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Original Research Article

Assessing the Potential of Heterogeneous Basic Catalysts for the Production of Biodiesel from Vegetable Oil Extracts

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Abstract

This study investigates the production of biodiesel, specifically fatty acid methyl esters (FAME), derived from waste orange peel oil (OPO) via transesterification. A mixed metal oxide heterogeneous catalyst was synthesized using a coprecipitation method in the presence of soluble sodium carbonate and evaluated for the conversion of OPO with methanol. The physicochemical properties of the catalyst were characterized using X-ray diffraction (XRD), Fourier transform-infrared (FT-IR) spectroscopy, scanning electron microscopy with energy-dispersive spectroscopy (SEM-EDS), and BET surface area analysis via N_2 adsorption-desorption at 77 K. Characterization results indicated that calcium oxide (CaO) particles formed and dispersed on the zinc oxide (ZnO) support, with higher ZnO content enhancing CaO dispersion. Under optimized transesterification conditions, the catalyst achieved a FAME yield of 73.04% at a CaO:ZnO ratio of 0.31 within 120 minutes. The resulting biodiesel exhibited a density of 0.872 g/cm^3 and a refractive index of 1.451. These findings demonstrate the potential of this mixed metal oxide catalyst for biodiesel production and highlight the importance of optimizing reaction parameters to enhance both yield and fuel quality.

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Keywords: Oil extraction; Biodiesel; Orange peel waste; Heterogenized catalysts; Transesterification; metal oxide

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1. Introduction

Rapid urbanization and industrialization are the main causes of the ongoing steady increase in energy demand [1]. Research has focused on renewable alternatives as global energy demand grows and fossil fuel supplies diminish [2]. Emissions from fossil fuels are increasing and affect the environment, human health, and the greenhouse effect. Furthermore, the supply of fossil fuels is low, and they are not renewable. Key elements of the energy transition, including developing energy technology, encouraging environmental sustainability, attaining clean energy, increasing energy efficiency, and combating climate change, are the focus of contemporary energy policy to address these issues [3]. These advancements have increased biomass utilization, as it is regarded as a renewable energy source with the potential to significantly contribute to the global primary energy supply, produce low-carbon emissions, and aid in mitigating climate change. One example of a biomass is biodiesel [4]. Currently, biodiesel, also known as fatty acid methyl esters (FAMEs) [5], is an alternative energy source produced from extracted oils from vegetable sources and animal fats [6,7]. This process employs various extraction methods, including distillation, ultrasonication, microwave techniques, Soxhlet extraction, and

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mechanical processes [8]. The biodiesel can be effectively manufactured via biotechnological techniques, such as transesterification, a chemical reaction of triglycerides and short-chain alcohols in the presence of catalysts to produce alkyl esters and glycerol [9]. Industrial biodiesel production primarily uses homogeneous base catalysts; however, separating these catalysts is challenging. Despite their widespread use, homogeneous base catalysts such as potassium hydroxide (KOH) and sodium hydroxide (NaOH) have several drawbacks, resulting in many problems, such as equipment corrosion, nonreusability, glycerol and biodiesel contamination, and sensitivity to free fatty acids (FFAs) and moisture [10,11]. Heterogeneous catalysts offer superior alternatives because of their lower production costs, ease of separation, lack of soap formation, reusability, reduced corrosiveness, and better environment [12,13].

Many works have been devoted to enhancing biodiesel production under optimum conditions. Taufiq-Yap *et al.* [14] tested the effectiveness of calcium-based binary oxide solid catalysts (CaMg and CaZn oxides) in the methanolysis of Jatropha curcas oil to produce biodiesel. The coprecipitation method was used to synthesize the catalysts, and the matching metal oxides were then created by calcination. The catalytic efficiency of CaMgO and CaZnO mixed oxides in the transesterification of Jatropha curcas oil was assessed and contrasted with that of reference catalysts (CaO, MgO, and ZnO). The results revealed that more than 80% JCO conversion was attained under ideal conditions. The catalysts also demonstrated good reusability, with CaMgO sustaining greater than 80% conversion over four cycles and CaZnO over three cycles. To estimate the potential of rocket seed oil for commercial biodiesel production, Tariq *et al.* [15] focused on converting it by base-catalyzed transesterification with alcohol into biodiesel. The physicochemical characteristics of rocket seed oil biodiesel suggest that it could be a useful alternative to petrodiesel. The production pathway of trace species detected in the diesel engine exhaust via biodiesel made from soybean oil is the main subject of a study by Wu and Lin [16]. Sodium hydroxide (NaOH) is commonly used as a catalyst. This process not only provides a renewable energy source but also helps minimize environmental pollution when utilized in diesel engines.

The study of Lee *et al.* [17] focused primarily on developing environmentally friendly heterogeneous alkaline-based mixed metal oxide (MMO) catalysts specifically designed for biodiesel production using crude jatropha oil as a feedstock. Several MMOs, such as MgO-ZnO, CaO-MgO, CaO-ZnO, and CaO-La_2O_3, have been created via a coprecipitation technique. The physicochemical characteristics of the catalysts were closely linked to the transesterification efficiency. The incorporation of different active metals in binary systems greatly affects the surface urea content, basicity, and stability of the catalyst. Among these materials, the most efficient catalyst for transesterification without the need for pretreatment is CaO-ZnO. Ouanji *et al.* [18] obtained 93.2% by weight conversion of waste cooking oil (WCO) into biodiesel using a base catalyst concentration of 1.2 wt% KOH, to produce a high-quality biodiesel suitable for use in diesel engines. Kataria *et al.* [10] focused on synthesizing a novel heterogeneous catalyst, utilizing zinc-doped calcium oxide, to enhance the transesterification process for biodiesel production from frying oil. The most effective catalyst was identified as the one prepared with 5 wt% zinc doped into CaO at 700 °C calcination temperature (5-ZnCaO-700). The optimal results achieved a yield of 98.5%. The studies of Sierra-Cantor *et al.* [19] examined mixed calcium and zinc oxide catalysts for biodiesel production from refined palm oil. Research has revealed that the alcohol/oil ratio is the most crucial factor in the transesterification, rather than the catalyst amount or Zn/Ca atomic ratio. The differences in yield between the catalysts were smaller than expected. The future improvements suggested by Sierra-Cantor *et al.* [19] should focus on optimizing calcium oxide dispersion to increase biodiesel production. A study conducted by Deep *et al.* [20] examined the use of orange peel oil and ethanol as alternative fuels for diesel engines, focusing on their performance and environmental impact. The results show that E20OPO80 has a slightly lower efficiency than D100 does, likely due to its higher orange peel oil content. For the production of biodiesel from waste cooking oil (WCO), Changmai *et al.* [21] developed a cost-effective and sustainable catalyst. This is achieved by synthesizing magnetic nanoparticles coated with Citrus sinensis peel ash (CSPA-Fe_3O_4), a biowaste-derived material. Using agricultural waste, such as rice husk, for catalyst production, as investigated by Foroutan *et al.* [22], offers a cost-effective solution and promotes waste management. Research indicates that rice husk ash (RHA) combined with CuO and K_2CO_3 (RHA/CuO/K_2CO_3) is a heterogeneous catalyst that is effective for biodiesel production. It achieved a maximum biodiesel yield of 99.2% from sunflower oil and 98.1% from castor oil. Ozor *et al.* [23] reported that an efficient solid basic catalyst was developed for fatty acid methyl ester (FAME) production through the transesterification of waste cooking oil (WCO) with methanol. The catalyst, derived from calcium oxide nanoparticles (CaOnp) obtained from oyster shells and chemically modified with silicon dioxide nanoparticles (SiO_2np), produced an impressive 97.8% yield under optimal conditions. This

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performance significantly exceeded that of unmodified CaO, which did not undergo pretreatment. Abdullahi *et al*. [24] investigated biodiesel synthesis from Allamanda seeds, yielding a 64% oil content via transesterification using a novel KOH-modified metakaolin catalyst. Through Box-Behnken design optimization, the model demonstrated a good fit (R^2 = 0.9123). This study demonstrated that Allamanda seed oil and kaolin-derived metakaolin can be effectively used for eco-friendly biodiesel production in Nigeria. Ngige *et al*. [25] optimized the production of biodiesel from paw paw seed oil through alkali-catalyzed transesterification. The optimal operating conditions for ameliorating biodiesel yield via response surface methodology (RSM) and the Box-Behnken design are identified. Agu *et al*. [26] investigated how leftover waste orange seeds (Citrus sinensis) can be turned into practical industrial bio-oil. This research highlights the potential of these waste seeds as sustainable substitutes for petroleum-based oils, offering a viable solution to environmental challenges such as climate change. Musyaroh *et al*. [27] explored sweet orange peel oil as an additive for low-octane gasoline blends. It examines the chemical composition of oil, highlighting key compounds such as limonene and eugenol, and investigates their impact on fuel properties, including the research octane number (RON), viscosity, density, and heating values. This investigation revealed that sweet orange peel oil serves as a viable additive for enhancing gasoline properties, combustion efficiency, and engine performance parameters while offering economic viability and environmental sustainability benefits.

The daily production of oranges is one of the most important fruits cultivated worldwide, and due to the seasonal consumption of fresh oranges, orange peels are widely available in large quantities. A few researchers focused on converting orange peel oil into fuel additives or engine-performance studies. There have been limited studies concerned with the transesterification of orange peel oil using a low-cost, reusable, heterogeneous mixed metal oxide catalyst. While CaO:ZnO mixed oxides have been reported as effective transesterification catalysts for other feedstocks such as palm kernel oil and refined palm oil [6,19], their performance with orange peel oil, a seasonal, low-cost feedstock with a distinct fatty acid profile compared with previously studied oils, has not been established; in particular, it is not known how the CaO:ZnO ratio governs oxide dispersion, surface area, and FAME yield for this specific feedstock. This is the knowledge gap that the present study addresses. Therefore, this research intends to determine how the oil extracted from orange peels could be converted into biodiesel using the Soxhlet extraction method. The objectives of this research are: (i) to physically characterize orange peel oil and its conversion into FAME through the transesterification process using a heterogeneously prepared CaO:ZnO catalyst, and (ii) to characterize the final fatty acid methyl ester produced. The results contribute to the development of more environmentally friendly biodiesel produced from orange peel oil by transesterification, offering significant advantages through catalyst selection and reaction sequence optimization. The present study focuses on identifying the optimal CaO:ZnO ratio for this feedstock under a single, literature-informed set of transesterification conditions, rather than on a full multi-parameter optimization; the individual effects of catalyst loading, methanol-to-oil ratio, reaction temperature, and reaction time were not investigated here and are identified as directions for future work.

2. Materials and Methods

2.1. Extraction of Orange Peel Oil

Orange peel oil (OPO) is a naturally occurring raw material that is converted into methyl ester [28]. OPO was obtained by extracting orange peels via the Soxhlet extraction method. Initially, the raw orange peels were thoroughly rinsed with running distilled water to eliminate dust and impurities, followed by a drying process. The washed, sun-dried (72-75 hours), and crushed orange peels were placed in a Soxhlet apparatus for oil extraction.

The system maintained a temperature of 60 °C for 4 hours, facilitating steaming and releasing the orange essence. The steam and aromatic orange vapors were then channelled into a cooling chamber, causing the steam to condense into liquid. The resulting mixture of condensed orange oil was gathered in a storage tank. Excess solvent was added to keep the oil below the Soxhlet, which was directly heated. Following extraction with n-hexane, the evaporation stage utilized a rotary evaporator at a temperature of 60-63 °C with constant agitation. The final oil product was stored in a tightly sealed bottle in a refrigerator, incorporating a small volume of solvent to ensure preservation until usage.

2.2. Catalyst Preparation

The calcium oxide and zinc oxide composite is prepared via co-precipitation of a mixture of two solutions: $Ca(NO_3)_2.4H_2O$ and $Zn(NO_3)_2.6H_2O$ (Merck, analytical grade). A solution of sodium carbonate (Na_2CO_3) is rapidly added under agitation, and the pH of the solution is adjusted between 7 and 8. Subsequently, the obtained mixture was stirred overnight at 70 °C.

A Teflon container was used in an autoclave, where the mixture was treated at 110 °C for 24

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hours. Following this, the mixture was subsequently filtered and washed with distilled water several times. The drying process at 120 °C was carried out in a thermal oven overnight. The final material was calcined at 650 °C for 4 hours. A white powder was obtained after calcination.

2.3. Catalyst Testing for the Production of FAME (Biodiesel)

Transesterification, which involves the reaction of triglycerides with methanol in the presence of a catalyst to produce fatty acid methyl esters (biodiesel), is a common approach for large-scale biodiesel production [8]. This study tested CaO:ZnO catalyst blends for their ability to catalyze the transesterification of orange peel oil triglycerides. The catalyst loading (5 wt%), methanol-to-oil molar ratio (3:1), reaction temperature (55 °C), and reaction time (120 min) used throughout this work were fixed at values commonly reported as effective for CaO- and ZnO-based heterogeneous catalysts in the literature [6,14,17]; these parameters were not varied independently in the present study, and their systematic optimization for orange peel oil is left for future investigation.

A mixture of the required amount of methanol and calcined catalyst (5 wt%) was stirred. In the next step, alcohol and catalyst were added to the oil, and the mixture was placed in a three-neck flask. Alcohol and oil at a molar ratio of 3:1 are not naturally miscible at room temperature, and the reaction is typically carried out at a relatively high temperature (55 °C) with continuous stirring. This increases the mass transfer between the alcohol and oil, allowing the reaction to proceed more effectively. The transesterification reaction was carried out for 120 min with continuous stirring at 350 rpm. After the transesterification reaction, centrifugation and rotary evaporation were used to isolate the FAME product from the catalyst and remove the methanol and water. The reaction mixture was transferred to a separating funnel and allowed to stand undisturbed for 24 hours. During this time, the liquid mixture separated into two layers. The bottom layer was yellow-colored glycerol. The top layer was the FAME, or biodiesel, which was the desired product. However, this FAME layer contained excess methanol that had not been fully consumed during the reaction [29]. This excess methanol was then distilled off and reused in subsequent batches, and magnesium sulfate was added as a drying agent to purify the biodiesel and remove residual water.

The final step involved weighing and characterizing the synthesized biodiesel samples. Various tests were conducted to analyze the physical and chemical properties of biodiesel, including gas chromatography to determine its composition, Fourier transform infrared spectroscopy (FT-IR), density measurements, and refractive index. The data obtained from these analytical techniques were compared against biodiesel quality standards from the United States (ASTM D6751) to evaluate compliance [30] for the assessment of key fuel performance metrics and suitability for commercial applications.

The yield was calculated as a percentage of the weight of the FAME produced compared to the initial amount of oil used [31]. This gravimetric yield reflects the mass of purified FAME recovered relative to the initial oil charge and is distinct from the fractional conversion of triglycerides, which was not independently quantified in this study; this distinction is maintained throughout the manuscript to avoid ambiguity between the two terms.

2.3.1. Mechanism of the transesterification reaction

The chemical reaction mechanism proposed by Karmakar and Halder [8], Roy *et al.* [29], and Naeem *et al.* [32] consists of three sequential, reversible reactions, as outlined in schemes of Equations (1-4).

From a stoichiometric perspective, each mole of triglyceride (TG), diglyceride (DG*), and monoglyceride (MG*) reacts with one mole of alcohol (R-OH), resulting in the formation of one mole of pure glycerol (GL) and three moles of fatty acid methyl ester (R_3-$COOCH_3$). The production of fatty acid methyl esters via this transesterification process involves three main steps: (i) adsorption of triglycerides onto the catalyst surface; (ii) surface reaction between the adsorbed triglycerides and the alcohol; and (iii) desorption of the glycerol byproduct from the catalyst surface.

$$TG + S \rightleftharpoons TG.S \quad (5)$$

$$R\text{-}OH + TG.S \rightleftharpoons GL.S + R\text{-}COOCH_3 \quad (6)$$

$$GL.S \rightleftharpoons S + GL \quad (7)$$

In these equations, S, TG.S, and GL.S represent the vacant catalytic surface site, the triglyceride adsorbed at the active site, and the glycerol adsorbed on the active site, respectively. This stepwise mechanism explains how the transesterification reaction converts the triglycerides and alcohol into the desired FAME product and glycerol [29,31].

3. Results and Discussion

3.1. Determination of Physical Properties

The physical properties of both the raw material and its fatty acid methyl ester are described as follow:

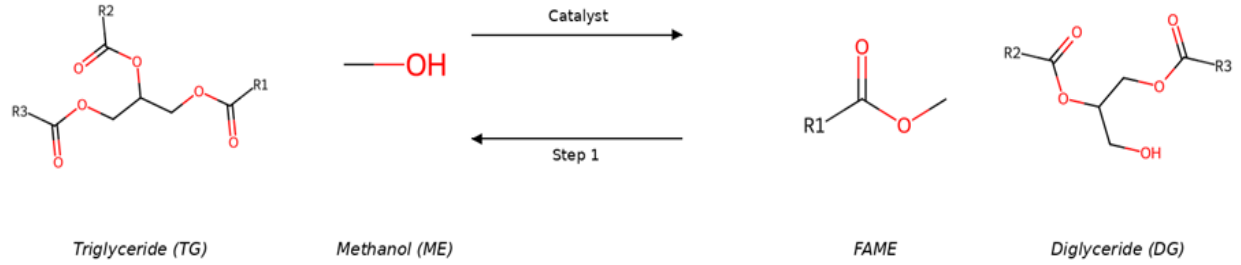
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Density (kg/m^3): The density was determined at 15 °C by weighing an empty pycnometer using an analytical balance. Subsequently, FAME was introduced into the pycnometer, and its weight was measured. The ratio of the mass of substance to the volume of the liquid was then calculated to obtain the density.

Refractive Index: The refractive index is a crucial physical property of a compound. These measurements are key in determining solution concentrations, assessing purity, and identifying compounds. The refractive index is susceptible to the solution purity. This study determined the refractive index for the raw material (OPO) and fatty acid methyl ester using a refractometer (KRUSS OPTRONIC, accuracy ±0.01) at 40 °C.

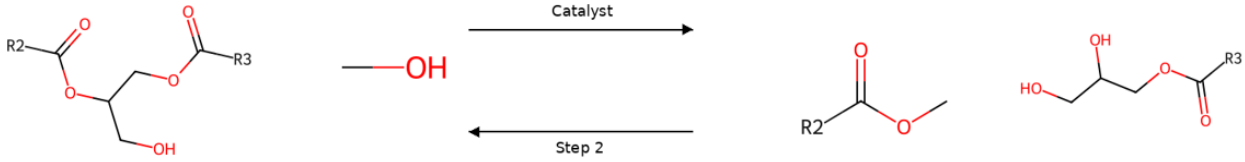
Potential for Hydrogen (pH): For pH, the oil and FAME were transferred individually to a clean, dry glass beaker, and hot distilled water was added while stirring continuously. The resulting mixture was then cooled in a cold water bath, after which the pH was determined using a calibrated pH meter equipped with a standard electrode. The measured pH values for oil and the synthesized methyl ester were 7.03 and 7.37, respectively. This aqueous-extraction pH measurement provides a qualitative, comparative indication of the acid–base character of the oil and

$$TG + R\text{-}OH \rightleftharpoons R_1\text{-}COOCH_3 + DG^* \quad (1)$$



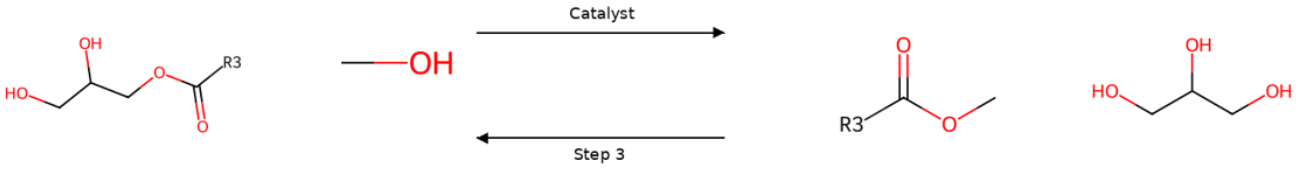
Triglyceride (TG) *Methanol (ME)* *FAME* *Diglyceride (DG)*

$$DG^* + R\text{-}OH \rightleftharpoons R_2\text{-}COOCH_3 + MG^* \quad (2)$$



Diglyceride (DG) *Methanol (ME)* *FAME* *Monoglyceride (MG)*

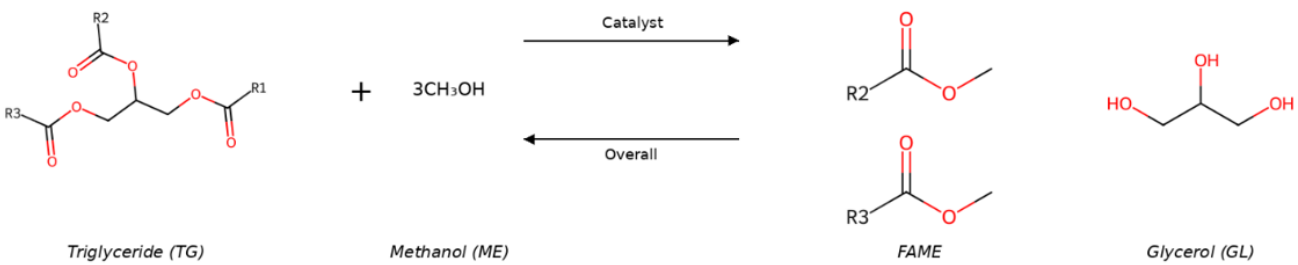
$$MG^* + R\text{-}OH \rightleftharpoons GL + R_3\text{-}COOCH_3 \quad (3)$$



Monoglyceride (MG) *Methanol (ME)* *FAME* *Glycerol (GL)*

The overall transesterification reaction can be summarized via the following equation:

$$TG + 3R - OH \rightleftharpoons 3R - COOCH_3 + GL \quad (4)$$



Triglyceride (TG) *Methanol (ME)* *FAME* *Glycerol (GL)*

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biodiesel; it is not a standard method for quantifying free fatty acids or acidity in fats and oils, and it does not substitute for the acid value titration prescribed in ASTM D664, which is recommended for a more rigorous assessment in future work.

The properties of orange peel oil as a feedstock and the resulting biodiesel are detailed in Table 1. The analysis revealed that the biodiesel meets ASTM D6751 (American Society for Testing and Materials) requirements, with its density falling within the specified range. The free fatty acid (FFA) content and moisture content of the orange peel oil, both of which strongly influence transesterification performance and the risk of soap formation, were not measured in this study; nor were other fuel-relevant properties such as viscosity, flash point, cloud point, acid value, and cetane number. These parameters were outside the scope of the present characterization and are acknowledged as a limitation; their determination is recommended for a fuller assessment of the biodiesel's compliance with ASTM D6751 in future work.

3.2. Characterizations of the Catalyst

3.2.1. Fourier Transform-Infrared Spectroscopy (FT-IR)

Figure 1 shows the FT-IR spectra of the CaO:ZnO catalyst samples with different ratios in the interval of 4000-400 cm^{-1} via an FT/IR-4700 type A JASCO spectrometer in transmission mode, to verify the presence of characteristic absorption bands of CaO and ZnO at a certain wavelength. Similarly, at an intensity ratio of 2.5, there is a significant peak intense at 1414.53, 872.63, 710.64 cm^{-1} attributed to the symmetric and asymmetric stretching vibrations of O-C-O bonds of unidentate carbonate at the reactive surface area of CaO with air during the calcination process, when considerable amounts of CO_2 and H_2O are produced [33]. For all the samples, the strong bands at 498.51-477 cm^{-1} could be attributed to the stretching vibration band of ZnO [34]. Therefore, both compounds can act as catalysts, as the FT-IR analysis shows characteristic bands of the two oxides, CaO and ZnO.

3.2.2. X-ray Diffraction (XRD)

Figure 2 shows the XRD diffraction pattern of the samples. The phases of the oxides of CaO and ZnO in the mixture CaO:ZnO were identified via X-ray powder diffraction (XRD) (EMPYREAN) via Cu-Kα radiation (λ = 0.15418 nm), with a scanning range of 10-100° (2θ) and a step size of 0.02°. The characterization results were compared with the CaO and ZnO standard diffractograms according to the Powder Diffraction File (PDF) database created by the Inorganic Crystal Structure for Diffraction Data (ICSD). Using the diffraction patterns of individual phases provided by the ICSD database, the crystalline phases of the samples were identified. The structural evolution of all three samples, i.e., CaO:ZnO, is shown in Figure 2.

The profiles revealed the presence of peaks corresponding to the ratio (2.5) of CaO to ZnO. The intensities of the diffraction peaks of CaO and ZnO were greater than those of the samples prepared with ratios of 0.31 and 0.63. The strongest diffraction peaks were recorded for the CaO and ZnO samples, which can potentially promote the growth of CaO and ZnO crystals. Peaks corresponding to ZnO appeared in the XRD patterns of all the samples; large crystals presented strong and narrow diffraction peaks. The peaks at angles 24°, 30.27°, 40.24°, and

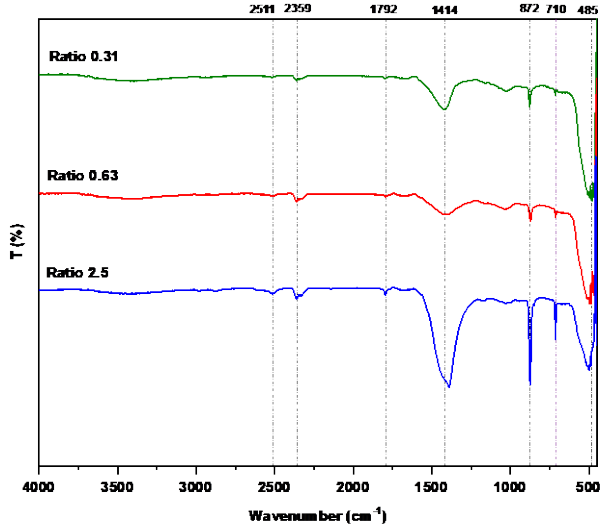


Figure 1. FTIR-ATR analysis of the CaO:ZnO catalyst (ratios of 0.31, 0.63, and 2.5).

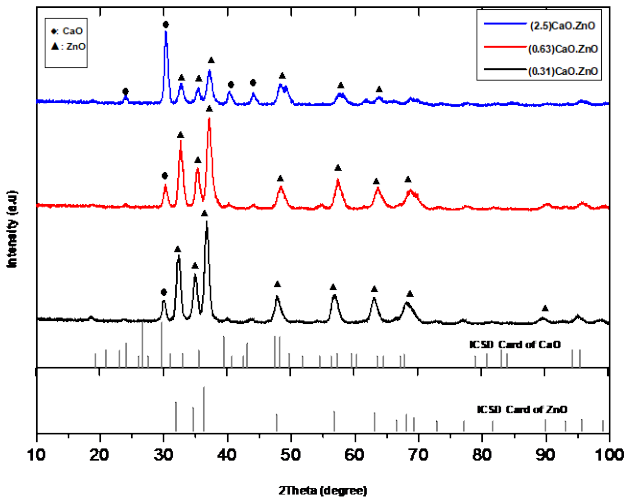


Figure 2. X-ray diffraction patterns of samples with different CaO: ZnO ratios.

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44.01° corresponded to the CaO phases, and peaks at 32.35°, 35.43°, 37.06°, and 68.09° matched the hexagonal phase of ZnO.

Compared with those of the primary peak, the diffraction peaks corresponding to the CaO phases with ratios of 0.63 and 0.31 are slightly lower in intensity than those corresponding to the 2.5 ratio. Moreover, for ZnO, a high peak intensity was observed in the XRD pattern at ratios of 0.63 and 0.31.

3.2.3. Scanning Electron Microscopy (SEM-EDS)

The surface morphology of the catalysts was thoroughly investigated by scanning electron microscopy. A Scios2 scanning electron microscope (SEM), integrated with an energy-dispersive spectroscopy (EDS) detector, was utilized for morphological examination and elemental analysis at ambient temperature. During image acquisition, the SEM was operated at an acceleration voltage ranging from 5 to 10 kV, maintaining a working distance of 7.1-7.3 mm.

The CaO:ZnO catalyst displayed a distinct morphology characterized by granularity and porosity. Its structure consists of aggregated units, forming an interconnected network resembling a porous mesh. This architectural arrangement increased the surface area, promoting more effective contact between the catalyst and reactants. The disparity became more evident as the desired mixed oxide ratio increased. Current preparation methods suggest that Zn^{2+} precipitates significantly faster than Ca^{2+} [6]. However, XRD analysis of the final CaO:ZnO catalyst revealed no additional oxide structures. The XRD patterns of the CaO:ZnO precipitates calcined at 650 °C showed only diffraction peaks for CaO and ZnO, resembling those of pure calcined CaO and ZnO, respectively.

This suggests that Ca and Zn mixed oxides do not form specific structures but exist as separate oxide clusters within precipitate particles. This aligns with the results of morphological studies by scanning electron microscopy (SEM-EDS), as shown in Figures 3-6. For ratios of 0.31 and 2.5, two particle types were observed: small, spherical particles similar in structure to pure ZnO, and larger particles resembling pure CaO in shape and size. These findings confirm the results of Ngamcharussrivichai *et al*. [6], indicating that the smaller particles are predominantly ZnO, whereas the larger ones are mainly CaO.

The energy-dispersive spectroscopy (EDS) microanalysis allowed for targeted examination of the sample area and shows the elemental distributions for the CaO:ZnO catalyst calcined at 650 °C, revealing that Ca, Zn, and O are present on the surface. The presence of the element carbon is due to the carbon support.

3.2.4. Surface analysis

The surface area and textural properties of the synthesized catalyst were measured via the Brunauer-Emmett-Teller (BET) method with a nitrogen gas (N_2) adsorption/desorption isotherm analyzer (Micromeritics, ASAP 2020). Before analysis, the catalyst was degassed at 300 °C for 5 hours to eliminate moisture and foreign gases. N_2 adsorption-desorption isotherms and the corresponding pore size distribution curves, calculated via the Barrett-Joyner-Halenda (BJH) method, are presented in Figure 7(a) and (b) for the catalyst. The surfaces were examined in a vacuum chamber at 77 K (-196 °C) over a relative pressure range of $10^{-3} \leq P/P_0 \leq 1$. The isotherms, when analyzed via the BET method, fit the characteristics of type IV isotherms in the IUPAC classification [35], indicating multilayer adsorption and H3 hysteresis loops, which are characteristic of mesopore filling and emptying through capillary condensation [36]. Using the experimental data obtained from the adsorption of N_2 at 77 K, the pore size distribution curve (shown in the insert of Figure 7) reveals a prominent peak in the 2.0-50 nm range for the CaO:ZnO catalyst, confirming its mesoporous structure [12,37]. Through these pores, OPO readily diffuses into and out of the catalyst, increasing its catalytic activity. The analysis revealed BET-specific surface areas of 12.929 m^2/g and 8.123 m^2/g, and BJH desorption pore volumes of 0.0785 cc/g and 0.0552 cm^3/g at 0.99 relative pressure, for ratios of 0.31 and 2.5, respectively. These results indicate that the catalyst has a high surface area and mesoporous structure, potentially enhancing the interface between the catalyst and solution by increasing the number of active contact sites. The results show that a ratio of 0.31 for the mixed oxides produces a moderate specific surface area (12.929

Table 1. Properties of orange peel oil and biodiesel products.

Parameters	OPO	Biodiesel product	ASTM specification
Density (g/cm^3, 15 °C)	0.96	0.872	0.870-0.890
Refractive Index (23 °C)	1.474	1.451	-
Solubility	Insoluble	Insoluble	-

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m^2/g) compared to a ratio of 2.5. The specific surface area plays a significant role in enhancing the conversion of triglycerides into methyl esters (biodiesel), thereby reducing the amount of unreacted oil and improving overall conversion efficiency.

Taken together, the XRD, SEM-EDS, and BET results indicate that the 0.31 CaO:ZnO ratio combines a higher proportion of finely dispersed, accessible CaO with a larger surface area than the 2.5 ratio, providing a plausible physicochemical basis for its higher FAME yield: a larger surface area and finer oxide dispersion are expected to expose more catalytically active sites and improve contact between the solid catalyst and the oil/methanol phases. However, the surface basicity of the catalysts (the quantity, strength, and accessibility of basic sites) was not directly measured in this study, for example by Hammett indicator titration or CO_2 temperature-programmed desorption; the link proposed here between CaO dispersion and catalytic activity is therefore inferred from structural and textural characterization rather than confirmed by direct basicity measurements, and this should be regarded as a limitation of the present characterization.

3.3. Characterization of the Biodiesel Product

Fourier transform infrared (FT-IR) spectroscopy was used to identify functional groups, further confirming the structural characteristics of the biodiesel. The results of the FT-IR analysis of orange peel oil and FAME are presented in Figure 8. For OPO, the band intensity between 3550 and 3000 cm^{-1} indicates the presence of stretching vibrations of hydroxyl groups (-OH). These results are attributed to the free-OH groups in carbohydrate and phenolic groups in fruit peels [38]. The characteristic absorption bands indicating the symmetric and asymmetric stretching of C-H in CH_2 and CH_3 groups were observed between 2922.59 and 2853.17 cm^{-1} [26,39]. The presence of an alkane is supported by two IR absorption peaks at

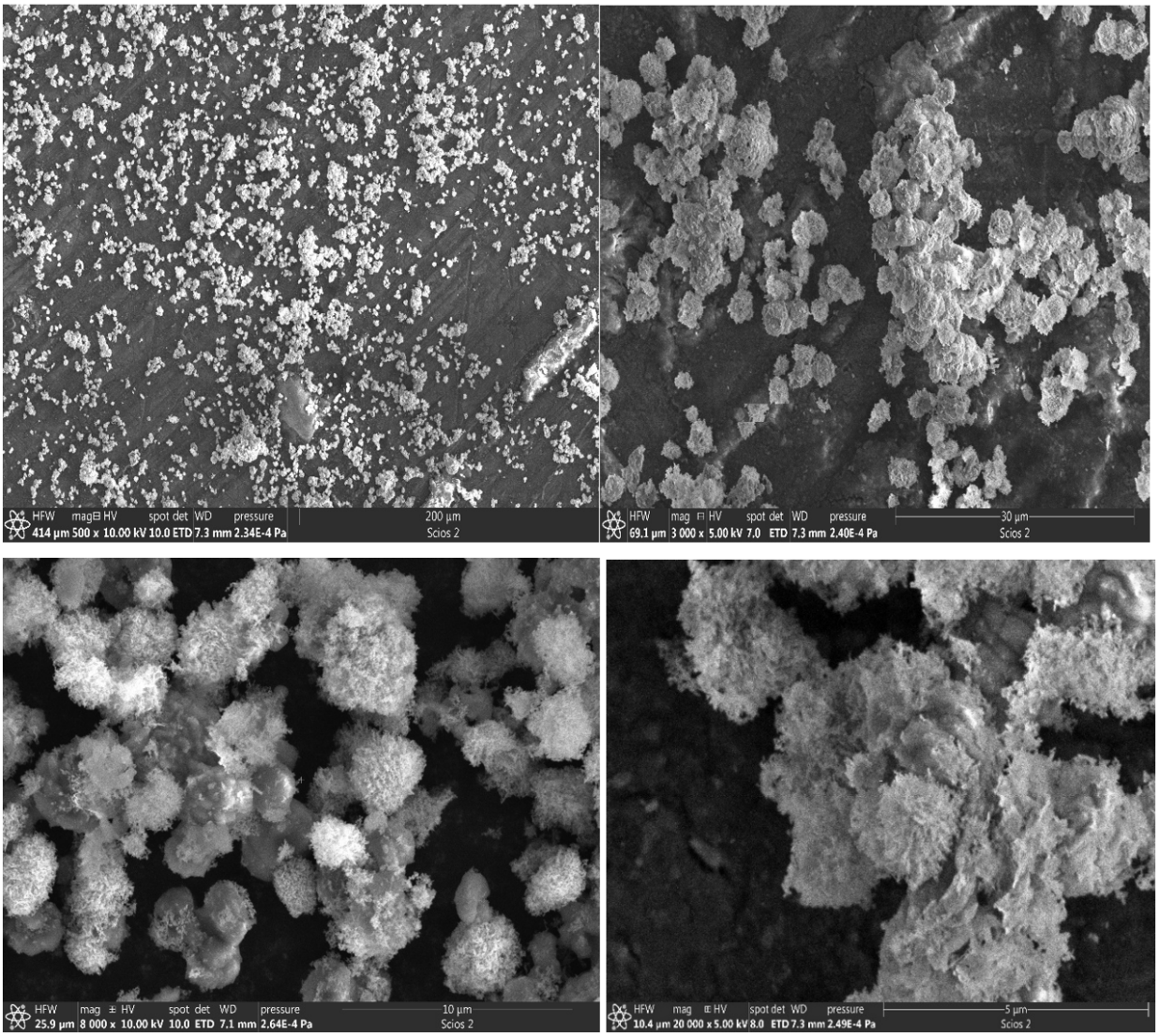


Figure 3. SEM micrographs of the catalyst at magnifications of 500X, 3000X, 8000X, and 20000X (CaO:ZnO ratio of 0.31).

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1462.74 and 1375.96 cm^{-1}, which arise from the asymmetric deformation vibrations of methyl (CH_3) groups [26]. The peak at 1640.16 cm^{-1} is due to the symmetrical stretching vibration of the carboxylic acid group [38,40] (not present in biodiesel). The band between 473 and 723 cm^{-1} (at 529.36 cm^{-1}) was attributed to the stretching of C-H [41].

For fatty acid methyl ester, an IR spectral peak at 723.1 cm^{-1} corresponds to CH_2 methylene group out-of-plane bending, which is indicative of olefinic compounds [26]. In the infrared spectrum, biodiesel methyl esters produce a distinctive absorption band at 1032 cm^{-1} (not present in pure samples). These bands are created by the asymmetric stretching movement of the carbon-oxygen (C-O) chemical bond [42], representing the methyl group stretch assigned to O-CH_3 [43]. The less intense absorption band between 3550 and 3000 cm^{-1} in the spectrum of biodiesel indicates that the orange peel oil-derived biodiesel contains minimal moisture. This is a positive finding, as water content is an important quality parameter for biodiesel.

Gas chromatography-mass spectrometry (GC-MS) was used to analyze the compound

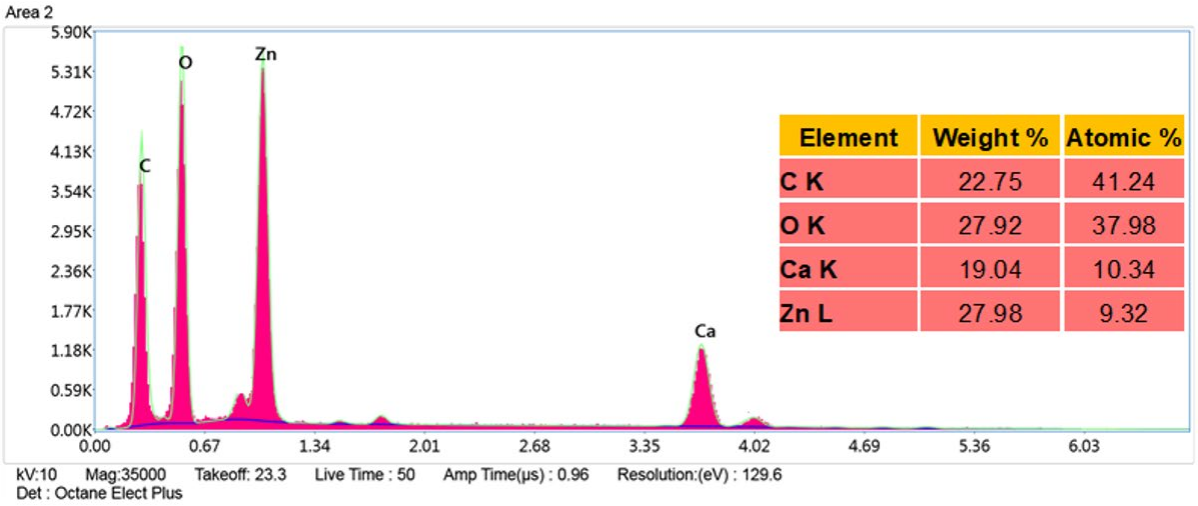


Figure 4. EDS spectrum of the catalyst (CaO:ZnO ratio of 0.31).

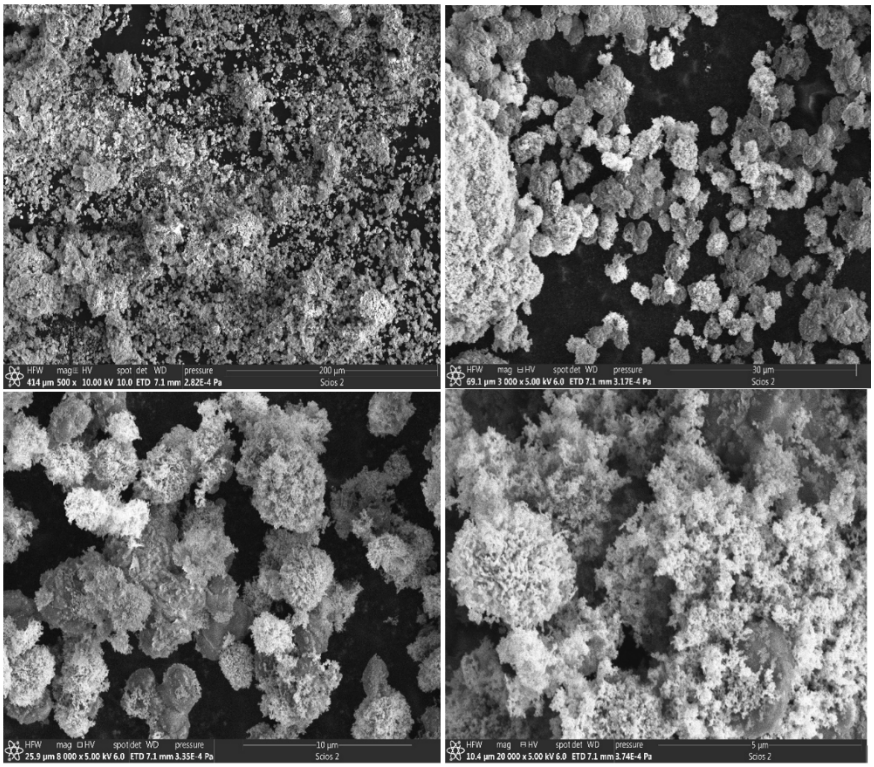


Figure 5. SEM micrographs of the catalyst at magnifications of 500X, 3000X, 8000X, and 20000X (CaO: ZnO ratio of 2.5).

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composition of the produced biodiesel. The synthesized fatty acid methyl ester components were identified via gas chromatography coupled with a mass spectrometer (GC-MS, Bruker) to determine the compounds formed during transesterification. The gas chromatography (GC) tool separates chemical compound components for analysis, whereas the mass spectrometry (MS) tool identifies them. The fatty acid methyl ester (biodiesel) was produced using a 5 wt% catalyst, with a 3:1 molar ratio of alcohol to orange peel oil, and was allowed to react for 120 minutes. The results produced spectrum peaks from an injected sample volume of 1 µL. Additionally, the GC-MS analysis provided information on the molecular structure and molecular weight of each constituent component of the FAME. The resulting chromatogram from the GC-MS analysis is listed in Table 2. The primary constituents of the fatty acid methyl ester products are 7,10-hexadecadienoic acid methyl ester ($C_{17}H_{30}O_2$), 9,12-octadecadienoic acid methyl ester ($C_{19}H_{34}O_2$, methyl linoleate), and 6-octadecenoic acid methyl ester, Z-isomer ($C_{19}H_{36}O_2$, methyl petroselinate / methyl oleate). The occurrence of non-FAME components is likely due to incomplete transesterification during the reaction and

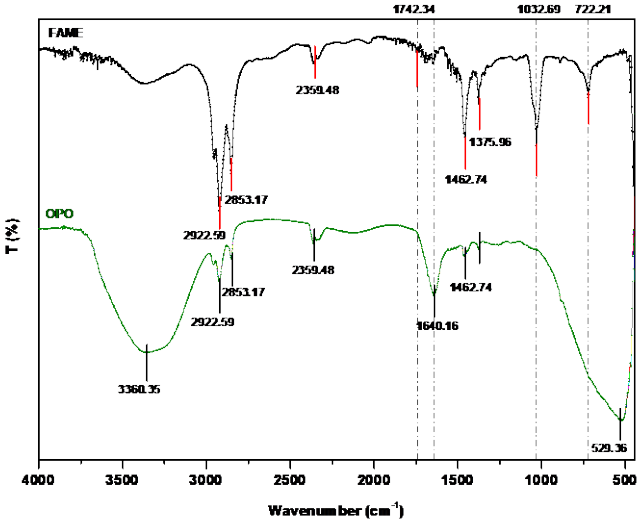


Figure 8. FT-IR analysis of OPO and FAME.

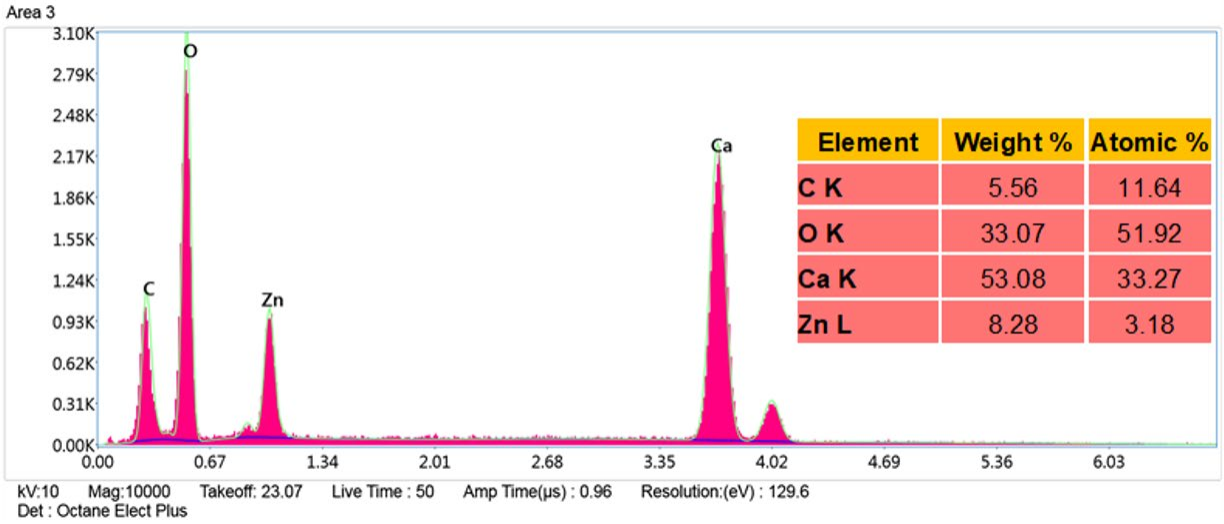


Figure 6. EDS spectrum of the catalyst (CaO:ZnO ratio of 2.5).

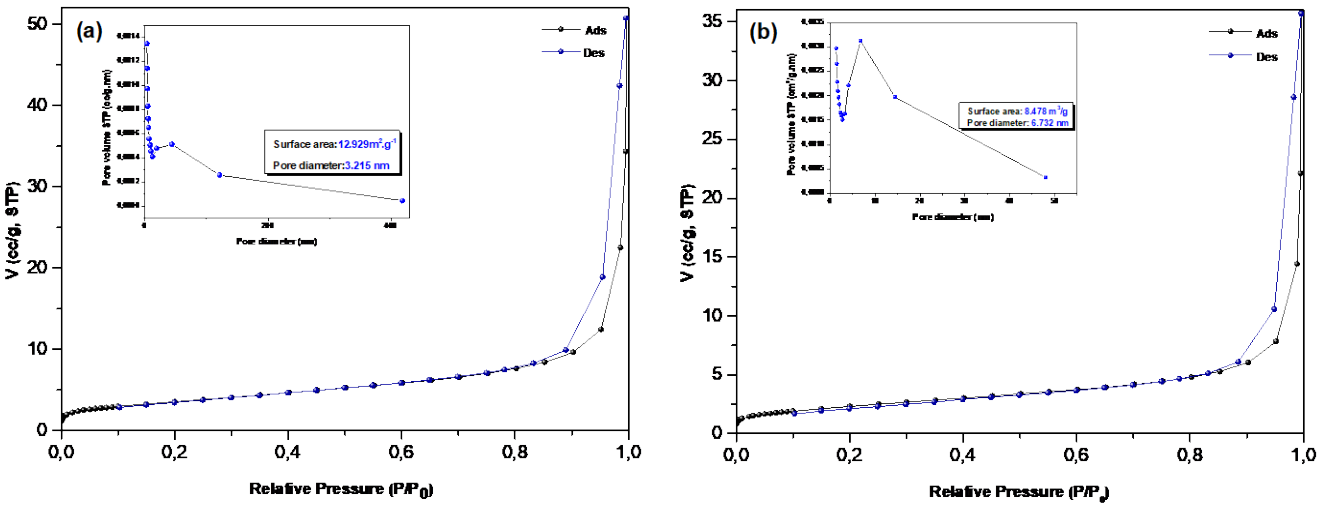


Figure 7. The nitrogen adsorption-desorption isothermal curves of the as-synthesized catalysts, (a) ratio of 0.31 and (b) ratio of 2.5. The insert illustrates the pore size distribution as determined by the BJH method.

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inadequate purification of the biodiesel after production. It should be noted that the area percentages reported in Table 2 are derived directly from GC-MS peak areas without the use of external calibration standards or compound-specific response factors; they therefore provide a semi-quantitative rather than absolute measure of composition, and quantification against certified reference standards is recommended for future analyses.

3.4. Effect of the Catalyst Ratio

Figure 9 shows how the catalyst ratio affects the OPO transesterification reaction. The FAME content was significantly dependent on the CaO:ZnO ratio. With a ratio of 2.5, minimal FAME production was observed. However, the FAME content significantly increased to 73.04% when the Zn amount (0.31 ratio) increased. The formation of Ca-Zn mixed oxides, most likely resulting from an increased surface area and the complete breakdown of mixed carbonate precursors, which produced active CaO sites (as shown in the XRD data), was responsible for this increase in transesterification activity. These results showed that the most efficient catalyst for the transesterification of orange peel oil was a CaO:ZnO ratio of 0.31.

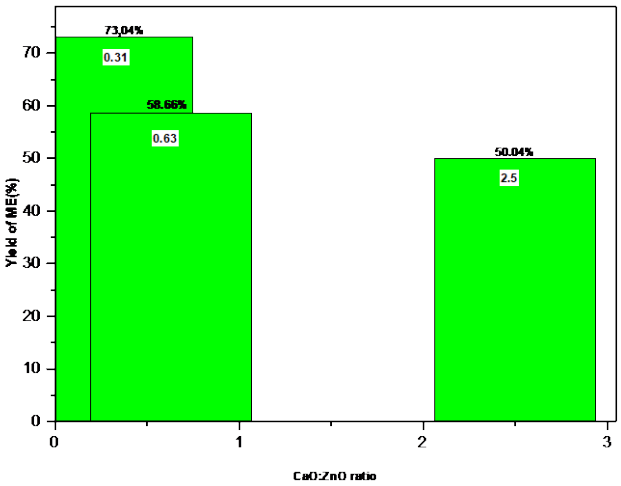


Figure 9. Effect of the catalyst ratio on the FAME yield (temperature reaction of 55 °C).

3.5. Comparison with Previously Reported Catalysts and Study Limitations

To place the present results in context, Table 3 compares the catalyst and conditions used in this work with previously reported heterogeneous (and one homogeneous) basic catalysts for biodiesel production, drawing on the studies discussed in the Introduction. As shown in Table 3, the FAME yield obtained in this work (73.04%) is lower than the yields reported for several CaO- or ZnO-based and other heterogeneous catalysts applied to other feedstocks (93–99%) [10,18,22,23]. However, a direct comparison of yield alone is not sufficient to assess catalytic performance, since these studies differ in catalyst loading, alcohol-to-oil ratio, reaction temperature, and time, and several used refined or pretreated feedstocks with lower free fatty acid and moisture content than the crude orange peel oil used here. The lower yield obtained in the present study may in part reflect the literature-derived, fixed reaction conditions used rather than conditions optimized specifically for this feedstock (Section 2.3), together with the unquantified FFA and moisture content of the orange peel oil (Section 3.1), both of which are known to affect transesterification efficiency [10, 11]. Reaction kinetics and the activation energy of the transesterification reaction were not determined in this study; their determination

Table 2. GC-MS identification of biodiesel derived from orange peel oil.

Peak	R Time (min)	Area (%)	Amount	Compound Name
1	2.452	0.35	0,347	beta-Pinene
2	3.007	16.79	16.794	Cyclohexene, 1-methyl-5-(1-methylethenyl)
3	6.769	0.57	0.572	2,4-Decadienal, (E, E)
4	7.095	0.95	0.949	2,4-Nonadienal, (E, E)
5	14.106	5.62	5.624	Tridecanoic acid, methyl ester
6	15.730	37.13	37.130	9,12-Octadecadienoic acid, methyl ester
7	15.796	15.55	15.549	6-Octadecenoic acid, methyl ester, (Z)
8	16.004	3.14	3.140	Tridecanoic acid, 12-methyl-, methyl ester
9	16.133	5.30	5.300	Linoelaidic acid
10	16.187	2.30	2.300	cis-Vaccenic acid
11	19.341	4.52	4.523	Phthalicacid, di(hept-3-yl) ester
12	20.522	1.67	1.673	7,10-Hexadecadienoic acid, methyl ester
13	20.557	1.67	1.666	2-Methyl-Z, Z-3,13-octadecadienoic
14	21.408	0.45	0.453	Squalene
15	23.514	1.16	1.156	1,6-Dimethyl-5-oxo-1,2,3,5-tetrahydroimi
16	25.074	2.82	2.824	3-Oxatricyclo [20.8.0.0(7,16)] triaconta-1

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would provide further mechanistic insight into the observed yield and is left for future work.

Catalyst recyclability is a key advantage claimed for heterogeneous catalysts and was demonstrated for related CaO-based mixed oxides in the literature, for example, CaMgO and CaZnO catalysts retained more than 80% conversion over four and three reaction cycles, respectively, when applied to Jatropha curcas oil [14]. Reuse experiments were not performed for the CaO:ZnO catalyst in the present study, and this is acknowledged as a significant limitation, since recyclability directly affects the practical and economic viability of the catalyst. Likewise, the possible leaching of Ca or Zn species into the reaction mixture, which can complicate the distinction between genuinely heterogeneous and partially homogeneous catalytic behavior, as previously investigated for calcium–zinc mixed oxides in palm oil transesterification [19], was not examined here. Catalyst reuse over multiple cycles and quantification of metal leaching (for example, by atomic absorption spectroscopy or ICP-OES analysis of the recovered biodiesel and glycerol phases) are recommended as priorities for future work on this catalytic system.

Scaling up this transesterification process would require addressing several practical considerations beyond those examined at laboratory scale, including efficient mixing and mass transfer between the immiscible oil, methanol, and solid catalyst phases at larger reactor volumes; the energy cost of the 650 °C calcination step used in catalyst preparation; the additional purification needed to remove the co-extracted non-FAME compounds evident in the GC-MS profile (Table 2); and the consistency of orange peel oil supply and composition across batches, given its origin as a seasonal agricultural waste stream. These factors were not evaluated experimentally in this work but are relevant considerations for future process-scale studies.

4. Conclusion

In this study, CaO:ZnO catalysts with ratios of 0.63, 0.31, and 2.5 were synthesized, and their catalytic performance in the production of fatty acid methyl esters (FAME) from orange peel oil (OPO) via the transesterification reaction was thoroughly evaluated. With a 0.31 ratio of

Table 3. Comparison of the CaO:ZnO catalyst used in this study with previously reported heterogeneous (and one homogeneous) basic catalysts for biodiesel production.

Catalyst	Feedstock	Key reaction conditions	Reported yield / conversion	Ref.
CaMgO / CaZnO (binary oxides)	Jatropha curcas oil	Coprecipitation-derived catalyst, methanolysis	>80% conversion; CaMgO reused >80% over 4 cycles, CaZnO over 3 cycles	[14]
CaO-ZnO (mixed metal oxide)	Crude jatropha oil	Coprecipitation, no pretreatment needed	Most efficient of the MMOs tested (yield not quantified in text)	[17]
KOH (homogeneous)	Waste cooking oil	1.2 wt% KOH	93.2 wt% conversion	[18]
5 wt% Zn-doped CaO, 700 °C calcination	Frying oil	Zinc-doped CaO	98.5% yield	[10]
Ca-Zn mixed oxides (various ratios)	Refined palm oil	Various Zn/Ca atomic ratios	Yield differences smaller than expected; alcohol/oil ratio more critical than catalyst amount or Zn/Ca ratio	[19]
CaOnp/SiO_2np (modified CaO nanoparticles)	Waste cooking oil	Oyster-shell-derived CaO modified with SiO_2 nanoparticles	97.8% yield	[23]
RHA/CuO/K_2CO_3	Sunflower oil / castor oil	Rice-husk-ash-derived heterogeneous catalyst	99.2% (sunflower); 98.1% (castor)	[22]
CaO:ZnO, ratio 0.31 (this work)	Orange peel oil	Coprecipitation; 5 wt% catalyst, 3:1 methanol:oil, 55 °C, 120 min (fixed, not optimized parametrically)	73.04% FAME yield	This study

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catalyst, the optimum results were obtained, achieving a FAME content of 73.04% under optimized transesterification conditions, including a reaction temperature of 55 °C, catalyst loading of 5 wt%, a methanol-to-oil molar ratio of 3:1, and a reaction time of 120 minutes. The findings revealed that the mixed oxide with a ratio of 0.31 had a significantly greater surface area (12.929 m^2/g) than that with a ratio of 2.5, underscoring the influence of composition on the surface characteristics of the catalyst. A greater surface area can potentially enhance the interface between the catalyst and the solution by increasing the number of active contact sites. Gas chromatography coupled with mass spectrometry (GC-MS) was employed to analyze the biodiesel and determine the compositional profile of the fatty acid esters formed. The analysis revealed that the biodiesel comprised approximately 37.13 wt% 9,12-octadecadienoic acid methyl ester ($C_{19}H_{34}O_2$), 15.54 wt% 6-octadecenoic acid methyl ester, Z-isomer ($C_{19}H_{36}O_2$), and 1.67 wt% 7,10-hexadecadienoic acid methyl ester ($C_{17}H_{30}O_2$). This demonstrates the catalyst's effectiveness and highlights its potential for efficient biodiesel production. While these results confirm the feasibility of using a CaO:ZnO heterogeneous catalyst for orange peel oil transesterification, several aspects were not addressed in the present study and are identified as priorities for future work: quantification of the free fatty acid and moisture content of the feedstock; determination of reaction kinetics and activation energy; assessment of catalyst recyclability and metal leaching over multiple reuse cycles; direct measurement of surface basicity; replicate measurements with reported standard deviations; and a systematic evaluation of catalyst loading, methanol-to-oil ratio, reaction temperature, and reaction time beyond the single condition set used here (Section 3.5). Addressing these points would strengthen the structure–activity understanding of this catalytic system and its comparison with other heterogeneous basic catalysts reported for biodiesel production.

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CRediT Author Statement

Author Contributions: Ghania Mecheri: Conceptualization, Methodology, Formal Analysis, Data Curation, Writing – Original Draft, Writing – Review and Editing. Assia Sid: Conceptualization, Investigation, Supervision, Writing – Review and Editing. Noureddine Gherraf: Conceptualization, Supervision, Writing – Review and Editing. Ahcene Bouchemma: Conceptualization, Investigation, Writing – Review and Editing. All authors have read and agreed to the published version of the manuscript.

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Declarations of Conflict of Interest

The authors declare that they have no affiliations with financial or other personal interests of any organization with any financial interest in the subject matter or materials discussed in this manuscript.

Declaration of Generative AI Use

During the preparation of this manuscript, the author(s) used "claude AI" solely to check the readability and language of the text. After using this tool, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the published article.

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