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Physicochemical Characterization and Recyclability of CaO/SiO₂Catalysts Derived from Eggshell and Rice Husk for Biodiesel Application

Eti Ayu Nurhana[1], Meka Saima Perdani[1*], Hilman Imadul Umam[2], Amalia Dian Fadilla[1], Christin Octaviani Sitanggang[1]

[1]Department of Chemical Engineering, Faculty of Engineering, Universitas Singaperbangsa Karawang, Jl. HS. Ronggowaluyo, Karawang 41361, Indonesia
[2]Department of Physics, Faculty of Engineering, Universitas Singaperbangsa Karawang, Jl. HS. Ronggowaluyo, Karawang 41361, Indonesia
Corresponding Author: meka.perdani@ft.unsika.ac.id

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Abstract

This study aims to evaluate the physicochemical characteristics and reusability of CaO/SiO_2 heterogeneous catalysts synthesized from eggshell and rice husk waste for biodiesel production. The catalyst, prepared with a 2:1 CaO to SiO_2 ratio, was applied in three transesterification cycles using waste cooking oil. Between cycles, the used catalyst was washed with n-hexane and dried at 80 °C for 12 hours. Catalyst characterization was performed using SEM-EDX, FTIR, and N_2 adsorption-desorption analysis. The biodiesel yield decreased from 65 ± 4.95% (first cycle) to 54 ± 1.41% and 46 ± 2.12% in subsequent cycles, indicating reduced catalytic activity. SEM-EDX revealed particle agglomeration, calcium content declined (from 24.78% to 19.22%), and increased silicon exposure (from 4.66% to 21.86%). FTIR analysis detected organic residue accumulation, while N_2 adsorption-desorption results showed a decrease in surface area (20.79 to 11.67 m²/g) and pore volume (0.03 to 0.02 cc/g), with increased pore size. The biodiesel showed a density of 1112–1119 ± 1.41 kg/m³ and a kinematic viscosity of 2.03–2.07 cSt, indicating it still requires purification to meet SNI 7182:2015 standards. These findings highlight the catalyst's promising initial performance and underscore the need for regeneration strategies to maintain catalytic efficiency over multiple uses.

Keywords: CaO/SiO_2 catalyst, eggshell, rice husk, biodiesel, physicochemical characterization, reusability

INTRODUCTION

The global reliance on fossil fuels has played a critical role in exacerbating the ongoing energy crisis and contributing to a range of environmental issues, including elevated greenhouse gas emissions, deteriorating air quality, and adverse effects on both public health and ecological integrity (Wang & Azam, 2024). In response to these issues, biodiesel is considered a more environmentally friendly and sustainable alternative fuel due to its biodegradable nature, non-toxicity, and lower exhaust emissions compared to conventional petrodiesel (Balpande et al., 2025; Pugazhendhi et al., 2020; Salihu et al., 2021). In Indonesia, biodiesel consumption shows a significant increase from 8.40 million kiloliters in 2020 to 13.16 million kiloliters in 2024, as shown in Figure 1, reflecting the growing urgency for renewable energy utilization and the need for a sustainable feedstock supply (Balpande et al., 2025; Kementerian ESDM, 2024). In this case, waste cooking oil (WCO) offers a sustainable and economically favorable feedstock for biodiesel production, as it does not interfere with edible oil supplies (Hamed et al., 2021). However, its successful conversion via transesterification largely depends on the effectiveness of the catalyst used (Basumatary et al., 2021; Pugazhendhi et al., 2020).

Catalysts are crucial for enhancing the transesterification reaction, and they are broadly divided into homogeneous and heterogeneous types based on their phase relative to the reactants (Ali et al., 2020; Hamed et al., 2021). Because of their great efficacy and affordable cost, Homogeneous catalysts like NaOH and KOH are frequently utilized to manufacture biodiesel (Hoang et al., 2021). However, they dissolve easily in alcohol, resulting in their presence in the final product, complicating the separation process between biodiesel and glycerol (Aslan & Eryilmaz, 2020). As an alternative,

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Heterogeneous catalysts have benefits, especially the ease with which they can be separated from the end product once the reaction is finished (Burmana et al., 2023). Commercial heterogeneous catalysts, such as pure CaO, MgO, and synthetic zeolites, are typically more costly than homogeneous catalysts despite these benefits (Zhang et al., 2025; Zhu et al., 2021; Zulkurnain et al., 2024).

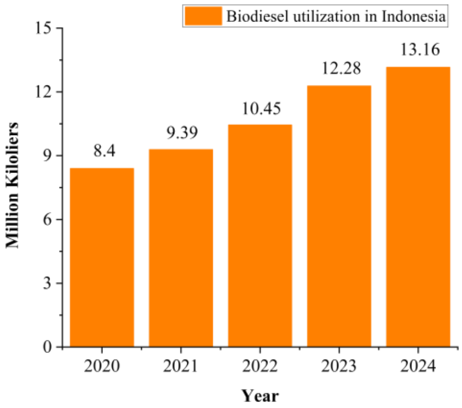


Figure 1. Domestic biodiesel utilization trend in Indonesia (Kementerian ESDM, 2024).

Biomass-based catalysts such as CaO from eggshells and SiO_2 from rice husk present a viable alternative to conventional homogeneous catalysts for converting WCO into biodiesel, while also offering the benefit of recyclability across several reaction cycles (Fatin et al., 2024). According to earlier research, CaO catalysts can be recycled up to five times while producing more than 60% biodiesel (Rahman et al., 2022). Similarly, silica as a support material in catalysts such as Zn/SiO_2 has demonstrated good regeneration performance, achieving yields greater than 80% after five cycles (Fatimah et al., 2022). Therefore, combining CaO and SiO_2 derived from biomass waste can produce heterogeneous catalysts that are cost-effective and environmentally friendly and exhibit high regeneration efficiency and stability in biodiesel production processes (Manurung et al., 2023).

However, the changes in the physicochemical characteristics of CaO/SiO_2 catalysts after multiple reuse cycles, including functional group analysis, surface morphology, elemental composition, surface area, and pore volume, all of which influence biodiesel yield, still require further investigation. The outcomes of this research may serve as a valuable reference for advancing the development of more efficient and environmentally friendly catalysts in biodiesel production technology.

METHODOLOGY

Materials and Instrumentals

The study's materials were rice husks and chicken eggshells collected from agricultural and domestic waste in Klari, Karawang, Indonesia. The chemicals used were a 20% KOH solution, a 6% NaOH solution, distilled water, methanol (CH_3OH, 99.9%, Merck), technical-grade n-hexane, and waste cooking oil collected from local food vendors in Karawang.

Experimental work was supported by several instruments, such as a furnace, drying oven, pH meter, thermometer, hotplate magnetic stirrer, analytical balance, 500 mL flat-bottom three-neck flask, and standard laboratory glassware. Catalyst characterization was conducted using SEM-EDX (Hitachi SU3500, EDX EDAX TEAM UF-240481), FTIR (Shimadzu IRSpirit-4600 ATR Pro One), and BET surface area analyzer (QuadraSorb Station 2). The quality of the produced biodiesel was evaluated by determining its density with a pycnometer and assessing its kinematic viscosity using an Ostwald viscometer.

Methods

Synthesis of CaO/SiO_2 Catalyst

Eggshells were washed with distilled water, dried in an oven at 110 °C for 24 hours, then crushed and sieved using a 60 mesh sieve. The shell powder was calcined at 800 °C for 4 hours, dried again at 100 °C, and then impregnated with 20% KOH solution at 60 °C for 24 hours. The mixture was dried for an hour at 100 °C, filtered through filter paper, and recalcined for 4 hours at 450 °C.

Rice husks were soaked in hot water and left overnight. The settled husks were then rinsed, oven-dried at 80 °C for 24 hours, and calcined in a furnace at 800 °C for 5 hours to produce rice husk ash (RHA). The resulting ash was ground to a fine powder, then mixed with calcined CaO powder at a 2:1 ratio. The mixture was recalcined at 500 °C for 4 hours to obtain the CaO/SiO_2 catalyst, as shown in Figure 2.

Pretreatment of Waste Cooking Oil

WCO is heated at a temperature of 100-115 °C until the water evaporates, then neutralized by mixing 6% NaOH to reduce the FFA content and pH to 6-7, as shown in Figure 2. The oil with a neutral pH is filtered using filter paper to separate large solid impurities and soap formed due to the addition of NaOH.

Transesterification and Biodiesel Characterization

The process of transesterification involved combining WCO, methanol, and CaO/SiO_2 catalyst

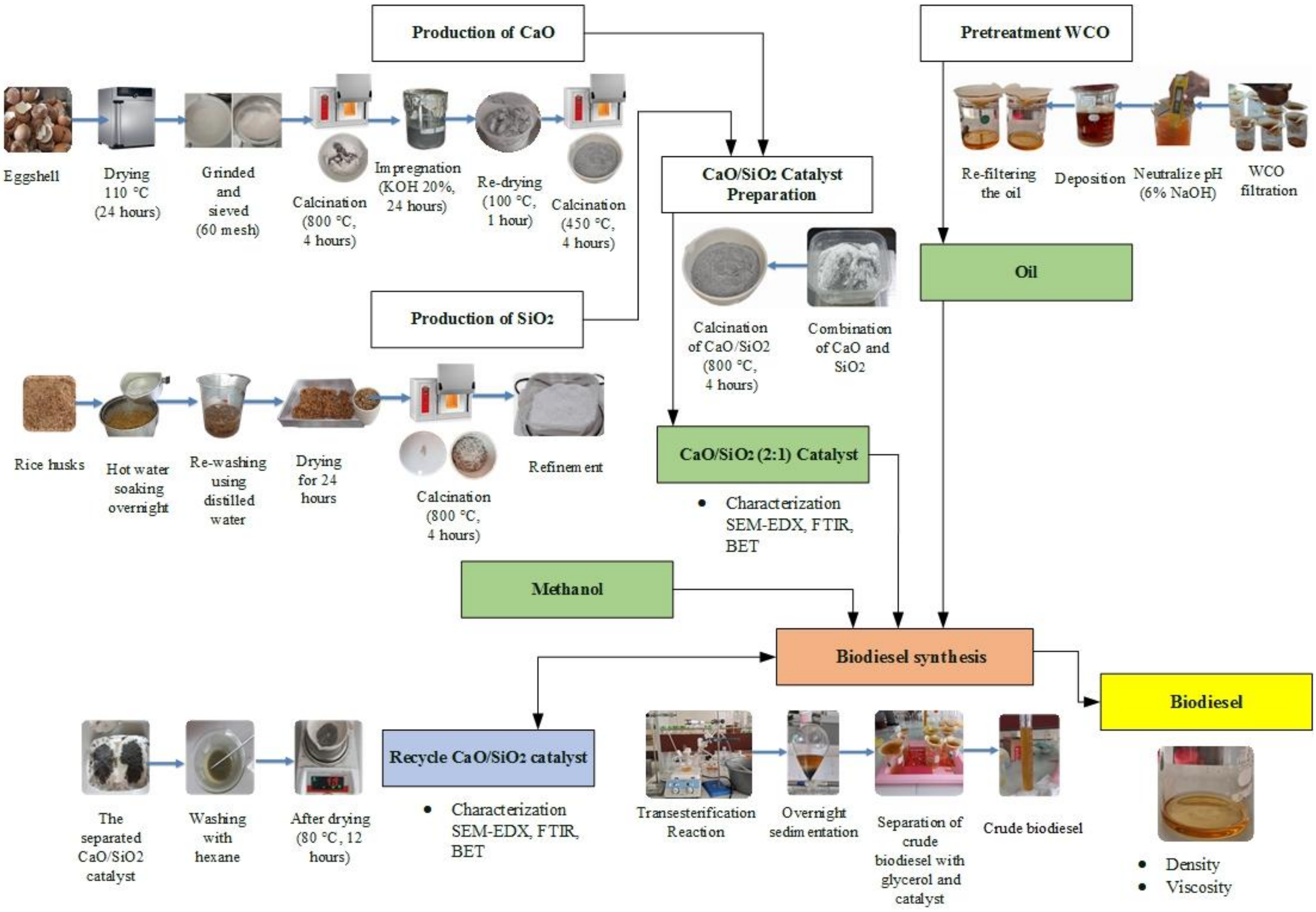


Figure 2. Process flow diagram of CaO/SiO_2 catalyzed biodiesel production and catalyst recycling.

into a 500 mL three-neck round-bottom flask equipped with a condenser, magnetic stirrer, and thermometer, as shown in Figure 2. The reaction was conducted at 65 ± 2 °C for 2 hours and 30 minutes, using an oil-to-methanol molar ratio of 1:9 and a catalyst loading of 4 wt% based on the oil mass. Following the completion of the reaction, the mixture was allowed to cool to ambient temperature before being placed into a separating funnel and allowed to settle for 12 hours, during that time three different layers formed. The catalyst was in the bottom layer, followed by glycerol in the middle and biodiesel at the top. The biodiesel phase was then separated, collected, and analyzed. The biodiesel yield was calculated using Equation (1). In addition, biodiesel quality testing was carried out by measuring density and kinematic viscosity at 40 °C.

$$Biodiesel\ yield\ \% = \frac{Mass\ of\ Biodiesel}{Mass\ of\ WCO\ Used} \times 100\% \quad (1)$$

Recycle CaO/SiO_2 Catalyst

The spent CaO/SiO_2 catalyst was separated from the reaction mixture and washed with n-hexane to remove residual triglycerides and by-products, as shown in Figure 2. It was then dried at 80 °C for 12 hours and reused in subsequent transesterification reactions for up to three cycles.

Characterization of CaO/SiO_2 Catalysts

Catalyst characterization was carried out to compare the CaO/SiO_2 (2:1) catalyst and the recycled catalyst, CaO/SiO_2, after the third transesterification cycle. SEM-EDX (Hitachi SU3500, EDAX TEAM UF-240481) was used to observe surface morphology and elemental composition. FTIR analysis (Shimadzu IRSpirit-4600 ATR Pro One) was performed to identify functional groups and detect structural changes after reuse. N_2 adsorption-desorption analysis (QuadraSorb Station 2) was used to quantify the surface and pore features of the catalyst, providing insight into its potential catalytic activity.

RESULTS AND DISCUSSION

Surface Morphology and Elemental Composition of CaO/SiO_2 Catalyst

This characterization was evaluated structural and compositional changes in the catalyst after multiple uses. Scanning Electron Microscopy (SEM) analysis of the CaO/SiO_2 (2:1) catalyst at 5000× magnification showed an irregular, rough, and agglomerated surface morphology, as shown in Figure 3. This morphology is consistent with the typical characteristics of amorphous RH-SiO_2 and CaO, which tend to form aggregated rectangular particles (Elfina et al., 2024). Agglomeration may also result from the hygroscopic nature of CaO, which absorbs moisture during sample handling (Manurung et al., 2023). The particle size distribution histogram in Figure 3 shows an average particle diameter of 3.15 µm with a standard deviation of 2.25 µm. Although the size reflects particle aggregates, it remains within an acceptable range for heterogeneous catalysts (Manurung et al., 2023; Nayak, 2020).

Significant morphological changes were observed after three reuse cycles, as shown in Figure 3. The surface became rougher and more inhomogeneous, with intensified particle aggregation, likely caused by sintering during repeated thermal exposure and reaction conditions. This morphological evolution contributed to pore narrowing and reduced active surface area (Song et al., 2023; Zhang et al., 2025). The average particle diameter increased to 4.12 µm with a standard deviation of 2.84 µm, indicating a decrease in specific surface area (Song et al., 2023). In addition, residual reactants or by-products, possibly including glycerol, may remain adsorbed on the catalyst surface even after washing, potentially causing pore blockage and limiting access to active sites (Burmana et al., 2023).

Energy Dispersive X-ray (EDX) analysis of the CaO/SiO_2 (2:1) catalyst, as shown in Figure 4, revealed the dominant presence of calcium (Ca) and oxygen (O), along with a significant amount of silicon (Si), indicating the contribution of SiO_2 in the catalyst matrix (Sameri et al., 2021). The high oxygen content comes from the oxide structure of CaO and SiO_2 (Hachem et al., 2023; Priya et al., 2020). However, in the reused catalyst, there was a decrease in calcium intensity to 19.22%; in comparison, silicon increased to 21.86%, indicating the dominance of the surface by SiO_2 due to the closure of the CaO phase in the silica matrix (Sameri et al., 2021). Ca leaching was inferred from EDX observations; ICP-OES/ICP-MS analysis was not performed. The oxygen content also decreased to 58.92%, indicating the degradation of the active CaO phase during the reaction (Saadiah et al., 2019), contributing to the overall decline in catalyst performance.

Functional Group Analysis of CaO/SiO_2 Catalyst

Functional group identification and structural assessment of the CaO/SiO_2 catalyst before and after reuse were performed using FTIR spectroscopy. This

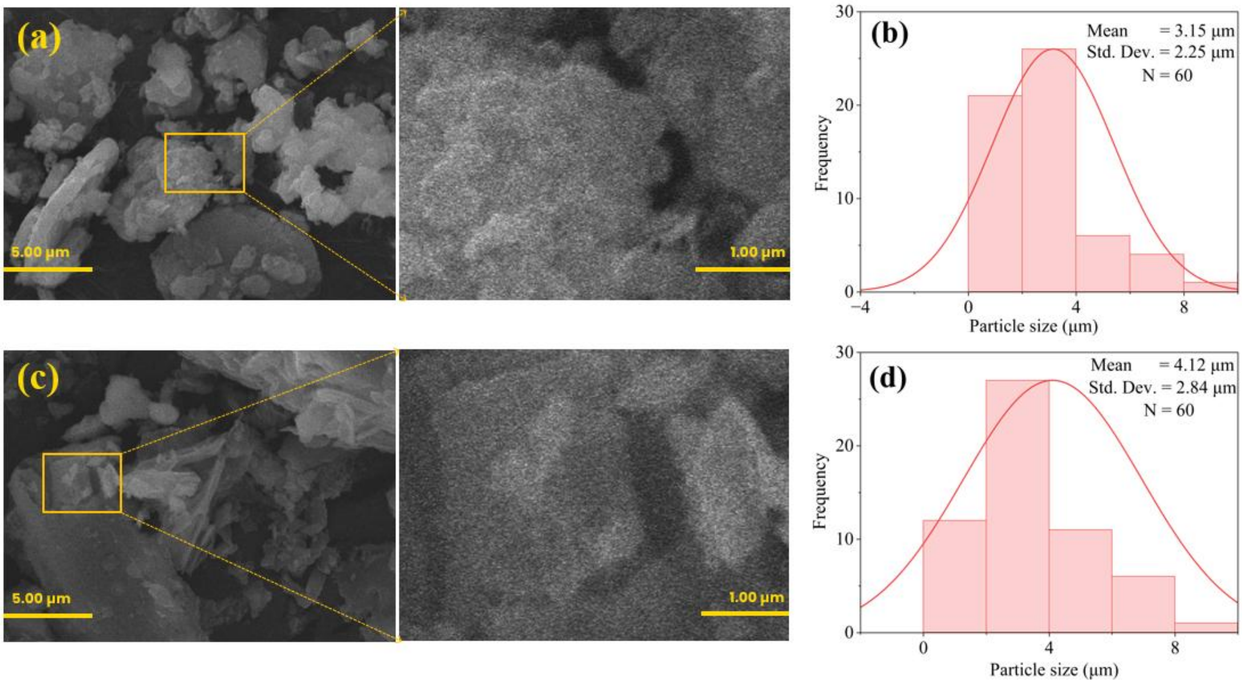
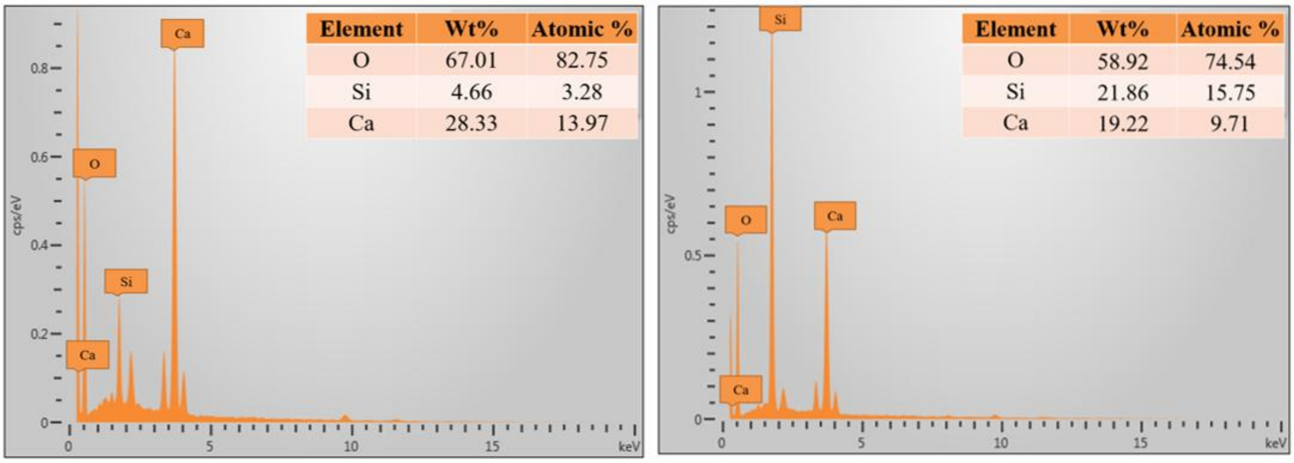


Figure 3. (a) SEM image of CaO/SiO_2 (2:1) catalyst, (b) particle size distribution of CaO/SiO_2 (2:1) catalyst, (c) SEM image of recycled CaO/SiO_2 (2:1) catalyst, and (d) particle size distribution of recycled CaO/SiO_2 (2:1) catalyst



Element	Wt%	Atomic %
O	67.01	82.75
Si	4.66	3.28
Ca	28.33	13.97

Element	Wt%	Atomic %
O	58.92	74.54
Si	21.86	15.75
Ca	19.22	9.71

(a) **(b)**

Figure 4. (a) EDX spectrum of CaO/SiO_2 (2:1) catalyst and (b) EDX spectrum of recycled CaO/SiO_2 (2:1) catalyst.

analysis is essential to understand the chemical interactions at the catalyst surface, which directly influence its activity and reusability (Hazmi et al., 2020; Manurung et al., 2023). The FTIR spectrum, presented in Figure 5, exhibited characteristic carbonate absorption bands at 874 and 1417 cm⁻¹ and in-plane vibrations at 711 cm⁻¹, indicating the presence of CO_3^{2-} groups bound to the CaO surface (Rahman et al., 2022; Saadiah et al., 2019). Additionally, peaks at 796 and 1057 cm⁻¹ correspond to Si–O–Si stretching vibrations, confirming the presence of siloxane groups typical of silica derived from rice husk (Febryzha et al., 2025; Hazmi et al., 2020; Manurung et al., 2023). A weak band at 2972 cm⁻¹ (C–H stretching) indicates the presence of organic residues (Wan et al., 2021).

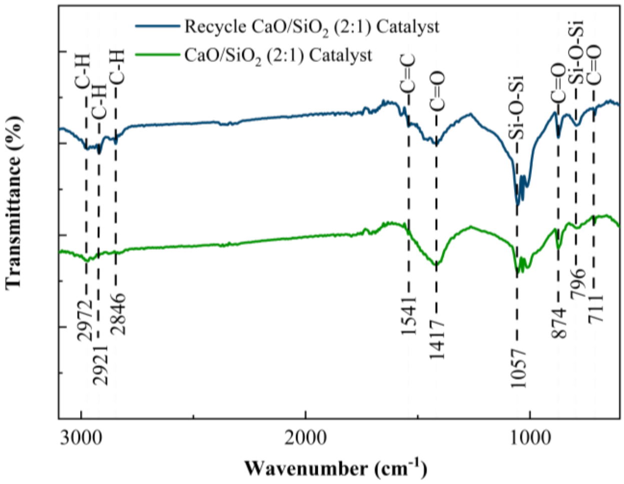


Figure 5. FTIR of CaO/SiO_2 (2:1) and Recycle CaO/SiO_2 (2:1) Catalyst.

The development of new bands at 2921 and 2846 cm⁻¹, which correspond to the asymmetric and symmetric stretching vibrations of C–H bonds in $–CH_2–$ groups, indicated minor modifications in the FTIR spectra after three reuse cycles (Sitanggang & M. Aras, 2024). These bands suggest the accumulation of organic residues, such as lipids or biodiesel reaction by-products, adsorbed on the catalyst surface (Kumar et al., 2020; Mansour et al., 2022). In addition, a peak at 1546 cm⁻¹, associated with C=C stretching, further indicates the presence of carbonaceous contaminants. This functional group likely originates from unsaturated organic compounds, such as unreacted feedstock or biodiesel by-products (Mansour et al., 2022; Sitanggang & M. Aras, 2024). Although the catalyst was washed and dried before characterization, such contamination may block active sites and reduce catalytic performance (Puspitasari, 2024). Therefore, more effective regeneration strategies, such as high-temperature calcination or specific solvent treatment, may be necessary to restore catalytic activity (Rahman et al., 2022). The complete assignment of functional groups observed in the FTIR spectra is summarized in Table 1.

Pore Structure Analysis of CaO/SiO_2 Catalyst

Surface analysis of the CaO/SiO_2 catalyst was carried out using N_2 adsorption–desorption analysis, which provided data on its specific surface area, overall pore volume, and average pore diameter. As presented in Table 2, N_2 adsorption–desorption analysis of the CaO/SiO_2 (2:1) catalyst revealed that its textural properties remain within the typical range reported in previous studies. The surface area of the CaO/SiO_2 (2:1) catalyst was 20.79 m²/g, which falls within the

Table 1. Results of Identification of Functional Groups of CaO/SiO2 Catalyst (2:1) Based on FTIR.

Wavenumber (cm^{-1})	Wavenumber Referance (cm^{-1})	Functional Group	Type of Vibration	Referance
711	711-712	C=O	In-plane bending	(Awogbemi et al., 2020; Saadiah et al., 2019)
796	788-797	Si-O-Si	Stretching	(Manurung et al., 2023; Nayak, 2020)
874	873-875	C=O	Out-of-Plane bending	(Awogbemi et al., 2020; Rahman et al., 2022; Saadiah et al., 2019)
1057	1057-1169	Si–O–Si	Stretching	(Hazmi et al., 2020; Taqiya & Samik, 2025)
1417	1403-1424	C=O	Asymmetric stretching	(Saadiah et al., 2019)
1541	1541-1546	C=C	Stretching	(Blaise et al., 2021)
2846	2846-2972	C-H	Symmetric stretching	(Blaise et al., 2021; Kumar et al., 2020; Wan et al., 2021)
2921			Asymmetric stretching	
2972			Stretching	

range of 14.41–21.48 m^2/g reported in previous studies (Manurung et al., 2023). Similarly, the total pore volume of 0.03 cm^3/g is consistent with previous reports on CaO/SiO_2 catalysts, which ranged between 0.01-0.05 cm^3/g (Khazaai et al., 2021; Manurung et al., 2023) The average pore size was 5.48 nm, which falls within the mesoporous range (2–50 nm) and is highly suitable for the transesterification process, as it can accommodate large molecules such as triglycerides (Mora et al., 2024). However, significant reductions in surface area and pore volume were observed after reuse. In this study, after three cycles of catalyst reuse, the surface area 11.67 m^2/g and pore volume 0.02 cm^3/g decreased, which is consistent with previous reports attributing this to possible pore blockage by product residues (Burmana et al., 2023). Although the catalyst was washed with n-hexane, some impurities are presumed to remain. Meanwhile, the average pore size increased to 6.50 nm, which still falls within the mesoporous range and remains suitable for transesterification (Mora et al., 2024).

Based on the nitrogen adsorption–desorption curve in Figure 6, both CaO/SiO_2 (2:1) catalysts and recycled CaO/SiO_2 (2:1) catalysts exhibit nitrogen adsorption–desorption behavior characteristic of Type IV isotherms with H3 hysteresis loops, as categorized by IUPAC. Such behavior is characteristic of mesoporous structures, which generally possess pore diameters between 2 and 50 nm and slit-shaped pores resulting from the aggregation of irregular particles (Lee et al., 2021). The isotherm displays a hysteresis loop between 0.5 and 0.8 P/Po, indicating capillary condensation within the mesopores (Manurung et al., 2023).

Table 2. N_2 adsorption–desorption analysis of CaO/SiO_2 Catalyst.

Catalyst	Multipoint BET Surface Area (m^2/g)	Total Pore volume (cc/g)	Average Pore Size (nm)
CaO/SiO_2 (2:1)	20.79	0.03	5.48
Recycle CaO/SiO_2 (2:1)	11.67	0.02	6.50

The presence of a mesoporous structure is critical in biodiesel production applications, as it enables the diffusion of large molecules such as triglycerides into the catalyst pores during the transesterification process. This enhances reactant accessibility to active sites and promotes catalytic efficiency (Lee et al., 2021; Manurung et al., 2023; Zhu et al., 2021). This study performed N_2 adsorption–desorption analysis on the CaO/SiO_2 catalyst after three reuse cycles. Average pore size was calculated from the average pore radius obtained from N_2 adsorption–desorption data.

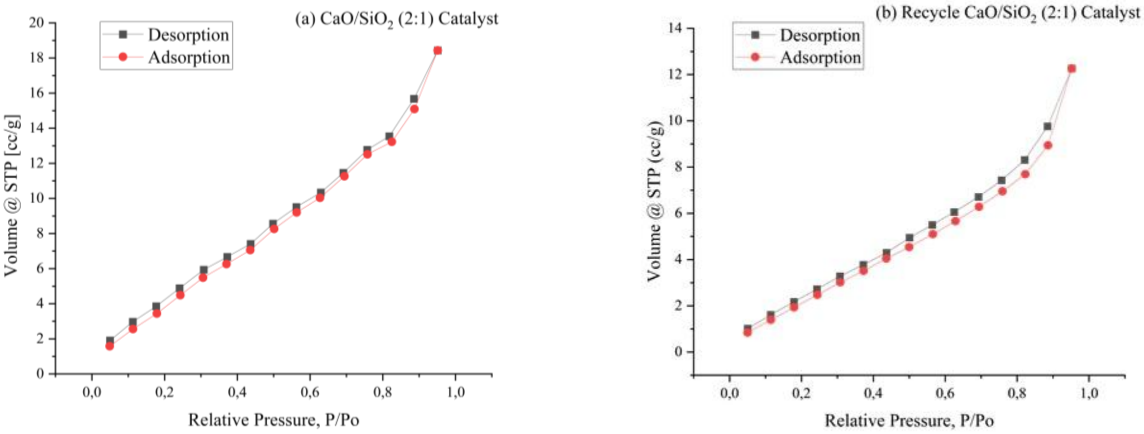


Figure 6. N_2 adsorption-desorption graph catalyst (a) CaO/SiO$_2$ (2:1) and (b) Recycle CaO/SiO$_2$ (2:1).

Reusability of CaO/SiO$_2$ Catalyst

Heterogeneous catalysts such as CaO/SiO$_2$ have the advantage of being reusable, but their effectiveness is greatly influenced by structural stability and resistance to degradation during the reaction (Palitsakun et al., 2021). In this study, the CaO/SiO$_2$ catalyst underwent regeneration through hexane washing and drying at 100 °C prior to reuse, indicated by a decrease in biodiesel yield from 65 ± 4.95% (cycle 1) to 54 ± 1.41% (cycle 2) and 46 ± 2.12% (cycle 3), as shown in Figure 7. The observed drop in performance may be attributed to a loss of catalytically active sites due to calcium leaching into the reaction phase and the presence of surface poisoning from the adsorption of glycerol and triglycerides (Rahman et al., 2022; Saadiah et al., 2019; Yusuff et al., 2021).

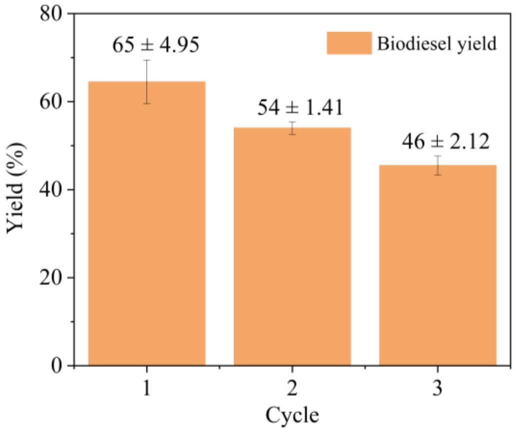


Figure 7. Catalyst Reusability on Biodiesel.

Changes in the catalyst's physicochemical characteristics also contribute to the decline in efficacy and chemical considerations. A decrease in the active surface area brought on by sintering and particle aggregation from repeated drying directly affects catalyst performance (Palitsakun et al., 2021). The washing process with hexane, although effective in removing residues, also risks reducing the amount of active material, so the effective catalyst mass decreases with each cycle (Saadiah et al., 2019). In this study, the catalyst mass decreased from 6 g initially to 4.34 ± 0.21 g after the first cycle, 3.34 ± 0.31 g after the second cycle, and 1.62 ± 0.64 g after the third cycle, confirming that the loss of catalyst mass also contributed to the observed decline in biodiesel yield.

Physicochemical Properties of Crude Biodiesel

According to its physicochemical characteristics, biodiesel was evaluated to determine the impact of catalyst reuse on fuel quality. As shown in Figure 8, the density values obtained in each cycle were 1119 ± 1.41 kg/m³ (cycle 1), 1115 ± 1.41 kg/m³ (cycle 2), and 1112 ± 0 kg/m³ (cycle 3), which exceed the SNI 7182:2015 standard range of 850–890 kg/m³. The high density suggests that the biodiesel remains unrefined, with residual impurities such as glycerol, methanol, and catalyst particles. This condition is likely due to the absence of additional purification steps (e.g., water washing) prior to measurement, leading to a higher density compared to pure FAME (Pugazhendhi et al., 2020; Ramanan et al., 2022; Taqiya & Samik, 2025).

The kinematic viscosity of biodiesel is presented in Figure 9, showing values of 2.065 ± 0.007 cSt (cycle 1), 2.05 ± 0 cSt (cycle 2), and 2.03 ± 0 cSt (cycle 3), which fall slightly below the SNI 7182:2015 requirement (2.3–6.0 cSt) but remain within the ASTM D6751 standard (1.9–6.0 cSt). Since viscosity is directly related to the purity of fatty acid methyl esters (FAME), the lower values can be attributed to residual methanol that reduces viscosity and incomplete transesterification due to declining catalyst activity after repeated use (Mora et al., 2024). Future studies should focus on implementing purification steps to improve biodiesel quality and ensure full compliance with SNI standards.

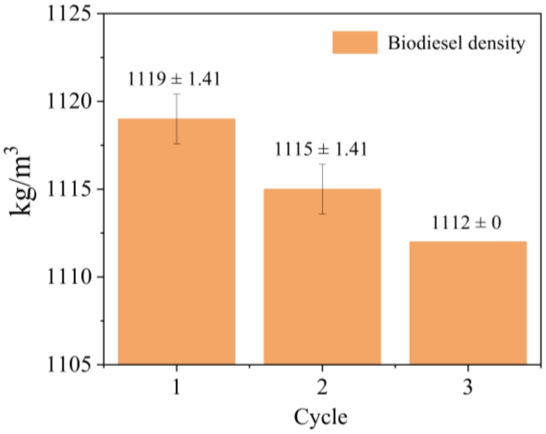


Figure 8. Biodiesel Density.

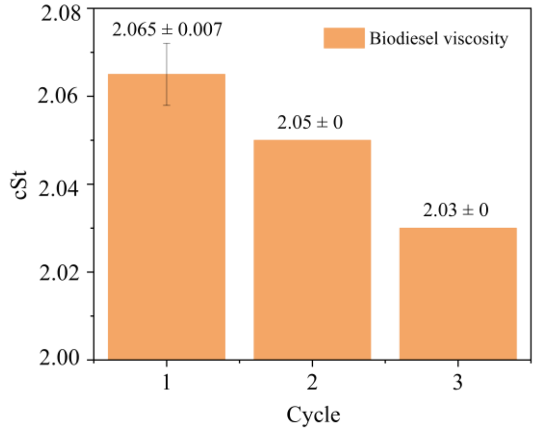


Figure 9. Biodiesel Viscosity.

CONCLUSION

The CaO/SiO_2 heterogeneous catalyst synthesized from waste eggshells and rice husks exhibited promising initial physicochemical characteristics, with a specific surface area of 20.79 m^2/g, pore volume of 0.03 cc/g, and a mesoporous structure favorable for transesterification. FTIR confirmed the presence of active carbonate and siloxane groups, while SEM-EDX revealed a calcium-rich surface. After three reuse cycles, significant deterioration was observed, including a reduction in surface area to 11.67 m^2/g, increased particle agglomeration, and a shift in elemental composition toward silica dominance. This structural degradation correlated with a progressive drop in biodiesel yield from 65 ± 4.95% to 46 ± 2.12%. Additionally, the resulting crude biodiesel exceeded the SNI 7182:2015 density limits and exhibited substandard viscosity, despite falling within ASTM D6751. These findings demonstrate that while the catalyst holds initial potential, its physicochemical instability during reuse critically limits its application, emphasizing the need for regeneration strategies to ensure sustained performance and fuel quality.

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