Biodiesel Synthesis From Jatropha Through Transesterification By Using Ba/ZnO Catalyst.

by

Mohd Najib bin Ramlan

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
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CERTIFICATION OF APPROVAL

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MOHD NAJIB BIN RAMLAN

TABLE OF CONTENTS

CHAPTER 1

INTRODUCTION

CHAPTER 2

LITERATURE REVIEW

CHAPTER 3

PROJECT WORK

CHAPTER 4

METHODOLOGY

CHAPTER 5

RESULT AND DISCUSSION

CHAPTER 6

LIST OF FIGURES

ABSTRACT

The objective of this project is to produce a Biodiesel from triglyceride of Jatropha oil by using conventional transesterification with the Ba/ZnO heterogeneous catalyst. The heterogeneous catalyst application can be applied in the industrial practice where it can be reuse and it is easy to separate from the products. The main objective in using heterogeneous catalyst in transesterification process is to reduce the production cost in separating biodiesel with the catalyst and also to reduce the water pollution. It is also as an alternative catalyst for the homogeneous catalyst biodiesel production system.

ACKNOWLEDGEMENT

A thousand appreciations to all that involving either directly or indirectly in this project to make it smoothly successful. A special thanks and congratulation to the Final Year Project coordinator for the successful planning and scheduling in order to smooth the progress flow.

Thank you to my Final Year Project supervisor, Assoc. Prof. Dr. Suzana Yusup for preparing such an interesting title and for giving me a chance to work with the great team of biodiesel researcher. Special thanks to all post-graduate student that always helping me in giving advise and guide to make sure the project were in the right track and smoothly successful.

Not to forget, the line of technician that always be with us to make sure our safety, and to give us guide and knowledge in handling equipment, chemical and other laboratory system.

The cooperation from all had made the project successful and completed within the expecting period. A thousand thanks again to all.

CHAPTER 1

1. INTRODUCTION

1.1. Background study

In early 1900, diesel engine had been invented by Rudolf Diesel and on 1911; he had proved that vegetable oil can be use for generating diesel engine.

In the era of decreasing petroleum fuel source and the price is keep increasing, the existing petroleum fuel will become unaffordable for the human use in the next few years. And this also will affect the world economies.

In order to prepare for the next fuel disaster, alternative fuels need to be find thus can give a consumers a choice for fuel sources. This will also decrease the demand for the petroleum fuel and in the same can control the price.

Biodiesel is produced by Transesterification of triglycerides present in animal fat or vegetable oils, by displacing glycerine with a molar mass alcohol. This resulting ester mixture has physico-chemical properties similar to those of petroleum diesel (2). Biodiesel is considered clean fuel since it has almost no sulphur, no aromatics and has about 10% built-in oxygen, which helps it to burn fully.

The Jatropha seeds are a suitable source for making the biodiesel in Malaysia, the ability of the plant in resistant to drought and pests had made it suitable for Malaysian. Jatropha is a native of Mexico & tropical South America but naturalized now through out tropical to sub tropical Asia & Africa. It is commonly known as physics nut. It can be very well grown in a wide range of agro-climatic conditions including moderate to high temperature areas. It can be planted in any kind of soil. It grows almost anywhere, in sandy and saline soils. Its water requirement is extremely low & it can withstand long period of drought by shedding most of its leaves to reduce transpiration loss (1).

1.2. Problem statement

In order to produce biodiesel, the process must be economic and efficient to generate more products and also reducing the production cost. Normal transesterification technique to produce biodiesel had been discovered since early 1900 and it is a well-known technique. The problems that come with this technique had effect the biodiesel price where the production cost is too high.

Normal conventional transesterification processes were using homogeneous catalyst, catalyst in form of liquid such as Sodium hydroxide or Potassium hydroxide dissolved in methanol as catalyst. In this conventional method, large amount of waste water was produced separate and clean the catalyst and the products. As the biodiesel production increase, the waste water produced also increased and this could harm the environment.

In early 1900, biodiesel is been produce by peanut oil as the triglycerides source. Nowadays, the biodiesel sources are come from a lot of agriculture product such as corn, soy beans, and palm oil. By using this kind of sources for producing biodiesel, will introduced to the food crisis problem in the future.

In order to avoid this matter, non-edible sources need to be use for the biodiesel production. Jatropha seed is the best choice for biodiesel source as the seed contain almost 40% oil and the seed can be grows in any weather. It is a non-edible source as it seed and leaves are poisonous and can't be consume by any living creatures.

1.3. Objective

The objective of this project is to produce biodiesel by synthesizing of Jatropha oil by using conventional transesterification with Ba/ZnO as a catalyst.

CHAPTER 2

2. LITERATURE REVIEW

2.1.The Jatropha seed

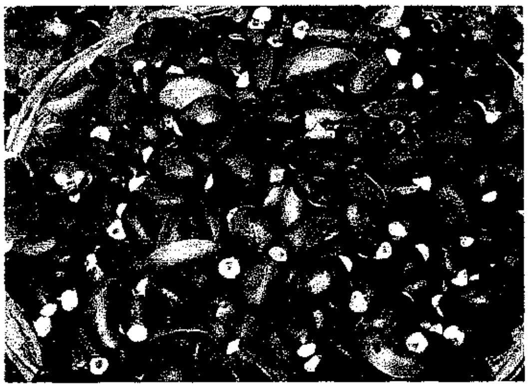


Figure 1: Jatropha seed

Jatropha is a native plant to Central America, and has become naturalized in many tropical areas.

Jatropha plant is a special plant because it is resistant to drought and pests. The seed and the leaves are poisonous and it works as the barrier to prevent from animals attack.

It is grows well under subtropical and tropical climates and can tolerate extremes of temperature but not frost.

Jatropha plantations require little attention and can produce plants and seeds for more than 50 years. This can reduce the maintenance cost and also starting cost in planting the Jatropha.

The plant also can grow in wide range of soils. The land that uses is always wasteland or land that is not used for anything else.

2.2. Heterogeneous catalyst

In usual technique of transesterification or in-situ transesterification, homogeneous catalyst such as Potassium Hydroxide or Sodium Hydroxide is use as a catalyst for reaction. The conversion of biodiesel is very high as a mass transfer between reactant and catalyst is high.

The problems that exist in using homogeneous catalyst is when come to the part of separating it from product. In laboratory scale, rotary evaporator with the temperature of catalyst boiling point. The process will take time to achieve high purity of product in separating it.

From the, University of Kentucky, project fact (14). By using KOH or NaOH as a catalyst can promote a formation of soaps, and other salts. In order to overcome this matter, a solid base catalyst such as mixed metal oxides is use for biodiesel production.

A.K Dalai (11), in his study had noted that by using heterogeneous catalyst, it will make the reaction in three phase system that is oil-methanol-catalyst. The use of heterogeneous catalyst facilitates the separation of post reaction mixture, saving on cost of separation of catalyst.

In the study of Wenlei Xei (15), had highlighted that in separating the catalyst from methanol, a large amount of waste water was produced to separate and clean the catalyst and the products. The development of heterogeneous catalyst could reduce the environmental pollution in producing biodiesel.

2.3. Ultrasonication technique

Ultrasonication is an alternative to high speed agitator technique. This technique offer great potential in the processing of liquids by improving the mixing and chemical reaction in various application and industries.

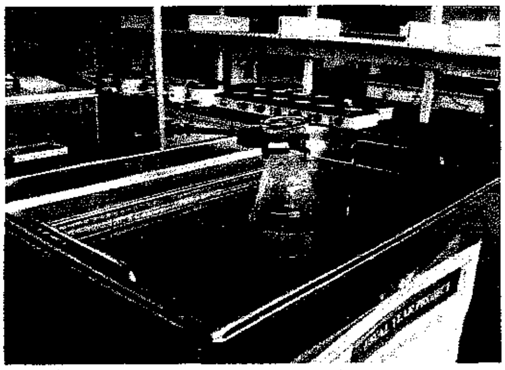


Figure 2: Experiment run in the Ultrasonic bath

From the Hielscher company (16), explained that ultrasonication increases the chemical reaction speed of the transesterification of vegetables oils and animals fats into biodiesel.

Biodiesel commonly produced in batch reactors using heat and mechanical mixing as energy input.

By introduced ultrasonic cavitational mixing, an effective alternative can direct to a better mixing in commercial processing.

In K.G Georgogoianni studies (12), he had compared the result in producing biodiesel between mechanical stirrer and ultrasonication. The result for the experiment is described below:

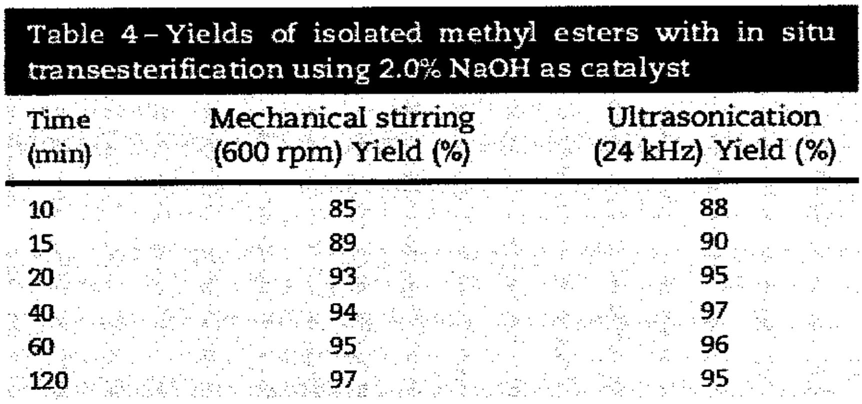


Table 4 – Yields of isolated methyl esters with in situ transesterification using 2.0% NaOH as catalyst

Time (min)	Mechanical stirring (600 rpm) Yield (%)	Ultrasonication (24 kHz) Yield (%)
10	85	88
15	89	90
20	93	95
40	94	97
60	95	96
120	97	95

Figure 3: Yield of isolated methyl esters with in-situ transesterification using 2.0% NaOH as a catalyst.

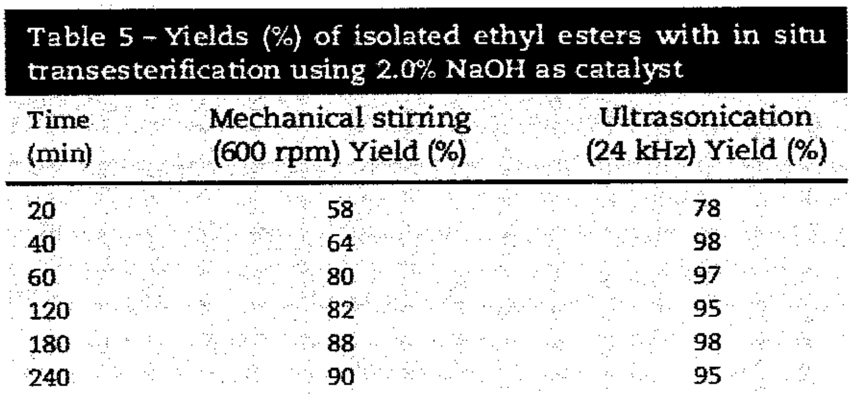


Table 5 – Yields (%) of isolated ethyl esters with in situ transesterification using 2.0% NaOH as catalyst

Time (min)	Mechanical stirring (600 rpm) Yield (%)	Ultrasonication (24 kHz) Yield (%)
20	58	78
40	64	98
60	80	97
120	82	95
180	88	98
240	90	95

Figure 4: Yield of isolated ethyl esters with in-situ transesterification using 2.0% NaOH as a catalyst.

He had concluded that in case of production of ethyl esters, ultrasonication was substantially efficient in triglycerides conversion.

CHAPTER 3

3. PROJECT WORK

3.1. The Heterogeneous catalyst.

In a study of a heterogeneous catalyst. A few pros and cons had been analyzed by referring to a literature reviews conclusion and discussion. The finding is:

Pros:

1. Heterogeneous catalyst can reduce the cost in separation part of catalyst with product.
2. Reusable as it can be recover and regenerate after the reactions.
3. Can prevent the formations the formation of soaps and other slats if compared to KOH or NaOH catalyst.
4. Can overcome environmental pollution.

Cons:

1. Reaction is slower compared to homogeneous catalyst
2. Introduce three phase system that cause a diffusional resistance.
3. Take longer time to achieve high conversion

By analyzing the pros and cons, it is possible to develop a heterogeneous catalyst as it can promise a low production cost.

A lot of modified heterogeneous catalyst had been tested by many researchers in finding the best catalyst for the optimum product result. Most of the catalyst is a mixed metal oxides such as Ba/ZnO, Li/CaO, KI/Al_2O_3, KF/ZnO, modified zeolites and many mores.

From the studies of Satoshi Furuta (17), had tested a tungstated zirconium alumina (WZA), TiO_2/ZrO_2, Al_2O_3/ZrO_2, and K_2O/ZrO_2 catalyst react with soybean oil and methanol at 250°C.

The results that come out from are below:

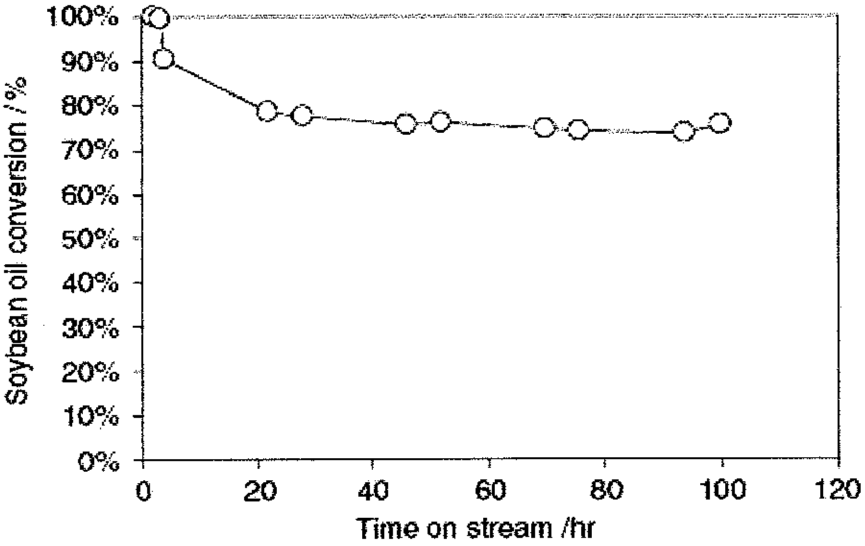


Figure 5: The Graph of Biodiesel conversion versus time taken

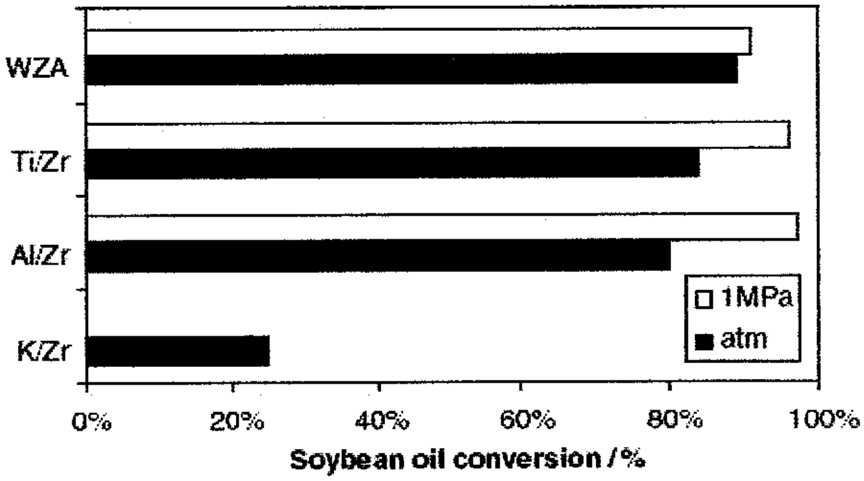


Figure 6: The biodiesel conversion for each catalyst in 1MPa and atm pressure.

In Figure 4, K_2O/ZrO_2 gave almost 100% conversions at the very beginning of reaction, but deactivated rapidly due to the leaching out of Potassium.

In Figure 5, he had summarized the conversion achieved after 20 hours with every catalyst in 1MPa and atmosphere pressure. Although the conversion is more than 80%, but the time taken is too long.

Wenlei Xei (15), had examined the Ba-ZnO catalyst amount range between 1-9% referred to the starting oil weight. The obtained result reported in Figure 6 below:

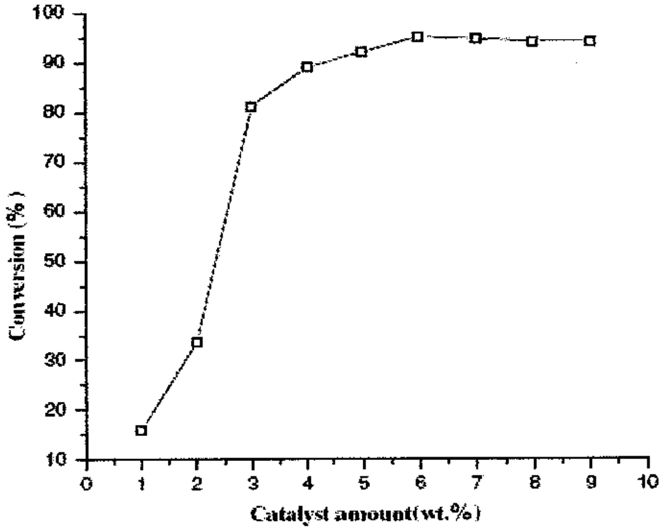


Figure 7: The Graph of Biodiesel conversion versus catalyst amount

From the figure above, at the amount of 6 wt% of catalyst, the conversion achieve at 95%, the highest conversion occur in the entire catalyst amount.

By analyzing the 6 wt% catalyst, the graph is explained below:

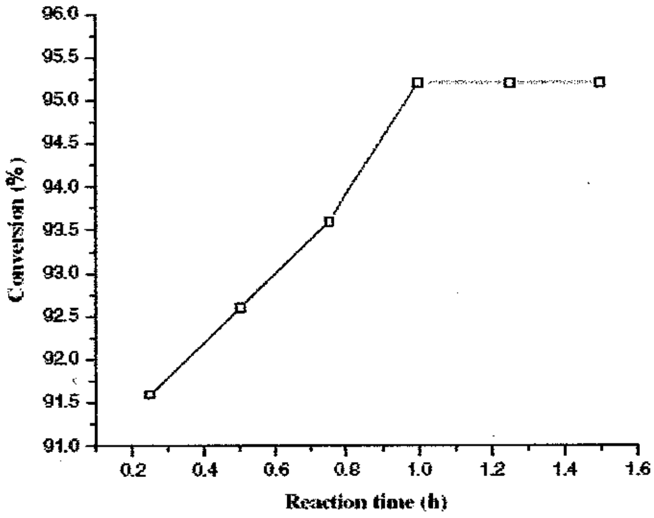


Figure 8: The Graph of biodiesel conversion versus reaction time

In a period of 1 hour, the reaction had achieved 95.5% conversion and it remains constant after 1 hour. The Ba-ZnO catalyst is an acceptable catalyst in achieving high conversion and lesser time.

In another work of Wenlei Xei (18), he had tested an economic catalyst and simple to prepare by modifying NaX zeolites impregnated with as aqueous solution of KOH.

The result in the effect of loading amount of KOH in biodiesel in 8 hours conversion is described below.

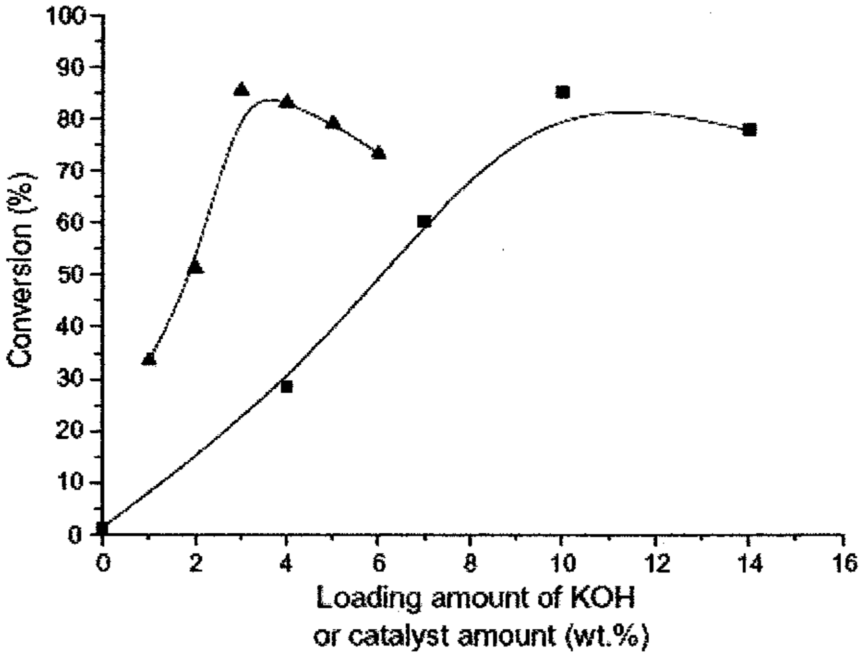


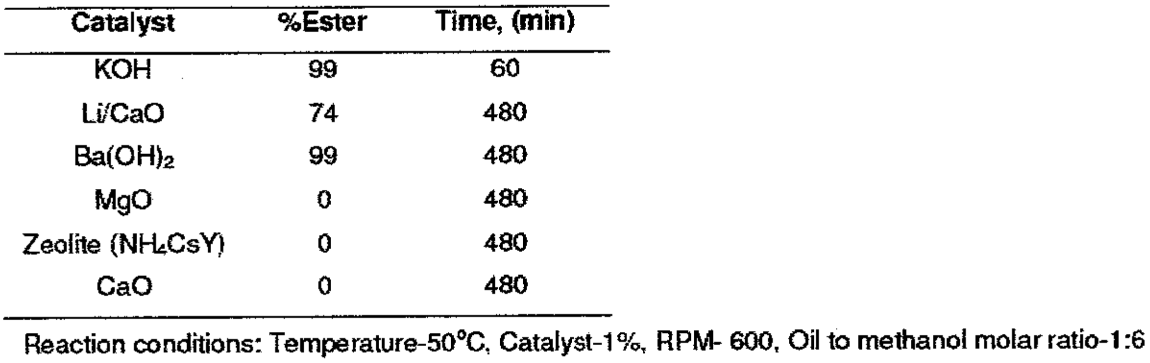
Figure 9: The Graph of Biodiesel conversion versus loading amount of KOH and catalyst amount.

He had realized that increasing amount of catalyst or loaded KOH not resulting in increasing of conversion. In loading the KOH, the breakthrough amount is at 10 wt% and the maximum of conversion is at 85%.

In catalyst amount, the breakthrough amount is at 3 wt% and the maximum conversion is at 85%. The conversion for this catalyst is still low and the reaction time takes too long for conversion of only 85% maximum.

Another study of heterogeneous catalyst that been done by A.K Dalai (11). He had tested a few catalyst to be compared and that is Li/CaO, $Ba(OH)_2$, MgO, zeolites and CaO.

The result in the experiment after 4 hours is described below:



Catalyst	%Ester	Time, (min)
KOH	99	60
Li/CaO	74	480
$Ba(OH)_2$	99	480
MgO	0	480
Zeolite (NH_4CsY)	0	480
CaO	0	480

Reaction conditions: Temperature-50°C, Catalyst-1%, RPM- 600, Oil to methanol molar ratio-1:6

Table 1: The biodiesel produce in percent for each catalyst.

It shows that $Ba(OH)_2$ catalyst is the only heterogeneous catalyst that can achieve 99% conversion 4 hours.

3.2. Literature Review Conclusion

From this study, we can conclude that the fastest conversion rate in biodiesel is by using Ba-ZnO.

The highest conversion rate in biodiesel is by using $Ba(OH)_2$.

From this study, it is a good starting point to identify which factor that can effect the highest conversion. It is either the existing of barium, metal oxide or hydroxide.

3.3. Preliminary Jatropha Selection

A preliminary test of Free Fatty Acid(FFA) need to be done to make sure the FFA is not too much as it will cause the soap formation with the alkaline catalyst. The soap can prevent separation of biodiesel from glycerin.

From Shashikant (19), had stated that amount of FFA that more than 1% w/w can cause soap forming in the absence of alkaline catalyst. This shows that FFA percentage contain is very crucial in biodiesel process as it can form an unwanted products.

In order to test the Jatropha oil Free Fatty Acid percentage, a titration of oil with the Sodium hydroxide need to be done to make sure it can produce a good result and maximum conversion of biodiesel.

The procedure for Free Fatty Acid test is described below:

The process for titration is as follows:

1) Place 10mL of iso-propanol in a 50ml flask
2) Add 1mL of vegetable oil (mix the oil thoroughly prior to drawing 1ml)
3) Mix the oil with the iso-propanol using the squirting action of the dropper
4) Add the ph indicator solution (usually 3 drops, check instructions)
5) Place 15ml 0.1% NaOH (know as titrant) solution in a 50ml flask
6) Draw exactly 5ml of the NaOH solution into the graduated pipette
7) Add the 0.1% NaOH to the iso-propanol/oil mixture one drop at a time. Mix the solution using a swirling action between the drops. Using the eyedropper to mix the solution may help if the oil forms drops in the bottom of the flask.
8) Continue to add 0.1% NaOH until a pH of 9 (blue-green color) is reached. This may require more than 5ml. Refill the pipette and continue. Note the amount in ml that was required.

Two samples of Jatropha oil were tested to determine the FFA %. The samples were put in two conditions before the oil been extracted:

Sample 1: Jatropha seeds in oven

Sample 2: Jatropha seed in sealed container.

Figure 10: The Comparison of Jatropha in the different conditions.

The titration procedure had been done and the amount of NaOH required to turn the phenolphthalein into a pink color is described below:

	NaOH required (mL)
Sample 1	12
Sample 2	3.1

Figure 11: NaOH required for Jatropha Oil to become neutralized

To determine the FFA %, the formula is:

Finding the moles of NaOH used,

$$\text{Mole of NaOH} = \frac{MV}{1000}$$

$$\text{Mole of NaOH} = \frac{(0.025)V}{1000}$$

In stoichiometric terms, 1 mole NaOH will neutralize 1 mole of oleic acid (OA)

Moles of NaOH = Moles of Oleic Acid

Mass of Oleic Acid = moles x molecular weight of OA (282.52)

%FFA = (mass of oleic acid) x amount of NaOH used (mL)

The value for the %FFA for each sample is:

	NaOH required (mL)	Mole of NaOH	Mass of OA	%FFA
Sample 1	12	0.0003	0.084756	10.17072
Sample 2	3.1	0.0000775	0.0218953	0.678754

Figure 12: Amount of FFA%

From the result above, we can conclude that the sample 1 can't be use in transesterification process as it contains high FFA. It could effect the product conversion and the soap that form can poison the heterogeneous catalyst.

To cater the problem, new samples of Jatropha need to be store in dry condition and a suitable temperature. The Jatropha need to be keeps in the room temperature in dry condition. The change in condition could increase the seed Free Fatty Acids.

CHAPTER 4

4. METHODOLOGY

4.1. Standard Procedure

The procedure in developing each part of the experiment is needed to make sure the quantity of the chemical add or the temperature for an experiment is right. The procedure is described below:

4.1.1. Catalyst Preparation

1. Prepare a 50 mL beaker
2. Prepare 500 mL distilled water
3. Prepare 25 mmol of $Ba(NO3)2$ = 6.5 g of Barium Nitrate
4. Prepare 10g of Zinc oxide.
5. Insert the Barium Nitrate into the beaker and fill with 500 mL distilled water.
6. Stir it until it become well-mixed.
7. Pour the zinc oxide into the beaker and mix it with the aqueous solution. The mixing will be stir using the stirrer for about 2 hours.
8. Dry the slurry overnight in the oven by setting the temperature to 393 K(120°C).
9. The dried material will be calcined at the temperature of 573 K(600°C) in the furnace for 5 hours.

4.1.2. Soxhlet Extractor Extraction Process

1. Prepare 140 mL of hexane.
2. 10g of Jatropha seed.
3. Insert the hexane in the 250 mL rounded bottom flask.
4. Put the thimble inside the Soxhlet chamber.
5. Fill the thimble with prepared Jatropha.

6. Install the condenser and other fitting as visual.
7. Set the temperature of the heater to 70°C (hexane boiling point)
8. Let the reflux process for about 2 hours.
9. Turn off the heater after 2 hours and let it cool.
10. Insert the hexane mixture into the beaker.
11. To separate the hexane from the Jatropha oil, put the mixture into the evaporator flask.
12. Set the bath temperature to 70°C.
13. Let the process going for 2 hours.
14. The amount of oil extract need to be record. Record the amount in unit of mL.

4.1.3. Transesterification process.

1. 10 g of Jatropha oil were put into 250mL flask.
2. Prepare the Ba/ZnO catalyst by 6 wt%. About 0.6g of catalyst was put into flask.
3. The Alcohol (Ethanol/Methanol) will be put into the flask with molar ratio of 10:1, 12:1, and 14:1.
4. Reactions were reflux at 60°C (Methanol boiling point) or 80°C (Ethanol boiling point).
5. Stir the solution by using ultrasonication for 1 hour.

The catalyst produced from impregnation of Barium nitrate in ZnO will be used for transesterification of Jatropha oil.

The experiment will use methanol & ethanol as a reactant for the comparison.

4.2. Solvent Extraction

20g of flaked Jatropha seed will be put in the Soxhlet thimble. A 250 mL reaction flask will be fill with 150 mL hexane as a extracting solvent.

The apparatus need to be setup as describe below:

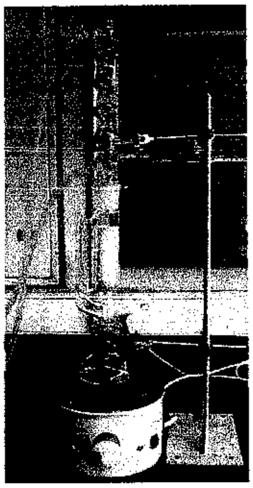


Figure 13: Soxhlet Extractor

The extraction will be run for 1 hour with the reflux temperature of hexane. The mixture of Jatropha oil and hexane will be separate by using rotary evaporator until the hexane evaporated completely.

4.3. Transesterification

A 250 mL flask will be use for the reaction. 10g of Jatropha oil will be used for the reaction and the volume of methanol or ethanol use is depending on the molar ratio of methanol to oil. The molar ratio used is 10:1, 12:1, and 14:1. The calculation for determine the methanol or ethanol volume is described below:

The sample for the calculation of volume of Ethanol need for 12:1 molar ratio

$$\begin{aligned}
&\text{Molar ratio} = 12:1\\
&\text{Mass of Jatropha} = 10.229\text{g}\\
&\text{mol of Jatropha} = \frac{10.229}{870} = 0.01176\,mol\\
&\text{mol of Ethanol} = \frac{0.01176}{1} \times 12 = 0.14112\,mol
\end{aligned}$$

$$\begin{aligned}
&\text{Mass of Ethanol} = \frac{0.14112}{6.5014} \times 46.07 = 6.5014\,g\\
&\text{volume of Ethanol} = \frac{6.5014}{790600 \,{}^{\text{g}}\!/_{\text{m}^3}}\\
&\qquad = 8.2234 \times 10^{-6}\,m^3 = 8.2234\,mL
\end{aligned}$$

The amount of Ba/ZnO catalyst used is about 6wt% of oil weight. The reactant and catalyst will be put into the flask and ultrasonic stirrer will be use by setting the temperature of alcohol reflux. The reaction will be set for 1 hour.

Figure 14: Vacuum filter separator

The mixture of catalyst, biodiesel, and glycerin will be separate by using vacuum filter. The oil filtered will need to undergo cleaning process to separate biodiesel from glycerin by adding hexane. The cleaning process will need to use separator funnel. The oil mixture will turn into two phases.

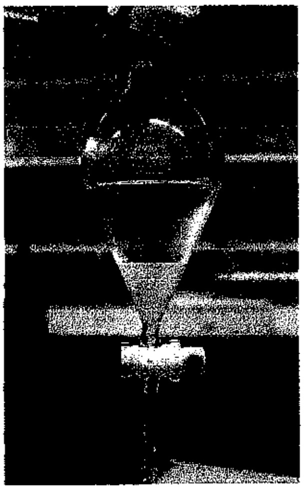


Figure 15: Separator funnel

4.4. TLC test

A TLC solution needs to be prepared by using Chloroform and hexane in the ratio of 1:1. TLC analysis was performed on TLC plate with the size of 2 x 10cm.

The prepared TLC plate will first dip horizontally into the chloroform: hexane solution in the TLC chamber until the plate is fully wetted.

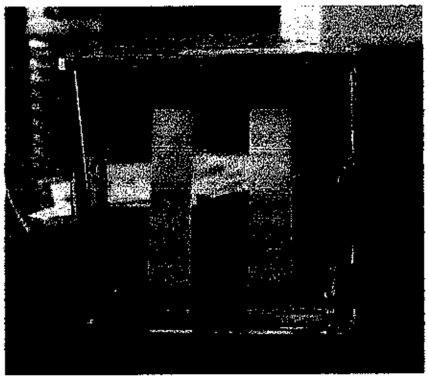


Figure 16: TLC plate in the TLC chamber

A capillary tube will be use for putting the sample spot on the TLC plate. The pattern will be visualized by using Iodine staining.

4.5. Catalyst characterization

The catalyst was characterized by using the XRD machine to characterize the component inside the catalyst. According to the Wenlei Xei (10), the catalyst will contain ZnO, $BaCO_3$, and $Ba(NO_3)_2$. The XRD pattern from her research is:

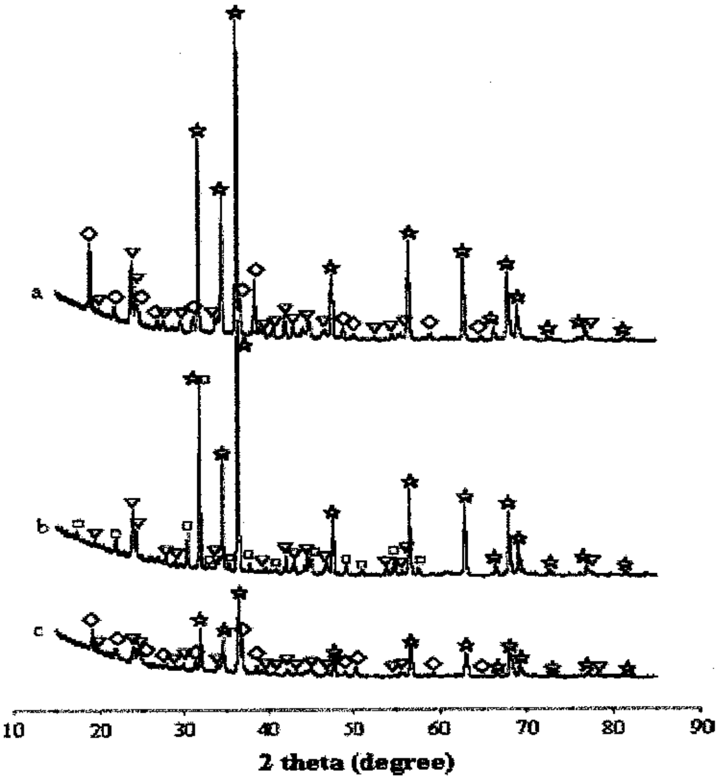


Figure 1. XRD patterns for samples: (a) 2.5 mmol/g Ba–ZnO calcined at 873 K, (b) 2.5 mmol/g Ba–ZnO calcined at 1073 K and (c) 4 mmol/g Ba–ZnO calcined at 873 K. (☆), ZnO; (▽), $BaCO_3$; (◇), $Ba(NO_3)_2$; (□), $BaZnO_2$.

Figure 17: XRD result from Wenlei Xei (10)

From this project, the XRD pattern is:

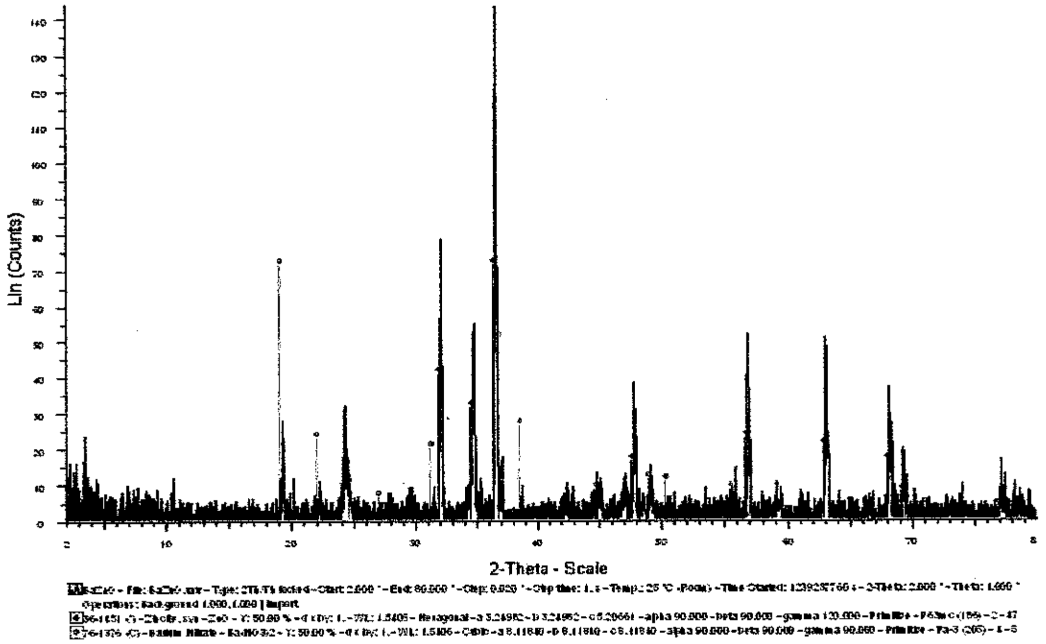


Figure 18: The XRD test result on Ba/ZnO catalayst. Green peak indicate Barium nitrate and Blue peak indicate Zinc oxide.

From the XRD, we can shows that the catalyst contains the desirable component to form a good Ba/ZnO catalyst. The green peak indicate the Barium nitrate and the blue peak for Zinc oxide. The black peak indicate the catalyst. In order to have Ba/ZnO catalyst, the component of Ba and ZnO need to be exist in the catalyst.

The pH test also indicates that the catalyst is a base catalyst as it indicates the pH 12. This basicity is required as the transesterification needs a base catalyst to react.

CHAPTER 5

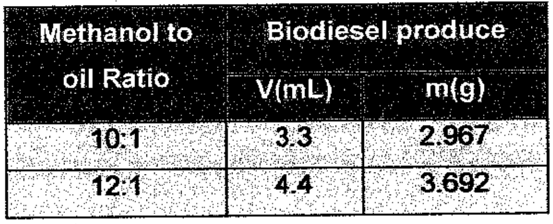
5. RESULT AND DISCUSSION

5.1. Experimental Result

5.1.1. The Effect of Alcohol

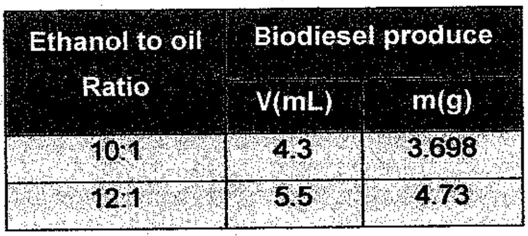
Four set of experiments had been ran that involved a study of Methanol and Ethanol comparison and also the comparison of the alcohol affect to the biodiesel conversion. The result is described below:

For Methanol



Methanol to oil Ratio	Biodiesel produce	
	V(mL)	m(g)
10:1	3.3	2.967
12:1	4.4	3.692

For Ethanol



Ethanol to oil Ratio	Biodiesel produce	
	V(mL)	m(g)
10:1	4.3	3.698
12:1	5.5	4.73

Figure 20: Result of Biodiesel produced with the different alcohol and molar ratio

The TLC pattern

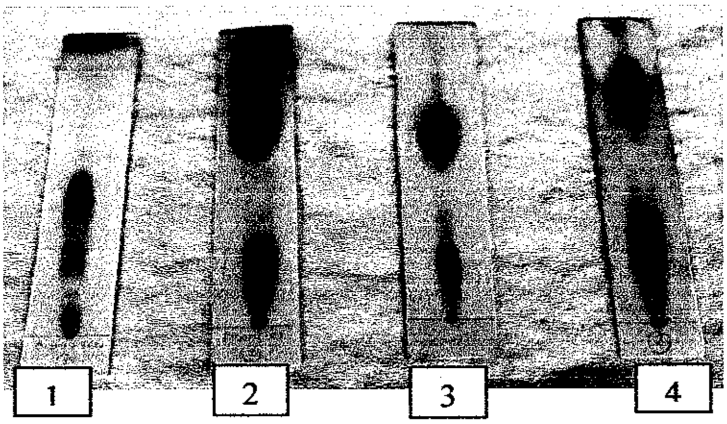


Figure 21: (1) 10:1 methanol ratio, (2) 12:1 methanol ratio, (3) 10:1 Ethanol ratio, (4) 12:1 Ethanol ratio

From the result, we can say that the higher the alcohol, the higher the biodiesel produced. By referring to Wenlei Xei (10), there will be a breakthrough point in adding the alcohol ratio where it will achieve the maximum conversion.

In the comparison of methanol and Ethanol, we can conclude that Ethanol resulting higher conversion then methanol. This is consistent to the study of biodiesel production by Suzana Yusup (21).

From the Suzana Yusup (21), the finding in using Ethanol and Methanol is:

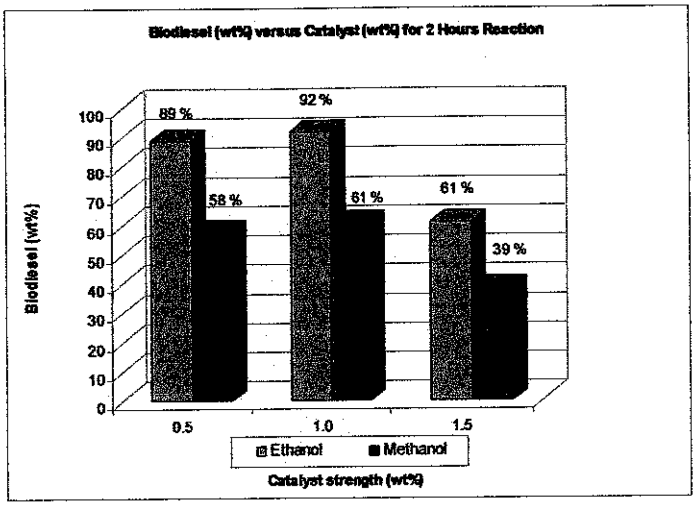


Figure 22: Effect of solvent on biodiesel yield

The biodiesel that yield with the same amount of catalyst and reaction time will be different when using different type of alcohol.

The explanation for this phenomena can be explain by referring to the A.K. Dalai (11), where he found that using Methanol as a reactant could form a three-phase system where consists of oil, methanol and catalyst. But, by using Ethanol, it can form a two-phase system that consists of Oil-Ethanol, and catalyst. The Ethanol has better solvent properties and a good mass transfer that can speed up the equilibrium.

As the Ethanol is been identified as a good reactant for the transesterification process, a further study need to be done to find the optimum condition for the reaction.

5.1.2. The Effect of Alcohol to oil molar ratio

The molar ratio of Ethanol to oil was study for Jatropha to find the optimum ratio for high conversion with the minimum reactant. From Wenlei Xei (10), had showed that the optimum molar ratio of alcohol to oil is 12:1.

From the experiment, the biodiesel yield is described from the table below:

Ethanol to oil Ratio	Biodiesel produce	
	V(mL)	m(g)
10:1	4.3	3.698
12:1	5.5	4.73
14:1	5.6	4.75
16:1	5.46	4.70

Figure 23: Biodiesel produce with the different molar ratio

As the Ethanol ratio more than 12:1, the yield is not increasing much as the increasing from 10: 1 to 12:1. In industry, alcohol were use in excess to maximize the biodiesel conversion. This could cost the producer more as it use more alcohol than what they need. From the study, we know that the reaction slowly stop producing the biodiesel after the molar ratio of alcohol to oil was 12:1.

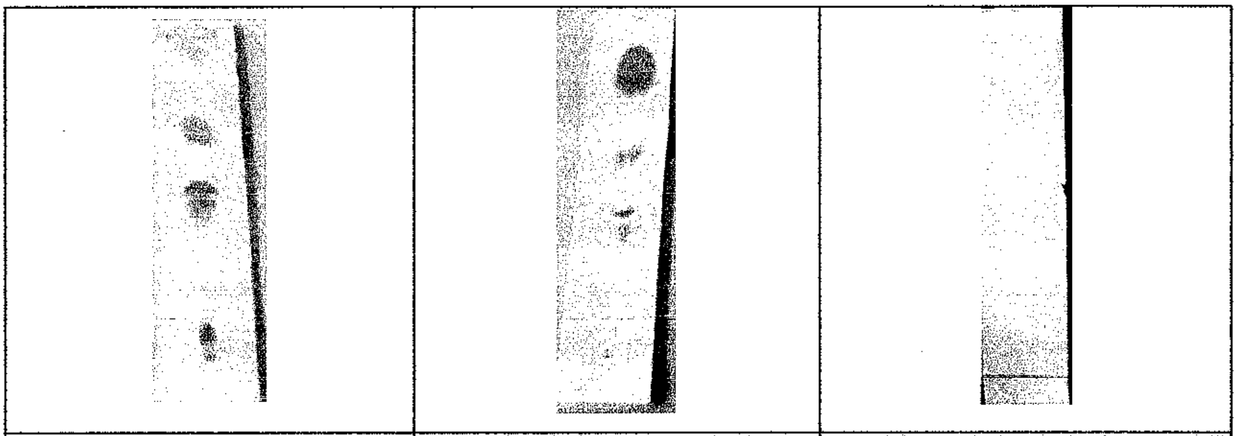
By this result, we can say that the optimum ratio that can achieve maximum conversion is by using 12:1 ratio.

5.1.3. The Effect of Reaction Time

From the Wenlei Xei (10), had stated that the in the reaction time of 1 hour, the transesterification can achieve highest conversion. By increasing the time after that will no effect the conversion. (Refer Figure 7)

In the experiment, three set of sample were taken for analyzing the biodiesel yield. The samples were taken at time 20 minutes, 40 minutes and 60 minutes.

The TLC pattern for each sample is described below:



t = 20 min

t = 40 min

t = 60 min

Figure 24: TLC pattern for each reaction time

The 3 spots before the last spot is indicate as glyceride (Monoglyceride, Diglyceride, and Triglyceride). The indication of the glyceride shows that the reaction is not complete yet. The triglyceride became lesser with the increasing of time.

At the time t=20 minutes, the spot of glyceride were large as same as biodiesel spot. This shows that the conversion is not complete yet. At the time of 40 minutes, the biodiesel spot is bigger than the glyceride spot. At the time of 60 min, the glyceride is nearly disappear and only left the biodiesel spot.

By increasing the reaction time more than one hour could increase the formation glycerol. The biodiesel produce will be hindered by the glycerol and it will form a gel where it is hard to filter the glycerol from biodiesel.

5.3. Problem encountered

A few problem that been encountered during the experimental process that might effect conversion of the product. The problem such:

1. Unrecovered biodiesel in the flask and vacuum filter.
2. Inaccurate temperature and fluctuate frequency setting at Ultrasonic stirrer.
3. Equipment failure in Vacuum Filter that reduce the volume of biodiesel flow by trapping the fuel in the filter.
4. Left the product too long in the flask after reaction that reaction might occur between glycerin and biodiesel that reduce the conversion.

5.4. Conclusion

From the result, we can conclude that Ba/ZnO catalyst can produce biodiesel from Jatropha. The conversion might be low if compared to the conversion of biodiesel produced from homogeneous catalyst but it is economical in the term of production cost and also can reduce pollution.

Ethanol can be use to maximize the conversion as it can create two-phase system and can increase the reaction mass transfer.

This heterogeneous catalyst could give a new prospect to the biodiesel industry to develop tremendously as the biodiesel production cost can be reduced.

By choosing Jatropha as the raw material for the biodiesel, we can prevent from any food crisis that might happen as Jatropha is a non-edible seed and it will not effect the food industry supply.

CHAPTER 6

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