UNIVERSITI TEKNIKAL MALAYSIA MELAKA

CONVERSION OF JATROPHA CURCAS OIL TO BIODIESEL USING HETEROGENEOUS CATALYST VIA MICROWAVE IRRADIATION METHOD

This report is submitted in accordance with the requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor of Mechanical Engineering Technology (Maintenance) with Honours.

by

AMIERA HUSNA BINTI ABDUL HALIM

B071610520

970112105374

FACULTY OF MECHANICAL AND MANUFACTURING ENGINEERING TECHNOLOGY

2019

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: CONVERSION OF JATROPHA CURCAS OIL TO BIODIESEL USING HETEROGENEOUS CATALYST VIA MICROWAVE IRRADIATION METHOD

Sesi Pengajian: 2019

Saya Amiera Husna binti Abdul Halim mengaku membenarkan Laporan PSM ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. **Sila tandakan (X)

☐ SULIT* Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia sebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972.

☐ TERHAD* Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan.

☒ TIDAK TERHAD

Yang benar,

.....

AMIERA HUSNA BINTI ABDUL HALIM

Alamat Tetap:

No 15, Jalan Kilauan 9/2A,

Taman Kilauan, 45600,

Bestari Jaya, Selangor

Tarikh:

Disahkan oleh penyelia:

.....

MAHANUM BINTI MOHD ZAMBERI

Cop Rasmi Penyelia

Tarikh:

DECLARATION

I hereby, declared this report entitled "Conversion of Jatropha Curcas Oil to Biodiesel Using Heterogeneous Catalyst Via Microwave Irradiation Method**"** is the results of my own research except as cited in references.

Signature:

Author : AMIERA HUSNA BINTI ABDUL HALIM

Date:

APPROVAL

This report is submitted to the Faculty of Mechanical and Manufacturing Engineering Technology of Universiti Teknikal Malaysia Melaka (UTeM) as a partial fulfilment of the requirements for the degree of Bachelor of Mechanical Engineering Technology (Maintenance) with Honours. The member of the supervisory is as follow:

Signature:

Supervisor : MAHANUM BINTI MOHD ZAMBERI

ABSTRAK

Satu ujikaji telah dijalankan untuk menghasilkan minyak biodiesel melalui proses transesterifikasi dengan bantuan radiasi gelombang mikro menggunakan minyak yang tidak boleh dimakan iaitu minyak biji pokok jarak sebagai sumber utama. Pengesteran asid menggunakan asid sulfurik dilakukan untuk mengurangkan tahap asid lemak bebas dalam minyak biji pokok jarak dan diikuti oleh proses transesterifikasi dengan bantuan teknik radiasi gelombang mikro. Kalsium Oksida diperolehi daripada kulit kerang (anadara granosa) yang telah digunakan sebagai pemangkin heterogen pepejal dan metanol (MeOH) sebagai alkohol utama dalam proses transesterifikasi. Proses ini telah berjaya menurunkan jumlah asid JCO daripada 30 mgKOH/g kepada 0.375 mgKOH/g. Hasil optimum metil ester asid lemak (FAME) telah berjaya mencapai 90.5% dengan keadaan optimum 9:1 nisbah metanol keatas minyak, kepekatan pemangkin sebanyak 7 wt%, masa reaksi selama 7 minit, dan kuasa gelombang mikro 350 W. Semua sifat-sifat minyak telah dianalisis mengikut piawaian ASTM D6751.

ABSTRACT

An experimental investigation was conducted to produce biodiesel oil via transesterification process with the help of microwave irradiation method by utilizing non edible Jatropha Curcas oil (JCO) as main feed stock. Acid esterification by using sulphuric acid (H_2SO_4), is performed in order to reduce the high free fatty acid level of JCO and followed by transesterification process by using batch microwave irradiation technique. Calcium oxide (CaO) derived from cockle shells (anadara granosa) were selected as solid heterogeneous catalysts with the help of methanol (MeOH) as main alcohol in the transesterification process. The process successfully reduced the acid value of JCO from 30 mgKOH/g to 0.375 mgKOH/g. The optimum yield of fatty acid methyl ester (FAME) was reached up to 90.5% under optimal conditions of 9:1 methanol to oil molar ratio, catalyst concentration of 7 wt%, 7 minutes of reaction time, and 350 Watt microwave power output. All fuel properties were analysed according to the ASTM D6751 standard.

DEDICATION

For my family.

ACKNOWLEDGEMENTS

First of all, all praised to Almighty Allah for the strengths given to me during completing this report. Apart from that, I would like to thank my family for their support and encouragements whether it is financially or morally. I want to express my gratitude to my supervisor, Madam Mahanum Mohd Zamberi who's always helping me in giving suggestions and information in finishing this report. A huge thanks to my fellow friends in 4 BMMM especially Nurul Husna and Farah Diyana that always encourage and share knowledges with me. Lastly, I would like to thank the authority of Universiti Teknikal Malaysia Melaka (UTeM) for the good facilities and environment to make this experiment and report, especially to Faculty of Mechanical and Manufacturing Engineering Technology (FTKMP) for giving me the chance to use the materials, apparatuses, and instruments provided in the laboratory.

TABLE OF CONTENTS

CHAPTER	TITLE		PAGE
	ABSTRAK		**iv**
	ABSTRACT		**v**
	TABLE OF CONTENTS		**vi**
	LIST OF TABLES		**viii**
	LIST OF FIGURES		**ix**
	LIST OF ABBREVIATIONS		**xii**
	LIST OF APPENDICES		**xiii**
1.0	**INTRODUCTION**		
	1.1	Background	1
	1.2	Problem statement	4
	1.3	Objectives	4
	1.4	Scopes	5
2.0	**LITERATURE REVIEW**		
	2.1	Biodiesel Background	6
		2.1.1 Sources of Biodiesel	7
		2.1.2 Benefits of Biodiesel	12
		2.1.3 Limitations of Biodiesel	12
	2.2	Raw Material	13

2.2.1 Jatropha Curcas Oil 13

2.2.2 Catalyst 14

2.2.3 Alcohols 16

2.3 Biodiesel Production Method 17

2.3.1 Conventional Method 17

2.3.2 Microwave Irradiation 17

2.3.3 Ultrasonic Heating method 18

2.4 Transesterification Process 20

2.5 Variables of Biodiesel 21

2.5.1 Reaction rate/Time 21

2.5.2 Reaction Temperature 22

2.5.3 Catalyst Concentration 22

2.5.4 Type of Alcohols Used and the Molar Ratio 23

2.5.5 Microwave Power Output 23

2.6 Standard Biodiesel Properties 24

3.0 METHODOLOGY

3.1 Raw Materials and Instrument 26

3.1.1 Jatropha Oil Preparation 26

3.1.2 Chemicals 27

3.1.3 Apparatus 27

3.2 Biodiesel Production 29

3.2.1 Acid Esterification 30

3.2.2 Catalyst Preparation and Characterization 32

3.2.3Transesterification 34

3.3 Gas Chromatography Mass Spectrometry 36

3.4 Fuel Properties 37

3.4.1 Acid Value and Free Fatty Acid 37

3.4.2 Kinematic Viscosity 38

3.4.3 Density 39

3.4.4 Flash Point 39

4.0 RESULTS AND DISCUSSION

4.1 Characteristic of Crude JCO 41

4.2 Preparation and Characterization of Catalyst 41

4.3 Acid Esterification 43

4.4 Transesterification by Microwave Irradiation 45

4.5 Effect of Variables on Transesterification 46

4.5.1 Effect of Methanol to Oil Molar Ratio 46

4.5.2 Effect of Catalyst Amount 48

4.5.3 Effect of Microwave Power (W) 50

4.5.4 Effect of Reaction Time 51

4.6 Gas Chromatography Mass Spectrometry 53

4.7 Fuel Properties 54

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion 54

5.2 Recommendation 56

REFERENCES 57

APPENDICES 60

LIST OF TABLES

TABLE	TITLE	PAGE
2.1	Potential feedstocks for biodiesel for selected countries	8
2.2	Identified Feedstocks for Biodiesel Productions	10
2.3	Comparison between homogeneous and heterogeneous catalysts activity	14
2.4	Comparison between conventional, microwave, and ultrasonic heating method	19
2.5	ASTM D6751 and EN 14214 properties	25
3.1	Crude JCO Specifications	26
4.1	Properties of Crude JCO	40
4.2	The XRF Analysis of the Calcined Samples Compared	41
4.3	Results of Acid Esterification	43
4.4	Transesterification Process Outcomes	44
4.5	Comparison of Microwave Power and Yield Percentage	49
4.6	The Comparison of Time Reaction and Percentage Yield	51
4.7	Comparison of Biodiesel Properties with ASTM D6751 Standard and EN 14212	52
4.8	Composition of FAME in Produced Biodiesel	53

LIST OF FIGURES

FIGURE	TITLE	PAGE
Figure 2.1	Transesterification Reaction	20
Figure 3.1	The schematic diagram of fabricated microwave	28
Figure 3.2	Flow chart for biodiesel production	29
Figure 3.3	The separation of solution	31
Figure 3.4	Catalyst Preparation	32
Figure 3.5	Modified Microwave for Transesterification Process	34
Figure 3.6	GCMS-Agilent Technologies 6890 N	36
Figure 3.7	Viscometer	38
Figure 3.8	Pycnometer	39
Figure 3.9	Pensky-marten closed cup tester	39
Figure 4.1	Effect of H_2SO_4 Concentration to the FFA Percentage	43
Figure 4.2	Effect of MeOH to Oil Molar Ratio to the Yield Percentage	46
Figure 4.3	Effect of Catalyst Concentration to the Yield Percentage	47
Figure 4.4	Effect of the Microwave Power to the Yield Percentage	50
Figure 4.5	Effect of Reaction Time to the Yield Percentage	51

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
Appendix 1	Gantt Chart PSM 1	60
Appendix 2	Gantt Chart PSM 2	61
Appendix 3	Molar Ratio Calculation	62
Appendix 4	Catalyst Concentration (wt%) Calculation	63
Appendix 5	Density Calculation	64
Appendix 6	GC-MS Analysis	65

LIST OF ABBREVIATIONS

FFA	Free Fatty Acid
JCO	Jatropha Curcas Oil
NaOH	Sodium Hydroxide
KOH	Potassium Hydroxide
MeOH	Methanol
CaO	Calcium Oxide
$CaCO_3$	Calcium Carbonate
H_2SO_4	Sulphuric Acid
ASTM	American Society of Testing and Materials
%wt	Weight Percentage
℃	Degree Celcius
AV	Acid Value
EN	European Standards
XRF	X Ray Fluorescence
GCMS	Gas Chromatography Mass Spectrometry

CHAPTER 1

INTRODUCTION

1.1 Background

It is acknowledged that fossil fuels are the main part in global energy demands. However, the demands appear to escalate at deliberate pace in this past years due to unaccustomed efficiencies made by new renewable technologies as well as the enforcement of strict energies policy and environmental laws. The need for to create a fossil alternative has caused large research in the few last years. Fossil fuels are non-renewable energy sources that produce pollutants and are related to global warming, some incurable diseases and climate change. In this condition, there is a strong need for renewable energy because of the new technologies, greater challenges to the environment, and speed of economy and geopolitics. It is widely known that igniting fossil fuels such as oil and coal leads to environmental problem and decarbonisation of energy system. Thus, it will increase the use of low carbon sources of energy like renewables. Environmental issues like climate change are one of the toughest challenges requiring full engagement by all parties involved. Therefore, there are efforts to alter vehicle fuel and this approach is believed to be one of the efficient solutions to environmental problems arising from combustion of fossil fuels. (Milano et al, 2018).

Biodiesel is regarded as alternative for non-renewable energy and has gained many attention due to its eco-friendliness, renewable behaviour, and likely to decrease deplete productions. Biodiesel is defined as the mono-alkyl esters of vegetable oils or animal fats. Biodiesel is contributing to the protection of the environment as it is reducing sulphur oxide

emissions, biodegradable, renewable, non-toxic, and less effect of greenhouse gasses. Thus, the global warming problems could be reduced (Chuah et al. 2015). Biodiesel is a renewable substitution fuel produced through the chemical process from vegetable oils, animal fats and other sources such as micro-algae. The chemical process includes reaction of crude oils with any types of alcohols and catalysts. After that, the mixture of the three substances is purified and tested to produce biodiesel yields that combustible in a diesel engine. Biodiesel fuel can be applied in pure diesel engine or blending the biodiesel and diesel fuel at any ratio for any diesel engines without modifications. The other characteristics of biodiesel are any modifications to a diesel engine are not necessary, biodegradable, safer to handle than petroleum diesel fuel and, quality of biodiesel is regulated by ASTM D 6751 quality specifications.

According to Lim & Teong, (2010), the development of vegetable oils in diesel engine could be started in the year of 1900 when Rudolf Diesel that invented the engine had performed peanut oil as fuel in Paris World Fair. In that time, diesel engine has become the engine of choice for power, reliability, and high fuel economy worldwide. Due to extensive availability and low price of petroleum diesel fuel, vegetable oil based fuels gained little rise, except in times of the high demands of oils and the shortages. However, the newer design of diesel engine could not use on traditional vegetable oil because of high viscosity of the oils. They need a way to lower the viscosity so that it can be combusted completely in the diesel engine. A Belgian inventor is the first person who projected the idea to use transesterification method to change the vegetable oils into fatty acid alkyl esters and use them as diesel substitute. The transesterification process made the vegetable oils less viscous and easy to burn because the oils had been converted to three smaller molecules.

Since early 1980s, the Malaysian government realized what is essential to the long-term development of biodiesel. As the world's biggest palm oil producer and exporter, it was required that in the palm biodiesel industry, Malaysia comes as one of the pioneer. This was accomplished through the ambitious stance by Malaysian palm Oil Board (MPOB) when the project of inventing palm biodiesel was started at laboratory scale in 1982. Henceforth, development of biodiesel in Malaysia had been progressing in fast pace (Lim & Teong, 2010).

In biodiesel production, transesterification is a crucial process that must be performed as the procedure that clarifies the respond comes from a lipid with alcohol to produce esters and by-product, glycerol. Basically, biodiesel is extracted from the vegetable oils or animal fats. Therefore, the chemical reactions during the process will converts the esters into a combination of fatty acid esters that forms the oil. Biodiesel is earned from the mixture purification of fatty acid methyl esters (FAME). Catalysts are available to speed up the reaction and type of catalysts can be different. In this experiment, the catalysts used is cockles while the oil feedstock used is Jatropha oil.

1.2 Problem Statement

Generally, biodiesel is known as an alternative way to replace the usage of diesel petroleum oil because biodiesel oil is more eco-friendly and not causing any visible pollution. However there are some limitations in order to develop biodiesel oil. The production of biodiesel is more pricy compared to the petroleum diesel. This is because the raw materials for biodiesel such as crude oil and alcohol is quiet expensive. Next, there are several parameters that need to be tested before producing biodiesel. The variables such as reaction time, molar ratio of alcohol to the oil and catalyst concentration have to be measured and tested repeatedly until the oil becomes biodiesel according to the ASTM D6751 standards. Therefore, the study of biodiesel uses more energy and time.

1.3 Objectives

1. To prepare and characterize the calcium oxide (CaO) from cockle(anadara granosa) as solid catalyst
2. To study the effects of process variables (microwave output power, reaction time, catalyst concentration and alcohol to oil ratio), production yield and apparatus embodiment.
3. To examine the performance and purity of the feedstock characteristics by performing the Gas Chromatography Mass Spectrometry (GCMS) and ASTM D6751 standard.

1.4 Scopes

1. Producing the pre-treatment or acid esterification of Jatropha oil (non-edible oil) as a feedstock for biodiesel production.
2. Converting calcium carbonate (CaCO3) of cockles and mussels to heterogeneous calcium oxide (CaO) by calcination process to produce solid catalyst.
3. Performing the transesterification process assisted by heat from the microwave irradiation.
4. To ensure that the production of biodiesel meets the standard preparation of ASTM D6751 test.

CHAPTER 2

LITERATURE REVIEW

2.1 Biodiesel background

A type of renewable alternative fuel or known as biodiesel is derived from vegetable oils or fats of plants such as rubber seeds, Jatropha Curcas or palm oil. According to Kotha et al., (2018), the main purpose of biodiesel production is to be used as replacement for petroleum diesel fuel and has gained a lot of attention due to its renewable nature, eco-friendliness, and potential toward reducing deplete emissions. Other than that, biodiesel is also suitable as alternative because it is fitting as energy demand and substitute to reduce petroleum consumes.

Biodiesel is briefly distinguished as animal fats or vegetable oil-based diesel fuel that consist of a long-chain (methyl, ethyl, or propyl) esters. According to National Biodiesel Board of United States of America, the technical definition for biodiesel based on American Society of Testing and Material (ASTM) D 6751 is a fuel that made of mono-alkyl esters of long chain fatty acids extracted from vegetable oils or animal fats, appointed as B100 (100% biodiesel) and meeting the ASTM D6751 standards. For the blending of biodiesel, the mixes with diesel fuel are composed as *Bx* where *x* is representing the percentage of biodiesel include in the mixture. For example, *B10* is indicated as 10% of biodiesel and 90% of diesel fuel. Therefore, *B100* stand for 100% pure biodiesel.

Just like diesel fuel, biodiesel can be applied for diesel compression engines like those used in transportations and stationary electrical generator units. In addition, no necessary engine modification is required as the biodiesel properties are resembling the payload capacity and range of petroleum diesel. Therefore, there is no need to use or create separate vehicles. The function of biodiesel fuel in the diesel engines results in a significant reduction of un-burnt hydrocarbons, carbon monoxide, and any particulate matters (Kotha et al., 2018).

2.1.1 Sources of biodiesel

The production of biodiesel is more reachable as substitution for renewable energy as it has extensive available feedstock or sources of raw materials. This is included different types of vegetable oils, animal fats, microbial oils, algal oils, and waste oil. Different sources will have different composition and purity for biodiesel productions. (Ambat, Srivastava, & Sillanpää, 2018). Thus, in production of biodiesel, widely range of sources is one of the most important factor in order to continue the uses of biodiesel. Atabani et al., (2012) stated that the best feedstock should fulfil the two main requirements as biodiesel sources which are low production costs and produce large scale of production. Other than that, the biodiesel sources should depends on crops that are compliant to the regional climate, geographical locations, local soils condition and agricultural practices from any country in order to determine the most suitable material for biodiesel production. Table 2.1 shows the primary biodiesel sources for some countries globally.