CONTINUOUS BIODIESEL PROCESS USING ULTRASONIC IN-LINE REACTOR FOR JATROPHA CURCAS OIL (JCO)

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Special dedication to my family who give me support:

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ABSTRACT

Biodiesel is an alternative fuel for replacing diesel fuel in compression ignition engines. Due to the complexity of the diesel fuel production and exhaust emissions from petroleum-fuelled engines will give negative impact to the environment. In this study, the sodium hydroxide as the catalyst was used to react with methanol for obtaining chemical compound that is called methyl ester which is known as biodiesel. The method used are *Ultrasonic*. Basically, this method will reduce the reaction time on the conversation of jatropha curcas oil (JCO) into biodiesel. The experiment was to determine the effect of esters contents by reaction time, molar ratio methanol (MeOH) to JCO, the amount of catalyst, frequency and power output of ultrasonic using ultrasonic in-line reactor. The optimum production of biodiesel was achieved at 7 minutes of reaction time, 1%wt of catalyst concentration and molar ratio methanol to oil 12:1, frequency ultrasonic of 20 KHz and ultrasonic output 600 Watt at temperature 65°C. The biodiesel produced by this method has been referred according to ASTM D6751. From the result, the biodiesel produced from this method has satisfied the requirement biodiesel standard. This optimum result in this research can be used to run larger pilot plant designed for industry.

ABSTRAK

Biodiesel adalah bahan api alternatif yang digunakan dalam pembakaran enjin. Hal ini kerana, kesukaran pengeluaran minyak diesel dan asap yang dilepaskan oleh pembakaran enjin petroleum mendatangkan kesan yang negatif kepada alam sekitar. Kajian ini menggunakan natrium hidroksida sebagai pemakin untuk bertindak balas dengan metanol bagi menghasilkan sebatian kimia yang dipanggil “methyl ester” dan juga dikenali sebagai biodiesel. Kaedah ultrasonik telah digunakan. Secara asasnya, kaedah ini dapat mengurangkan masa tindak balas bagi penukaran minyak jatropha curcas (JCO) kepada biodiesel. Kajian ini dijalankan adalah untuk menentukan kandungan “ester” dengan masa tindak balas, nisbah molar metanol (MeOh) kepada JCO, jumlah pamangkin, frekuensi dan keluaran kuasa ulrasonik “in-line”. Penghasilan biodiesel yang optimum dicapai dengan masa tidak balas 7 minit, 1% wt kepekatan pemangkin dengan nisbah molar 12:1 metanol kepada minyak, frekuensi ultrasonik 20 KHz dan keluaran ultrasonic 600watt pada suhu 65°C. Biodiesel yang dihasilkan melalui kaedah ini telah dirujuk dengan piawaian biodiesel iaitu ASTM D6751. Keputusan yang diperolehi daripada perbandingan biodiesel yang dihasilkan menggunkan kaedah ini adlah menepati piawaian biodiesel. Penghasilan optimum biodiesel yang diperolehi dari kajian ini boleh diaplikasikan pada loji pandu biodiesel yang dihasilkan bagi kegunaan industri.

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LIST OF SYMBOLS AND ABBREVIATIONS

Δ Uc,m	molar internal energy
ASTM	American Standard Testing Method
$Ca(OH)_2$	calcium hydroxide
$CISO_3OH$	chlorosulphonic acid
CO	carbon monoxide
DEE	diethyl ether
DME	dimethyl ether
EU	European Nations
FAME	fatty acids methyl ester
FFA	free fatty acid
FID	flame ionization detector
GC/MS	gas chromatography mass spectrometry
GHz	giga hertz
H_2SO_4	sulphuric acid
HC	hydrocarbons
HCl	hydrochloric acid
HI	hydrogen iodide
JCO	jatropha curcas oil
KOH	potassium hydroxide
MeOH	methanol
MHz	mega hertz
MPa	mega pascal
MPOB	Malaysian Palm Oil Board
Na_2SO_4	sulphate
$NaOCH_3$	sodium methoxide
NaOH	sodium hydroxide
NOx	nitrogen oxides
USA	United States of America

LIST OF APPENDICES

LIST OF PUBLICATIONS

Journals:

i. Nurrul Rahmah bt Mohd Yusoff, Sulaiman Haji Hasan and Nor Hazwani bt Abdullah, "The Properties Of Biodiesel Using Ultrasonic Continues Process" Australian Journal of Basic and Applied Sciences, Vol. 8 Issue 15, pp 269-276, 2014 ISSN1991-8178

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Renewable energy is the development of new technologies that has become interesting topics around the world over a decades ago. These technologies are the alternatives for replacement of conventional (fossil) fuels. Biodiesel is one of the alternatives fuel which substitutes the petroleum-based diesel (Benavides *et al.*, 2013). According to Gude *et al.*, (2013) alternative fuel sources such as biodiesel is needed in transportation and industrial sectors to drive the machinery and engines. Biodiesel is an alternative fuel for replace diesel fuel in compression ignition engines because today storage of diesel fuel decreases and exhaust emissions from petroleum-fuelled engines give bad impact to environment (Berchmans and Hirata, 2008).

Since 2003 the total production of biodiesel in the world was around 1.8 billion liters. In European Nations (EU) alone, the demand for biodiesel was projected to increase from 3 million tons in 2005 to 10 million tons in 2010. Table 1 depicts the top 10 countries in terms of absolute biodiesel production potential with Malaysia far ahead among the rest. Therefore, Malaysia does not need to rely on foreign import for raw materials to develop its own biodiesel industry feedstock available for the development of biodiesel in these nations are 28% of soybean oil, 22% of palm oil, 20% of animal fats, 11% of coconut oil, while rapeseed, sunflower and olive oils constitute 5% each. Malaysia is one of countries which is richness in oil palm is the primary driving force for its development of biodiesel industry. Therefore, Malaysia does not need to rely on foreign import for raw materials to develop its own biodiesel industry (Ong *et al.,* 2011).

Table 1.1 :Top 10 countries in terms of absolute biodiesel production (Ong *et al.*, 2011)

No	Country	Volume(million litres)	Production cost (USD/L)
1	Malaysia	14,450	$0.53
2	Indonesia	7,595	$0.49
3	Argentina	5,255	$0.62
4	USA	3,212	$0.70
5	Brazil	2,567	$0.62
6	Netherlands	2,496	$0.75
7	Germany	2,024	$0.79
8	Philippines	1,234	$0.53
9	Belgium	1,213	$0.78
10	Spain	1,073	$1.71

The overall production of biodiesel includes two major challenges which are cost of the feedstock and conversion process of oils to biodiesel. Furthermore, using raw materials from own plantations will enable biodiesel developers to control the cost and quality of the biodiesel production more efficiently because process improvements and optimization help reduce the biodiesel conversion process costs. The extraction of oil from the feedstocks and conversion (transesterification) of oils (fatty acids) to biodiesel (alkyl esters) are the main steps involves in biodiesel production. This steps are important because without these steps biodiesel production is not possible. Commonly the method ulitize to indicate these two steps simultaneously or in series include conventional heating, high pressure and temperature reactions such as thermal liquefaction and pyrolysis. These methods are employed based on the feedstock type and quality (Gude *et at.,* 2013) . With the increase demand of biodiesel, so the new novel methods for increasing and enchanging biodiesel production being researched in the context of enchanging and increasing the biodiesel production time and energy consuming which leads higher cost production.

The conventional method have some disadvantages such as low level of triglycerides conversion. It is because the reaction ony occur interfacial region between the liquids as an alkali catalyst are essentially insoluble in the oil phase due to of limitation in mass and heat transfers. Therefore, vigorous mixing and heating using sonochemistry approach are required to increase the area contact between two spaces and causing to increase the rates of reaction. According to Agustian, (2012) the conventional process the reaction tempature required maintain at 60°C for one until two hours for get 97% until 98% yield of biodiesel. Besides that, the yield of biodiesel is rise up to 97% in one hour reaction time at 70°C with molar ratio 6:1 (methanol to oil).

1.2 Problem statement

The mechanical stirring is one method of biodiesel production which is takes long reaction times of chemical reaction in transesterification process. Transesterification is a mass transfer-limited reaction, it happen between the interface of feedstock oil and alcohol, which are only partially miscible. Mechanical stirring method only helps to a degree by improving contact between the reagents, but the reaction still very slow because stirring cannot sufficiently emulsify the two liquids in order to increase the interface surface area between them. Thus, there is a need to investigate new production methods which can overcome this disadvantage. Currently ultrasonic method which is the new technologies in biodiesel production can be approach to increase reaction rate in the process. The high intensity ultrasound creates acoustic cavitation, which produces violently and asymmetrically imploding vacuum bubbles, causing shock waves, micro-jets and strong shear forces as well as extreme local temperature and pressure. This results in extremely efficient mixing, leading to ultra-fine, kinetically stable emulsion (nano-emulsions) of miscible liquids. The interfacial surface area between the liquid is, therefore, increased by orders of magnitude which greatly promotes mass transfer-limited reaction such as transesterification. According to Singh *et al*, (2007) the time reaction by using ultrasonic method can be decreased from hours to minutes.

1.3 Objectives of study

The objectives of this research were focused on:

i. To study the application of ultrasonic method in biodiesel production using Jatropha Curcas Oil (JCO).
ii. To development of ultrasonic in-line biodiesel rig used in lab scale.
iii. To determine the optimum yield of biodiesel production using ultrasonic in-line method.
iv. To evaluate the physical properties of the biodiesel produced using jatropha curcas oil compared to American Standard Testing Method (ASTM) standard and chemical properties test to confirm the content of the biodiesel.

1.4 Scopes of study

The scopes of this study focused on:

i. The ultrasonic use constant frequency, variable time and different molar ratio in the rig and is based in biodiesel plant, Universiti Tun Hussein Onn Malaysia (UTHM).
ii. The biodiesel product would be tested using properties standard of American Standard for Testing and Materials (ASTM D6751) biodiesel and Gas Chromatography Mass Spectrometer (GC/MS) to confirm the content of biodiesel.
iii. The transesterification process was performed at 3, 5 and 7 minutes for the reaction time with molar ratio 6:1, 9:1 and 12:1
iv. The rig used would be the one fabricated by the Prototype Research Grant Scheme (PRGS) team, located in Universiti Tun Hussein Onn Malaysia (UTHM).
v. This project would utilize ultrasonic reactor to reduce the process time. The miniature rig that will be used is at the Universiti Tun Hussein Onn Malaysia (UTHM) biodiesel plant.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will focus on a few options used to increase conversion rates and yields of the esters to bring save production cost and improve the overall quality of the product of biodiesel. The types of feedstock, factors affecting transesterification reaction, biodiesel properties and technology of biodiesel production will be discussed. From the current technology of biodiesel production ultrasonic method was selected to process biodiesel improve yielding and short time reaction in processing biodiesel. The previous studies related to biodiesel production and physical and chemical properties will be used as a guideline to conduct an improvement to the ultrasonic process.

2.2 Definition of biodiesel

Biodiesel is a non-oil based diesel fuel, comprises of the monoalkyl esters of long chain fatty acids which originated from renewable lipid sources. Biodiesel is commonly produced through the reaction of a vegetable oil or animal fat with methanol within the vision of a catalyst to yield glycerine and biodiesel (chemically called methyl esters). According to United State Environmental Protection Agency, biodiesel can be defined as a pure fuel or as a fuel additive and is a legal fuel for marketing. Biodiesel is an option fuel which can be used as a part of neat form or mixed with petroleum diesel for use in compression ignition (diesel) motors. It is physical and chemical properties can be identified through the operation of diesel motors such as petroleum-based diesel fuel. The determination for biodiesel is endorsed by the American Standards for Testing and Materials (ASTM) under code number 6751 (Biodiesel Definition and Benefits).

2.3 Malaysian National Biodiesel Policy

According to Chin, (2011) Malaysia is the largest exporter and the second largest producer (after Indonesia) of crude palm oil (CPO) which is in 2009 Malaysia produced 17.6 million tonnes of CPO. The aim of replacing biodiesel for fossil fuels used at the moment should not lead to higher consumption of diesel by the thought of availability and renewability of this kind of fuel. In order to promote more renewable energy resources, Malaysia has launched a National Biofuel Policy. This policy was finally established in 21st of March 2006 and is supported by five strategic thrusts with the period of the implementation of short term, medium term and long term. This policy is interchangeably known as the National Biodiesel Policy and is formulated after extensive consultation with all stakeholders and as a result of research findings by Malaysian Palm Oil Board (MPOB) since 1982. Figure 2.1 shows an overview of the National Biofuel Policy (Chin, 2011).

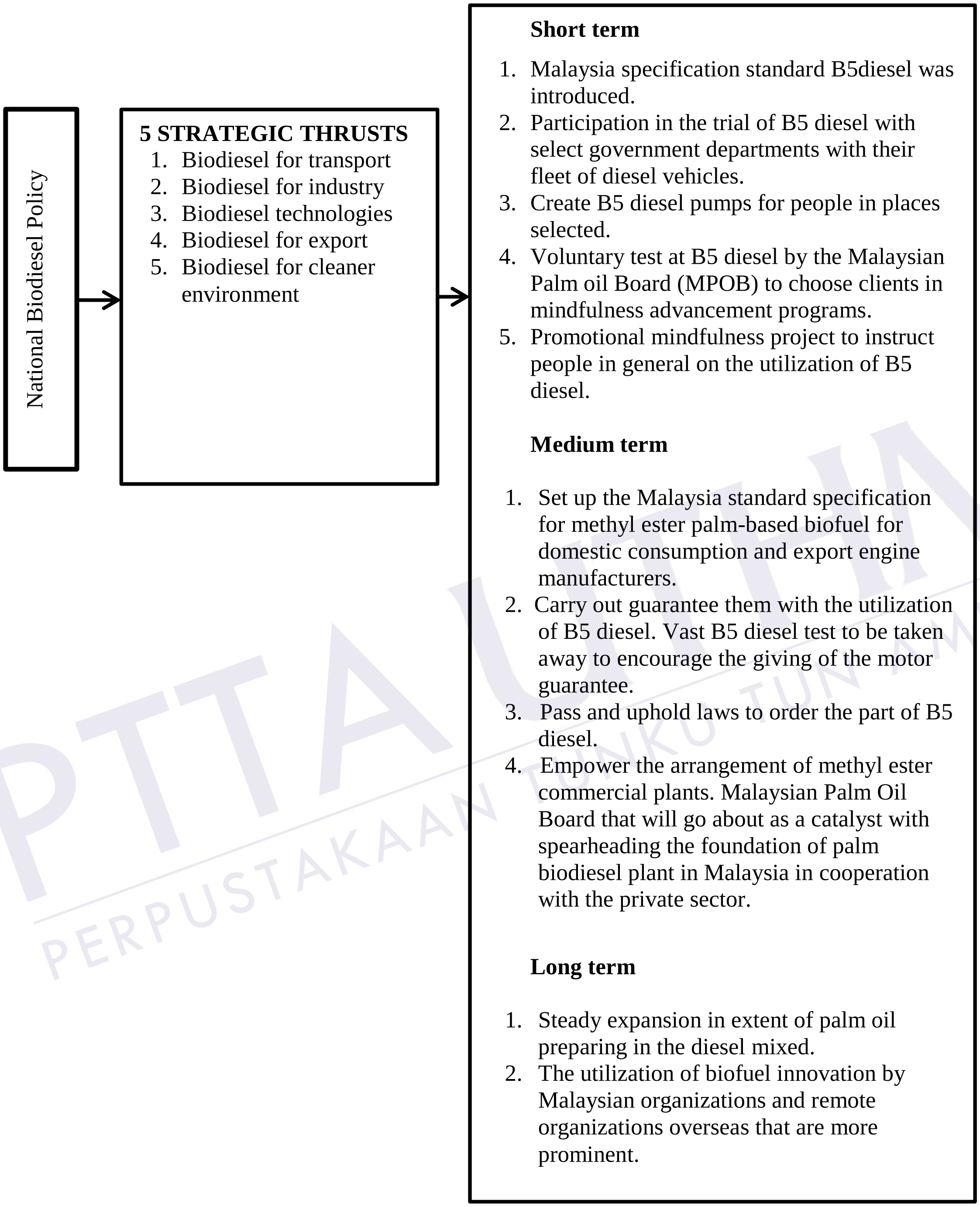

Figure 2.1: Implementation of National Biofuel Policy in Malaysia (Chin, 2011)

2.4 Feedstock of biodiesel production

Nowadays, the world is currently producing biofuel in search of replacement diesel fuel (Fernandes *et al.,* 2013). Samniang *et al.,* (2014) also reported that biodiesel as an alternatives fuel for diesel engines for over a decade. The biodiesel concept as an alternative diesel fuel has been gaining great importance worldwide for its good quality exhaust, sustainability and biodegradability. Malaysia is also one of the countries that suffered from this crisis. According to by British Petroleum statics, oil production in Malaysia in 2001 has decreased from 32.9 million tons to 32.1 million tons in 2010. Besides that, Yunus *et al.,*(2013) also reported the corresponding consumption of oil has increased from 22 million tons in 2001 to 25.3 million tons in 2010. So the production of biodiesel also impact by government subsidy and other political factors which is the types of feedstock one of the factor.

2.4.1 Types of feedstock

In order to solve the petroleum minimization and pollution, biodiesel from biomass can be one of realistic alternative renewable fuel. The feedstock in biodiesel production such as algae, animal fats and vegetable oil such as palm, jatropha, rapeseed and sunflower. Brazil and United States are encouraging ethanol as a potential biodiesel which derives from sugar cane and corn; meanwhile, Asian countries such as Malaysia, Indonesia, and India have been encouraging comprehensively on palm oil and jatropha as source as biodiesel. Table 2.1 show the types of feedstocks that used for production biodiesel in Malaysia.

Table 2.1: Types of feedstocks that used for production biodiesel in Malaysia.

Feedstocks	Characteristic	Region	Author
Palm oil	i. Height of 20–25 m with a life cycle of about 25 years and full production is reached 8 years after planting ii. Used for cooking, and as a raw material for margarine production and as an additive for butter and bakery products iii. Pure palm oil is semisolid at room temperature (20–22°C), and in many applications is mixed with other vegetable oils, sometimespartially hydrogenated	Malaysia, Indonesia	(Ahmad *et al.*, 2012 ; Romano and Sorichetti, 2011; Lim and Teong, 2010)
Jatropha	i. Jatropha plants become productive after 3 or 4 years, and their lifespan is about 50 years ii. Oil yield depends on the method of extraction; it is 28–32% using presses and up to 52% by solvent extraction iii. Variety; it requires little water or additional care; therefore, it is adequate for warm regions with little fertility iv. Productivity may be reduced by irregular rainfall or strong winds during the flowering season	Asia / Australia	(Ahmad *et al.*, 2012 ; Romano and Sorichetti, 2011; Lim and Teong, 2010)
Soybean	i. Depending on environmental conditions and genetic varieties, the plants show wide variations in height ii. Biodiesel production form soybean yields other valuable sub-products in addition to glycerin: soybean meal and pellets (used as food for livestock) and flour (which have a high content of lecithin, a protein). iii. Grain yield varies between 2,000 and 4,000 kg/hectare. iv. Since the seeds are very rich in protein, oil content is around 18%.	East Asia United States, Brazil, Argentina, China, and India	(Ahmad *et al.*, 2012 ; Romano and Sorichetti, 2011; Lim and Teong, 2010)

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