



Novel Calcined Activated Anthill as Catalyst for the Synthesis of Environmentally friendly Fuel (Biodiesel)

Nwodi, N.F., Ikoru, M.A., Vwioko, I.R., Umuerrri, A.K., Umukoro, B., Ogbimi, E.V., Adepoju, T. F^{2*}

¹Department of Architecture, Faculty of Environmental Sciences, Southern Delta University, Ozoro, Delta State, Nigeria

²Chemical Engineering Department, Southern Delta University, Ozoro, Delta State, Nigeria

*Chemical Engineering Department Southern Delta University, Ozoro, Delta State, Nigeria

Date of Submission: 07-05-2025

Date of Acceptance: 17-05-2025

Abstract

In an attempt to solve the problem of global warming and the waste disposal problem due to agricultural wastes, this study employed waste cooking oil (WCO) for the synthesis of biodiesel using a novel catalyst derived from anthill. The properties of the oil as well as the quality of the biodiesel were evaluated and compared with international standards.

Observations on the results showed that the WCO has some fuel properties with high pour (17.53°C) and flash points (215.00°C). The viscosity (12.30 Cst) of the oil ensures efficient oil flow, proper sealing, and effective protection against wear and tear. The calcined activated anthill (CAA) was found to be rich in calcium with 43.60 %, iron with 28.70%, potassium with 17.10%, silicon with 10.12%, and manganese with 0.2%. These are the main components of CAA. Biodiesel yield was found to be maximum at run 4 with 92.64% (wt./wt.) at a reaction temperature of 75 min., a CAA weight of 3.5 g, and a methanol-to-oil molar ratio of 6:1. The produced biodiesel qualities were found to be in agreement with international standards ASTM D6751 and EN 14214.

The study concluded that WCO is a good raw material for the synthesis of a cost-effective, environmentally friendly biodiesel for use in internal combustion engines.

Keywords: Waste cooking oil (WCO); Anthill; Calcined; Activation; Biodiesel; Physicochemical properties; Internal combustion engine

I. Introduction

As human existence increases and the growth rate of plants and animals increases, the industrial revolution also increases, hereby causing the energy scarcity and the increasing cause of

global warming. It is no longer a hidden fact that the depletion of the ozone layer is sporadically increasing, and the causes have not ceased to exist (Barbosa et al., 2022). One major cause of this environmental danger is the release of hazardous chemicals from the use of energy in terms of fuel. Fuel is a liquid substance derived from underground decayed molten debris that has existed long before now. The products and by-products are the source of income for many countries such as Qatar, Nigeria, Brazil, etc. (Adepoju et al., 2018). But this fuel has been identified as the source of the major cause of global warming, causing more death to human nature as well as plants and animals. Hence, there is an urgent need to replace conventional fuel with alternative fuel derived from biomass wastes such as vegetable oils, algae, fat/pork lard, etc. (Eyibio et al., 2023). Nevertheless, waste cooking oil generated from the household has been identified as a major cause of environmental problems owing to the disposal problem. This oil, when harnessed as the raw material for biodiesel synthesis, will not only solve the disposal problem but also solve the negative effect of global warming (Akhavue et al., 2023).

Biodiesel can be obtained by the reaction of triglyceride (oil) with alcohol in the presence of a base catalyst (NaOH/KOH) (Mabel et al., 2023). The uses of bio-base derived from solid agricultural wastes have been identified to have various advantages over the NaOH and KOH salts. Such advantages include ease of recovery, low cost of production, environmental friendliness, and increased biodiesel yield (Onwuachi-Iheagwara et al., 2024; Ozioko et al., 2024).

An anthill is a mound of soil, leaves, and other debris built by ants as they create their nests underground (Fig. 1). The specific elemental composition of anthills can vary depending on the surrounding environment but generally includes



elements like silicon, aluminum, iron, calcium, and potassium. Ant hills are mainly composed of soil, sand, clay, and organic matter collected by the ants,

with a high proportion of silica, alumina, and iron oxide.



Fig. 1: Ant-hill

Hence, this study employed waste cooking oil (WCO) for the synthesis of biodiesel. The catalyst was activated from waste anthill and was characterized using SEM and XRD before being used. The properties of biodiesel were examined and compared with the biodiesel international recommended standard.

II. Resources and Procedures

2.1 Resources

Waste cooking oil (WCO) was collected from an eatery located at Eket, in Akwa Ibom, Nigeria. The collected WCO was preheated and filtered with a sieve to remove the dirt associated with long storage and exposure to open air. The filtered oil was kept in a tight 5 L container for further processing.

Ant-hill was collected from nearby land within the school premises (Akwa Ibom State University, Nigeria). The collected anthill was blended with an industrial manual to a small particle size and was made into a smaller particle size with a mesh sieve (20 μm). The sieved sample was kept in a crucible for further processing.

2.2 Procedures

2.2.1 Physicochemical properties of the oil

The refined oil obtained from WCO was used to determine the properties of the oil before biodiesel production. The procedures can be found in what

was earlier reported by Adepoju and Eyibio (2016) with little modification.

2.2.3 Catalyst activation and characterization

The sieved anthill sample was activated by calcining it in a furnace at 450°C for 4 h. The calcined activated anthill (CAA) was then allowed to stand for 24 h to allow proper cooling. The cooled CAA was then characterized using SEM and an XRD analyzer.

2.2.3 Quality of biodiesel

The quality of biodiesel was examined through its properties, and these properties were compared with the biodiesel recommended standards ASTM D6751 and EN 14214.

III. Results and Discussions

3.1 Physicochemical properties of WCO

Table 1 displays the properties of the WCO. Physical characteristics of the oil that can be used as raw materials for the production of biodiesel include its moisture content, specific gravity, viscosity, and refractive index. All of these characteristics were found to be present in moderate amounts. The oil's viscosity (14.20 Cst.) demonstrated that it has a low flow resistance and will not congeal if left at room temperature for a period of time. The oil is of low acid, edible, and requires direct transesterification ($\text{FFA} < 1.5$) with activated catalyst, as indicated by its acid value (1.80 mg KOH/g oil), which indicates an FFA of 0.90%. The oil's tendency to form soap



during production was indicated by the saponification value; the higher value (108.900 mg KOH/g oil) found in this study suggested that the WCO has the potential to form soap if improperly treated at high temperatures in a CAA medium.

Table 1: Properties of the WCO

| Properties | Blended oil |
|--|--------------|
| Colour | Brownish red |
| Moisture content (%) | 0.001 |
| Specific gravity | 0.890 |
| Viscosity @ 40 °C (Cst) | 12.30 |
| Acid value (mg KOH/g oil) | 1.800 |
| %FFA (as oleic acid) | 0.900 |
| Iodine value (g I ₂ /100g oil) | 11.510 |
| Saponification value (mg KOH/g oil) | 108.900 |
| Peroxide value (meq. O ₂ /kg oil) | 5.620 |
| Pour point (°C) | 17.530 |
| Flash point (°C) | 215.00 |

The oil's pour and flash points, expressed in temperature units, provide information about its

energy capacity when used as a raw material for biodiesel production (Mandari and Davari, 2021). Consequently, WCO turned out to be a good option for biodiesel production.

3.2 SEM analysis of calcined anthill (CAA)

The morphological features, size, shape, texture, and other attributes of the CAA analysis are displayed in Fig. 2. The white appearance of the analysis demonstrated that the CAA had entirely changed into highly activated carbon ash. The rigid-pour gaps that were observed were caused by the anthill's tiny size when it was converted to CAA; this observation can be due to an interface that was present during the interfacial reaction of gaseous evolution during heat treatment. The fractured structure at 10,000x magnification was found to be caused by thermal furnace heat treatment; anthill totally disintegrates to produce its heterogeneous atoms, which give it its porous void sizes and shapes. The color showed that during the furnace thermal heat treatment, ash was produced at a high temperature, causing the CAA to achieve the base contents required for biodiesel synthesis.

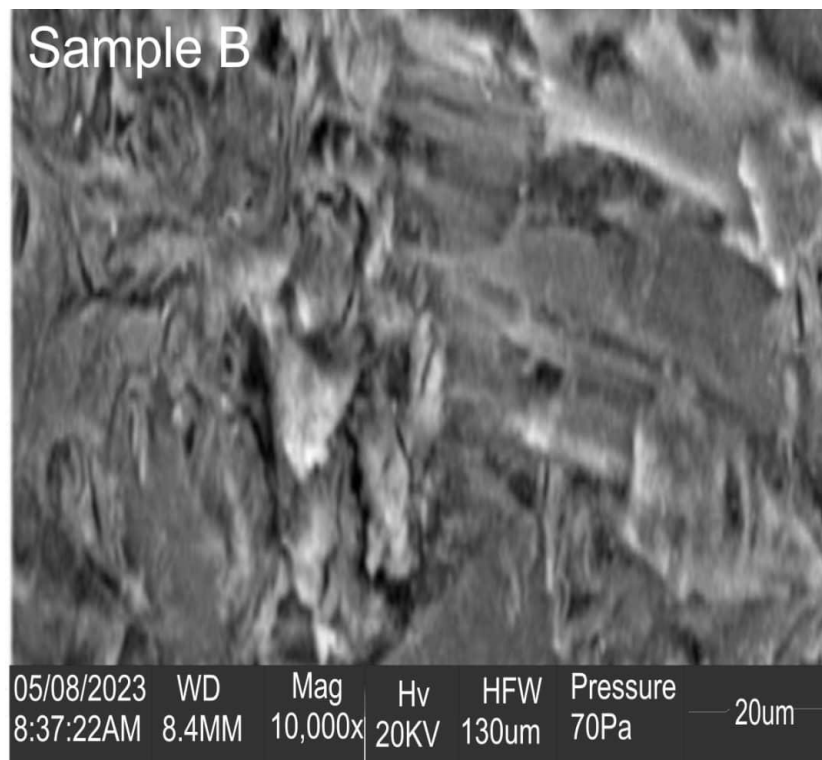


Fig. 2: SEM analysis of CAA



3.3 XRD analysis of calcined activated anthill (CAA)

The XRD analysis of the CAA, which was performed to look at the elemental compositions inside the CAA, is shown in Fig. 3. The figure's observations show that calcium with 43.60 %, iron with 28.70%, potassium with 17.10%, silicon with

10.12%, and manganese with 0.2% are the main components of CAA. These are the compositions of heterogeneous catalysts. It's crucial to keep in mind that CAA is an exfoliant, which absorbed the water content of the WCO during biodiesel synthesis to avoid avert formation.

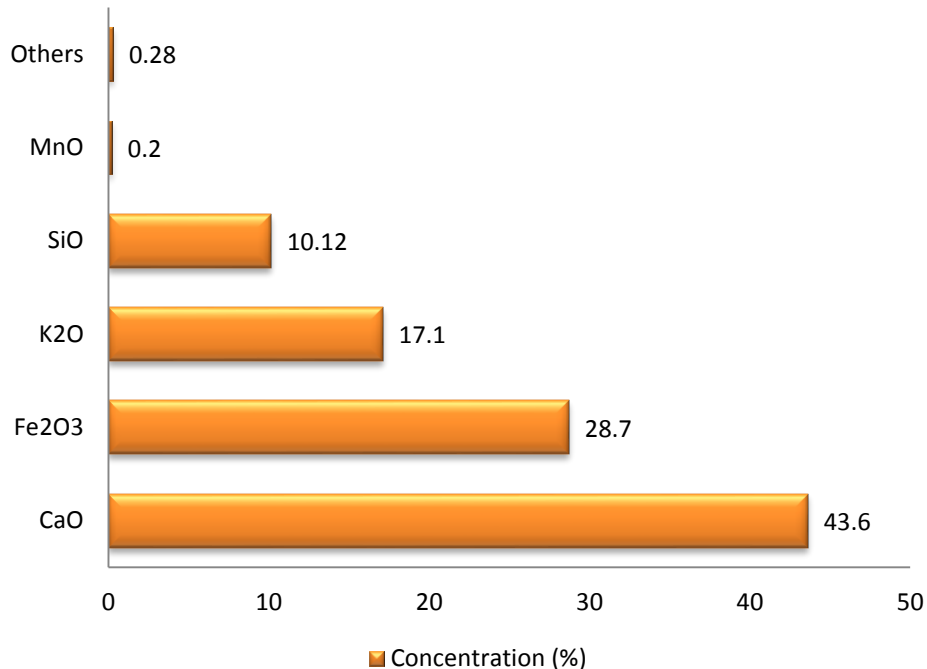


Fig. 3: Percentage concentration of elemental compound in CAA

3.4 Biodiesel synthesis

Presented in Table 2 were the results of biodiesel yield obtained at various reaction times, CAA weights, and methanol-to-oil molar ratios. The reaction temperature was kept constant at 55°C. The yield of the biodiesel indicated that all variables contributed positively to the high yield observed in

the output. It was observed that the highest biodiesel produced was at run 4 with 92.64% (wt./wt.), while the lowest biodiesel yield of 88.64% (wt./wt.) was reported at run 1. This report supported what was earlier reported by Nyorere et al. (2024), that twice the molar ratio of alcohol produced a higher yield of biodiesel based on stoichiometric ratio.

Table 2: The variables and the biodiesel yields

| Runs | Reaction time (min) | CAA weight (g) | Methanol-oil molar ratio (vol./vol.) | Biodiesel yield |
|------|---------------------|----------------|--------------------------------------|-----------------|
| 1 | 60 | 2.0 | 3 | 88.64 |
| 2 | 65 | 2.5 | 4 | 90.12 |
| 3 | 70 | 3.0 | 5 | 90.74 |
| 4 | 75 | 3.5 | 6 | 92.64 |
| 5 | 80 | 4.0 | 7 | 91.20 |

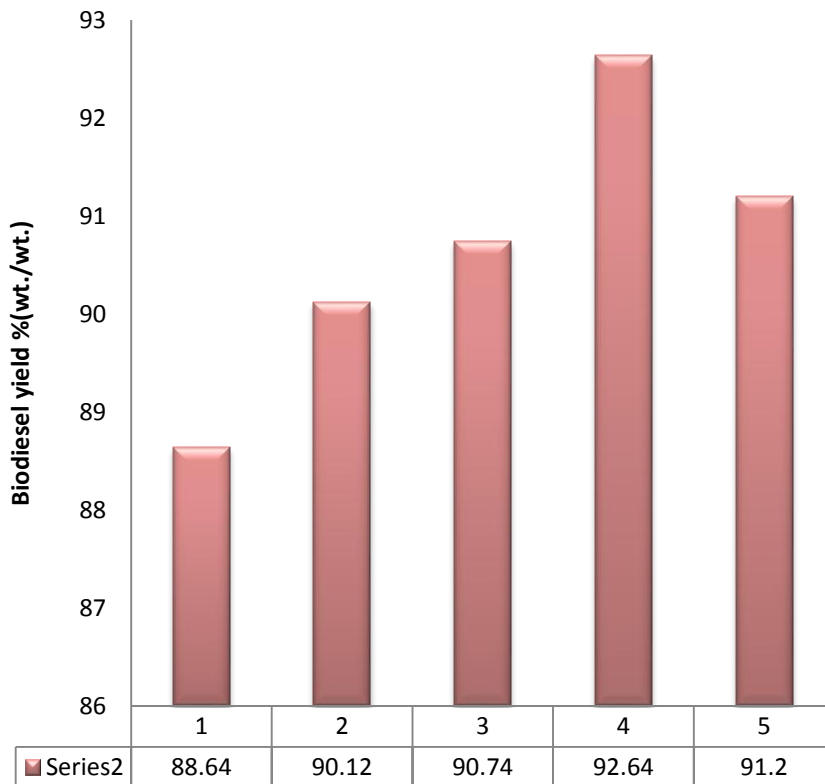


Fig. 4: Plot of biodiesel yield Vs. Runs

3.5 Quality of Biodiesel Synthesis

Table 4 displays the quality of biodiesel properties and a comparison with the international standard (ASTM & EN). It was obvious that the produced biodiesel qualities are in line with what

was recommended by international standards. Hence, the produced biodiesel from WCO may be used as an alternative environmentally friendly fuel in internal combustion engines.

Table 10: Properties of biodiesel as compared with recommended standard

| Parameters | BOB | ASTM D6751 | EN 14214 |
|--|--------|------------|----------|
| Specific gravity | 0.865 | - | 860-900 |
| Viscosity @ 25 °C/ (mm ² /s) | 2.80 | 1.9-6.0 | 3.5-5.0 |
| Moisture content (%) | <0.001 | <0.03 | 0.02 |
| %FFA (as oleic acid) | 0.34 | 0.40 max | 0.25 max |
| Acid value (mg KOH/g oil) | 0.68 | 0.80 max | 0.50 max |
| Iodine value (g I ₂ /100g oil) | 56.20 | - | 120 max |
| Saponification value (mg KOH/g oil) | 102 | - | - |
| Peroxide value (meq. O ₂ /kg oil) | 4.87 | - | 12.85 |
| HHV (MJ/kg) | 44.41 | - | - |
| Cetane number | 87.17 | 57 min | 51 min |
| API | 32.08 | 39.95 max | - |
| Diesel index | 106.14 | 50.4 min | - |
| Cloud point (°C) | +18 | 9 min | - |
| Flash point (°C) | 114 | 50 min | - |



Pour point

-10

-2

-28

IV. Conclusion

In this study, WCO has been successfully converted to biodiesel through the activated anthill. The properties of the oil showed that the oil has a low acid value and can be easily converted to biodiesel in a single-step approach. The activated catalyst was found to be rich in bio-base. The yields of biodiesel were found to be at maximum at twice the methanol-oil molar ratio. The qualities of biodiesel were found to be in line with the biodiesel recommended standard.

Declarations

Ethics approval and consent to participate

Not Applicable

Consent for publication

All authors consented and agreed to take part in this study as research participant.

Competing interests

Authors declares no competing interests whatsoever

Funding

This research receive no funds either from private or government organizations

References

- [1]. Barbosa, S.L., Rocha, A.C.P., Nelson, D.L., de Freitas, M.S., Mestre, A.A.P.F., Klein, S.I., Clososki, G.C., Caires, F.J., Flumignan, D.L., Dos Santos, L.K., Wentz, A.P., Pasa, V.M.D., Rios, R.D.F. (2022). Catalytic Transformation of Triglycerides to Biodiesel with SiO₂-SO₃H and Quaternary Ammonium Salts in Toluene or DMSO. *Molecules*. 2022 Jan 30;27(3):953. doi: 10.3390/molecules27030953. PMID: 35164218; PMCID: PMC8840453.
- [2]. Adepoju T. F., Olatunbosun B. E., Olatunji O. M., Ibeh. M. A. (2018). Brette Pearl Spar Mable (BPSM): A potential recoverable catalyst for renewable source of biodiesel from *Thevetia peruviana* seed oil for sustainable development in West Africa. *Energy, Sustainability and Society*, 8(3): 1-17. DOI: 10.1186/s13705-018-0164-1.
- [3]. Eyibio, U. P., Ukanwa, K. S., Amabogba, E., Adepoju, T. F., Adebayo, A. D., Balogun, T. A., Eloka-eboka, A. C. (2022). Biodiesel synthesized from the blend of *Thai Red* and *Elaeis guineensis* oil: an application of Calcined Base, Optimization, Kinetics, and Thermodynamic Parameters Studies. *Heliyon*. <https://doi.org/10.1016/j.heliyon.2022.e12608>.
- [4]. Akhabue., E.R., Eyibio, U. P., Ukanwa, K. S., Adepoju, T. F., Amabogba, E. (2023). Osamilite (K-Na-Ca-Mg-Fe-Al-S): A derived base catalyst for the synthesis of biodiesel from blends of pumpkin seed oil-goat fat-poultry waste fat. 7(2023)100347. *Case studies in chemical and Environmental Engineering*
- [5]. Mabel Keki., Rockson-Itiveh David Emoefe., Ozioko Fabian Chidiebere., Adepoju Tunde Folorunsho (2023). Hetero-Alkali Catalyst for Production of Biodiesel from Domesticated Waste. *ABUAD International Journal of Natural and Applied Sciences*. 3(2), 16-24 . DOI: 10.53982/aijnas.2023.0302.02-j
- [6]. Onwuachi-Iheagwara., P. N., Kperegbeji, J. I., Ekanem, U., Nwadiolu, R., Okolotu, G. I., Balogun, T. A., Adepoju, T. F., Oboreh, J. S. (2024). Bio-adsorbent of *Jatropha curcus* oil in sugar cane bagasse ash for the synthesis of biodiesel catalyzed by calcined Sartaj maize stalk powder (CSMSP). *Case Studies in Chemical and Environmental Engineering* 10 (2024) 100879. <https://doi.org/10.1016/j.cscee.2024.100879>
- [7]. Ozioko, F.C. Onwuachi-Iheagwara, P.N. Cyril, A. Mabel, K. Nwadiolu, R. Oboreh, J.C. Adepoju, T.F. Oboreh. J.S. (2024). Synthesis of biofuel from *Luffas cylindrical-Dennettiatripetala* oil blend (BT40) using catalytic sweet corn stock acidified with iron (III) sulfate (Fe₂(SO₄)₃). *South African Journal of Chemical Engineering*, 49, 2024, Pages 200-209, ISSN 1026-9185, <https://doi.org/10.1016/j.sajce.2024.05.003>.
- [8]. Adepoju T. F. and Eyibio, U. P. (2016): Comparative study of Response Surface Methodology (RSM) and Artificial Neural Network (ANN) on Oil Extraction from *Citrus sinensis* oilseed and Its Quality Characterization. *Chemistry Research Journal*, 1(5):37-50. ISSN: 2455-8990
- [9]. Mandari, V. and Devari, S. K. (2021). Biodiesel production using homogeneous, heterogeneous, and enzyme catalysts via transesterification and esterification reactions: A critical review. *BioEnergy*, 15:935-961. <https://doi.org/10.1007/s12155-021-10333>



- [10]. Nyorere, O., Oluka, S.I., Onoji, S.E., Nwadiolu, R., Adepoju. T. F. (2024). Production of Biodiesel from Biocatalysis of Agro-wastes in Acidic Environment, *Scientific African*, 2024, e02154. ISSN. 2468-2276.
<https://doi.org/10.1016/j.sciaf.2024.e02154>.