



METHANOLYSIS OF TRIGLYCERIDES USING JATROPHA OIL AND KOH CATALYST

A. Ayodeji Ayoola*

C. Vincent Efevbokhan*

A. Opeyemi Adeeyo*

O. Oluwatosin Johnson*

Abstract: *In this study, Jatropha curcas oil was used as feedstock for biodiesel production by alkali-catalyzed methanolysis. To establish the optimum conditions for biodiesel production, effects of certain factors were investigated, these factors are oil-to-methanol molar ratios (1:4 - 1:8), KOH catalyst concentrations (0.5 - 1.5% w/w oil), reaction temperatures (50 - 70°C), and reaction times (55 - 90minutes). Biodiesel produced were analyzed to determine its viscosity, flash point and pour point. The experimental work revealed that optimum conditions for Jatropha curcas biodiesel production were oil-to-methanol molar ratio of 1:6, a catalyst concentration of 1.0% w/w oil, a reaction temperature of 60 °C, and a reaction time of 80 minutes. The methyl ester produced under these optimum conditions was 93.75 % w/w.*

Keywords: *Biodiesel, Jatropha oil, Methanol, Methanolysis, Trans-esterification, Triglycerides.*

*Member of Chemical Engineering Department, Covenant University Nigeria.



1 INTRODUCTION

Biodiesel is an alternative renewable diesel fuel that has properties comparable to diesel obtained from petroleum processing [7]. Since biodiesel is renewable and it creates less harmful exhaust emissions when combusted compared to that of petroleum diesel, the use of this fuel is a shift towards sustainable energy [12,6]. Biodiesel can be produced from vegetable oil, animal fat, organisms (such as microalgae) through a chemical reaction called trans-esterification with short chain alcohols [7,5,2]. Methanol is the most preferred alcohol, because it is cheap, readily available and reacts faster than any other alcohol.

Fats and oils are members of the lipids family. Lipids may either be a solid or liquid at room temperature, depending on their structure and composition. Normally, "oil" refers to a lipid that is liquid at room temperature, while "fat" refers to a lipid that is solid or semi-solid at room temperature. Fats and oils primarily consist of esters of glycerol (mono-, di-, and tri-glycerides) and low to moderate contents of free fatty acids (carboxylic acids) [14]. Other compounds such as phospholipids, polypeptides, sterols, water and other impurities can be found in crude oils and fats [15]. The structures of mono-, di-, and tri-glycerides (MGs, DGs, and TGs) consists of glycerol (a backbone of carbon, hydrogen, and oxygen) esterified with fatty acids (chains of carbon and hydrogen atoms with a carboxylic acid group at one end). Free fatty acids (FFAs) can contain 4-24 carbon atoms with some degree of unsaturation, typically 1-3 C-C double bonds [15]. High percentage of FFA ($\geq 5\%$) in oil or fat adversely affect the yield of biodiesel and increase production cost.

Oils containing a low percentage of FFA are currently being used for commercial biodiesel production [3,11,10]. *Jatropha curcas* oil is a good example of these kinds of oils. In recent years *Jatropha curcas* oil, non-edible oil, has gained much attention as a feedstock for biodiesel production. Many literatures indicates that the fatty acid methyl ester (biodiesel) of *Jatropha curcas* is one of the 26 fatty acid methyl esters of oils that are most suitable as biodiesel,¹¹ and results into an increase in revenues for agriculturists as well as the creation of new jobs.

The study of biodiesel production from *Jatropha curcas* oil has been conducted by several researchers, but with differing production processes, optimum conditions, and methyl ester yields therefore, it is necessary to further investigate the optimum conditions for biodiesel



production from *Jatropha curcas* oil. These are important reasons to make further studies of biodiesel production from crude *Jatropha curcas* oil by alkali-catalyzed methanolysis [9].

2 METHOD

Water content and density of the *Jatropha curcas* oil used were determined in order to determine amount of catalyst (KOH) required for the transesterification of oil. The experimental work involved investigations on the effects of methanol/oil ratios, concentration of KOH catalyst, reaction temperature and reaction time on both the quality and quantity of biodiesel (fatty acid methyl ester) produced.

In each experimental set up, calculated gram of KOH was added to methanol and stirred continuously and also warmed gently until it was completely dissolved resulting in a clear solution. Required quantity of oil was measured into a flat bottom flask and then heated to 55°C while stirring using a magnetic stirrer, the clear solution obtained from potassium hydroxide and methanol was then carefully added to the oil (considering the required methanol/oil ratio) and the flask was capped, to avoid evaporation of methanol into the atmosphere since the mixture is warmed. Stirring of the solution was done continuously, but gently to avoid formation of vortex.

The reaction was kept at the required temperature for a specified period of reaction time. At the end of the reaction time, the heat source was turned off and the mixing was stopped. The mixture was then poured into a separating funnel and allowed to cool and left for 24 hours, after which two clear layers were observed. The top layer was a golden yellow fatty acid methyl ester (biodiesel) and the bottom layer was a dark brown glycerol. Separation of these two layers was carefully done.

Crude biodiesel obtained was purified by washing with 50°C warm water thrice, in each experimental set up, to remove impurities like residual catalysts, soap and glycerol present. Two drops of H₂SO₄ was added to the warm water used for the first washing, to aid total removal of KOH present in the crude biodiesel. Pure biodiesel obtained was dried by heating to 120°C to remove water content present and then cool.

3 RESULTS AND DISCUSSIONS

3.1 Effect of Reaction Temperature on Biodiesel yield

Figure 1 shows the methyl ester (biodiesel) yield during methanolysis of *Jatropha curcas* oil at various temperatures. The results show that biodiesel yield increased steadily from the



starting reaction temperature of 50°C to 55°C and then to 60°C. Slight increase in biodiesel yield was still experienced when the reaction temperature was further raised to 65°C, although at this point the increase in biodiesel yield was 0.12%. Also, when the temperature of reaction was increased to 70°C, the increase in the yield was a meagerly 0.13% increase opposed to 2.13% increase in biodiesel yield experienced when varying temperature from 55°C to 60°C. These results are in agreement with those obtained by other researchers [4,1,13]. The increase in the biodiesel yield might have been due to decrease in oil viscosity with an elevation of reaction temperature, which resulted in an increase in the solubility of the oil in the methanol, leading to an improvement in the contact between the oil and the methanol. Although reaction temperatures above 60°C provided higher biodiesel contents, these reaction temperatures should be avoided for economic reasons because the increase in yield is very negligible as compared to the increase in biodiesel yield experienced at reaction temperatures of 60°C and that below it and would just be a waste of energy resulting into increase in production costs. So the optimum reaction temperature is 60°C.

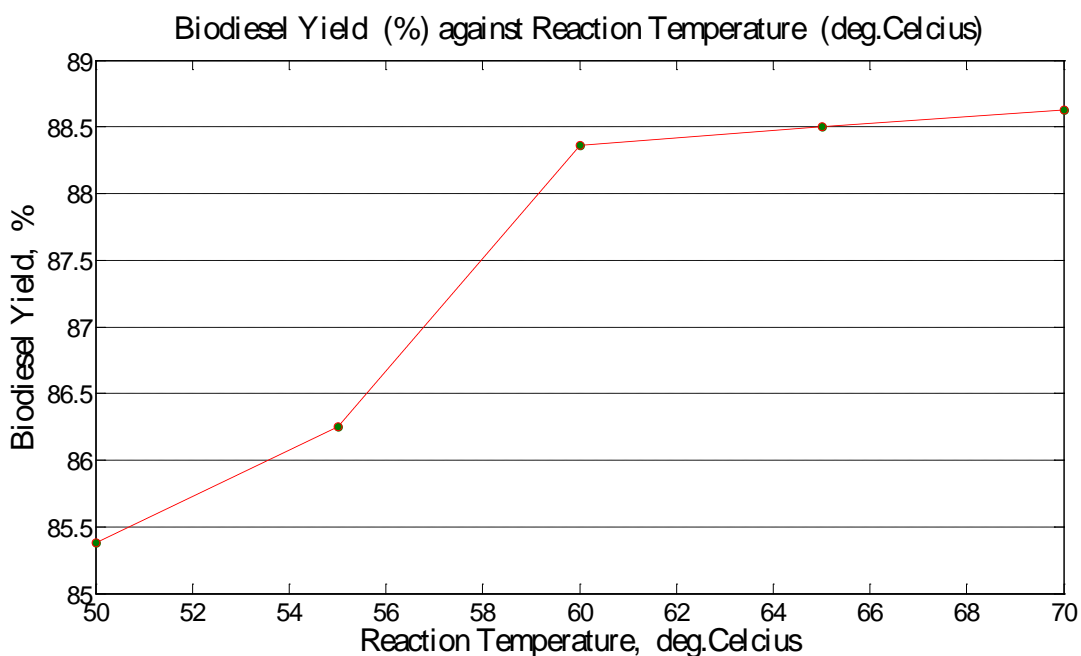


Figure 1: Yield of biodiesel against Reaction Temperature

3.2 Effect of Molar ratio of Oil/Methanol on Biodiesel yield

The reaction was carried out using a catalyst concentration of 1% w/w of oil and a reaction temperature of 60°C and the results of biodiesel yield at the various ratios are shown in Figure 2. It was observed that the biodiesel yield increased as the oil-to-methanol ratio



increased. The biodiesel yield increased steadily from 1:4 to 1:6 thus indicating a 3.83% increase. Although there was equally an increase in biodiesel yield from ratio of 1:6 to 1:7 and then to 1:8, but the increase was insignificant compared to what had been experienced when raising the ratio from 1:4 to 1:5 and then 1:6. Thus, the oil-to-methanol molar ratio of 1:6 is the best oil/methanol molar ration for economical reasons. In industrial applications, the methanol consumption in the production can be reduced by recycling methanol recovered from the biodiesel and glycerol layers using a distillation apparatus, by so doing operating costs would be reduced.

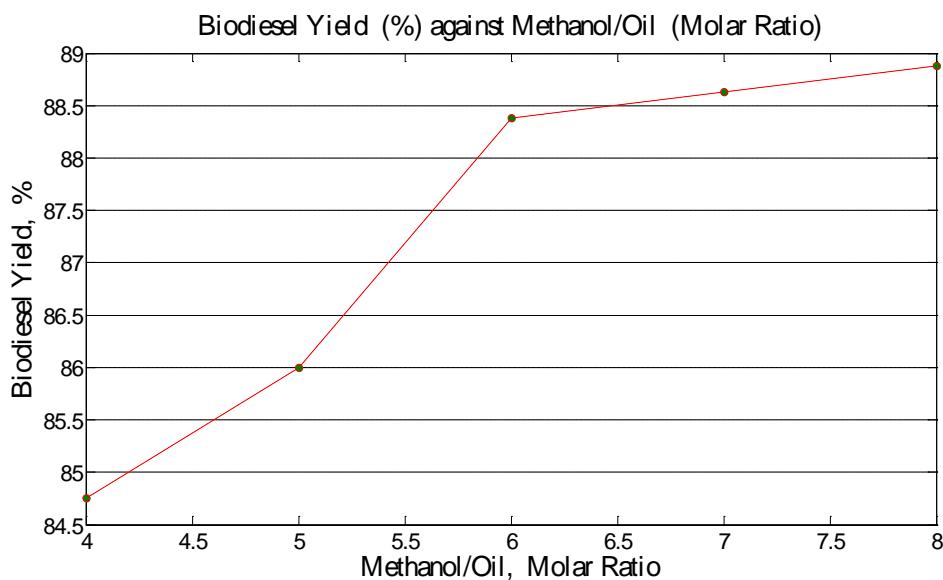


Figure 2: Biodiesel yield against weight of methanol

3.3 Effect of KOH Catalyst

The experiments were carried out with varied concentrations of KOH using an oil-to-methanol ratio of 1:6 and a reaction temperature of 60°C. The effect of the variation in the concentration of KOH catalyst on biodiesel yield is as represented in **Figure 3**. The results show that the concentration of KOH had a significant effect on the alkali-catalyzed methanolysis. The biodiesel yield increased with higher catalyst concentrations. This has also been reported by researchers who had previously delved into this area [5,4,1,13,16] Biodiesel yield increased as the catalyst concentration increased from 0.5 to 0.8 and then to 1.0% w/w by percentage increase of 1.13 and 2.87% respectively. The percentage increase in biodiesel yield began to reduce after 1% w/w as indicated by the 0.25 and 0.13% obtained for 1.0 to 1.2 and then to 1.5% w/w. 1.0% catalyst concentration was chosen as the optimum concentration because the resulting increase in the input of KOH was not



adequately reflected in the biodiesel yield which means that the additional KOH is just being under-utilized and thus wasted when it could be used for another production run. Also, these concentrations should be avoided because of the cost of removing the residual catalyst in the biodiesel layer. Moreover, the biodiesel obtained from using these catalyst concentrations has to be washed several times resulting into loss of biodiesel to emulsion formation.

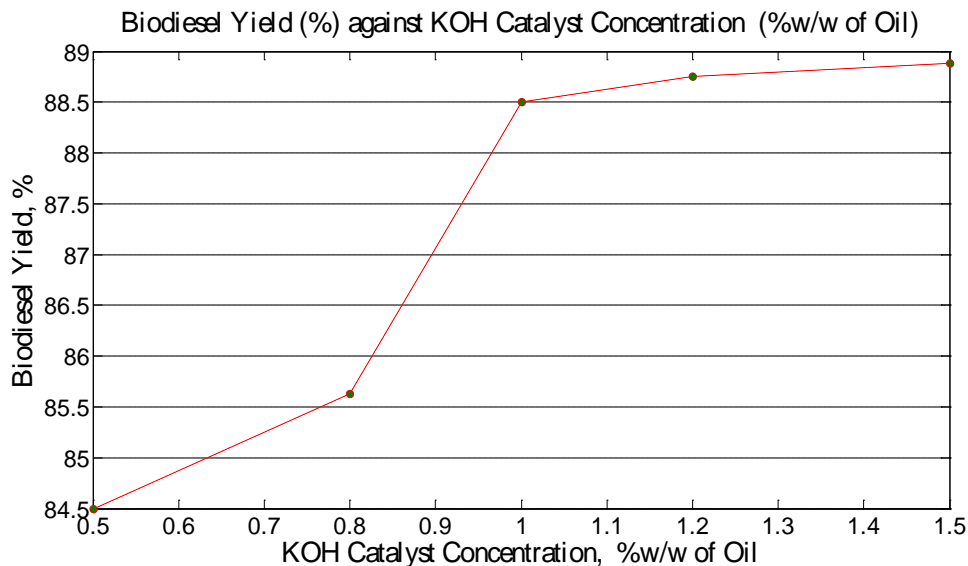


Figure 3: Biodiesel yield against catalyst concentration

3.4 Effect of Change in Reaction Time on Biodiesel yield

Having obtained optimum conditions of 60°C reaction temperature, 1% catalyst w/w of oil concentration, oil-to-methanol molar ratio of 1:6, experimental runs (at different time interval) were then carried out to determine the optimum reaction time for biodiesel production. The results obtained were displayed in Figure 4. The yield of biodiesel increased steadily from reaction time of 55minutes to 90minutes. Reaction time of 80minutes was chosen as the optimum reaction, since just a little increase in weight of biodiesel (0.3g) was observed when the reaction was allowed to linger for a further 10minutes after 80minutes of reaction opposed to the 0.5, 2.8 and 1.4g increase experienced when raising reaction time from 55 to 60, 70 and finally to 80minutes respectively.

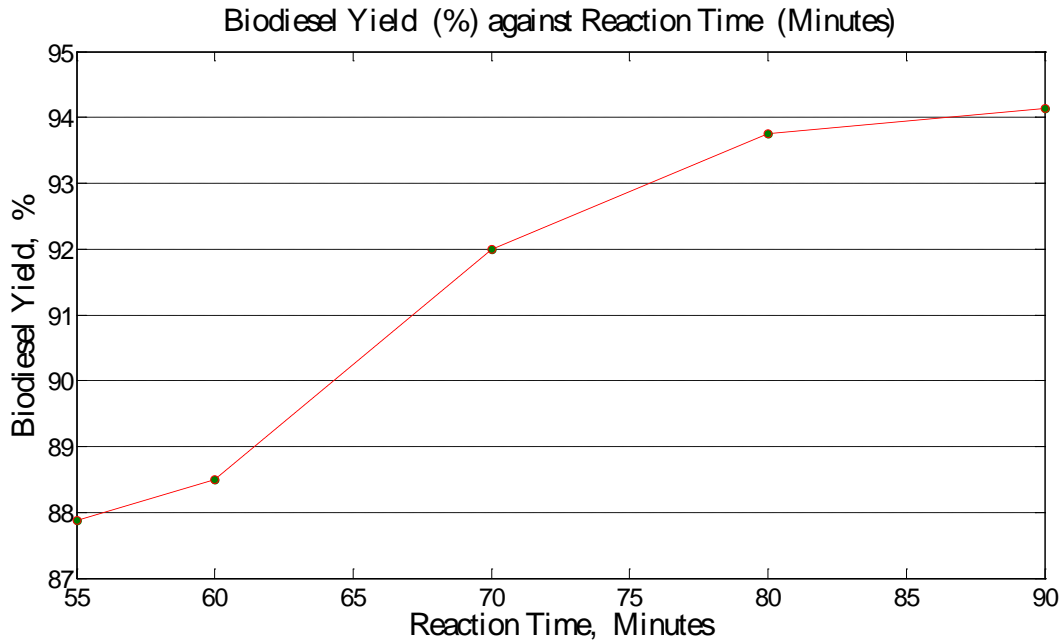


Figure 4: Biodiesel yield against Reaction Time

4 PROPERTIES OF JATROPHA BIODIESEL

The quality of biodiesel is very important for the performance and emission characteristics of a diesel engine. Thus, the *Jatropha curcas* biodiesel produced at optimum experimental conditions was sent to the Chemical Engineering Department laboratory of the University of Lagos (UNILAG, Nigeria) where tests were carried out.

The results showed that the alkali-catalyzed methanolysis produced *Jatropha* oil with the specific gravity of 0.855, viscosity of $4.000\text{mm}^2/\text{s}$ which is slightly higher than that of petroleum diesel ($3.068\text{mm}^2/\text{s}$). Pour point, an important parameter associated with the performance of diesel engine in cold weather conditions, was obtained to be -3.5°C . Also, the flash point of the biodiesel fuel was obtained to be 139.5°C which is higher than that of petroleum diesel (67.5°C) and blending even a small percentage of biodiesel with diesel would increase the flash point of petroleum diesel. Therefore, *Jatropha curcas* biodiesel or a blend of *Jatropha curcas* biodiesel and petroleum diesel would be safer than pure diesel for storage, transport, energy generation and handling.

TABLE 1: Some Properties of Pure *Jatropha* Oil

| Property | Value |
|--|--------|
| Density (g/L) at 20°C | 874 |
| Specific gravity | 0.874 |
| Water content (w/w of oil) | 0.870 |
| Saponification value of oil (mg KOH/g oil) | 202.50 |



TABLE 2: Some Properties of Biodiesel Produced at Optimum Conditions

| Property | Method | Jatropha Biodiesel @ Optimum Conditions | | |
|--|-----------|---|----------|---------------|
| | | Sample 1 | Sample 2 | Average Value |
| Flash point (°C) | ASTM D-93 | 138.0 | 141.0 | 139.5 |
| Pour point (°C) | ASTM D-97 | -3.0 | -4.0 | -3.5 |
| Viscosity at 40°C (mm ² /s) | | 4.000 | 4.000 | 4.000 |
| Specific gravity | | 0.860 | 0.850 | 0.855 |

5 CONCLUSION

This study shows that biodiesel can be produced successfully from *Jatropha curcas* oil by alkali-catalyzed methanolysis. These experimental investigations revealed that the optimum conditions for *Jatropha curcas* biodiesel production are a reaction temperature of 60°C, KOH catalyst concentration of 1% w/w of oil, oil-to-methanol ratio of 1:6 and reaction time of 80minutes. Produced under these conditions, the biodiesel yield obtained was 93.75% w/w. due to the low price of KOH, the short reaction time and high biodiesel yield obtained, these optimum conditions can be used in large-scale production.

Further work can be done on the use of some other catalysts and crude *Jatropha curcas* oil.

REFERENCES

- [1] Ankapong, E, The Influence of Physicochemical Characteristics of Vegetable Oils on the Quality of Biodiesel Produced from Palm Oil, Palm Kernel Oil, Refined Soyabean Oil, Unrefined Soyabean Oil and *Jatropha Curcas* Oil, MSc. Dissertation, Kwame Nkrumah University of Science and Technology, Ghana, 2010.
- [2] Cheng, J, Biomass to Renewable Energy Processes, 2-5, pp.338-341, 2010.
- [3] Duncan, J, The Cost of Biodiesel Production, Energy Efficiency and Conservation Authority, 2003.
- [4] Gerpen, J.V, Biodiesel Processing and Production, Fuel Processing Technology, 86, pp. 1097-1107, 2005.
- [5] Issariyakul, T, Development of Biodiesel Production Processes from various Vegetable Oils, PhD. Thesis, University of Saskatchewan, 2011.
- [6] Marchetti, J.M, Miguel, V.U and Errazu, A.F, Techno-Economic Study of different Alternatives for Biodiesel Production, Fuel Process. Technology, 89, pp.740 – 748, 2008.



- [7] Meher, L.C, Sagar, D.V and Naik, S.N, Technical aspects of biodiesel production by Transesterification—A Review, *Renewable Sustainable Energy Revolution* 10/3, pp. 248-268, 2006.
- [8] Mohibbe, A.M and Waris, A.N, Prospects and Potential of Fatty Acid Methyl Esters of some Non-Traditional Seed Oils for Use as Biodiesel in India, *Biomass Bioenergy*, 29, pp.293-302, 2005.
- [9] Nakpong, P and Wootthikanokkhan, S, *Journal of Sustainable Energy & Environment*, 1, pp.105-109, 2010.
- [10] Omar, W.N and Amin, N.A, Biodiesel Production from Waste Cooking Oil over Alkaline modified Zirconia catalyst, *Fuel Process Technology*, 92, pp.397–2405, 2011.
- [11] Ramadhas, A.S, Jayaraji S and Muruleedharam, C, Biodiesel Production from high FFA Rubber Seed Oil, *Fuel*, 84, pp.335-340, 2005.
- [12] Ramadhas, A.S, Muraleedharan, C Jayaraj, S, Performance and Emission Evaluation of a Diesel Engine Fueled with Methyl Esters of Rubber Seed Oil, *Renewable Energy*, 30, pp.1789-1800, 2005.
- [13] Richard, C.E, Determination of Optimal Methanol: Oil Volume Ratio for maximum Biodiesel Production from Waste Cooking Oil, *Journal of Physical Sciences and Innovation*, 2, pp.38-43, 2010.
- [14] Srivastava, A and Prasad, R, Triglycerides-based Biodiesel Fuels, *Renewable Sustainable Energy Review*, 4, pp.111-133, 2000.
- [15] Suwannakarn, K, Biodiesel Production from High Free Fatty Acid Content Feedstocks, PhD. Thesis, Clemson University, 2008.
- [16] Zhou, W, Konar, S and Boocock, D.G, Ethyl Esters from the Single-Phase Base-Catalyzed Ethanolysis of Vegetable Oils. *J. Am Oil Chem. Soc.* 80, 4, pp.367–371, 2003.