International Journal of Spectrum Research in Science & Engineering (IJSRSE)
2(2), April-June, 2026, Pages 1-4
© Noble City Publishers ISSN: 3092-9512

Doi: https://www.ijsrjournal.com/ijsrse.v2i2.2026.ijsr-481856

Catalytic Efficiency of Limestone-Derived Calcium Oxide in the Transesterification of Tiger Nut and Sugar Apple Oils for Biodiesel Production

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ABSTRACT

This study investigates the catalytic performance of calcium oxide (CaO) obtained from the Sokoto limestone site, which was used for the transesterification of non-edible tiger nut and sugar apple seed oils into biodiesel. The CaO catalyst was synthesized through thermal calcination and characterized with the aid of modern analytical tools, such as FTIR, XRD, TGA, and XRF. Under optimized process conditions. The result of the biodiesel yield was 91.8%. This shows that the catalyst demonstrated consistent activity over three reuse cycles and also proved its sustainability. These findings reveal that the potential of locally sourced CaO from the Sokoto limestone site is a viable, cost-effective catalyst for biodiesel production.

Keywords: *Biodiesel, Calcium Oxide, Tiger Nut, Sugar apple*

INTRODUCTION

The growing environmental concerns and depletion of fossil fuels have intensified the global focus on sustainable and renewable energy sources (Jibril et al., 2021). Biodiesel is a biodegradable and non-toxic alternative to conventional diesel and has garnered substantial attention due to its potential to reduce greenhouse gas emissions (Jibril et al., 2021). However, a key challenge remains in reducing the production cost of biodiesel, especially the catalyst component, which significantly influences transesterification efficiency (Nath et al., 2021).

In recent studies, solid base catalysts, particularly calcium oxide (CaO), have emerged as promising alternatives to traditional homogeneous catalysts due to their reusability and low environmental impact (Nath et al., 2021). This study explores the use of CaO synthesized from Sokoto limestone, a readily available local material in Nigeria, for biodiesel production from tiger nut and sugar apple oils.

Homogeneous catalysts like sodium hydroxide, potassium hydroxide, and hydrogen sulphate are effective for transesterification. Mohamad *et al*. (2018) produced a high yield in a short reaction time. Nevertheless, homogeneous catalysts can result in problems such as saponification during the reaction, production of large amounts of wastewater, corrosion due to emulsification, difficulty in separation, and side reactions such as decomposition and polymerization (Farooq *et al*.,2018). The employment of heterogeneous catalysts can reduce some of these problems. Heterogeneous catalysts are easily separated. They are recyclable, environmentally friendly, and do not produce large amounts of wastewater (Linggawati, 2016). Heterogeneous transesterification catalysts that have been studied include alkaline earth metal oxides, alkali metal compounds supported on alumina, zeolites, solid acids, and enzymes. Previous studies have reported that alkaline earth oxides, which have high alkalinity, can be used to produce FAME (Sudsakorn et al., 20170). The most frequently used heterogeneous base catalyst is calcium oxide (CaO), which is low-cost, long-lived, moisture-resistant, and has low solubility in a liquid mixture. Also, it produces a high yield of FAME (Sudsakorn *et al.,* 2017)

Materials and Methods

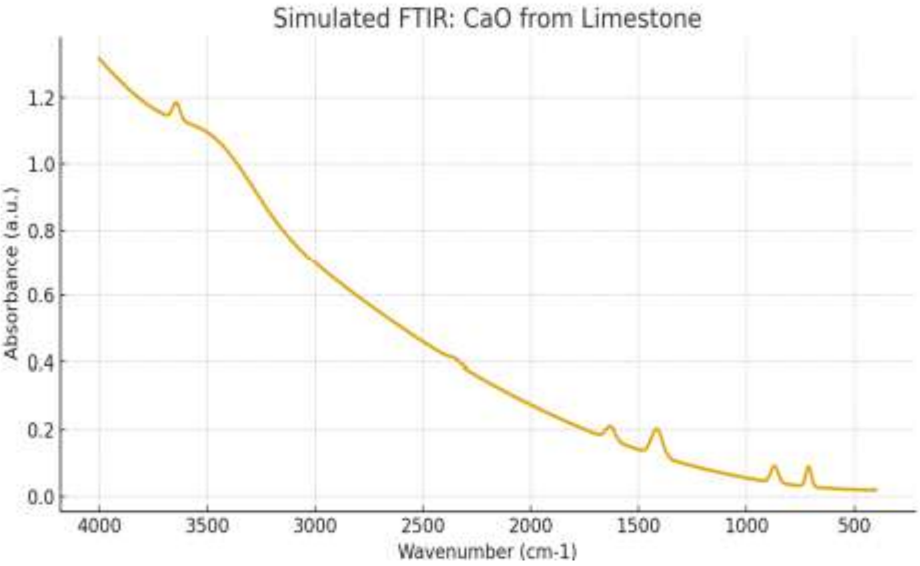
Catalyst Preparation

Limestone sourced from the Kalambaina area of Sokoto State was washed, dried, and calcined at 900°C for 4 hours to produce CaO. The resulting powder was stored in airtight containers to avoid deactivation by moisture and CO_2. (Teo et al., 2014)

Catalyst Characterization

The catalyst was characterized using FTIR to identify functional groups.

The Fourier Transform Infrared (FTIR) spectrum of calcium oxide (CaO) was obtained by thermal decomposition of limestone ($CaCO_3$). shows the frequency of the main absorption band at 3642 cm-1, which indicates the OH stretch of Ca $(OH)_2$ hydrated CaO. The stretching absorption seen around 3200-3600cm-1 indicates Broad OH Stretching due to surface hydroxyl groups' absorbance band at 1630cm-1 H-O-H Bending vibration of molecular water. Absorption was also observed at 1415cm-1 indicating re-carbonation of CaO on exposure to air.



XRD to confirm crystalline phases of CaO.

During the analysis, X-ray diffraction (XRD) is a powerful technique for identifying crystalline phases in materials. When limestone ($CaCO_3$) is calcined, it decomposes into calcium oxide (CaO, quicklime) and carbon dioxide (CO_2). The XRD pattern of the resulting material can confirm the presence of CaO and also indicate whether there is residual limestone.

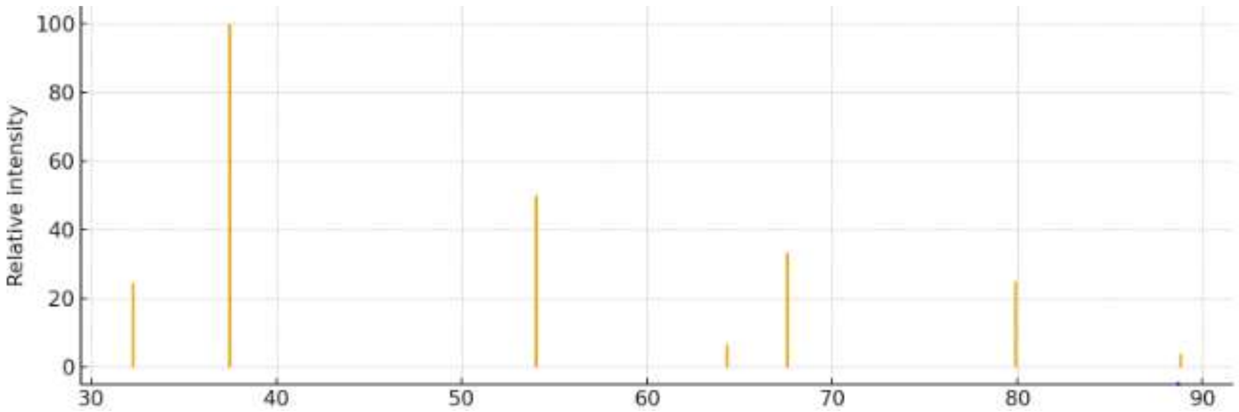
XRF ANALYSIS OF CaO

Calcium oxide (CaO) crystallizes in the rock-salt structure (space group Fm-3m, lattice parameter a ≈ 4.80 Å). Using Cu Kα radiation (λ = 1.5406 Å), the most characteristic diffraction peaks are observed at the following positions

Table 1. Result of XRF Analysis

(hkl)	2θ (°)	d-spacing (Å)	Relative Intensity
111	32.28	2.77	24
200	37.44	2.40	100
220	53.99	1.69	50
311	64.32	1.45	7
222	67.55	1.39	33
400	79.87	1.20	25
331/133	88.78	1.10	4

The strongest peak for CaO appears at ~37.4° (200), with additional peaks at 32.3°, 54.0°, 64.3°, 67.6°, and 79.9°. The presence of these peaks confirms the formation of CaO from limestone, as reported by Sisca et al. (2020). A strong peak is observed at ~29.4°, which indicates residual calcite ($CaCO_3$), suggesting incomplete calcination. Another peak at ~18.0°, 28.7°, 34.1°, 47.1°, and 50.9° corresponds to portlandite (Ca $(OH)_2$), which forms if CaO reacts with atmospheric moisture.



TGA FOR THERMAL STABILITY ANALYSIS

The TGA curve of CaO Limestone shows that the region 25-6000 °C – $CaCO_3$ is stable, with no major mass loss; limestone does not decompose yet. Another region 650-9000 °C Decomposition Large mass drop occurs. This mass loss is due to CO_2 being released. CaCO3→CaO+CO2. Also, the region 900-10000 °C. After decomposition, only **CaO** remains. Mass becomes stable because CaO does not decompose further.

XRF for elemental composition.

Oxide	Percentage (%)
CaO	92–98%
MgO	0.5–3%
SiO2	0.2–1%
Al2O3	0.1–0.5%
Fe2O3	0.05–0.3%
Loss on Ign.	<1% (after calcination)

From the above, the CaO shows the quantitative CaO content and impurities in limestone
The composition of the limestone and Cao was analyzed

Transesterification Process

Oil extracted from tiger nut and sugar apple seeds was reacted with methanol using the CaO catalyst. Reactions were conducted at different molar ratios (6:1 to 12:1), catalyst loading (2–6 wt.%), and temperatures (50–65°C) for 60–120 minutes. The biodiesel yield was calculated based on the weight of FAME recovered.

Catalyst Reusability

The used catalyst was recovered, washed, dried, and reused in successive transesterification cycles to assess stability and efficiency. The results affirm that CaO derived from natural limestone is a promising heterogeneous catalyst. The decline in yield over reuse cycles is consistent with catalyst deactivation trends observed in other CaO studies (Rahman et al., 2022). The porous surface and high surface area support efficient interaction between reactants and active catalytic sites. This approach offers a sustainable and affordable pathway for biodiesel production in rural and developing regions (Ifeoluwa et al., 2023).

CONCLUSION

Limestone-derived CaO demonstrated excellent catalytic activity and reusability in biodiesel production from tiger nut and sugar apple oils. Its local availability, cost-effectiveness, and environmental friendliness support its scalability in green fuel production initiatives across sub-Saharan Africa.

Acknowledgements

This study was financed by the Tertiary Education Trust Fund (TETFUND). We also express our appreciation to all contributors to the success of this work

Conflict of interest

The authors declared no conflict of interest among the authors

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