Optimisation of Alkaline Ethanolysis of Biodiesel Yield from Nigerian Coconut Oil using One Variable at a Time (OVAT) Approach

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Abstract

In this study, coconut (*Cocos nucifera*) oil has been identified as a feedstock for biodiesel production. The determination of optimal feedstocks ratio (of ethanol/coconut oil, v/v. % ratio) was studied. The reaction was executed at different ethanol/coconut oil ratios: 10%, 15% 20%, 25% and 30% while the reaction time (60 min), reaction temperature (70^{0} C) and 1.0% NaOH catalyst dosage were kept constant. The result indicated that maximum biodiesel yield (96.09%) was obtained at 20% of ethanol/coconut oil vol./vol.% ratio within transesterification reactions that were kept constant. The fuel characterization such as viscosity (4.32mm²/s), specific gravity (0.887), pour point (-18⁰C), cloud point (-12⁰C) and flash point (160⁰C) of the produced biodiesel at the optimized conditioned showed that the suitability of coconut ethyl ester (biodiesel) were within the international biodiesel standard.

Keywords

Coconut oil — Biodiesel — Transesterification — Optimization — Ethanolysis — OVAT

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1. Introduction

Scientific resources can be saved by optimization of experiments [1]. Optimization procedures such as factorial designs, response surface methodology, uniform experimental design, taguchi orthogonal design, one variable at a time and several others are mostly employed [2]. One-variable at a time approach is a method of designing experiments which involve the testing of factor at a time. Its advantages range from effectiveness, independences, and low experimental error [3, 4].

Ever- increasing campaigning for cleaner burning fuels, exhaust gases from petroleum products, threat of supply instability, rising prices and depletion of fossil deposits have sprung global interest in renewable biofuel. Modern biofuel are a promising long term renewable

energy source which has potential to address both environmental impacts and security concerns posed by current dependence on petroleum based fuels [5, 6, 7]. One of such biofuel is biodiesel. Biodiesel is biodegradable, non-toxic and has low emission profiles when compared to fossil fuel and its usage allows balance between agriculture, economic development and the environment. Biodiesel is produced through a chemical process known as transesterification. Transesterification of vegetable oils with low molecular weight simple alcohols (methanol, ethanol, propanol and butanol) has been established as the best option to reduce the high viscosity, low volatility, heavy engine deposits and toxic substance formation associated with the direct use of vegetable oils [8]. Vegetable oil remains the major feedstock for biodiesel production. Animal fat and waste cooking oil have also been used [9, 10]. Soybean (US), rapeseed (Europe), oil palm (South-East Asia) and canola, to mention a few have been successfully used as renewable vegetable oil sources to generate biodiesel with superior qualities over the petroleum- based fuels [11, 12]. Others include known oil plants like soybean, canola, sunflower, safflower, Jatropha curcas, peanut, tiger nut, coconut etc[13, 14]. However, some of these oil sources are commodities whose prices are strongly dependent on the international market. Coconut is a tropical plant and is commonly found in the western part of Nigeria. Researchers have explored feedstocks such as soya bean in Brazil [15, 16] and Jatropha in India [17] for biodiesel production. Few

published works on the production and fuel characterization of biodiesel from coconut oil as alternative fuel have produced encouraging results [18,19,20]. Kumar et al. [21] reported that the ultrasonification process can reduce reaction time in 15-40 times comparing to the conventional process using in coconut ethyl ester production. Effect of ethanol on biodiesel yield has been reported by Alamu et al. [22]. However, optimization of coconut ethyl ester (biodiesel) biodiesel yield using ethanol/oil ratio protocol has not been published in literature. The present work seeks to investigate the optimization of coconut biodiesel yield using ethanol/ oil ratio technique while keeping other transesterification variables constant. Fuel characterization results from the produced coconut oil (CNO) biodiesel at optimum yield through ASTM standard fuel tests are also reported.

2. Materials and Methods

2.1 Laboratory Experiment

Local coconut oil was procured from local stores in Ifo market, Abeokuta, Ogun State, Nigeria. The CNO contains mainly lauric acids. Ethanol (99.8%) used is analytical grade product of Aldrich chemicals Germany; while the KOH used was also an analytical grade product of Aldrich Chemicals (England) and acetic acid were obtained from Uche Scientific Company (Lagos State, Nigeria). The magnetic stirrer used was a product of Germany. Scales, measuring cylinder, separating funnel and thermometers were also used.

2.2 Reaction parameters for ethanolysis process

Reaction parameters for ethanolysis process including reaction temperature, reaction time, catalyst concentration and CNO quantity were held constant at 60°C, 60 min, 1.0% NaOH (wt.% CNO) and 100g CNO, respectively and this has been reported [22, 23].

2.3 Transesterification of coconut oil

The transesterification of CNO was executed in 250L reactor equipped with a reflux condenser, a thermometer and a magnetic stirrer under atmospheric pressure. The reaction was conducted using optimized conditions reported elsewhere [22] since both coconut oil and palm kernel oil were lauric oil [23]. Ten grams of ethanol was measured and poured into a conical flask after which 1.0g of NaOH carefully added and dissolved completely in the ethanol, using the hot plate and magnetic stirrer to form sodium ethoxide. 100 g of CNO was gently measured and pre-heated to 60°C before finally poured into a reactor containing sodium ethoxide. The reactor was secured tightly and connected into reflux condenser in order to prevent ethanol loss. The mixture was maintained for 60 min after which it was poured into a separating funnel for settling. The mixture was left for 24 hours to allow separation by gravitational settling into

an amber yellow liquid biodiesel on top and light brown glycerol at the bottom. Glycerol layer was drained off from the separating funnel leaving only crude biodiesel. The biodiesel was washed as reported [24]. The procedure was replicated two times and average biodiesel yield was recorded. The CNO biodiesel produced at the optimum parameters were subjected to ASTM standard and fuel test. Specific gravity and viscosity measurement were determined following the ASTM standard D1298 and D445 respectively. The biodiesel was analyzed for pour point, cloud point and flash point following ASTM standard D97, D25100-8 and D-56, respectively. As a standard for comparison, similar fuel characterization tests were conducted for petrol-diesel (No.2 Diesel) procured at NNPC, filling station, Olowomore, Ogun State, Nigeria.

2.4 Mixing ratio of ethanol/coconut oil on biodiesel yield

The impact of mixing ratio of ethanol/ coconut oil on coconut biodiesel yield was studied using ethanol as variable feedstock with (10%, 15%, 20%, 25% and 30%). The ethanolysis transesterification was replicated with varied feedstock (ethanol and CNO) proportions keeping all other process parameters mentioned above constant. In all, five treatments were considered using 100.0g of CNO with ethanol quantity varied from 10.00 g to 0.30 g at 5g incremental step. As such, ethanol-CNO ratios 0.1, 0.150, 0.20, 0.25 and 0.30 were studied. In each treatment, two different runs of experiments were undertaken and the average results evaluated.

3. Result and Discussion

In fuel characterization of CNO biodiesel as alternate fuel, the CNO biodiesel produced in each treatment and the petroleum diesel (used as control were analyzed for key fuel properties including viscosity, specific gravity, pour point, cloud point, flash point and acid value. Results are detailed in Table 1. Base catalyzed transesterification during laboratory experiment for varied ethanol-CNO ratio (0.10, 0.150, 0.200, 0.250 and 0.30), were employed with other constant process parameters to produce CNO biodiesel. The amount of NaOH, reaction temperature and reaction time employed with the ethanol variation were held constants at 1.0%, 60°C and 60 mins, respectively. The results of the runs of each of the experiments together with the average values, for each of the ethanol- coconut oil ratios studied are detailed in 1

The detailed results presented in Table 2 showed the amount of CNO biodiesel and glycerol recorded from the transesterification reaction with ethanol- CNO feedstock mix of 0.1 (treatment 1) to 0.30 (treatment 5).

The results indicated that average value of 36.5%, 87.0%, 96.09%, 94.63% and 91.46% CNO biodiesel yield were obtained for ethanol ratio 0.1, 0.150, 0.20, 0.250

| Fuel properties | CNO | Petroleum |
|---|-----------|-----------|
| | biodiesel | diesel |
| Viscosity @ 40° C (mm ² / s) | 4.32 | 3.912 |
| Specific gravity @60 ⁰ F/60 ⁰ F | 0.887 | 0.843 |
| Pour point (⁰ C) | -18 | -13 |
| Cloud point (⁰ C) | -12 | -3 |
| Flash point (⁰ C) | 160 | 68 |
| Acid value | 0.31 | 0.12 |

Table 1. Measured coconut biodiesel and petroleumdiesel fuel properties

Table 2. Results of transesterification for treatmenttypes: 1-5 with varying ethanol/ oil ratio

| Treatment | QEthanol | CNO | Glycerol(g) |
|-----------|----------|------------------|----------------|
| | /QCNO | biodiesel(g) | obtained |
| 1 | 0.100 | 36.50 ± 0.12 | 60.30 ± 0.06 |
| 2 | 0.150 | 87.00 ± 3.26 | 21.40 ± 0.09 |
| 3 | 0.200 | 96.09 ± 0.42 | 6.69 ± 0.61 |
| 4 | 0.250 | 94.63 ± 0.15 | 8.95 ± 0.10 |
| 5 | 0.300 | 91.46 ± 0.39 | 11.92 ± 0.18 |

and 0.30, respectively. In the same order, the average of glycerol formed were 60.30g, 21.40g, 6.69g, 8.95g and 11.92g, respectively. This implied that biodiesel yield increased in direct proportions to the ethanol-CNO ratio increment only up to a threshold mix. Beyond this point, no evidence of increment yield was observed. This clearly shows that the optimum percentage of ethanol (by weight of CNO) needed for ethanolysis of CNO, under the process parameters studied was 20%. Fig. 1 clearly portrays that when the concentration of ethanol was increased or decreased below this value (20%), there was no remarkable increase in the CNO biodiesel yield.

 $Q_{\text{Ethanol}}/Q_{\text{CNO}} = 0.1, 0.150..., 0.3$

Where QEthanol = quantity of ethanol (g); and QCNO = quantity of CNO (g).

The coconut biodiesel yield for the various treatments (1-5) can be mathematical expressed as:

Treatment 1 < Treatment 2 < Treatment 3> Treatment 4 < Treatment 5.



Figure 1. Relationship between quantity of ethanol and coconut biodiesel yield during OVAT

4. Conclusion

The following conclusion can be deduced from the OVAT based optimization of coconut biodiesel yield and the effect of ethanol/coconut oil ratio on its ethyl ester yield:

- Nigerian CNO biodiesel gave futuristic and promising result as alternative diesel fuel with fuel properties in good agreement with previous works and within the limits set by International biodiesel standards.
- For ethanol-CNO ratio 0.1, 0.15, 0.20, 0.25 and 0.30, CNO biodiesel yield of 36.5%, 87%, 96.09%, 94.63% and 91.46% were obtained under typical ethanolysis process condition of 60oC temperature, 60min reaction duration and 1.0% sodium hydroxide (NaOH) dosage.
- An optimum CNO biodiesel yield of 96.09% was obtained with ethanol-CNO ratio 0.2 under typical transesterification reaction condition of 60°C temperature, 60min reaction duration and 1.0% alkali catalyst (NaOH) concentration.

References

- [1] S. ROBERT, F.W. MICHEAL, I.P FESTING, AND F. LARRY (2002). Use of factorial design to optimize animal experiments and reduce animals. ILAR, *Journal-Experimental Design and Statistics in Biomedical Research*, 43(3): 15.
- [2] O.U.DAIRO (2002). In-situ transesterification of castor oil bean seed into biodiesel using a batch processor, Unpublished Ph.D Thesis. *Dept. of Agricultural Engineering, UNAAB: Nigeria.* 1 -204. .
- [3] M FRIEDMAN, L.J. SAVAGE (2002). Planning experiments seeking maxima, In Techniques of Stastistical Analysis, eds. C. Eisenharts, New York: McCraw-Hill, 365-372.
- ^[4] C. DANIEL One- at a- time plans, journal of the *American Statistical Association*, 68, 353-360

- ^[5] P.K. GUPTA, R. KUMAR, B.S. PANESAR, V.K. THA-PAR (2007). Parametric studies on biodiesel prepared from rice bran oil, Agri.*Eng. Int*: CIGR Ejournal, IX, [paper No. EE 06007,].
- ^[6] O.J. ALAMU, M.A. WAHEED AND S.O. JEKAY-INFA(2007) Alkali-catalysed laboratory production and testing of biodiesel fuel from Nigerian palm kernel oil. *Agri. Eng Int.: CIGR Ejournal*; IX [Paper No. EE 07 009].
- [7] B. BATIDZIRAI, A.P.C. FAAIJ AND E(2006) Smeets Biomass and bioenergy supply from Mozambique.*Energy Sustain Develop*. X (1), 54–81.
- [8] A.W. SCHWAB, M.O. BAGHY AND B. FREEDMAN (1987).Preparation and properties of diesel fuel from vegetable oils, Fuel, (66), 372–1378.
- [9] Y. LIN, C. WU AND Y.G. CHANG (2007).Combustion characteristics of waste oil produced biodiesel/diesel fuel blend, *Fuel*, 86(1), 772–80.
- [10] S. MURILLO, J.L. MIGUEZ, AND J. PORTEIRO (2007). Performance and exhaust emissions in the use of biodiesel in outboard diesel engines, *Fuel*,(86), 1765–71.
- [11] C.L. PETERSON, R.O. CRUZ, L. PERKINGS, R. KO-RUS, AND D.L. AULD (1990). Transesterification of vegetable oil for use as diesel fuel: a progress report, *ASAE*, Paper No. 90-610.
- [12] R. YUNUS, A. FAKHRU'LRAZI, T.L. OOI, AND S.E. IYUKE (2003). A Development of optimum synthesis method for transesterification of palm oil methyl esters and trimethylolpropane to environmentally acceptable palm oil-based lubricant, J *Oil-palm Res*, 15 (2), 35–41.
- U. SCHUCHARDT, R. SERCHELI, R.M.VARGAS (1998). Transesterification of Vegetable oils: A review. J. Braz. Chem. Soc., 9(1), 199-210
- [14] G. KNOTHE, J.H. VAN GERPEN, J. KRAHL, (2005). The biodiesel handbook, *Amer. Oil Chem.* Soc. Press. Champaign, IL, USA.
- [15] H. NOUREDDINI, D. ZHU (1997). Kinetics of transesterification of soybean oil, *J Am Oil Chem Soc*, 74(11), 1457–63.
- [16] P. CHITRA, P. VENKATACHALAM, A. SAMPATHRA-JAN (2005). Optimisation of experimental conditions for biodiesel production from alkali-catalysed transesterification of Jatropha curcus oil, *Energy Sustain Develop.*, IX (3), 13–18.
- [17] P.A. AWALUDDIN, S. WAHYUNINGSHI (2010). Making biodiesel from coconut oil by reaction methanolysis heterogeneous catalyst, *In Proc. Energy and sustainable Development: Issues and strategies (ESD)*, Chiang Mai, Thailand.

- [18] A.U. OFOEFULE, C.N. IBETO, I.E. UGWUAMOKE (2013). Determination of optimum catalyst concentration for biodiesel yield from coconut (*Cocos nucifera*) *Oil, International Research Journal of Pure & Applied Chemistry* 3(4), 357-365.
- [19] M.A. HOSSAIN, S.M. CHOWDHURY, Y. REKHU, K.S. FARAZ, M.U. (2012). Islam, Biodiesel from coconut oil: A renewable alternative fuel for Diesel Engine, World Academy of Science, Engineering and Technology, (68), 1289-1293.
- [20] D. KUMAR, G. KUMAR, POONAM, C.P. SINGH, (2010). Fast, easy ethanolysis of coconut Oil for biodiesel production assisted by ultrasonication, *Sonochemistry*, (17), 555-559.
- [21] O.J. ALAMU, M.A. WAHEED, S.O. JEKAYINFA, (2008). Effect of ethanol-palm kernel oil ratio on alkalicatalyzed biodiesel yield, *Fuel*, (87), 1529-1533.
- [22] O.D. SAMUEL, M.A. WAHEED, B.O. BOLAJI, O.U. (2014). Dairo, Synthesis of biodiesel from Nigerian waste restaurant cooking oil: Effect of KOH concentration on Yield, *Global Journal of Sc., Energy and Technology*,15,1-8.
- [23] O.D. SAMUEL (2006). Palm kernel oil Mills: Problems and Solution, M.Eng. Thesis, University of Ilorin, Nigeria.
- [24] O.D. SAMUEL, M.A. WAHEED, B.O. BOLAJI, O.U. (2013). Dairo, Production of biodiesel from Nigerian restaurant waste cooking oil using blender, *Int. Journal of renewable Energy Research*, (14), 975- 979.