UNIVERSITI TEKNIKAL MALAYSIA MELAKA

HIGH FREE FATTY ACID REDUCTION FROM JATROPHA CURCAS OIL VIA ACID TRANSESTERIFICATION

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A thesis submitted
in fulfilment of the requirements for the degree of
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DECLARATION

I declare that this Choose an item. entitled “High Free Fatty Acid Reduction From Jatropha curcas Oil Via Acid Transesterification” is the result of my own research except as cited in the references. The Choose an item. has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature	:	
Name	:	Muhammad Ariff Bin Noor Salleh
Date	:	18 JANUARY 2022

APPROVAL

I hereby declare that I have checked this thesis and, in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Mechanical Engineering Technology (Maintenance Technology) with Honours.

Signature :

Supervisor Name : Mahanum Binti Mohd Zamberi

Date : 18 JANUARY 2022

DEDICATION

This research is dedicated to my dear parents who have served as a source of inspiration and strength when me have felt like giving up.

To my dear supervisor, teammates, classmates, and brothers who encouraged me to finish this study with their words of advice and encouragement.

Finally, I want to express my gratitude to the almighty god for his guidance, strength, mental, power, and protection.

ABSTRACT

Biodiesel has earned a remarkable of attention as a renewable, biodegradable, and non-toxic alternative to fossil fuels. The multipurpose plant known as *Jatropha curcas* which contains a lot of oil and can be consider as non-edible oil. The main objective of the current research work is to study the effects of acid esterification of this highly Free Fatty Acid (FFA) content of oil in order to reduce acid content during transesterification process. Acid esterification process would be introduce using sulphuric acid (H_2SO_4) to reduce the high fatty acid of the feedstock. The best molar ratio alcohol to oil is 12:1 with 1% v/v acid sulfuric (H_2SO_4), which is value FFA is 0.18 mg KOH/g. Reduction percentage of FFA will be analysed according to ASTM D6751 and EN 14214.

Keywords: Biodiesel; *Jatropha curcas*; acid esterification

ABSTRAK

Biodiesel menarik perhatian luar biasa sebagai salah satu sumber alternatif yang boleh diperbaharui, biodegradasi, dan tidak beracun sebagai bahan bakar fosil. Tumbuhan serbaguna yang dikenali sebagai minyak biji pokok jarak yang memiliki kandungan minyak yang banyak dan boleh dianggap sebagai minyak yang tidak boleh dimakan. Objektif utama penyelidikan ini adalah untuk mengkaji kesan pengesteran asid kandungan Asid Lemak Bebas minyak yang tinggi dengan tujuan mengurangkan kandungan acid semasa proses transesterifikasi. Proses pengesteran asid diperkenalkan menggunakan asid sulfurik (H_2SO_4) untuk mengurangkan asid lemak tinggi bahan mentah. Nisbah molar terbaik alkohol kepada minyak ialah 12:1 dengan 1% isipadu asid sulfurik (H_2SO_4), iaitu nilai FFA ialah 0.18 mg KOH/g. Peratusan pengurangan FFA akan dianalisis mengikut ASTM D6751 dan EN 14214.

Kata kunci: Biodiesel; Minyak biji pokok jarak; Pengesteran asid

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LIST OF SYMBOLS AND ABBREVIATIONS

w/w	-	Weight per weight
N	-	Normality
ASTM D6751	-	American Society for Testing and Material
EN 14214	-	European Nation
FAME	-	Fatty Acid Methyl Ester
FFA	-	Free Fatty Acid
KOH	-	Potassium hydroxide
H_2SO_4	-	Sulphuric acid
v/v	-	Volume per volume
GCMS	-	Gas Chromatography-Mass Spectrometry (GCMS)

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CHAPTER 1

INTRODUCTION

1.1 Background

Biodiesel is a renewable fuel made primarily from domestic feedstock derived from agricultural or nutrition products, as well as the recycling of products such as cooking and vegetable oils (both edible and non-edible oil) and algae. Biodiesel had recently gained popularity due to its greenhouse advantages and the concept of making something out of renewable resources and to be more cost-effective than fossil fuel production. Soybean, sunflower, palm kernel, rapeseed, cotton seed, and jatropha oils are the most widely used oils for biodiesel processing (S. P. Singh & Singh, 2010).

Biodiesel production is thought to be more cost-effective than production of fossil fuels. Biodiesel made from energy crops is environmentally friendly because it is biodegradable, reduces the amount of acid rain, and reduces the environment impact brought by combustion process. It also reduces the amount of sulfur dioxide (SO_2) and hydrocarbons that have not been burned released at the process of combustion (Agarwal, 2007). More than 95% of biodiesel is currently made from vegetable oil as a feedstock (soya bean oil, sunflower oil, rapeseed oil, palm oil, and sesame oil), resulting in a significant out of balance in the human nutrition chain versus diesel (Muhammada et al., 2018).

As a matter of fact, an extensive variety of non-edible oil producing plants are considered for biodiesel production. Non-edible seed oil, such as that from *Jatropha curcas* seed can be used for commercial biodiesel production to prevent these situations and it is a possible feedstock for biodiesel extraction. Biodiesel that meets international fuel standards such as ASTM D6751 (American Society for Testing and Material) specified standards and requirements for blended biodiesel with medium petroleum fuels and EN 14214 (European Nation) that specifies the specification and test methodologies for fatty acid methyl ester has been acknowledge as a substitute for fossil diesel. This international standard detailed for B100 (biodiesel 100%).

One of most common method of making biodiesel is transesterification. Transesterification occurs when a lipid reacts with an alcohol in existence of potassium hydroxide (KOH) as catalyst to produce esters and glycerol as a by-product. Before that acid treatment was proposed firstly because of high content free fatty acid in crude *Jatropha curcas* oil. Alcoholysis is described as the action of one alcohol displacing another from an ester in principle (cleavage by an alcohol) (Anitha & Dawn, 2010). Several factors influence this reaction, including the molar ratio alcohol to oil, reaction time, catalyst concentration, reaction temperature, and stirring influence, and among others. One among the most popular raw materials used in the manufacturing of biodiesel is alcohol. The most frequently used alcohols in biodiesel production are methanol and ethanol. Methanol, on the other hand, is selected due to its physical and chemical properties. It also has a rapid reaction with triglycerides and is quickly dissolved in KOH. In addition, methanol is used in the manufacture of biodiesel because cheap and reactive. Biodiesel fuels that were made from methanol and ethanol have minor differences in terms of their characteristics as fuels. As a comparison purposes, biodiesel made from methanol have slightly higher pour and cloud points and slightly lower viscosities compared with those made from ethanol (Yusuf et al., 2011).

In this study, the high FFA of *Jatropha curcas* seed oil will be fully utilized as the feedstock in producing a green and quality biodiesel. An acid transesterification will be used to perform the reduction of FFA. The value FFA will be set to meet the requirement determined by the ASTM D6751 and EN 14214.

1.2 Problem Statement

The relationship between current issues in biodiesel production and this study is demonstrated through problems statement. The study of objectives and scope are intended to address such various challenges in reduction of Free Fatty Acid of feedstocks. There are rarely of detailed study especially on the use of *Jatropha curcas* oil in acid transesterification method has been reported. Furthermore, *Jatropha curcas* produces a large amount of oil despite being an inedible fruit. If this benefit is not utilized or disregarded, it will be a loss. Amongst the most important problem should be highlight is the greenhouse impact caused by combustion process such as gas emission, sulfur dioxide and unburned hydrocarbons released during the combustion phase.

1.3 Research Objective

The main aim of this research from *Jatropha curcas* seed oil having a high free fatty acid are as follows:

a) To reduce Free Fatty Acid of *Jatropha curcas* oil using an acid transesterification process according ASTM D6751 and EN 14214.

b) To perform acid sulfuric (H_2SO_4) as acid catalyst in acid transesterifcation process.

c) To study the influences of variable including alcohol to oil molar ratio, catalyst loading, effect of temperature, and reaction time on the reduction of Free Fatty Acid.

1.4 Scope of Research

The scopes of this study consist of three important elements. They are: -

a) Develop an overall overview of the current construct of FFA reduction with the conventional method, including its process, variable parameters, and raw material that involved.

b) Performed acid-treatment process with an acid catalyst, which is sulfuric acid (H_2SO_4).

c) Acid value reduced are subjected to a thorough chemical and physical analysis, which was carried out in accordance with ASTM D6751 and EN 14214 standards.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In recent years, several studies have focused on biodiesel as alternative fuel made from animal fat, vegetable oil (edible or non-edible oil), and recycled cooking oil. Biofuels are seen as a part of the option to issues such as sustainable development, energy, and carbon reduction. The methods used, the type of oil, production process, sort of alcohol, the type of catalyst, the stirring effect, the reaction time, the reaction temperature, a molar ratio of alcohol to oil and acid value were all the factors that need to be considered in the production based on the criteria and factors. *Jatropha curcas* has been identified as one of the possible commercial sources of renewable energy that have a lot of benefit especially on the production output.

Free fatty acids (FFA), phospholipids, water, and other impurities are present in crude oil obtained by pressing natural vegetable oils. Even with these components, oil cannot be used as a fuel directly. Chemical adjustments to the oils are required to resolve the issues raised so that can meet those properties of biodiesel. Performed acid-treatment process with an acid catalyst, which is sulfuric acid (H_2SO_4) to reduced FFA of feedstock before Fatty acid methyl ester (FAME) was produced from reaction alcohol with triglyceride of raw *Jatropha curcas* oil (JCO) via 2-step pretreatment process and base-transesterification process. Acid value reduced were subjected to a thorough chemical and physical analysis, which was carried out in compliance with ASTM D6751 and EN 14214 standards.

2.2 Ishikawa Diagram

An Ishikawa diagram is used to represent the primary elements that influence a final outcome, which is frequently related to a production or design issue. These charts are commonly referred to as ‘Fishikawa’ diagrams since are shaped like a fish. Branch bones are mean variables in this study that influence an output, and the result was reduction in FFA (refer Figure 2.1).

There are six parameters to reduce free fatty acids of *Jatropha curcas* oil (JCO). First, effect of production, where acid transesterification was proposed first before base transesterification process because high free fatty acid content in JCO would cause separation issue and formation of soap. Effect of alcohol, where methanol was chosen to react with JCO in acid transesterification process. In addition, effect of catalyst was one of the most important influences, such as percentage concentration of acid sulfuric give high impact to the resultant JCO. Furthermore, effect of temperature means that reaction temperature must below and not exceed boiling point of methanol, where the range is 60℃ to 70℃. Next, effect of molar ratio alcohol to oil, where stoichiometric ratio for acid transesterification process. For example, molar ratios are 3:1, 6:1, 9:1, and 12:1. Effect of time means that the time needed for acid transesterification until reaction completed.

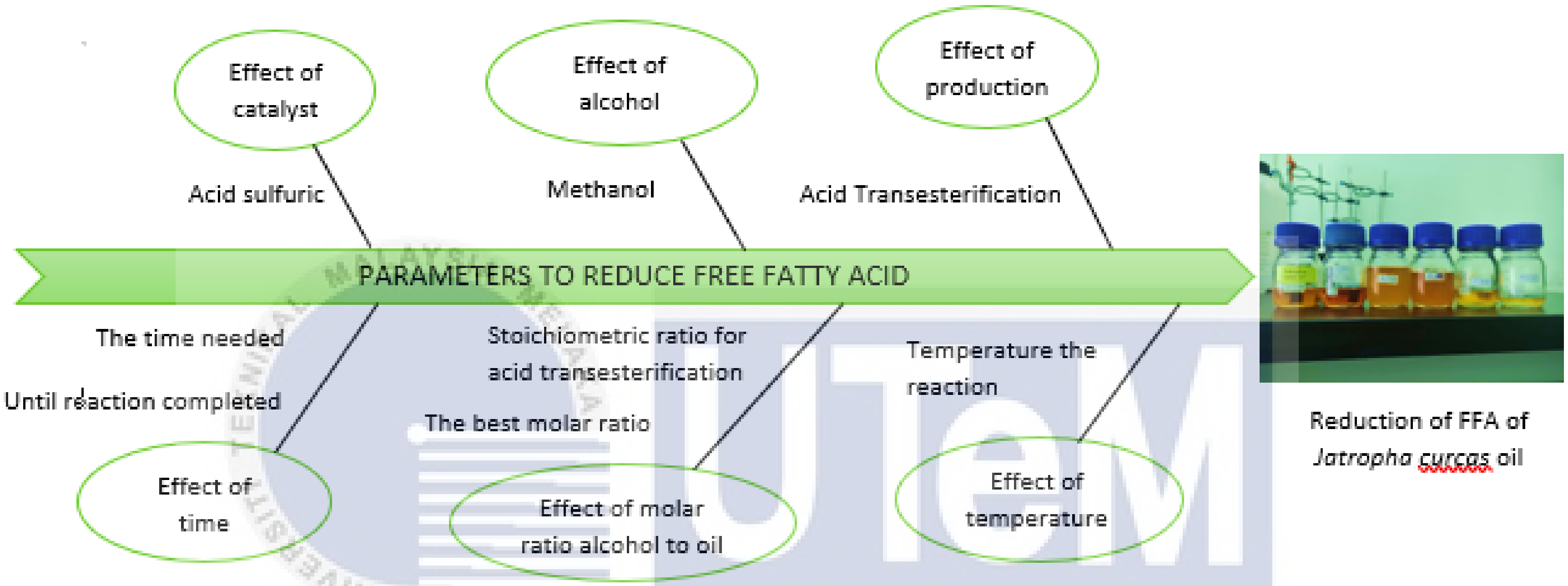

Figure 2.1 Parameters to reduce Free Fatty Acid

2.3 Raw Jatropha curcas

The tropical tree *Jatropha curcas* that originated in Mexico and has now spread over Asia and Africa. Since *Jatropha curcas* belongs to the *Euphorbiaceae* family, it has traditionally been used for medicinal purposes. *Jatropha curcas* is distinguished by the presence of 3±0 seeds in each fruit, which are black in colour. Figure 2.2 shows *Jatropha curcas* fruit and seeds. The average seed length is 18 mm (Abdulla et al., 2011). Mechanical pressing is one of the most popular methods for extracting oil. Crude oil is obtained by pressing vegetable oil, which include free fatty acids, moisture and other impurities (Abdulla et al., 2011). *Jatropha curcas* is non-edible oil, it does not pose a threat to food supplies once used to make biodiesel, and it also produces a lot of oil. The leaves and seeds of *Jatropha curcas* are harmful and animals, among other things.

Table 2.1 shows a properties specification of *Jatropha curcas* oil and *Jatropha curcas* biodiesel. Properties specification that has been highlighted are density at 15 °C, cloud point, flash point, pour point, viscosity at 40 °C, cetane number, iodine number, and acid value.

Figure 2.2 *Jatropha curcas* fruit and seeds (Tuttosemi, 2021)

Table 2.1 Properties of *Jatropha curcas* oil and *Jatropha curcas* biodiesel (D. Singh et al., 2019).

Properties specification	***Jatropha curcas* oil**	***Jatropha* biodiesel**
Density at 15 ℃	916	865
Cloud point (℃)	-	5.66
Flash point (℃)	211.7	175.5
Pour point (℃)	-	6
Viscosity at 40 ℃ (mm^2/s)	37.28	4.52
Cetane number	21	55.43
Iodine number	97.9	96.75
Acid value (mg/g)	-	0.24

2.4 Effect of Production Process

Free fatty acids (FFA), phospholipids, water, and other impurities are present in crude oil obtained by pressing natural vegetable oils. Even with these components, oil cannot be used as a fuel directly. Chemical adjustments to the oils are required to resolve the issues raised so that can meet those properties of biodiesel. Transesterification is a method that involved reaction of oil with an alcohol in the presence of catalyst to produce fatty acid methyl ester, to produce a good biodiesel and glycerol (Abdulla et al., 2011). Acid transesterification process was used to reduce FFA and eliminate other impurities were present in crude oil before start transesterification for production of biodiesel. So other impurities did not to disturb a process production of biodiesel.

In another study, Rajalingam et al., (2016) examined the process of thermal cracking to convert a hydrocarbon's complex structure into its simplest structure, either with or without the use of a catalyst. Oil density and viscosity will be reduced as a result of this process. Another study by Rajalingam et al., (2016) were direct use to engine and blending. In direct injection engines, animal fat or vegetable oil can be utilized as a fuel because it has