiesel makes it safer to handle and be stored as compared to petroleum diesel. Next, the lube content in biodiesel preserves the engine shelf-life. Meanwhile, biodiesel could be served as domestic renewable energy sources and is possibly inexhaustible with the energy ability and properties close to diesel fuel (Yusuf *et al*., 2011).

Other significant advantage is that biodiesel contains lower sulphur compared to petroleum diesel by which it will cause lower sulphur oxides emission. The Cetane number that is present in biodiesel displays higher value by which this property will ensure clean and efficient engine combustion compared to petroleum diese (Lujan *et al*., 2009).

2.5.4.5 Disadvantages of Biodiesel Production

Even though biodiesel provides a lot of benefits to humans and ecosystem, it in fact also has its own drawbacks such as offering adverse cold flow properties since it will crystallize at low temperature, which could cause clogging on filters or even unable to pump the fuel to the engine if the crystallization becomes thicker. Furthermore, its oxidation value is lower than petroleum and can cause acidity in the fuel which will produce sediments that can clog filters (Demirbas, 2008).

Meanwhile, the cost of production tends to rise since there are factors that need to be reconsidered such as raw materials resources, food security, and research development tools. The factor of geography also plays a role since different places are only adaptable to certain biodiesel properties, for example, cold weather countries cannot use biodiesel with highly saturated fatty content (Fontaras *et al*., 2010).

Next is the invasiveness issue. It is important to control the growth of foreign biomass sources from affecting the existence of native species. It is feared that without proper management, the invasive species will disturb the stability of ecology and environmental system (Bassam, 2013).

2.5.4.6 Environmental Impact

Biodiesels should be produced in the most economical and environmentally friendly technique in order to make them more preferable than petroleum diesel fuel. This is

because petroleum diesel fuel is a non-renewable energy that cannot be replaced in a short period of time and it is limited on earth (Luque *et al*., 2008; Peiró *et al*., 2010). Instead of its huge impact on the environment, the constant use of petroleum diesel fuel will surely make them to be depleted and disappear from time to time. Biodiesel, which is known as biomass energy, can be very beneficial to the environment as it is cleaner and greener to be used as fuel to motor vehicles compared to petroleum diesel fuel; however, there are concerns about its economic viability (Mekhilef *et al*., 2011; Oh *et al*., 2017).

Biodiesel is classified as biofuel and each biofuel has some benefits and potential costs. Research on biofuel has come out with a conceptual scheme to evaluate different biofuels by using just two criteria, which are greenhouse-gas emissions and overall environmental impact (Abdullah *et al*., 2009; Savvanidou *et al*., 2010). They compare gasoline, diesel, and natural gas with 26 different biofuels that are produced from different feedstock and livestock such as corn, sugar cane, soy, palm oil, and recycled oil (Adi *et al*., 2009; Yee *et al*., 2009; Fontaras *et al*., 2010). The findings of the research stated that most biofuels reduce greenhouse gasses emissions by more than 30% relative to gasoline; however, nearly half that is 12 out of 26 biofuels include the most important biofuels economically such as the U.S. corn ethanol, Brazilian sugarcane ethanol and soy diesel, and Malaysian palm-oil diesel, which have greater aggregate environmental costs than fossil fuels (Jayeed *et al*., 2009; Ghobadian *et al*., 2009; Lujan *et al*., 2009; Duda *et al*., 2018).

Recently, there is a concern among humankind on the production of biodiesel. They have claimed that biodiesel production has disturbed their food supply as it is mainly produced from the feedstock (Chhetri & Islam, 2008). Production of biodiesel will reduce the availability of human feedstock. This problem can be reduced by making use of waste or residue from the feedstock to produce biodiesel instead of using the whole feedstock like copra residue from Cocos nucifera or corn leftover or residue from cultivation

activities such as stalks and cobs. Even though the production of biodiesel from leftover or residue of feedstock will not be as much as the production from the whole feedstock, it will however be beneficial to the environment (Ahmad *et al*., 2011; Sani *et al*., 2013).

Other concern on biodiesel production includes the cost of biodiesel to be used as fuel for motor vehicles as biodiesel is more expensive than petroleum diesel fuel. This is because it requires more energy, processes, and time to produce biodiesel from feedstock. This concern results in petroleum diesel fuel to be more preferable than biodiesel as the economical aspect is also very important to any authorities; even its impact on the environment can be huge. The other good effects on the environment are its biodegradable ability, non-toxic, and produce low emission profile including hazardous gases. Furthermore, the continuous biomass feedstock grown ability reduces the contribution for greenhouse gases that causes climate change (Ong *et al*., 2011; Liu *et al*., 2012).

CHAPTER 3: METHODOLOGY

3.1 Background

In this study, the samples of matured *Cocos nucifera* (coconut) copra residue were collected from the agro-industrial markets in Bangsar, Kuala Lumpur. The copra residue that was intact to the shell during collection was separated and cleaned properly (Figure 3.1). Then, the copra would be dried (oven dry, 60ºC) till reaching constant moisture content. The copra was ground using a Wonder® blender (WB-1) into powder form. The copra powder was refrigerated (7ºC) until extraction.

(a) (b)

Figure 3.1: Collection of coconut residue: (a) coconut residue (shell with copra); (b) separated copra residue

The copra was then extracted with 3 different methods of extraction (soxhlet extraction, ultrasonic-assisted extraction, and microwave-assisted extraction) to identify the most efficient technique in extracting oil yield. Then, gas-chromatography (GC) was used to analyse the composition of fatty acid. Consequently, the observation for the presence of free fatty acid in extracted oil was to determine the most suitable technique to extract the oil for biodiesel conversion.

Next, for the biodiesel utilization, two different transesterification methods (in-situ and conventional) and three techniques (stirring hot-plate-assisted, ultrasonic-assisted, and

microwave assisted) were employed. Then, ANFIS and RSM were used to optimize the biodiesel synthesis yield. GC had also been used to check the availability of fatty acid methyl acid (FAME) in producing biodiesel yield. Figure 3.2 is the overall flow of methodology that is summarized for better understanding.

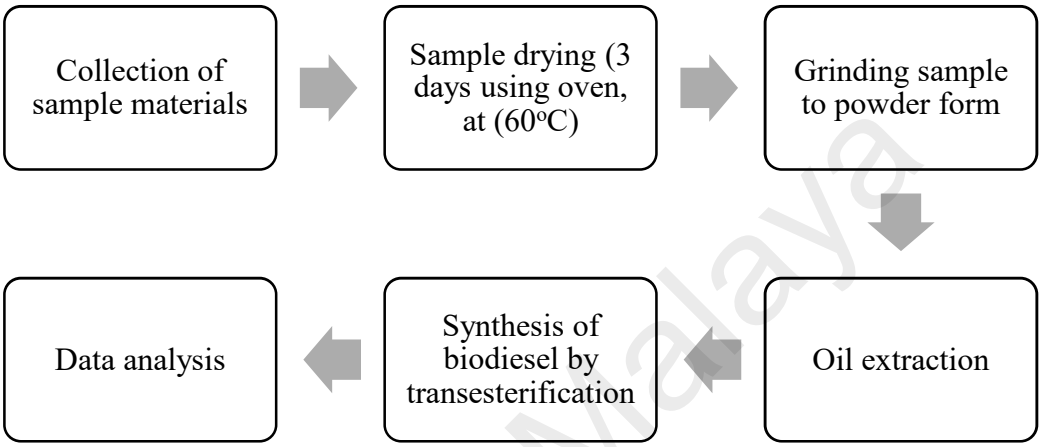


Figure 3.2: Methodology flowchart

3.2 Copra Residue Oil Extraction

3.2.1 Apparatus and Reagents

Apparatus used for extraction techniques was Soxhlet (supplied by Favorit®), ultrasonic water bath (Thermo-Line), and modified domestic microwave (Panasonic). The other apparatuses were weighing balance (Shimadzu), cellulose extraction thimble (Favorit®), heating mantle (TOPS®), rotary evaporator (IKA®), laboratory bottle (Schott Duran), round-bottom and flat-bottom flask (Favorit®). Further, the solvent used for the extraction of hexane was supplied by Friendmann Schmidt.

3.2.2 Extraction Preparation

The copra oil extraction process in this study involved three types of extraction techniques, namely Soxhlet extraction (SXE), ultrasonic-assisted extraction (UAE), and microwave-assisted extraction (MAE) as illustrated in Figure 3.3 (a-c). Oil extraction was carried out according to the AOCS Method (Am 2-93) (American Oil Chemists' Society,

2009). Hexane (analytical reagent grade) was used as solvent, 10g of copra powder and 60°C (±5) of temperature was applied in all extraction procedures. After extractions were done, the copra oil was separated from the sample (by filtering) and solvent (using rotary evaporator). All extractions were performed in triplicates. The summary of copra residue oil extraction was illustrated in Figure 3.4.

(a) ***Soxhlet Extraction (SXE)***

In this SXE technique, the extraction was done under different conditions (volume and time). As shown in Figure 3.3 (a), the sample was located inside the cellulose thimble. The volumes of solvent (hexane) used were 200, 300, 400, 500, and 600 mL while the extraction time of copra oil involved were 6, 12, 24, 48, and 72 hrs.

(b) ***Ultrasonic-Assisted Extraction (UAE)***

For the UAE method, ultrasonic water bath (ultrasonic power 120W, with frequency 40 KHz, 3L capacity, and 240x140x100mm of internal size dimension) was used for this study (Figure 3.3 (b)). The UAE was done under the following conditions: solvent volume (50, 100, 200, 300, and 400 mL) and time (10, 30, 45, 60, and 90 min). For experimental procedure, the copra powder was mixed with solvent in the laboratory bottle with blue cap. Then, the bottled mixture was put in ultrasonic water bath according to time as mentioned above.

(c) ***Microwave-Assisted Extraction (MAE)***

In the MAE technique, modified domestic microwave (800W for power, 200 rpm magnetic stirring, 25 L capacity, and 315x227x349 mm of interior dimension) was utilized for this extraction procedure as illustrated in Figure 3.3 (c). The MAE was performed by mixing copra powder with solvent in flat bottom flask together with magnetic bar under influence of the following conditions: volume of solvent (40, 70, 100, 130, and 150 mL) and time (5, 10, 15, 25 and 35 min).

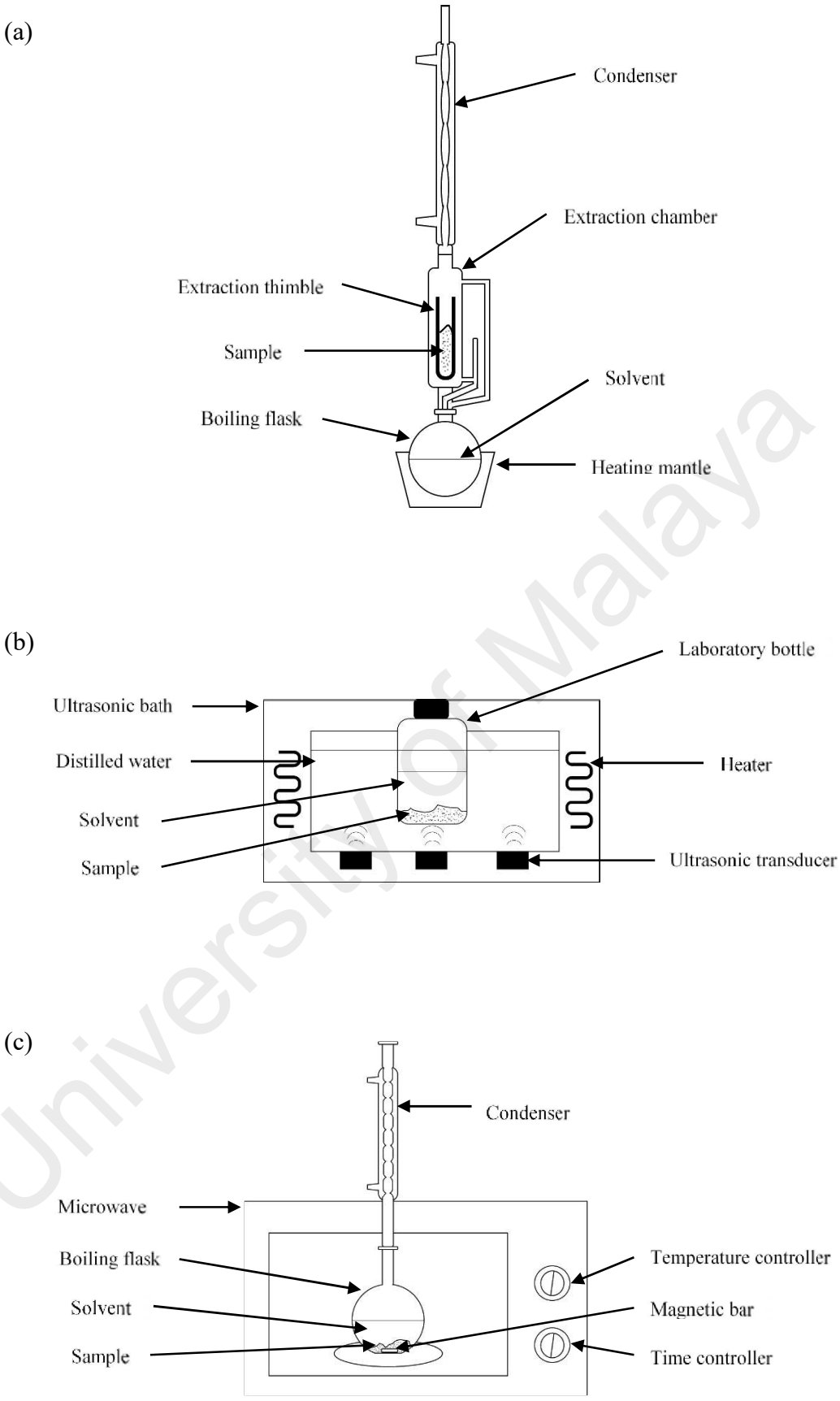


Figure 3.3: Schematic diagram of extraction techniques: (a) SXE; (b) UAE; (c) MAE

3.2.3 Statistical Analysis

Percentage of copra residue oil yield will be calculated using the following formula:

$$Yield = \frac{W_x}{W_u} \times 100 \quad (1)$$

Where W_x is weight of oil and W_u is weight of sample used. For statistical analysis, the results of 25 experimental runs for each extraction technique (SXE, UAE, and MAE) were analysed using Originpro 2016 and Minitab version 18 software. Analysis of variance (ANOVA) was conducted to validate the results.

3.2.4 Chemical Properties Profile

Fatty acid composition was analysed by gas chromatography (GC). The analysis was done using the Agilent 7000 GC-QQQ gas chromatography coupled to Ion trap mass spectrometer with HP-5MS column (5% phenyl methyl silox). The initial temperature of oven was programmed at 70ºC for 0 minute and was gradually increased with 10ºC/min up to 300ºC (held for 6 minutes). Helium as carrier gas was used at 1 mL/min in constant flow mode with splitless injection temperature of 250ºC, auxiliary temperature at 280ºC, and threshold at 150. The sample was prepared according to AOCS Official Method AOCS Ce2-66 (American Oil Chemists' Society, 2007).

For free fatty acid (FFA), the concentration of extracted CRO from SXE, UAE, and MAE was evaluated by referring to AOCS Ca 5a-40 method (American Oil Chemists' Society, 2007).

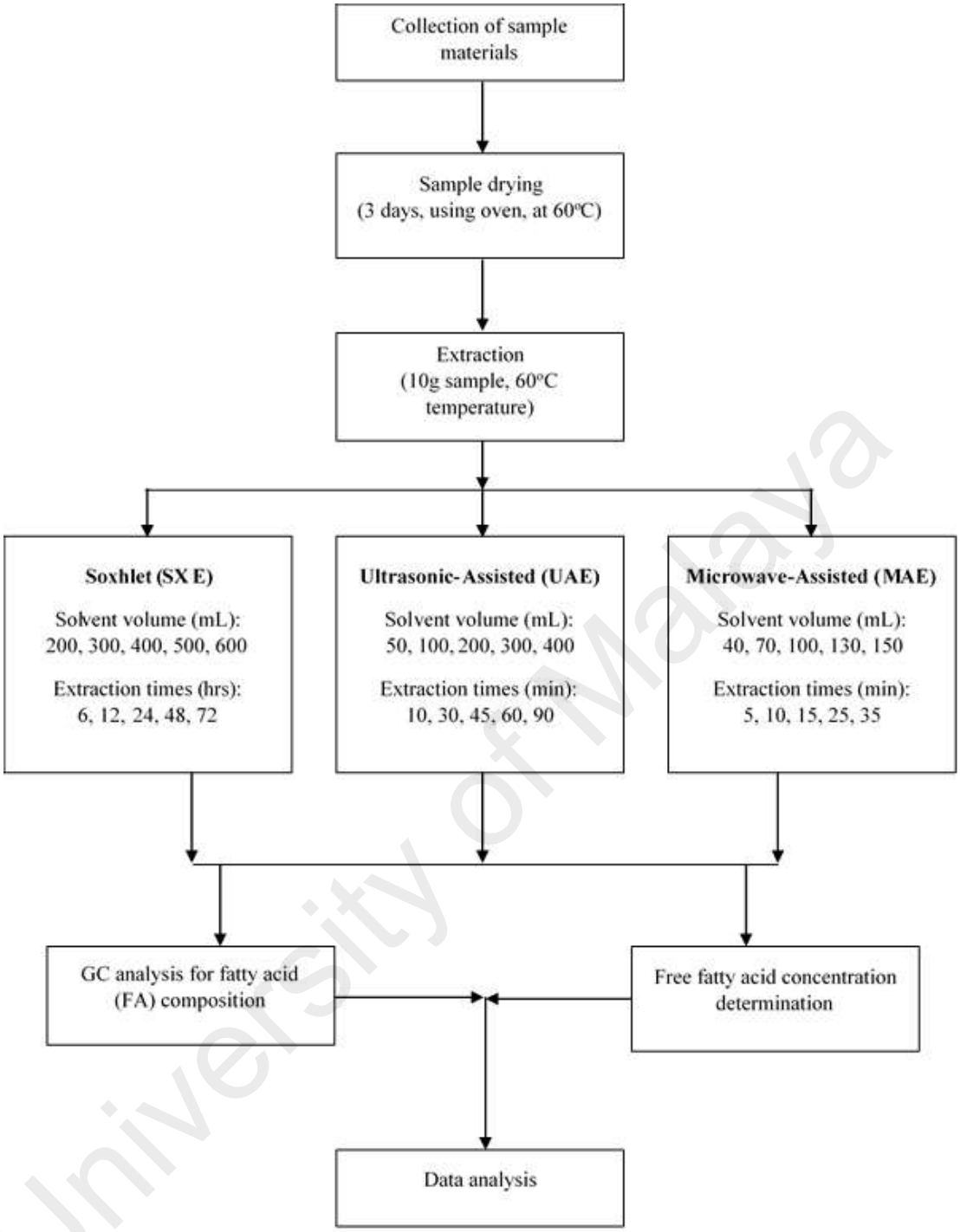


Figure 3.4: Copra residue oil extraction flow chart

3.3 Synthesis of Biodiesel

3.3.1 Apparatus and Reagents

Apparatus used for transesterification techniques was stirring hot-plate (supplied by IKA®), ultrasonic water bath (Thermo-Line), and modified domestic microwave (Panasonic). The other apparatuses were weighing balance (Shimadzu), rotary evaporator (IKA®), laboratory bottle (Schott Duran), separtory funnel (Schott Duran), magnetic bar, round-bottom and flat-bottom flasks (Favorit®). Furthermore, the chemicals used for the transesterification were methanol (Friendemann Schmidt), potassium hydroxide pallet (Systerm®), and hexane which was supplied by Friendemann Schmidt.

3.3.2 Transesterification Preparation

The conversion of the copra oil into biodiesel was done using two different transesterification methods: conventional and *in situ*. Besides, the transesterification will be assisted by three different techniques, namely stirring hot-plate-assisted (HT), ultrasonic-assisted (UT), and microwave-assisted (MT) as depicted in Figure 3.5 (a-c) and Figure 3.6 (a-c). Methanol will be served as alcohol and potassium hydroxide (KOH) as catalyst. The transesterification reaction will use 3:1, 6:1, 9:1, 12:1, and 15:1 methanol to oil molar ratio (v/w) with 0.5%, 1.0%, 1.5%, 2.0%, and 2.5% of KOH to oil (w/w). Before mixing the sample, the KOH needs to dissolve in methanol first to form sodium methoxide solution. The reaction was set at 65^{o}C temperature and stirred for 30 minutes or until the reaction occurs. Once esterified, the sample was transferred to the separatory funnel for 24 hours. The final product of transesterification reaction produced two layers of compounds known as biodiesel or fatty acid methyl acid (FAME) at the top and glycerol at the bottom. The FAME was siphoned off for analysis. The copra residue biodiesel synthesis process was as illustrated in Figure 3.8.

3.3.2.1 Conventional Method of Transesterification

Conventional transesterification refers to method that should undergo two steps. The first step, the oil should be extracted from the copra. The second step was transesterification, which used extracted oil yield from the first step.

3.3.2.2 *In Situ* Method of Transesterification

Next, for the *in situ* step, the copra will directly mix with alcohol and catalyst for transesterification reaction to utilize the biodiesel. Oil extraction is not required for this method. In addition, hexane was introduced during the transesterification to assist the reaction to synthesis high yield since the presence of hexane does not disturb the transesterification reaction instead.

3.3.3 Statistical Analysis

The collected biodiesel yield was calculated using the formula as follows:

$$Yield = \frac{A_y}{[(\frac{O_m}{O_{mw}}) \times 3] \times B_{mw}} \times 100 \quad (2)$$

Where A_y is actual yield, O_m is oil mass, O_{mw} is oil molecular mass, and B_{mw} is biodiesel molecular weight. All the 25 experiments being run for all techniques were statistically analysed using Originpro 2016 and Minitab 18 by employing ANOVA to verify the models' accuracy. All the experiments were performed in triplicate (n=3).

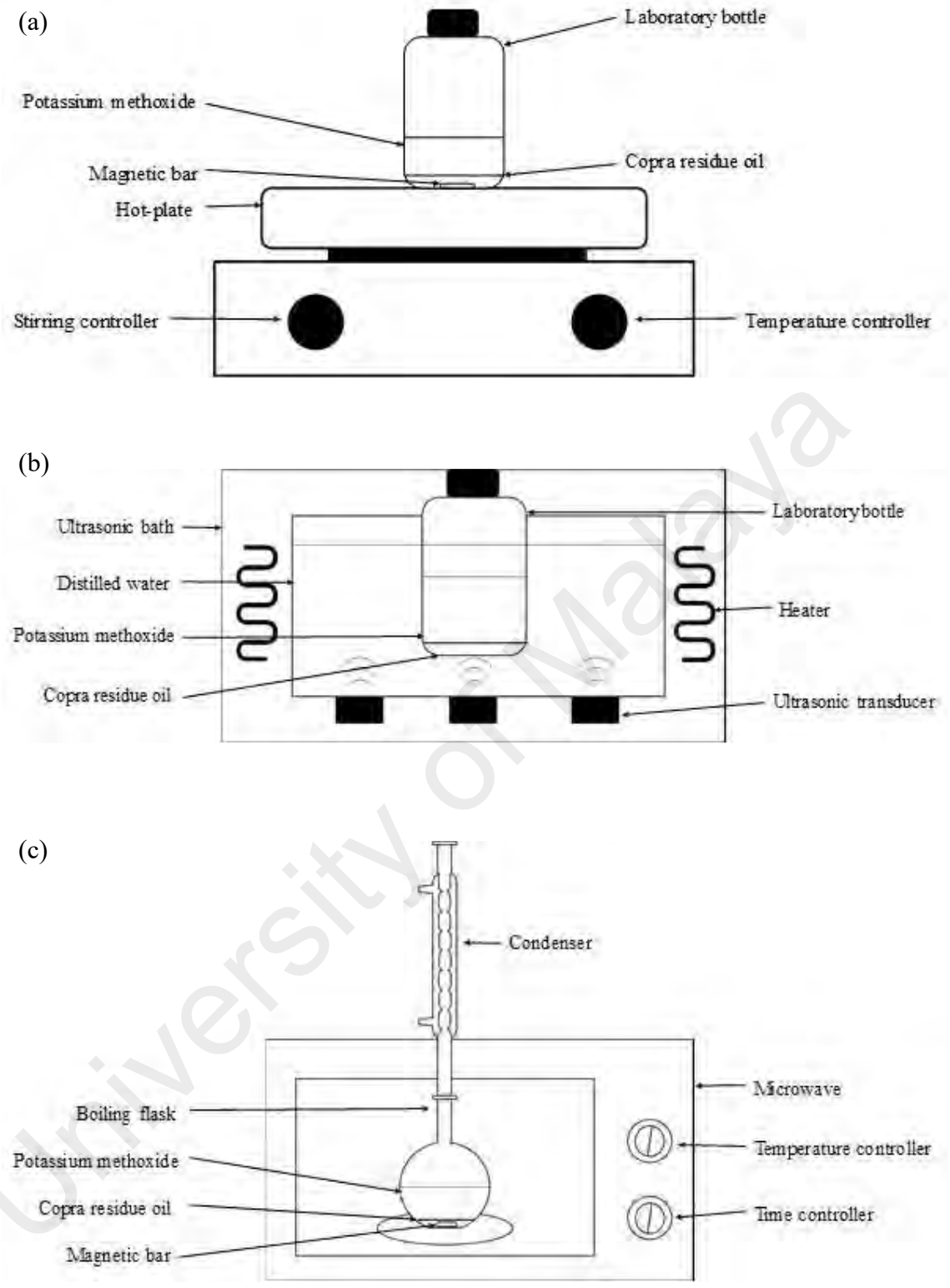


Figure 3.5: Copra residue oil transesterification using conventional method and different techniques: (a) HT; (b) UT; (c) MT

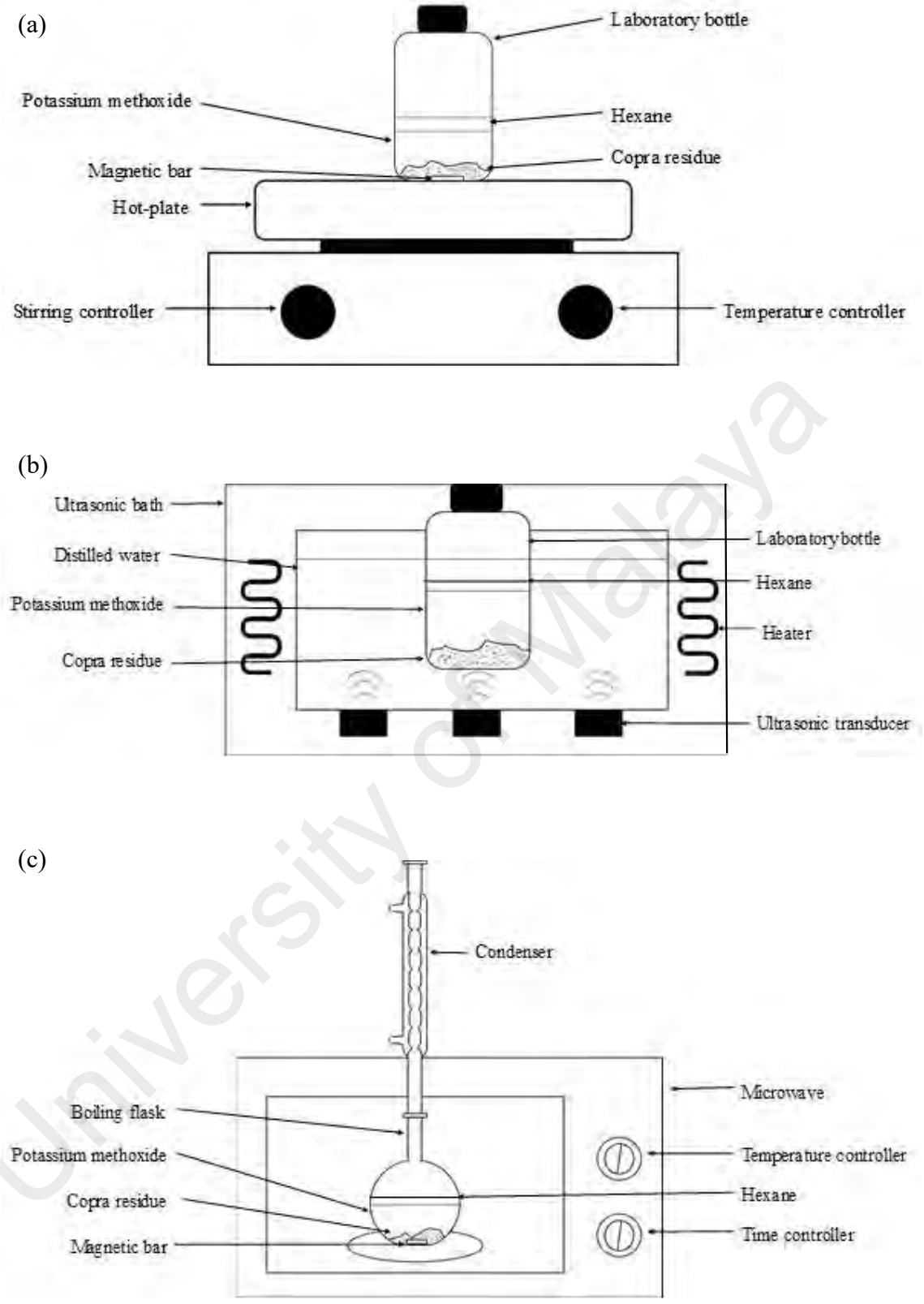


Figure 3.6: Copra residue transesterification using *in situ* method and different techniques: (a) HT; (b) UT; (c) MT

3.3.4 Optimization of Experiment

The collected biodiesel yield from the experiment was then compared with artificial intelligence software, ANFIS and RSM to optimize the biodiesel production.

3.3.4.1 Adaptive Neuro-Fuzzy Inference System (ANFIS)

Adaptive neuro-fuzzy inference system (ANFIS) is the integration of fuzzy logic system with neural network that could act as hybrid intelligent system. With the advantage of hybrid system, it was proven from the previous study for its efficiency. Using neuro-fuzzy toolbox in MATLAB R2015b with Sugeno method and training setting, data set from experimental run was set up. This study will use hybrid membership which integrates back-propagation algorithm with a least squares method type. The inserted data were composed of two inputs (solvent to oil molar ratio and catalyst concentration) and one output. Figure 3.7 depicted the fuzzy inference system (FIS) that consists of 9 rules with logical connector for all rules; this rule uses the linear defuzzifier formula. After the data were run, fuzzy inference computation was performed, and the predicted data were compared with the experimental data.

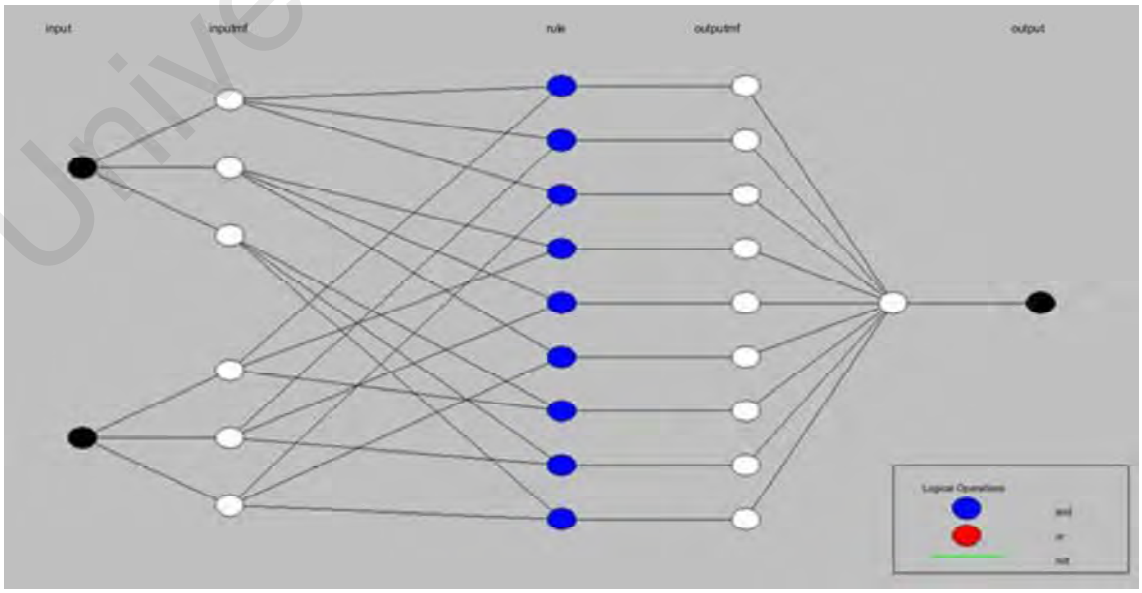


Figure 3.7: Adaptive neuro-fuzzy inference system (ANFIS) model structure

3.3.4.2 Response Surface Methodology (RSM)

Design-Expert 10.0 software was used to run the response surface methodology (RSM). RSM with mathematical and statistical modelling techniques are usually utilized for analysis of multiple regressions. Furthermore, it also functions in quantifying the measured responses and the factors of vital input interactions. This study will use 2 level factors using central composite design (CCD) in investigating independent variables effect (solvent to oil molar ratio and catalyst concentration) on transesterification reaction for converting the coconut residue oil to biodiesel. The least squares method was used to analyse the data and to fit the quadratic polynomial. The predicted data were the compared with experimental data. The data prediction data were computed using following formula:

$$Y = B_0 + \sum_{i=1}^{k} B_i X_i + \sum_{i=1}^{k} B_{ii} X_i^2 + \sum_{i<j}^{k} B_{ij} X_i X_j + e \quad (3)$$

Where Y is predicted yield, B_0 is intercept value (i = 1, 2, 3 … k), B_i is model coefficient, X is level of the factor, B_{ii} is quadratic coefficient of X_i, B_{ij} is effect of interaction, and e refer to error.

3.3.5 Fatty Acid Methyl Ester (FAME) Profiles

The biodiesel will be analysed using Gas Chromatography-Flame Ionization Detector (GC-FID). The oven temperature will be programmed to start and hold at 150ºC for 15 minutes, increase to 210ºC at 2ºC/min, the 50ºC/min to 220ºC, and then hold at 200ºC for 5 minutes with nitrogen as the carrier gas for GC-FID. Injector and detector temperatures will be set at 240ºC and 28ºC respectively. The sample was prepared according to AOCS Official Method AOCS Ce2-66 (American Oil Chemists' Society, 2007).

3.3.6 Prediction of Biodiesel Physicochemical Properties

Determining the physicochemical properties of biodiesel is really important in identifying the quality of the biodiesel. Due to financial situation and lack of sufficient amount of oil, the BiodieselAnalyzer® version 1.1, which was introduced by Biofuel Research Team will be used to observe the physicochemical properties (Talebi *et al*., 2014). This software will analyse the biodiesel physicochemical properties using the fatty acid methyl ester (FAME) profiles and analysed by GC as mentioned before. Then, the data will be compared with international standards, which are ASTM D6751 (American Society for Testing Materials, 2008) and EN 14214 (European Standardization Organization, 2008). The prediction of biodiesel physicochemical properties were computed using following formula:

$$CN = 46.3 + \left(\frac{5.458}{\sum \frac{560 \times n}{m}}\right) - \left(0.225 \times \sum \frac{254 \times d \times n}{m}\right) \quad (4)$$

Where *CN* is cetane number, *n* is particular FAME percentage, *m* is FAME molecular weight, and *d* is double bonds presence in FAME.

$$CFPP = \left[3.1417 \times \left((0.1 \times C_{16}) + (0.5 \times C_{18}) + (1 \times C_{20}) + (1.5 \times C_{22}) + (2 \times C_{24})\right)\right] - 16.477 \quad (5)$$

Where *CFPP* is cold filter plugging point and $C_{16\text{-}24}$ is percentage of long-chain saturated FAME.

$$CP = (0.526 \times C_{16}) - 4.992 \quad (6)$$

Where *CP* is cloud point and C_{16} is percentage of presence in FAME.

$$\ln(V) = \sum N_i(-12.503 + (2.496 \times \ln Mw_i) - 0.178 \times D_i) \quad (7)$$

Where *V* is kinematic viscosity, *N* is percentage of particular FAME profiles, *Mw* is molecular weight of FAME, and *D* is double bonds present in FAME. Meanwhile, density (ρ) of biodiesel was computed using following formula:

$$\rho = \sum N_i(0.8463 + \left(\frac{4.9}{Mw_i}\right) + 0.0118 \times D_i) \quad (8)$$

Last but not least, the biodiesel higher heating value (HHV) is estimated by following equation:

$$HHV = \sum N_i(46.19 - \left(\frac{1794}{Mw_i}\right) - 0.21 \times D_i) \quad (9)$$

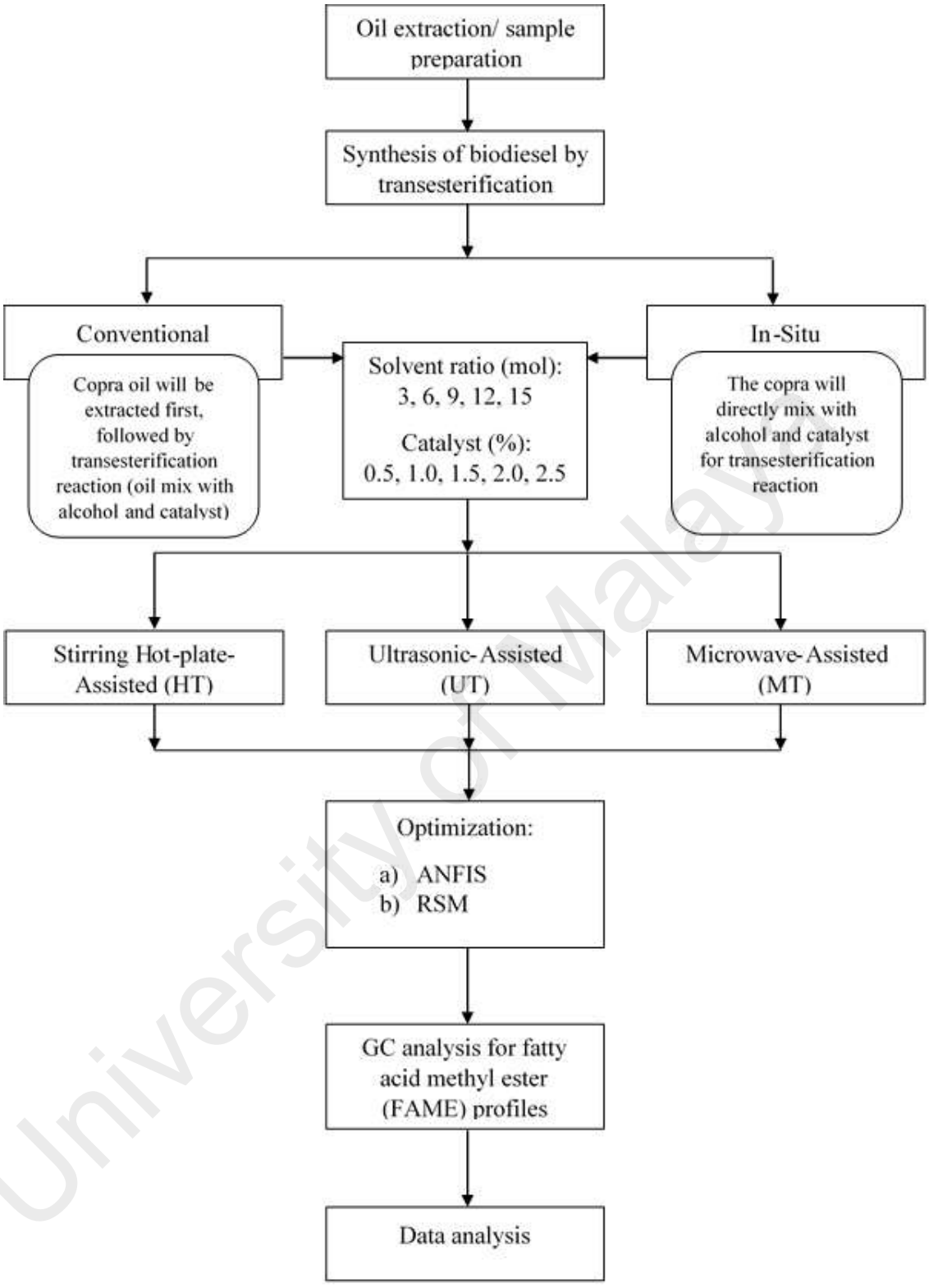


Figure 3.8: Transesterification of copra residue biodiesel flow chart

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Background

This chapter contains the obtained results and discussion. The chapter is divided into two majors of study's findings. The first part will display results for extraction of copra residue oil (CRO), which includes the oil yield, the effect of solvent volume and the effect of extraction time, the effect of different extraction techniques, fatty acid composition, and free fatty acid concentration in extracted CRO. Next, part two will explain the findings of conversion of copra residue biodiesel (CRB). This part will indicate the study's findings on CRB, which includes the biodiesel yield, optimization using ANFIS and RSM, the effect of solvent to oil molar ratio, the effect of catalyst concentration, the effect of different methods, the effect of different techniques, the fatty acid methyl ester (FAME) chemical composition and predicted CRB physicochemical properties.

4.2 Copra Residue Oil

This part of study will discuss the results regarding the CRO yield extracted using different extraction techniques namely Soxhlet (SXE), ultrasonic-assisted (UAE), and microwave-assisted (MAE). The full results will be discussed in the further section.

4.2.1 Statistical Analysis

*(a) **Soxhlet Extraction (SXE)***

The study for 25 of experiments using Soxhlet extraction (SXE) for CRO yield were done. The results observed that the optimal CRO yield was 81.39% (±0.60 of SEM) by employing 48 hours of extraction time and 400 mL of solvent volume. Further, in Table 4.1, it was depicted that the use of extraction time, solvent volumes, and interaction as SXE's parameters in terms of experimental models were all significant by which the p-value was less than 0.05. The result also shows the regression of fit goodness (R^2 =

0.9902) and prediction goodness (pred. R^2 = 0.9779), by which the SXE model developed quite splendidly since its value was almost to unity 1.

(b) Ultrasonic-Assisted Extraction (UAE)

The results for ultrasonic-assisted extraction (UAE) indicated that the use of 30 min of extraction time and 300 mL of solvent produced the optimum CRO yield with 75.80% (±0.75 of SEM). Further, in Table 4.2, it was evinced that the models using extraction time, solvent volumes, and interaction between the parameters were all significant (p-value ≤ 0.05). The result also depicted the regression of fit goodness (R^2 = 0.9846) and prediction goodness (pred. R^2 = 0.9652), by which the UAE model development was good.

(c) Microwave-Assisted Extraction (MAE)

There were 25 experiments being run to investigate the significance of microwave-assisted extraction (MAE) model in extracting CRO yield in this study. From the results, it was observed that the optimal CRO yield was 62.97% (±0.55 of SEM) by employing 15 min of extraction time and 100 mL of solvent volume. Further, in Table 4.3, it was mentioned that the use of extraction time, solvent volumes, and interaction as MAE's parameters in terms of experimental models were all significant with p-value less than 0.05 and the regression of fit goodness (R^2 = 0.9662) and prediction goodness (pred. R^2 = 0.9239), by which the MAE model has developed quite well.

Table 4.1: Analysis of variance (ANOVA) of SXE

Source	df	Adj. SS	Adj. MS	F-value	P-value
Solvent volume	4	4278.73	1069.68	719.60	0.00
Extraction time	4	2787.19	696.80	468.75	0.00
Solvent volume*Extraction time	16	440.46	27.53	18.52	0.00
Error	50	74.32	1.49		
Total	74	7580.71			
Mean – 65.12, Std – 10.120, SEM – 1.17, Variance – 102.44, R^2 – 0.9902, adj. R^2 – 0.9855, pred. R^2 – 0.9779					

Table 4.2: Analysis of variance (ANOVA) of UAE

Source	df	Adj. SS	Adj. MS	F-value	P-value
Solvent volume	4	1397.87	349.47	188.92	0.00
Extraction time	4	4079.95	1019.99	551.40	0.00
Solvent volume*Extraction time	16	417.05	26.07	14.09	0.00
Error	50	92.49	1.85		
Total	74	5987.36			
Mean –57.55, Std – 8.995, SEM – 1.04, Variance – 80.91, R^2 – 0.9846, adj. R^2 – 0.9771, pred. R^2 – 0.9652					

Table 4.3: Analysis of variance (ANOVA) of MAE

Source	df	Adj. SS	Adj. MS	F-value	P-value
Solvent volume	4	752.20	188.06	93.02	0.00
Extraction time	4	2013.40	503.35	248.97	0.00
Solvent volume*Extraction time	16	123.70	7.73	3.83	0.00
Error	50	101.10	2.02		
Total	74	2990.40			
Mean – 50.27, Std – 6.360, SEM – 0.73, Variance – 40.41, R^2 – 0.9662, adj. R^2 – 0.9500, pred. R^2 – 0.9239					

4.2.2 Effect of Different Techniques

The comparison of oil yield between SXE, UAE, and MAE extraction techniques are shown in Figure 4.1. Results revealed that copra residue extracted 81.39% of oil yield when using SXE technique (48 hrs/ 400 mL solvent). Next, MAE (15 min/ 100 mL) showed the lowest oil yield and UAE (30 min/ 300 mL solvent) with moderate results, which were 62.97% and 75.8% respectively. The low yield in MAE technique could be due to the effect of used solvent type, hexane. Hexane, as reported low of dieletric constant and dieletric loss which affecting the ability to absorb the microwave energy (Veggi *et al*., 2013). However, despite of low yield, hexane is the most suitable solvent for oil extraction. Furthermore, few previous researchers using mixture of hexane with other solvents which high of dieletric such as isopropanol and methanol for oil extraction using microwave-assisted (Zigoneanu *et al*., 2008; Veggi *et al*., 2013).

Table 4.4 depicts the summary of comparisons between extraction techniques. As for the MAE technique, the heat and mass gradients worked in the same direction during the transport phenomena (Chemat *et al*., 2009). In fact, the extraction process resulted from changes in the cell structure caused by electromagnetic waves (Veggi *et al*., 2013). In addition, microwave power will increase the temperature pressure and result in high yield recovering until it becomes decrease or insignificant (Zigoneanu *et al*., 2008; Michel *et al*., 2011; Li *et al*., 2012; Veggi *et al*., 2013). MAE could also accelerate the extraction by enhancing the desorption and dissolution of active compound that is bound to matrix's sample with continuous assisted-stirring function, in comparison to those conventional extraction techniques such as SXE, which only involve the interaction between solid and liquid that is between the sample and solvent respectively (Chemat *et al*., 2009, Perino-Issartier *et al*., 2011; Veggi *et al*., 2013). The SXE involved the transfer of heat from outside to inside of the substrate, while mass transfer occurred reversely from inside to outside while the UAE attributed by acoustic cavitation occurrence resulting from

generation of high-intensity of acoustic waves in fluid. The UAE technique involved physical phenomenon which is diffusion of cell walls and the cells were then washed out of the content once the cell wall was disrupted. The sonic wave modified the physical and chemical structure during the interaction with the sample and extractable was released once the cell walls disrupted as resulted from the cavitation activity (Jensen, 2007; Chemat & Khan, 2011; Gaete-Garretón *et al*., 2011; Veggi *et al*., 2013) .

Table 4.5 shows the comparison of coconut oil with different vegetable sources from the previous study (Azeez, 2007; Zhang *et al*., 2008; Tan *et al*., 2009; Sumaram *et al*., 2013; Taghvaei *et al*., 2014; Menezes *et al*., 2017). Most of the oil in the table was commercially produced in different countries. The study revealed that coconut recovered relatively high yield of oil (60-72%) but slightly lower than flaxseed (65-85%) compared to the other vegetable oils. However, green coffee bean oil also showed a slightly similar result of oil recovery to coconut oil with 64-71%. Meanwhile, oil extraction from others are soybean (20-40%), cottonseed (32-35%), palm oil (37-39%) and papaya seed (76.1-79.1). In addition, it can be noticed that differences in extraction techniques give different results of oil yield (Samaram *et al*., 2013; Veggie *et al*., 2013).

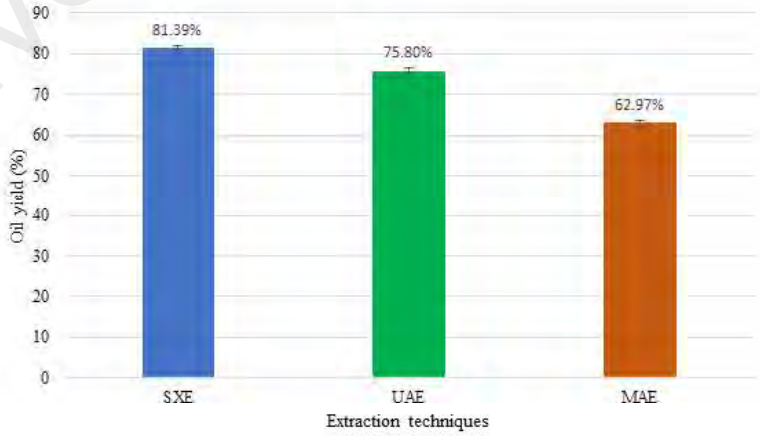


Figure 4.1: Oil yield comparison between SXE (48 hrs, 400mL), UAE (30 min, 300 mL), and MAE (15 min, 100 mL)

Table 4.4: Comparison of extraction techniques (SXE, UAE, and MAE)

Characteristics	Extraction techniques		
	Soxhlet (SXE)	Ultrasonic-assisted extraction (UAE)	Microwave-assisted extraction (MAE)
Extraction procedure	Sample put inside cellulose thimble and placed in Soxhlet extractor. The solvent placed inside round bottom flask and heated using mantle.	Sample immersed in solvent in a laboratory bottle before being placed in ultrasonic water bath.	Sample immersed in solvent with magnetic bar in a flat bottom flask before being placed in microwave.
Extraction interaction	a. Interaction between solid and liquid. Sample continuously percolated by re-condensed solvent vapor. b. Heat transfer from outside to inside. Next, mass transfer inside to outside.	a. Interacted with acoustic cavitation occurrence which was caused by acoustic waves in fluid. b. Involved physical interaction by which cell structure disrupted and caused chemical content to wash out.	a. Electromagnetic waves affected the cell structure and caused physical changes on cell. b. Microwave power increased over time and pressured the sample to wash out the chemical compound. c. Stirring function assisted in extraction acceleration.
Sample size	1 – 30 g	1 – 30 g	1 – 10 g
Solvent amount	200 – 500 mL	30 – 300 mL	10 – 100 mL
Extraction time	3 - 48 hrs	10 – 30 min	3 – 15 min
Advantages	Moderate sample amount, no filtration needed.	Easy to handle, high sample amount, moderate extraction time and multiple extractions.	Fast, low solvent consumption and elevated temperature.
Disadvantages	Long extraction time and high solvent volume.	Need filtration and high solvent amount.	Need filtration and vessel should cool down before next extraction.
Investment	High	Moderate	Low

Source: Jensen, 2007; Zigoneanu *et al*., 2008; Chemat *et al*., 2009; Chemat & Khan, 2011; Gaete-Garretón *et al*., 2011Michel *et al*., 2011; Perino-Issartier *et al*., 2011; Li *et al.,* 2012*;* Veggi *et al*., 2013

Table 4.5: Oil yield from different plant sources

Oil Source	Oil Content (%)	Extraction Techniques
Coconut	60-72	Soxhlet
Palm oil	37-39	Screw press
Papaya seed	76.1-79.1	Ultrasound-assisted, Soxhlet
Soybean	20-40	Ultrasound-assisted
Flaxseed	65-85	Ultrasound-assisted
Cottonseed	32-35	Microwave-assisted, Soxhlet
Green coffee bean	64-71	Microwave-assisted

Source: Azeez, 2007; Zhang *et al*., 2008; Tan *et al*., 2009; Sumaram *et al*., 2013; Taghvaei *et al*., 2014; Menezes *et al*., 2017

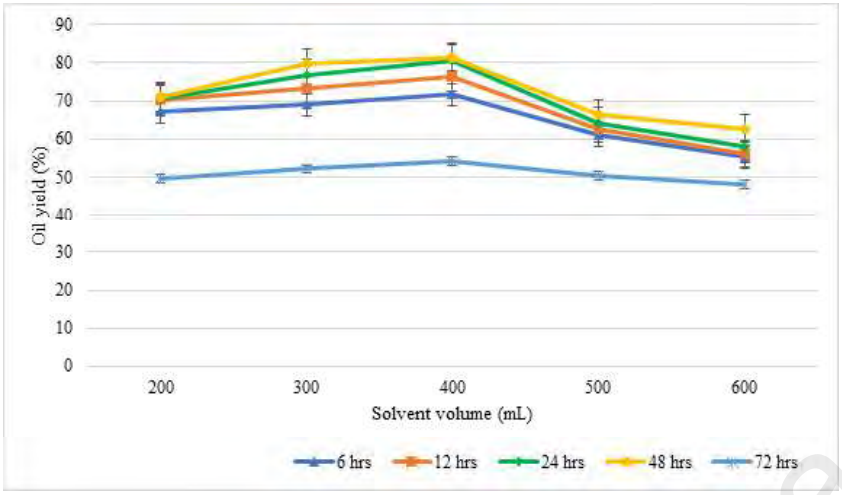
4.2.3 Effect of Solvent Volume

Optimization of parameter using solvent to solid (sample) ratio is an important condition to ensure high extraction of oil yield. Figure 4.2 (a-c) show the effect of volume of solvent (hexane) on CRO yield obtained from different techniques namely SXE, UAE, and MAE. From the results, it was shown that the oil yield increased as the solvent volume increased in the range of 70.88% to 81.39%, 49.97 to 75.8%, and 52.13 to 62.97% respectively. This is due to the fact that high solvent volume can increase the rate of chemical reaction occurred in the extraction process. In fact, the previous study reported that the use of high volume of solvent affects the inclination of extraction recovery (Veggie *et al*., 2013; Taghvaei *et al*., 2014; Tsukui *et al*., 2014).

However, it was observed that each of technique reached maximum oil yield once the solvent hit the maximum of volume usage. As SXE, the oil yield decreased between solvent usage of 400 mL and 500 mL, while UAE and MAE of 300 to 400 mL and 100 to 130 mL, respectively. In addition, the results depicted that MAE used the lowest volume of solvent to achieve the highest oil yield compared to SXE and MAE, which were 100 mL, 300 mL and 400 mL, respectively.

In the MAE technique, the solvent volume should be sufficient enough to guarantee that sample immersed completely throughout the extraction process, especially when using sample that will absorb solvent and become inflated such as copra during the extraction (Li *et al*., 2010; Tatke & Jaiswal, 2011; Veggie *et al*., 2013). The MAE and UAE gave a moderate and low oil yield probably due to non-consistency of distribution and exposure to microwave and sonic power (Veggie *et al*., 2013). Further, SXE (conventional) technique extracted the highest oil yield due to the consistency of interaction between the solid and solvent (Jensen, 2007).

(a)



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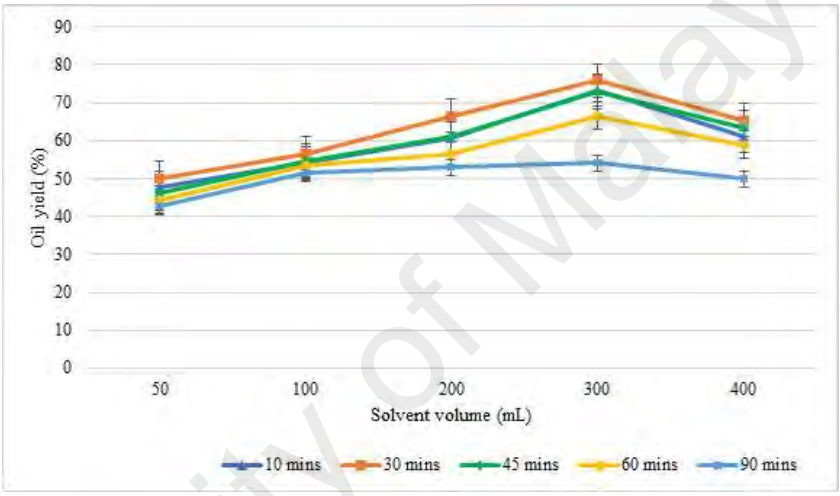

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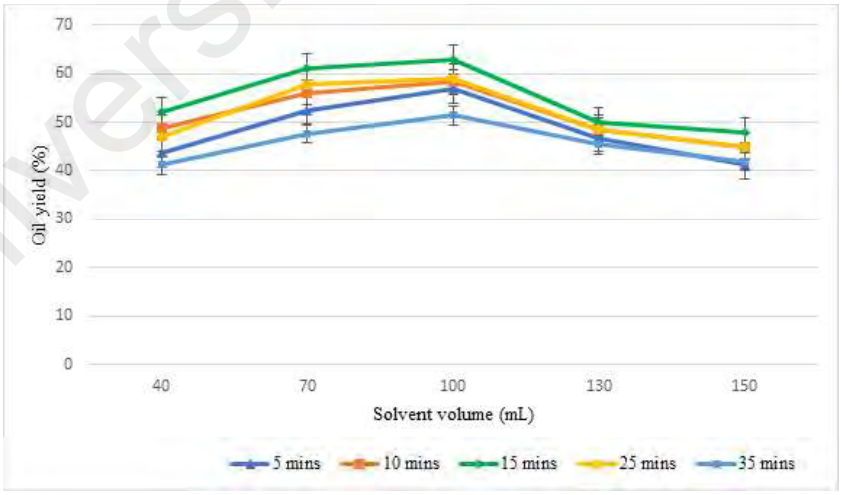
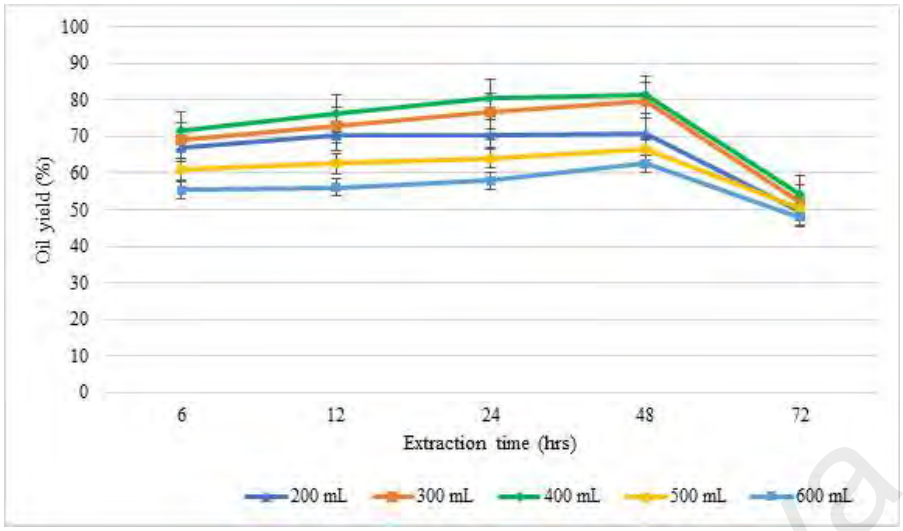


Figure 4.2: Effect of oil extraction using different solvent volumes: (a) SXE; (b) UAE; (c) MAE

4.2.4 Effect of Extraction Time

Extraction time is one of the important conditions that needs to be considered to optimize oil recovery (Tatke & Jaiswal, 2011; Veggie *et al*., 2013; Taghvaei *et al*., 2014). Figure 4.3 (a-c) depicts the effect on CRO yield from SXE, UAE, and MAE techniques based on different extraction times, respectively. The results showed that in SXE, the CRO yield increased until 48 hours (71.56% to 81.39) and then the yield started to decrease. For the UAE, the CRO recovered optimum yield when reached 30 minutes of extraction (56.57%) and for MAE, the copra oil yield only increased until 15 minutes of extraction (62.97%). Thus, by increasing the time for oil extraction through all of the techniques, it could not result in high oil yield. In addition, the study revealed that MAE consumed the shortest time to achieve optimum oil yield, followed by UAE, while SXE required the longest time to reach the maximum yield (Tatke & Jaiswal, 2011; Veggie *et al*., 2013; Taghvaei *et al*., 2014).

(a)



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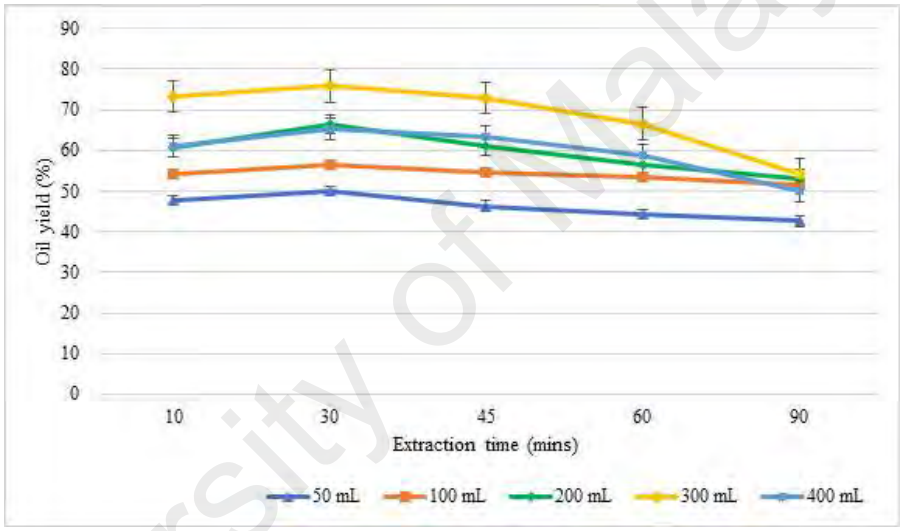

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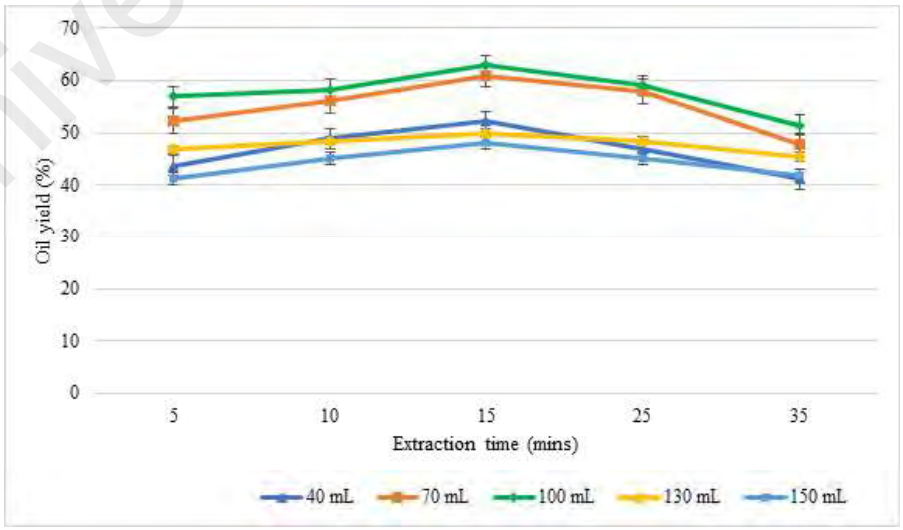


Figure 4.3: Effect of oil yield using different extraction times: (a) SXE; (b) UAE; (c) MAE

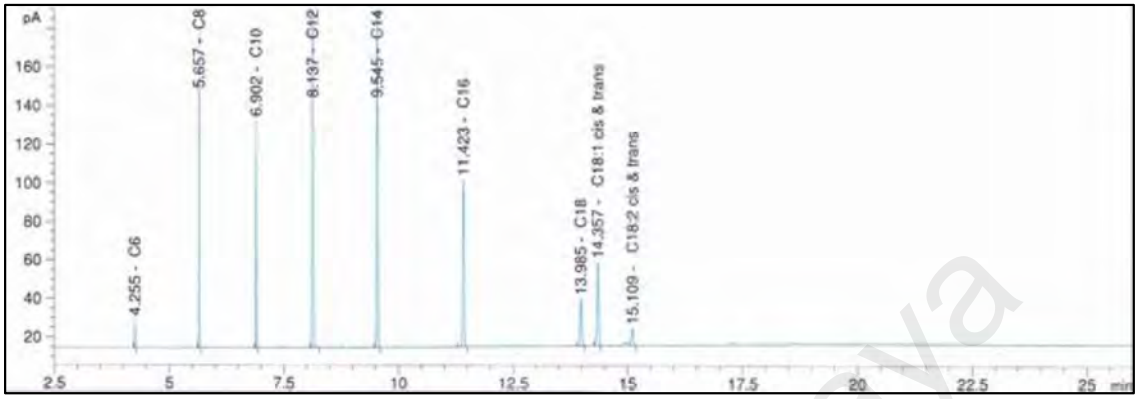
4.2.5 Fatty Acid (FA) Composition

Coconut copra oil as other vegetable oil and animal fats are also triglyceride (Alamu *et al*., 2010; DebMandal & Mandal, 2011). The *Cocos nucifera* oil mainly contains more than 91% of fatty acid with medium chain fatty acid being the major part (DebMandal & Mandal, 2011). The FA contents in oil are really important since it will affect human health and vehicle engine's efficiency when the oil is consumed and used. Figure 4.4 (a-c) and Table 4.6 show FA composition of CRO from different extraction techniques (SXE (48 hrs/ 400 mL solvent), UAE (30 min/ 100 mL solvent), and MAE (15 min/ 100 mL solvent) analysed using Gas Chromatography-Mass Spectrometry (GC/MS). The result indicated the FA composition profile between extracted CRO using SXE, UAE, and MAE. The FA composition present in CRO are caproic acid (C6:0), caprylic acid (C8:0), capric acid (C10:0), lauric acid (C12:0), myristic acid (C14:0), palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1), and linoleic acid (C18:2). This is as stated by previous studies by which most abundant fatty acids that can be found in the coconut oil are lauric acid and myristic acid, followed by linoleic acid (DebMandal & Mandal, 2011; Kumar & Krishna, 2015; Pontoh, 2016).

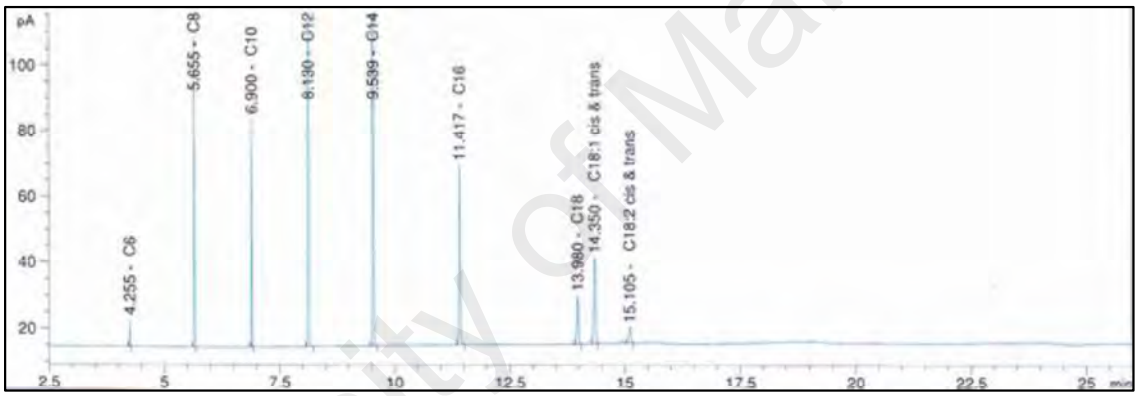
All extraction techniques showed lauric acid as the highest compound of FA with about 47.44% (SXE), 47.67% (UAE), and 47.11% (MAE) as in comparison to other compounds. On the other hand, caproic acid was shown as the lowest compound for CRO extracted from all the techniques; SXE, UAE, and MAE, with only about 0.68%, 0.65%, and 0.65% respectively. The study shows that MAE recovered slightly high compared to SXE and UAE for palmitic acid (MAE (9.13%), SXE (8.84%), and UAE (9.07%)), stearic acid (MAE (3.17%), SXE (3.10), UAE (3.15%)) and oleic acid (MAE (5.96%), SXE (5.85), UAE (5.84)). Hence, the previous studies and current research revealed that the effect of the different techniques on FA composition was not really significant since it

was only slightly different in FA composition concentration and recovered the same FA profiles (Tan *et al*., 2009; Samaram *et al*., 2013).

(a)



(b)



(c)

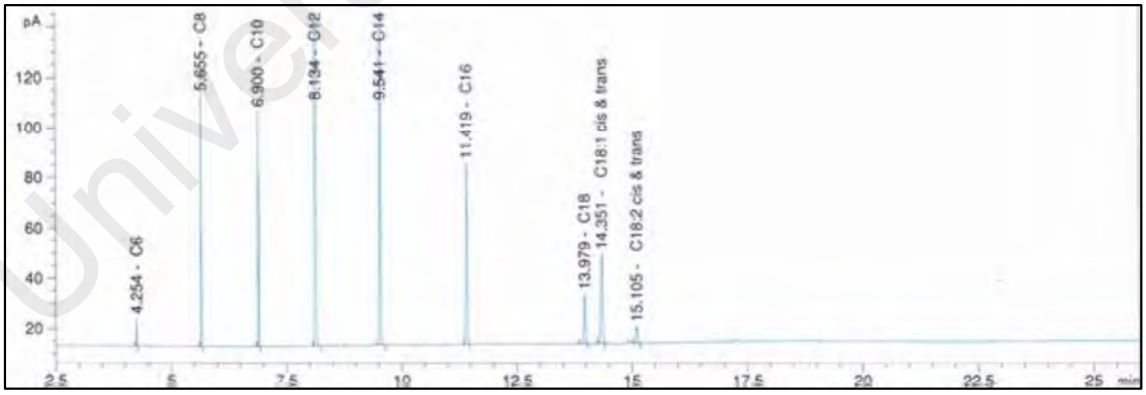


Figure 4.4: Chromatogram of copra residue oil Fatty acid (FA) extracted from different techniques (a) SXE (400 mL, 48 hrs); (b) UAE (300 mL, 30 min); (c) MAE (100 mL, 15 min)

Table 4.6: Fatty acid (FA) composition using different extraction techniques: SXE, UAE and MAE

No.	Fatty Acid Composition		Fatty Acid (%)		
			SXE	UAE	MAE
1.	C6:0	Caproic acid	0.68	0.65	0.65
2.	C8:0	Caprylic acid	8.14	7.78	7.83
3.	C10:0	Capric acid	6.18	6.11	6.10
4.	C12:0	Lauric acid	47.44	47.67	47.41
5.	C14:0	Myristic acid	18.53	18.55	18.56
6.	C16:0	Palmitic acid	8.84	9.07	9.13
7.	C18:0	Stearic acid	3.10	3.15	3.17
8.	C18:1	Oleic acid	5.85	5.84	5.96
9.	C18:2	Linoleic acid	1.24	1.18	1.19

4.2.6 Free Fatty Acid (FFA) Concentration

Free Fatty Acid (FFA) concentration (as lauric) in the CRO by different extraction methods are compared in Figure 4.5. CRO extracted through UAE (90 min/ 300 mL solvent) technique contained the lowest FFA which was 0.18%, while SXE (48 hrs/ 400 mL solvent) containing the highest, 8.03% and UAE technique showed moderate FFA concentration. This might be due to the long extraction time in the SXE technique that took hours compared to other techniques (UAE and MAE), which consumed shorter extraction times (minutes) (Zhang *et al*., 2008; Chan *et al*., 2011; Song *et al*., 2011). In addition, the sonic and electromagnetic wave involved in the extraction process of copra oil through UAE and MAE techniques could possibly affect the FFA content in the oil instead of the heat factor only (Song *et al*., 2011; Taghvaei *et al*., 2014).

The concentration of FFA in oil is really important as low content of FFA could prolong the shell-life of oil and at the same time might affect the next possible product from the oil such as biodiesel, since the FFA of the oil must be below 5% to avoid saponification during the conversion process (Hakimi *et al*., 2017).

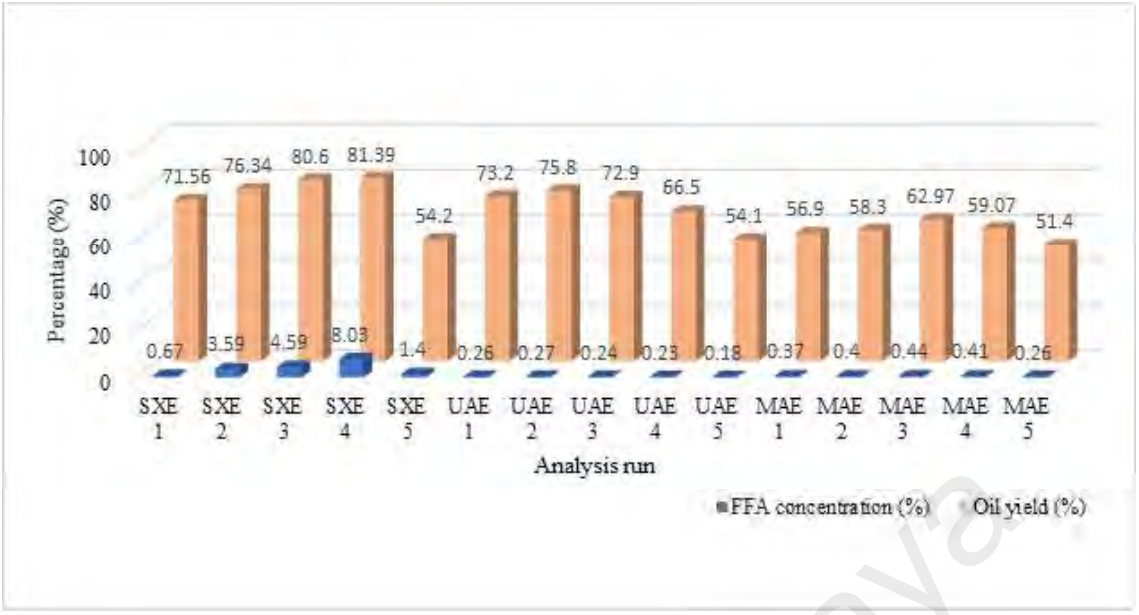


Figure 4.5: Free fatty acid (FFA) concentration in extracted oil yield: SXE1 (SXE, 400 mL, 6 hrs); SXE2 (SXE, 400 mL, 12 hrs); SXE3 (SXE, 400 mL, 24 hrs); SXE4 (SXE, 400 mL, 48 hrs); SXE5 (SXE, 400 mL, 72 hrs); UAE1 (UAE, 300 mL, 10 min); UAE2 (UAE, 300 mL, 30 min); UAE3 (UAE, 300 mL, 45 min); UAE4 (UAE, 300 mL, 60 min); UAE5 (UAE, 300 mL, 90 min); MAE1 (MAE, 100 mL, 5 min); MAE2 (MAE, 100 mL, 10 min); MAE3 (MAE, 100 mL, 15 min); MAE4 (MAE, 100 mL, 25 min); MAE5 (MAE, 100 mL, 35 min)

4.3 Copra Residue Biodiesel

This part of research will illustrate the results of biodiesel yield from transesterification reaction with employment of different methods (conventional and *in situ*) and techniques (stirring hot-plate-assisted, ultrasonic-assisted, and microwave-assisted). The CRO used in this experiment was extracted using the ultrasonic-assisted extraction since the technique displayed consistency of moderate oil yield and low free fatty acid content (<5). The data of coconut copra biodiesel (CRB) yield were discussed in the further section.

4.3.1 Statistical Analysis

4.3.1.1 Conventional Method

(a) ***Stirring Hot-Plate-Assisted Conventional Transesterification (HTC)***

The study has done 25 of transesterification experiments to convert CRO into CRB using conventional method with stirring hot-plate-assisted technique (HTC). The results indicated that the use of 3:1 solvent to oil ratio (v/w) and 1% of catalyst to oil (w/w) produced the optimum CRB yield with 96.85% (±0.40 of SEM). Further, in Table 4.7, it was evinced that the models using solvent to oil ratio and catalyst percentage were significant (p-value ≤ 0.05). The result also depicted that the regression of fit goodness ($R^2 = 0.9858$) and prediction goodness (pred. $R^2 = 0.9681$), by which the HTC model development is good.

(b) ***Ultrasonic-Assisted Conventional Transesterification (UTC)***

There were 25 experiments being run to investigate the significance of ultrasonic-assisted conventional transesterification (UTC) model in synthesis CRB yield in this study. From the result, it was observed that the optimal CRB yield is 93.79% (±0.58 of SEM) by employing 3:1 solvent to oil ration (v/w) and 1% catalyst to oil (w/w). While in Table 4.8, it was mentioned that the use of solvent to oil ratio, catalyst percentage, and interaction of parameters in terms of experimental models were all significant by which

the p-value was less than 0.05. Further, the result shows the regression of fit goodness (R^2 = 0.9860) and prediction goodness (pred. R^2 = 0.9685), by which the UTC model development is quite good.

(c) ***Microwave-Assisted Conventional Transesterification (MTC)***

The results for 25 experiments using conventional methods with microwave-assisted transesterification technique (MTC) revealed that the optimal CRB yield was 91.76% (±0.50 of SEM) by employing 3:1 solvent to oil ratio (v/w) and 1% catalyst to oil (w/w). While in Table 4.9, it was depicted that the use of solvent to oil ratio, catalyst percentage, and parameters interaction in terms of experimental models was significant (p-value ≤ 0.05). Then, for the regression of fit goodness (R^2 = 0.9884) and prediction goodness (pred. R^2 = 0.9740), by which the MTC model developed quite splendidly since its value was almost to unity 1.

4.3.1.2 *In Situ* Method

(a) ***Stirring Hot-Plate-Assisted In Situ Transesterification (HTI)***

There were 25 of experiments being run to investigate the significance of stirring hot-plate-assisted *in situ* transesterification (HTI) model in converting CRO into CRB in this study. From the results, it was seen that the optimal CRO yield was 71.76% (±0.36 of SEM) by employing 3:1 solvent to oil ratio (v/w) and 1.5% catalyst to oil (w/w). While in Table 4.10, it was mentioned that the use of solvent to oil ratio, solvent percentage, and interaction of parameters in terms of experimental models were all significant by which the p-value was less than 0.05. Next, the result shows the regression of fit goodness (R^2 = 0.9865) and prediction goodness (pred. R^2 = 0.9696), by which the HTI model development was quite good.

(b) ***Ultrasonic-Assisted In Situ Transesterification (UTI)***

The results for 25 experiments using the *in situ* method with ultrasonic-assisted transesterification techniques (UTI) showed that the optimal CRB yield is 65.93% (±0.54 of SEM) by employing 3:1 solvent to oil ratio (v/w) and 0.5% catalyst to oil (w/w). Furthermore, to prove the validity of results using 0.5% catalyst to oil (w/w) as the optimum parameter for UTI, additional triplicate experiments was carried out using 0.4% catalyst to oil (w/w). It was observed that the results for 3:1, 6:1, 9:1, 12:1 and 15:1 of solvent to oil ratio (v/w) were 59.02% (±0.29), 53.33% (±1.03), 49.47% (±0.71), 47.81% (±0.67), and 43.90% (±0.67) respectively. Table 4.11 depicts that the use of solvent to oil ratio, catalyst percentage and parameters interaction in terms of experimental models were all significant (p-value ≤ 0.05) and, the regression of fit goodness (R^2 = 0.9905) and prediction goodness (pred. R^2 = 0.9787), by which the UTI model development was good since its value was almost to unity 1.

(c) ***Microwave-Assisted In Situ Transesterification (MTI)***

The results for 25 experiments in converting CRO into CRB using the *in situ* method with microwave-assisted transesterification techniques (MTI) indicated that the use of 3:1 solvent to oil ratio (w/w) and 1% catalyst to oil (w/w) produced the optimum CRB yield with 63.23% (±0.32 of SEM). While in Table 4.12, it was evinced that the models using solvent to oil ratio and catalyst percentage were significant (p-value ≤ 0.05). Next, the result depicts the regression of fit goodness (R^2 = 0.9939) and prediction goodness (pred. R^2 = 0.9862), by which the UAE model development was quite good.

Table 4.7: Analysis of variance (ANOVA) of HTC

Source	df	Adj. SS	Adj. MS	F-value	P-value
Solvent ratio	4	984.33	246.083	356.26	0.000
Catalyst	4	1403.37	350.842	507.93	0.000
Solvent ratio*Catalyst	16	12.16	0.760	1.10	0.380
Error	50	34.54	0.691		
Total	74	2434.39			
Mean – 85.48, Std – 5.74, SEM – 0.66, Variance – 32.90, R^2 – 0.9858, adj. R^2 – 0.9790, pred. R^2 – 0.9681					

Table 4.8: Analysis of variance (ANOVA) of UTC

Source	df	Adj SS	Adj MS	F-value	P-value
Solvent ratio	4	1301.12	325.280	415.88	0.000
Catalyst	4	1427.81	356.952	456.38	0.000
Solvent ratio*Catalyst	16	25.58	1.599	2.04	0.028
Error	50	39.11	0.782		
Total	74	2793.61			
Mean – 81.12, Std – 6.14, SEM – 0.71, Variance – 32.75, R^2 – 0.9860, adj. R^2 – 0.9793, pred. R^2 – 0.9685					

Table 4.9: Analysis of variance (ANOVA) of MTC

Source	df	Adj. SS	Adj. MS	F-value	P-value
Solvent ratio	4	1535.29	383.822	464.79	0.000
Catalyst	4	1948.91	487.228	590.01	0.000
Solvent ratio*Catalyst	16	44.29	2.768	3.35	0.001
Error	50	41.29	0.826		
Total	74	3569.78			
Mean – 77.44, Std – 6.95, SEM – 0.80, Variance – 48.24, R^2 – 0.9884, adj. R^2 – 0.9829, pred. R^2 – 0.9740					

Table 4.10: Analysis of variance (ANOVA) of HTI

Source	df	Adj SS	Adj MS	F-value	P-value
Solvent ratio	4	1582.00	395.500	480.68	0.000
Catalyst	4	1370.93	342.732	416.55	0.000
Solvent ratio*Catalyst	16	53.38	3.336	4.05	0.000
Error	50	41.14	0.823		
Total	74	3047.45			
Mean – 58.70, Std – 6.41, SEM – 0.74, Variance – 41.18, R^2 – 0.9865, adj. R^2 – 0.9800, pred. R^2 – 0.9696					

Table 4.11: Analysis of variance (ANOVA) of UTI

Source	df	Adj. SS	Adj. MS	F-value	P-value
Solvent ratio	4	3013.06	753.264	897.20	0.000
Catalyst	4	1337.05	334.263	398.13	0.000
Solvent ratio*Catalyst	16	37.54	2.346	2.79	0.003
Error	50	41.98	0.840		
Total	74	4429.62			
Mean – 47.45, Std – 7.73, SEM – 0.89, Variance – 59.86, R^2 – 0.9905, adj. R^2 – 0.9860, pred. R^2 – 0.9787					

Table 4.12: Analysis of variance (ANOVA) of MTI

Source	df	Adj. SS	Adj. MS	F-value	P-value
Solvent ratio	4	2886.13	721.533	1077.85	0.000
Catalyst	4	2511.08	627.769	937.79	0.000
Solvent ratio*Catalyst	16	15.18	0.948	1.42	0.172
Error	50	33.47	0.669		
Total	74	5445.85			
Mean – 43.87, Std – 8.58, SEM – 0.99, Variance – 73.59, R^2 – 0.9939, adj. R^2 – 0.9909, pred. R^2 – 0.9862					

4.3.2 Biodiesel Synthesis Optimization

4.3.2.1 Adaptive Neuro-Fuzzy Inference System (ANFIS)

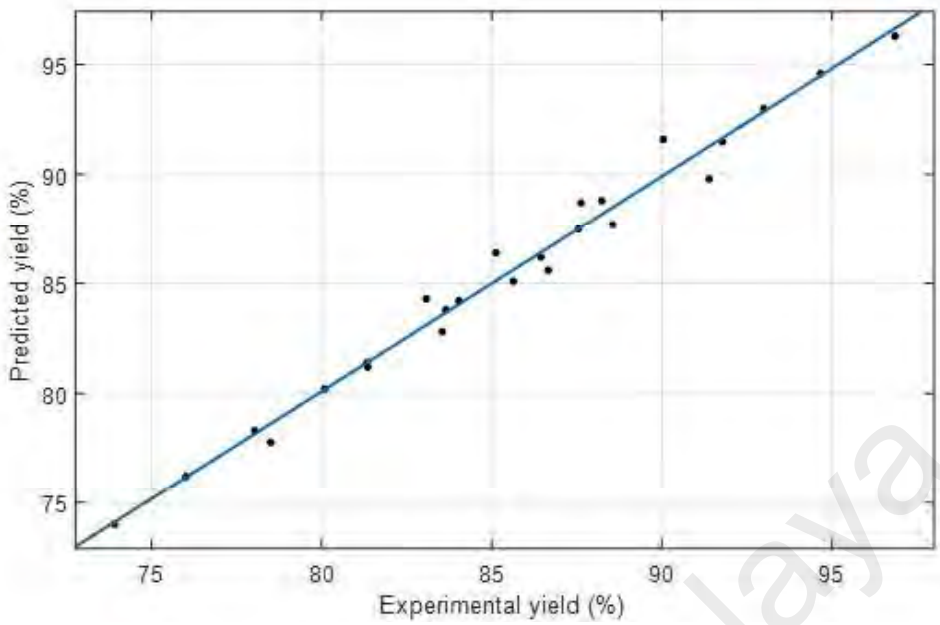
(a) ***Stirring Hot-Plate-Assisted Conventional Transesterification (HTC)***

CRB yields for the 25 experiments carried out using HTC. In this study, the accuracy of the model was observed by the computed values of R^2 (0.8212) and adjusted R^2 (0.8131). The values ($R^2 > 0.8$), which showed R^2 and adjusted R^2 by the model indicated good fit of the model. Furthermore, the plot of the experimented against predicted values shown in Figure 4.6 (a) and experimental against residual values ($-3 \geq \text{residual} \leq +3$) in Figure 4.6 (b), which verified the precision and accuracy of the model. In addition, the training data plotted versus FIS output evinced good compatibility and less testing error was achieved (e = 0.69).

(b) ***Ultrasonic-Assisted Conventional Transesterification (UTC)***

The CRB yields for the 25 experiments using UTC were identified and the accuracy of the used model was studied by observing the computed values of R^2 (0.8494) and adjusted R^2 (0.8426), which indicated good fit of the model ($R^2 > 0.8$). Besides, Figure 4.7 (a) displays the plot of the experimental against prediction values, which verified the precision and accuracy of the model. Furthermore, in Figure 4.7 (b), the plot of experimental against residual values exhibited the goodness of the model by which the residue value was between ±3. In fact, the training data plotted shows good compatibility with testing error value (e = 0.73).

(a)



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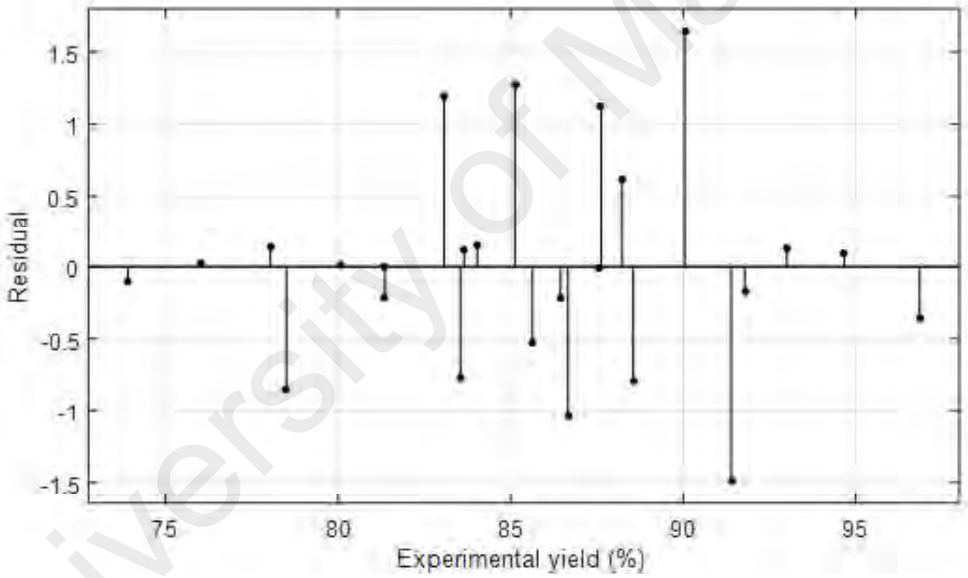
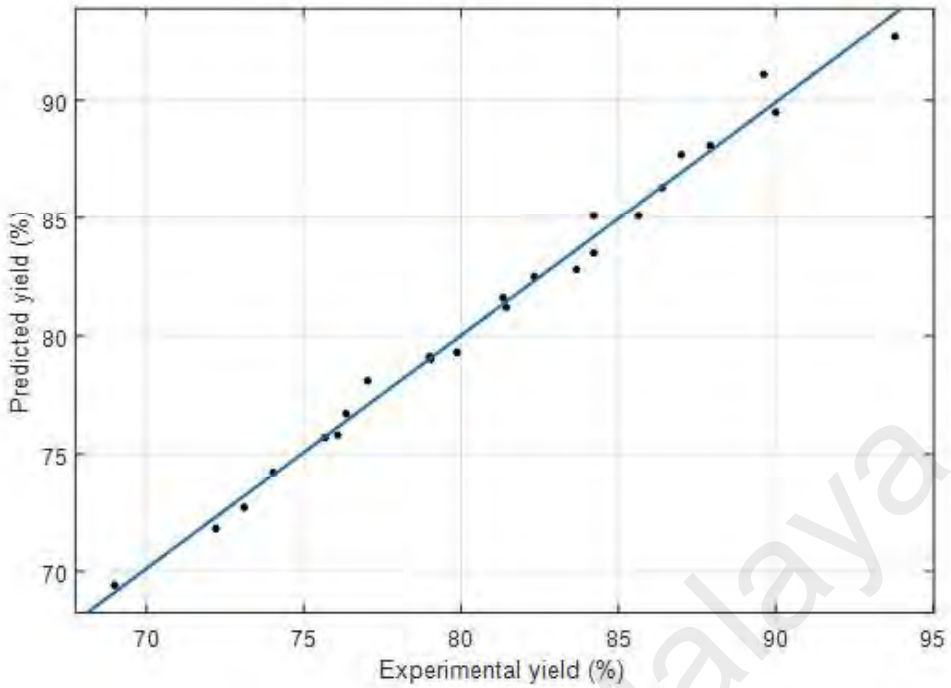


Figure 4.6: Correlation of HTC techniques using ANFIS: (a) experimental yield against predicted yield; (b) experimental yield against residual

(a)



(b)

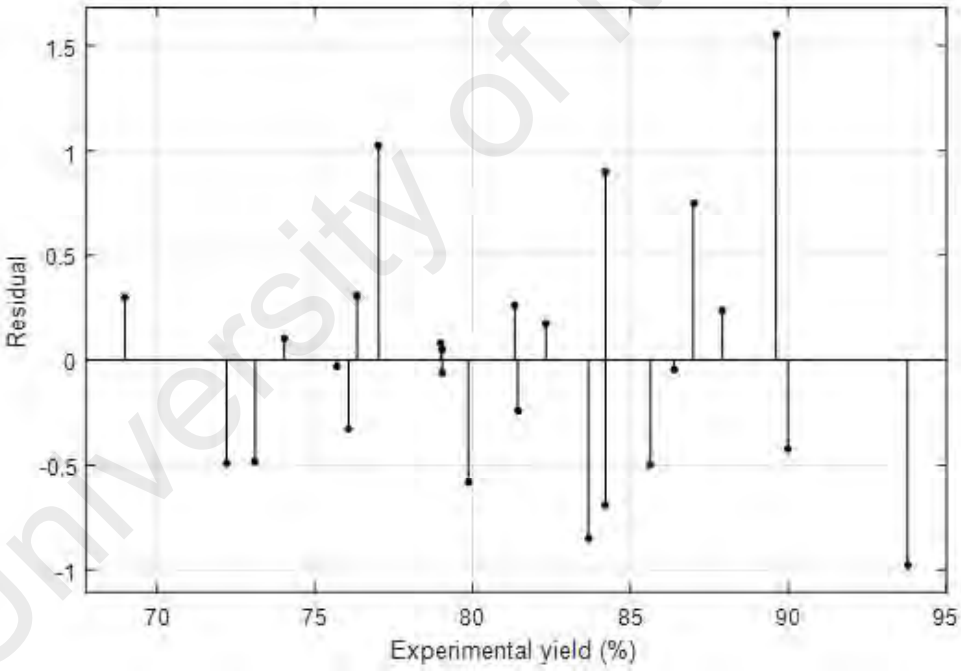


Figure 4.7: Correlation of UTC techniques using ANFIS: (a) experimental yield against predicted yield; (b) experimental yield against residual

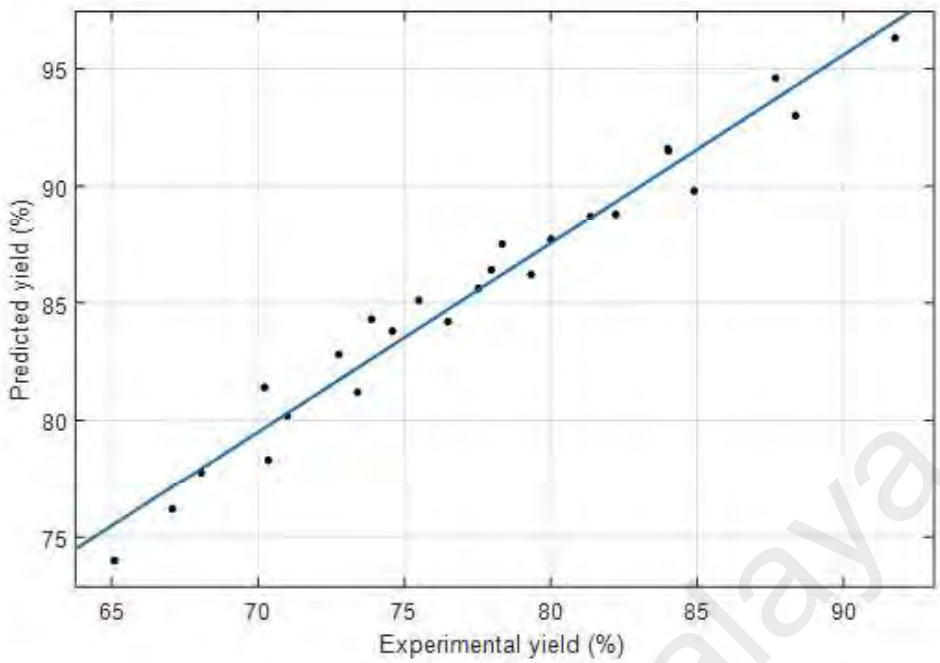
*(c) **Microwave-Assisted Conventional Transesterification (MTC)***

The results of the 25 experiments using MTC for CRB synthesis showed the value of R^2 (0.8567) and adjusted R^2 (0.8502), which indicated good fit of the model ($R^2 > 0.8$). The plot of the experimental against prediction values is depicted in Figure 4.8 (a), which affirmed the precision and accuracy of the model. Figure 4.8 (b) shows the plot of experimental against residual values (-3≥residual≤+3) which indicated that the model is good. Furthermore, the training data shows good compatibility with testing error value (e = 0.77).

*(d) **Stirring Hot-Plate-Assisted In situ Transesterification (HTI)***

The CRB yield results derived from 25 of experiments using HTI indicated the values of R^2 (0.9585) and adjusted R^2 (0.9567), which verified the fitness of the model ($R^2 > 0.8$). Figure 4.9 (a) shows the plot of experimental against prediction values, which affirmed the precision and accuracy of the model. Figure 4.9 (b) shows the plot of experimental against residual values (-3≥residual≤+3), which displayed the goodness of the model. Besides, the training data shows good compatibility with testing error value (e = 0.79).

(a)



(b)

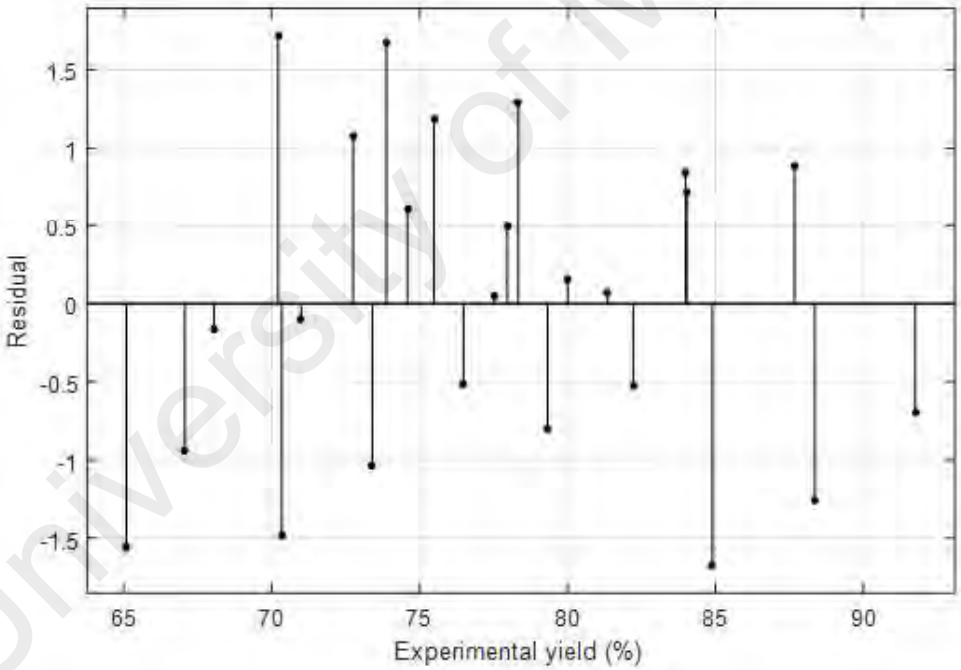


Figure 4.8: Correlation of MTC techniques using ANFIS: (a) experimental yield against predicted yield; (b) experimental yield against residual

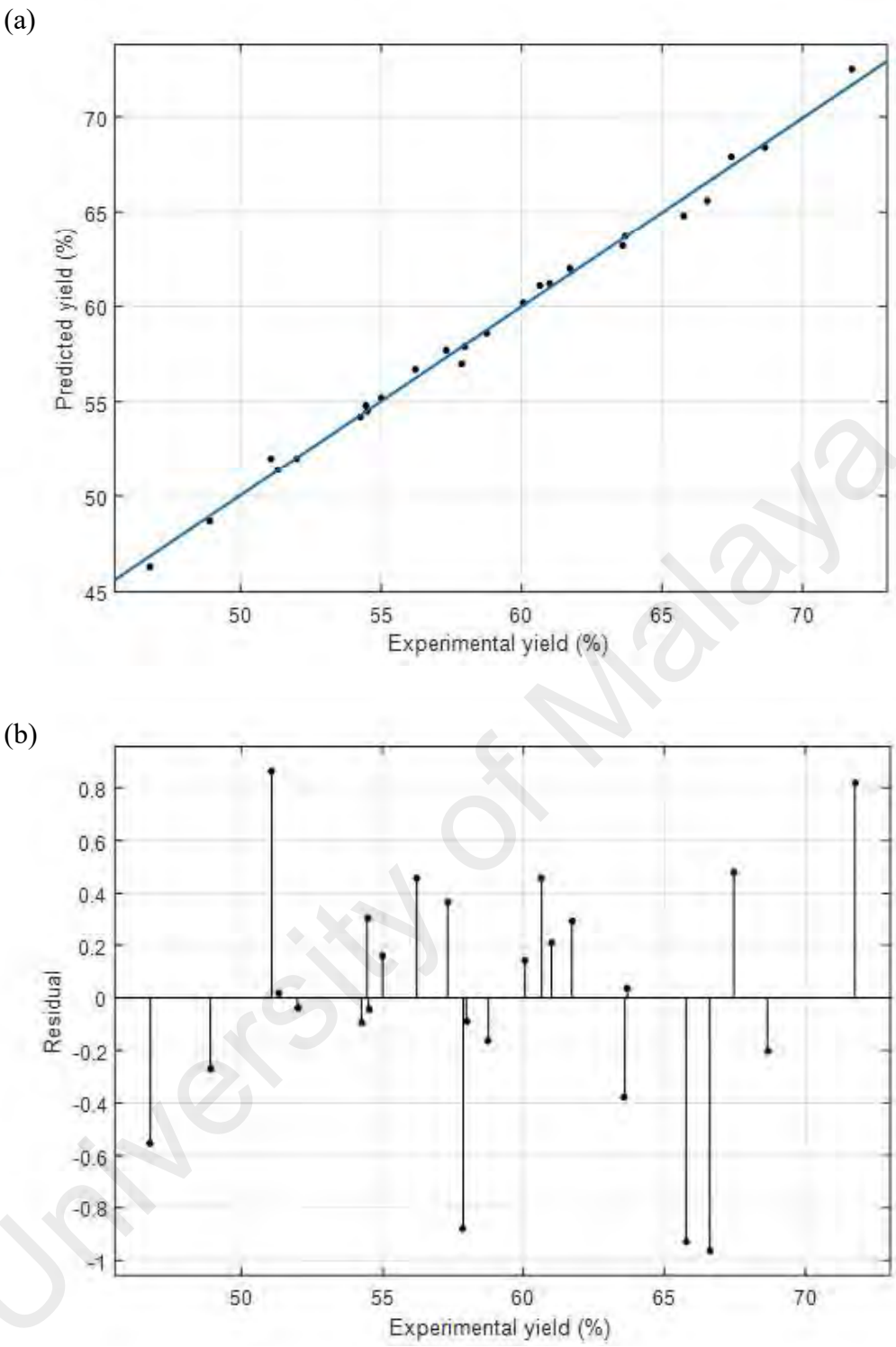


Figure 4.9: Correlation of HTI techniques using ANFIS: (a) experimental yield against predicted yield; (b) experimental yield against residual

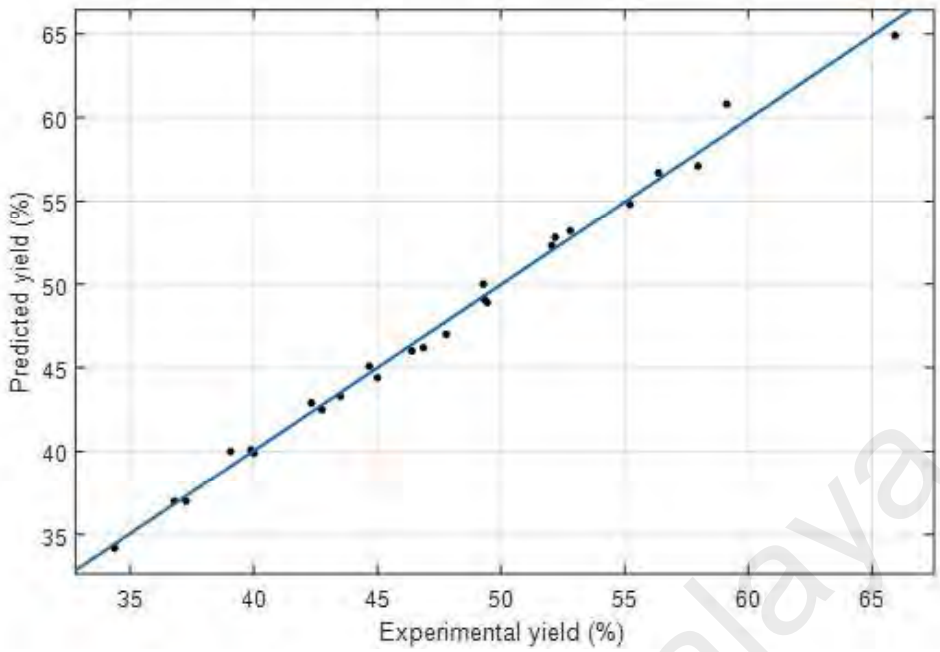
*(e) **Ultrasonic-Assisted In Situ Transesterification (UTI)***

The CRB yields for the 25 experiments by using UTI were carried out. In this study, the accuracy of the model was remarked by the calculated values of R^2 (0.9790) and adjusted R^2 (0.9780). The values ($R^2 > 0.8$), which were evinced R^2 and adjusted R^2 values of the model showed good fit of the model. Furthermore, the plot of the experimental against prediction values is shown in Figure 4.10 (a) and experimental against residual values (-3≥residual≤+3) in Figure 4.10 (b), which affirmed the precision and accuracy of the model. Further, the training data indicated good compatibility and less testing error was achieved (e = 0.79).

*(f) **Microwave-Assisted In Situ Transesterification (MTI)***

The CRB yields for the 25 experiments were observed using MTI. The accuracy of the used model was studied by perceiving the calculated values of R^2 (0.8608) and adjusted R^2 (0.8545), which indicated the good fit of the model ($R^2 > 0.8$). Next, Figure 4.11 (a) displays the plot of the experimental against prediction values, which verified the precision and accuracy of the model. Furthermore, in Figure 4.11 (b), the plot of experimental against residual values exhibited the goodness of the model (-3≥residual≤+3). In fact, the training data showed good compatibility with testing error value (e = 0.68).

(a)



(b)

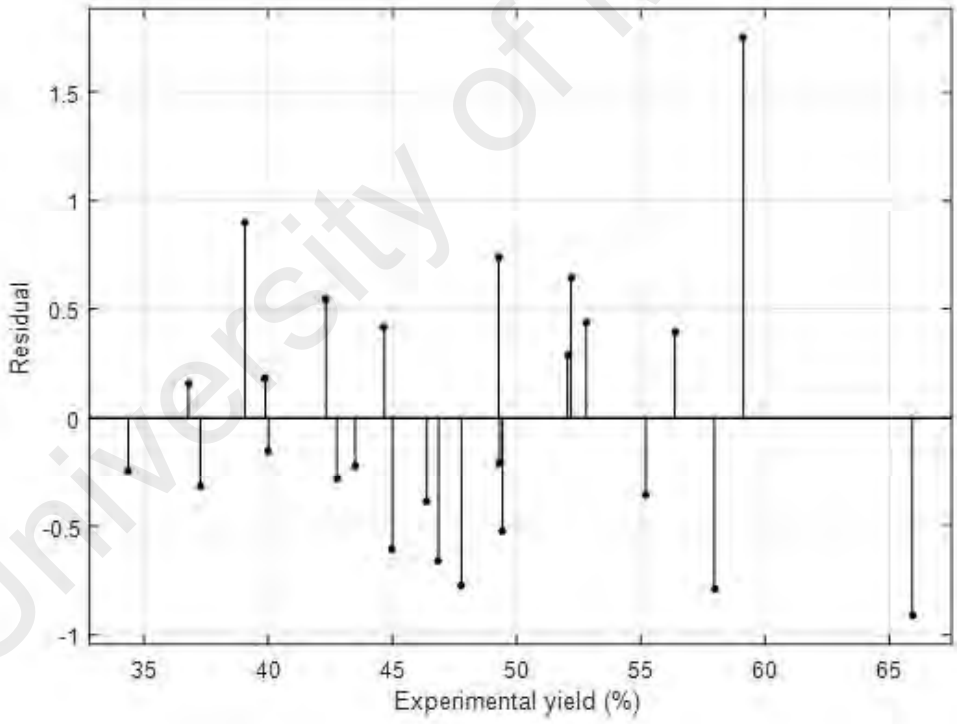
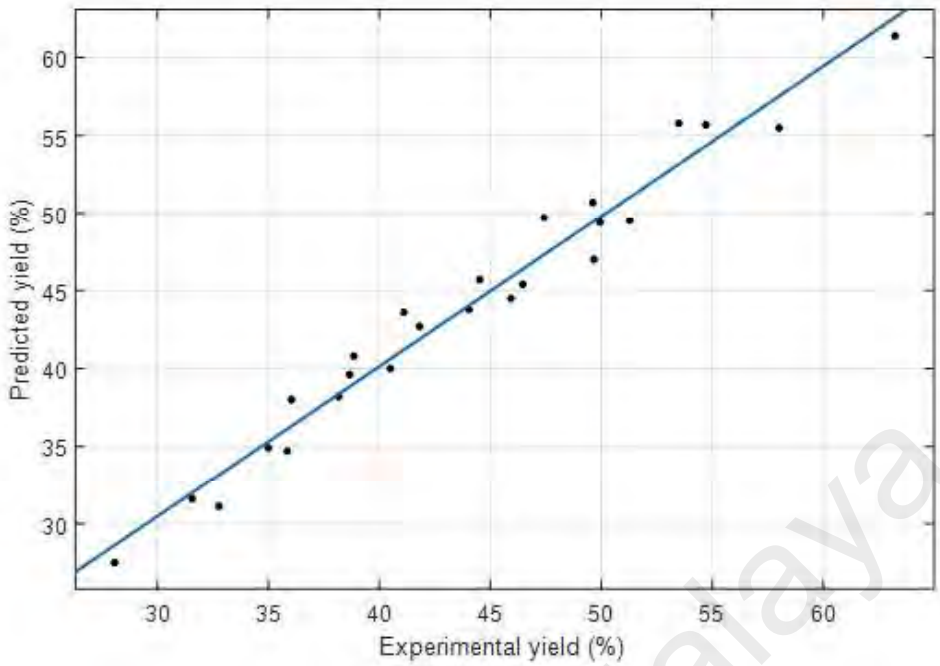


Figure 4.10: Correlation of UTI techniques using ANFIS: (a) experimental yield against predicted yield; (b) experimental yield against residual

(a)



(b)

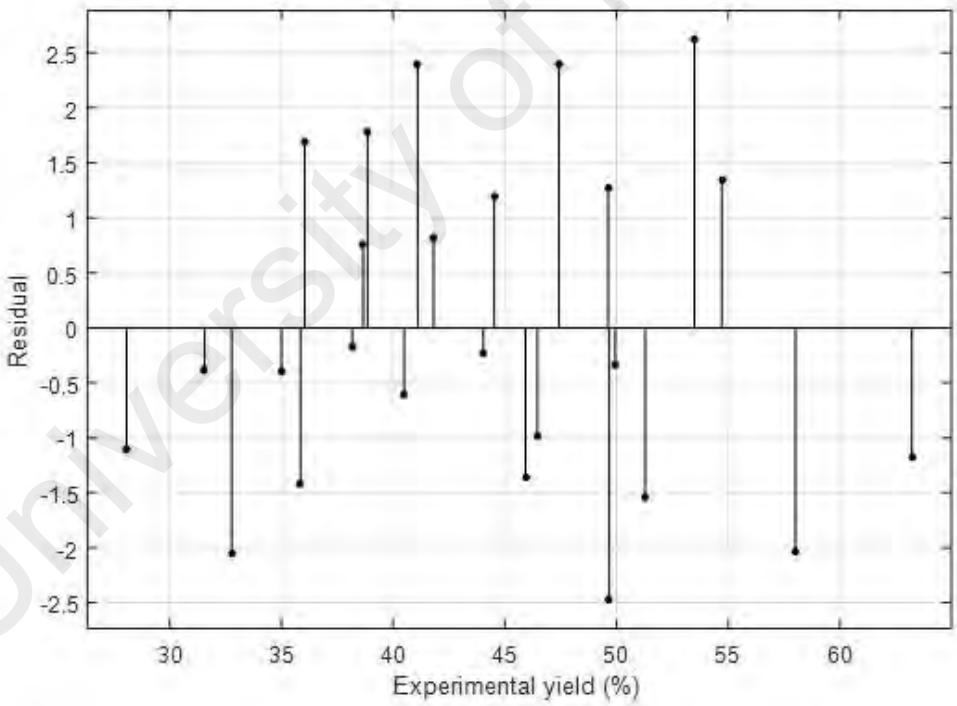


Figure 4.11: Correlation of MTI techniques using ANFIS: (a) experimental yield against predicted yield; (b) experimental yield against residual

4.3.2.2 Response Surface Methodology (RSM)

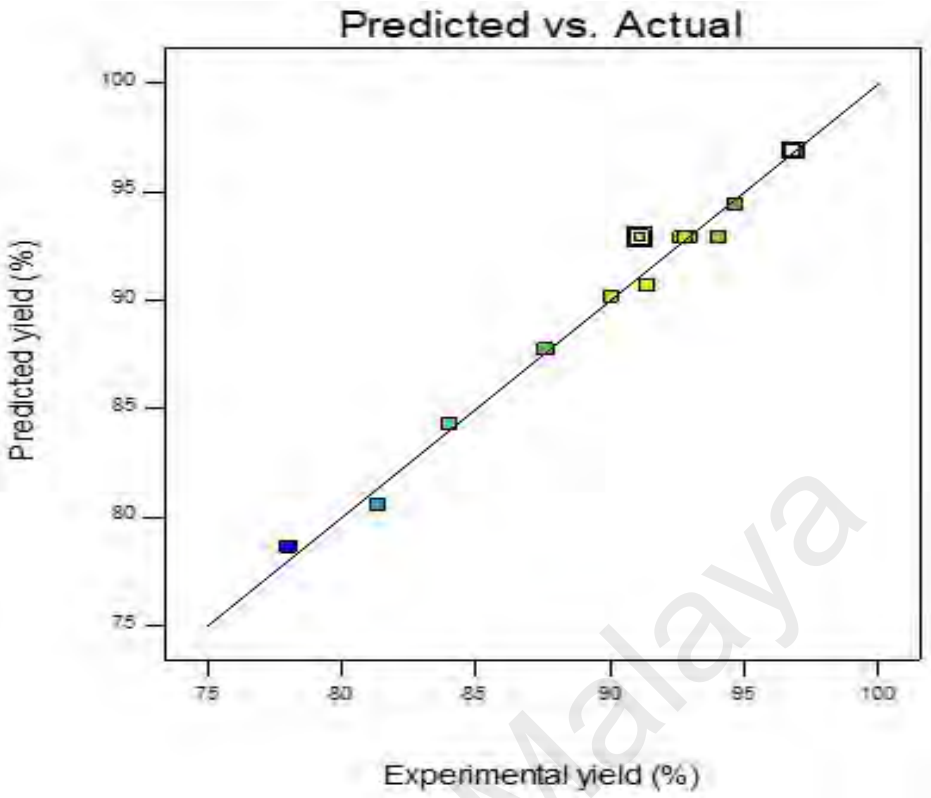
*(a) **Stirring Hot-Plate-Assisted Conventional Transesterification (HTC)***

The results in Table 4.13 indicated R^2 (0.9832), adjusted R^2 (0.9712), and predicted R^2 (0.9409), by which the HTC model development was good ($R^2 > 0.8$). The result also evinced that the models using solvent to oil ratio and catalyst percentage were significant (p-value ≤ 0.05). The insignificancy of lack of fit (p-value ≥ 0.5) showed the goodness of the model. In addition, the adequate precision value was 28.44, which indicated the signal to noise ratio. Normally, the ratio greater than 4 is desirable. Next, Figure 4.12 (a) displays the plot of the experimental against prediction values, which verified the precision and accuracy of the model. Furthermore, in Figure 4.12 (b), the plot of experimental against residual values exhibiting the model was good (-3≥residual≤+3).

Table 4.13: Analysis of variance of HTC using RSM

Source	SS	df	MS	F-value	P-value	Remarks
Model	365.07	5	73.01	81.90	< 0.0001	significant
A-Solvent	57.04	1	57.04	63.98	< 0.0001	
B-Catalyst	139.30	1	139.30	156.25	< 0.0001	
AB	0.27	1	0.27	0.30	0.5989	
A^2	2.27	1	2.27	2.55	0.1543	
B^2	156.09	1	156.09	175.09	< 0.0001	
Residual	6.24	7	0.89			
Lack of Fit	1.74	3	0.58	0.51	0.6943	Not significant
Pure Error	4.50	4	1.13			
Cor. total	371.31	12				
Std = 0.9442, mean = 89.82, C.V. % = 1.05, R^2 = 0.9832, adj. R^2 = 0.9712, pred. R^2 = 0.9409, adeq. precision = 28.44						

(a)



(b)

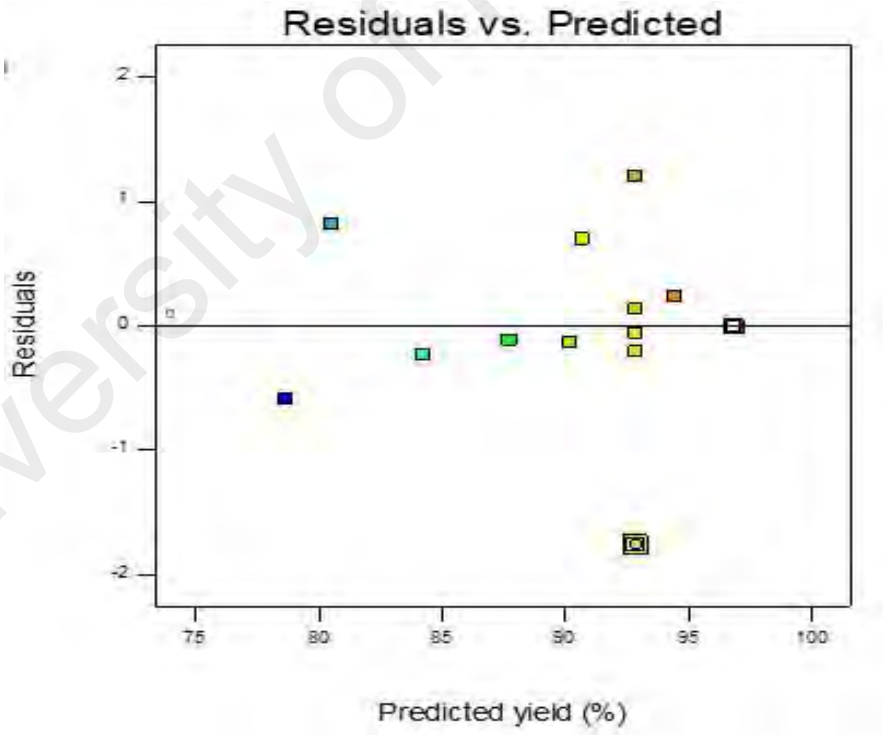


Figure 4.12: Correlation of HTC technique using RSM: (a) experimental yield against predicted yield; (b) experimental yield against residual

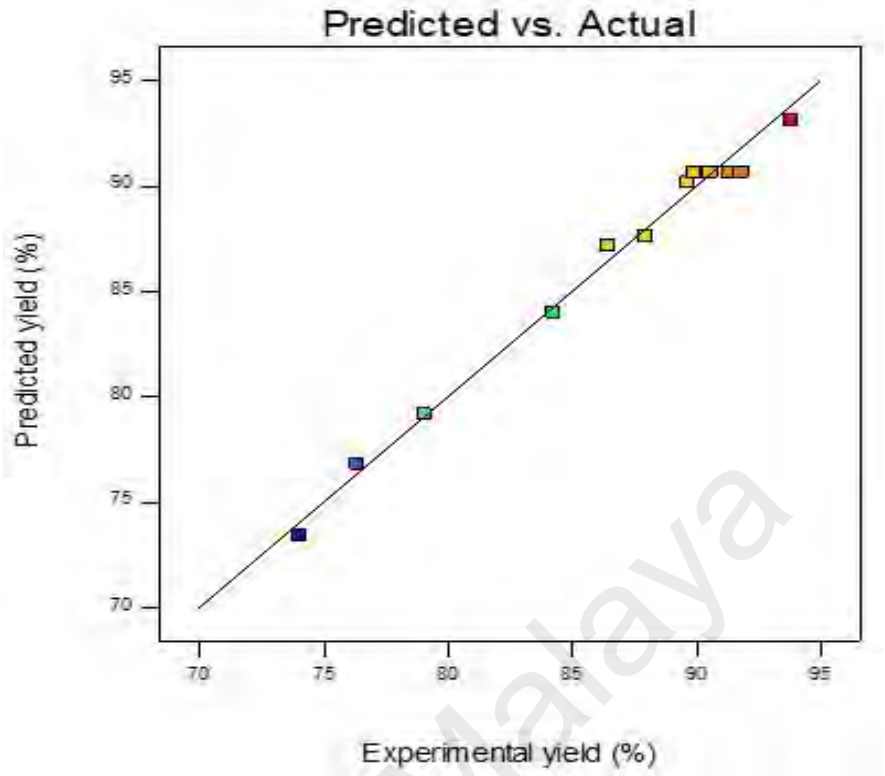
(b) ***Ultrasonic-Assisted Conventional Transesterification (UTC)***

The result in Table 4.14 shows R^2 (0.9900), adjusted R^2 (0.9828), and predicted R^2 (0.9469), by which the UTC model being developed was quite good ($R^2 > 0.8$). The results also depicted that the models using solvent to oil ratio and catalyst percentage were significant (p-value ≤ 0.05). The insignificancy of lack of fit (p-value ≥ 0.5) showed the goodness of the model. Next, the adequate precision value that was higher than 4 showed lack of noise with 35.15. Figure 4.13 (a) shows the plot of experimental against prediction values, which affirmed the precision and accuracy of the model. Figure 4.13 (b) shows the plot of experimental against residual values (-3≥residual≤+3) that displayed the goodness of the model.

Table 4.14: Analysis of variance of UTC using RSM

Source	SS	df	MS	F-value	P-value	Remarks
Model	470.10	5	94.02	138.30	< 0.0001	significant
A-Solvent	52.81	1	52.81	77.68	< 0.0001	
B-Catalyst	174.42	1	174.42	256.57	< 0.0001	
AB	0.04	1	0.04	0.06	0.8153	
A^2	0.71	1	0.71	1.05	0.3397	
B^2	197.83	1	197.83	291.00	< 0.0001	
Residual	4.76	7	0.68			
Lack of Fit	2.10	3	0.70	1.06	0.4605	Not significant
Pure Error	2.66	4	0.66			
Cor. total	474.86	12				
Std = 0.8245, mean = 86.52, C.V. % = 0.9529, R^2 = 0.9900, adj. R^2 = 0.9828, pred. R^2 = 0.9469, adeq. precision = 35.15						

(a)



(b)

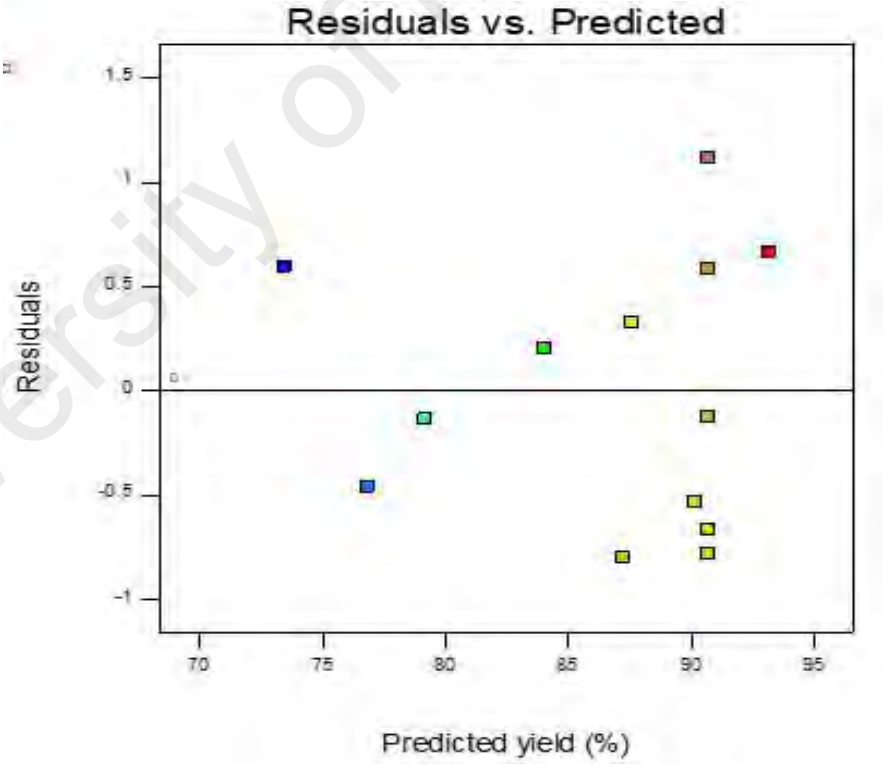


Figure 4.13: Correlation of UTC technique using RSM: (a) experimental yield against predicted yield; (b) experimental yield against residual

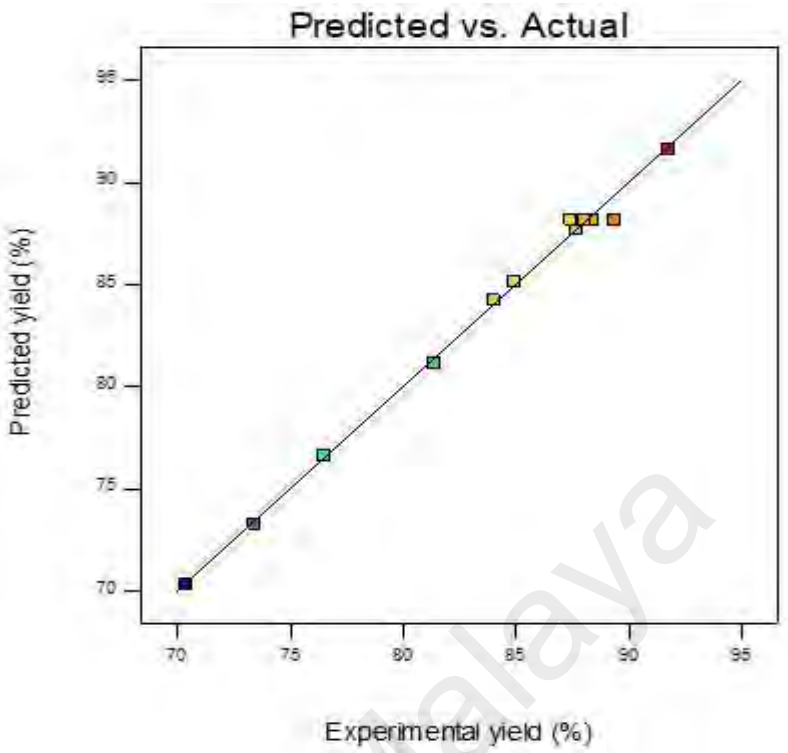
*(c) **Microwave-Assisted Conventional Transesterification (MTC)***

The results observed in Table 4.15 showed the R^2 (0.9957), adjusted R^2 (0.9926), and predicted R^2 (0.9900) of which the MTC model being developed was of great ($R^2 > 0.8$). In addition, the results depicted that the use of solvent to oil ratio and catalyst percentage in terms of experimental models was significant by which the p-value was less than 0.05. The insignificancy of lack of fit (p-value ≥ 0.5) indicated that the model was good. Furthermore, the adequate precision showed result higher than the desirable value (>4) with 55.05, which was quite good to indicate the lack of noise. Besides, the plot of the experimented against predicted values was shown in Figure 4.14 (a) and experimental against residual values (-3≥residual≤+3) in Figure 4.14 (b), which verified the precision and accuracy of the model.

Table 4.15: Analysis of variance of MTC using RSM

Source	SS	df	MS	F-value	p-value	Remarks
Model	524.76	5	104.95	323.69	< 0.0001	significant
A-Solvent	62.21	1	62.21	191.87	< 0.0001	
B-Catalyst	179.31	1	179.31	553.01	< 0.0001	
AB	0.01	1	0.01	0.03	0.8789	
A^2	0.10	1	0.10	0.32	0.5880	
B^2	245.89	1	245.89	758.37	< 0.0001	
Residual	2.27	7	0.32			
Lack of Fit	0.23	3	0.08	0.15	0.9241	Not significant
Pure error	2.04	4	0.51			
Cor. total	527.03	12				
Std = 0.5694, mean = 83.92, C.V. % = 0.6785, R^2 = 0.9957, adj. R^2 = 0.9926, pred. R^2 = 0.9900, adeq. precision = 55.05						

(a)



(b)

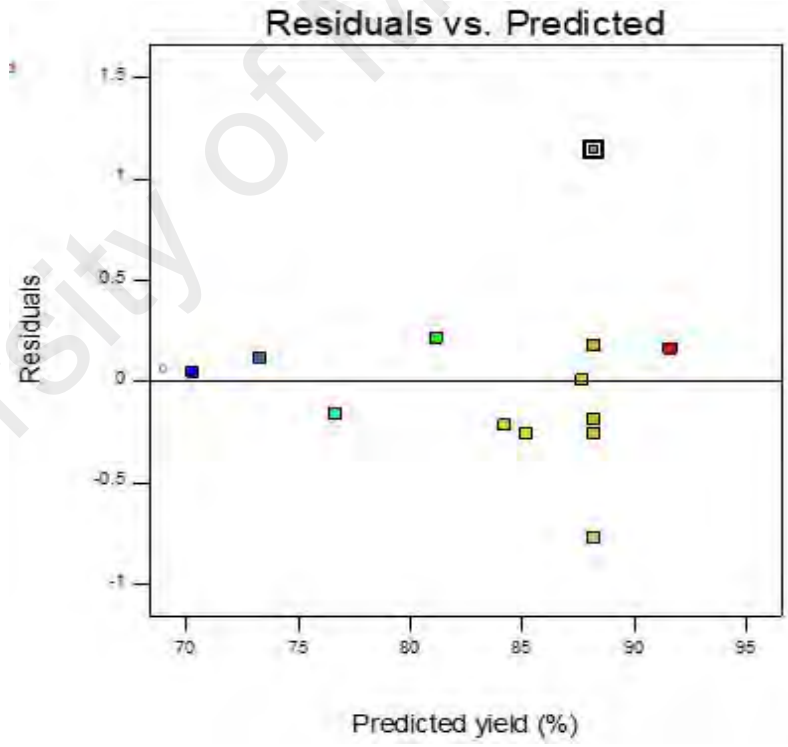


Figure 4.14: Correlation of MTC technique using RSM: (a) experimental yield against predicted yield; (b) experimental yield against residual

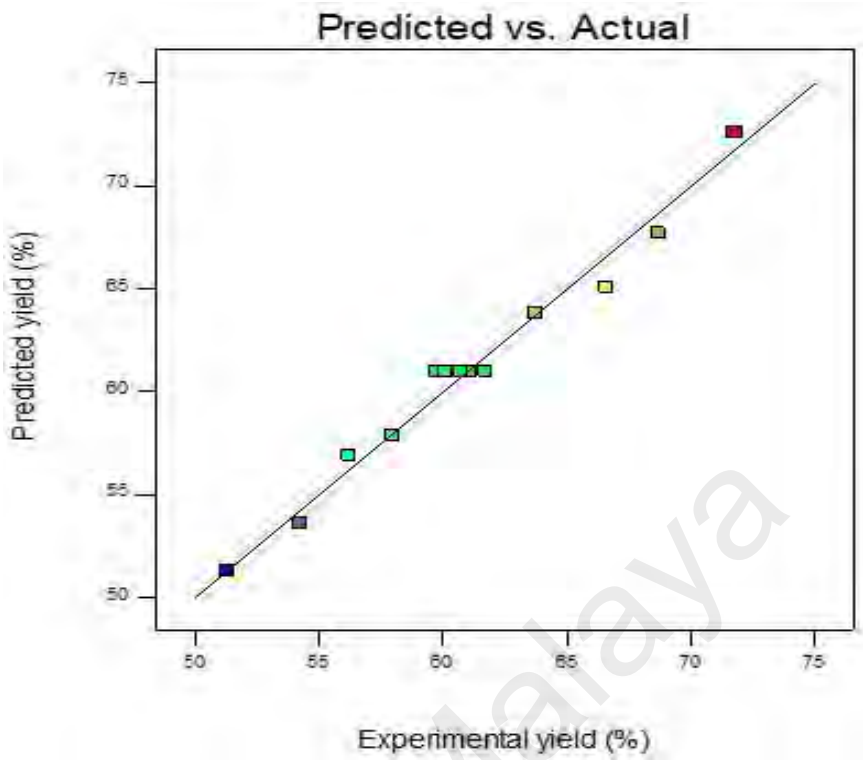
(d) ***Stirring Hot-Plate-Assisted In Situ Transesterification (HTI)***

From the results in Table 4.16, it was shown that the R^2 (0.9795), adjusted R^2 (0.9649), and predicted R^2 (0.8812) that the HTI model developed was quite good ($R^2 > 0.8$). In addition, it was mentioned that the use of solvent to oil ratio and solvent percentage in terms of experimental models were all significant (p-value ≤ 0.05). The insignificancy of lack of fit (p-value ≥ 0.5) showed the goodness of the model. Furthermore, the adequate precision value that was higher than 4 showed lack of noise with 29.47. Besides, Figure 4.15 (a) displays the plot of the experimental against prediction values, which verified the precision and accuracy of the model. Furthermore, in Figure 4.15 (b), the plot of experimental against residual values exhibited the goodness of the model (-3≥residual≤+3).

Table 4.16: Analysis of variance of HTI using RSM

Source	SS	df	MS	F-value	P-value	Remarks
Model	379.20	5	75.84	67.01	< 0.0001	significant
A-Solvent	77.69	1	77.69	68.64	< 0.0001	
B-Catalyst	298.21	1	298.21	263.49	< 0.0001	
AB	2.53	1	2.53	2.23	0.1787	
A^2	0.7044	1	0.70	0.62	0.4561	
B^2	0.3008	1	0.30	0.27	0.6221	
Residual	7.92	7	1.13			
Lack of Fit	5.42	3	1.80	2.88	0.1668	Not significant
Pure error	2.51	4	0.67			
Cor. total	5387.12	12				
Std = 1.0600, mean = 61.06, C.V. % = 1.74, R^2 = 0.9795, adj. R^2 = 0.9649, pred. R^2 = 0.8812, adeq. precision = 29.47						

(a)



(b)

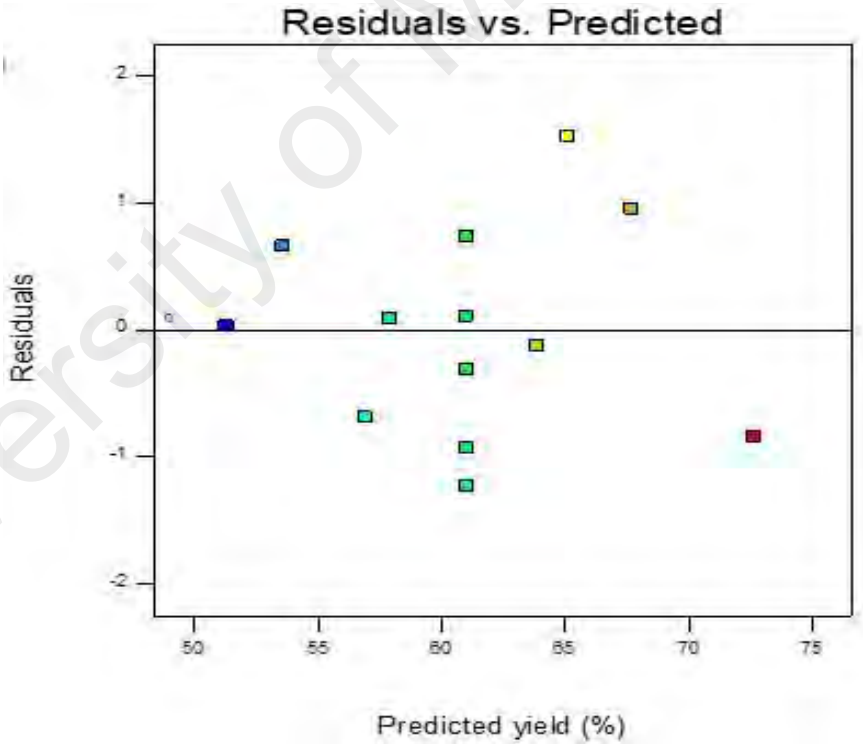


Figure 4.15: Correlation of HTI technique using RSM: (a) experimental yield against predicted yield; (b) experimental yield against residual

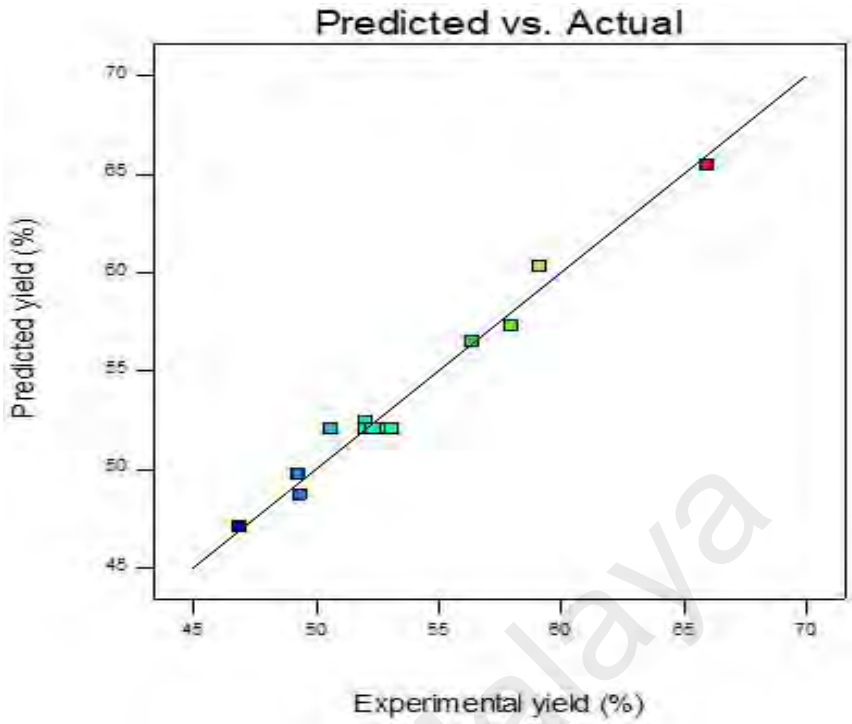
*(e) **Ultrasonic-Assisted In Situ Transesterification (UTI)***

The results in Table 4.17 revealed the values of R^2 (0.9787), adjusted R^2 (0.9635), and predicted R^2 (0.89), by which the UTI model being developed was good ($R^2 > 0.8$). In addition, the use of solvent to oil ratio and catalyst percentage in terms of experimental models was all significant (p-value ≤ 0.05). The insignificancy of lack of fit (p-value ≥ 0.5) showed that the model was good. Furthermore, the adequate precision showing result higher than desirable value (>4) with 27.89 was quite good to show lack of noise. The plot of the experimental against prediction values depicted in Figure 4.16 (a) affirmed the precision and accuracy of the model. Figure 4.16 (b) shows the plot of experimental against residual values (-3≥residual≤+3), which indicated that the model was good.

Table 4.17: Analysis of variance of UTI using RSM

Source	SS	df	MS	F-value	P-value	Remarks
Model	301.51	5	60.30	64.32	< 0.0001	significant
A-Solvent	68.34	1	68.34	72.90	< 0.0001	
B-Catalyst	201.72	1	201.72	215.18	< 0.0001	
AB	1.93	1	1.93	2.06	0.1942	
A^2	3.09	1	3.09	3.29	0.1125	
B^2	16.67	1	16.67	17.79	0.0039	
Residual	6.56	7	0.94			
Lack of Fit	2.93	3	0.98	1.08	0.4537	Not significant
Pure error	3.63	4	0.91			
Cor. total	308.07	12				
Std = 0.9682, mean = 53.67, C.V. % = 1.80, R^2 = 0.9787, adj. R^2 = 0.9635, pred. R^2 = 0.8908, adeq. precision = 27.89						

(a)



(b)

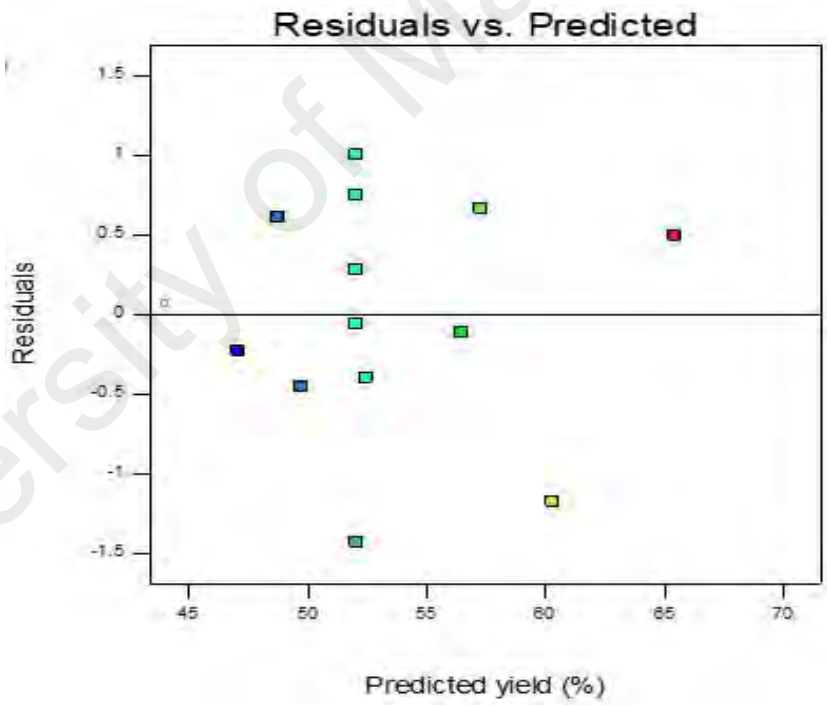


Figure 4.16: Correlation of UTI technique using RSM: (a) experimental yield against predicted yield; (b) experimental yield against residual

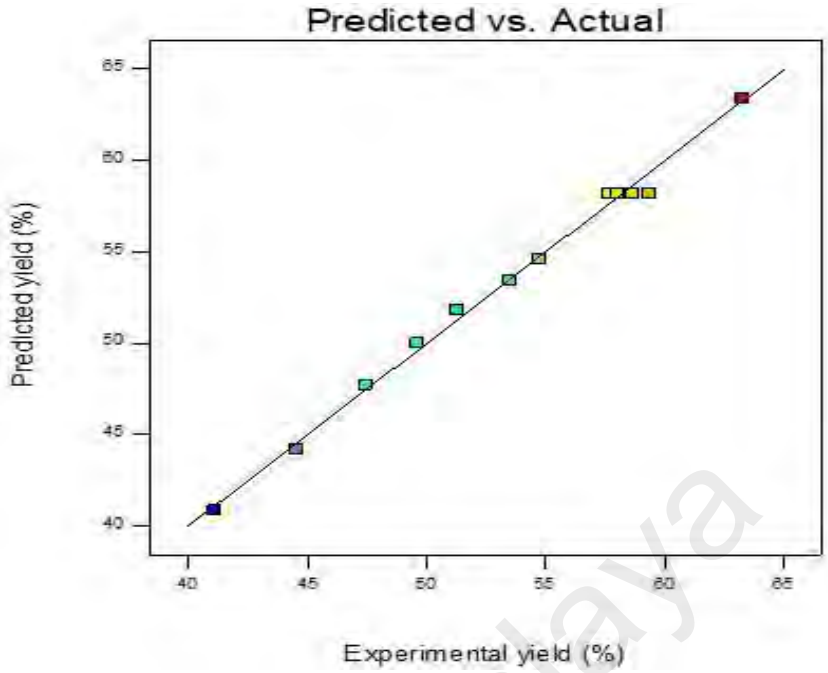
(f) Microwave-Assisted In Situ Transesterification (MTI)

In Table 4.18, it depicted the R^2 (0.9948), adjusted R^2 (0.9910), and predicted R^2 (0.9822), by which the UTC model being developed was quite good ($R^2 > 0.8$). The results also indicated that the models using solvent to oil ratio and catalyst percentage was significant (p-value ≤ 0.05). The insignificancy of lack of fit (p-value ≥ 0.5) indicated that the model was good. Furthermore, the adequate precision showed result higher than the desirable value (>4) with 53.88, which was quite good to indicate lack of noise. Next, Figure 4.17 (a) displays the plot of the experimental against prediction values, which verified the precision and accuracy of the model and Figure 4.17 (b) shows the plot of experimental against residual values exhibited the goodness of the model (-3≥residual≤+3).

Table 4.18: Analysis of variance of MTI using RSM

Source	SS	df	MS	F-value	P-value	Remarks
Model	503.37	5	100.67	265.83	< 0.0001	significant
A-Solvent	198.72	1	198.72	524.72	< 0.0001	
B-Catalyst	7.80	1	7.80	20.59	< 0.0001	
AB	1.21	1	1.21	3.19	0.1170	
A^2	0.96	1	0.96	2.54	0.1548	
B^2	240.19	1	240.19	634.22	<0.0001	
Residual	2.65	7	0.38			
Lack of Fit	0.86	3	0.29	0.64	0.6270	Not significant
Pure error	1.79	4	0.45			
Cor. total	506.03	12				
Std = 0.6200, mean = 53.6200, C.V. % = 1.15, R^2 = 0.9948, adj. R^2 = 0.9910, pred. R^2 = 0.9822 adeq. precision = 53.88						

(a)



(b)

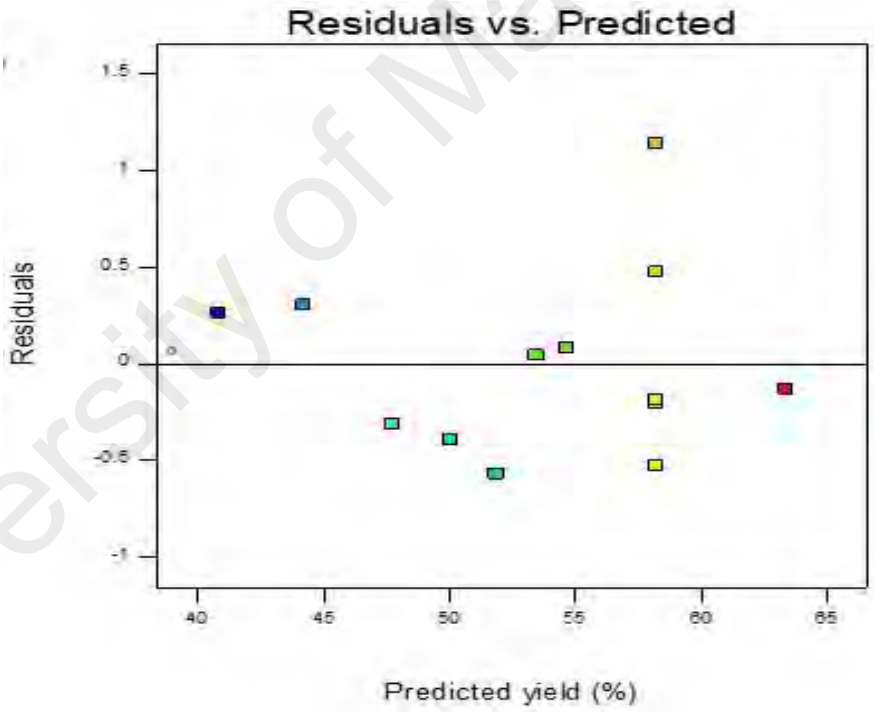


Figure 4.17: Correlation of MTI technique using RSM: (a) experimental yield against predicted yield; (b) experimental yield against residual

4.3.2.3 Comparison of ANFIS and RSM

In order to ensure that the ANFIS and RSM results were comparable, additional analysis for ANFIS was run using developed experiments by RSM. The results for experimental yield, ANFIS and RSM values were depicted in Table 4.19. The coefficient of determination was used to verify the accuracy of both ANFIS and RSM performance. From the results, the R^2 values for HTC, UTC, MTC, HTI, UTI, and MTI were 0.9863, 0.9944, 0.9961, 0.9935, 0.9882, and 0.9964 respectively and for RSM R^2, the values were 0.9832, 0.9900, 0.9967, 0.9795, 0.9787, and 0.9948 respectively. Even though the results showed that both of the models fitted well for experiments; however, the R^2 values for ANFIS was slightly higher compared to RSM. Besides, both models could predict the experimental data. However, the prediction ability of ANFIS was quite good because it indicated the residual values lower as compared to RSM, by which it was in agreement with the previous studies. This might be due to the ANFIS model being constituted by the integration of both fuzzy logic and neural network. Besides artificial neural network (ANN), the ANFIS is capable of estimating non-linear functions compared to RSM that is only limited to second-order polynomial (Salamatinia *et al*., 2010; Rajkumar *et al*., 2011; Yang & Enthchev, 2014; Mostafei *et al*., 2016; Hasni *et al*., 2017; Ighose *et al*., 2017).

Table 4.19: Optimization comparison between ANFIS and RSM

Order	Solvent to oil ratio (mol)	Catalyst concentration (%)	HTC					UTC				
			Experimetal yield (%)	ANFIS prediction		RSM prediction		Experimental yield (%)	ANFIS prediction		RSM prediction	
				Yield (%)	Residual	Yield (%)	Residual		Yield (%)	Residual	Yield (%)	Residual
1	3	0.5	84.03	84.00	0.03	84.26	-0.23	79.03	79.00	0.03	79.17	-0.14
2		1.0	96.85	96.80	0.05	96.86	-0.01	93.79	93.80	-0.01	93.12	0.67
3		1.5	94.66	94.70	-0.04	94.42	0.24	89.62	89.60	0.02	90.15	-0.53
4	6	0.5	81.35	81.30	0.05	80.53	0.82	76.35	76.30	0.05	76.81	-0.46
5		1.0	93.00	92.70	0.30	92.87	0.13	90.00	90.70	-0.70	90.66	-0.66
6		1.0	92.81	92.70	0.11	92.87	-0.06	91.78	90.70	1.08	90.66	1.12
7		1.0	91.11	92.70	-1.59	92.87	-1.76	90.54	90.70	-0.16	90.66	-0.12
8		1.0	92.66	92.70	-0.04	92.87	-0.21	91.25	90.70	0.55	90.66	0.59
9		1.0	94.07	92.70	1.37	92.87	1.20	89.88	90.70	-0.82	90.66	-0.78
10		1.5	90.04	90.00	0.04	90.17	-0.13	87.92	87.90	0.02	87.59	0.33
11	9	0.5	78.03	78.00	0.03	78.62	-0.59	74.03	74.00	0.03	73.43	0.60
12		1.0	91.39	91.40	-0.01	90.69	0.70	86.39	86.40	-0.01	87.19	-0.80
13		1.5	87.62	87.60	0.02	87.73	-0.11	84.22	84.20	0.02	84.02	0.20
R^2			0.9879	0.9863		0.9832		0.9944	0.9944		0.9900	
Adj. R^2			0.9636	0.9814		0.9712		0.9832	0.9913		0.9828	

Table 4.19, continued

Order	Solvent to oil ratio (mol)	Catalyst concentration (%)	MTC					HTI				
			Experimetal yield (%)	ANFIS prediction		RSM prediction		Experimental yield (%)	ANFIS prediction		RSM prediction	
				Yield (%)	Residual	Yield (%)	Residual		Yield (%)	Residual	Yield (%)	Residual
1	3	0.5	76.49	76.50	-0.01	76.65	-0.16	56.22	56.20	0.02	56.91	-0.69
2		1.0	91.76	91.80	-0.04	91.60	0.16	66.61	66.60	0.01	65.08	1.53
3		1.5	87.68	87.70	-0.02	87.68	0.00	71.76	71.80	-0.04	72.60	-0.84
4	6	0.5	73.40	73.40	0.00	73.28	0.12	54.26	54.30	-0.04	53.60	0.66
5		1.0	88.36	88.20	0.16	88.19	0.17	60.05	60.70	-0.65	60.98	-0.93
6		1.0	88.00	88.20	-0.20	88.19	-0.19	61.72	60.70	1.02	60.98	0.74
7		1.0	87.41	88.20	-0.79	88.19	-0.78	61.09	60.70	0.39	60.98	0.11
8		1.0	89.33	88.20	1.13	88.19	1.14	60.67	60.70	-0.03	60.98	-0.31
9		1.0	87.93	88.20	-0.27	88.19	-0.26	59.75	60.70	-0.95	60.98	-1.23
10		1.5	84.00	84.00	0.00	84.22	-0.22	68.66	68.70	-0.04	67.70	0.96
11	9	0.5	70.35	70.30	0.05	70.30	0.05	51.33	51.30	0.03	51.30	0.03
12		1.0	84.90	84.90	0.00	85.16	-0.26	57.98	58.00	-0.02	57.89	0.09
13		1.5	81.36	81.40	-0.04	81.15	0.21	63.69	63.70	-0.01	63.81	-0.12
R^2			0.9961	0.9961		0.9957		0.9935	0.9935		0.9795	
Adj. R^2			0.9884	0.9940		0.9926		0.9806	0.9900		0.9649	

Table 4.19, continued

Order	Solvent to oil ratio (mol)	Catalyst concentration (%)	UTI					MTI				
			Experimetal yield (%)	ANFIS prediction		RSM prediction		Experimental yield (%)	ANFIS prediction		RSM prediction	
				Yield (%)	Residual	Yield (%)	Residual		Yield (%)	Residual	Yield (%)	Residual
1	3	0.5	65.93	65.90	0.03	65.43	0.50	54.71	54.70	0.01	54.62	0.09
2		1.0	56.37	56.40	-0.03	56.48	-0.11	63.23	63.20	0.03	63.36	-0.13
3		1.5	52.05	52.00	0.05	52.44	-0.39	53.49	53.50	-0.01	53.44	0.05
4	6	0.5	59.13	59.10	0.03	60.30	-1.17	49.62	49.60	0.02	50.01	-0.39
5		1.0	52.33	52.20	0.13	52.05	0.28	58.01	59.30	-0.29	58.20	-0.19
6		1.0	51.99	52.20	0.21	52.05	-0.06	57.99	58.30	-0.31	58.20	-0.21
7		1.0	50.62	52.20	-1.58	52.05	-1.43	58.67	58.30	0.37	58.20	0.47
8		1.0	52.80	52.20	0.60	52.05	0.75	59.34	58.30	1.04	58.20	1.14
9		1.0	53.05	52.20	0.85	52.05	1.00	57.67	58.30	-0.63	58.20	-0.53
10		1.5	49.32	49.30	0.02	48.71	0.61	47.82	47.80	0.02	47.73	-0.31
11	9	0.5	57.96	58.00	-0.04	57.29	0.67	44.52	44.50	0.02	44.21	0.31
12		1.0	49.28	49.30	-0.02	49.73	-0.45	51.28	51.30	-0.02	51.85	-0.57
13		1.5	46.86	46.90	-0.04	47.08	-0.22	41.10	41.10	0.00	40.83	0.27
R^2			0.9882	0.9882		0.9787		0.9964	0.9964		0.9948	
Adj. R^2			0.9646	0.9817		0.9635		0.9893	0.9945		0.9910	

4.3.3 Effect of Different Methods

Figure 4.18 depicts CRB yield using different methods and techniques: HTC, UTC, MTC, HTI, UTI, and MTI. The results were compared. From the results, it was displayed that transesterification using conventional method gave quite high CRB yield compared to the in-situ method. The difference in optimal values for stirring hot-plate-assisted transesterification (HT), ultrasonic-assisted transesterification (UT), and microwave-assisted transesterification (MT) between the conventional and in-situ methods were approximately 25.09%, 27.86%, and 28.53% respectively. The low CRB yield synthesised from the in-situ method might be due to extra effort that was taken to extract oil and converting to biodiesel simultaneously. The low biodiesel yield derived from in-situ is might due to methanol effect which regard as poor oil extraction (Georgogianni *et al*., 2008; Guldhe *et al*., 2014). Meanwhile, the introduced hexane does not disturbed the conversion reaction as reported by previous studies. (Georgogianni *et al*., 2008, Rahimi *et al*., 2018)

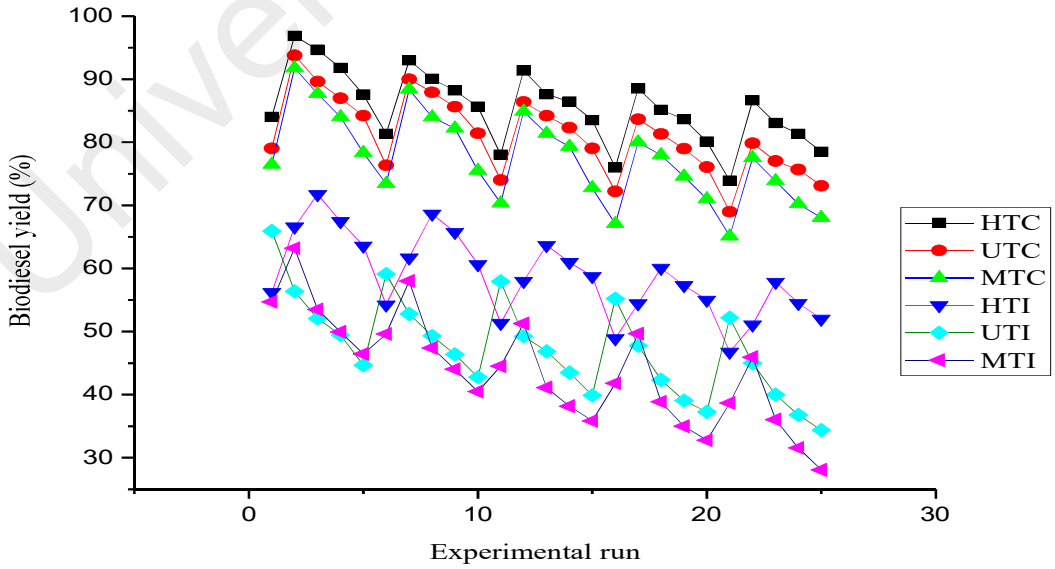


Figure 4.18: Comparison of biodiesel yield between conventional and in-situ method

4.3.4 Effect of Different Techniques

The comparisons of CRB yield between HT, UT, and MT transesterification techniques are shown in Figure 4.19. Results observed optimal value of 96.85% of CRB yield when using HT technique (3:1 solvent to oil ratio, 1% catalyst to oil). Even though the study employed two different methods (conventional and in-situ), the results for HT still displayed the highest CRB yield among the techniques for each method. On the other hand, MT showed the lowest and UT with moderate CRB yield result.

Table 4.20 depicts the summary of comparisons between transesterification techniques. HT technique by using heat transferring from the plate to the reactant from outside to inside and stirring ability instigated the conversion reaction of CRO to CRB (Teixira *et al*., 2009; Da Porto *et al*., 2013). Further, the UT with its ultrasound irradiation produced cavitational impact and at the same time, it enhanced the reactant surface to facilitate the transesterification. For MT, the microwave power affected the molecules polarity and ions (alcohol) by altering the magnetic field. In addition, due to high frequency, vibration that caused the molecules to collide and heating occurred from rapid rotation, the reaction of separation methyl ester from glycerol occurred (Patil *et al*., 2011; Sajjadi *et al*., 2014). Furthermore, HT consists of ability in synthesising biodiesel in high scale compared to UT and MT. In addition, UT contains more advantages such as low-cost production, short time reaction, and can run a few experiments simultaneously without affecting the quantity and quality of yield. In terms of disadvantages, MT is not recommended since it produces the lowest biodiesel yield, hard to control, and it is not possible to imply in large scale industry (Veljković *et al*., 2012; Badday *et al*., 2013; Salamatinia *et al*., 2013; Vinatoru *et al*., 2017)

Table 4.21 shows the comparison of biodiesel yield using coconut oil with different feedstock from the previous study. Most of the feedstock in the table was commercially

produced in different countries. The study revealed that coconut oil produced relatively high biodiesel yield (98.00%) but slightly lower compared to waste vegetable oil (100.00%), soybean oil (99.00%), and castor oil (99.00%) compared to the other feedstock. However, cottonseed oil showed a similar result as coconut oil for producing biodiesel yield with 98.00%, even though both were synthesised using different transesterification methods. However, the data showed that biodiesel is derived from microalgal (*Scenedemus* sp.), which is quite low as compared to others (51.94 – 71.37%). In brief, it can be noticed that differences in transesterification methods and techniques do give different results of biodiesel conversion yield (Karmakar *et al*., 2010; Kumar *et al*., 2010; Demirbas & Demirbas, 2011; Hsiao *et al*., 2011; Barekati-Goudarzi *et al*., 2017).

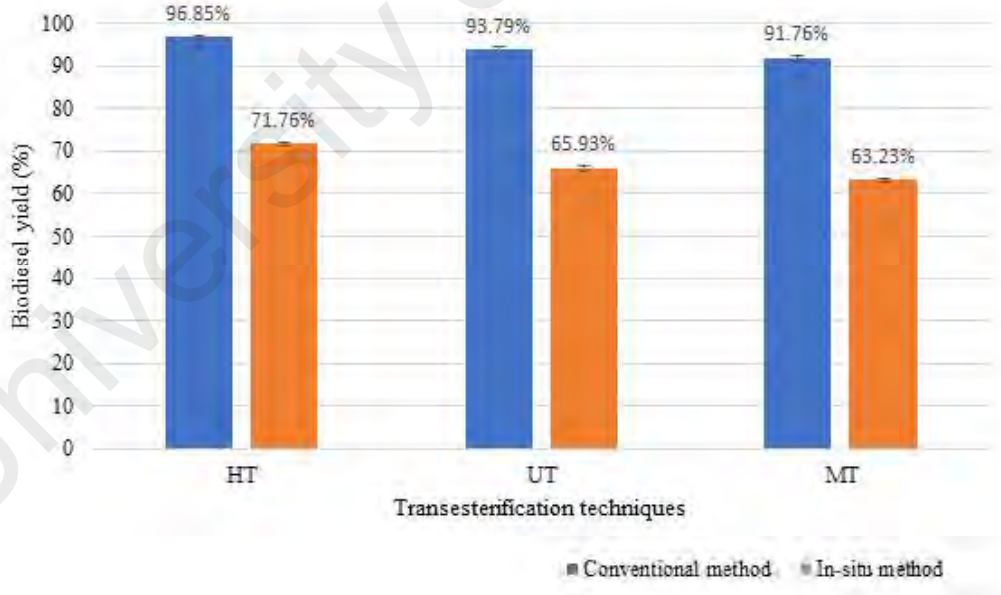


Figure 4.19: Biodiesel yield comparison between different techniques: HT, UT and MT

Table 4.20: Comparison of transesterification using different techniques: HT, UT and MT

Characteristics	Transesterification techniques		
	Stirring hot-plate-assisted (HT)	Ultrasonic-assisted (UT)	Microwave-assisted (MT)
Reaction interaction	a. Heat transfer from outside to inside. b. Stirring enhanced the reaction.	a. Ultrasound waves produced cavity effect and escalated reaction process. b. Optimizing reactant surface.	a. Microwave irradiation affecting molecules of polar and alcohol ions by altering magnetic field. b. Molecules collided due to high frequency of vibration. c. Heating occurred due to rapid rotation.
Sample size	Large	Moderate	Small
Yield (%)	High	Moderate	Low
Solvent to oil molar ratio (v/w)	3:1	3:1	3:1
Catalyst concentration	Conventional - 1 % In-situ – 1.5%	Conventional - 1 % In-situ – 0.5%	Conventional - 1 % In-situ – 1%
Advantages	Can conduct large amount of transesterification, easily controllable.	Fast reaction, can run a few experiments simultaneously without negative impact, economical.	Fast reaction, consistent.
Disadvantages	Long reaction time	Consumed time to produce heat	Hard to control, not possible to apply in large scale industry.

Source: Teixira *et al*., 2009; Patil *et al*., 2011; Veljković *et al*., 2012; Badday *et al*., 2013; Da Porto *et al*., 2013; Salamatinia *et al*., 2013; Sajjadi *et al*., 2014; Vinatoru *et al*., 2017

Table 4.21: Comparison of biodiesel yield with other sources

Feedstock	Biodiesel generation	Method	Technique	Biodiesel yield (%)
Coconut oil	1	Conventional	Ultrasonic	98.00
Soybean oil	1	Conventional	Ultrasonic	99.00
Soybean oil	1	Conventional	Mechanical stirring	97.00
Castor oil	1	Conventional	Ultrasonic	99.00
Rapeseed oil	1	Conventional	Mechanical stirring	96.00
Sunflower seed oil	1	Conventional	Ultrasonic	90.00
Waste vegetable oil	2	Conventional	Microwave	100.00
Waste cooking oil	2	Conventional	Ultrasonic	96.78
Used frying oil	2	Conventional	Mechanical stirring	85.30
Algae oil	3	Conventional	Mechanical stirring	94.27
Sunflower seed	1	*In situ*	Ultrasonic	97.00
Sunflower seed	1	*In situ*	Mechanical stirring	97.00
Cotton seed	1	*In situ*	Ultrasonic	98.00
Microalgal (*Scenedemus* sp.)	3	*In situ*	Microwave	51.94
Micralgal (*Scenedemus* sp.)	3	*In situ*	Ultrasonic	71.37
Microalgal (*Nannochloropsis* sp.)	3	*In situ*	Microwave	80.13

Source: Karmakar *et al*., 2010; Kumar *et al*., 2010; Demirbas & Demirbas, 2011; Hsiao *et al*., 2011; Barekati-Goudarzi *et al*., 2017

4.3.5 Effect of Solvent Molar Ratio

Figure 4.20 shows the effects of the solvent to oil molar ratio (v/w) for all transesterification techniques: HTC, UTC, MTC, HTI, UTI, and MTI. From the results, it was shown that the optimum solvent to oil molar ratio was 3:1 as validated by all techniques. The results depicting 3:1 solvent to oil ratio synthesised the highest CRB yield for HTC, UTC, MTC, HTI, UTI, and MTI with 96.85%, 93.79%, 91.76%, 71.76%, 65.93%, and 63.23% respectively. On the other hand, the results displayed that if the solvent ratio increased, the biodiesel yield showed a reversed trend. The results were supported by a previous study, which stated that solvent to oil ratio is an important parameter since it gives major effect in producing biodiesel because low ratio could cause the transesterification reaction to be incomplete whereas if the ratio is too high, it would decrease the yield (Salaminiti *et al*., 2013; Sajjadi *et al*., 2014). The usage of methanol as solvent for transesterification was suggested by the previous study because of its reactivity characteristic, but excessive ratio could lower the yield because of the solubility of glycerol that is affected by increasing polarity, which makes the separation process of methyl ester become harder (Badday *et al*., 2013; Vinatoru *et al*., 2017).

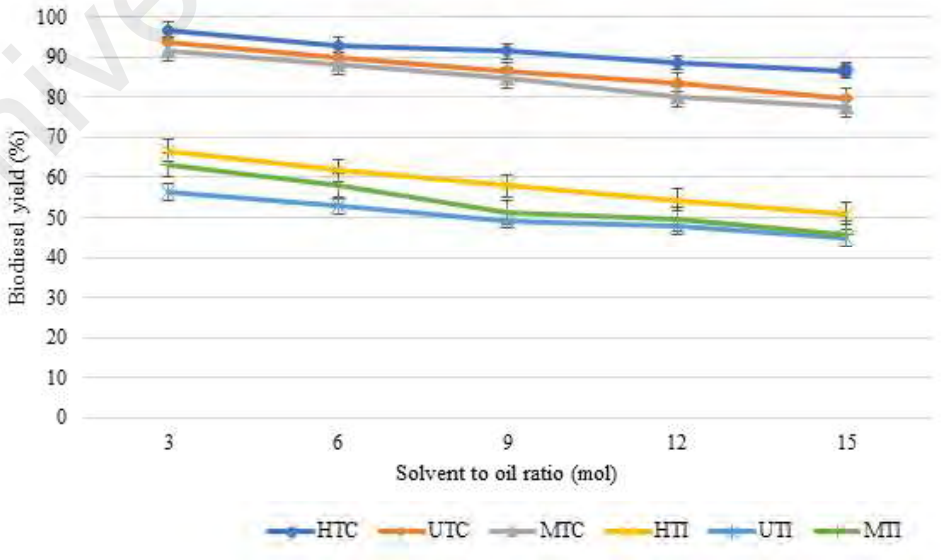


Figure 4.20: Effect of solvent to sample molar ratio (1% catalyst)

4.3.6 Effect of Catalyst Concentration

The employment of catalyst concentration as parameter in synthesis of biodiesel is important because the percentage of catalyst could affect the transesterification by which it could cause saponification reaction to occur (Demirbas, 2008). Figure 4.21 depicts the effect of catalyst concentration used in the experiment. The results displayed consistency of optimal CRB yield for techniques using conventional method by employing 1% catalyst to oil (w/w). On the other hand, unexpected result was observed when the in-situ method was employed. The results showed different catalyst concentration needed for each technique. The ultrasonic-assisted transesterification (UTI) indicated that it only needed 0.5% to reach optimum CRB yield. Further, the stirring hot-plate-assisted transesterification (HTI) showed that higher concentration of catalyst produced the highest CRB yield. Besides, the MTI as a technique employed in conventional method displayed an optimum point of CRB yield when 1% catalyst to oil was introduced. The effect that occurred when the in-situ method was used could be because the extraction and transesterification were done simultaneously. Overall, the results observed decrease in the pattern of CRB yield after optimal value was reached. This is supported by previous studies, which mentioned that too low or too high catalyst concentration could cause incomplete reaction and give lower yield (Deshmane & Adewuyi, 2013; Gulde *et al*., 2014; Rahimi *et al*., 2018).

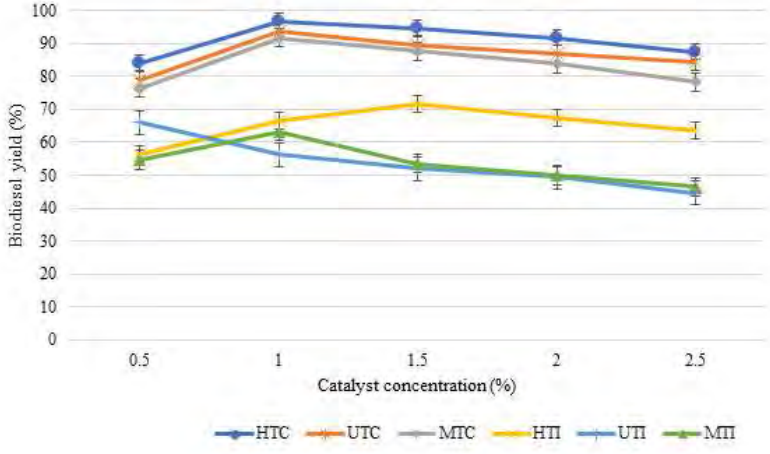


Figure 4.21: Effect of catalyst concentration (3:1 solvent ratio)

4.3.7 Fatty Acid Methyl Ester (FAME) Profiles

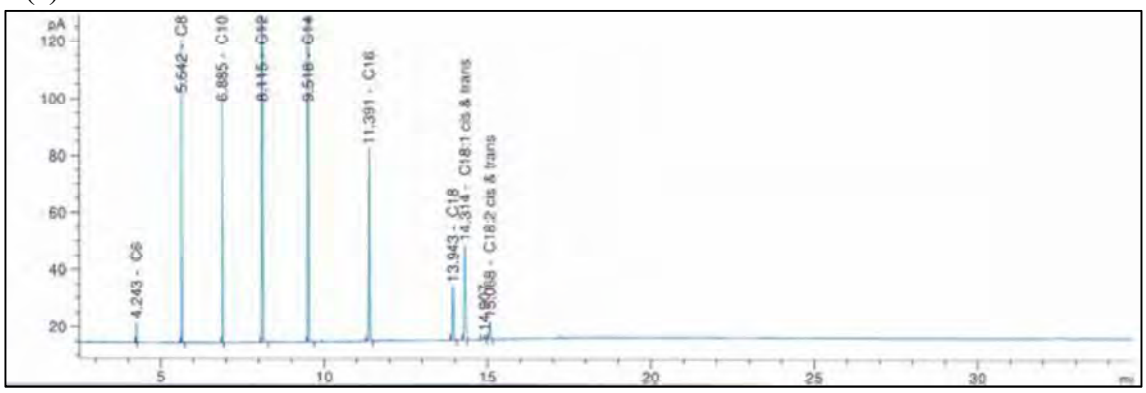
Table 4.22 and Figure 4.22 show FAME composition of CRB from different transesterification techniques, which are HTC, UTC, MTC, HTI, UTI, and MTI. All the analysed FAME used CRB derived using 3:1 solvent to oil molar ratio (v/w) and 1% catalyst concentration to oil (w/w). The results indicated that there were 9 types of FAME found in CRB from all techniques, namely methyl caproate (C6:0), methyl caprylate (C8:0), methyl caprate (C10:0), methyl laurate (C12:0), methyl myristate (C14:0), methyl palmitate acid (C16:0), methyl stearate (C18:0), methyl oleate (C18:1), and methyl linoleate (C18:2). All transesterification techniques depicted methyl laurate as the highest compound of FAME with 47.8% (HTC), 48.1% (UTC), 47.9% (MTC), 47.9% (HTI), 47.7% (UTI), and 47.9% (MTI) in comparison to other compounds. On the other hand, methyl caproate was displayed as the lowest compound in CRB as converted using all of the techniques with approximately ≤0.8% (Azeez, 2007; Kumar *et al*., 2010).

Table 4.22: Comparison of fatty acid methyl ester profiles using different techniques

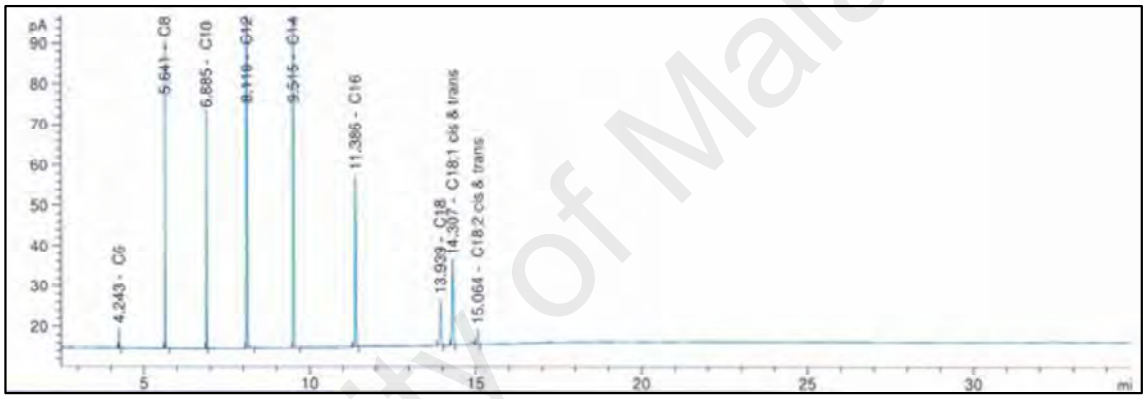
FAME Composition		Techniques						Coconut
		HTC	UTC	MTC	HTI	UTI	MTI	
C6:0	Methyl Caproate	0.5	0.6	0.6	0.8	0.8	0.8	0-0.8
C8:0	Methyl Caprylate	7.9	8.6	8.1	8.9	8.9	8.9	5.0-9.0
C10:0	Methyl Caprate	6.3	6.7	6.3	6.7	6.7	6.6	6.0-10.0
C12:0	Methyl Laurate	47.8	48.1	47.9	47.9	47.7	47.9	44.0-52.0
C14:0	Methyl Myristate	18.0	17.5	17.9	17.4	17.4	17.5	13.0-19.0
C16:0	Methyl Palmitate	9.4	9.0	9.3	8.9	9.0	9.0	8.0-11.0
C16:1	Methyl Palmitoleate	-	-	-	-	-	-	2.5
C18:0	Methyl Stearate	3.1	2.9	3.0	2.9	3.0	2.9	1.0-3.0
C18:1	Methyl Oleate	5.8	5.5	5.7	5.3	5.4	5.3	5.0-8.0
C18:2	Methyl Linoleate	1.2	1.1	1.2	1.2	1.1	1.1	0-1.0
C20:0	Methyl Eicosanoate	-	-	-	-	-	-	0-0.5

Source: Azeez, 2007; Kumar *et al*., 2010

(a)



(b)



(c)

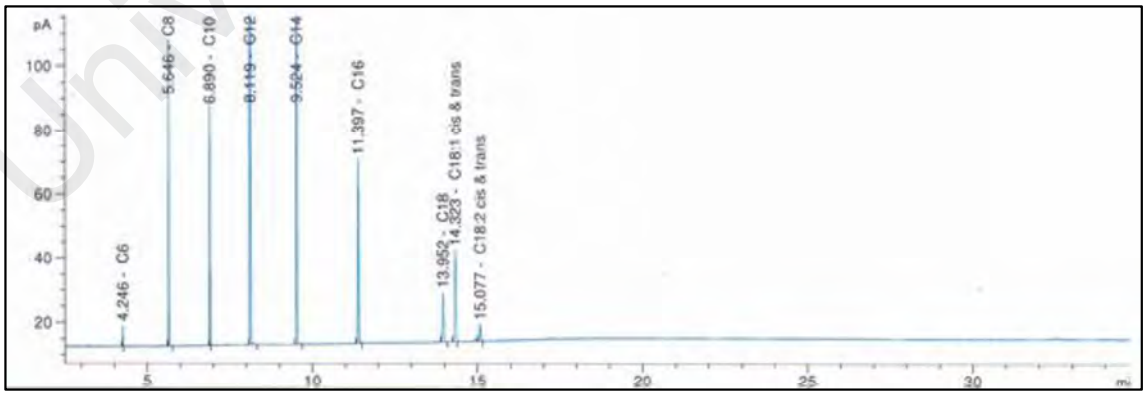
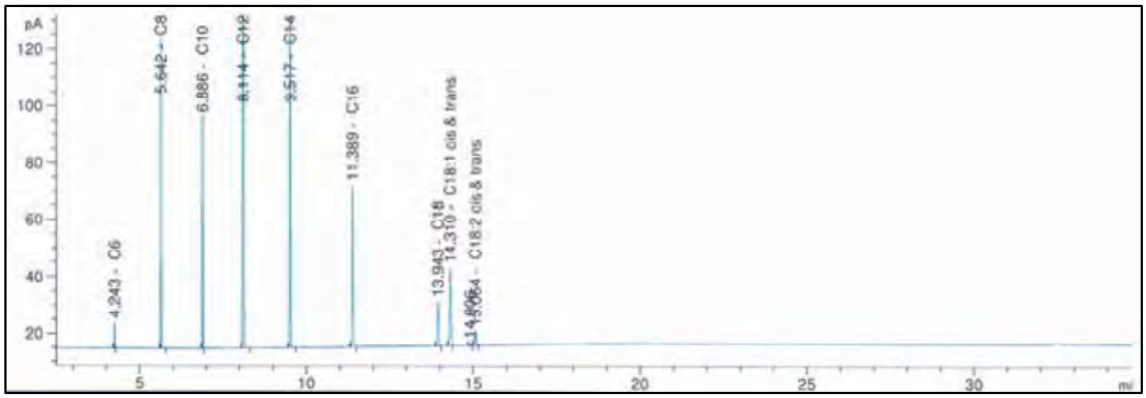
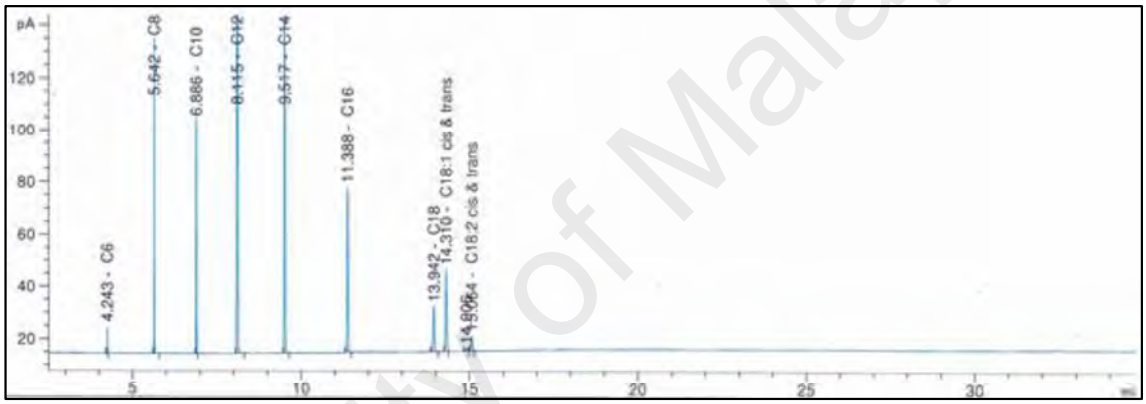


Figure 4.22: Chromatogram of FAME profiles transesterified using different techniques: (a) HTC (3:1 solvent ratio, 1% catalyst); (b) UTC (3:1 solvent ratio, 1% catalyst); (c) MTC (3:1 solvent ratio, 1% catalyst); (d) HTI (3:1 solvent ratio, 0.5% catalyst); (e) UTI (3:1 solvent ratio, 1.5% catalyst); (f) MTI (3:1 solvent ratio, 1% catalyst)

(d)



(e)



(f)

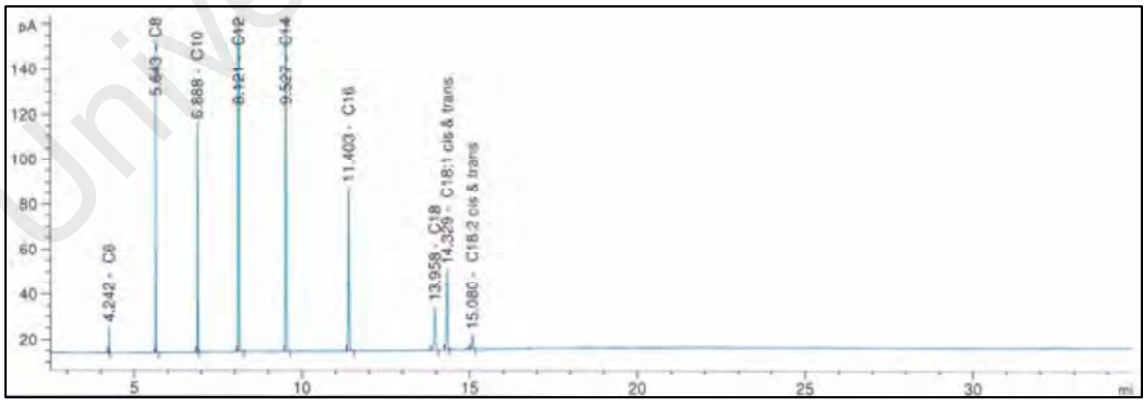


Figure 4.23, continued

4.3.8 Biodiesel Physicochemical Properties Prediction

Table 4.23 shows the prediction of CRB physicochemical properties and comparison with international standard: ASTM D6751 and EN 14214 with other feedstock (American Society for Testing and Materials, 2008; European Standardization, 2008; Kumar *et al*., 2010; Talebi *et al*., 2014). The physicochemical properties such as cetane number, cloud point, oxidation stability, and so on play major roles in determining the quality of produced biodiesel. The cetane number (CN) affects the ignition of fuel delay; the higher the number of cetane, the less occurrence of delay in fuel ignition. The table shows that ASTM and EN put the standard for cetane number where the value should be more than ≥47 to have good ignition delay. From the study, it was predicted that all of CRB yield should contain cetane number of more than 64. The cetane number value is influenced by the presence of saturated fatty acid such as palmitic acid. Next, the results displayed the predicted cloud point (CP) result for CRB yield (-0.3 to 0ºC). The cloud point is the point where solidification occurred. As CP, the cold filter plugging point (CFPP) is a temperature where crystallization occurs and will cause clogging inside the fuel filter. Hence, as cetane number, the saturated fatty acids found in CRB yield gave high impacts to CP and CFPP values (Karmakar *et al*., 2010; Vyas *et al*., 2010).

Furthermore, oxidation stability is also important in determining biodiesel quality because it will determine the extensiveness of biodiesel usage. The stability of oxidation is influenced by the presence of air, heat, light, and especially the fatty compound structure itself (double-bond structure). Previous study mentioned that high presence of saturated fatty acids concentration could give high stability (Karmakar *et al*., 2010; Salamatinia *et al*., 2010; Vyas *et al*., 2010). In brief, the presence of saturated fatty acids compound in CRB potentially produced high quality of biodiesel production.

In terms of comparison with other feedstock, the CRB was potentially explored more widely as an alternative in producing biodiesel since most of the predicted physicochemical properties complied with the ASTM and EN standard. Also, the predicted properties contents were mostly in agreement with the previous study of coconut biodiesel.

Table 4.23: Comparison of predicted physicochemical properties of copra residue biodiesel with international standards (ASTM D6751 and EN 14214) and other biodiesel sources.

Standard/ Diesel/ Biodiesel Properties		Density (g/cm3)	Kinematic viscosity at 40ºC (mm2/s)	Cetane Number	Heating value (Mj/kg)	Cloud point (ºC)	Cold filter plugging point (ºC)	Flash point (ºC)	Pour point (ºC)	Oxidation stability (hrs)
Biodiesel specification standard	ASTM D6751	-	1.90-6.00	≥47.00	-	-3.00 to 12.00	-	≥93	-15 to 10	≥3
	EN14214	0.860-0.900	3.50-5.00	≥51.00	-	-	-	≥101	-	≥6
Petroleum diesel		0.820-0.845	2.00-4.50	≥51.00	-	-15.00 to 5.00	-	≥55	-15 to -35	-
Techniques	HTC	0.871	2.27	64.71	-	-0.05	-8.65	-	-6.873	100.865
	UTC	0.871	2.23	64.67	-	-0.26	-9.09	-	-7.101	109.799
	MTC	0.871	2,26	64.68	-	-0.10	-8.84	-	-6.93	100.865
	HTI	0.871	2.22	64.60	-	-0.31	-9.13	-	-7.158	100.865
	UTI	0.871	2.22	64.63	-	-0.26	-8.94	-	-7.101	109.799
	MTI	0.880	2.25	64.50	-	-0.26	-7.52	-	-7.101	109.799
Other feedstocks	Coconut	0.807	2.73	-	-	-	-4.00	110	-	35.5
	Palm	0.876	5.70	62.00	33.5	13.00	12.00	164	-	4
	Rapeseed	0.882	4.44	54.40		-3.30	-13.00	-	-	7.6
	Sunflower	0.880	4.44	49.00	33.5	3.40	-3.00	183	-	0.9
	Jatropha	0.880	4.80	-	39.2	2.70	-	135	-	2.3
	Corn	0.885	4.40	53.00	-	-2.80	-12.00	170	-	2.2

Source: American Society for Testing and Materials, 2008; European Standardization, 2008; Karmakar *et al*., 2010; Kumar *et al*., 2010; Salamatinia *et al*., 2010; Vyas *et al*., 2010; Talebi *et al*., 2014

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Research Conclusion

The aim of the study is to evaluate the potential of copra residue as renewable energy resources, biodiesel. Hence, the study was divided into three objectives. The first objective is to enhance the copra residue oil (CRO) yield by using three different methods of extractions namely; Soxhlet (SXE), ultrasonic-assisted (UAE), and microwave-assisted (MAE). The results illustrated that the use of SXE technique is the most efficient in extracting CRO yield, in comparison to UAE and MAE techniques. The SXE technique extracted the highest CRO yield, which was about 81.39% (400 mL hexane, 48 hrs), compared to UAE and MAE which showed optimum yield of only 75.80% (300 mL hexane, 30 mins) and 62.97% (100 mL hexane, 15 mins). However, the MAE technique showed great performance in extracting the optimal yield with 15 minutes. Meanwhile, for free fatty acid (FFA) content, the CRO yield extracted through UAE displayed the lowest and the most consistent fatty acid concentration between 0.18% to 0.27% as compared to the other techniques, SXE and MAE.

The second objective is to enhance the biodiesel yield through different transesterification methods (conventional and *in situ*) and techniques, namely: stirring hot-plate-assisted (HT), ultrasonic-assisted (UT), and microwave-assisted (MT). The results indicated that the use of conventional stirring hot-plate-assisted transesterification (HTC) produced the optimal yield compared to other techniques, with 96.85% (3:1 solvent to oil molar ratio, 1% catalyst concentration to oil weight). From the results, it was observed that conventional method is better than in-situ method in producing copra residue biodiesel (CRB). Further, for techniques undoubtedly, the use of HT is the most efficient in both conventional and *in situ* methods to convert the CRO to biodiesel as compared to other techniques.

The third objective discusses the best intelligence software in optimizing the CRB yield which is between adaptive neuro-fuzzy inference system (ANFIS) and response surface methodology (RSM). Undeniably, ANFIS is the best tool in optimizing the CRB yield by which almost all techniques showed high R2 value with almost unity 1 as compared to the other techniques.

In brief, as depicted in Table 5.1, the use of UAE technique is the best method in extracting oil yield for biodiesel conversion purpose since it extracts moderate oil yield and lowest FFA concentration in comparison to other techniques. Furthermore, for biodiesel conversion, the conventional method and HT techniques showed high potential to be employed in producing or synthesising CRB. For CRB yield optimization, the ANFIS software is recommended. Hence, the study concluded that CRO is potentially to be used as sustainable and renewable energy resources, biodiesel.

Table 5.1: Summary of study findings

No.	Objectives	Findings
1.	To enhance the copra residue oil yield using different techniques of extraction.	i. The Soxhlet extraction (SXE) technique extracted the highest oil yield (81.39%). ii. The ultrasonic-assisted extraction (UAE) technique extracted the lowest and consistent free fatty acid (FFA) (0.18 – 0.27%). iii. Hence, the oil extracted from using the UAE technique is the most suitable to be converted into biodiesel.
2.	To enhance copra residue biodiesel yield using different methods and techniques.	i. The conventional method was illustrated as the most efficient method to synthesis biodiesel. ii. The stirring hot-plate-assisted (HT) transesterification demonstrated the best technique to convert the oil to biodiesel (96.85%).
3.	To optimize the copra residue biodiesel yield using adaptive neuro-fuzzy inference system (ANFIS) and Response Surface Methodology (RSM).	i. The adaptive neuro-fuzzy inference system or ANFIS showed the ability as the most accurate intelligence system in optimizing the biodiesel yield.

5.2 Future Research Recommendations

For future research, the following topics should be considered in order to improve the quantity and quality of the copra residue oil as well as to produce better quality of copra residue oil:

1) For microwave-assistant extraction, there should be experiments on various types of solvents including mixture of solvent which have high microwave energy absorbance such as methanol or ethanol.

2) The use of temperature and surface area as parameters should be carried out in order to fully optimize both copra residue oil and biodiesel production. In addition, the type of catalyst is also important to identify the most suitable catalyst to enhance the copra residue biodiesel yield to the maximum.

3) To ensure copra residue oil and biodiesel good shelf life, it is recommended to carry out the antioxidant components analysis. The antioxidant components is responsible not just for the shelf life of the oil, but also determine its quality.

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1) Hakimi, M. I., Ilham, Z., & Kohar, R. A. A. (2018). Enhancement of agro-industrial copra residue oil yield using microwave-assisted extraction. *Waste and Biomass Valorization*. https://doi.org/10.1007/s12649-018-0274-1.

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1) Rabiatul Adawiyyah Abdul Kohar, Mohd Idham Hakimi., & Zul Ilham. (2017, May). *Enhancement of copra oil yield by microwave-assisted extraction*. Poster presented at the Asia International Multidisciplinary Conference 2017 (AIMC2017), University of Technology Malaysia, Johor.