MedCrave
Step into the World of Research
Material Science & Engineering International Journal

Research Article
Open Access
CrossMark

Influence of esterification and neutralization in the production of biodiesel: a comparison study

Abstract

The challenges of emission of greenhouse gases (GhG) have triggered researchers all over the world to come up with alternative fuel sources that reduce greenhouse gas emissions. One of such alternatives is Biodiesel production from Neem and Jatropha seed oils. However, the presence of Free Fatty Acids (FFA) in the oil obtained from these seeds reduces the yield of produced biodiesel. Two common methods proposed in previous literature to reduce the free fatty acids value are the Esterification and Neutralization methods. This study focused on comparing the esterification and neutralization method as a preliminary stage of biodiesel production from Neem seed and Jatropha seed oils, in an effort to advance biodiesel production in terms of yield from its process. The Neem and Jatropha seed oils were esterified with concentrated sulphuric acid and were tested for free fatty acids. Both seed oils were then refined via the degumming process, after which they were neutralized with sodium hydroxide. The study shows the neutralization method with a yield of 96% and 94% for Neem seed oil and Jatropha seed oil respectively to be more efficient than the Esterification method which produced a yield of 93.2% for Neem seed oil and 91.4% for Jatropha seed oil.

Keywords: biodiesel, free fatty acid, esterification, neutralization, transesterification, density

Volume 6 Issue 2 - 2022

Ajadi IK,[1] Oyegoke T,[1] Abdullahi NB,[1] Musa Q,[1] Hassan Y[2]
[1]Chemical Engineering Department, Ahmadu Bello University Zaria, Nigeria
[2]Petroleum Engineering Department, University of Port Harcourt, Nigeria

Correspondence: Ajadi IK, Chemical Engineering Department, Ahmadu Bello University Zaria, Nigeria, Email kazeemolawale634@gmail.com

Received: June 15, 2022 | **Published:** June 27, 2022

Introduction

The adverse effects of fossil fuel on the environment and its high price have encouraged governments and researchers to consider alternative renewable sources of fuels.[1] Fossil fuel usage creates pollution. The pollutants include the emissions coming from vehicles such as carbon monoxide, sulfur and nitrogen oxides, and suspended fine particles. The emission of greenhouse gas to the atmosphere as a result of combustion from fossil fuels is also blamed for global warming. Both human health and the environment can be threatened by fossil fuel usage. Thus, environment-friendly and nontoxic fuel is needed.[2] Current work on energy has been focused on saving the planet and meeting man's need for energy in the most cost-effective way. An example of biofuel which is well known as an alternative to petroleum diesel fuel, is biodiesel,[1] which is produced from renewable biological sources, like algae, plant oils (Neem seed oil, jatropha oil, sunflower, soybeans, and many others) and animal fats.[3,4] Edible feed stocks are discouraged in biofuel production, while non-edible oil like jatropha oil, Neem seed oil, and others are promoted. This is because it rules out the chances of competing with food chain.[5]

A survey of literature indicates that many research works have attempted to investigate ways of addressing the fossil fuel hazard by providing alternative fuels, known as biofuels, which entails biodiesel, bioethanol, and biogas. Biodiesel, an example of biofuel is well known for being a combination of alkyl ether derived via transesterification reaction. Among the reported studies in the literature that gave attention to the study of biodiesel production using esterification and neutralization means of reducing the free fatty acid (FFA) include Ajadi et al.,[5] where esterification of Neem oil results to an FFA value of 1.6 % from 20.035 % using two-step esterification processes. Another study by Haziret et al.,[6] investigated that the esterification of jatropha oil gave an FFA value of 1% using the sulphuric acid (H_2SO_4) as the catalyst at 0.225% (v/v), and a Methanol-to-oil ratio of 12.29% (w/w) and a reaction time of 149.76 min are the optimum process parameters for the esterification reaction. Novizar et al.,[7] reviewed the extraction, pretreatment and characterization of jatropha oil using neutralization method using KOH 1.5 wt-%, Methanol-to-oil ratio 4.50:1 mol, a reaction time of 30 min and yield of 92%. This study was aimed at comparing the esterification and neutralization method as preliminary stage of biodiesel production from Neem seed and jatropha seed oils, in other to advance biodiesel production in terms of yield from its process. By comparing with Ajadi et al.,[5] and Hazir et al.,[6] the findings from this study would go a long way to explore effective means of producing eco-friendly fuel with a more economical process that would suitably replace the current usage of fossil fuels that has largely been contributing to the rising greenhouse gases (GHG) in our environment.

Methods and materials

Materials and reagents

Hydrochloric acid (95%, analytical grade BDH), potassium hydroxide (analytical grade M&B), phenolphthalein (indicator), methanol (99.5%, analytical grade BDH), isopropyl alcohol (99.5%, analytical grade, BDH).

Esterification of neem and jatropha oil using sulfuric acid catalyst

Esterification of FFA is of great importance because, when % FFA is high, soap is being formed in the course of the transesterification, which increases the solution's viscosity and subsequently reduces the yield of biodiesel.[8] The reaction is reported to be carried out without much complication, when the % FFA is below 1% w/w.[9] For this study, the oils were esterified by the following method: 100 g of jatropha oil was esterified with 2.25g of methanol, and 0.05g of concentrated sulfuric acid. Also, 500g of Neem oil was esterified using same method. The oils were first preheated in a conical flask to 60°C before methanol/sulfuric acid mixtures were added, stirred and heated at 60°C using a magnetic stirrer for 60 minutes. After the esterification, the content was transferred into a separating funnel where the ester

was drawn, leaving behind a water and acid mixture. The separated esters were tested for free fatty Acid by titration method.[10]

Refining of neem and jatropha oil via degumming process

The degumming of crude Neem and jatropha oils which were purchased from National Research Institute for Chemical Technology was carried out as follows, for each of the oils; 200 ml was measured in a flat bottom beaker and heated to 65 °C. Thereafter, 800 ml of deionized water was poured. The mixture was heated to 65 °C and stirred at 400 rpm using a magnetic hotplate for 30 minutes. The mixture was allowed to cool and later poured into a separating funnel for water removal.

Neutralization of degummed neem and jatropha oil

The neutralization of the degummed oils was done as follows; to both degummed Neem and jatropha oil, 0.125 M NaOH solution was added, heated to 80 °C and stirred at 400 rpm using a magnetic hotplate for 40 minutes. The neutralized oil was washed of the soap with deionized water in the separating funnel. The collected oil was heated to 120°C at 30 minutes to remove any traces of water. 120 ml of refined Neem and jatropha oil was gotten from this process. The refined oils were tested for free fatty Acid by titration method.[10]

Acid value and free fatty acid determination

In determining the acid value and free fatty acid, 0.1 M KOH was titrated against 2g (2.1 ml) of oil sample, thereafter average volume of KOH consumed for complete reaction was determined. The acid values and free fatty acid were calculated using Equation 1 and 2 in with method deployed in existing literature,[5] ASTM-D 974). The acid value is computed as:

$$AV = \frac{A \times M \times 56.10}{W} \quad (1)$$

$$FFA = \frac{AV}{2} \quad (2)$$

Where A, Vol. of 0.1M KOH consumed by sample (average titer value), M, molarity of KOH (0.1 M), Molar mass of KOH, 56.11g/mol; W, weight in grams of the sample (2g); FFA, free fatty acid of the oil; and AV, acid value of the oil.

Transesterification

A modified biodiesel reaction method was employed in terms of timing, catalyst, temperature, methanol to oil ratio, and a biodiesel reactor according to Bello et al.,.[11] To 12 g of the pretreated oil, 6:1g methanol to oil ratio with 1% w/w of the catalyst was added. It was then heated and stirred at 60°C and 500 rpm respectively for 60 minutes. The yield of biodiesel using the expression is presented in Equation 3 (Figure 1).

$$Yield = \frac{AB \times 100}{AO} \quad (3)$$

Where, Y, yield; AB, amount of biodiesel produced; AO, amount of oil used

Density determination

The density of Neem and jatropha oil and their esterified and neutralized biodiesel produced were determined using density bottle. Initial weight of the bottle was measured on a digital scale and recorded, the bottle filled with samples was also measure and the difference between empty bottle and bottle filled with samples is the real density. The values of various density obtained were recorded in Table 2 below.

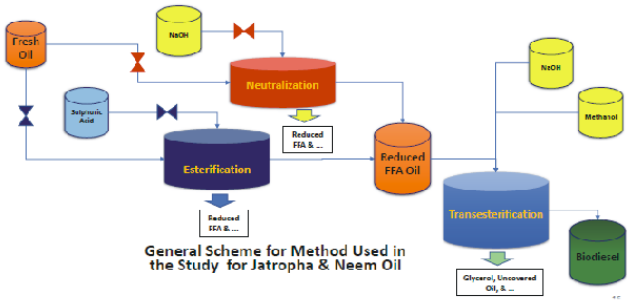

Figure 1 workflow diagram for both pretreatment and transesterification of the oils.

Result and discussions

Result of the free fatty acid

The free fatty acid (FFA) of Neem and jatropha oils, for the control and after the 1st, and 2nd esterification processes where, 13.9mgKOH/g,1.43mgKOH/g, 0.701mgKOH/g, 2.1mgKOH/g, 0.561mgKOH/g respectively while that of the control and after the 1st and 2nd of neutralizations processes gave 14.2mgKOH/g, 1.222mgKOH/g, 1.02mgKOH/g, 1.2mgKOH/g, 0.813mgKOH/g respectively. FFA was also determined on the biodiesel produced from esterified and neutralized Neem and jatropha oils, the values obtained respectively are; 0.695mgKOH/g, 0.557mgKOH/g and 0.705mgKOH/g, 0.57mgKOH/g. The two-step esterification process was adopted to reduce the FFA which has been reported by Ibrahim et al.,[10] to be a potential inhibitor that would aid in reducing the yield of methyl esters. The lower the free fatty acid of the oil, the higher the yield of the methyl esters.

The result in Table 1 obtained from the drop FFA indicated that the oil gets transformed into biodiesel via the transesterification process. Etelvina et al.,[13] reported in the biodiesel analysis that the free fatty acid (acid number) of the biodiesel produced using sunflower oil was 0.7mgKOH/which in line with what was reported for both esterifying and neutralized free fatty acid (acid number) of Neem and jatropha biodiesel in this research. For Neem oil it was observe from the first and second esterification that the rate at which the FFA reduces in second esterification is half of the first esterification and for neutralization the second neutralization is four times lower than the first neutralization which as a result of longer reaction time than esterification. Also, for jatropha oil it was noticed that the second esterification is just about 0.2 differences and for second neutralization the value is almost half reduced of the first neutralization. There is no much difference in esterifying and neutralizing of jatropha oil is as a result of initial high FFA value of jatropha. Ajadi et al.,[5] esterification of the Neem seed oil gave a free fatty acid value of 1.6%, and Hazir et al.,[6] esterification of jatropha oil gave a free fatty acid value of 1%, carried out esterification on Neem and jatropha oil and the above result were gotten. In this work the value of FFA obtained for both esterification of Neem and jatropha were 0.701g and 1.02g respectively, which is bellow the findings reported by Ajadi et al.,[6] using Neem oil and the value is the same as what is reported by Hazir et al.,[5] using jatropha oil.

Citation: Ajadi IK, Oyegoke T, Abdullahi NB, et al. Influence of esterification and neutralization in the production of biodiesel: a comparison study. *Material Sci & Eng.* 2022;6(2):64–66. DOI: 10.15406/mseij.2022.06.00181

Table 1 Free fatty acid (FFA) of neem and jatropha oil using esterification and neutralization

Oil type	Free Fatty Acid (FFA), mgKOH/g						
	Control	First esterification	Second esterification	First neutralization	Second neutralization	Esterified biodiesel	Neutralized biodiesel
Neem oil	13.9	1.43	0.701	2.1	0.561	0.695	0.557
Jatropha oil	14.2	1.222	1.02	1.2	0.813	0.705	0.57

Result of the density analysis

The density of Neem and jatropha oil and their esterified and neutralized biodiesel produced was found to be 0.9073, 0.8700 & 0.8233 g/m^3, and 0.9200, 0.9000 & 0.809g/m^3 respectively. The result in Table 2 obtained from the densities indicated that a drop in the densities of biodiesel after transesterification process shows that a reaction has taking place and biodiesel is produced. This density result is in line with the value gotten by Estelvina et al.,[13] where it was reported that the density of biodiesel using sunflower oil was 0.87g/m^3. It was observed that the esterified and neutralized biodiesel is lower compare to initial density of Neem and jatropha oil. Also, it was observed that the density of neutralized biodiesel in both oils are lower compare to esterified biodiesel, this is due to lower FFA in the neutralized oil as compared to esterified oil before transesterification. Ajadi et al.,[5] reported that the density of biodiesel Produced using Neem oil was 0.823 kg/m^3 and is in line with that obtained in this work which is 0.8233 kg/m^3.

Table 2 Density of neem and jatropha oil and biodiesel

Density	Unit	Control	Esterify bio diesel	Neutralize biodiesel
Neem oil	g/m3	0.9073	0.87	0.8233
Jatropha oil	g/m3	0.92	0.9	0.809

Effect of esterified and neutralized in biodiesel yield

The free fatty acid (FFA) has a significant impact on biodiesel yield as the lower the FFA the higher the yield, in line with results presented in Table 3. From the result presented in the table below it shows that neutralized has the highest biodiesel yield compare to esterified due to the lower FFA of neutralized oil compare to esterified oil, this is due to the fact that neutralization has lower FFA in both oils as reported above.

Table 3 Yield of neem and jatropha biodiesel

Yield	Unit	Esterified biodiesel	Neutralized biodiesel
Neem oil	%	93.2	96
Jatropha oil	%	91.4	94

Conclusions

The research findings confirmed that the FFA of esterified and neutralized oils were determined and it was observed that neutralized is lower compared to esterified in both oil and biodiesel produced due to longer reaction time. This low FFA also resulted in the lower density of the neutralized biodiesel. The yield of biodiesel in neutralized oil is a bit higher than esterified oil. Also, the amount of feed stock lost during esterification is about 5% while the amount of feed stock lost during neutralization is about 20%.

Acknowledgments

None.

Conflicts of interest

The authors state that there is no conflict of interest.

Funding

None.

References

1. Zewude DA. Transesterification of neem seed oil into fatty acid methyl ester using Na_2O/CaO catalyst. *Addis Ababa: Addis Ababa Institute of Technology*. 2015.
2. Chen S. Design and synthesis of mixed oxides nanoparticles for biofuel applications. *Masters Thesis*. 2010.
3. Umaru M, Aberuagba F. Characteristics of a typical Nigerian *jatropha curcas* oil seeds for biodiesel production. *Research Journal of Chemical Sciences*. 2012;2(10):7–12.
4. Athar M, Zaidi S. A review of the feedstocks, catalysts, and intensification techniques for sustainable biodiesel production. *Journal of Environmental Chemical Engineering*. 2020;8(6):1–5.
5. Ajadi IK, Oyegoke T, Geoffrey TT, et al. Biodiesel production from neem seed oil: catalyst synthesis, the effect of time, and temperature on biodiesel yield. *Materials Science & Technology Society of Nigeria*. 2020;1:8–10.
6. Hazir Farouk, Seyed Mojib Zahraee, Atabani AE, et al. Optimization of the esterification process of crude jatropha oil (cjo) containing high levels of free fatty acids: a Malaysian case study. *Biofuels*. 2020;11(6):655–662.
7. Novizar Nazir, Nazaruddin Ramli, Djumali Mangunwidjaja, et al. Extraction, transesterification and process control in biodiesel production from Jatropha curcas. *Eur J Lipid Sci Technol*. 2009;111(12):1185–1200.
8. Tiwari AK, Kumar A, Raheman H. Biodiesel production from jatropha oil with high free fatty acids: an optimized process. *Biomass Bioenergy*. 2007;31(8):569–575.
9. Ghadge SV, Raheeman H. Biodiesel production from Mahua (*Madhuca indica*) oil having high free fatty acids. *Biomass Bioenergy*. 2005;28:601–605.
10. Ibrahim H, Mohamed F, Verlaine N. Effect of high free fatty acid feedstock on methyl esters yield using bulk calcium oxide catalyst. *International Journal of Scientific & Technology Research*. 2015;4(3):186.
11. Bello EI, Daniyan IA, Akinola AO, et al. Development of a biodiesel processor. *Research journal in engineeringand applied sciences*. 2013;2(3):182.
12. Estelvina Rodríguez Portillo, Araceli Amaya Chávez, Arturo Colín Cruz, et al. Qualitative Characteristics of Biodiesel Obtained from Sunflower Oil. *Biofuels*. 2015.

Citation: Ajadi IK, Oyegoke T, Abdullahi NB, et al. Influence of esterification and neutralization in the production of biodiesel: a comparison study. *Material Sci & Eng.* 2022;6(2):64–66. DOI: 10.15406/mseij.2022.06.00181