

Coconut-Shell Activated Carbon-Supported CaO for Biodiesel Production from Waste Cooking Oil: Catalyst Preparation and Process-Parameter Screening

¹Abubakar Usman and ^{2,3}Jiya Mohammed

¹Department of Chemistry, Federal University Lokoja, Lokoja, Kogi State, Nigeria.

²Department of Plant Biology, Federal University of Technology, Minna, Niger State, Nigeria.

³Department of Forestry, Ministry of Environment, Ilorin, Kwara State, Nigeria.

*Corresponding authors' email: mjiya0811308016@gmail.com

ABSTRACT

Valorising waste cooking oil and agricultural residues provides a practical route to integrate low-cost biodiesel production with the circular use of biomass resources. In this study, we produced activated carbon (AC) from waste coconut shell and used it as a support for a calcium-based heterogeneous catalyst prepared by wet impregnation with calcium nitrate, followed by thermal treatment. We evaluated the resulting CaO/AC material for transesterification of pretreated waste cooking oil with methanol at 60 °C. A one-factor-at-a-time screening examined nominal oil:methanol ratios of 1:4, 1:8, and 1:16; catalyst loadings of 0.05, 0.10, and 0.20 g; and reaction times of 30, 60, and 120 min. We used FTIR and atomic absorption spectroscopy (AAS) to characterise the prepared catalyst. FTIR bands were consistent with oxygen-containing surface groups and calcium-containing species, while AAS confirmed calcium in the supported material. The highest gravimetric yield of recovered biodiesel-rich product was approximately 90%, obtained at an oil: methanol ratio of 1:16, a catalyst loading of 0.10 g, and a reaction time of 60 min. Under the selected conditions, CaO/AC substantially outperformed activated carbon alone (approximately 90% versus 32%). These findings support the feasibility of coconut-shell-derived activated carbon as a low-cost support for calcium-based transesterification catalysts. Because the source dataset lacked replicate variability, XRD/SEM-EDX confirmation, FAME quantification, and standardised fuel-property data, the results are interpreted as process screening rather than definitive statistical optimisation or proof of fuel-standard compliance.

Received: 11 Oct. 2026

Accepted: 25 Oct. 2026

Published: 27 Oct. 2026

Keywords:

Biodiesel, Waste Cooking Oil, Calcium Oxide, Activated Carbon, Coconut Shell, Heterogeneous Catalyst, Transesterification

INTRODUCTION

Biodiesel consists primarily of fatty-acid alkyl esters produced by transesterification of triglyceride-rich feedstocks with a short-chain alcohol, most commonly methanol. Its environmental and economic performance depends strongly on the feedstock, catalyst, alcohol demand, separation requirements and product quality. Waste cooking oil (WCO) is attractive because it is inexpensive, does not directly compete with edible oils and can otherwise become an environmental burden when poorly managed (Lam et al., 2010; Singh et al., 2021; Monika et al., 2023; Rocha Meneses et al., 2023).

Homogeneous alkali catalysts are highly active for refined, low-acidity oils, but they can generate soap, complicate catalyst recovery, and increase washing and wastewater requirements. Solid heterogeneous bases can simplify separation and may enable catalyst reuse. Among these materials, CaO has received sustained attention because it is inexpensive, strongly basic and effective for transesterification under comparatively mild conditions. However, free fatty acids, moisture, atmospheric carbonation, leaching and particle agglomeration can reduce its practical performance (Kouzu et al., 2008; Lam et al., 2010).

Immobilising calcium-containing active phases on porous supports is one strategy for improving accessibility and handling. Activated carbon derived from agricultural residues is particularly attractive because it combines high porosity with the valorisation of a second waste stream. Coconut-shell

activated carbon has already been used to support alkaline catalysts for biodiesel production, and related high-impact studies have shown that calcium-containing species dispersed within carbonaceous matrices can achieve high transesterification activity when pore structure and active-site accessibility are well controlled (Buasri et al., 2012; Sun et al., 2021; Ibrahim et al., 2022). Recent reviews likewise identify activated-carbon and biochar-supported catalysts as a promising direction for more sustainable biodiesel catalysis (Abo El-Khair et al., 2026).

This study aims to prepare and characterise coconut-shell-derived activated carbon-supported calcium-based catalysts and evaluate the effects of selected transesterification parameters on recovered biodiesel yield from waste cooking oil. The study uses sequential one-factor-at-a-time screening rather than factorial or response-surface optimisation.

MATERIALS AND METHODS

Materials

The study obtained waste cooking oil from a local snack vendor. Waste coconut shells collected in Lokoja, Kogi State, Nigeria served as the carbon precursor. We used methanol, calcium nitrate hydrate, sodium chloride, and distilled water during catalyst preparation and transesterification.

Preparation of Coconut-Shell Activated Carbon

Coconut shells were washed with distilled water, cleaned of extraneous matter and cut into smaller pieces. The material

was air-dried for 3 days and then oven-dried at 120 °C for 4 h. After size reduction, the precursor was treated with NaCl at a coconut-shell: NaCl mass ratio of 4:1 (120 g coconut shell to 30 g NaCl). The mixture was stirred and carbonised at 600 °C for 30 min. The carbonised material was washed three times with distilled water, oven-dried at 80 °C for 4 h, ground and sieved through an 850 µm mesh. After processing, we recovered approximately 30 g of powdered activated carbon. Preparation of CaO/activated-carbon catalyst

The supported catalyst was prepared by wet impregnation. Approximately 0.33 g of calcium nitrate hydrate was

dissolved in 20 mL of distilled water. Activated carbon (8 g) was placed in a beaker, and the calcium nitrate solution was gradually added with continuous stirring at 60 °C for 1 h. The resulting slurry was oven-dried at 120 °C for 24 h, ground and calcined at 600 °C for 2 h under static air. The final calcium-containing supported material is denoted CaO/AC for convenience; its crystalline CaO phase was not independently confirmed by X-ray diffraction. Figure 1 summarises the catalyst preparation and transesterification workflow.

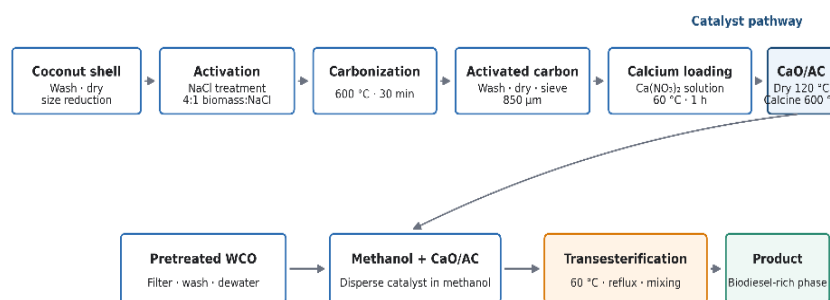


Figure 1: Experimental workflow for coconut-shell activated-carbon preparation, calcium loading and waste-cooking-oil transesterification. The diagram summarises the procedure used in this study; it is not a mechanistic scheme

Pretreatment of Waste Cooking Oil

The collected WCO was filtered to remove food particles and suspended solids, then allowed to settle. It was washed three to four times with distilled water until the wash water appeared clear. The aqueous phase was removed using a separating funnel. The oil was then heated to approximately 100 °C to remove residual water, cooled and stored in a closed container to minimise moisture uptake.

Transesterification and Process-Parameter Screening

Transesterification was conducted under reflux at 60 °C. We first dispersed CaO/AC in methanol under continuous stirring, then gradually added the resulting suspension to preheated WCO. After the specified reaction period, we centrifuged the mixture to separate the solid catalyst from the liquid phases, then recovered the biodiesel-rich phase. The report shows nominal oil: methanol ratios of 1:4, 1:8, and 1:16; catalyst loadings of 0.05, 0.10, and 0.20 g; and reaction times of 30, 60, and 120 min. The available experimental record does not specify whether the nominal oil:methanol ratios are mass-, volume- or mole-based. Catalyst loadings are therefore reported as measured masses because the initial mass of oil was not documented consistently for every screening run. Recovered product yield (%) = (mass of recovered biodiesel-rich phase / initial mass of oil) × 100. This gravimetric measure does not establish the fatty acid methyl ester (FAME) content or conversion.

Catalyst Characterization

FTIR spectra were recorded over approximately 400–4000 cm⁻¹ to identify surface functional groups and calcium-

associated bands. Atomic absorption spectroscopy was used to detect and estimate calcium in the supported catalyst.

RESULTS AND DISCUSSION

FTIR and AAS Characterization

The FTIR spectrum of CaO/AC contained a band near 1416 cm⁻¹ tentatively associated with Ca–O–C/carbonate-related vibrations, a feature near 872 cm⁻¹ associated with calcium-containing oxygen species, and a lower-wavenumber band near 552 cm⁻¹ attributed to Ca–O vibration. The activated-carbon spectrum also showed a broad O–H-associated feature around 3220 cm⁻¹ and a band near 1565 cm⁻¹ consistent with oxygenated/aromatic carbon surface functionality.

AAS detected calcium in the CaO/AC sample. The laboratory record reports a calculated value of 19.54 against a target value of 20, but the reporting basis and units are not defined clearly enough in the available record for rigorous quantitative interpretation. Accordingly, we use the AAS result here only as supporting evidence for calcium incorporation, not as a definitive loading measurement.

Process-Parameter Screening

Increasing the nominal oil: methanol ratio from 1:4 to 1:16 increased recovered biodiesel yield from 65.9% to 90%, with little difference between 1:4 and 1:8. At the selected 1:16 ratio, catalyst loading showed a maximum within the tested range: yield increased from 78% at 0.05 g to 90% at 0.10 g and then decreased to 64% at 0.20 g. Reaction time showed an even stronger response, with yields of 37%, 90% and 20% at 30, 60 and 120 min, respectively. Figure 2 summarises the screening trends.

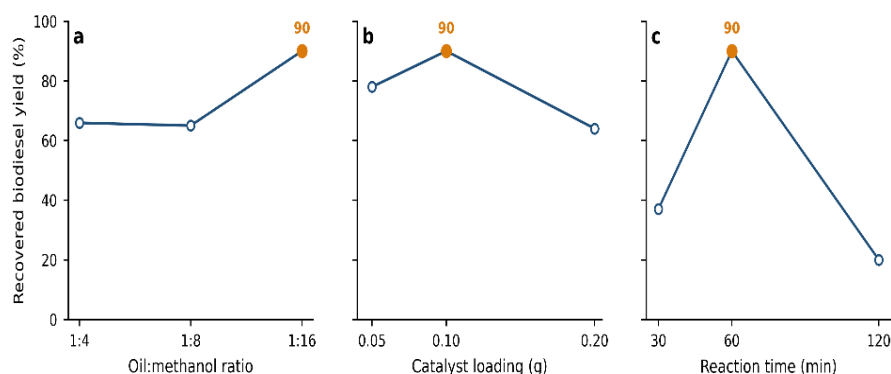


Figure 2: Effect of the screened process variables on recovered biodiesel yield: (a) nominal oil:methanol ratio, (b) CaO/AC catalyst loading, and (c) reaction time. The highlighted point marks the highest yield within each tested range. Values are single measurements from the available experimental dataset; error bars are therefore not shown

Table 1: Summary of the Process-Screening Dataset used in this Manuscript

Screening variable	Level	Fixed conditions	Recovered yield (%)
Oil: methanol ratio	1:4	0.10 g; 60 min	65.9 ^a
Oil: methanol ratio	1:8	0.10 g; 60 min	65
Oil: methanol ratio	1:16	0.10 g; 60 min	90
Catalyst loading	0.05 g	1:16; 60 min	78
Catalyst loading	0.10 g	1:16; 60 min	90
Catalyst loading	0.20 g	1:16; 60 min	64
Reaction time	30 min	1:16; 0.10 g	37
Reaction time	60 min	1:16; 0.10 g	90
Reaction time	120 min	1:16; 0.10 g	20

Note: ^aRecalculated from the reported masses (5.4 g recovered biodiesel-rich product from 8.2 g oil). Selected levels used in the screening sequence are shown in bold.

Comparison of CaO/AC with activated carbon

Under the selected conditions (nominal oil: methanol ratio 1:16, catalyst loading 0.10 g, 60 °C and 60 min), CaO/AC produced approximately 90% recovered biodiesel yield,

whereas activated carbon alone produced approximately 32% (Figure 3). The 58-percentage-point difference supports a dominant catalytic contribution from the calcium-containing phase rather than from the carbon support alone.

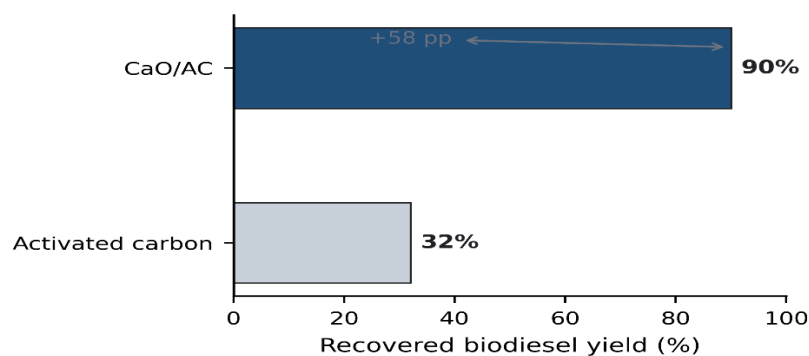


Figure 3: Comparison of recovered biodiesel yield for activated carbon and CaO/AC under the selected screening conditions (nominal oil:methanol ratio 1:16, catalyst loading 0.10 g, 60 °C, 60 min). The arrow indicates the 58-percentage-point gain obtained with CaO/AC

Discussion

The approximately 90% maximum recovered yield demonstrates the potential of coconut-shell activated carbon as a support for a calcium-based solid catalyst. A possible explanation is that calcium oxide, if present, can provide strongly basic sites for methoxide formation and triglyceride transesterification, while a porous carbon matrix can improve dispersion, handling and accessibility of the active phase. Earlier work with CaO established high transesterification activity but also identified deactivation and calcium leaching as practical concerns, particularly when WCO contains free

fatty acids (Kouzu et al., 2008). Carbon-supported systems aim to address these limitations by improving active-species distribution and mass transfer.

The present trend with increasing methanol is consistent with the reversible nature of transesterification: increasing alcohol availability can shift the equilibrium toward ester formation and improve contact between the alcohol and triglyceride phases. However, very high methanol concentrations can complicate glycerol separation and increase recovery costs, so the 1:16 condition is considered the best-performing level within the tested range, not a verified optimum. Modern

WCO studies commonly optimise alcohol ratio alongside catalyst amount, reaction time, and temperature using factorial or response-surface approaches rather than sequential one-factor screening (Mohadesi et al., 2022; Rocha Meneses et al., 2023).

Catalyst loading showed a typical optimum-type response. Increasing the loading from 0.05 to 0.10 g increased yield, consistent with greater availability of basic sites; the decline at 0.20 g may reflect poorer mixing, agglomeration or mass-transfer limitations at higher solids content. Similar interactions between catalyst dosage, alcohol ratio and reaction conditions have been reported for supported CaO and other heterogeneous WCO catalysts (Ibrahim et al., 2022; Mohadesi et al., 2022). Because the current dataset lacks replicate experiments and rheological or mass-transfer measurements, we cannot definitively assign the mechanism underlying the decline at 0.20 g.

The pronounced decrease in recovered yield after 120 min is also noteworthy. Once the reaction approaches equilibrium, prolonged contact may increase reverse reactions, hydrolysis, soap formation or handling losses. Importantly, this study measured recovered product mass rather than chromatographically quantified FAME conversion; therefore, the observed decline should not be interpreted as direct evidence of a specific chemical pathway without GC-based composition data.

The large performance difference between CaO/AC and AC alone is consistent with a catalytic role for the calcium-containing phase. Sun et al. (2021) reported a hierarchically porous coconut-shell-derived CaO/AC catalyst with highly dispersed CaO nanoparticles and high FAME yield, while Ibrahim et al. (2022) demonstrated strong WCO conversion using CaO-containing activated-carbon nanocatalysts. More recent work continues to emphasise structure–performance relationships in CaO/AC systems and the importance of combining phase identification, morphology, porosity and reusability with catalytic measurements (Rashad et al., 2026; Abo El-Khair et al., 2026). These studies support the rationale for the present catalyst design, although differences in feedstock composition, catalyst preparation, loading, reactor conditions, and the analytical definition of yield limit direct comparisons of absolute yields.

The study has several limitations. XRD and SEM-EDX analyses were unavailable, so we could not confirm the crystalline CaO phase, catalyst morphology, or the spatial distribution of calcium; FTIR and AAS alone support only the presence of calcium-containing species. The experimental record did not support the reported oil:methanol ratios, catalyst loading relative to the mass of oil, detailed product-purification and catalyst-recovery procedures, or replicate counts. The record provided no replicate variability, catalyst reusability or leaching measurements, GC-FID/GC-MS confirmation of fatty acid methyl esters, or standardised fuel-property measurements. Therefore, recovered product yield should not be interpreted as chemically verified FAME yield, and the study cannot establish compliance with ASTM D6751 or EN 14214.

Conclusion

Coconut-shell-derived activated carbon was used as a support for a calcium-containing heterogeneous catalyst for transesterification of waste cooking oil. Among the conditions screened at 60 °C, a nominal oil: methanol ratio of 1:16, a CaO/AC loading of 0.10 g, and a reaction time of 60 min gave the highest recovered biodiesel-rich product yield of approximately 90%. CaO/AC also markedly outperformed activated carbon alone, which gave approximately 32% under

the selected conditions. FTIR and AAS results were consistent with chemical modification of the carbon support and incorporation of calcium-containing species. The study therefore provides preliminary evidence that dual valorisation of waste cooking oil and coconut-shell biomass is feasible. Future work should incorporate replicated factorial or response-surface experiments, XRD and SEM-EDX characterisation, surface-area/basicity measurements, FAME quantification, catalyst reusability and leaching tests, and complete fuel-property evaluation.

REFERENCES

- Abo El-Khair, M. A., Yadav, K. K., Aldosari, F. M., Fallatah, A. M., Alreshidi, M. A., Hameed, T. A., Ewies, E. F., & Morshedy, A. S. (2026). Activated carbon and biochar-based catalysts for sustainable biodiesel production: A comparative insight into transesterification efficiency, mechanism, and design. *Materials Research Bulletin*, 201. <https://doi.org/10.1016/j.materresbull.2026.114119>
- Buasri, A., Chaityut, N., Loryuenyong, V., Rodklum, C., Chaikwan, T., Kumphan, N., Jadee, K., Klinklom, P., & Wittayaronayut, W. (2012). Transesterification of waste frying oil for synthesizing biodiesel by KOH supported on coconut shell activated carbon in packed bed reactor. *ScienceAsia*, 38(3), 283–288. <https://doi.org/10.2306/scienceasia1513-1874.2012.38.283>
- Ibrahim, N. A., Rashid, U., Hazmi, B., Moser, B. R., Alharthi, F. A., Rokhum, S. L., & Ngamcharussrivichai, C. (2022). Biodiesel production from waste cooking oil using magnetic bifunctional calcium and iron oxide nanocatalysts derived from empty fruit bunch. *Fuel*, 317. <https://doi.org/10.1016/j.fuel.2022.123525>
- Kouzu, M., Kasuno, T., Tajika, M., Sugimoto, Y., Yamanaka, S., & Hidaka, J. (2008). Calcium oxide as a solid base catalyst for transesterification of soybean oil and its application to biodiesel production. *Fuel*, 87(12), 2798–2806. <https://doi.org/10.1016/j.fuel.2007.10.019>
- Lam, M. K., Lee, K. T., & Mohamed, A. R. (2010). Homogeneous, heterogeneous and enzymatic catalysis for transesterification of high free fatty acid oil (waste cooking oil) to biodiesel: A review. *Biotechnology Advances*, 28(4), 500–518. <https://doi.org/10.1016/j.biotechadv.2010.03.002>
- Mohadesi, M., Aghel, B., Gouran, A., & Razmehgir, M. H. (2022). Transesterification of waste cooking oil using clay/CaO as a solid base catalyst. *Energy*, 242. <https://doi.org/10.1016/j.energy.2021.122536>
- Monika, Banga, S., & Pathak, V. V. (2023). Biodiesel production from waste cooking oil: A comprehensive review on the application of heterogeneous catalysts. *Energy Nexus*, 10. <https://doi.org/10.1016/j.nexus.2023.100209>
- Rashad, M. A., Hussain, M., Jamil, F., Hamayun, M. H., Ahsan, A., Akhter, P., & Park, Y.-K. (2026). Dual-waste valorization of eggshell and coconut shell–pith biomass into CaO/activated carbon catalysts: Synthesis, characterization, and structure–performance correlations in biodiesel transesterification. *Carbon Letters*, 36(3), 1321–1335. <https://doi.org/10.1007/s42823-026-01065-y>
- Rocha Meneses, L., Hari, A., Inayat, A., Yousef, L., Alarab, S., Abdallah, M., Shanableh, A., Ghenai, C., Sabarathinam,

S., & Kikas, T. (2023). Recent advances on biodiesel production from waste cooking oil (WCO): A review of reactors, catalysts, and optimization techniques impacting the production. *Fuel*, 348. <https://doi.org/10.1016/j.fuel.2023.128514>

Singh, D., Sharma, D., Soni, S. L., Inda, C. S., Sharma, S., Sharma, P. K., & Jhalani, A. (2021). A comprehensive review of biodiesel production from waste cooking oil and its use as

fuel in compression ignition engines: 3rd generation cleaner feedstock. *Journal of Cleaner Production*, 307, Article 127299. <https://doi.org/10.1016/j.jclepro.2021.127299>

Sun, H., Sun, K., Wang, F., Liu, Y., Ding, L., Xu, W., Sun, Y., & Jiang, J. (2021). Catalytic self-activation of Ca-doped coconut shell for in-situ synthesis of hierarchical porous carbon supported CaO transesterification catalyst. *Fuel*, 285. <https://doi.org/10.1016/j.fuel.2020.119192>



©2026 This is an Open Access article distributed under the terms of the Creative Commons Attribution 4.0 International license viewed via <https://creativecommons.org/licenses/by/4.0/> which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is cited appropriately.