

**BIODIESEL PRODUCTION FROM JATROPHA CURCAS SEED IN BATCH  
REACTOR USING MICROWAVE CATALYZED BY ZEOLITE CATALYST**

**POUYE PANAHI**

**UNIVERSITI TEKNOLOGI MALAYSIA**

BIODIESEL PRODUCTION FROM JATROPHA CURCAS SEED IN BATCH  
REACTOR USING MICROWAVE CATALYZED BY ZEOLITE CATALYST

POUYE PANAHI

A dissertation submitted in partial fulfillment of the  
requirements for the award of the degree of  
Master of Engineering (Chemical)

Faculty of Chemical Engineering  
Universiti Teknologi Malaysia

AUGUST 2013

*To my beloved mother and father*

## **ACKNOWLEDGEMENTS**

I would like to express my great sincere appreciation to my main thesis supervisor, Professor Dr. Nor Aishah Saidina Amin for encouragement, guidance, critics and friendship

My grateful thanks also extend to all academic staff and postgraduate classmates especially students in membrane and fuel cell laboratory for providing assistance at various occasions, their kindness and moral support during my study. Thanks for the friendship and memories.

Last but not least, my deepest gratitude goes to my beloved parents. Without their endless love, continued support, prayers and interest I would not be able to present the same as presented here.

## ABSTRACT

The purpose of present study is direct production of biodiesel from *Jatropha curcas* seed into methyl ester in batch reactor using microwave catalyzed by ZSM-5 catalyst. The triglycerides were converted to biodiesel by in-situ transesterification conducted by microwave in presence of methanol and ZSM-5. The performance of transesterification was investigated with four variables by response surface methodology (RSM) based on central composite design (CCD). RSM was applied to design the experiments and analyzed the effects of variables on biodiesel yield such as irradiation time, agitation speed, methanol to *Jatropha curcas* seed ratio and catalyst loading. The ranges of independence variables contain irradiation time (15-35 min), agitation speed (100-500 rpm), methanol to *Jatropha curcas* seed ratio (6.5-9.5 v/w %) and catalyst loading (5-15 w%) . The highest yield was 88.34% at the optimum conditions of 26.13 min irradiation time, 318.43 rpm agitation speed, 10.53 v/w % methanol / *Jatropha curcas* seed and 10.53 w% catalyst loading. Finally, the quality of the methyl ester product was compared to EN-14214. The ISO-3104 (2003) and EN-14214 were used to determine the density and kinematic viscosity at 15°C and 40°C respectively. In addition, water content and acid value were determined with ASTM 6751.

## ABSTRAK

Kajian ini adalah untuk menghasilkan biodiesel secara langsung daripada biji *Jatropha curcas* kepada ester metil di dalam reaktor kelompok dengan menggunakan gelombang micro yang dimungkinkan oleh ZSM-5. Penukaran triglisirida kepada biodiesel adalah dengan menggunakan kaedah *in-situ transesterification* dimana ia telah dilaksanakan dengan kehadiran methanol dan pemangkin ZSM-5 di dalam gelombang mikro. Prestasi transesterification telah dikaji dengan menggunakan empat pembolehubah iaitu kaedah respon permukaan (RSM) berdasarkan reka bentuk komposit pusat (CCD). RSM telah digunakan untuk mereka bentuk eksperimen dan menganalisis kesan pembolehubah pada hasil biodiesel seperti masa radiasi, kelajuan pergolakan, nisbah metanol kepada biji *Jatropha* dan suapan pemangkin. Julat pembolehubah ketidakbergantungan adalah masa radiasi (15-35 min), kelajuan pergolakan (100-500 rpm), nisbah metanol kepada benih *Jatropha curcas* (6.5-9.5 v/w%) dan suapan pemangkin (5-15 w%) . Hasil tertinggi pada keadaan optimum adalah 88.34% iaitu pada 26.13 min masa radiasi, 318.43 rpm kelajuan pergolakan, 10.53 v/w% nisbah metanol kepada biji *Jatropha curcas* dan 10.53 w% suapan pemangkin. Kualiti produk ester metil telah dibandingkan dengan kaedah EN-14214. ISO-3104 (2003) dan EN-14214 telah digunakan untuk menentukan ketumpatan dan kelikatan kinematik pada 15°C dan 40 °C. Selain itu, kandungan air dan nilai asid juga telah ditentukan dengan kaedah ASTM 6751.

## TABLE OF CONTENTS

CHAPTER NO.	TITLE	PAGE
	<b>DECLARATION</b>	ii
	<b>DEDICATION</b>	iii
	<b>ACKNOWLEDGEMENTS</b>	iv
	<b>ABSTRACT</b>	v
	<b>ABSTRAK</b>	vi
	<b>TABLE OF CONTENTS</b>	vii
	<b>LIST OF TABLES</b>	x
	<b>LIST OF FIGURES</b>	xi
	<b>LIST OF ABBREVIATIONS</b>	xiii
	<b>LIST OF SYMBOLS</b>	xiv
	<b>LIST OF APPENDICES</b>	xv
<b>1</b>	<b>INTRODUCTION</b>	<b>1</b>
	1.1 Background of research	1
	1.2 Problem statement	6
	1.3 Hypothesis of research	7
	1.4 Objective of research	8
	1.5 Scope of research	8
	1.6 Significance of research	9
<b>2</b>	<b>LITERATURE REVIEW</b>	<b>10</b>
	2.1 Introduction	10
	2.1.1 Biodiesel	10
	2.1.2 Biodiesel production	11

	2.1.3	Base catalyzed transesterification	13
	2.1.4	Acid transesterification reaction	15
	2.1.5	Two-step catalyzed transesterification	15
	2.2	Microwave	16
	2.3	Potential feedstock	17
	2.4	Jatropha curcas	21
	2.5	In Situ method	23
	2.6	Summary	25
<b>3</b>		<b>METHODOLOGY</b>	<b>26</b>
	3.1	Research methodology approach	26
	3.2	Equipment	28
	3.3	Material	28
	3.4	Experimental	29
	3.4.1	Jatropha seeds preparation	29
	3.3.2	Experimental set up	31
	3.5	Experimental procedure	32
	3.6	Experiment design and process optimization	33
	3.7	Sample analysis	34
	3.7.1	Kinematic viscosity	35
	3.7.2	Density at 15°C	36
	3.7.3	Acid value	36
	3.7.4	Water content	36
<b>4</b>		<b>RESULTS AND DISCUSSION</b>	<b>37</b>
	4.1	Introduction	37
	4.2	Jatropha curcas seed analysis	37
	4.3	Biodiesel properties	38
	4.4	Design of experiment	39
	4.4.1	Regression model and statistical analysis	41
	4.4.2	Interaction of process variables	45
	4.5	Process optimization	50
<b>5</b>		<b>CONCLUSION AND RECOMMENDATION</b>	<b>51</b>



5.1	Conclusion	51
5.2	Recommendation	52
REFERENCES		53-57
Appendices A-B		58-60

**LIST OF TABLES**

<b>TABLE NO.</b>	<b>TITLE</b>	<b>PAGE</b>
2.1	Oil yields based on crop type	21
2.2	Chemical/physical properties and composition of Jatropha oil	23
3.1	Experimental design of alkyl ester production from Jatropha seed using microwave	33
4.1	Extracted oil from 20 g Jatropha curcas seed by microwave and soxhlet	38
4.2	Major properties of the biodiesel in this study	39
4.3	Experimental design results	40
4.4	ANOVA analysis	42
4.5	Predicted analysis of optimum condition for FAME yield	50

## LIST OF FIGURES

<b>FIGURE NO.</b>	<b>TITLE</b>	<b>PAGE</b>
1.1	U.S biodiesel production, net export and consumption between 2001 and 2011	2
1.2	Worldwide biodiesel production and capacity	3
1.3	Overall transesterification reaction of triglycerides by alcohol, R'OH-alcohol; R -long chain alkyl groups	4
1.4	Stepwise triglycerides transesterification reactions by alcohol	4
1.5	General process flow schematic for biodiesel production via homogeneous base catalyzed transesterification	5
2.1	Basic scheme for produce biodiesel	12
2.2	Types of biodiesel production via transesterification	13
2.3	Prices of potential biodiesel feedstock	20
2.4	Procedure for producing biodiesel from fresh vegetable oil	20
2.5	Jatropha curcas plant and seed	21
2.6	Comparison of (a) the conventional process and (b) the in situ process to produce biodiesel	24
3.1	Research methodology approach	27
3.2	Soxhlet extractor apparatus	30
3.3	Rotary evaporator	30

3.4	experimental set up	31
3.5	Final product before separating	35
4.1	(a) Normal % probability and studentized residual plot. (b) The studentized residuals and predicted response plot. (c) The actual and predicted plot (d)The outliert	45
4.2	RSM Plot of the combine methanol/Jatropha curcas seed ratio and catalyst loading	46
4.3	Counter of agitation catalyst loading and methanol/Jatropha curcas seed oil ratio on FAME	47
4.4	RSM plot of the combine irradiation time and methanol/Jatropha curcas seed Ratio	48
4.5	Response surface plot of the combine catalyst loading and irradiation time.	49

**LIST OF ABBREVIATIONS**

ANOVA	-	Analysis of variance
ASTM	-	American society of testing and materials
AV	-	Acid value
CCDs	-	Central composite designs
DG	-	Diglycerides
DOE	-	Design of expert
EBP	-	End boiling point
E	-	Ester
FAEE	-	Free fatty ethyl ester
FAME	-	Fatty acid methyl ester
FFA	-	Free fatty acid
GC	-	Gas chromatography
GC-MS	-	Gas chromatography-mass spectroscopy
GL	-	Glycerol
J.C	-	Jatropha curcas
KOH	-	Potassium hydroxide
MG	-	Mono glycerides
ml	-	Milli liter
mm	-	Milli meter
N	-	Normality
rpm	-	Revolutions per minute
RSM	-	Response surface methodology
SEM	-	Scanning electron microscope
TG	-	Triglyceride
W	-	Watt

**LIST OF SYMBOLS**

$R^2_{adj}$	-	Adjust R-square
[ROH]	-	Alcohol concentration
$\beta_0$	-	Constant coefficient
X	-	Conversion
DF	-	Degree of freedom
$\varepsilon$	-	Error
MSE	-	Mean square error
$\eta$	-	Response
$R^2$	-	R-square
SS	-	Sum of square
t	-	Time

**LIST OF APPENDICES**

<b>APPENDIX</b>	<b>TITLE</b>	<b>PAGE</b>
A	Standard specification for biodiesel	59
B	Example calculation of methyl ester yield	61

## CHAPTER 1

### INTRODUCTION

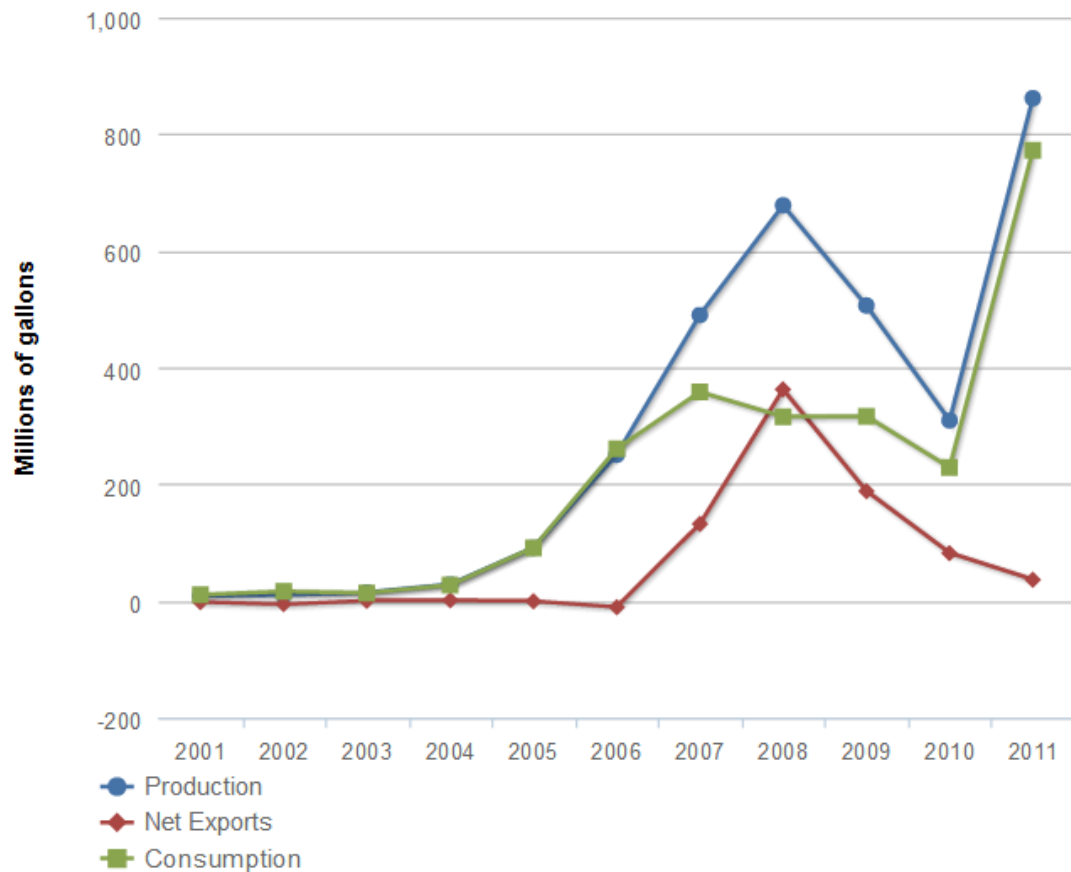
#### 1.1 Background of Research

Economic development comes with energy which has become significant in the world. Consequently, because of increasing of human population and industries, the energy consumption has been strictly rising. The energy sources are petroleum, natural gas and coal from fossil fuel. Moreover limited resource of crude oil, fossil-based fuel price growing and waning of gas reserves has led to focus on energy security in every countries (Agarwal and Agarwal, 2007; Jain and Sharma, 2010). The statistics of international agencies demonstrate that energy consumption will grow 53% by the 2030. Requirements of petroleum derivatives have predicted that will rise from 84.4 to 116.0 million barrels per day till 2030 only in U.S (Leverett and Bader, 2005; "US Energy Department Website, "). Environmental concerns like air pollution and global warming due to combustion of fossil fuels drove people to seek non-toxic and environmentally benign fuels to reduce the pollution emissions.

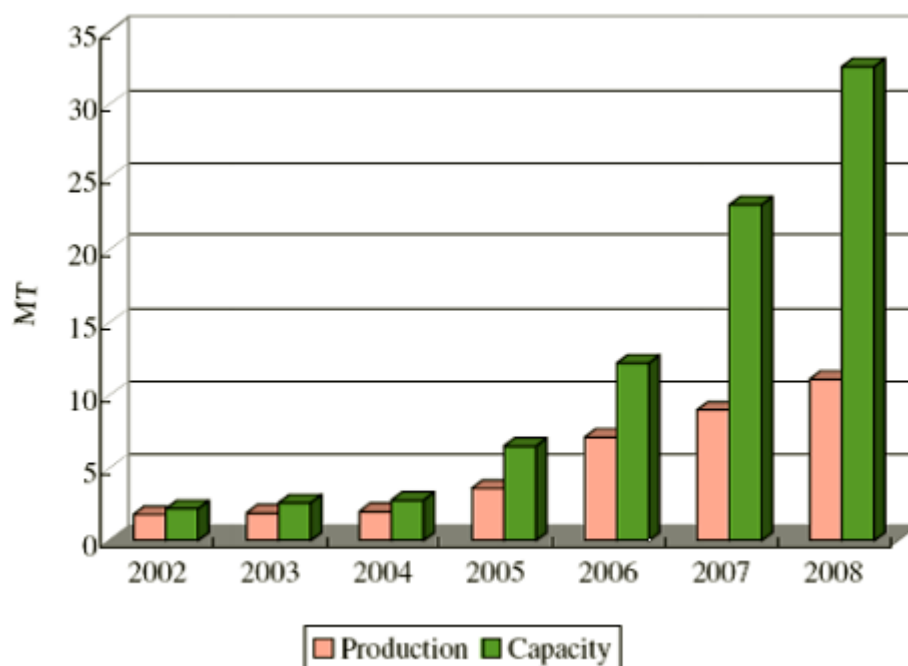
Biofuels have become significant in cater the problems. Currently biodiesel and bio-ethanol are common biofuels which are being used. Alkyl ester gains from renewable resource like animal fat oils (Fangrui and Hanna, 1999; Sharma. *et al.*, 2008). Compared to conventional diesels, low emission profile, biodegradable and non-toxic biodiesel can decrease pollutant gases especially SO<sub>x</sub> emission (Angina, 2000; Pinzli 2009) because feedstocks for biodiesel are free of or low in N<sub>2</sub> and S, and also CO<sub>2</sub> (Fangrui and Hanna, 1999; Sharma., *et al.*, 2008). Owing to these



benefits, methyl ester has experienced a main surge worldwide in the 21st century. Figure 1.1 illustrates producing of methyl ester in the U.S, net export and consumption data over the 9 years from 2001 to 2010 which shows production and consumption of biodiesel have considerable growth. Figure 1.2 illustrates producing biodiesel and installed capacity in the world. Fast growing in renewable fuel market and production has estimated by the world.



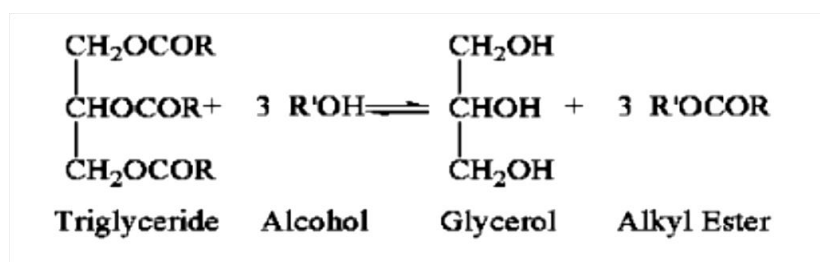
**Figure 1.1** U.S. biodiesel production, net export and consumption between 2001 and 2011 (Sharma *et al.*, 2013)



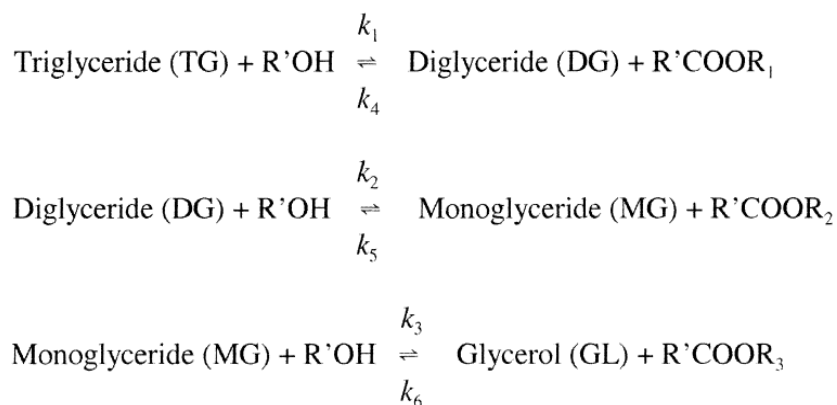
**Figure 1.2** Worldwide biodiesel production and capacity (Vonortas and Papayannakos, 2013)

Biodiesel is made up from free fatty alkyl esters (FFAE). Transesterification, blending, micro emulsification, pyrolysis and cracking are procedures to produce biodiesel. Transesterification reaction is the foremost way to convert edible vegetable oil, non-edible vegetable oil or animal fat with alcohol and a catalyst to fatty acid methyl ester (FAME) (Fangrui and Hanna, 1999; Sharma, *et al.*, 2008). Figure 1.3 demonstrates the totally transesterification stoichiometry with alcohol and catalyst. Figure 1.4 has shown a reversible and stepwise reaction (Freedman, 1986). Therefore, an extra amount of alcohol commonly utilized to break the equilibrium to gain a complete reaction. The main process to produce biodiesel is utilizing homogeneous base catalysts such as alkaline metal alkoxides (Schwab *et al.*, 1987) or hydroxides (Tanabe and Hölderich, 1999). Figure 1.5 demonstrates a typical conventional process flow schematic for biodiesel production by homogeneous transesterification (Gerpen, 2005). The whole process consists of the reaction followed by a downstream processing. The process includes separation section (biodiesel and glycerol, biodiesel and methanol), neutralization of homogeneous base catalysts, washing steps and the catalyst recovery and excess methanol. Although, homogeneous catalysts cause a better reaction rate than heterogeneous catalysts, complicated downstream processing results in low production efficiency.

Indeed, excess alcohol recovery and catalysts causes consume more energy and cost (Corma and Iborra, 2006).



**Figure 1.3** Overall transesterification reaction of triglycerides by alcohol, R'OH-alcohol; R -long chain alkyl groups (Otera, 1993)

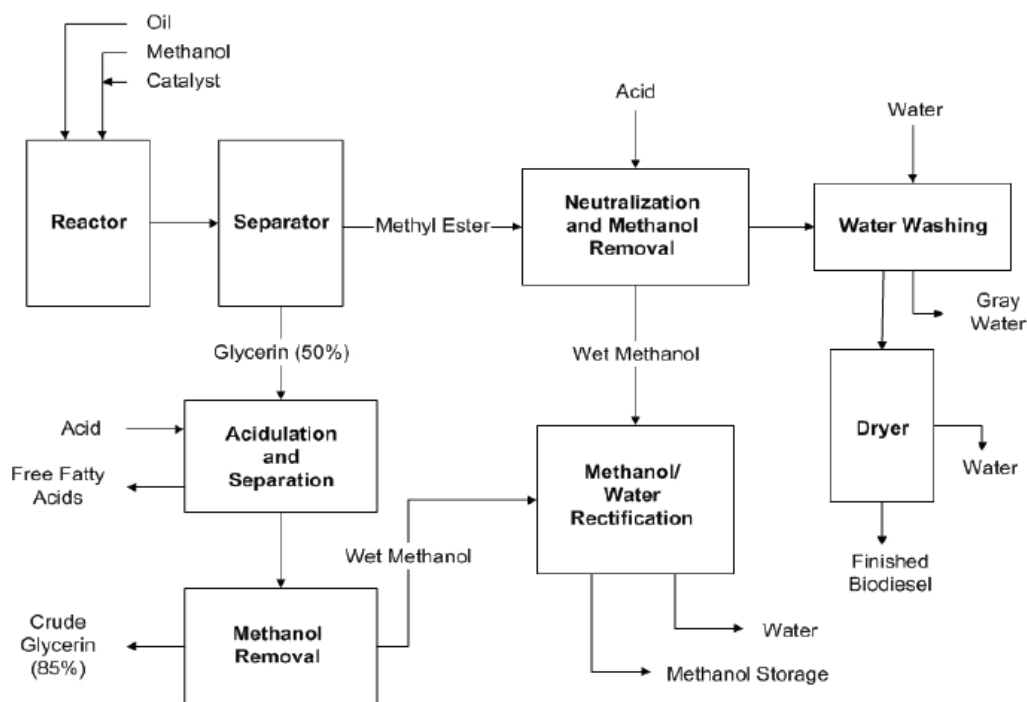


**Figure 1.4** Stepwise triglycerides transesterification reactions by alcohol (Freedman, 1986)

In addition, the washing step to remove homogeneous catalysts discharges a enormous amount of wastewater (Fukuda *et al.*, 2001) which is not environmentally benign. Using homogeneous catalysts instead of homogeneous catalysts is the main option to overcome drawback of them.

Economically and environmentally heterogeneous catalysts have some advantages over catalysts. Easier separations from the liquid and long life times are advantages of heterogeneous catalysts which can cause cost reduction of biodiesel production (Gryglewicz, 1999). They are also more environmentally benign for the reason that they present fewer disposal problems (Helwani *et al.*, 2009) due to these advantages over homogeneous catalysts; heterogeneous catalysts for biodiesel

production have obtained increased attention lately. Both solid acid and solid base catalysts have been used as catalysts in transesterification reactions.



**Figure 1.5** General process flow schematic for biodiesel production via homogeneous base catalyzed transesterification (Gerpen, 2005)

The cost of biodiesel commercialization is roughly 1.5 times more than producing petroleum diesel fuel which is the significant issue because of vegetable oil cost (Chen *et al.*, 2012). 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.

Some factors such as alcohol/oil ratio; reaction temperature, reaction time and quantity of catalyst are significant to produce biodiesel. Catalytic reactions can use alkali catalysts, acid catalysts, or enzymatic transesterification, of which the first achieves the best results. The conventional sample heating has some important problems, like heterogenic heating of the surface, limitations dependent on the thermal conductivity of materials, specific heat, and density compared to microwave irradiation (Metaxas, 1996), and thus many research groups have recently focused on the latter approach (Chen, *et al.*, 2012; Motasemi and Ani, 2012). Previous studies

announced that microwave-assisted chemical reactions are better than those gained using other synthetic techniques and that microwave heating systems can increase the reaction rate, product yields, and products purity (Geuens *et al.*, 2007; Yuan *et al.*, 2008; Zhang *et al.*, 2010).

## 1.2 Problem Statement

Due to the petroleum reserves waning, rising requirement of energy, increasing environmental concerns because of rising toxic gas emission, and petroleum prices skyrocketing there is more need to renewable, biodegradable and alternative energy sources such as 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 enzyme catalyst is costly and also become inactive after reaction. With homogenous base catalysts, elimination catalyst from the biodiesel requires a more purification step, which leads 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, acid heterogeneous catalysts are can be rapidly separated from the product by filtration, which reduces the washing requirement. In addition, acid heterogeneous catalysts can stimulatingly catalyze the transesterification and esterification reaction that can avoid the pre-esterification step, thus these catalysts are particularly useful for those feedstocks with high free fatty acid content

(Mangesh, 2006). Therefore acid heterogeneous catalyst can be considered as one of the best preferences for transesterification reaction of biodiesel production.

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 (Wali *et al.*, 2013). 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 products purity (Barnard *et al.*, 2007; Chen, *et al.*, 2012; K.-S. Chen *et al.*, 2012; Motasemi and Ani, 2012).

### **1.3 Hypothesis of Research**

The hypothesis of the research is that ZSM-5 catalyst will have high activity, as well as separating will be easy in order to carry very well out in biodiesel production from *Jatropha curcas* which have a high free fatty acid (FFA). Directly in situ esterification followed by transesterification using heterogeneous acid catalyst can be led to produce biodiesel in short period of time from *jatropha* seed by microwave assisted.

Producing biodiesel in microwave will increase biodiesel yield as well as simplify converting *Jatropha* oil to better quality production.

## 1.4 Objective of Research

The objectives of research are:

- i. To test feasibility of using ZSM-5 as a heterogeneous catalyst to produce fatty acid methyl ester from *Jatropha curcas* seed by microwave
- ii. To investigate effect of different variables such as irradiation time, methanol/oil ratio, catalyst loading and agitation speed on yield of methyl ester.
- iii. To optimize the effective variables on biodiesel production by using response surface methodology.
- iv. Application of in-situ process for biodiesel production

## 1.5 Scope of Research

Scope of this study was focused on designing batch reactor in microwave oven at the laboratory scale for in-situ transesterification of *Jatropha curcas* seeds. This procedure was carried out by ZSM-5 as heterogeneous acid catalyst to produce cost effective and sustainable biodiesel with high yield.

*Jatropha curcas* seed was used as the raw material for 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 curcas* seed, in-situ transesterification and purification (evaporation, filtration, etc.) were carried out.

Process parameters including, methanol /*Jatropha curcas* seed ratio, catalyst concentration, reaction time, and agitation speed 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 Significance of Research**

This research comes up for the first time to produce biodiesel from *Jatropha curcas* seed in a microwave system with ZSM-5 catalyst, which helping for ease of converting seed to high quality biodiesel in a single reactor, thus reducing number of process units found in other processes. Furthermore, finding of this research work shows the possibility of biodiesel production by one step in-situ method from *Jatropha curcas* seed, non-edible sources with economical and high free fat acid feedstock with heterogeneous acid 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 transesterification method. By this study we made an attempt to discover one of the best methods to reduce the cost biodiesel synthesis with inedible feedstock by elimination of oil extraction step from seed.



## REFERENCES

- Agarwal, D. and Agarwal, A. K. (2007). Performance and emissions characteristics of Jatropha oil (preheated and blends) in a direct injection compression ignition engine. *Applied Thermal Engineering*, 27(13), 2314-2323.
- Anand, K., Sharma, R. P. and Mehta, P. S. (2011). A comprehensive approach for estimating thermo-physical properties of biodiesel fuels. *Applied Thermal Engineering*, 31(2-3), 235-242.
- Angina, S. a. R. P. (2000). Triglycerides-based diesel fuels. *Renewable and Sustainable Energy Reviews*, 4, 111-113
- Balat, M. and Balat, H. (2010). Progress in biodiesel processing. *Applied Energy*, 87(6), 1815-1835.
- Banapurmath, N. R., Tewari, P. G. and Hosmath, R. S. (2008). Performance and emission characteristics of a DI compression ignition engine operated on Honge, Jatropha and sesame oil methyl esters. *Renewable Energy*, 33(9), 1982-1988.
- Barnard, T. M., Leadbeater, N. E., Boucher, M. B., Stencel, L. M. and Wilhite, B. A. (2007). Continuous-Flow Preparation of Biodiesel Using Microwave Heating. *Energy & Fuels*, 21(3), 1777-1781.
- Canakci, M. (2007). The potential of restaurant waste lipids as biodiesel feedstocks. *Bioresource Technology*, 98(1), 183-190.
- Chen, Lin, Hsu, K.-H. and Wang, H.-K. (2012). Improving biodiesel yields from waste cooking oil by using sodium methoxide and a microwave heating system. *Energy*, 38(1), 151-156.
- Chen, Xiao, B., Chang, J., Fu, Y., Lv, P. and Wang, X. (2009). Synthesis of biodiesel from waste cooking oil using immobilized lipase in fixed bed reactor. *Energy Conversion and Management*, 50(3), 668-673.
- Chen, K.-S., Lin, Y.-C., Hsu, K.-H. and Wang, H.-K. (2012). Improving biodiesel yields from waste cooking oil by using sodium methoxide and a microwave heating system. *Energy*, 38(1), 151-156.
- Chen, Y., Xiao, B., Chang, J., Fu, Y., Lv, P. and Wang, X. (2009). Synthesis of biodiesel from waste cooking oil using immobilized lipase in fixed bed reactor. *Energy Conversion and Management*, 50(3), 668-673.
- Chi, Z., Pyle, D., Wen, Z., Frear, C. and Chen, S. (2007). A laboratory study of producing docosahexaenoic acid from biodiesel-waste glycerol by microalgal fermentation. *Process Biochemistry*, 42(11), 1537-1545.

- Chisti, Y. (2007). Biodiesel from microalgae. *Biotechnology Advances*, 25(3), 294-306.
- Contran, N., Chessa, L., Lubino, M., Bellavite, D., Roggero, P. P. and Enne, G. (2013). State-of-the-art of the *Jatropha curcas* productive chain: From sowing to biodiesel and by-products. *Industrial Crops and Products*, 42(0), 202-215.
- Corma, A. and Iborra, S. (2006). Optimization of Alkaline Earth Metal Oxide and Hydroxide Catalysts for Base-Catalyzed Reactions. In C. G. Bruce & K. Helmut (Eds.), *Advances in Catalysis* (Vol. Volume 49, pp. 239-302): Academic Press.
- Demirbas, A. (2008). Comparison of transesterification methods for production of biodiesel from vegetable oils and fats. *Energy Conversion and Management*, 49(1), 125-130.
- Di Serio, M., Tesser, R., Dimiccoli, M., Cammarota, F., Nastasi, M. and Santacesaria, E. (2005). Synthesis of biodiesel via homogeneous Lewis acid catalyst. *Journal of Molecular Catalysis A: Chemical*, 239(1-2), 111-115.
- Encinar, J. M., González, J. F., Martínez, G., Sánchez, N. and Pardal, A. (2012). Soybean oil transesterification by the use of a microwave flow system. *Fuel*, 95(0), 386-393.
- Fangrui and Hanna, M. A. (1999). Biodiesel production: a review. *Bioresource Technology*, 70(1), 1-15.
- Felizardo, P., Neiva Correia, M. J., Raposo, I., Mendes, J. F., Berkemeier, R. and Bordado, J. M. (2006). Production of biodiesel from waste frying oils. *Waste Management*, 26(5), 487-494.
- Freedman, B. R. O., Pryde, E.H. (1986). Transesterification kinetics of soybean oil. *Journal of American Oil Chemists Society*, 63, 1375-1380.
- Fukuda, H., Kondo, A. and Noda, H. (2001). Biodiesel fuel production by transesterification of oils. *Journal of Bioscience and Bioengineering*, 92(5), 405-416.
- Furuta, S., Matsushashi, H. and Arata, K. (2004). Biodiesel fuel production with solid superacid catalysis in fixed bed reactor under atmospheric pressure. *Catalysis Communications*, 5(12), 721-723.
- Gerpen, J. V. (2005). Biodiesel processing and production. *Fuel Processing Technology*, 86(10), 1097-1107.
- Geuens, J., Kremsner, J. M., Nebel, B. A., Schober, S., Dommissie, R. A., Mittelbach, M., et al. (2007). Microwave-Assisted Catalyst-Free Transesterification of Triglycerides with 1-Butanol under Supercritical Conditions. *Energy & Fuels*, 22(1), 643-645.
- Gryglewicz, S. (1999). Rapeseed oil methyl esters preparation using heterogeneous catalysts. *Bioresource Technology*, 70(3), 249-253.
- Gui, M. M., Lee, K. T. and Bhatia, S. (2008). Feasibility of edible oil vs. non-edible oil vs. waste edible oil as biodiesel feedstock. *Energy*, 33(11), 1646-1653.
- Haaland, P. D. (1989). Experimental Design in Biotechnology. *Marcel Dekker Inc.*

- Haas, M., Scott, K., Foglia, T. and Marmer, W. (2007). The General Applicability of in Situ Transesterification for the Production of Fatty Acid Esters from a Variety of Feedstocks. *Journal of the American Oil Chemists' Society*, 84(10), 963-970.
- Haas, M., Scott, K., Marmer, W. and Foglia, T. (2004). In situ alkaline transesterification: An effective method for the production of fatty acid esters from vegetable oils. *Journal of the American Oil Chemists' Society*, 81(1), 83-89.
- Halim, S. F. A., Kamaruddin, A. H. and Fernando, W. J. N. (2009). Continuous biosynthesis of biodiesel from waste cooking palm oil in a packed bed reactor: Optimization using response surface methodology (RSM) and mass transfer studies. *Bioresource Technology*, 100(2), 710-716.
- Harrington, K. and Catherine. (1985). A comparison of conventional and in situ methods of transesterification of seed oil from a series of sunflower cultivars. *Journal of the American Oil Chemists Society*, 62(6), 1009-1013.
- Helwani, Z., Othman, M. R., Aziz, N., Fernando, W. J. N. and Kim, J. (2009). Technologies for production of biodiesel focusing on green catalytic techniques: A review. *Fuel Processing Technology*, 90(12), 1502-1514.
- Jain, S. and Sharma, M. P. (2010). Prospects of biodiesel from *Jatropha* in India: A review. *Renewable and Sustainable Energy Reviews*, 14(2), 763-771.
- Kildiran, G., Yücel, S. and Türkay, S. (1996). In-situ alcoholysis of soybean oil. *Journal of the American Oil Chemists' Society*, 73(2), 225-228.
- Knothe, G. (2005). Cetane Numbers-Heat of Combustion-Why Vegetable Oils and Their Derivatives Are Suitable as a Diesel Fuel. *The Biodiesel Handbook*, 76-80.
- Leadbeater, N. E. and Stencel, L. M. (2006). Fast, Easy Preparation of Biodiesel Using Microwave Heating. *Energy & Fuels*, 20(5), 2281-2283.
- Leung and Guo. (2006). Transesterification of neat and used frying oil: Optimization for biodiesel production. *Fuel Processing Technology*, 87(10), 883-890.
- Leung, Xuan and Leung, M. K. H. (2010). A review on biodiesel production using catalyzed transesterification. *Applied Energy*, 87(4), 1083-1095.
- Leung, D., Wu, Xuan. (2010). A review on biodiesel production using catalyzed transesterification. *Applied Energy*, 87(4), 1083-1095.
- Leverett, F. and Bader, J. (2005). Managing China-U.S. energy competition in the Middle East. *The Washington Quarterly*, 29(1), 187-201.
- Lewis, T., Nichols, P. D. and McMeekin, T. A. (2000). Evaluation of extraction methods for recovery of fatty acids from lipid-producing microheterotrophs. *Journal of Microbiological Methods*, 43(2), 107-116.
- Mangesh, R. G., Lekha Charan Meher and Ajay Kumar Dalai. (2006). Solid acid catalyzed biodiesel production by simultaneous esterification and transesterification. *Green Chem*, 8, 1056-1062.
- Marchetti, J. M., Miguel, V. U. and Errazu, A. F. (2007). Possible methods for biodiesel production. *Renewable and Sustainable Energy Reviews*, 11(6), 1300-1311.

- Meng, X., Chen, G. and Wang, Y. (2008). Biodiesel production from waste cooking oil via alkali catalyst and its engine test. *Fuel Processing Technology*, 89(9), 851-857.
- Metaxas, A. C. (1996). Foundations of electroheat. A unified approach. *Fuel and Energy Abstracts*, 37(3), 193-193.
- Morita, E., Kumon, Y., Nakahara, T., Kagiwada, S. and Noguchi, T. (2006). Docosahexaenoic Acid Production and Lipid-Body Formation in *Schizochytrium limacinum* SR21. *Marine Biotechnology*, 8(3), 319-327.
- Motasemi, F. and Ani, F. N. (2012). A review on microwave-assisted production of biodiesel. *Renewable and Sustainable Energy Reviews*, 16(7), 4719-4733.
- Otera, J. (1993). Transesterification. *Chemical Reviews*, 93(4), 1449-1470.
- Özgül-Yücel, S. and Türkay, S. (2003). FA monoalkylesters from rice bran oil by in situ esterification. *Journal of the American Oil Chemists' Society*, 80(1), 81-84.
- Patil, P. D. and Deng, S. (2009). Optimization of biodiesel production from edible and non-edible vegetable oils. *Fuel*, 88(7), 1302-1306.
- Pinzli, I. G., Lopez-Gimenez, M. D., Luque de Castro, G., Dorado, M.P., Dorado (2009). The ideal vegetable oil-based biodiesel composition: A review *Energy and Fuel*, 23, 2325-2341.
- Predojević, Z. J. (2008). The production of biodiesel from waste frying oils: A comparison of different purification steps. *Fuel*, 87(17-18), 3522-3528.
- Qian, J., Wang, F., Liu, S. and Yun, Z. (2008). In situ alkaline transesterification of cottonseed oil for production of biodiesel and nontoxic cottonseed meal. *Bioresource Technology*, 99(18), 9009-9012.
- Schwab, A. W., Bagby, M. O. and Freedman, B. (1987). Preparation and properties of diesel fuels from vegetable oils. *Fuel*, 66(10), 1372-1378.
- Sharma, B., Ingalls, R. G., Jones, C. L. and Khanchi, A. (2013). Biomass supply chain design and analysis: Basis, overview, modeling, challenges, and future. *Renewable and Sustainable Energy Reviews*, 24(0), 608-627.
- Sharma, Singh, B. and Upadhyay, S. N. (2008). Advancements in development and characterization of biodiesel: A review. *Fuel*, 87(12), 2355-2373.
- Siler-Marinkovic, S. and Tomasevic, A. (1998). Transesterification of sunflower oil in situ. *Fuel*, 77(12), 1389-1391.
- Singh, R. N., Vyas, D. K., Srivastava, N. S. L. and Narra, M. (2008). SPRERI experience on holistic approach to utilize all parts of *Jatropha curcas* fruit for energy. *Renewable Energy*, 33(8), 1868-1873.
- Srilatha, K., Lingaiah, N., Devi, B. L. A. P., Prasad, R. B. N., Venkateswar, S. and Prasad, P. S. S. (2009). Esterification of free fatty acids for biodiesel production over heteropoly tungstate supported on niobia catalysts. *Applied Catalysis A: General*, 365(1), 28-33.
- Tanabe, K. and Hölderich, W. F. (1999). Industrial application of solid acid-base catalysts. *Applied Catalysis A: General*, 181(2), 399-434.

- Tsai, W.-T., Lin, C.-C. and Yeh, C.-W. (2007). An analysis of biodiesel fuel from waste edible oil in Taiwan. *Renewable and Sustainable Energy Reviews*, 11(5), 838-857.
- . US Energy Department Website. Retrieved 12 August 2010 from <http://www.energy.gov>
- Vicente, G., Coteron, A., Martinez, M. and Aracil, J. (1998). Application of the factorial design of experiments and response surface methodology to optimize biodiesel production. *Industrial Crops and Products*, 8(1), 29-35.
- Wali, W. A., Hassan, K. H., Cullen, J. D., Shaw, A. and Al-Shamma'a, A. I. (2013). Real time monitoring and intelligent control for novel advanced microwave biodiesel reactor. *Measurement*, 46(1), 823-839.
- Wan Omar, W. N. N. and Saidina Amin, N. A. (2011). Optimization of heterogeneous biodiesel production from waste cooking palm oil via response surface methodology. *Biomass and Bioenergy*, 35(3), 1329-1338.
- Wang, S. O., P.Liu,Z.Zhang. (2007). Preparation of biodiesel from waste cooking oil via two- step catalyzed process. *Energy Conversion and Management* 48, 184-188
- Yan, S., Salley, S. O. and Simon Ng, K. Y. (2009). Simultaneous transesterification and esterification of unrefined or waste oils over ZnO-La<sub>2</sub>O<sub>3</sub> catalysts. *Applied Catalysis A: General*, 353(2), 203-212.
- Yuan, H., Yang, B. L. and Zhu, G. L. (2008). Synthesis of Biodiesel Using Microwave Absorption Catalysts. *Energy & Fuels*, 23(1), 548-552.
- Zhang, Dubé, M. A., McLean, D. D. and Kates, M. (2003). Biodiesel production from waste cooking oil: 1. Process design and technological assessment. *Bioresource Technology*, 89(1), 1-16.
- Zhang, Zu, Y.-G., Fu, Y.-J., Luo, M., Zhang, D.-Y. and Efferth, T. (2010). Rapid microwave-assisted transesterification of yellow horn oil to biodiesel using a heteropolyacid solid catalyst. *Bioresource Technology*, 101(3), 931-936.
- Zullaikah, S., Lai, C.-C., Vali, S. R. and Ju, Y.-H. (2005). A two-step acid-catalyzed process for the production of biodiesel from rice bran oil. *Bioresource Technology*, 96(17), 1889-1896.