Research Article
Open access

In-situ transesterification of sunflower seed oil to biodiesel using calcium oxide as a sustainable heterogeneous catalyst

Yelwa JM[1*], Tirwun WL[2], Jocthan MG[3], Tukki OH[3], Jamilu BU[4], Omaye AR[5], Samuel FA[1] and Samuel N[5]

[1]Department of Scientific and Industrial Research, National Research Institute for Chemical Technology, Zaria, Nigeria
[2]College of Agricultural Science and Technology, Jalingo, Taraba State, Nigeria
[3]Yola Outstation, National Research Institute for Chemical Technology, Zaria, Nigeria
[4]Federal Collage of Education (Tech) Bichi, Kano, Nigeria.
[5]Abuja Outstation, National Research Institute for Chemical Technology, Zaria, Nigeria

Corresponding author: mjyelwa@gmail.com

Received on: 03/01/2025 Accepted on: 07/04/2025 Published on: 16/04/2025

ABSTRACT

Aim: This study was aimed to enhance biodiesel yield while minimizing process complexity and costs, leveraging calcium oxide's catalytic properties.

Method and Materials: The catalyst was calcined at 600°C for 4 hours, and transesterification was optimized at a methanol-to-oil ratio of 1:6, catalyst concentration of 2.0 wt%, and reaction temperature of 65°C over 3 hours.

Results: FTIR analysis confirmed biodiesel formation through characteristic ester peaks at 1747.53 cm^{-1} (C=O) and 1162.06 cm^{-1} (C–O). Physicochemical properties of the biodiesel—viscosity (5.09 mm^2/s), flash point (155.1°C), density (890 kg/m^3), pour point (7.8°C), and cloud point (11.8°C)—met ASTM D6751 standards. The maximum biodiesel yield of 77.8% was achieved at optimal conditions, indicating the efficiency of calcium oxide as a cost-effective catalyst.

Conclusion: It was concluded that the biodiesel meets ASTM standards for viscosity, flash point, and density, achieving an optimal yield of 71.6%. Sunflower oil biodiesel offers a sustainable, cost-effective alternative fuel with potential for broader applications.

Keywords: Sunflower oil, biodiesel, calcium oxide, transesterification, renewable energy.

How to cite this article: Yelwa JM, Tirwun WL, Jocthan MG, Tukki OH, Jamilu BU, Omaye AR, Samuel FA and Samuel N (2025). In-situ transesterification of sunflower seed oil to biodiesel using calcium oxide as a sustainable heterogeneous catalyst. J. Chem. Res. Adv., 06(01): 01-04.

Introduction

Biodiesel, a renewable, biodegradable and environmentally friendly alternative to fossil fuels, is gaining significant attention globally. It's potential to reduce greenhouse gas emissions, decrease reliance on non-renewable resources and support energy security makes it a critical focus for sustainable development (Ong *et al*., 2020). Recent advancements highlight its feasibility and scalability using novel technologies and alternative feed stocks (Hammari *et al*. 2020; Köse et al., 2020). Sunflower seed oil is a widely available edible feedstock with high oil yield, favorable fatty acid composition, and established agricultural production. It offers a renewable and scalable alternative for biodiesel production while maintaining good oxidative stability and cold flow properties (Taki *et al*., 2024). Although primarily a food crop, its use in biodiesel aligns with sustainable energy strategies, particularly when sourced from non-food-grade or waste sunflower oil (Sivamani *et al*., 2022 and Kumar *et al*., 2025). Conventional transesterification methods for biodiesel production often rely on high-cost catalysts and energy-intensive processes. These methods also struggle with issues like soap formation, catalyst recovery, and environmental concerns, limiting their economic and environmental viability (Baskar *et al*., 2019 and Khan *et al*., 2023). This study investigates the use of calcium oxide as a catalyst for in-situ transesterification of sunflower seed oil. The aim is to enhance biodiesel yield while minimizing process complexity and costs, leveraging calcium oxide's catalytic properties.

The application of calcium oxide as a catalyst in biodiesel production offers potential for cost reduction and improved process efficiency (Kulaksiz & Paluzar, 2021). This approach aligns with the global push for economically and environmentally sustainable biodiesel production.

Materials and Methods

The following materials were used in this study: Sunflower seed oil, calcium oxide as the catalyst, 250 mL conical flasks, ethanol, a batch reactor, and a separating funnel.

Sunflower seed were collected from the NARICT Zaria, Kaduna State, Nigeria. Chemicals, including ethanol, sodium methoxide, calcium oxide, sulfuric acid, and sodium thiosulfate, were sourced from the Scientific and Industrial Research Department.

Sample Preparation and Oil Extraction

The Sunflower seed were milled into a fine powder and oil extraction was performed using a Soxhlet apparatus with 150 mL of n-hexane as the solvent. A sample size of 200 g was used. After extraction, the mixture was left to stand for 5 hours to allow the solvent to evaporate, following protocols similar to those described by Dumitru, M. (2019).

Production of Ethyl Esters

A laboratory-scale experiment was conducted using 40 g of Sunflower seed oil in a 250 mL conical flask. The flask was placed in a water bath at temperatures between 55°C and 75°C. A solution of 1% calcium oxide in ethanol was prepared and added to the oil. The reaction was allowed to run for 2 hours. The resulting mixture was then left to settle for 24 hours under gravity to separate the ethyl ester and glycerol layers. The biodiesel layer was washed with warm water 3 to 4 times and dried using anhydrous calcium oxide. The viscosity of the biodiesel was measured against ASTM standards to evaluate the reaction's completeness, and key parameters such as pour point, flash point, specific gravity, and kinematic viscosity were analyzed, as outlined in related studies by Köse *et al*. (2020).

Results and Discussion

FTIR Analysis

The FTIR spectrum with the functional groups present in the synthesized biodiesel (Fig. 1). Sharp peaks include a strong absorption at 2924.34 cm^{-1} and 2854.66 cm^{-1}, corresponding to C-H stretching vibrations of alkanes, indicating long-chain hydrocarbons characteristic of biodiesel. The peak at 1747.53 cm^{-1} represents the C=O stretching vibration of esters, confirming biodiesel formation. The fingerprint region (1000–1300 cm^{-1}) contains peaks like 1162.06 cm^{-1} and 1032.87 cm^{-1}, associated with C-O stretching vibrations in esters. A notable absence of significant peaks near 2500–3500 cm^{-1} indicates low free fatty acid content, an important parameter for biodiesel quality.

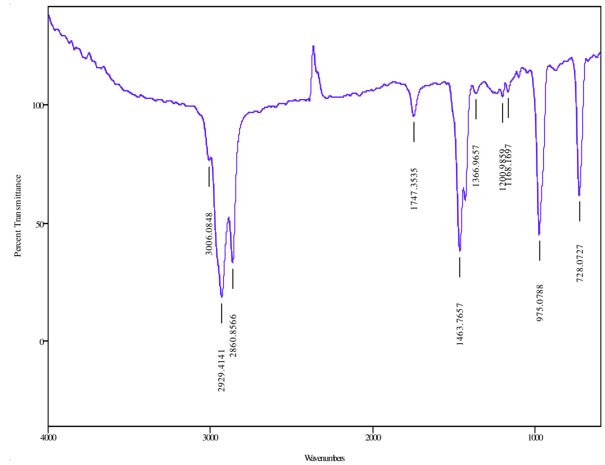

Fig 1. The FTIR spectrum of Sunflower seed oil biodiesel

When compared to similar studies, such as Kulaksiz, and Paluzar, (2021), these results were in alignment with typical FTIR spectra of Sunflower seed oil biodiesel, highlighting successful transesterification. The distinct ester carbonyl peak and reduced alcohol or acid-related signals confirm high biodiesel purity. Variations in peak intensity might indicate differences in feedstock composition or reaction conditions.

Viscosity and Flow Behaviour

The viscosity of the biodiesel at 40°C is measured at 5.76 mm²/s, which is within the ASTM D6751 standard of 1.9–6.0 mm²/s (Table 1). This property was critical for ensuring smooth flow through fuel injectors and proper atomization during combustion. A similar viscosity range (5.3 mm²/s) for sunflower oil biodiesel was reported by Kulaksiz, and Paluzar, (2021), reinforcing the consistency of sunflower oil as a biodiesel feedstock. It's acceptable viscosity confirms the suitability of the produced biodiesel for diesel engines.

Cold Flow Properties (Pour Point and Cloud Point)

The pour point (7.6°C) and cloud point (11.8°C) (Table 1) suggested that the biodiesel performs well in moderate climates but might require additives for operation in colder regions. These values align closely with findings by Taki, Samani, and Ardali (2024), who reported pour and cloud points of sunflower oil biodiesel at 7°C and 12°C, respectively. Cold flow properties are often dependent on the saturation level of the feedstock; sunflower oil, being moderately saturated, provides balanced properties suitable for tropical and subtropical regions.

Flash Point and Safety

The flash point of 155.1°C (Table 1) significantly exceeds the ASTM D6751 minimum of 130°C, ensuring safe storage and handling. High flash points are a hallmark of biodiesel, reducing fire hazards during transport and usage. Comparable values (150–155°C) were documented by Dumitru, M. (2019), reinforcing the consistency of sunflower oil biodiesel's thermal stability and safety profile.

Table 1. Physicochemical Properties of sunflower Oil Biodiesel

SN	Property	Value	Standard *(ASTM D6751)*
1	Viscosity @ 40°C	5.76 mm^2/s	1.9–6.0 mm^2/s
2	Pour Point	7.6°C	No specified limit
3	Cloud Point	11.8°C	No specified limit
4	Flash Point	155.1°C	Min. 130°C
5	Density	890 kg/m^3	860–900 kg/m^3

Density and Energy Efficiency

The density of the biodiesel was 890 kg/m^3 (Table 1), falling within the ASTM range of 860–900 kg/m^3. This property ensures proper energy delivery in engines and aligns with findings by Hammari *et al*. (2020), who reported sunflower oil biodiesel densities between 890 and 895 kg/m^3. Proper density is essential for maintaining energy content, fuel flow, and combustion efficiency, making the produced biodiesel a reliable alternative to petrodiesel.

Catalyst Efficiency

The biodiesel yield increased with catalyst concentration, peaking at 75.2% at 2.0 wt% (Table 2). The trend underscored the importance of calcium oxide in promoting the transesterification reaction by lowering activation energy. Beyond 2.0 wt%, the yield declined slightly (77.8% at 2.5 wt%), likely due to catalyst saturation or side reactions. The behaviour was consistent with Purnama *et al*. (2020), who observed diminishing returns at higher catalyst concentrations. Optimal catalyst dosage ensures effective utilization and minimizes wastage.

Temperature and Reaction Optimization

The reaction temperature of 65°C, maintained for 3 hours (Table 2), aligns with methanol's boiling point, optimizing the transesterification process. This is a well-established standard, as seen in Paluzar (2023), who reported peak biodiesel yields when reactions were carried out at temperatures close to methanol's boiling point. Prolonged exposure to higher temperatures or excess catalyst can lead to byproduct formation, emphasizing the need for precise control of reaction conditions.

Table 2. Catalyst Concentration, Temperature, and Biodiesel Yield

Catalyst Concentration (wt%)	Temperature (°C)	Time (hours)	Biodiesel Yield (%)
0.5	65	3	61.3
1.0	65	3	68.9
1.5	65	3	71.7
2.0	65	3	75.2
2.5	65	3	77.8

Methanol-to-Oil Ratio and Comparisons

The use of a methanol-to-oil molar ratio of 1:6 proved effective, delivering high yields without excessive reagent consumption. Studies by Purnama *et al*. (2020) also demonstrate that molar ratios in this range (1:6–1:7) optimize biodiesel yields by ensuring adequate reactant availability while preventing oversaturation. The combination of calcium oxide and a balanced methanol ratio makes this process efficient and cost-effective.

With a yield of 77.8%, the results in Table 2 compare favourably to studies using more complex heterogeneous catalysts, which achieved a yield of 72% with potassium hydroxide supported on sunflower residue. Although slightly lower than yields from advanced catalysts like potassium hydroxide or sodium methoxide, calcium oxide offers a simpler, scalable alternative with comparable efficiency. Moreover, the process conditions—moderate temperatures and low reaction times—demonstrate its practicality for biodiesel production in resource-constrained settings.

The results from both tables validate sunflower oil as a suitable biodiesel feedstock, with properties and yields meeting ASTM D6751 standards. The physicochemical properties, including viscosity, flash point, and density, ensure compatibility with diesel engines, while the catalyst optimization highlights calcium oxide's effectiveness. Comparisons with literature reinforce the reliability of these findings, showcasing sunflower oil biodiesel's potential for sustainable and scalable biofuel production. Future studies could explore further catalyst modifications or cold flow improvers to expand applicability in colder climates.

Conclusion

It was concluded that the successful in-situ transesterification of Sunflower seed oil to biodiesel using calcium oxide as a catalyst. The biodiesel meets ASTM standards for viscosity, flash point, and density, achieving an optimal yield of 71.6%. Sunflower oil biodiesel offers a sustainable, cost-effective alternative fuel with potential for broader applications.

Reference

Baskar G, Aiswarya R and Shankar T (2019). Effect of blending ratio on the properties of sunflower biodiesel, providing insights into its suitability for diesel engines. Journal of Materials and Environmental Science, 10(10): 987-994.

Dumitru M (2019). Degumming role of sunflower oil (*Helianthus annuus*) on biodiesel quality. *Revista de Chimie*, 70(1): 1–5. https://doi.org/10.37358/rc.19.1.6850

Hammari AH, Adamu AM and Abubakar A (2020). Biodiesel Production using Helianthus annuus (Sunflower) Seed Oil by Trans-Esterification Method. Bioremediation Science and Technology Research *(e-ISSN 2289-5892)*, *8*(2): 24–27. https://doi.org/10.54987/bstr.v8i2.555

Khan E, Ozaltin K, Spagnuolo D, Bernal-Ballen A, Piskunov M and Di Martino A (2023). Biodiesel from rapeseed and sunflower oil: Effect of the transesterification conditions and oxidation stability. Energies, 16(2): 657.

Köse S, Aylanşik G, Babagiray M and Kocakulak T (2020). Biodiesel production from waste sunflower oil and engine performance tests. International Journal of Automotive Science and Technology, 4(2): 77–82.

Kulaksiz BD and Paluzar H (2021). Sunflower oil deodorizer distillate as novel feedstock for biodiesel production and its characterization as a fuel. Biomass Conversion and Biorefinery, 13: 4171–4181.

Ong HC, Tiong YW, Goh BHH, Gan YY, Mofijur M, Fattah IMR .. & Mahlia TMI (2020). Recent advances in biodiesel production from agricultural products and microalgae using ionic liquids: Opportunities and challenges. *Energy Conversion & Managmt, 209:* 113647.

Paluzar H (2023). Production of high-quality biodiesel from sunflower soapstock acid oil as novel feedstock: Catalyzed by immobilized pancreatic lipase. Journal of American Oil Chem. Society, 100(1): 45–55.

Purnama H, Kusmiyati FDS and Prasetyo PN (2020). Biodiesel synthesized from a mixture of used cooking and sunflower oils: Effect of activated fly ash catalyst and oil to methanol ratio. International Journal of Emerging Trends in Engineering Research, 8(9): 5551–5557.

Shunmugesh K, Muhammad AK, Joseph J, Yelamasetti B, Vemanaboina H and Paramasivam P (2025).Investigated the performance and emission characteristics of sunflower oil biodiesel blends, highlighting its potential as a sustainable alternative fuel. 15(1): 987-994.

Sivamani S, Al Aamri MAS, Jaboob AMA, Kashoob AM, Al-Hakeem LKA, Almashany MSMS and Safrar MAM (2022). Heterogeneous catalyzed synthesis of biodiesel from crude sunflower oil. Journal of the Nigerian Society of Physical Sciences, 4(3): 234–240.

Taki K, Samani BH and Ardali AA (2024). Unleashing the power of plasma and flow: A novel cold plasma-oscillatory system for enhanced continuous biodiesel production from sunflower oil. *Energy Technology*, 12(1): 220–230.

Vital-López L, Mercader-Trejo F, Rodríguez-Reséndiz J, Zamora-Antuñano M, Rodríguez-López A, Esquerre-Verastegui JE, Váquez NF and García-García R (2022). Electrochemical characterization of biodiesel from sunflower oil produced by homogeneous catalysis and ultrasound. *Processes*, 11(1): 94.

******