UNIVERSITI TEKNIKAL MALAYSIA MELAKA

EFFECT OF WASTE MUSSELS ON BIODIESEL PRODUCTION FROM RUBBER SEED OIL USING MICROWAVE 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

MUHAMMAD FAIZ BIN MOHAJIR
B071510386
960410016899

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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:

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ABSTRACT

Rubber seed oil (RSO) has been investigated as a potential source for an alternative feedstock of biodiesel production. The primary aim of this study is to investigate the effects of using waste mussel shells as a heterogeneous catalyst on the transesterification process of very high free fatty acid (FFA) rubber seeds oil with methanol under microwave irradiation method. Waste shells are used as a source of calcium oxide (CaO) heterogeneous catalyzing process and their effect on transesterification process has been studied. The waste mussel was experience process of calcinations to obtain CaO. Then, the process of acid esterification was first used to reduce the high FFA rubber seed oil. The transesterification process was carried out afterward with the used of domestic household microwave by Samsung brand, followed by the drying process to remove all excess water and methanol content. The maximum reaction conditions in the microwave batch reactor are recorded around molar ratio of methanol to oil of 6:1, 12 wt% mussel catalyst and 9 minute of reaction time with 400 watt microwave power. All biodiesel properties are analyzed in accordance with ASTM D 6751 and EN 14124 standards for further analysis. It can be concluded that transesterification assisted by microwave irradiation is effective to improve biodiesel yield and produce superior quality biodiesel. It is believed that the discovery of this study would benefit the biodiesel synthesis assisted by microwave irradiation at industrial scale.

ABSTRAK

Minyak biji getah (RSO) telah dikaji sebagai sumber yang berpotensi sebagai bahan mentah alternatif bagi pengeluaran biodiesel. Tujuan utama kajian ini adalah untuk mengkaji kesan menggunakan sisa cengkerang sebagai pemangkin heterogen pada proses transesterifikasi minyak biji getah yang sangat tinggi asid lemak dengan metanol di bawah kaedah penyinaran gelombang mikro. Cengkerang terbuang digunakan sebagai sumber kalsium oksida (CaO) bagi proses katalis heterogen dan kesannya terhadap proses transesterifikasi telah dikaji. Kulit kupang akan mengalami proses kalsinasi untuk mendapatkan CaO. Kemudian, proses pengesteran asid pertama kali digunakan untuk mengurangkan minyak biji getah yang mempunyai FFA yang tinggi. Proses transesterifikasi dilakukan selepas itu dengan menggunakan ketuhar gelombang mikro jenama Samsung, diikuti dengan proses pengeringan untuk membuang semua kandungan air dan metanol yang berlebihan. Kondisi reaksi maksima dalam reaktor kelompok ketuhar gelombang mikro dicatatkan pada nisbah molar minyak ke metanol 1: 6, 12 wt% pemangin kupang,9 minit masa reaksi dengan 400 watt kuasa gelomabang mikro. Semua sifat biodiesel dianalisis mengikut piawaian ASTM D 6751 dan EN 14124. Dapat disimpulkan bahawa transesterifikasi dibantu oleh penyinaran gelombang mikro yang berkesan untuk meningkatkan hasil biodiesel dan menghasilkan biodiesel berkualiti tinggi. Adalah dipercayai bahawa penemuan kajian ini akan memberi manfaat kepada sintesis biodiesel yang dibantu oleh penyinaran gelombang mikro pada skala yang lebih besar.

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CHAPTER 1

INTRODUCTION

1.1 Background

Petroleum is the one of the major source of energy used in most parts of the world that plays important role in human being development. However, petroleum consumption has enlarged over the last few decades due to human and industrial growth, which has resulted in running down of fossil fuel reserves and rising petroleum price. Therefore, explore an alternative source of sustainability and renewable energy such as water, wind, sunlight, geothermal heat and biofuel has get the interest with the possible to solve many social issues as recently like the increasing crude oil prices and environmental concerns such as global warming and air pollution.

The biodiesel fuel has recently increased attention and has been proposed as a candidate for replacing petroleum diesel. Petroleum diesel can be mixed with biodiesel because it has the same characteristics as a less hazardous release of exhaust. Animal fats and vegetables oil are some of the biodiesel that is made from sources of renewable biological. The oils from animal fats and vegetables crop and are processed or extracted to get the crude oils that pass through transesterification process. There is great diversity of raw material in biodiesel production that can be used such as jatropha, soybean, palm, tallow, waste cooking oil as well as rubber seed. Biodegradability, bigger cetane number, renewability and good quality exhaust gas emissions is the advantages of biodiesel as a petroleum diesel. American Society for Testing and Material (ASTM) standard and EN standard are the referred standard that used for test quality of biodiesel in U.S (United States) and EU (European Union).

At present, the Brazil, EU and U.S are the main global player of bio fuel. In the middle of 1970s, the United States and Brazil have started their bio fuel development when a fuel price is increasing and the concerns over domestic energy security. By year 2000, annual production of biodiesel and bio ethanol has reached respectively 15 and 0.8 million tons. After that, bio fuel production has grown to 80.1 million tonnes since then 2009, with major output from the United States, the EU and Brazil (Mardhiah et al. 2017). Globally, the influential of bio fuel production are US and Brazil and followed by some Asian countries that actively promote bio fuel production.

Tactically, Malaysia as the world's second-largest manufacturer of palm oil is an vital member of the global dynamics of biodiesel production that have produces 40 % of global palm oil volume requiring the country's. For years, from 1960 to 2013 Malaysia has increased substantially from 2.6 million tons to 19.7 million tons in crude palm oil production. Malaysia is worried about the release of greenhouse gases and the dangers of global warming when petroleum dependence is increasing every year. These apprehensions lead to the announcement of long-term recovery policies such as BIA (Biofuel Industry Act) and NBP (National Biofuel Policy) that aimed in sustainable palm oil production and palm based biodiesel in Malaysia, promoting renewable energy use and decreasing fossil fuel dependence (Johari et al. 2015).

Main obstacle to the product commercialization is the higher biodiesel costs than petro diesel. In industrial countries, the cost of biodiesel production become higher than the cost of producing fossil-derived fuels due to the high demand for edible oil. Commonly, biodiesel production costs are strongly influenced by seasonal crops yield, crop geographic areas, local crude petroleum prices and other factors. The produce of crude oil from non-edible plant oils are found to guarantee for biodiesel production. In recent years, there have been numerous works reported on the production of biodiesel using higher rubber seed oils used as alternative diesel fuel as it is an inexhaustible oil that can produce enough oil for the industry.

Malaysia is become one of the largers rubber suppliers in the world with rubber estates of about 1,021,540 reported in 2009 which produces more than 120 kilos of rubber per year (Muhamad et al. 2013). So far, this seed is less used and has been discarded as a waste over the years. Thus, for renewable fuels it is considered as a cheap raw material. However, rubber oil is the high amount of free fatty acid that is unwanted in catalyzed transesterification reaction. As the consequences, the conversion of rubber seed oil to biodiesel becomes more complicated. As a result, further study is necessary to find out the most appropriate parameter for rubber oil transesterification reaction process.

1.2 Problem Statement

i. A good quality biodiesel with a proper method was developed by using any variable source from the daily life that can be used as a raw material.

ii. The usage of waste mussel catalyst as the enhancement in the oil reducing the cost of using acidic/alkali type of catalyst

iii. Each process parameters in biodiesel production give the unique effects on biodiesel yield and characteristics

1.3 Objective

i. To prepare the good quality quality biodiesel from crude Rubber Seed Oil through transesterification via microwave.

ii. To prepare and characterize mussel shell as a heterogeneous catalyst

iii. To study the effect of process variable (reaction time, amount of catalyst, microwave power and molar ratio of oil to methanol), on the production yield.

1.4 Scope

i. To ensure that the biodiesel production meet all the standard (ASTM D6751 and EN 14214)

ii. To convert a calcium carbonate, $CaCO_3$, mussels to heterogeneous calcium oxide, CaO via calcinations process in catalyst preparation.

iii. To study the feedstock properties that may influence the final properties of the biodiesel

CHAPTER 2

LITERATURE REVIEW

2.1 Biodiesel

In the past three decades, studies and development in green energy has exploded, build up hundreds of promising new technologies that can decrease our reliance on coal, oil, and natural gas. Green energy comes from natural sources such as sunlight, rain, wind, plants, tides, geothermal heat and algae. These power resources are renewable, meaning they are in nature replenished. Renewable energy sources also have a minor impact on the surroundings than fossil fuels, which generate pollutants such as greenhouse gases as a side-effect that contributing to climate change.

Rather than burning biomass to create energy, sometimes these renewable organic materials are transformed into fuel. In order to remain the environment clean and fresh, environmentally friendly fuels are required to be produced. Biodiesel is clean renewable bio energy as it can be produced from renewable sources such as animal fats, vegetables oil and micro-algal oil. According to Mardhiah et al. (2017), state that property of biodiesel for example low emission, carbon neutral biodegradable and non-toxic are better to those of non-renewable fuels

Soy bean oil, olive oil, sunflower oil, peanut oil, colza oil, sesame oil, cotton oil, palm oil and corn oil are the first-generation biodiesels that are created from edible vegetable oils. Nevertheless, biodiesel production using edible vegetable oils is not a practicable solution in the long run as this approach leads food and global fluctuations and confusion in food prices. Therefore, biodiesel formed from non-edible plant-based oils such as *Madhuca indica (mahua) , Sterculia feotida, Calophyllum inophyllum, Pongamia glabra (koroch seed), Pongamia pinnata (karanja), Hevea brasiliensis (Rubber seed), Azadirachta indica (neem), Camelina sativa, Linseed, Lesquerella fendleri, Jatropha curcas* and *Nicotiana tabacum* come to replace as the second generation biodiesel. According to Milano et al.2018, second-generation biodiesel is verified to have good oxidation stability, high yield biodiesel and favorable cooling properties. Moreover, fuel specifications give in ASTM D6751 and EN 14214 standards can be meet by second-generation biodiesel that making this fuel promising as a diesel substitute.

Vegetable oil that has been regarded as raw materials for biodiesel production is second-generation food stocks or non-edible. Non edible oil that used for the production of biodiesel such as palm oil and rubber seeds is due to their ability to overcome food problems over and fuel crises. Furthermore, they are economically comparable to that edible oil and these are readily available in many parts of the world, especially homeland that are not suitable for food crops, reduce the rate of deforestation, more efficient, more environmentally friendly, produce useful by-products.

2.2 Raw Material

The biodiesel production process begins with pre-treatment for the raw material, as the quality feedstock brings significant cause of biodiesel yield. The extraction of rubber seeds are well known to be high in oil (Hassan et al. 2014). Comparable to drying oils commonly used in surface coating, the content in the dried kernel varies from 35 to 45 % and it is semi-drying and consists of 17 to 22 % saturated fatty acids and 17 to 82% unsaturated fatty acids. (Abubakar et al. 2014). Grains are collected by peeled the rubber seeds manually and 2.90 grams and 1.73 grams are respectively weights for rubber and kernel seeds. 3.25% (average) weight loss was observed after the kernels were dried for 12 hours at 50 ˚C before the intake of oil

2.2.1 Rubber oil

Rubber oil contains 80 to 90% hydroxyl fatty acids (double bonds and hydroxyl groups), enhances lubrication compared with ordinary vegetable oils and becomes the main candidate as an additive for diesel fuel. Because of the higher grain content in unsaturated fatty acids, a step of acid esterification is used to reduce higher free fatty acids (FFA). Two stages for the production of biodiesel from rubber seed oil that is using esterification reaction method and then followed by transesterification reaction (Widayat et al. 2013). Acid esterification was done in order to reduce the value of oil acid less than 5 mg KOH/g of oil, on rubber seed oil prior to trans-esterification process

Biodiesel is a combination of alkyl esters of fatty acids produced from animal fats or vegetables oil. In order to produce an ester mixture (biodiesel), transesterification reaction of vegetable oils having a number of fatty acids responds with the right alcohol with the presence of alkyl catalysts. Acid value is measure to calculate the amount of acid present in the biodiesel sample. The free fatty acids in oil are approximately half the acid value. This is highly undesirable and will produce soap when the acid value of oil is high and free fatty acids in oil will react with alkyl catalysts. Pre-treated will be done crude oil in present of suitable catalyst to reduce the acid value at lower level that less than 4 and it will avoid from saponification reaction from happen. (C. S. Sabarish, et al. 2016).

2.2.2 Alcohol

One of the most important raw materials for biodiesel production is alcohol. Alcohol is a primary and secondary aliphatic alcohol consisting of 1 to 8 carbon atoms (I.A. Musa, 2016). Acylation acceptors are methanol and to a slight extent, ethanol that is the most widely used of alcohols for biodiesel production is. Short chain alcohol like butanol, propanol, octanol, tert-butanol, branched alcohol and isopropanol is the other alcohols that are used in producing biodiesel, however these alcohols are costly.

The mainly used alcohols in biodiesel production are methanol and ethanol. The mostly often preferred is methanol because of its chemical and physical advantages. Beside, can be smoothly dissolved in sodium hydroxide (NaOH) and its response with triglycerides is fast. A. Demirbas, (2008) remarked that compared to ethanol, methanol also recognized as ‘‘wood alcohol”, is generally easy and simpler to find. In addition variety of alcohol can reacted triglycerides but the short-chain alcohols provide better conversions under the same reaction time.

Nevertheless, in the case of methanol alcohols such as iso-propanol, and propanol are regularly obtained from petrochemical materials it is resulting from natural gas. The advantages due to being fewer toxic, carbon dioxide neutral, environmentally, and due to the renewability make the ethanol the mostly suitable alternate to substitute methanol. Based on I.A. Musa in 2016 stated that as long as the completed biodiesel product meets ASTM D6751, biodiesel that formed from a variety of alcohols, make the nature of alcohol used in the production process does not make any chemical changes. Nevertheless, due of steric hindrance effect, through transesterification reaction, higher chain molecular alcohols are usually avoided.

2.2.3 Catalyst

Catalyst is something that makes a chemical reaction react more quickly without itself being changed. Transesterification and production can be affect by one of the major factors that is catalyst concentration. In the presence of catalysts, biodiesel, an alternative diesel fuel is synthesized by direct transesterification of animal fat or vegetable oil with short chain alcohol. With using of homogeneous catalysts (alkali and acids) or heterogeneous catalysts (solid or basic acids), transesterification reactions can be catalyzed

The use of heterogeneous catalysts can improve the efficiency of the reactor design, enabling the continuous process, and improving the biodiesel production economy. The most advantage of is heterogeneous catalysts does not produce soap through neutralizing free fatty acids, which facilitates the process of purification and separation. Calcium oxide (CaO) is among the heterogeneous catalysts that most studied and has the great potential for biodiesel production due to the desired activity and low price (Istadi et al. 2015). The physical characteristics of CaO-based oxides showed progression of thermal stability and surface area. This may because of the large increase in oil dispersion into the catalyst surface which has been converted into hydrophobic. Although homogeneous base catalysts give faster reaction rates than heterogeneous catalysts, separation of homogeneous catalysts from the reaction mixture is very expensive and hard makes homogenous catalyst less selected (Sree and Kuriakose 2015). The homogeneous catalyst catalysts are also not preferred as they are difficult to recycle and operate at high temperatures as well as pose a risk to serious corrosion and environment problems. Table 2.1 shows the comparison between homogeneous and heterogeneous catalysts for biodiesel production.

In terms of solid acid catalysts, the catalysts based on zirconia and calcium oxide show the potential to be used as recyclable catalysts after mild regeneration without any loss in activity. Zirconia sulfates show good catalysts for vegetable oil transesterification because of its strong acid properties, high activity and selectivity for fatty acids by alcohol selection. Acid catalysts require high catalysts concentrations and alcohol to oil molar ratio to get acceptable transesterification conversions (Istadi et al. 2015). Acid catalysts at the same time can running esterification of free fatty acids (FFA) and oil transesterification, they may help in low processing quality feedstock, low-cost, thus decreasing overall production cost. Alkali catalyzed transesterification reaction is the mainly adopting methods for production of biodiesel because it is easy to extract the respective esters (Meher, L.C. et al. 2006)