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Biodiesel Conversion from Used Cooking Oil: An Eggshell-Based ZnO/CaO Supported on Activated Carbon Catalyst with Microwave Heating

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Abstract

Fossil fuels, such as petroleum, are non-renewable and have been exploited for decades, necessitating alternative energy solutions. Biodiesel reduces reliance on fossil fuels and mitigates their environmental impact. This research converts used cooking oil into biodiesel through chemical reactions—esterification and transesterification—that transform oils into biodiesel. Microwave heating accelerates reaction rates and reduces process times compared to conventional heating. A CaO catalyst derived from eggshells, impregnated with ZnO and supported by activated carbon, was chosen for its sustainability and enhanced catalytic activity. The study aims at determining the optimal conversion time for transforming used cooking oil into biodiesel according to SNI 7182-2015, using FT-IR and GC-MS analyses to assess chemical composition and purity. XRD and SAA confirmed the formation of CaO and a surface area of 3.822 m^2/g. Microwave heating times of 4-8 minutes at 600 watts were tested, with the highest yield (89.62%) achieved in 5 minutes. This meets SNI 7182-2015 standards for density, kinematic viscosity, acid number, and saponification number. GC-MS identified cis-13-octadecenoic acid methyl ester (52.69%), pentadecanoic acid 14-methyl methyl ester (31.89%), and methyl stearate (6.14%) as the main components. These results demonstrate sustainable biodiesel production from waste cooking oil using environmentally friendly catalysts, reducing reliance on fossil fuels.

Keywords: Biodiesel, Eggshell Based Catalyst, Microwave Heating, Used Cooking Oil

1 Introduction

Indonesia's diesel consumption has risen with economic growth and population expansion, leading to increased pollution and a decline in foreign exchange reserves due to substantial diesel imports. [1]. Data from the BPH Migas organization, as reported in 2017, shows a significant annual increase of 6% in diesel distribution. Between 2016 and 2018, the average annual consumption reached 14.6 million kiloliters [2]. The escalating demand for diesel presents a formidable challenge that necessitates immediate attention. This situation demands a comprehensive and effective solution to address the pressing issue [3]. Biodiesel, a promising alternative fuel, is one of Indonesia's potential biofuel production pathways. It's environmentally friendly, has no adverse health effects, and reduces emissions compared to diesel oil. [4]. Biodiesel can be made from used cooking oil. The quality and quantity of this raw material will be analyzed before conversion. [5]. The oil's quality has deteriorated due to free radical formation, leading to various diseases. [6].

The formation of biodiesel typically requires catalysts. Common challenges include saponification reactions that increase viscosity, reduce yield, and complicate catalyst separation. A potential solution is to use a solid catalyst, such as calcium oxide (CaO). [7]. The production of CaO can be achieved through the calcination of calcium carbonate ($CaCO_3$). A readily available source of $CaCO_3$ is eggshells, which contain

approximately 94% $CaCO_3$, 1% $MgCO_3$, 1% $Ca_3(PO_4)_2$, and 4% organic matter [8].

The transesterification process using heterogeneous catalysts has been extensively studied to replace the role of homogeneous catalysts [9], [10], [11], thereby improving the efficiency of biodiesel production. Zinc Oxide is a mineral-derived substance, appearing as a fine, pale particulate solid, which shows a negligible ability to break down in an aqueous medium but readily goes into solution within either acidic or alkaline environments [12], Through doping of ZnO into metal oxides, it was demonstrated that the incorporation of ZnO can augment the activity of heterogeneous catalysts in transesterification reactions [13]. Consequently, based on these research findings, the utilization of ZnO/CaO catalysts can elevate the yield of methyl esters from avocado seed oil to a satisfactory level of 90.882%, conforming to SNI biodiesel standards [14].

Activated carbon (CA) is employed as a foundational medium for catalytic substances to expand its accessible reactive area and refine its targeting efficiency. This method produces high-efficiency catalysts with maximum specific surface area of the metal, improved thermal stability, and extended lifespan. [15]. Microwave technology has unique characteristics compared to conventional methods, such as shorter heating times. [11]. Notable advantages include reduced power requirements, improved product output, and decreased reliance on alcoholic dissolving agents. [16].

This research aimed to assess the impact of varying microwave heating duration and the ZnO/CaO-AC catalyst on the chemical transformation of used cooking oil into biodiesel, specifically fatty acid methyl esters. Notably, used cooking oil, a hazardous and toxic waste, was used as the primary raw material for biodiesel production. The CaO catalyst source was unused eggshells, contributing to waste reduction. The microwave-based thermal process is another key feature of this investigation.

2 Method

2.1 Materials

The materials utilized in this study encompassed 200 grams of used cooking oil, aquades, zinc oxide solids (Prevest DenPro), sodium hydroxide solids (NaOH, Merck), potassium hydroxide solids (KOH, Merck), 1000 gram eggshells, methanol (99.8%, Merck), ethanol (96%, Merck), Phenolpthalein (SmartChem), Sulfuric acid (95-97%, Merck), anhydrous sodium sulfat (Mitra Wacana Media), acetic acid (100%, Emsure), hydrochloric acid (37%, Emsure), and benzoic acid (99.5%, SRL).

2.2 Instrumentation

Two types of characterization were performed in this research. The first was to characterize ZnO/CaO supported on activated carbon. Catalyst characterization was conducted using three instrumental methods: X-ray diffraction (XRD, XPert Pro), surface area analysis (SAA, St 3 on Nova 800), and thermogravimetric analysis (TGA, Shimadzu). XRD was used to analyze the crystalline structure, SAA to determine surface area, and TGA to assess the thermal stability of the catalyst. The second objective was to characterize the biodiesel. Characterization was performed using Fourier Transform Infrared Spectroscopy (FTIR, Invenio-S) to identify functional groups and Gas Chromatography-Mass Spectrometry (GC-MS, Thermo-Scientific) to determine the composition of fatty acid methyl esters.

2.3. Preparation of Cao catalyst from eggshells

The eggshell undergoes a meticulous cleaning process utilizing warm water to eliminate any dirt or membranes. Subsequently, the eggshell is immersed in a 10% NaClO solution for a duration of 10 minutes, facilitating the removal of the membrane from the eggshell. The eggshell is then rinsed thoroughly with aquades water until the pH level reaches neutrality. Subsequently, it is dehydrated in an oven at 100 degrees Celsius for 24 hours to remove moisture. The dehydrated eggshell is then crushed using a mortar and a blender to form a powder. The powder is sieved through an 80 mesh sieve and subjected to a five-hour high-temperature thermal treatment in a furnace at 900 degrees Celsius. This calcination process results in the formation of CaO solids. The CaO solids are analyzed using X-ray diffraction (XRD) techniques to determine their crystalline structure and phase composition.

2.4 Preparation of ZnO/CaO-AC Catalyst

The catalyst was prepared using the wet impregnation method. The catalyst consists of three components: calcium oxide (CaO), zinc oxide (ZnO), and activated carbon. The preparation process begins by synthesizing two components: a solid mixture of 21 grams of calcium oxide and 14 grams of activated carbon, and a mixture of 10 grams of zinc oxide and 100

mL of purified water. Zinc oxide in purified water is in the slurry form. These components are combined in an Erlenmeyer flask and subjected to continuous agitation for an hour. Afterward, the slurry is dried for five hours at 100°C in an oven. Finally, the dried material is calcinated at 500°C for five hours. The impregnated catalyst is then analyzed using X-ray diffraction (XRD) techniques to assess its crystalline structure and Brunauer–Emmett–Teller (BET) analysis to determine its specific surface area.

2.4 Esterification Reaction

The esterification procedure commenced by introducing 117 mL of post-consumer frying oil into a three-necked reaction vessel, to which a combination of methanol and concentrated sulfuric acid (H_2SO_4) was added to function as the catalytic agent. The reagent quantities were established to create a 1-to-12 molar proportion of oil to methanol, with the catalyst dosed at 5 wt% of the oil. Subsequently, the reaction was initiated under microwave irradiation at a 450-Watt power level for a seven-minute duration, during which the contents were continuously agitated.

Following this process, the upper phase of the resulting mixture was isolated. The upper phase consists of the mixture of oil and ester, while the lower phase comprises the mixture of H_2SO_4, water, and excess methanol. The upper phase was separated using liquid-liquid extraction. Subsequently, the upper phase was washed using preheated water at 60°C. The process was followed by adding Na_2SO_4 to the upper phase to remove water. To eliminate Na_2SO_4, the mixture was filtered using filter paper. The upper phase is now referred to as "washed oil." Finally, the washed oil underwent a thermal drying stage in an oven before being analyzed to determine its free fatty acid (FFA) content [13].

2.5 Transesterification Reaction

The procedure began by combining a ZnO/CaO-CA catalytic agent, dosed at 3% of the oil's total mass (w/w), with methanol to establish a 1-to-10 molar proportion of oil to alcohol; this mixture was then introduced into a double-necked boiling flask holding the pre-measured oil. The reaction proceeded under continuous agitation using a high microwave power setting of 600 W, with the duration systematically varied across five setpoints: 4, 5, 6, 7, and 8 minutes. To ensure the temperature remains constant at 60°C, a thermometer was inserted into the reaction vessel. Consequently, the reaction temperature can be monitored in real-time.

Following the reaction, the resulting biodiesel underwent a two-hour thermal treatment in an oven at 105°C [14]. Subsequently, the produced oil was separated and subjected to two analytical techniques: FTIR was utilized to identify the functional groups present, while GC-MS was employed to determine the biofuel's specific components and quantify the amount of fatty acid methyl esters that had formed.

2.6 Physical Characterization of Biodiesel

The procedures for characterizing biodiesel include density, viscosity, acid number, saponification number, and water content determination, as described by Boangmanalu & Ginting [17].

3 Result and Discussion

3.1 Catalyst Preparation and characterization

In powder form, calcium oxide (CaO) has a predominantly white color distinguished by subtle gray tones. After the filtering process, the eggshell powder weighed 645.12 g, and the subsequent burning process reduced the powder's weight to 410.13 g. The chemical decomposition of calcium carbonate ($CaCO_3$) into calcium oxide (CaO), which is the result of heating at high temperatures, can be expressed using the symbolic representation in the **Eq. 1**.

$$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)} \qquad \text{(Equation 1)}$$

An X-ray diffraction (XRD) analysis was performed on the Calcium Oxide (CaO) material to qualitatively identify the constituent phases produced during the catalyst's formation. This was accomplished by examining the resulting diffractogram, which is presented in **Fig. 1**. The pattern reveals several prominent peaks at 2θ angles of 32.19°, 37.34°, 53.85°, 64.14°, and 67.36°. The locations of these peaks are characteristic of Calcium Oxide, as they are in direct agreement with the established standard reference data for pure CaO.

XRD analysis was performed using ICDD data to identify and characterize the crystalline components in the ZnO/CaO-CA and CaO catalysts. The characteristic peaks of ZnO show strong peaks at approximately 2θ = 31.7°, 34.4°, and 36.2°, consistent with ICDD No. 36-1451. The characteristic peaks of CaO show strong peaks at approximately 2θ = 32.2°, 37.3°, and

53.8°. The ZnO/CaO-CA catalyst phase in this research is a crystalline phase because there are three dominant CaO diffraction peaks with relatively high intensity. High crystallinity can also be observed based on the pattern in the XRD diffractogram, which shows narrow and sharp peaks [9].

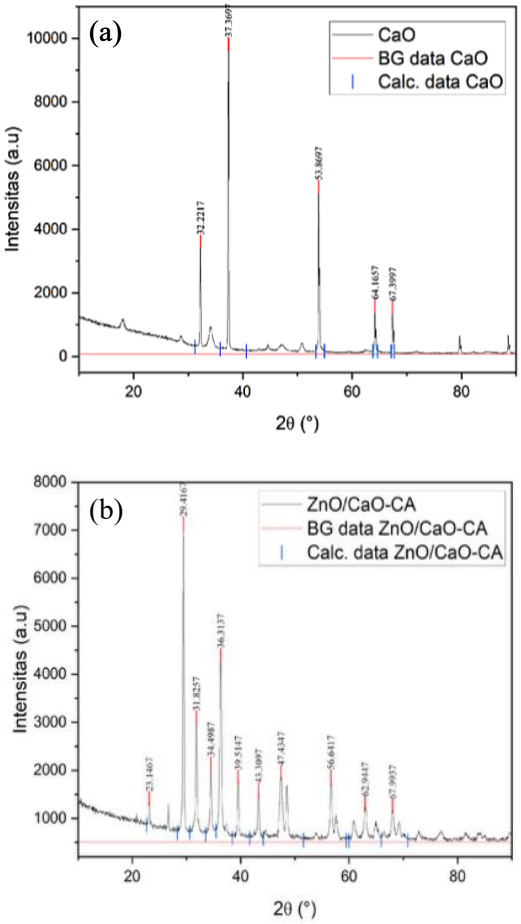

Figure 1. XRD diffractogram of (a) CaO catalyst and (b) ZnO/CaO-CA catalyst

In addition to the primary identified phases, a weak signal at an angle of approximately 2θ = 19° indicates the presence of a minor component or contaminant. This observation is consistent with research by Ismida and Bahri [18], who reported that trace quantities of other substances, such as aluminum oxide (alumina), can exist alongside the main $CaCO_3$ component in materials derived from eggshells. Similarly, an alumina compound was detected at a comparable 2-theta value of 19.80° and exhibited a faint signal in the Siregar et al. study [19].

Thermogravimetric Analysis (TGA) is a thermo-analytical method used to quantify variations in a material's mass. This measurement is recorded either as a response to controlled changes in temperature or as a function of time, all while the sample is maintained within a precisely regulated atmosphere [20]. In this research, TGA (Thermogravimetric Analysis) measures changes in the catalyst's weight as it is heated, allowing us to determine its thermal stability and ensure it does not decompose or undergo unwanted phase transformations at high reaction operating temperatures. The purpose of employing TGA in this study as a preparatory step for the BET methodology is to ensure the accuracy of the temperature chosen during the BET process. If TGA is not conducted prior to BET, the temperature selected during BET may not be precise, resulting in the inability to obtain reliable BET data. This clear connection between the analytical technique and its purpose helps clarify the reasoning behind our methodological choices and will be maintained when discussing other characterization methods throughout this study. In this research, the ZnO/CaO-CA catalyst has a midpoint of 135.56°C.

The Brunauer-Emmett-Teller (BET) methodology was utilized to measure key physical properties of the ZnO/CaO-CA catalyst, namely its specific surface area, total pore volume, and mean pore diameter. The data gathered from this Surface Area Analysis (SAA) are summarized in **Table 1**.

Table 1. Results of Analysis of ZnO/CaO-AC Catalyst Using BET and BJH Methods

Catalyst	Surface area (m^2/g)	Pore Volume (cc/g)	Average Pore Diameter (nm)	Reference
CaO	1.10	0.031	32.00	Haryono et al.,2018
ZnO/CaO-AC	3.82	0.017	22.13	This research

Table 1 presents the surface area of the ZnO/CaO-AC catalyst, which is 3.82 m^2/g, and the surface area of the single CaO catalyst, which is 1.1 m^2/g. This indicates an enhancement in the surface area of the catalyst after the impregnation of ZnO metal with activated carbon support. The determination of the surface area is based on the perfectly adsorbed N_2 molecules on the monolayer layer.

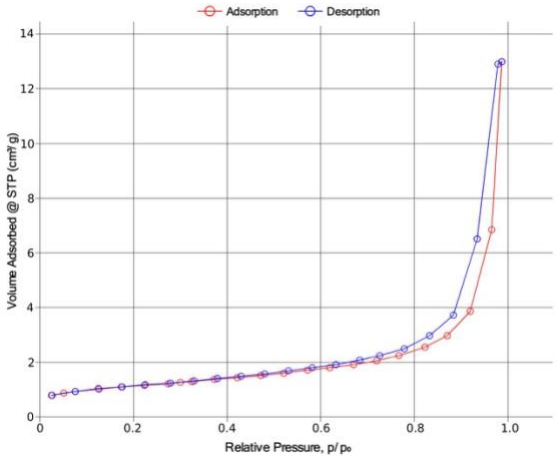

Figure 2. Isotherm curve of ZnO/CaO composite supported on activated carbon.

The isotherm curve depicted in **Fig. 2** exhibits a type III profile, which indicates that the lateral forces of attraction between adsorbed molecules are significantly stronger than the adhesive forces connecting those molecules to the adsorbent's surface [21]. According to the International Union of Pure and Applied Chemistry (IUPAC) system—which categorizes materials as microporous (< 2 nm), mesoporous (2–50 nm), or macro-porous (> 50 nm)—the catalyst produced in this study is classified as a mesoporous material. This categorization is based on its measured pore diameter of 22.1270 nm, a value falling directly within the established mesoporous range.

3.2 Biodiesel Production and characterization

The transesterification reaction was conducted at a microwave power level of 600 Watts, with heating durations carefully selected at 4, 5, 6, 7, and 8 minutes. The selected time intervals were based on preliminary tests indicating optimal reaction progress within this range, ensuring effective conversion and yield. Upon completion of the reaction, two distinct liquid phases formed: a clear, yellow upper layer consisting of fatty acid methyl esters (biodiesel), and a yellowish-brown lower layer containing the glycerol by-product. Phase separation was visually observed and confirmed by decanting the upper layer, followed by further analysis to verify purity. The biodiesel yield can be determined using the **Eq. 2**.

$$yield\ (\%) = \frac{weight_{biodiesel}}{weight_{used\ cooking\ oil}} \times 100\%$$

(Equation 2)

The biodiesel yield from various reaction times (4, 5, 6, 7, and 8 minutes) is determined, and the biodiesel yield is presented in **Table 2**.

Table 2. Biodiesel yield from heating times of 4, 5, 6, 7, and 8 minutes at 600 watts

Time Variation (Minutes)	Yield (%)
4	88.20 %
5	89.62 %
6	87.89 %
7	87.58 %
8	86.77 %

From **Table 2**, the most optimal biodiesel yield from the five microwave heating time variations (4, 5, 6, 7, and 8 minutes) at 600 watts was obtained at 5 minutes with a biodiesel yield of 89.62%. The optimal time in this study is 5 minutes, based on the trial-and-error method conducted by the researcher. If the heating time exceeds 5 minutes, the temperature during the transesterification process at 600 watts may exceed the optimal temperature for biodiesel

The analytical technique of Fourier Transform Infrared Spectroscopy (FTIR) is employed to characterize the composition of biodiesel. It is used to identify compounds, detect functional groups, and analyze samples or mixtures in biodiesel [22].

Fig. 3 presents the FTIR spectra of biodiesel synthesized at 5 and 8 minutes, allowing a direct comparison of the organic functional groups present after different synthesis durations. Biodiesel produced with a reaction time of 8 minutes exhibits identical FTIR spectra to biodiesel produced with a reaction time of 5 minutes. The disparity in physical characteristics arises from the presence of impurities or the potential for biodiesel decomposition at extended reaction times.

Functional groups formed in 600-watt biodiesel at a heating time of 5 minutes, as identified in this study, are fatty acid methyl ester functional groups (biodiesel). These functional groups contain methyl (C-H) and ester (C-O) functional groups. The wavelengths of 3006.148 cm^{-1} and 2920.66 cm^{-1} are included in the 3000-2840 cm^{-1} absorption area. These wavelengths correspond to saturated aliphatic C-H sp^3 groups (-CH_3 and -CH_2-) The FTIR spectrum graph shows a relatively strong peak intensity for these methyl groups.

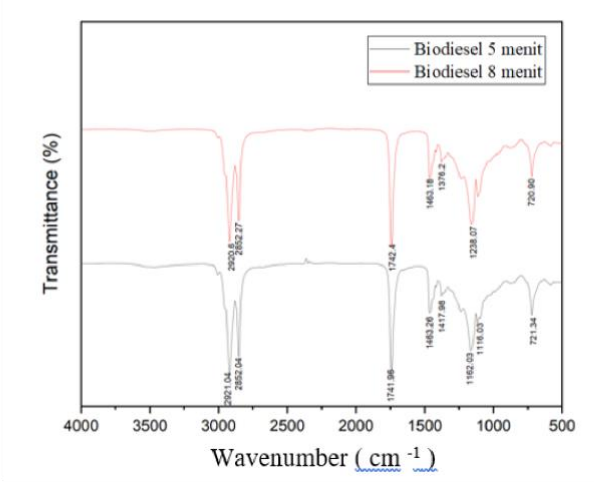

Figure 3. FTIR spectrum of 600-watt biodiesel after 5 and 8 minutes of microwave heating

Examination of biodiesel was performed using Gas Chromatography coupled with Mass Spectrometry (GC-MS) to identify the components resulting from the 5-minute transesterification using the ZnO/CaO-CA catalyst. Among the common fatty acid methyl esters (FAME) found in biodiesel, major compounds such as methyl palmitate, methyl oleate, and methyl stearate were detected.

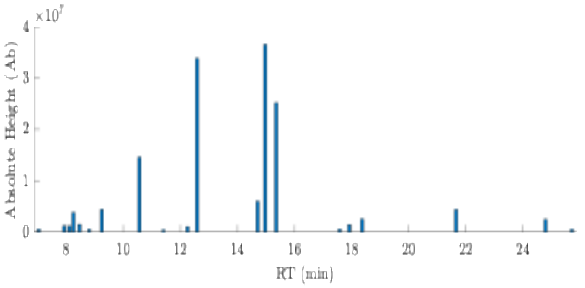

Figure 4. Chromatogram of Biodiesel Produced by Transesterification at 600 watts with a Heating Time of 5 minutes

Based on the chromatogram, dominant peaks were identified among the 20 main components (compounds) in biodiesel, including methyl palmitate and methyl oleate. These compounds are significant because they are indicative of high-quality biodiesel, owing to their favorable combustion properties and efficient energy output. The presence and abundance of these FAMEs reflect the effective conversion of the feedstock into biodiesel and confirm the suitability of the transesterification process under the given conditions. **Fig. 4** presents a comprehensive analysis of the products present in the biodiesel after a heating time of 5 minutes. The figure clearly delineates three distinct peaks, each occurring at specific intervals: 12.58 minutes, 14.97 minutes, and 15.36 minutes.

According to the findings obtained through Gas Chromatography-Mass Spectrometry on previously utilized frying oil, the selected composition of the biodiesel compounds was obtained as shown in **Table 3**.

Table 3. Selected Composition of 5 minutes biodiesel (FAME content) using GC-MS analysis

Rt	Compound Name	Chemical Formula	% Area
12.581	Pentadecanoic acid, 14-methyl ester	$C_{17}H_{34}O_2$	31.89
14.973	cis-13-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	52.69
15.356	Methyl stearate	$C_{19}H_{38}O_2$	6.14

Based on the **Table 3**, it can be said that the transesterification results with a heating time of 5 minutes at 600 watts have been converted into biodiesel. The trio of compounds exhibiting the greatest relative abundance or most prominent peaks consists also belong to the FAME (Fatty Acid Methyl Ester) group. Components belonging to the FAME group include: Pentadecanoic acid, 14-methyl ester (31.89%), cis-13-Octadecenoic acid, methyl ester (52.69%), and Methyl stearate (6.19%).

The FAME group derived from used cooking oil comprises several components, including nonanoic acid, 9-oxo-, methyl ester (0.05%), dodecanoic acid, methyl ester (0.40%), methyl tetra decanoate (1.78%), pentadecanoic acid, methyl ester (0.06%), 6-pentadecenoic acid, 13-methyl ester (0.36%), pentadecanoic acid, 14-methyl ester (31.89%), 9.12-octadecadienoic acid, methyl ester (1.39%), cis-13-octadecenoic acid, methyl ester (52.69%), and methyl stearate (6.19%). The methyl ester content in biodiesel produced by heating a mixture for 5 minutes using a ZnO/CaO-AC catalyst with a purity of 99.19% is determined. This result complies with the SNI standard for a minimum required level of methyl esters at 96.50%.

Biodiesel parameters are analyzed to ensure its quality and characteristics as an alternative fuel that meets established standards [10]. These parameters include viscosity, density, water content, saponification number, and acid number tests. This analysis is conducted to ensure optimal engine performance and to meet applicable

biodiesel quality standards in accordance with SNI biodiesel.

Biodiesel production through transesterification reaction was optimized with a 5-minute heating time based on density, viscosity, acid value, saponification value, and water content. If the heating time is longer, the density, viscosity, acid value, saponification value, and water content of the biodiesel will increase and exceed the SNI value. **Table 4** indicates that the optimal heating duration for biodiesel production from waste cooking oil utilizing ZnO/CaO supported on activated carbon is 5 minutes.

Table 4. Analysis of Biodiesel Product Characteristics Against SNI 7182-2015 Biodiesel

Physical Characteristics	Heating Time (min)					SNI
	4	5	6	7	8	
Density (kg/m^3)	848	897	898	916	919	850-890
Kinematic Viscosity (cst)	4.08	3.91	4.17	5.09	5.08	2.3-6.0
Acid Number (mg KOH/g)	0.35	0.44	0.50	0.62	0.84	Max 0.6
Saponification Number (mg KOH/g)	143	130	123	123	108	Max 500
Water Content (%)	0.05	0.04	0.06	0.11	0.17	Max 0.05

Table 4 reveals that despite the diverse time reactions presented, the density remains outside the acceptable range defined by the SNI standard. Notably, there is a significant increase in the density value between reaction times of 4 minutes and 5 minutes. This suggests that the reaction is not yet fully complete at 4 minutes. The GC chromatogram at 5 minutes of reaction time indicated the presence of several components related to unreacted used cooking oil. These components are decanal and dodecanal. Both unreacted components exhibit a density range of 0.841-0.848 g/mL [23]. This density range aligns with the density of biodiesel at 4 minutes of reaction time.

4 Conclusions

The ZnO/CaO-AC catalyst demonstrated a significantly enhanced surface area of 3.822 m²/g compared to the conventional CaO catalyst, which exhibited a surface area of only 1.5 m²/g. During the transesterification process, reactions were conducted at a temperature of 60°C, employing a oil-to-methanol molar ratio of 1:10 and a catalyst loading of 3 wt%. Under these specific conditions, an optimal reaction time of 5 minutes was determined among the five tested intervals, resulting in an exceptionally high biodiesel yield of 89.62%. The biodiesel produced at this optimal duration demonstrated superior quality, with its key physical and chemical properties closely aligning with the specifications of the SNI 7182-2015 standard. GC-MS (Gas Chromatography-Mass Spectrometry) analysis confirmed a methyl ester content of 99.19%, indicating a high purity level of biodiesel. This exceptional quality means the biodiesel is highly suitable for commercial use and can reliably meet regulatory requirements, potentially improve engine performance and reduce emissions. These findings underscore the significance of achieving such a high methyl ester content and demonstrate the effectiveness of the transesterification process under the optimized conditions.

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