



In-Situ Transesterification of Biodiesel Synthesized from *Ampelocissus Africana* Seed Oil Using (MgO/Al₂O₃) Catalyst

A. Y. Zubairu^{1&2*}, M. Mukhtar¹, I. Saidu², I. A. Khalil¹, I. Garba³

¹Department of Pure and Applied Chemistry, UsmanuDanfodiyo University, P.M.B, 2346, Sokoto, Nigeria

²Department of Science Laboratory Technology, Umaru Ali Shinkafi Polytechnic, P.M.B. 2356, Sokoto, Nigeria

³Department of Mechanical Engineering, Faculty of Engineering, Bayaro University Kano, Nigeria

*Corresponding Author

A. Y. Zubairu

Department of Pure and Applied Chemistry, UsmanuDanfodiyo University, P.M.B, 2346, Sokoto, Nigeria

Article History

Received: 03.08.2021

Accepted: 06.09.2021

Published: 02.10.2022

Abstract: Biodiesel is an unconventional fuel composed of mono-alkyl esters also achieved essentially from the base catalyzed transesterification reaction of oils or fats. It is utilized clean haven't require single alteration in the diesel engine and also biodiesel does not contain in consequential amount of sulphur. Consequently, biodiesel is presently the preeminent alternate for petro diesel. Biodiesel was synthesized using one step trasesterification reaction using magnesium oxide over aluminum oxide (MgO/Al₂O₃) at different ratio 5%, 10% and 15%. Moreover the biodiesel were analyzed using 6890N network GC/MS and physicochemical analysis cared out are compared according to international standard via American Society of Testing Material (ASTMD-6751) the results obtain were: Acid value (0.1, 0.1, 0.2mgKOH/g), Density(0.64, 0.82, 0.71g/cm³) Iodine value (0.70, 0.52, 0.70mgI₂/100g), Saponification value (0.10, 0.28, 0.63mgKOH/g), and Water & Sediment level (0.002, 0.002, 0.002) for sample A, B, and C, more ever the percentage yield obtained were (51%, 50%, 50.5%) for A, B, and C.

Keywords: Ampelocissus Africana Seed; Biodiesel; Insitu-transesterification; Non-edible oils.

Copyright © 2022 The Author(s): This is an open-access article distributed under the terms of the Creative Commons Attribution 4.0 International License (CC BY-NC 4.0) which permits unrestricted use, distribution, and reproduction in any medium for non-commercial use provided the original author and source are credited.

1.0 INTRODUCTION

Rising uncertainty about global energy production and consumption, environmental pollution due to the use of fossil fuels, and the elevated cost of petroleum products are the most important reasons to search for substituent to petro diesel (Yang *et al.*, 2007).

In recent times, world has been affected with an energy catastrophe due to fossil fuel reduction and environmental dreadful conditions. Biodiesel is one of the mainly hopeful substitute fuels to meet up these tribulations. It is renewable, recyclable, non-hazardous, and has nearly secure

property to that of petrodiesel fuel (Kulkarni *et al.*, 2006).

Biodiesel is environmental gracious since it habitually burns 75% clean than conservative fuel complete from fossil fuel. As well it reduces unburned hydrocarbon, carbon monoxide and particulate matter in tire out fumes. Sulphur dioxide emission is removed because it has no Sulphur. Its manufacture is simple and uncomplicated as sources of feed are willingly accessible and renewable not like the fossil diesel (Almustapha *et al.*, 2009).

More effort is place on vegetable oils as well as their derivatives (ethyl or methyl esters) referred

to biodiesels. They are proposed to substitute fossil fuels. They are moreover measured as being renewable and environmental closeness. They do not affect the generally steadiness of CO₂ in the environment (Wanignon, *et al.*, 2011).

Biodiesel be in advance consideration similar to substitute energy and is naturally synthesized from transesterification of vegetable oils, animal fats otherwise worn scorching oils which contain mostly triglycerides. The transesterification can be produce by means of single-hydric (monohydric) alcohols similar to methanol and ethanol by the occurrence of an alkali catalyst. Biodiesel and its blends with petro-leum-based diesel fuel can be used in diesel engines with no some considerable modifications toward the engines (Rushang *et al.*, 2007).

Biofuels generate from vegetable oils can be characterized via the subsequent parameters: viscosity, density, cetane number, cloud and pour points, distillation range, flash point, ash content, sulphur content, carbon residue, acid value, copper corrosion and higher heating value (Wanignon, *et al.*, 2011).

It has been identified to facilitate a substantial figure of biodiesel are synthesized from oils fit for human consumption although the wide employ of oils fit for human consumption is in the direction of several unhelpful impacts for instance hunger and higher cost food within develop countries for those reason there is 70% percent price increased for oils fit for human consumption (Yunus khan *et al.*, 2014).

Various researchers boast out with the aim of rising the technology to ward adapt cellulosic resources to synthesized biofuels this considerably resolve decrease scarcity tribulations of food, besides to this waste edible oil probably prepared as the major raw material and the new edible and non-edible oils be supposed to be prepared as extra raw material. Which will resolve scarcity tribulations of food decrease extensively, on the other hand numerous of the researchers concur to facilitate non-edible oils are the appropriate substitute to edible oils for biodiesel synthesis. Due to this reason, the current attention is to discover non-edible oil as raw material intended for synthesizing biodiesel (Galadima *et al.*, 2009).

Industrial sections with the intention of synthesis biodiesel rely on decontamination steps to make sure the invention of ideal fuel, a provision with the aim of critical intended for biofuel promotion motive behind trade (Gomes *et al.*, 2015).

This is the first report of *Ampelocissus Africana* used as the feed stocks in production of biodiesel, will focus on this research.

1.1 LITERATURE REVIEW

Zi-ZheCai, Yong Wang, Ying-Lai Tengb, Ka-Man Chongb, Jia-Wei Wangb, Jie-Wen Zhangb, De-Po Yanga, (2015) A two-step biodiesel production process from waste cooking oil via recycling crude glycerol esterification catalyzed by alkali catalyst, in the literature two-step biodiesel production methods have been developed: firstly FFAs are esterified catalyzed by acid as a pretreatment, and then the acylglycerols are transesterified using a homogeneous alkali catalyst (typically NaOH) in this prospective research attempt has been made to synthesis biodiesel using a one step process (insitu-transesterification) from seeds of (*Ampelocissus Africana*) using Magnesium oxide over Aluminum oxide (MgO/Al₂O₃) heterogeneous catalyst and the effect of the catalyst in various ratio, the total percentage yield on the final biodiesel (methyl esters) was also considered and characterization of biodiesel properties and physicochemical analysis.

2.0 EXPERIMENTAL

2.1 Materials and analysis methods

Sample of (*Ampelocissus Africana*) was purchased from sokoto state market Nigeria.



2.2 Sample Treatment

The Sample of (*Ampelocissus Africana*) were dry using oven at 35°C at about 24 hours, the dry sample were grinded to powder form using electric blinder.

2.3 Preparation of Catalyst

Catalysts were prepared using magnesium oxide over aluminium oxide (MgO/Al₂O₃) in various ratios (0.5:0.95, 1:9, 1.5:8.5, and 2:8) after agitation of the catalyst one gram (1g) of each ratio was used

in the transesterification of (*Ampelocissus Africana*) (Galadima *et al.*, 2009).

2.4 Transesterification

20g of powdered sample of (*Ampelocissus Africana*) were used in the transesterification, one gram (1g) of catalyst prepared was mix with the 10ml of methanol (MeOH) and 90ml of n-hexane. But extraction and tranesterification was achieved in a single step for about 90 minutes with reaction temperature 65°C via soxh let extractor connected with CA1112Shanghai Eyela co. Ltd refrigerator to regulate the condenser at ambient temperature maintained at 35°C. This method was used for the all ratio of the prepared catalysts.

2.5 Analyses

2.5.1 Water and Sediment Level

After transesterification the biodiesel produce was transferring into centrifuge machine for centrifuging. After the centrifuging the remaining sediment was weight using electrical weight balance which was used for calculation of water and sediment level of biodiesel according to.

2.5.1 Physicochemical Analysis

3.0 RESULTS

Parameters	Sample A	Sample B	Sample C	ASTM-D6571(B100)
Acid value (mgKOH/g)	7.0 ±0.1	4.4±0.1	4.2 ±0.2	0.5 max
Cetane number	1315.45	3684.84	1562.27	47min
Density (g/cm ³)	0.63±0.007	0.83±0.007	0.72±0.007	0.86-0.90
High heating value(MJ/Kg)	49.27	49.36	49.27	
Iodine value (mgI ₂ /100g)	0.68±0.13	0.56±0.04	0.63±0.06	115max
Percentage yield (%)	51	50	50.5	
Saponification value (mgKOH/g)	4.3±0.10	1.5±0.28	3.6±0.63	
Water & Sediment level %(w/w)	0.002	0.002	0.002	0.05max

3.1 DISCUSSION

The percentage yield of biodiesel were found to be 51%, 50% and 50.5% when compare with the result 57.92 % obtain by (Daramola *et al.*, 2015) there are in line with previous study.

Acid value is the number of milligrams of potassium hydroxide required to defuse fatty or rosin acids in 1 gram of sample (S.T.A.M 2013). The acid value ware obtained to be 7.0 ±0.1, 4.4±0.1 and 4.2 ±0.2 for A, B and C the results were not higher than range given by ASTM-D6751 (0.50max).

The acid value results are contradict with the results 0.11, 0.15 and 0.31 for Safflower, Soybean and Used cooking reported by (Mucino *et al.*, 2014).

The physicochemical analyses were carried according stand analytical method reported by surfa tech (2013).

While the high heating value was determined using the following equation reported by Huseyin *et al.*, (2014) present an expression to estimate the high heating value of vegetable oils in terms of saponification value (SV) and iodine value (IV).

$$HHV = 49.43 - (0.041 (SV) + 0.015 (IV))$$

Where

IV = Iodine value

SV = Saponification value

Also the cetane numbers were calculated from equation reported by Sokoto *et al.*, (2011).

$$CN = 46.3 + \frac{5458}{SV} - 0.225 \times IV$$

Where

IV = Iodine value

SV = Saponification value

The Cetane number this used to determine the ignition excellence of diesel fuel. The higher the Cetane number, the effortless the fuel ignites after introduced in the engine. The higher the Cetane number the additional fuel well-organized the fuel. Biodiesel contain a lager Cetane number than petrodiesel due to its large oxygen content (Demirbas 2008).

The cetane number is a basic property, and defined as a measurement of the ignition performance of a fuel. This parameter is influenced by structural features of fatty acid alkyl esters, such as chain length, degree of unsaturation and branching of the chain. It should be emphasized that the higher the cetane number, the better will be the combustion (Sivaramakrishnan *et al.*, 2012).

The cetane numbers were found to be 1315.45, 3684.84 and 1562.27 for A, B, and C. The results were contradicted with the results 49 and 53 for Biodiesel and Diesel obtained by (Qi *et al.*, 2014). The cetane numbers obtained exceed minimum level needed establish by ASTM-D6751 (47min) this indicate that biodiesel produced from *Ampelocissus Africana* seed oil contain long range of degree of unsaturated.

The cetane number were found to be high which indicate that the biodiesel has good ignition properties, fuel with excellent ignition value contain high cetane number, where the ignition interruption period linking the begin of fuel injection and the start of automobile ignition is little (Sivaramakrishnan *et al.*, 2012).

Density, its compute of the mass per unit volume (kg/m³) is a significant fuel factor which is unbiased in the lead of biodiesel oxidation (Zahira *et al.*, 2014).

The density, were obtain to be 0.63±0.007, 0.83±0.007 and 0.72±0.007 for sample A, B, and C. all the density are bellow the range giving by ASTM-D6751 (0.86-0.90), while only the sample B is within the range with a density (0.83±0.007).

The high heating value (HHV), expressed in kcal, represents the amount of heat required to completely burn a mass of 1 kg of solid or liquid fuel. It is the single most important factor that represents the performance of the fuel, the higher the high heating value, the higher the efficiency. Generally, the high heating value of a saturated fatty acid alkyl ester increases with increasing carbon double bonds (Wanignon, *et al.*, 2011).

The high heating value (HHV) for sample A, B, and C were found to be 49.27, 49.36 and 49.27 the results were conform with the result reported by (Hong *et al.*, 2014).

Iodine value determines the amount of unsaturation in fuel and also articulated as the level in mass of iodine engaged by mass of biodiesel. Hence, iodine value rises by increasing number of double bonds. Higher iodine value can signify chemical unsteadiness, because the double bonds are relatively reactive regions of the molecule (Martinez *et al.*, 2014).

Iodine Value obtain to be 0.68±0.13, 0.56±0.04 and 0.63±0.06 for sample A, B and C, the results did not exceed the maximum limit 115max reported by ASTM-D6751.

Saponification value is the quantity of alkali required to saponify a specific quantity of the sample. It is articulated as the amount of potassium hydroxide (KOH) required saponifying (dissolved) 1g of oil (STACM, 2013).

Saponification value was found to be 4.3±0.10, 1.5±0.28 and 3.6±0.63. Water & Sediment level in biodiesel is an important factor in the quality control. Water & Sediment can endorse microbial development, direct to tank corrosion, involve in the development of emulsions, and cause hydrolysis or hydrolytic oxidation. As a result, the content of water and sediments limited to 0.05% (w/w) establish by EN 14214 and ASTM-D6751 standards (Marcos *et al.*, 2008).

The water and sediment were found to be 0.002 for all samples which indicate that there is no effect of corrosion or is very minor, since the water and sediment found were bellow the maximum standard limit reported by ASTM-D6751.

4.0 CONCLUSION

In conclusion one step transesterification or insitu-tranesterification (process) of *Ampelocissus Africana* seeds oil was achieved, the biodiesel generated were characterized using physicochemical analysis indicate the present of fatty acid alkyl esters. Since this method can be utilize in the production of biodiesel, can serve as easier method in the production of biodiesel in biofuels refinery also reduce available step in the production of biodiesel, more ever this method will reduce cost of production, as well as reduce long time of production which are the important factors for commercial purpose if utilize.

4.1 RECOMMENDATIONS

It is recommended that further research has to be done, in order to further test this method using differences non edible seed and to determine Flash Point, Cloud Point, Oxidation Stability, Pour point and Lubricity which are important parameters required by ASTM-D6751(B100) for biodiesel quality.

REFERENCE

- Almustapha, M. N., Hassan, L. G., Ismaila, A., Galadima, A., & Ladan, M. M. (2009), Comparative Analysis of Biodiesel From *Jatropha Curcas* Seed Oil With Conventional Diesel Fuel (A.G.O). *Chemclass Journal*, 6, 43-046.
- American Society of Testing Materials. (2011). ASTM-D6751-11b, Standard.
- AOAC. (2005). Official method of analysis Association of analytical Chemist. Wash. Dc, 15th ed. Pp11-14.

- Association of Official Analytical Chemists. (2010). Official Methods of Analysis of AOAC International. 18th ed. Washington DC: Association of Official Analytical Chemists.
- Belewu, M. A., Adekola, F. A., Adebayo, G. B., Ameen, O. M., Muhammed, N. O., Olaniyan, A. M., Adekola, O. F., & Musa, A. K. (2010). Physico-chemical characteristics of oil and biodiesel from Nigerian and Indian *Jatropha curcas* seeds. *International Journal Biological and Chemical Sciences*, 4(2): 524–529.
- Birnin-Yauri, U. A., & Garba, S., (2011). Comparative Studies on Some Physicochemical Properties of Baobab, Vegetable, Peanut and Palm Oils. *Nigerian Journal of Basic and Applied Science*, 19(1), 64-67. Available online at www.ajol.info/index
- Daramola M. O., Mtshali K., Senokoane L., & Fayemiwo, O. M. (2015). Influence of Operating Variables on the Transesterification of Waste Cooking oil to Biodiesel over Sodium Silicate Catalyst: A statistical approach. *Journal of Taibah University for Science*, 15, 1-26. Available at <http://dx.doi.org/10.1016/j.jtusci.2015.07.008>
- Demirbas, A. (2008). Relationships Derived From Physical Properties of Vegetable Oil and Biodiesel Fuels. *Fuel*, 87, 1743–1748. Available online at www.sciencedirect.com
- Demirbas, A. (2008). Relationship Derived from Physical properties of Vegetable Oil and Biodiesel Fuel. *Fuel*, 87, 1743-1748.
- Galadima, A., Ibrahim, B. M., Umar, K. J., & Garba, Z. N. (2009). Homogeneous and Heterogeneous Methanolysis of cotton seed oil locally produced in northern Nigeria. *Biological and Environmental Science Journal for the Tropics*, 6(3), 71-75.
- Gomes, F. J. C., Peiter, A. S., Pimentel, W. R. O., Soletti, J. I., Carvalho, S. H. V., & Meili, L. Biodiesel Production From *Sterculia Striata* Oil By Ethyl Transesterification Method. *Industrial Crops and Products*. 74, 767–772. Available online at www.sciencedirect.com
- Hong I. K., Jeon G. S., & Lee S. B. (2014). Prediction of Biodiesel Fuel Properties From Fatty Acid Alkyl Ester. *Journal of Industrial and Engineering Chemistry*, 20, 2348–2353. Available at www.elsevier.com/locate/jiec
- Huseyin S., Mustafa C., & Ertan A. (2014). Predicting the Higher Heating Values of Waste Frying Oils as Potential Biodiesel Feedstock. *Fuel*, 115, 850–854.
- Kulkarni, M. G., & Dalai, A. K. (2006), Waste Cooking Oils an Economical Source for Biodiesel, A Review. *Ind Eng Chem Res*, 45, 2901-2913.
- Ma, X. (2012). Biodiesel production from Algae through *In situ*-Transesterification Technology. A Thesis submitted to the faculty of the Graduate School of the University of Minnesota. Pp1-70.
- Marcos R. M., Alessandra R. P. A., Luciano M. L., & Antonio G. F. (2008). Critical Review on Analytical Methods for Biodiesel Characterization. *Talanta*, 77: Pp593–605. Available online at www.elsevier.com/locate/talanta
- Marcos R. M., Alessandra R. P. A., Luciano M. L., & Antonio, G. F. (2008). Critical Review on Analytical Methods For Biodiesel Characterization. *Talanta*, 77, 593–605. Available online at www.elsevier.com/locate/talanta
- Martinez G., Sanchez N., Encinar J. M., & Gonzalez J. F. (2014). Fuel Properties of Biodiesel From Vegetable Oils and Oil Mixtures. Influence of Methyl Esters Distribution. *Biomass and Bioenergy*, 63, 22-32. Available online at www.sciencedirect.com
- Mucino G. G., Romero R., Ramirez A., Martinez S. L., Baeza-Jimenez, R., Reyna, N. (2014). Biodiesel Production from Used Cooking Oil and Sea Sand as Heterogeneous Catalyst. *Fuel*. 07: Pp53. Available online at www.sciencedirect.com
- Mukhtar, M., & Zubairu, A. Y. (2016). Biodiesel Production from Moringa Oleifera Seed Oil Using MgO/Al₂O₃ Catalyst, *International Journal of Science for Global Sustainability*, 2(3), 116-122.
- Mustafa, B., & Havvat, B. (2008): A Critical review of bio-diesel as a vehicular fuel. *Energy Conversion and Management*, 49, 2727-2741. Available online at www.sciencedirect.com
- NNPC, (2007). Official Gazette of the Nigerian Biofuels Policy and Incentives: Renewable Energy. Nigeria National Petroleum Corporation. <http://www.nnpcrd.com/images/policy.pdf> Accessed 08 July 2008.
- Qi, D. H., & Lee, C. F. (2014). Influence of Soybean Biodiesel Content on Basic Properties of Biodiesel-Diesel Blends. *Journal of the Taiwan Institute of Chemical Engineers*, 45, 504–507. Available online at www.elsevier.com/locate/jtice
- Rushang, M. J., & Michael, J. (2007). Flow Properties of Biodiesel Fuel Blends At Low Temperatures. *Fuel*, 86, 143–151. Available at www.sciencedirect.com
- Sivaramakrishnan, K., & Ravikumar, P. (2012). Determination Of Cetane Number Of Biodiesel And It's Influence On Physical Properties. *Journal of Engineering and Applied Sciences*. 7: Pp2. Available online at www.arpnjournals.com
- Sokoto, M. A., Hassan, L. G., Dangoggo, S. M., Ahamad, H. G., & Uba, A. (2011). Influence of Fatty Acid Methyl Esters on Fuel Properties of Biodiesel Production From the Seed Oil of Curcubitapepo. *Nigerian Journal of Basic and Applied Science*. ISSN0794-5698, 19: Pp.

- 81–86. Available online at <http://www.ajol.info/browse-journals>.
- Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels, International, West Conshohocken, USA, Available online at <http://dx.doi.org/10.1520/D6751-11B>.
 - Surfa Technology Corporation Analytical Method. (2013). Method of Analysis of STCAMPp. 3-8. Available at <http://www.surfatech.com/pdfs/SurfaTech%20Analytical%20Method.pdf>
 - Wanignon, F. F., Laurent, V. D. S., Siaka, T., & Eric, M. (2011). What Correlation Is Value Appropriate To Evaluate Biodiesels And Vegetable Oils Higher Heating (HHV)?. *Fuel* 90: Pp3398–3403. Available online at www.elsevier.com/locate/fuel
 - Yang, H. H., Chien, S. M., Lo, M. Y., Lan, J. C. W, Lu, W. C., & Ku, Y. Y. (2007). Effects of biodiesel on emissions of regulated air pollutants and polycyclic aromatic hydrocarbons under engine durability testing. *Atmospheric Environment*, 41, 7232-7240.
 - Yunuskhan, T. M., Atabani, A. E., Irfan, A. B., Ahmad, B., Khayoon, M. S., & Triwahyono, S. (2014). Recent Scenario and Technologies to Utilizenon-Edible Oils for Biodiesel Production. *Renewable and Sustainable Energy*, 37, 840–851. Available online at www.sciencedirect.com
 - Zahira, Y., Binitha, N. N., Silija, P., Surya, U. K., & Mohammed, A. P. (2014). A review on the Oxidation Stability of Biodiesel. *Renewable and Sustainable Energy Reviews*, 35, 136–153. www.elsevier.com/locate/rser
 - Zi-ZheCai, Yong, W., Ying-Lai, T., Ka-Man C., Jia-Wei, W., Jie-Wen, Z., & De-Po, Y. (2015). A two-Step Biodiesel Production Process From Waste Cooking Oil Via Recycling Crude Glycerol Esterification Catalyzed By Alkali Catalyst. *Fuel Processing Technology*, 137: Pp 186–193. Available online at www.sciencedirect.com