MICROWAVE ASSISTED TWO STEP IN-SITU METHOD FOR BIODIESEL PRODUCTION FROM JATROPHA CURCAS L. SEED

HAMIDREZA JALILIANNOSRATI

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ABSTRACT

This study explored the development of two steps process in a batch reactor system capable of operating in a microwave oven irradiation for biodiesel production from jatropha seed by using in–situ method. The first step, in-situ esterification, converted the free fatty acid by H_2SO_4 as acid catalyst and the second step, in-situ transesterification converted the triglycerides by KOH 5N in ethanol as base catalyst in the microwave system. This was also the first investigation, which carried out two-step process from jatropha seed with out any washing and purification between esterification step and transesterification step. Furthermore, microwave irradiation assisted extraction of oil from Jatropha curcas seed with different solvent was carried out for two size seed with diameters of A<0.5 mm & 0.5<B<1.1 mm. The study concluded that maximum oil efficiency of 77.78% and 67.76 % were obtained for jatropha curcas seeds for size A and B respectively in 20 minute with ethanol by microwave system. Response surface methodology (RSM) was employed to examine the relationship between process variables and predicting the optimal conditions. The highest conversion of 93.84% at optimum reaction conditions of 31.07 minute irradiation time, 10.32% (V/W) ratio of ethanol to seed, 10.32 %wt catalyst amount, and 270 rpm agitation speed was achieved for size A in step 1. For size B the highest conversion of 90.19% at optimum reaction conditions of 35.5-minute irradiation time, 11.2%(V/W) ratio of ethanol to seed, 7.95%wt catalyst amount, and 285.12rpm agitation speed was reached. The highest final biodiesel conversion of 92.95% at optimum reaction conditions of 33.07 minute irradiation time, 13.82ml catalyst amount, and 202.64rpm agitation speed was achieved for size A in step 2. For size B the highest biodiesel conversion 92.67 at the same optimum reaction conditions for seed size. The final product was found to be in a reasonable agreement with EN-14214 European standard for physical properties.

ABSTRAK

Kajian ini meneliti perkembangan dua langkah proses dalam sistem reaktor kelompok yang boleh beroperasi dalam sinaran ketuhar gelombang mikro untuk penghasilan biodiesel daripada biji jatropha dengan menggunakan kaedah in-situ.Langkah pertama, pengesteran in-situ iaitu penukaran asid lemak bebas oleh H_2SO_4 sebagai pemangkin asid dan langkah kedua, transesterifikasi in-situ menukar trigliserida oleh KOH 5N dengan etanol sebagai pemangkin alkali dalam sistem gelombang mikro. Ini merupakan penyiasatan pertama,yang menjalankan dua langkah proses dari biji jatropha tanpa membasuh dan penulenan antara langkah pengesteran dan langkah transesterifikasi .Tambahan pula, penyinaran gelombang mikro disertai dengan pengekstrakan minyak dari benih Jatropha curcas dengan pelarut yang berbeza telah dijalankan dengan menggunakan dua saiz benih dengan diameter A<0.5mm& 0.5<B<1,1mm. Kajian ini menyatakan bahawa kecekapan maksimum minyak ialah 77.78% dan 67.76% yang diperolehi bagi jatropha curcas benih untuk saiz A dan B masing-masing dalam masa 20 minit melalui etanol dengan menggunakan sistem gelombang mikro Kaedah Respon Permukaan(RSM) telah digunakan untuk mengkaji hubungan antara pembolehubah-pembolehubah proses dan menjangka keadaan optimum. Penukaran tertinggi adalah 93.85% pada keadaan tindak balas optimum 31.07 minit masa penyinaran, 10.32%(V/W) nisbah etanol pada benih, 10.32%wt berat jumlah pemangkin, dan 270rpm kelajuan pengacauan telah diperoleh untuk saiz A dalam langkah 1. Untuk saiz B penukaran tertinggi ialah 90.19% pada keadaan tindak balas optimum 35.5minit masa penyinaran, 11.2%(V/ W) nisbah etanol pada benih, 7.95% berat pemangkin,dan kelajuan pengacauan 285.12rpm telah diperoleh. Penukaran akhir biodiesel tertinggi adalah 92.95 pada keadaan tindak balas optimum 20 minit masa penyinaran, 9% berat jumlah pemangkin, dan 250rpm kelajuan pengacauan telah dicapai untuk saiz A dalam langkah 2. Untuk saiz B, penukaran tertinggi biodiesel 92.67% dan pada keadaan tindak balas optimum 15minit masa penyinaran,. Produk akhir telah didapati menepati dengan standard EN-14214 Eropah untuk beberapa sifat-sifat fizikal.

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LIST OFABBREVIATION

AR	-	Analytical Regent
ANOVA	-	Analysis of Variance
ASTM	-	American Society of Testing and Materials
AV	-	Acid Value
C	-	Heat capacity
CCDs	-	Central composite designs
DEC	-	Dielectric constant
DOE	-	Design of expert
FAME	-	Free fatty methyl ester
FAEE	-	Free fatty ethyl ester
FFA	-	Free fatty acids
JCL	-	Jatropha curcas L.
H_2SO_4	-	Sulfuric Acid
GC	-	Gas Chromatography
GC-MS	-	Gas Chromatography-Mass Spectroscopy
KOH	-	Potassium Hydroxide
MAE	-	Microwave-assisted extraction
$MgSO_4$	-	Magnesium Sulfate
ml	-	Milli Liter

mm	-	Milli Meter
N	-	Normality
NaOH	-	Sodium Hydroxide
rpm	-	Round Per Minute
RSM	-	Response Surface Methodology
SEM	-	Scanning Electron Microscope
TG	-	Triglyceride
W	-	Watt

LIST OF SYMBOLS

ε'	-	Dielectric constant
ε''	-	Dielectric loss factor
δ	-	Loss tangent
ϵ	-	Error

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Nowadays, the global industrialization and population growth make the world energy demand to be enhanced considerably. Energy known as catalyst for development has been divided into non-renewable (fossil fuel) and renewable energy. Lately, fossil fuel is known as the main source of energy in the world. Malaysia has consumed fossil fuel of 160000 per day, in 1980. Figure 1.1 indicates this number has risen to 554000 per day in 2009 (Independent statistics & Analysis, eia, website).

Figure 1.2 indicates the percentage of fossil fuel types consuming in United State America (USA) in 2007. It is obvious that the fossil fuel such as coal, natural gas, and petroleum include the most energy consumption in USA as establishment the 86% of energy consumption in 2007.

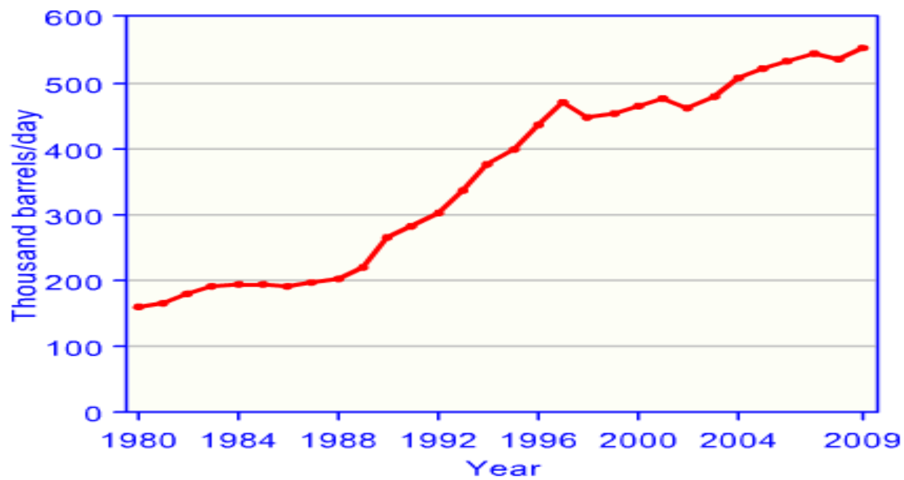

Figure 1.1 Petroleum consumption in Malaysia

(Independent statistics & Analysis, eia, website)

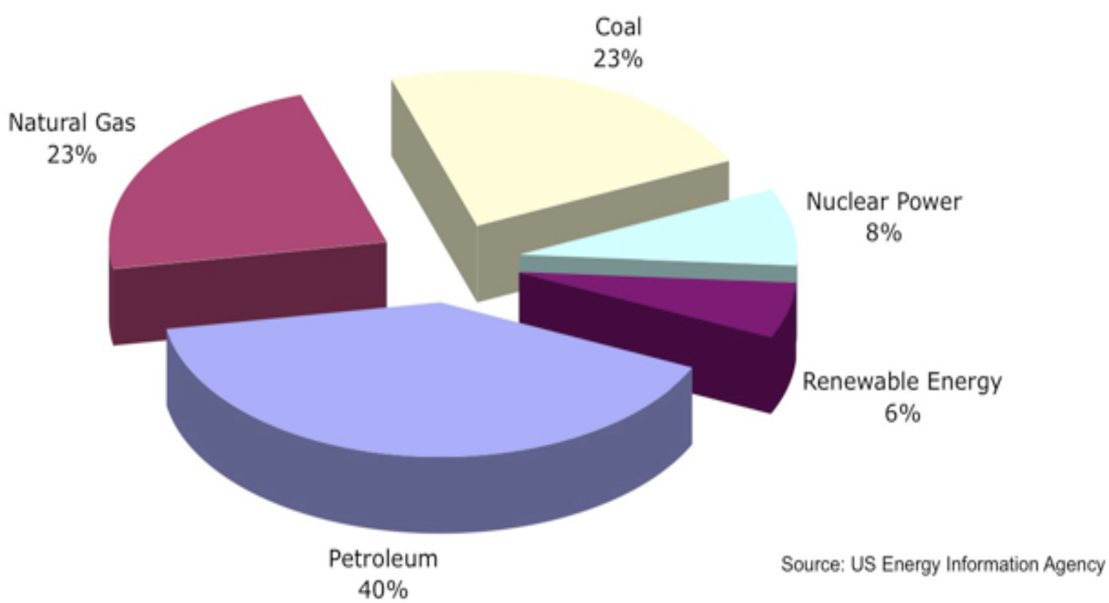

Figure 1.2 Energy consumption in USA in 2007

(The Oil Drum website, 2007)

Over 100 years the fossil fuel has been used in spite of it sources are restricted in the environment. Thus, finding a proper substitution of fossil fuel with new energies should take into account since increasing renewable energy has become essential to decrease the reliance on petroleum. In addition, fast-depleted and restricted resources as well as the increasing demands of diesel fossil fuel lead to price rises of crude oil.

Environmental problems might be one the important reasons for developing green and environmentally safe fuels (Demirbas, 2009). Greenhouse gases, which made by fossil fuel, can affect the environment. Therefore, renewable fuels have received a lot of attention as an alternative to fulfill an increasing energy demand. Biofuel can be considered as a major sustainable energy and contributor in the future. Nowadays, around 90% of the biofuel market is confined by bioethanol and biodiesel. Biodiesel usually known as a transesterification of triglycerides (animal fats and vegetable oils) with alcohols in the presence of catalyst (acid, alkali or enzymes) which leads to configuration of fewer viscous fatty acid alkyl esters.

Biodiesel is known as a “green fuel” since it is a benign, renewable, harmless and biodegradable substance. Furthermore, an oxygenated fuel (over 10% oxygen), which improves fuel combustion does not include sulphur constituents therefor its sulphur emission is slight (Gerpen, 2005). Major decline in hydrocarbons, CO and soot appear in the exhaust gases, after combustion are other problems of using these resources. Moreover, biodiesel can combine with common diesel in diesel engines with no slight modifications. Figure 1.3 indicates the chemical distinction between the biodiesel and petro diesel.

$CH_3.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_3$
Cetane, a linear diesel fuel molecule.

$CH_3.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.CH_2.\mathbf{COOCH_3}$
Methyl Palmitate, a typical biodiesel fuel molecule.

Figure 1.3 Chemical difference between the petro diesel and biodiesel

The background of diesel and employing vegetable oil as fuel in engine was planned in Rodulf Diesel in 1878 (Diesel R., 1912). Then various feedstock such as soybean, palm oil, castor oil, cottonseed oil and were studied in the “significant” times (Mayne, 1920; Mathot 1923; Manzella, 1939). Nevertheless, the term ‘Biodiesel’ was known in Chinese paper in 1988 (Knothe 2005).

There are various procedures which can be implemented to produce biodiesel: (1) based-catalyzed transesterification (Dorado et al., 2002), (2) acid-catalyzed transesterification (Mittelbach et al. 1996; Lotero et al., 2005) (3) integrated acid-catalyzed pre-esterification of FFAs and base-catalyzed transesterification (Canakci and Van Gerpen, 2003; Ramadhas 2005) (4) enzyme-catalyzed transesterification (Wei et al., 2004; Du et al., 2004) (5) hydrolysis and acid-catalyzed esterification (Kusdiana and Saka, 2005) (6) pyrolysis (Damirbas A., 2003) (7) supercritical alcohol transesterification (Saka and Kusdiana, 2001) (8) based-catalyzed in-situ transesterification (Kasim and Harvey, 2011; Ginting et al.,2012), (9) acid-catalyzed in-situ esterification (Shiu et al , 2009) (10) enzyme-catalyzed in-situ transesterification (Erzheng et al., 2009) (in situ bas cat thesis) and integrated acid-catalyzed in situ esterification of FFAs and base-catalyzed in situ transesterification (Shiu et al , 2009) . The increase of biodiesel productions can be seen in figure 1.4.

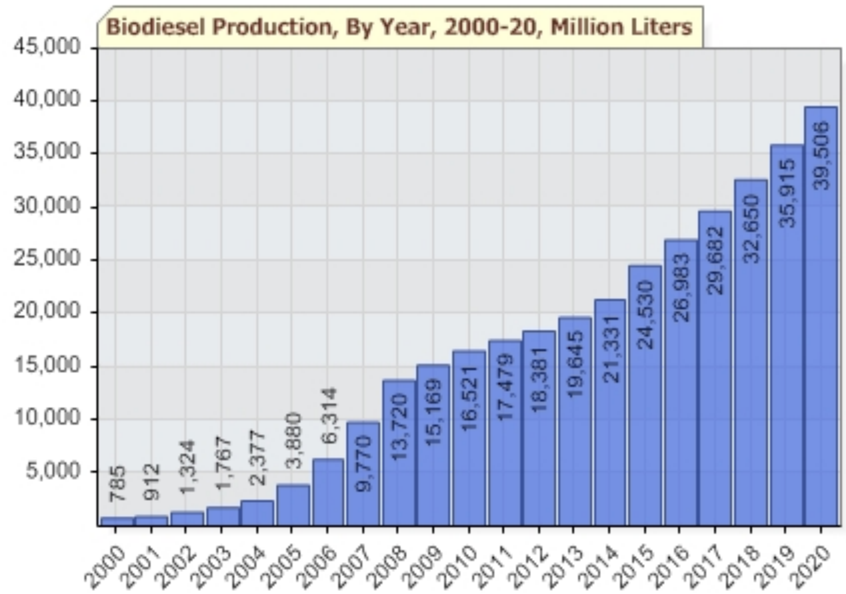

Figure 1.4 Biodiesel production (Earth trends website, 2009)

Biodiesel is composed of alkyl esters that usually made from either the transesterification of triglycerides (TG) in oils and fats or esterification of free fatty acids (FFA) with short-chained alcohols such as methanol (methanolysis reaction) (Jacobson et al., 2008). Triglycerides are considered as the main constituent of vegetable oil, which has three long chains of fatty acid. In this reaction three fatty acid chains are released from the triglycerides and combined with the alcohol to produce biodiesel or methyl esters and the glycerol as the byproduct.

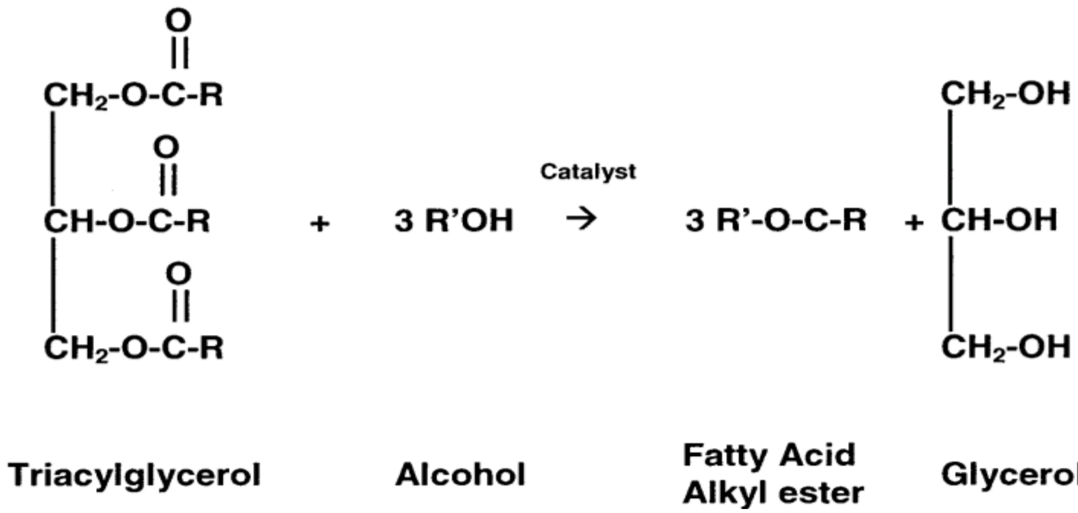

Figure 1.5 Transesterification reaction, where R represents fatty acid chains.

To complete the reaction stoichiometrically, a 3:1 molar ratio of alcohol to triglyceride is essential. In fact, extra amount of alcohol is included to increase the biodiesel yield. Several factors can be affected the transesterification process such as presence of moisture and FFA, the molar ratio of alcohol to oil, reaction time and temperature, catalyst type and concentration, etc. (Freedman et al., 1984).

Transesterification can be catalyzed by acid, base or enzyme (Williams et al., 2007; Zullaikah et al., 2005; Rashid and Anwar, 2008; Ranganathan et al., 2008). Though, enzyme and acid catalysis is usually slower and enzyme catalyst is further costly than base catalyst (Shimada et al., 2002). Hence, base catalysts show a strong preference for the industrial scale. Common base catalysts comprise potassium methoxide ($KOCH_3$), sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium methoxide ($NaOCH_3$). Hydrolysis of the formed alkyl ester to FFA can take place in the absence of moisture in the transesterification reaction. Similarly, due to triacylglycerols ester, the reaction of triacylglycerols can form FFA with water. However, low free fatty acid content in the oil and anhydrous alcohol is essential in the usage of base catalysts to avoid soap formation (Ni and Meunier, 2007). While high FFA-including oil is used as a feedstock of acid-catalyzed the process is further cost-effective for one-step process of transesterification and esterification (Canakci and Van Gerpen, 1999). Hence, in order to gain a low level of free fatty acid the vegetable oil should be refined to produce biodiesel.

Free fatty acid from feedstock was removed by Esterification reaction then converted it to methyl ester by reacting with alcohol in the presence of acid catalyst. Various acids could be commonly used such as sulfuric acid (H_2SO_4), phosphoric acid, hydrochloric acid (HCl) or organic sulfonic acid. H_2SO_4 and HCl Besides, various solid acid catalysts have been used including tungstophosphoric acid and heteropoly acid (Mbaraka and Shanks, 2005). However, there are some problems including slow kinetics, high cost, imperfect conversions, corrosion, and restricted life acid catalyst for esterification reaction. One mole of water was produced from each mole of free fatty acid as shown in Figure1.6

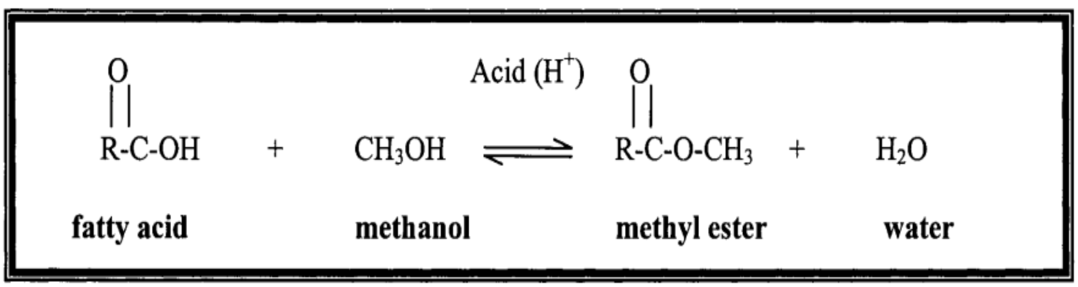

Figure 1.6 Esterification Reaction

Most of the raw material has been derived from edible or non-edible oil of vegetable resources including rapeseed, soybean, palm oil, Jatropha, sunflower, algae and waste oil cooking. Edible oil has broadly used as a feedstock, which can create unsteadiness of the food supply. This feature can be replaced by using non-edible and economical feed stocks such as jatropha, used animal fats, or waste cooking oils. Among non-edible oils, Jatropha has been considered as the most appropriate feedstock for sustainable biodiesel production (Achten et al. 2008;Foidl et al. 1996; Francis et al., 2005; Ye et al. 2009). Due to sociological, environmental implications and economical value Jatropha biodiesel has valuable and proficient for biodiesel production (Juan et al., 2011).

Consequently, the production of biodiesel is costly because the extraction and the refinery of the oil give the highest percentage to the total biodiesel production cost roughly above 70% of the final cost of biodiesel (Zhang et al., 2003; Vyas et al., 2009; Hass et al., 2006). Significant factors such as raw material selection and process used in the biodiesel production influence the production cost. In situ transesterification or reactive extraction known as a single step and integrating the extraction and reaction can reduce the biodiesel production cost.

Other options to decrease the biodiesel cost are accelerating the transesterification reaction and esterification by assisted microwave irradiation. Microwave or radio frequency radiation has been applied to homogeneous alkali-catalyzed ester synthesis reactions (Hernando et al., 2007; Azcan and Danisman, 2007) and for both homogeneous and heterogeneous reaction catalyze by either alkali or acid (Portnoff et al., 2005). Microwave irradiation accelerates the reaction rate. This increase of rate is assumed to be happening due to the catalyst particularly absorbs the applied energy, raising the local reaction speed (Portnoff et al., 2005). Beside the increase of reaction rate, microwave can enhance the purity and yield of biodiesel (Hayes, 2002;Leadbeater and Stencel, 2006; Groisman, and Gedanken, 2008)

Along with the previous studies, this project is intended to investigate biodiesel production from Jatropha curcas with two-step in-situ esterification and transesterification technique by microwave assisted irradiation. This project was also carried out in an ethanolysis batch process and at a laboratory scale with heterogeneous acid catalyst (H_2SO_4) and conventional alkali-catalyzed (KOH).

1.2 Statement of problem

The fast decreasing and restricted resources of fossil fuel, price rises of crude oil and environmental concerns are different reasons to substitute the petroleum products such as diesel and gasoline with green fuel "Biodiesel".

Vegetable oils, edible and non-edible vegetables oils, as renewable energy become important feedstock for biodiesel production in nature. Biodiesel growth, increasing food supply cost and the negative of food versus fuel competition for lipids lead to focus on the development of new resources for lipids, often with stress on non-edible feed stocks such as jatropha.

The catalyst for transesterification process can be homogeneous or heterogeneous, basic, acid or enzyme catalyst. The higher temperature reaction is one of the disadvantages of acid catalyst for transesterification reaction, which also involves corrosion of liquid acid and costly separation. Furthermore, Enzyme catalyst is costly and also become inactive after reaction. Moreover, enzyme catalytic and acid reactions are so slower compared to alkali catalysts (Saifuddin and Chua, 2004; Vicente et al., 2004). With homogenous base catalysts, elimination catalyst from the biodiesel requires a more purification step, which lead to price rises of biodiesel production. Regarding to heterogeneous base catalyzed transesterification still some disadvantages exist due to formation soap for feedstock with FFA levels exceeding 4% and elimination of soaps from the products, which requires great amount of water. However, heterogeneous base catalyzed can be considered as one of the best preferences for transesterification reaction of biodiesel production.

The major barrier to biodiesel commercialization is the high cost of biodiesel rather than petroleum diesel. Minimizing the raw material is one of the methods broadly recognized to reduce the biodiesel cost. However, using waste cooking oil or low cost feedstock such as jatropha cannot resolve the whole problem. In addition, a biodiesel production procedure, which removes organic solvent use for oil extraction and biomass drying, might cause cost savings and significant energy. Prior research made an attempt to remove some of these steps. Another way to reduce the biodiesel cost is in situ esterification and transesterification method that uses the original agricultural component as the resource of triglycerides and free fatty acid for direct esterification and transesterification. By conducting this method the esterification and transesterification reagents may reach triglycerides and free fatty acid resident in oilseeds and attain their esterification and transesterification in a direct way.

An alternative method of biodiesel production is to utilize an acid catalyzed esterification to convert the FFA to ester, followed by a base-catalyzed transesterification reaction to convert the triglycerides into ester. At present this two-step procedure is in primary step of commercialization. Most studies noted that in biodiesel production utilizing a two-step process, a separation step (water washing) was essential for improving the esterification product. Then, the consequential natural phase from the first step was utilized as the initiating material for the next step (transesterification). This study is the first investigation reported on the biodiesel production from jatropha seed using an in-situ process in which esterification was followed directly by transesterification without employing a separation step in between.

In addition, the common heating of a sample leads to important problems such as specific heat, limitations dependent on the thermal conductivity of materials, heterogenic heating of the surface, and density in comparison with microwave irradiation (Metaxas, 1996). Therefore, more studies have lately stressed on the new approach such as microwave irradiation and ultrasound. Survey showed that microwave-assisted chemical reactions are preferred rather than utilizing other synthetic methods since microwave-heating systems can enhance product yields, the reaction rate, and purity of products (Loupy, 2006; Hayes, 2002).

Time consuming run tests and costly equipment are needed to perform the optimization of the parameters included the esterification and transesterification reaction. Therefore, to develop biodiesel production for the industrialization process optimization of the study is significant. (Lee et al., 2011). Hence, software such as Response Surface Methodology (RSM) can decrease or remove a great amount of related costs and laboratory tests.

1.3 Research Objective

The objectives of this research are:

i. To find suitable solvent for oil extraction from jatropha curcas in microwave system.

ii. To evaluate the possibilities of doing acid-catalyzed in-situ esterification and directly followed by base-catalyzed in-situ transesterification technique with H_2SO_4 and KOH respectively to produce fatty acid ethyl ester from jatropha curcas seed by using microwave system and characterize properties of achieved product.

iii. To investigate the effect of time, alcohol/seed ratio, concentration of catalyst, speed of stirrer and size of seed on reducing the free fatty acid (FFA) in the first step and to investigate the effect of time, concentration of catalyst, speed of stirrer and size of seed on conversion of biodiesel in step two.

iv. To determine optimized variety of variables on biodiesel production by employing response surface methodology (RSM) for each step.

1.4 Hypothesis

The hypotheses of the research are:

1. Directly in situ esterification followed by transesterification by using conventional acid and base catalyst can be led to produce biodiesel in short period of time from jatropha seed by microwave assisted.
2. Microwave irradiation system can be enhanced the reaction rate of biodiesel production and the conversion of product by two-step in-situ method.
3. Carrying out in-situ esterification and instant in-situ transesterification method from Jatropha seeds can be reduce the total process time of biodiesel production and as well as the cost by eliminating the oil extraction phase.
4. The optimum condition of biodiesel production can be determined using response surface methodology.

1.5 Scope of the Study

This study was focused on designing ethanolysis batch reactor in microwave oven at the laboratory scale for in-situ esterification followed by in situ transesterification of jatropha seeds. This procedure was carried out by the H_2SO_4 as acid catalyst for the first step and KOH as base catalyst for the second step to produce cost-effective and sustainable biodiesel with high conversion.

Jatropha seed was used as the raw material for in-situ esterification and transesterification. Microwave irradiation heating systems was used to enhance the purity of products, the reaction rate and product yields. To conduct the process preparation of jatropha seed, in-situ esterification, in-situ transesterification and purification (evaporation, neutralize, alcohol recovery, water washing, etc.) were carried out.

Process parameters including, alcohol/seed ratio, catalyst concentration, reaction time, the size of the seed and speed of stirrer were studied. In addition, design expert software was used to design and optimize the process. Biodiesel production was performed to decrease the reaction time and cost hopefully.

1.6 Significant of the Study

The finding of this research work show the possibility of biodiesel production by two-step in-situ method from jatropha seed, non-edible sources with economical and high free fat acid feedstock with conventional acid and base catalyst in a batch reactor..

Moreover, this study by using RSM method indicated the optimization and modeling of the biodiesel production by utilizing microwave irradiation for in-situ esterification and transesterification method. By this study we made an attempt to discover the one of the best method to reduce the cost biodiesel synthesis with inedible feedstock by elimination of oil extraction step from seed. The two-step in situ reaction of with microwave assisted was expected as a cost-effective way to solve the economic problems of biodiesel production by enhancing the rate reaction and biodiesel conversion.

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