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# Microwave-Assisted Transesterification of Waste Cooking Oil Using Modified CaO Catalyst for High-Purity Biodiesel Production

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**Abstract**

Biodiesel is a renewable alternative fuel capable of partially or fully replacing conventional diesel in compression ignition engines. Waste cooking oil (WCO) represents a sustainable feedstock for biodiesel production; however, its high free fatty acid (FFA) content must be reduced below 1% to prevent emulsion formation during methanolysis. In this study, WCO was pretreated and converted into biodiesel *via* a two-stage process (esterification followed by transesterification) using a microwave-assisted heating system and ammonium carbonate-modified CaO catalyst. The effects of reaction time (2–10 min) and catalyst loading (1–5 wt%) on biodiesel yield and quality were investigated. Under optimal conditions, a biodiesel yield of 82.80% was achieved at 4 wt% catalyst and 6 min reaction time. The resulting biodiesel exhibited a density of 0.8617 g/mL, kinematic viscosity of 2.863 $mm^2/s$, and a calorific value of 36.78 kJ/g (8.81 kcal/g), all complying with SNI 7182:2015 standard. Gas chromatography-mass spectroscopy analysis confirmed the formation of high-purity fatty acid methyl esters, with a total methyl ester content of 97.71%, dominated by oleic, palmitic, linoleic, stearic, and ricinoleic methyl esters. These findings indicate that microwave-assisted transesterification with modified CaO catalyst is an efficient, rapid, and sustainable method for producing high-quality biodiesel from WCO, suitable for application as a renewable diesel alternative.

**Keywords**

waste cooking oil, biodiesel, microwave-assisted transesterification, modified CaO catalyst, fatty acid methyl esters

## 1 Introduction

Indonesia dependence on fossil-based fuels continues to be a major barrier to achieving national energy resilience and decarbonization. According to the Ministry of Energy and Mineral Resources (EMR), 42% of national energy consumption is still supplied by petroleum fuels, with diesel use showing a dramatic increase from 877000 $m^3$ in 2020 to 1.59 million $m^3$ in 2021, representing an 80.86% escalation [1]. Such a significant rise not only places pressure on finite fossil fuel reserves but also contributes to escalating greenhouse gas emissions and environmental degradation. These conditions have strengthened the strategic relevance of developing renewable and low-carbon fuels that are technically and economically viable for large-scale deployment. Biodiesel is widely recognized as one of the most promising alternatives due to its compatibility with existing diesel engines, biodegradability, and potential to reduce lifecycle emissions when produced from sustainable feedstocks [2].

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Among various renewable feedstocks investigated globally, waste cooking oil (WCO) has gained substantial attention, particularly in developing countries. A study conducted by the International Council on Clean Transportation (ICCT) reported that Indonesia produces approximately 1.64 billion liter of WCO annually, sourced from households, restaurants, hotels, and food service industries [3]. Utilizing WCO not only addresses waste management challenges but also reduces the environmental and health risks associated with dumping or reusing degraded oil [4, 5]. WCO typically contains high levels of free fatty acids (FFA), ranging from 5–30% (w/w) [6], which pose a barrier to direct transesterification because FFAs react with alkaline catalysts to form soap, reducing biodiesel yield. To overcome this challenge, a two-step approach is required: acid-catalyzed esterification to reduce FFA content below 1%, followed by alkaline-catalyzed transesterification to convert triglycerides into fatty acid methyl esters (FAME) [7, 8]. These consecutive reactions are essential to ensure acceptable biodiesel conversion efficiency and adherence to national and international quality standards.

Catalytic performance plays a decisive role in determining the efficiency, cost, and scalability of biodiesel production. While homogeneous catalysts remain widely used, heterogeneous catalysts have gained priority due to their reduced environmental footprint, ease of separation, reusability, and lower operating costs [9, 10]. There are many catalysts that have been applied for biodiesel production, one of which is calcium oxide, due to its high basicity, low cost, and wide availability [11, 12]. CaO is one of the most studied heterogeneous catalysts owing to its high basicity and suitability for transesterification. However, the reactivity of CaO can be further enhanced by modifying its surface structure to produce super-alkaline CaO-based catalysts. Super-alkaline CaO catalysts have shown exceptional catalytic activity, with biodiesel conversion rates reaching 99.9% and methyl ester contents between 98.82–99.07%. This modification is commonly achieved by contacting CaO with ammonium bicarbonate, forming $CaCO_3$, which thermally decomposes back into highly active CaO [13]. The efficiency of CaO is strongly correlated with its purity; higher-purity CaO typically results in faster reaction rates and delayed catalyst deactivation, making the evaluation of CaO purity an essential preliminary step in catalyst development [14, 15].

Process intensification has emerged as a promising strategy to improve biodiesel production efficiency, and microwave-assisted transesterification is among the most actively explored methods. Microwaves generate heat through dipolar polarization, ionic conduction, and interfacial polarization, enabling rapid volumetric heating and shorter reaction times compared with conventional heating [9, 16]. These characteristics can accelerate reaction kinetics and reduce energy consumption. However, excessive microwave power may cause undesirable effects, including methanol evaporation, overheating, and degradation of organic molecules, which collectively reduce biodiesel yield [17]. Oko et al. [8] demonstrated that biodiesel produced under extreme microwave power conditions (180–800 W for 4 min) failed to comply with the Indonesian biodiesel quality standard SNI 7182:2015 [18], highlighting the importance of optimizing microwave power and reaction time [17]. Therefore, fine-tuning microwave-assisted processes remains a critical research challenge. Ensuring that biodiesel meets the Indonesian National Standard (SNI 7182:2015) [18] is essential for its adoption as a commercial fuel. Quality parameters such as density, viscosity, cetane number, flash point, sulfated ash, sulfur content, acid number, glycerol content, and methyl ester composition must fall within strict limits to guarantee safe and efficient engine performance [2]. Analytical verification of biodiesel properties, especially methyl ester composition, is typically carried out using gas chromatography–mass spectrometry (GC–MS), a powerful analytical technique capable of identifying molecular structures based on mass fragmentation patterns [19]. Through GC–MS, the extent of transesterification can be precisely quantified, providing insights into reaction completeness and overall biodiesel quality.

Given the increasing availability of WCO in Indonesia, the need for more efficient catalyst systems, and the potential of microwave-assisted heating to improve processing efficiency, further exploration of optimized biodiesel production pathways is both timely and necessary. Therefore, this study aims to examine the effects of CaO-based heterogeneous catalyst loading and microwave-assisted transesterification reaction time on the physicochemical properties and overall quality of biodiesel derived from waste cooking oil. The outcomes are expected to strengthen the knowledge base on catalyst modification and alternative heating technologies, while providing practical recommendations for enhancing WCO-based biodiesel production in accordance with national quality standards.

## 2 Materials and methods

### 2.1 Materials

The primary feedstock consisted of used waste cooking oil collected from street vendors in the Rungkut district of Surabaya. The WCO was collected periodically (once every month) from the same group of vendors under similar operating conditions to minimize variability in feedstock quality. rior to use, the collected oil was filtered to remove food residues and stored under controlled conditions.

Analytical-grade methanol 99% from CV Nirwana Abadi Surabaya, calcium oxide from online shop ROFA Laboratorium Center, ammonium carbonate from online shop Nanyang Chemical, sulfuric acid from CV Nirwana Abadi Surabaya, activated charcoal from online shop Anugerah Abadi Kimia, and distilled water (aquadest) from CV Nirwana Surabaya were obtained from certified chemical suppliers in Surabaya. All chemicals were used without further purification. The major equipment included a microwave-assisted heating system, an electric oven (Electrolux-EMM20K22B, Singapore), a muffle furnace (Thermo Scientific Barnsted Premium F6010-Large Muffle Furnace, New Jersey, USA), mechanical stirrer, double-neck glass reactor (Duran, Indonesia), and a GC–MS instrument (Shimadzu GCMS-QP2010, Japan). The independent variables were the CaO catalyst loading (1, 2, 3, 4, and 5 wt%) and microwave-assisted transesterification reaction time (2, 4, 6, 8, and 10 min). Fixed parameters included microwave power (340 W), methanol-to-oil molar ratio (8:1), and stirring speed (350 rpm) [5].

### 2.2 CaO catalyst modification with ammonium carbonate

CaO modification was performed to enhance catalyst basicity and surface reactivity. A total of 3.5 g of CaO was immersed in 50 mL of ammonium carbonate solution (0.57 g/mL) (Fig. 1). The suspension was stirred until solid precipitates formed. The resulting solid was separated, washed, and dried in an oven at 110 °C until constant weight was achieved. Subsequently, the dried precursor was calcined in a muffle furnace at 900 °C for 1.5 h to decompose carbonate species and regenerate highly active CaO. The modified catalyst was stored in a desiccator prior to use. Catalyst purity and CaO content were determined through standard CaO assay methods to evaluate its active component fraction [5].

### 2.3 Preparation and pretreatment of waste cooking oil

Collected waste cooking oil was filtered using fine-grade filter paper to remove suspended solids. Determination of free fatty acid (FFA) content was carried out by titration

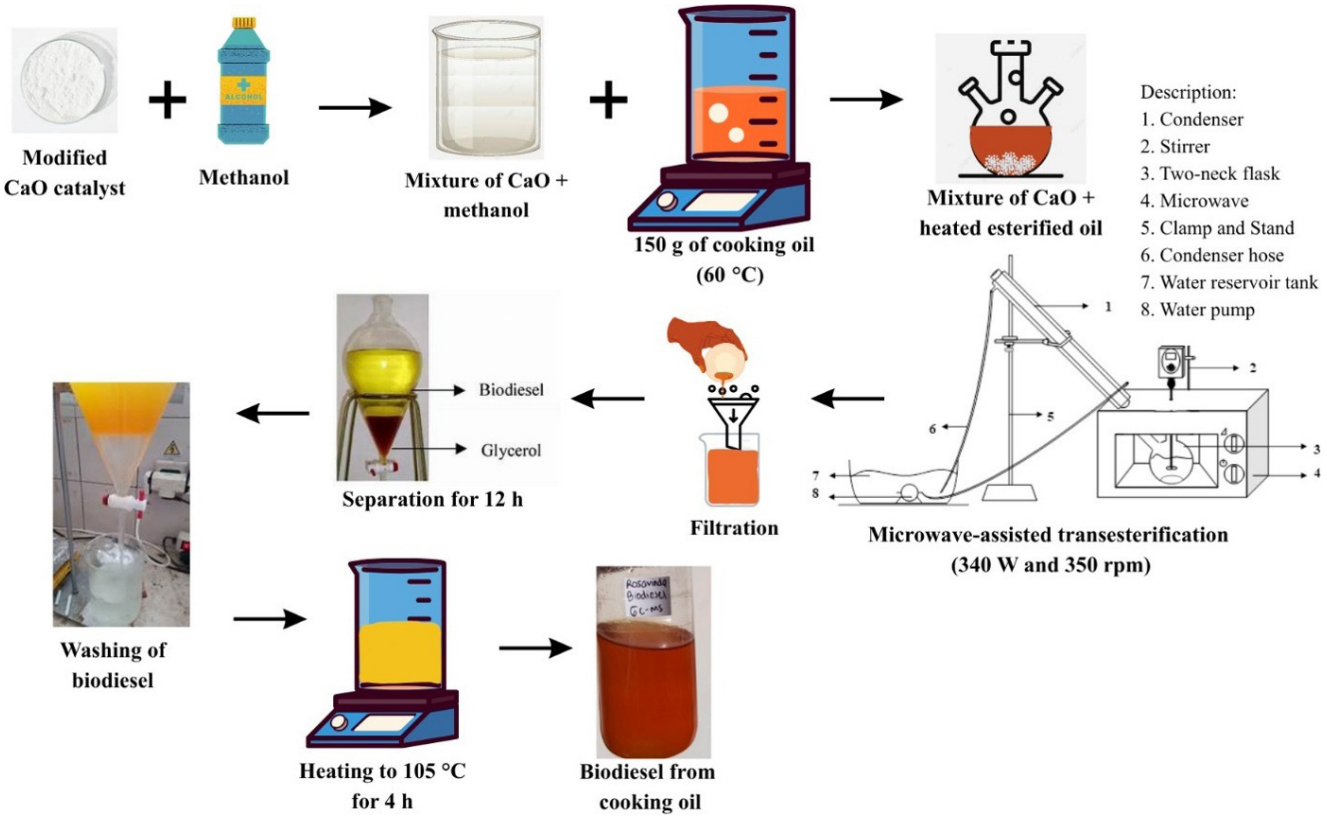


**Fig. 1** Scheme of the microwave-assisted transesterification process for biodiesel production using a modified CaO catalyst

using a phenolphthalein (PP) indicator and standardized NaOH solution. To reduce pigments and impurities, an adsorption pretreatment was conducted using 7 g activated charcoal for every 40 mL of waste cooking oil. The mixture was heated to 150 °C and stirred for 60 min. The treated oil was allowed to cool, filtered, and stored for subsequent esterification. The free fatty acid content was calculated using the following Eq. (1):

$$\mathrm{FFA}(\%) = \frac{V_{\mathrm{NaOH}} \times M_{\mathrm{NaOH}} \times 25.6}{m_{\mathrm{oil}}}, \quad (1)$$

where $V_{\mathrm{NaOH}}$ is the volume of NaOH solution (mL), $M_{\mathrm{NaOH}}$ is the molarity of NaOH, and $m_{\mathrm{oil}}$ is the mass of oil (g). The constant 25.6 is a conversion factor derived from the molecular mass of palmitic acid (256.4 g/mol), with FFA expressed as % palmitic acid.

### 2.4 Microwave-assisted transesterification of waste cooking oil

To reduce the FFA content to below 1%, an acid-catalyzed esterification step was performed. Approximately 150 g of pretreated oil was placed in a beaker and mixed with 78 mL of methanol and 1.5 mL of concentrated $H_2SO_4$. The mixture was heated at 50–60 °C for 1 h under continuous stirring. After completion, the reaction mixture was transferred into a separatory funnel and allowed to settle until two distinct layers were formed. The lower layer, containing water and impurities, was discarded, while the upper esterified oil layer was retained. The post-esterification FFA content was re-evaluated to ensure its value met the requirement for the subsequent transesterification stage.

### 2.5 Catalytic reaction mechanisms

The production of biodiesel from waste cooking oil involves two main catalytic reactions: esterification of free fatty acids and transesterification of triglycerides. In the acid-catalyzed esterification step, sulfuric acid protonates the carbonyl oxygen of the carboxylic (–COOH) group present in free fatty acids (FFAs), enhancing its electrophilicity and enabling nucleophilic attack by methanol to form fatty acid methyl esters and water, as shown in Fig. 2 (a). Subsequently, the main conversion of triglycerides occurs *via* base-catalyzed transesterification using modified CaO as a heterogeneous catalyst. CaO generates surface methoxide species ($CH_3O^-$) through methanol activation, which then attack triglyceride molecules to produce FAME and glycerol in a stepwise reaction, as presented in Fig. 2 (b). In this system, microwave irradiation enhances reaction kinetics by improving molecular collisions, reducing mass transfer limitations, and accelerating both esterification and transesterification processes. The combined acid–base catalytic route is therefore effective for high-FFA waste cooking oil, enabling high biodiesel yield and purity.

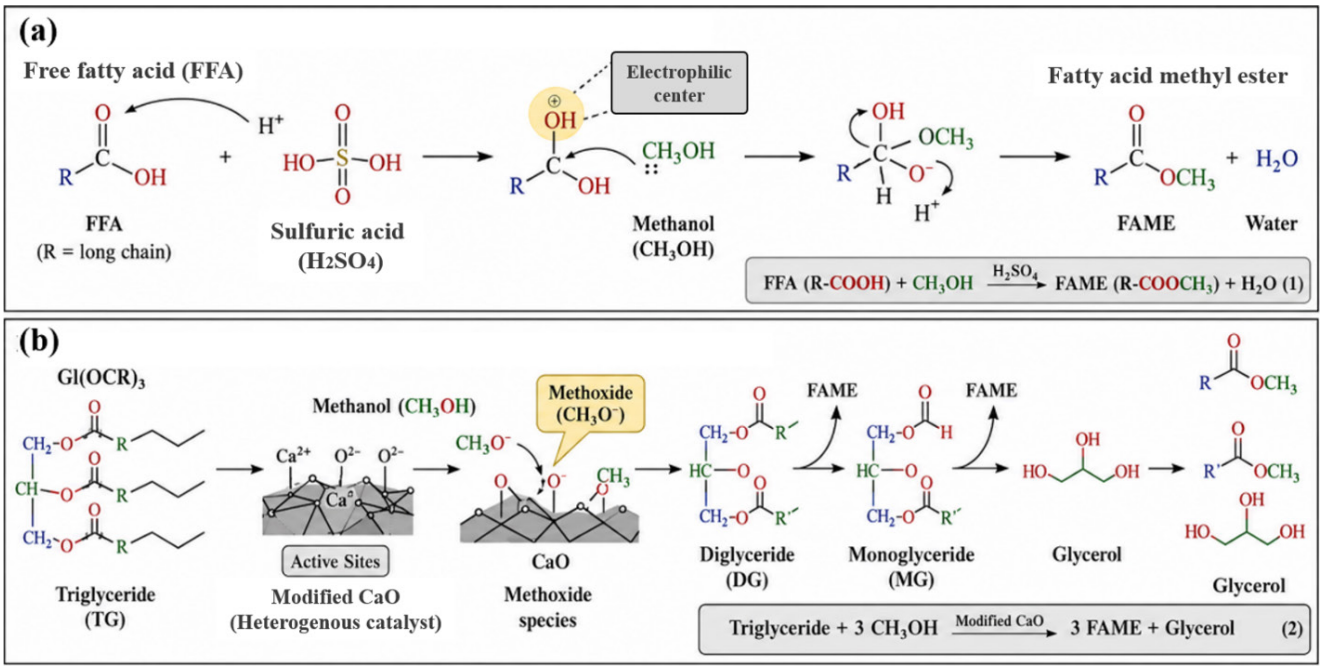


**Fig. 2** Schematic representation of biodiesel production *via* (a) acid-catalyzed esterification of free fatty acids with methanol to form fatty acid methyl esters and water as a by-product, and (b) base-catalyzed transesterification of triglycerides with methanol to produce FAME and glycerol as a by-product

### 2.6 Characteristics of biodiesel production from waste cooking oil

#### 2.6.1 Yield of biodiesel

The biodiesel yield was calculated based on the mass ratio of methyl ester produced to the initial oil mass, as expressed in Eq. (2):

$$\text{Yield}(\%) = \left( \frac{\text{Mass of biodiesel obtained}}{\text{Mass of oil used}} \right) \times 100. \quad (2)$$

#### 2.6.2 Density of biodiesel

Biodiesel density was measured using a pycnometer at room temperature. The density was calculated using Eq. (3):

$$\text{Density} = \frac{\left( m_{\text{filled}} - m_{\text{empty}} \right)}{V_{\text{pycnometer}}}, \quad (3)$$

where $m_{\text{filled}}$ and $m_{\text{empty}}$ are the masses of the filled and empty pycnometer, respectively, and $V_{\text{pycnometer}}$ is the pycnometer volume.

#### 2.6.3 Kinematic viscosity

The kinematic viscosity of biodiesel was determined using an Ostwald viscometer at 40 °C. The viscosity was calculated according to Eq. (4).

$$v = K \times t, \quad (4)$$

where $v$ is the kinematic viscosity ($mm^2/s$), $K$ is the viscometer constant, and $t$ is the flow time (s). The measured viscosity values were compared with ASTM D6751-24 [20] and EN 14214:2012+A2:2019 [21] biodiesel standards.

### 2.7 Analytical methods

The elemental oxide composition of the catalyst was analyzed using X-ray fluorescence (XRF) spectroscopy. The analysis was performed using an energy-dispersive X-ray fluorescence (EDXRF) technique, where the sample is excited by primary X-ray radiation to determine its elemental composition. The measurements were carried out using a PANalytical MiniPal 4 XRF spectrometer (PANalytical, Almelo, Netherlands) at the Laboratory of Universitas Negeri Malang, Indonesia.

The FAME composition of the produced biodiesel was determined using GC–MS. The analysis was conducted using a Shimadzu GC–MS system (GCMS-QP2010 SE, TQ8030 model) at the Laboratory of Universitas Diponegoro, Indonesia. GC was used for component separation, while MS was employed for molecular identification of the separated compounds. The calorific value of the biodiesel samples was determined using the constant volume combustion method. The measurements were performed using a Parr 6400 Automatic Isoperibol Bomb Calorimeter (Parr Instrument Company, USA) at the Integrated Laboratory Unit of Universitas Diponegoro, Indonesia. This method was used to evaluate the energy content of the produced biodiesel under controlled combustion conditions.

## 3 Result and discussion

### 3.1 Physicochemical properties of waste cooking oil

The physicochemical characteristics of waste cooking oil before and after esterification pretreatment are summarized in Table 1. Esterification led to substantial improvements in all measured properties, indicating successful feedstock upgrading prior to transesterification.

A decrease in density from 0.9372 to 0.8879 $g/cm^3$ was observed following esterification. This reduction is primarily attributed to the conversion of free fatty acids into fatty acid methyl esters and the removal of polar degradation products generated during repetitive thermal oxidation. Similar trends have been reported in previous studies [22, 23], where esterification pretreatment improved feedstock homogeneity and fuel flow characteristics.

The viscosity of the oil decreased significantly from 8.75 to 6.20 $mm^2/s$, reflecting the breakdown of polymerized triglycerides and oxidized compounds typically formed during prolonged frying. High viscosity is detrimental to mass transfer during transesterification, particularly in heterogeneous catalytic systems. The observed viscosity reduction is consistent with reports by Azahar et al. [24], confirming the effectiveness of esterification in improving rheological properties of waste cooking oil. Qualitative improvements were also evident. The transformation of the oil from dark brown and turbid to clear orange, along with the elimination of rancid odor, suggests efficient removal of suspended solids, pigments, and volatile oxidation byproducts. Such purification is critical, as residual impurities may deactivate catalysts and reduce ester yield [25].

Most notably, the FFA content decreased from 1.74% to 0.82%, falling below the widely accepted threshold of 1% required for base-catalyzed transesterification. Elevated

**Table 1** Characteristics of waste cooking oil before and after esterification pretreatment. Before esterification refers to the oil after pretreatment and prior to esterification.

| Parameter | Before esterification | After esterification |
|---|---|---|
| Density ($g/cm^3$) | 0.9372 | 0.8879 |
| Viscosity ($mm^2/s$) | 8.75 | 6.2 |
| Color | Dark brown, cloudy | Clear orange |
| Smell | Rancid smell | No sting |
| FFA (%) | 1.74 | 0.82 |
| References | This study | This study |

FFA levels promote soap formation in alkaline systems, leading to emulsion formation and poor phase separation. The achieved FFA reduction aligns well with previous findings that emphasize acid esterification as an essential pretreatment for high-FFA feedstocks [26].

From a standards perspective, there is currently no specific regulation governing the physicochemical quality of WCO as a biodiesel feedstock. However, several international biodiesel standards, including Indonesian National Standard (SNI 7182:2015) [18], ASTM D6751-24 standard [20], and EN 14214:2012+A2:2019 standard [21], implicitly define acceptable feedstock quality through limits on final biodiesel properties and pretreatment requirements.

One critical parameter indirectly regulated by these standards is free fatty acid content. High FFA levels in feedstock are known to cause soap formation during base-catalyzed transesterification, leading to poor ester yield and difficult phase separation. Consequently, numerous studies have adopted an FFA threshold of ≤1 wt% as a practical criterion for feedstock suitability prior to alkaline transesterification, a value consistent with recommendations derived from ASTM D6751-24 [20] and EN 14214:2012+A2:2019 [21] specifications [27, 28].

In the present study, esterification pretreatment reduced the FFA content of waste cooking oil from 1.74% to 0.82%, thereby satisfying the commonly accepted feedstock requirement for subsequent base-catalyzed transesterification. This improvement is essential to ensure that the resulting biodiesel can meet key quality parameters regulated under SNI 7182:2015 standard [18], including ester content, viscosity, and acid value, as well as international standards such as ASTM D6751-24 [20] and EN 14214:2012+A2:2019 [21].

### 3.2 Modification of CaO with ammonium carbonate

The influence of ammonium carbonate modification on CaO catalyst properties was assessed through changes in CaO content and alkalinity, as presented in Table 2.

The modified CaO catalyst exhibited a significantly higher CaO content (97.61%) compared to the unmodified catalyst (91.72%). This increase indicates that ammonium carbonate treatment followed by high-temperature calcination effectively removed impurities and regenerated highly active CaO phases. During calcination, ammonium carbonate decomposes into $NH_3$ and $CO_2$, promoting pore formation and exposing additional basic active sites on the CaO surface.

**Table 2** Chemical composition and alkalinity of CaO catalyst before and after ammonium carbonate modification

| Parameter | Initial CaO catalyst | Modified CaO catalyst |
|---|---|---|
| CaO Component (%) | 91.72 | 97.61 |
| pH | 12 | 13 |
| References | This study | This study |

The increase in pH from 12 to 13 further confirms the enhanced basicity of the modified catalyst. Catalyst basicity plays a critical role in transesterification reactions, as stronger basic sites facilitate methoxide ion formation, accelerating triglyceride conversion into methyl esters. Similar enhancements in CaO basicity and catalytic activity after chemical modification have been reported by [29], who observed improved biodiesel yields when CaO was modified using carbonate or hydroxide precursors.

Moreover, studies by Sierra-Cantor et al. [30] demonstrated that modified CaO catalysts exhibit higher resistance to leaching and better reusability compared to untreated CaO, making them more suitable for heterogeneous catalysis. The higher CaO purity and alkalinity observed in this study suggest that ammonium carbonate modification enhances catalyst stability and activity under microwave-assisted reaction conditions. The modification of CaO using ammonium carbonate significantly improves its chemical composition and alkalinity, leading to a more active and effective heterogeneous catalyst for biodiesel production. These findings are consistent with previous studies and support the use of modified CaO as a low-cost, environmentally friendly catalyst for waste cooking oil transesterification.

In addition, the detailed oxide composition obtained from X-ray fluorescence analysis is presented in Table 3.

The detailed XRF results presented in Table 3 further confirm that ammonium carbonate modification effectively reduced or eliminated minor oxide impurities such as $SO_3$, MnO, ZnO, SrO, and $Re_2O_7$, while significantly decreasing the $Fe_2O_3$ content. The presence of such impurities is

**Table 3** XRF analysis of CaO catalyst before and after ammonium carbonate modification

| Oxide component | CaO catalyst before modification (%) | Modified CaO catalyst (%) |
|---|---|---|
| CaO | 97.00 | 99.05 |
| $SO_3$ | 0.48 | – |
| MnO | 0.33 | – |
| $Fe_2O_3$ | 0.67 | 0.32 |
| ZnO | 0.29 | – |
| SrO | 0.47 | – |
| $Tm_2O_3$ | – | 0.45 |
| $Lu_2O_3$ | 0.48 | 0.17 |
| $Re_2O_7$ | 0.27 | – |

known to reduce catalyst basicity and promote catalyst deactivation during transesterification reactions [31].

The enhancement in CaO purity can be attributed to the thermal decomposition behavior of ammonium carbonate during calcination. Upon heating, ammonium carbonate decomposes into $NH_3$ and $CO_2$, temporarily promoting $CaCO_3$ formation on the catalyst surface. Subsequent high-temperature calcination induces the decarbonation of $CaCO_3$ back to CaO, facilitating impurity removal and generating a more porous structure with a higher density of exposed $O^{2-}$ basic sites. Similar pore-forming and basicity-enhancing effects have been reported for chemically modified CaO catalysts prepared using carbonate precursors [32, 33].

Previous studies have also demonstrated that chemically modified CaO catalysts exhibit improved structural stability, reduced leaching, and enhanced reusability compared to untreated CaO [34]. Although catalyst reusability was not evaluated in this study, the high CaO purity (99.05%) and increased alkalinity observed suggest enhanced resistance to deactivation, making the modified catalyst suitable for heterogeneous biodiesel production.

Moreover, ammonium carbonate modification significantly enhances both the chemical composition and alkalinity of CaO, as confirmed by XRF and pH analysis. This simple, low-cost, and environmentally benign modification strategy produces a highly active and stable heterogeneous catalyst, well suited for microwave-assisted biodiesel production from waste cooking oil. The results obtained in this study are in good agreement with previous findings and further support the application of modified CaO as an efficient catalyst for sustainable biodiesel synthesis.

This research has been conducted to determine the influence of variables on yield by Wati et al. [5]. And regarding the results of these characteristics, they are in accordance with previous researcher, these and both studies consistently report very high biodiesel yields in the range of approximately 95–96%, confirming that modified Calcium oxide catalysts are highly effective in achieving near-complete conversion of triglycerides present in waste cooking oil into fatty acid methyl esters. Specifically, nano-CaO catalysts demonstrate an optimized yield of about 96%, while CaO/hectorite heterogeneous catalysts achieve approximately 95% biodiesel yield under optimized reaction conditions. This strong agreement across different modified CaO systems indicates that catalyst structural enhancement plays a critical role in improving transesterification performance. The increased surface area in nano-CaO systems provides greater exposure of active sites, allowing improved accessibility of methanol and triglyceride molecules to the catalytic surface. increased basicity is a key factor in improving catalytic efficiency, as stronger $O^{2-}$ basic sites promote more rapid formation of methoxide ions, which are the active nucleophilic species responsible for attacking triglyceride carbonyl groups. Improved dispersion of CaO particles in both catalyst systems further prevents agglomeration, ensuring that more active sites remain available for reaction and thereby accelerating overall transesterification kinetics [35]. Waste cooking oil initially exhibits very high viscosity values in the range of approximately 25–40 $mm^2/s$, which is unsuitable for direct use in diesel engines due to poor atomization and incomplete combustion.

However, after catalytic conversion into FAME, the viscosity is markedly reduced to approximately 4–6 $mm^2/s$, which falls within ASTM D6751-24 [20] and EN 14214:2012+A2:2019 [21] biodiesel standards. The mechanistic explanation for this behavior is strongly linked to the efficiency of methoxide ion formation on the catalyst surface. Strong basic $O^{2-}$ sites on modified CaO facilitate rapid deprotonation of methanol, generating active nucleophiles that efficiently cleave ester bonds in triglycerides. This results in complete ester bond conversion and the formation of lower molecular weight methyl esters, which inherently possess lower viscosity compared to long-chain triglycerides [35]. Biodiesel produced using modified Calcium oxide catalysts exhibits a significant reduction in density compared to raw waste cooking oil, while remaining fully compliant with international biodiesel fuel standards such as ASTM D6751-24 [20] and EN 14214:2012+A2:2019 [21].

Waste cooking oil typically shows relatively high-density values in the range of approximately 910–965 $kg/m^3$ due to its high content of triglycerides, long-chain fatty acids, and degradation products formed during repeated thermal processing. In contrast, after catalytic conversion through transesterification, the resulting biodiesel (fatty acid methyl esters) displays lower density values, generally within the range of 850–890 $kg/m^3$, which falls comfortably within the standard acceptable range of 860–900 $kg/m^3$. The underlying mechanism is strongly linked to improved catalytic activity, where enhanced basicity and increased surface area of modified CaO promote rapid formation of methoxide ions, leading to more complete transesterification of triglycerides into methyl esters. This efficient molecular transformation directly results in a lighter fuel composition with more uniform physical properties [35].

### 3.3 Characteristics of biodiesel

#### 3.3.1 Yield of biodiesel

Biodiesel yield is a critical indicator of transesterification efficiency, as it reflects the extent of triglyceride conversion into fatty acid methyl esters. The effect of reaction time and CaO catalyst loading on biodiesel yield is summarized in Fig. 3 (a). Based on the result, biodiesel yield increased with increasing reaction time and CaO catalyst loading up to an optimum condition, beyond which a decline was observed also in some data in Wati et al. [5]. The increase in yield from 2 to 6 min indicates that longer reaction times and higher catalyst concentrations provide a greater number of accessible basic active sites, thereby enhancing methoxide ion formation and accelerating the transesterification reaction. Similar trends have been reported by Bharti et al. [36] who demonstrated that increasing CaO catalyst loading improves biodiesel conversion until an optimal level is reached.

The maximum biodiesel yield of 82.80% was achieved at a catalyst loading of 4 wt% and a reaction time of 6 min, indicating an optimal balance between catalyst availability, reaction kinetics, and microwave energy input. Beyond this condition, further increases in reaction time (8–10 min) resulted in a reduction in biodiesel yield. This decline can be attributed to the reversible nature of the transesterification reaction, in which methyl esters may react back with glycerol once equilibrium is reached. In addition, prolonged microwave irradiation can induce localized overheating, potentially leading to thermal degradation of methyl esters and reduced biodiesel yield [37]. These results demonstrate that excessive reaction time and catalyst dosage do not necessarily enhance biodiesel production and may instead promote side reactions or degradation, underscoring the importance of process parameter optimization in microwave-assisted transesterification systems.

#### 3.3.2 Density of biodiesel from waste cooking oil

Biodiesel density was measured using a 10 mL pycnometer, and the results as a function of reaction time and CaO catalyst loading. As shown in Fig. 3 (b), biodiesel density exhibited a decreasing trend with increasing reaction time and CaO catalyst concentration. This behavior reflects the progressive conversion of high-molecular-weight triglycerides into lower-density fatty acid methyl esters. In addition, the increase in reaction temperature associated with longer microwave irradiation reduces intermolecular interactions,

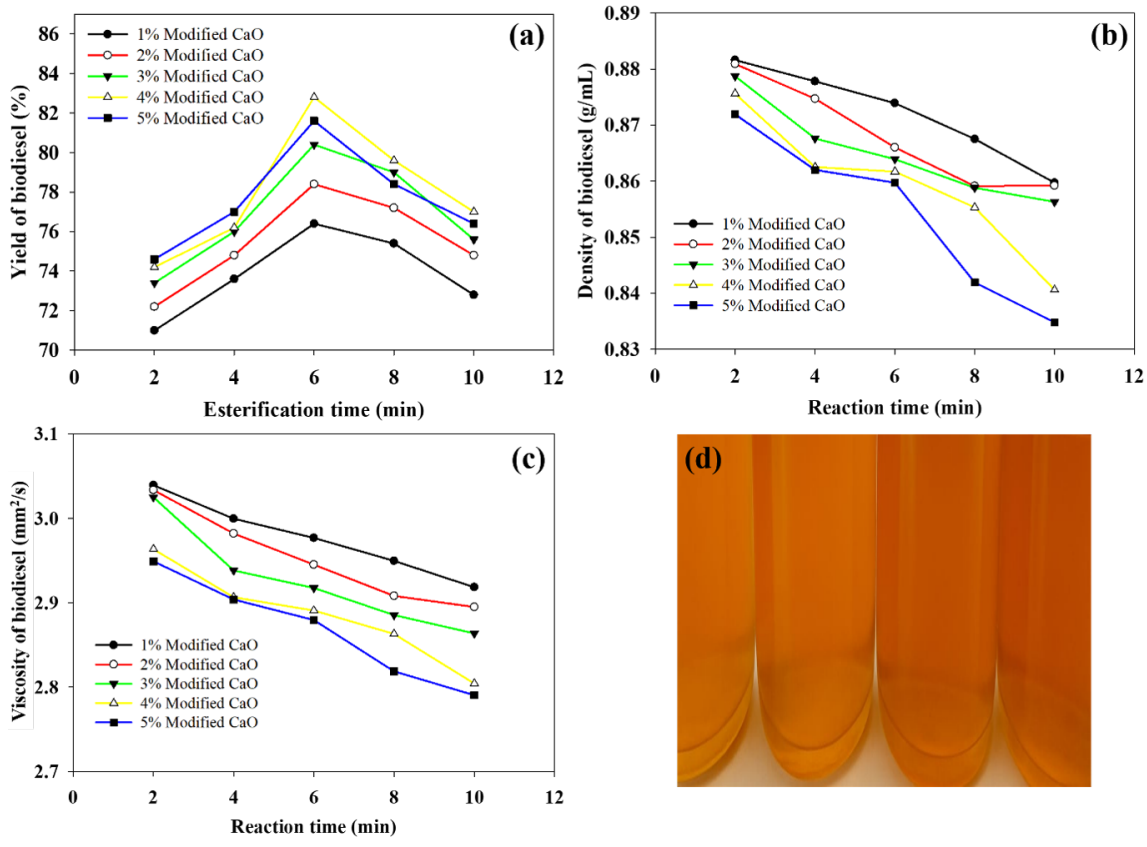


**Fig. 3** Effect of modified CaO catalyst concentration (1–5%) and reaction time on the physical and chemical properties of produced biodiesel: (a) yield of biodiesel (%); (b) density (g/mL); (c) kinematic viscosity (mm²/s); (d) visual appearance of the final biodiesel sample obtained under the optimum reaction conditions of 4 wt% CaO catalyst and 6 min reaction time

further contributing to the observed decrease in density. The biodiesel density values obtained at reaction times of 2, 4, 6, 8, and 10 min fall within the density range specified by Indonesian National Standard (SNI 7182:2015) [18] (0.85–0.89 g/mL). This indicates that biodiesel produced under all investigated conditions satisfies the national density requirement. Moreover, the measured density values are also consistent with ASTM D6751-24 standard [20] and EN 14214:2012+A2:2019 standard [21], which specify similar density ranges for biodiesel fuels. The observed trend aligns with previous studies reporting that increased ester content and higher conversion efficiency result in lower biodiesel density due to the breakdown of triglycerides into three methyl ester molecules with reduced molecular mass [38].

### 3.3.3 Kinematic viscosity of biodiesel from waste cooking oil

The kinematic viscosity of biodiesel was determined using an Ostwald viscometer, as illustrated in Fig. 3 (c), both CaO catalyst loading and reaction time significantly influence the viscosity of the produced biodiesel. Increasing catalyst concentration and longer reaction times result in lower viscosity values, indicating improved conversion of oil into methyl esters. This reduction in viscosity occurs because methyl ester molecules possess shorter carbon chains and fewer intermolecular interactions compared to triglycerides, leading to enhanced flow properties [39].

Other physicochemical properties, including density, flash point, and calorific value, were also analyzed to evaluate the overall fuel quality. The biodiesel density ranged from 0.88 to 0.83 g/mL, complying with both SNI 7182:2015 [18] (850–890 kg/m$^3$) and EN 14214:2012+A2:2019 [21] (860–900 kg/m$^3$) standards. The flash point was measured at 100 °C, meeting SNI requirements. The calorific value of 8.80 kcal/g, reported in Table 4, further confirms the biodiesel high energy potential and suitability as an alternative fuel source.

The viscosity values obtained for reaction times ranging from 2 to 10 min are within the acceptable range specified by SNI 7182:2015 standard [18], which requires biodiesel viscosity to be between 2.3 and 6.0 mm$^2$/s. These results demonstrate that the microwave-assisted transesterification process using modified CaO catalyst effectively improves fuel flow characteristics, which is critical for engine performance and fuel injection systems. Overall, the combined analysis of yield, density, and viscosity confirms that the optimal operating condition, such as 4 wt% CaO catalyst and 6 min of reaction time provides high biodiesel yield while maintaining physicochemical properties that comply with national biodiesel standards.

### 3.3.4 Calorific value

The calorific value represents a critical parameter for evaluating the energy potential of biodiesel as a fuel. It indicates the amount of thermal energy released per unit mass of fuel upon complete combustion. The calorific value is a key indicator of biodiesel quality because higher calorific values correspond to greater energy output, directly influencing engine efficiency and fuel performance. In this study, biodiesel was produced *via* transesterification under optimal conditions, specifically a reaction time of 6 mins and catalyst loading of 4 wt%, and its calorific value was determined using a Parr bomb calorimeter 6400 at the Integrated Laboratory (UPT), Diponegoro University, as shown in Table 5 [40–43]. The results, summarized in Table 4, indicate that the biodiesel exhibits a calorific value of 36.78 kJ/g equivalent to 8.80 kcal/g.

The measured calorific value meets the Indonesian National Standard (SNI 7182:2015) [18], which specifies a minimum calorific value of 36.64 kJ/g (8760 cal/g), confirming that the produced biodiesel is suitable for use as an alternative fuel in diesel engines without requiring significant modifications.

When compared to other studies, the calorific value obtained in this work is competitive. For instance, Plata et al. [44] reported calorific value of approximately 39.9 ± 0.1 MJ/kg for palm oil-based biodiesel. Literature summarized in Table 5 [40–43] shows that biodiesel produced from various feedstocks, including sunflower oil, karanja oil, and waste cooking oil using different catalysts, exhibits calorific values ranging from 36.78 to 40.00 kJ/g. The biodiesel synthesized in the present study thus falls within the typical range of industrially relevant biodiesel and aligns well with literature-reported energy values.

**Table 4** Physicochemical properties of biodiesel produced from waste cooking oil under optimal microwave-assisted transesterification conditions (4 wt% CaO catalyst, 6 min reaction time) compared with SNI 7182:2015 [18] and EN 14214:2012+A2:2019 [21] standards

| Parameter | Biodiesel in this study | SNI 7182:2015 [18] | EN 14214:2012+A2:2019 [21] |
|---|---|---|---|
| Kinematic viscosity (mm$^2$/s) | 3.03–2.79 | 2.3–6.0 | 3.5–5 |
| Density (kg/m$^3$) | 0.88–0.83 g/mL | 850–890 | 860–900 |
| Calorific value (kJ/g) | 36.78 | 36.64 | – |

**Table 5** Calorific value of biodiesel produced under optimal conditions (6 min reaction time and 4 wt% catalyst)

| Raw material | Catalyst | Calorific value | Method/Instrument | Reference |
|---|---|---|---|---|
| Waste cooking oil | CaO catalyst | 36.78 kJ/g | Parr Bomb Calorimeter 6400 | This study |
| Waste cooking oil | Alkali catalyst | 37.2 kJ/g | Oxygen bomb calorimeter | [40] |
| Sunflower oil | – | 39,500 kJ/g | – | [41] |
| Waste cooking oil | CaO catalyst | 37.09 MJ/kg | Automatic calorimeter | [42] |
| Karanja oil | KOH | 40 MJ/kg | Bomb calorimeter | [43] |
| Waste cooking oil | KOH | 39.85 MJ/kg | Bomb calorimeter | [43] |

The calorific value of biodiesel is influenced by multiple factors, including fatty acid composition, catalyst type, and reaction conditions. Biodiesel with higher saturated fatty acid content tends to exhibit greater calorific values due to the higher energy released from the combustion of saturated hydrocarbon chains compared to unsaturated chains. The optimized conditions applied in this study (6-min reaction time and 4 wt% catalyst) ensured efficient transesterification, achieving a high conversion to fatty acid methyl esters, which directly contributed to the elevated calorific value.

From a practical perspective, the calorific value of 8.80 kcal/g indicates that the biodiesel produced can deliver energy comparable to commercial vegetable oil-derived biodiesel. This suggests a strong potential for industrial application as a renewable and sustainable fuel, contributing to reduced reliance on fossil fuels and supporting national biofuel programs, such as the B30 or B40 biodiesel blending initiatives in Indonesia.

### 3.4 GC–MS analysis of biodiesel from waste cooking oil

GC–MS was conducted to determine the chemical composition and quantify the fatty acid methyl ester content of biodiesel produced under optimal conditions, specifically a 6 min reaction time and 4 wt% CaO catalyst. The analysis was performed using a GCMS/Simadzu system at the Integrated Laboratory (UPT), Diponegoro University, and the results are summarized in Fig. 4 and Table 6.

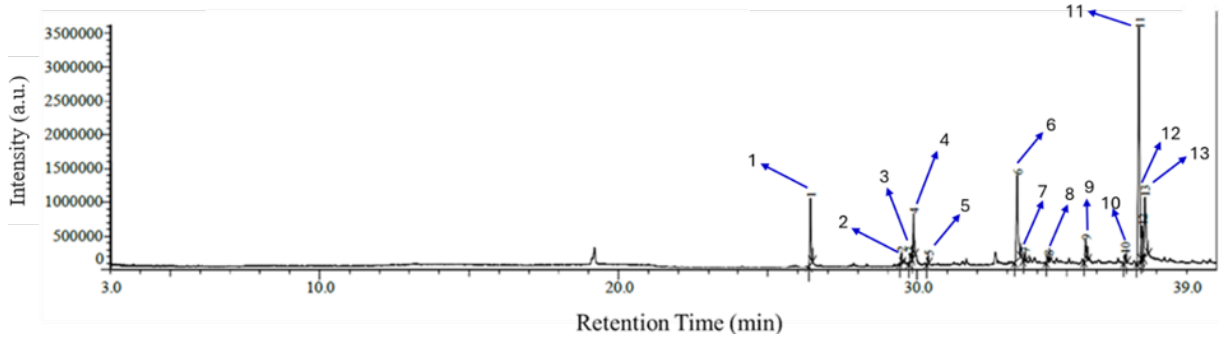


**Fig. 4** Gas chromatography-mass spectrometry profile of waste cooking oil biodiesel. The chromatogram displays the distribution of methyl esters. The numbers are identified in Table 6.

**Table 6** GC–MS analysis of biodiesel produced under optimal conditions (6 min reaction time, 4 wt% CaO catalyst)

| Peak # | Retention time (min) | Area (a.u.) | Area (%) | Height (a.u.) | Compound name |
|---|---|---|---|---|---|
| 1 | 26.40 | 382,324 | 11.62 | 975,120 | 9,12-Octadecadienoic acid, methyl ester (linoleic acid) |
| 2 | 29.42 | 112,171 | 0.34 | 157,022 | Oxirane, tetradecyl |
| 3 | 29.73 | 79,209 | 0.24 | 135,450 | 1,2-Benzenedicarboxylic acid |
| 4 | 29.83 | 287,808 | 8.75 | 637,260 | Octadecanoic acid, methyl ester (stearic acid) |
| 5 | 30.31 | 78,335 | 0.24 | 109,633 | Acetic acid |
| 6 | 33.30 | 559,518 | 17.00 | 1,255,700 | Hexadecanoic acid, methyl ester (palmitic acid) |
| 7 | 33.55 | 96,140 | 0.29 | 101,498 | 2-Pentadecanone, trimethyl- |
| 8 | 34.32 | 78,626 | 0.24 | 85,592 | Oleyl alcohol |
| 9 | 35.60 | 63,894 | 0.19 | 300,620 | 9-Tetradecenal, (Z)- |
| 10 | 36.92 | 53,158 | 0.16 | 126,977 | 2-Heptadecanone |
| 11 | 37.38 | 1,643,477 | 49.94 | 3,464,562 | 9-Octadecenoic acid, methyl ester (oleic acid) |
| 12 | 37.48 | 19,287 | 0.59 | 492,289 | Ethanol, 2-(9-octadecenyloxy) |
| 13 | 37.58 | 342,076 | 10.40 | 888,273 | Hydroxy methyl ester of ricinoleic acid (ricinoleic acid methyl ester) |

The GC–MS analysis identified several major methyl esters. Linoleic acid methyl ester (9,12-octadecadienoic acid, methyl ester) was present at 11.62% with a retention time of 26.4 min while stearic acid methyl ester (octadecanoic acid, methyl ester) was detected at 8.75% at 29.832 min. Palmitic acid methyl ester (hexadecanoic acid, methyl ester) accounted for 17.00% at 33.301 min, and oleic acid methyl ester (9-octadecenoic acid, methyl ester, (E)) represented the highest proportion at 49.94% with a retention time of 37.382 min. Ricinoleic acid methyl ester (hydroxy methyl ester of ricinoleic acid) was observed at 10.40% at 37.577 min. Minor compounds, including oxirane derivatives, acetic acid, and ketones, were present at 2.29%, indicating a high overall purity of the biodiesel. The total methyl ester content was calculated as 97.71%, exceeding the minimum requirement of 96.5% according to SNI 7182:2015 standard [18], confirming that the biodiesel complies with national quality standards.

The fatty acid composition identified in this study is consistent with previous reports on waste cooking oil biodiesel. Similar studies have shown that oleic, palmitic, and linoleic acid methyl esters are the major components, with oleic acid generally being the most abundant [45, 46]. The predominance of oleic acid methyl ester contributes to favorable fuel properties, including improved oxidative stability and cold flow characteristics, as mono-unsaturated methyl esters are more resistant to degradation than polyunsaturated esters and maintain fluidity at lower temperatures [47]. Saturated methyl esters, such as palmitic and stearic acids, enhance the calorific value, complementing the energy performance discussed in Section 3.3.4. Linoleic acid methyl ester, while beneficial for low-temperature flexibility, slightly reduces oxidative stability due to the presence of double bonds, and ricinoleic acid methyl ester contributes to hydroxyl groups that may influence viscosity and lubrication properties [48].

The high methyl ester content observed demonstrates that microwave-assisted transesterification using CaO catalyst is highly efficient, achieving nearly complete conversion of triglycerides to FAME in a short reaction time. The low proportion of impurities indicates minimal post-processing or purification requirements, supporting the feasibility of this method for industrial-scale biodiesel production.

Overall, this study successfully improved the fuel properties of waste cooking oil through transesterification using modified CaO catalyst, producing biodiesel characteristics that were within or close to the biodiesel quality standards of ASTM D6751-24 [20] and EN 14214:2012+A2:2019 [21]. The synthesized biodiesel exhibited lower density and viscosity compared with the original waste cooking oil, indicating an effective conversion of triglycerides into fatty acid methyl esters. The reduction in viscosity and density after transesterification confirms the effective catalytic activity of modified CaO in converting high-molecular-weight triglycerides into lower-viscosity methyl esters, thereby improving fuel atomization and combustion characteristics. In addition, the produced biodiesel showed improved physical appearance, including a clearer color and reduced rancid odor, demonstrating enhanced fuel quality. A comparison with previous studies is presented in Table 7 [49, 50], confirming that the fuel properties obtained in this work are comparable with those reported for biodiesel derived from other feedstocks and catalytic systems.

**Table 7** Fuel properties of produced biodiesel from cooking oil using CaO as a catalyst compared to literature value

| Parameter | Waste cooking oil | Biodiesel | WCO | Biodiesel (B5) | Ricinus communis oil + UCO | Biodiesel (RCO + UCO) |
|---|---|---|---|---|---|---|
| Density (kg/m$^3$) | 937.2 | 850–890 | 923.3 ± 1.5 | 830.2 ± 0.6 | 875 | 875 ± 1 |
| Viscosity (mm$^2$/s) | 8.75 | 2.3–6.0 | 33.12 ± 0.25 | 3.194 ± 0.015 | 6.24 | 6.24 ± 0.14 |
| Color | Dark brown, cloudy | Clear yellow | – | – | – | – |
| Odor | Rancid | No pungent odor | – | – | – | – |
| FFA (%) | 1.74 | – | – | – | – | – |
| Calorific value (kcal/g) | – | 8.80 | – | – | – | – |
| Heating value (MJ/kg) | – | – | – | 42.74 ± 0.20 | 38.77 | 38.77 ± 0.15 |
| References | This study | This study | Abobakr et al. [49] | Abobakr et al. [49] | Kodgire et al. [50] | Kodgire et al. [50] |

Note: – indicates that the corresponding data were not available or were not reported in the cited reference.

## 4 Conclusion

This study demonstrated that biodiesel produced from waste cooking oil using microwave-assisted transesterification with modified CaO catalyst exhibits excellent physicochemical properties and high methyl ester content under optimized conditions. Among the tested reaction times (2–10 min) and catalyst loadings (1–5 wt%), the optimal condition was achieved at 4 wt% catalyst and 6 mins, resulting in a biodiesel yield of 82.80%. Under these conditions, the biodiesel showed a density of 0.8617 g/mL, a kinematic viscosity of 2.863 $mm^2/s$, and a calorific value of 36.78 kJ/g (8.80 kcal/g), all of which comply with SNI 7182:2015 standard [18]. Characterization of the catalyst indicated that ammonium carbonate-modified CaO had a higher calcium oxide content (97.61%) compared to unmodified CaO, contributing to the enhanced transesterification efficiency. GC–MS analysis confirmed the formation of high-purity biodiesel, with a total methyl ester content of 97.71%, exceeding the minimum requirement of 96.5% specified by SNI 7182:2015 standard [18]. The fatty acid profile was dominated by oleic acid methyl ester (49.94%), followed by palmitic, linoleic, stearic, and ricinoleic methyl esters, which collectively contributed to favorable fuel properties, including oxidative stability, energy content, and cold flow performance. Overall, these findings indicate that microwave-assisted transesterification using modified CaO catalyst is an effective, rapid, and environmentally friendly method for producing high-quality biodiesel from waste cooking oil. The resulting biodiesel not only meets national standards but also demonstrates potential as a renewable and sustainable alternative fuel suitable for diesel engines, supporting both energy efficiency and environmental sustainability. Nevertheless, this study was limited to FTIR-based catalyst characterization, while detailed morphological and crystalline analyses such as SEM, EDX, XRD, and reusability catalyst were not conducted. Therefore, future studies should include comprehensive physicochemical characterization to better understand the relationship between catalyst structure and biodiesel production performance.

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