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Optimization of Biodiesel Production from Jatropha and Waste Cooking Oil Blends Using Mixed Metal Oxide Nanocatalysts: A Response Surface Methodology Approach

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Abstract

Biodiesel production has emerged as a sustainable alternative to fossil fuels, addressing both environmental concerns and the need for renewable energy. This study investigates the optimization of biodiesel production from underutilized vegetable oils, specifically jatropha seed oil and waste cooking oil, through transesterification using three synthesized nano-mixed metal oxide catalysts (n-CaO, n-CuO/CaO, and n-CaO/ZnO). A key challenge addressed is the inefficiency of conventional catalysts and the limited use of low-cost, sustainable feedstocks. Physicochemical analyses of crude, refined, and blended oils revealed improved fuel properties upon blending, notably a reduction in free fatty acid (FFA) content and acid value. Among the catalysts, CaO/ZnO exhibited the highest catalytic performance, achieving a biodiesel yield of 91.70 % and demonstrating strong stability over multiple reaction cycles. Optimization using a Full Factorial Design further improved the fatty acid methyl ester (FAME) yield to 94.76 %. Structural and functional analyses via FTIR and XRD confirmed the catalysts' crystallinity and functional groups, while GC-MS identified key FAME components in the produced biodiesel. This comparative analysis of three nanocatalysts underscores their potential in enhancing biodiesel quality and yield, offering a scalable, eco-friendly solution for sustainable energy development.

Keywords: Biodiesel, vegetable oils, nanocatalysts, transesterification, optimization

Introduction

The increasing global demand for renewable and sustainable energy sources has intensified the exploration of biodiesel as a viable alternative to fossil fuels. This shift is primarily driven by the rising cost of petroleum, escalating environmental concerns, and the urgent need to curb greenhouse gas emissions (Al-Muhtaseb et al, 2021; Hafeez et al, 2020; Kasirajan et al, 2021). Biodiesel, a biodegradable and non-toxic fuel, offers a lower carbon footprint than traditional petroleum-derived diesel and can be utilized in existing diesel engines without major modifications (Adenuga et al, 2021). Biodiesel is produced via transesterification, a chemical process that converts triglycerides in oils or fats into fatty acid methyl esters (FAMEs), using alcohol and a catalyst (Anbessie et al, 2019; Borah et al, 2019). While biodiesel production has several advantages, it faces challenges including high production costs, limited availability of suitable feedstocks, and catalyst inefficiencies (Bharti et al, 2021; Qureshi et al, 2021). Addressing these issues is crucial to enhancing large-scale adoption. Non-edible vegetable oils such as jatropha oil and waste cooking oils represent promising low-cost, sustainable feedstocks for biodiesel production, avoiding competition with food

However, these oils often have high free fatty acid content, requiring robust catalyst systems and optimized reaction conditions for effective conversion. In alignment with global environmental goals, particularly the United Nations Sustainable Development Goals (SDGs), biodiesel supports SDG 7 (Affordable and Clean Energy) and SDG 13 (Climate Action). These goals emphasize the global need to expand renewable energy access and mitigate climate change through reduced carbon emissions. According to the International Energy Agency (IEA, 2022), global biodiesel production is expected to rise from 42.7 billion liters in 2022 to over 52.5 billion liters by 2027 to meet increasing demand.

Recent research has shown that mixed metal oxide-based nanocatalysts offer significant advantages over conventional catalysts. These include enhanced surface area, thermal stability, high catalytic activity, and recyclability, which contribute to improved biodiesel yields and reaction efficiency (Pratiwi *et al*, 2019; Rahmani *et al*, 2017). Nanocatalysts also exhibit superior resistance to saponification and can operate effectively under milder conditions, making them ideal for processing low-quality or waste feedstocks. Despite growing interest in nanocatalyst applications, few studies have thoroughly investigated the use of blends of underutilized oils (such as jatropha oil and waste cooking oil) with multiple mixed metal oxide nanocatalyst**s** in a comparative framework. This study addresses that gap by evaluating the performance of three nanocatalysts (CaO, CuO/CaO, and CaO/ZnO) in enhancing the transesterification process of blended feedstocks. By optimizing reaction conditions and characterizing the catalyst and biodiesel properties, this work contributes to the development of a cost-effective and scalable biodiesel production process.

Materials and Methods

Materials and Feedstock Collection

Jatropha seeds were sourced from an agro-processing farm in Oke-Oyi, Ilorin East LGA, while waste cooking oil (WCO) was collected from household and commercial kitchens in Agric area of Ilorin, Kwara State, Nigeria, over a period of three weeks to minimize seasonal variability. The collected feedstocks were filtered to remove particulates and stored in clean, airtight containers at 4 °C until use. All chemicals and reagents used in this study were of analytical grade, procured from Cerrilliant (Round Rock, TX, USA), Sigma-Aldrich (UK), and Teddington Middlesex (UK).

Oil Extraction and Refining

Crude jatropha oil (CJO) was extracted via Soxhlet extraction using n-hexane (analytical grade) as solvent. A seed-to-solvent ratio of 1:5 (w/v) was maintained, and the extraction was carried out at 45 °C, for 20 hours. The extracted oil was dried at 40 °C in a vacuum oven to remove residual solvent and stored at −20 °C until further use.

Both crude jatropha oil (CJO**)** and crude waste cooking oil (CWO) were refined through a standardized three-step procedure involving degumming, neutralization, and bleaching. In the degumming stage, 21 mL of aqueous 300 g/mL $NaCl_{(aq)}$ solution was added to the crude oil at 60 °C and stirred at 800 rpm for 60 minutes, followed by

centrifugation at 1000 rpm for 30 minutes to remove phosphatides and gums. For neutralization, the oil was treated with 2.5 M sodium hydroxide (NaOH) at 60 °C for 30 minutes under continuous stirring to neutralize free fatty acids. Finally, during the bleaching stage, 2% activated clay was added to the oil, and the mixture was heated to 90 °C for 45 minutes. The treated oil was then filtered to obtain the refined product. The refined jatropha oil (RJO) and refined waste cooking oil (RWO) were blended in three ratios (75:25, 25:75, and 50:50) by volume and stirred at room temperature (25 °C) for 15 minutes to ensure homogeneity (Olaoluwa et al., 2017).

Physicochemical Characterization of Oils and Blends

The density, kinematic viscosity, acid value (AV), iodine value (IV), and saponification value (SV) of all individual and blended oils were determined following ASTM D6751 and AOAC Official Methods (AOAC, 2012). The measurements were performed in triplicate and averaged to ensure accuracy. The most suitable blend was selected based on its improved physicochemical characteristics and compliance with biodiesel standards.

Catalyst Synthesis and Characterization

Three nano-mixed metal oxide catalysts (n-CaO, n-CuO/CaO, and n-CaO/ZnO) were synthesized using standard wet chemical procedures. The n-CaO catalyst was synthesized via the sol-gel method by dissolving 0.5 M calcium nitrate tetrahydrate [$Ca(NO_3)_2.4H_2O$] in distilled water and mixing it with in 25 ml of ethylene glycol solution. The pH of the solution was adjusted to 7 using sodium hydroxide, The gel solution was left at a static condition for approximately 5 hours in order to obtain nanoparticles of uniform size after being stirred for 10 minutes. The resulting gel was dried overnight at 110 °C and then calcined at 500 °C for 4 hours (Degfie, et al., 2019).

The n-CuO/CaO catalyst was prepared by co-precipitation. A 1:3 mixture of copper acetate and sodium hydroxide were combined at a flow rate of 2 mL/min for 4 hours until the solution turned completely black. The resulting precipitate were continuously washed with ethanol and de-ionized water in order to get rid of any residual impurities after centrifuging them for 20 minutes at 4000 rpm. The recovered precipitate was filtered, dried at 105 °C, and calcined at 600 °C for 3 hours. The synthesized CuO-NP and n-CaO powder (1:1) were combined with 50 mL of deionized water, and the mixture was stirred at 40 °C for six hours. After placing the retentate onto Whatman No. 1 filter paper, it was calcined for three hours at 600 °C (Niju et al., 2019).

For the n-CaO/ZnO catalyst, 10 grams of calcined CaO was combined with 1% ZnO. The mixture was stirred at 300 rpm, 80 °C for 2 hours, then dried at 100 °C, and finally calcined at 450 °C for 1.5 hours (Pratiwi et al., 2019). Each catalyst was stored in airtight containers to prevent moisture absorption before characterization and use in transesterification reactions. The structural and functional properties of the synthesized nanocatalysts were confirmed using X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR).

Transesterification Process

Transesterification was carried out in a 500 mL three-necked round-bottom flask fitted with a reflux condenser, thermometer, and mechanical stirrer. The optimal oil blend was reacted with methanol at a methanol-to-oil molar ratio of 9:1, using 0.45; 0.65; 0.85; 1.05; 1.25 % wt/wt catalyst loading. The reaction mixture was stirred at 600 rpm, maintained at 60 °C, and allowed to proceed for 90 minutes. Upon completion, the mixture was allowed to settle, and the upper biodiesel layer was separated. The biodiesel was purified and dried before analysis (Leevijit et al., 2016). To determine the best nanocatalyst, the biodiesel produced was weighed and the yield was determined. Biodiesel yield was computed by the equation below.

$$\text{Biodiesel Yield (\%)} = \frac{\text{(Weight of Biodiesel)}}{\text{(Weight of Oil Used)}} \times 100 \quad \ldots. (1)$$

Following the determination of the optimal nanocatalyst, the catalyst was extracted using centrifugation. The catalyst was washed with methanol and n-hexane (1:1) to remove residual reactants, and dried at 105 °C for 3 hours. In order to monitor the catalyst stability and reusability, the cleaned catalyst was reused and 10 runs of the procedure were carried out in a row.

Optimization and Experimental Design

The design of the experiment was carried out using a 2^4 factorial-based response surface methodology (RSM) experimental design to evaluate the influence of four independent variables: oil blend ratio, catalyst concentration, reaction temperature, and reaction time. A total of 16 experimental runs were conducted to determine optimal conditions for maximum biodiesel yield. Statistical analysis was performed using ANOVA, and the optimal conditions were determined based on the generated model. The physical, chemical and fuel properties of the produced biodesel, including density, viscosity, flash point, and acid value, were determined by the Leevijit methodology (Leevijit et al., 2016). Gas chromatography-mass spectrometry (GC-MS) analysis was conducted to determine the fatty acid methyl ester (FAME) composition (AOAC, 2012). Fourier Transform Infrared Spectroscopy (FTIR) was used to determine the functional group present.

Results and Discussion

Physicochemical Properties of Oils and Blends

The physicochemical properties of crude and refined oils [crude jatropha oil (CJO), refined jatropha oil (RJO), crude waste cooking oil (CWO), and refined waste cooking oil (RWO)] were analyzed and are summarized in Table 1. Parameters such as density, saponification value (SV), and iodine value (IV) varied depending on the oil type and processing state. For instance, the density values were 0.881 g/cm³ (CJO), 0.875 g/cm³ (RJO), 0.910 g/cm³ (CWO), and 0.890 g/cm³ (RWO), with a standard deviation of ±0.002 g/cm³ based on triplicate analyses. Refining significantly reduced acid value (AV) and free fatty acid (FFA) content, which is beneficial for biodiesel production by minimizing soap formation. Saponification values decreased slightly

after refining, indicating a shift in fatty acid chain lengths. These findings align with prior studies (Mustapha *et al*., 2021; Zakaria *et al.*, 2022), which observed improved oil quality post-refinement.

Blending of RJO and RWO in three volumetric ratios (Blend 1: 75:25, Blend 2: 25:75, Blend 3: 50:50) revealed enhanced fuel properties. Table 2 shows that Blend 3 had the most favorable characteristics, including AV of 1.12 ± 0.03 mg KOH/g, % FFA of 0.56 ± 0.01 %, and a density of 0.874 ± 0.003 g/cm³. The specific gravity of the blends (0.90–0.91 g/cm³) fell within or close to the typical biodiesel range (0.86–0.90 g/cm³) outlined in ASTM D6751, with Blend 3 slightly exceeding the upper range, possibly due to higher unsaturation. The oil blends show similar properties when compared to reported literature (Fadhil *et al*., 2017; Milano *et al*., 2018).

Interestingly, Blend 3 had the highest iodine value (86.49 ± 0.5 I_2/100g), indicating a greater degree of unsaturation. While unsaturation is beneficial for cold flow properties, it may reduce oxidative stability over time. Thus, the improved oxidative properties observed in Blend 3 are attributed to the balance between saturated and unsaturated fatty acids, not merely the iodine value alone. These results suggest that oil blending enhances physicochemical stability and optimizes fuel properties for biodiesel synthesis. FTIR analysis (Figure 1) of Blend 3 revealed a prominent ester peak at 1118 cm^{-1}, and peaks at 1639 cm^{-1} and 1461 cm^{-1} corresponding to carbonyl and unsaturated hydrocarbon groups, respectively, confirming biodiesel potential.

Characterization and Performance of Nanocatalysts

X-ray diffraction (XRD) shown in Figure 2 confirmed the crystalline phases of the synthesized catalysts. For n-CaO (A), peaks at 29.93°, 32.9°, and 53.63° matched JCPDS card No. 04-0777 for face-centered cubic CaO. The n-CuO/CaO (B) catalyst displayed additional peaks at 38.30°, 47.74°, and 74.56° corresponding to monoclinic CuO (JCPDS No. 05-0661), verifying the dual-phase composition. Likewise, n-CaO/ZnO (C) showed peaks matching hexagonal wurtzite ZnO (JCPDS No. 36-1451) and CaO, confirming successful composite formation. These findings are in accordance with previously reported studies of Niju *et al*., (2019) and Degfie *et al*., (2019).

FTIR spectra shown in Figure 3 further validated metal-oxide bonding: peaks at 708 cm^{-1} and 872 cm^{-1} (Ca-O), 775 cm^{-1} (Cu-O), and 711 cm^{-1} (Zn-O), indicating successful incorporation of metal oxides. These absorption bands are in line with reported literature ranges for corresponding bonds (Asgari *et al*., 2014; Wei *et al*., 2012; Bordbar *et al*., 2014). In addition to the major metal-oxide absorption bands, the FTIR spectra were examined for signs of residual impurities. No significant peaks associated with carbonate (1400–1500 cm^{-1}) or nitrate species (1250–1380 cm^{-1}) were observed, suggesting high catalyst purity. Minor peaks below 500 cm^{-1} were attributed to metal–oxygen lattice vibrations.

The average biodiesel yields achieved using n-CaO, n-CuO/CaO, and n-CaO/ZnO were 80.02 ± 1.1 %, 87.24 ± 1.0 %, and 91.52 ± 0.8%, respectively. Among the three, n-CaO/ZnO demonstrated the highest

catalytic efficiency due to the synergistic interaction between CaO and ZnO, which likely improves active site availability and stability. The reusability study (Figure 4) revealed that n-CaO/ZnO maintained yield above 78.7 % after eight cycles, with only a 0.01 % reduction in catalyst mass. This stability is attributed to minimized active site leaching and better thermal resistance. These results validate the structural integrity and superior reactivity of the n-CaO/ZnO nanocatalyst, making it an excellent candidate for large-scale biodiesel production from blended non-edible oils
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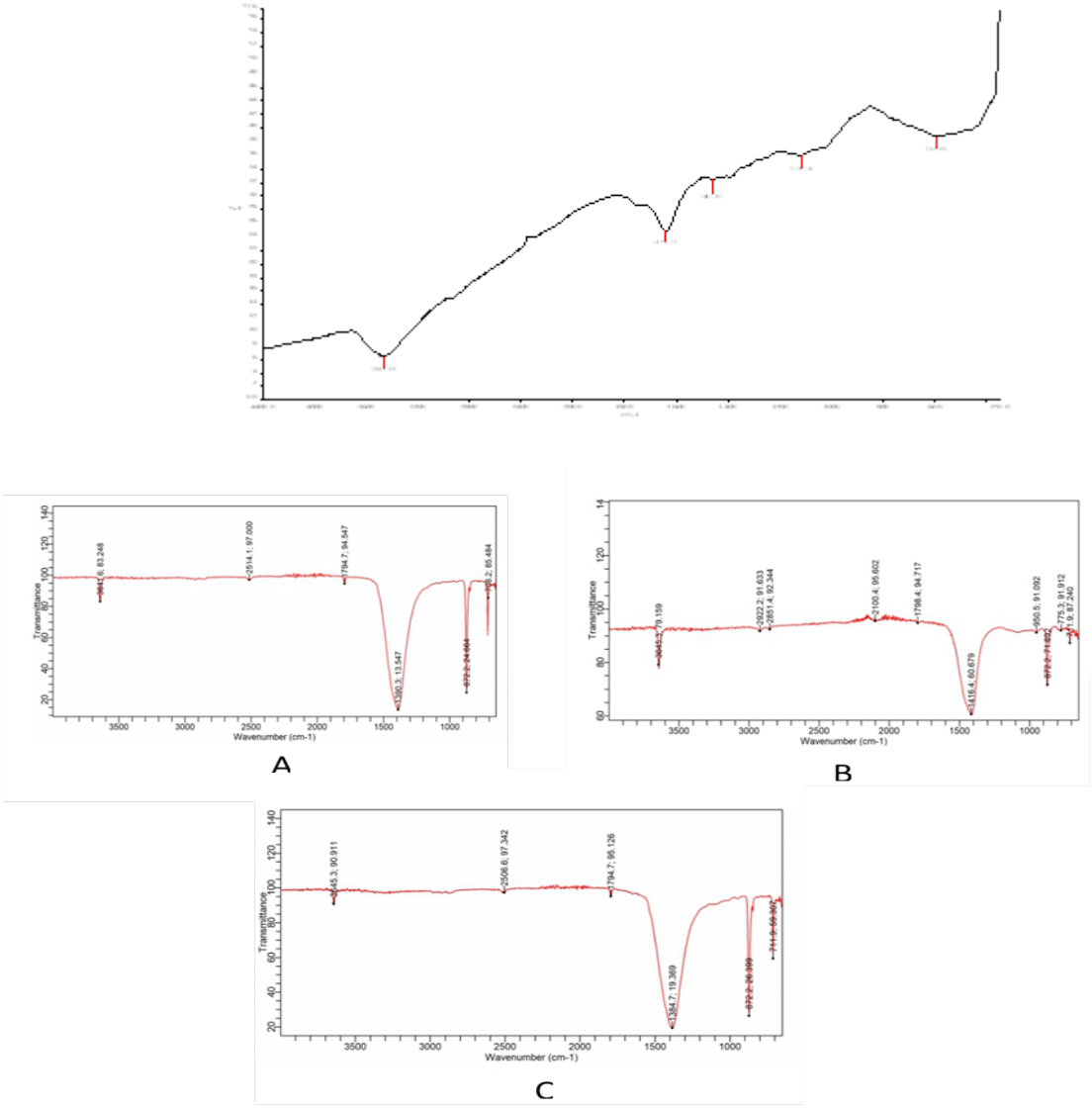


Figure 1: The Ideal Oil Blend's Fourier Transform Infrared (FTIR) Spectrum

Table 1: Physicochemical Properties of CJO, RJO, CWO and RWO Compared With Seed Oils.

Physicochemical Properties	Jatropha Oil		Waste Cooking Oil		Waste cooking Oil[a]		Jatropha Oil[b]	Cashew Nut Shell Liquid[c]	Castor seed oil[d]	ASTM Standard
	CJO	RJO	CWO	RWO	Crude	Refined				
Acid Value (mg KOH/g)	47.69	1.68	5.611	1.6837	5.89	2.42	39.89	13.2	0.92	0.40-4.00
Density at 30^{o}C (g/cm^{3})	0.881	0.875	0.905	0.89	0.93	0.91	_	_	_	0.88-0.95
Specific Gravity (g/cm^{3},30^{o}C)	0.950	0.945	0.910	0.90	0.95	0.91	0.902	0.935	0.96	0.957-0.968
Saponification Value (mg/KOH)	194.87	187.60	186.90	179.45	197.50	181.14	72.94	66.8	186	175-187
Iodine Value (I_2/100g)	85.3	82.4	80.00	59.76	_	_	11.065	124.4	91.00	82-88
% FFA	23.98	0.84	2.8	0.28	2.96	1.21	_	6.6	_	0.3-1.0
Average Molecular Weight (gmol^{-1})	815.96	895.44	894.87	936.18	846.26	926.69	2267.5	2506.26	903.92	_

Keynote *: (Mustapha et al., 2021[a]; Mustapha et al., 2020[b]; Zakaria et al., 2022[c] and Pradhan et al., 2021[d])*

Table 2: Physicochemical Properties of Blend Oils Compared with Some Seed Blend Oils.

Physicochemical Properties	Blend 1 (25:75)	Blend2 (75:25)	Blend 3(50:50)	Waste cooking oil and Jatropha blend (70:30)[a]	Palm kernel and G. oil (70:30)[b]	Rubber seed and Neem oil (40:60)[c]	Waste fish oil and Castor seed oil (50:50)[d]	ASTM Standard
Acid Value (mgKOH/g)	0.5611	1.34	1.12	13.26	1.42	35.34	0.51	0.40-4.00
Density at 30ºC (g/cm^3)	0.860	0.850	0.874	0.902	0.910	0.931	0.951	0.88-0.95
Specific Gravity (g/cm^3, 30 ºC)	0.905	0.90	0.91	0.95	_	_	_	0.957-0.968
Saponification Value (mg/KOH)	180.45	185.98	182.50	_	_	188.64	_	175-187
Iodine Value (I_2/100g)	79.80	80.04	86.49	_	_	96.64	_	82-88
% FFA	0.28	0.68	0.56	6.66	0.17	_	_	0.3-1.0

Keynote: (Milano et al., 2018[a]; Giwa et al., 2016[b]; Falowo et al., 2019[c]; Fadhil et al., 2017[d])

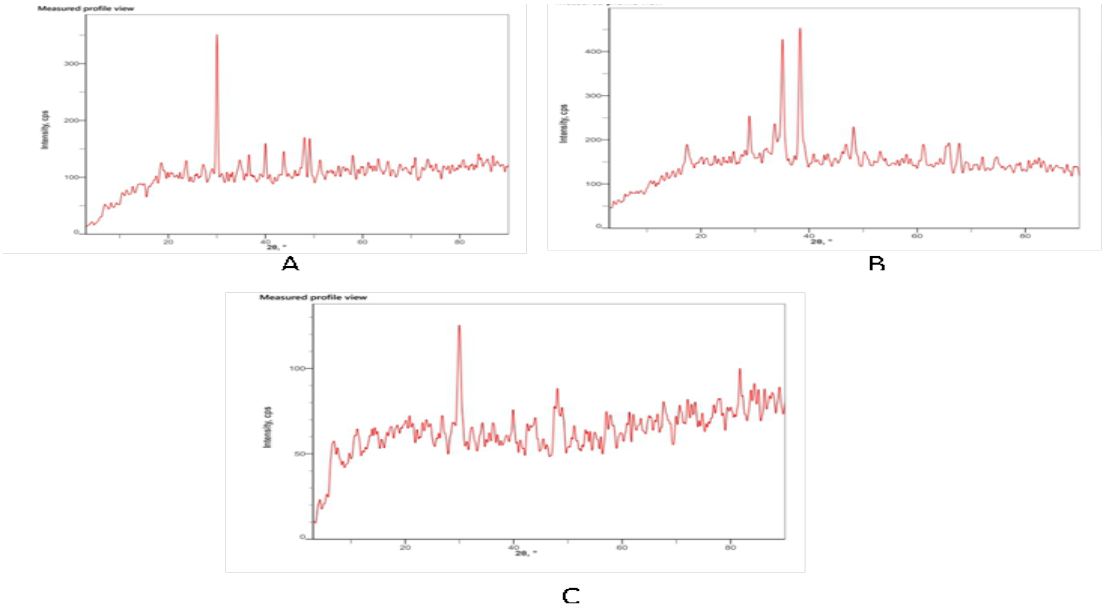


Figure 3: X-ray Diffractogram of CaO {A}, CuO/CaO {B} and CaO/ZnO {C} Nanocatalysts.

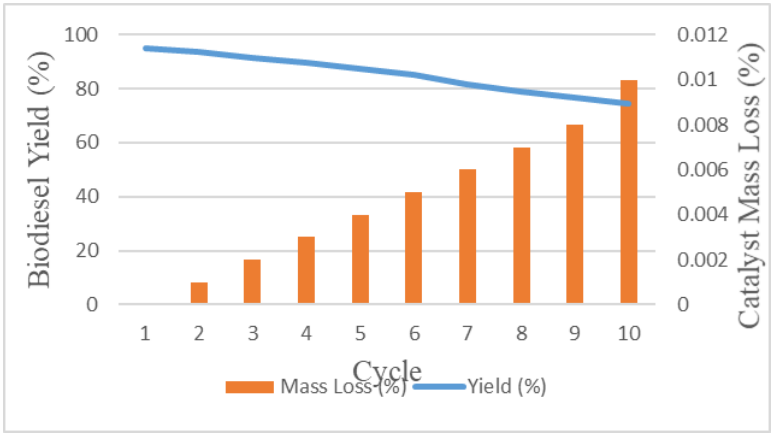


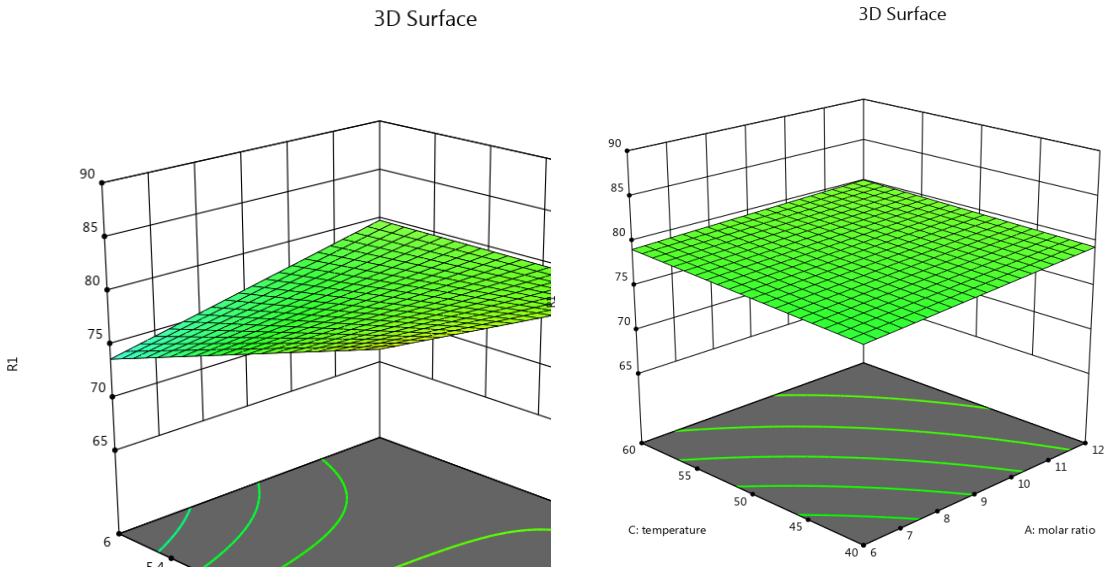
Figure 4: Reusability and Stability of n-CaO/ZnO catalyst over Ten Cycles

Table 3: Types and Concentrations of Catalyst on the Yield of Biodiesel.

Catalyst	**Biodiesel yield (%)**		
Dosage (%)	n-CaO	n-CaO/CuO	n-CaO/ZnO
0.45	71.90	86.70	88.70
0.65	80.70	89.70	91.70
0.85	79.10	82.20	85.20
1.05	87.70	88.30	93.40
1.25	80.70	89.30	90.6
Mean	80.02	87.24	91.52

Optimization of Reaction Conditions

Using the factorial experimental design, it was demonstrated that not all four independent variables significantly affected biodiesel yield at the 95 % confidence level ($p < 0.05$). The analysis of variance (ANOVA) revealed that interactions between the methanol-to-oil molar ratio and catalyst loading ($F = 56.13$), molar ratio and time ($F = 104.17$), temperature and time ($F = 15.70$), and the three-way interaction among mole ratio, temperature, and time ($F = 7.09$) had significant effects on biodiesel yield. Additionally, the individual effects of catalyst loading ($F = 51.02$) and reaction time ($F = 67.14$) statistically has the most significant influence on the biodiesel yield. In contrast, oil-to-methanol ratio ($F = 4.71$), temperature ($F = 3.07$), and their interaction ($F = 0.4343$) did not show statistically significant influence on the yield ($p > 0.05$). The optimal conditions for biodiesel production were determined to be a methanol-to-oil molar ratio of 12:1, catalyst loading of 6.0 wt%, reaction temperature of 40 °C, and reaction time of 90 minutes. Under these conditions, the maximum biodiesel yield of 94.76 % was achieved. Figure 5 and 6 illustrate how interactions between production parameters affects the biodiesel yield.



(A) (B)

Figure 5: (A) Molar Ratio and Catalyst Level Shown against OBME Yield in a Three Dimensional Response Surface. (B) Molar Ratio and Temperature against OBME Yield in a 3D Response Surface

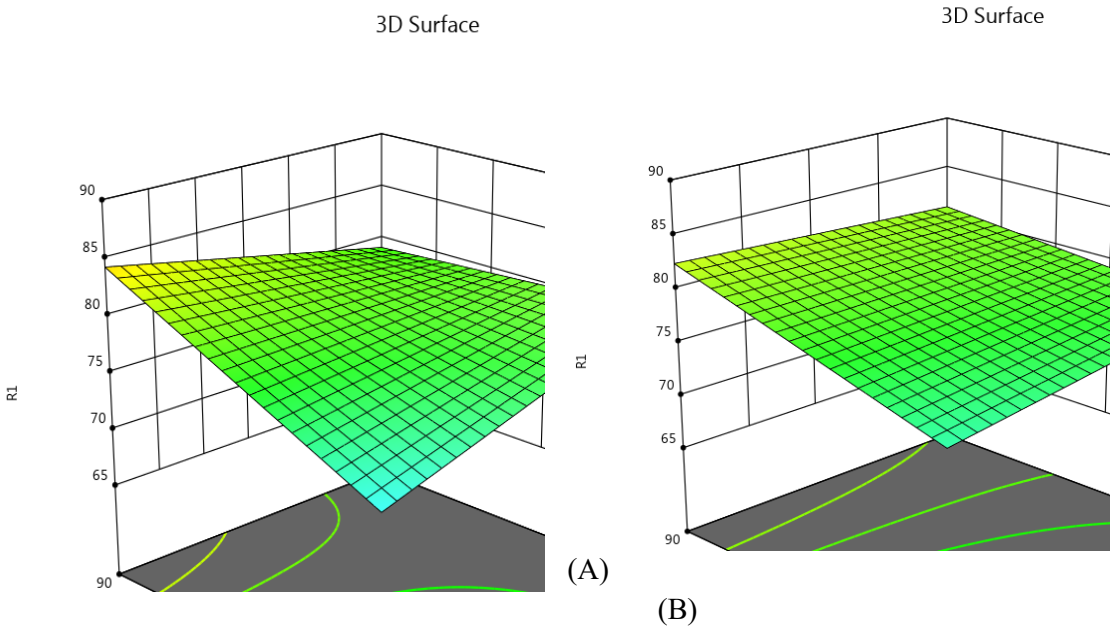


Figure 6: (A) Molar Ratio and Reaction Time Plotted in 3D against OBME Yield. (B) Plotting Temperature and Reaction Time in a 3D versus FAME Yield.

The optimized biodiesel met international standards, with a density of 0.812 kg/m³, viscosity of 4.39 mm²/s, flash point of 150 °C, calorific value of 41.31 and acid value of 0.23 mg KOH/g. The FTIR spectra of the final biodiesel as shown in Figure 7 confirmed the successful transesterification process. With the dominant ester carbonyl peak at 1726 cm^{-1}, indicating complete ester formation. Absorption at 1175 cm^{-1} corresponded to C-O stretching vibration, further verifying the biodiesel quality. This discovery aligns with the findings of Mokhtar *et al.* (2018). GC-MS analysis confirmed the presence of major FAME components, including methyl palmitate, methyl oleate, and methyl linoleate. The result showed a greater proportion of mono and poly unsaturated fatty acids (61.86 %) compared to saturated fatty acid (30.64 %). Increased levels of unsaturated fatty acids reduce viscosity (Deshmukh 2019). The high concentration of methyl oleate and methyl linoleate suggests that the biodiesel possesses excellent combustion properties and oxidative stability, making it a viable alternative to petroleum diesel

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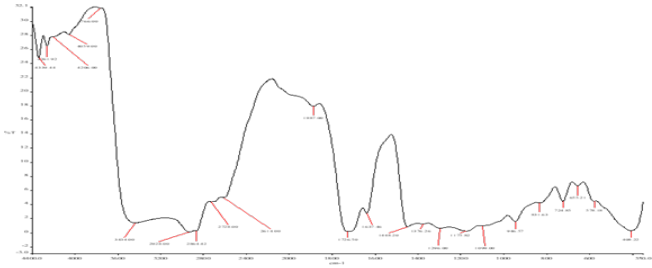

Figure 7: FTIR Spectrum of Optimal Blend Methyl Ester (OBME).

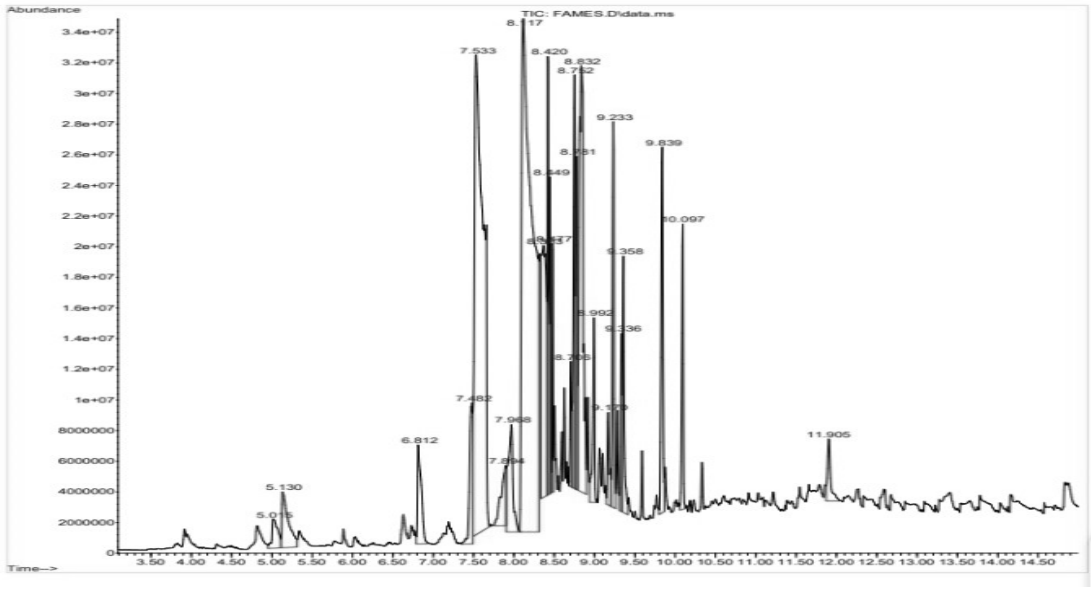

Figure 8: GC-MS Chromatograph of OBME.

Conclusion

This study demonstrated the feasibility of producing high-quality biodiesel from underutilized vegetable oil blends—specifically jatropha oil and waste cooking oil—using mixed metal oxide-based nanocatalysts. The synthesized nanocatalysts (n-CaO, n-CuO/CaO, and n-CaO/ZnO) exhibited strong catalytic performance, with n-CaO/ZnO delivering the highest biodiesel yield and stability over multiple reuse cycles. Beyond confirming their catalytic efficiency, this research emphasizes the potential of nanocatalyst-driven processes in transforming low-cost, non-edible feedstocks into sustainable biofuels. The integration of catalyst optimization with oil blending strategies enhanced biodiesel quality, bringing it within ASTM specifications. Importantly, the findings have broader implications for scaling up biodiesel production in resource-limited settings, reducing dependency on fossil fuels while promoting energy diversification. The use of waste and non-edible oils also minimizes the food-versus-fuel conflict, enhancing economic and environmental sustainability. Future work should focus on techno-economic analysis, pilot-scale validation, and lifecycle environmental assessment of the process. Additionally, exploring the compatibility of these nanocatalysts with other regional feedstocks could further strengthen the case for decentralized, community-level biodiesel production.

Conflict of interest

There is no conflict of interest in this work.

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