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Original Article

Modification of Calcium Oxide Catalyst Derived from Chicken Eggshell Waste with Magnesium Oxide to Enhance Biodiesel Synthesis from Crude Palm Oil

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Abstract— Biodiesel is a promising renewable energy source to replace conventional diesel fuel. This study aims to improve biodiesel production efficiency by utilizing a heterogeneous catalyst derived from chicken eggshell-based calcium oxide (CaO), modified with magnesium oxide (MgO). The catalyst was synthesized via co-precipitation and applied in both transesterification and interesterification reactions using crude palm oil (CPO) as feedstock. The optimum reaction was conducted at 65°C for 180 minutes with a 9:1 methyl acetate/methanol to oil molar ratio and 2.5 wt% catalyst loading. The modified CaO.MgO catalyst outperformed unmodified CaO, yielding 89.63% and 99.96% biodiesel via interesterification and transesterification, respectively. The catalyst characterization was performed using FTIR, XRD, and GC-MS, confirming the enhancement in basicity, crystallinity, and FAME content. This study demonstrates the potential of waste-derived catalysts in sustainable biodiesel production while addressing environmental waste issues.

Keywords— *Biodiesel; Calcium oxide; Catalyst; Chicken eggshell; Magnesium oxide.*

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1. INTRODUCTION

Indonesia is the world's largest contributor of palm oil commodities, accounting for 59% of total global production with an annual production volume of 45.5 million tons [1]. However, an embargo by the European Union [2] has caused palm oil commodities to flood the market. This has led to the empowerment of palm oil to be used as biodiesel. The synthesis method used is interesterification. Interesterification is a chemical process in which ester groups in two or more different molecules exchange places, producing a new mixture of esters [3].

This method is another alternative to transesterification for producing biodiesel. Transesterification uses alcohol to supply alkyl groups in producing esters, and also produces glycerol as a by-product that is difficult to utilize further [4]. In this study, the interesterification process uses methyl acetate as an alkyl group supplier so that the by-product is superior for further use compared to the common transesterification method, namely triacetin.

Several studies on the biodiesel interesterification process use heterogeneous and homogeneous catalysts. Homogeneous catalysts such as potassium hydroxide and sodium hydroxide provide fast reaction rates [5], but heterogeneous catalysts receive greater attention due to their separation effectiveness. One example of a heterogeneous catalyst is calcium oxide. Calcium oxide (CaO) can be obtained from the conversion of calcium carbonate ($CaCO_3$) through a calcination process. One source of $CaCO_3$ is chicken eggshells. Indonesia itself produced 170,000 tons of chicken eggshell waste in 2019 [6].

With hundreds of thousands of tons of eggshell waste, there is great potential to utilize it as an environmentally friendly and low-cost CaO catalyst. CaO catalyst has added value as a potential source of calcium, which is abundant and inexpensive, and has a relatively low impact because its separation is simple [7]. However, CaO catalysts have low stability against moisture and high temperatures, so modifications using magnesium

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oxide were made in this study to improve their catalytic performance. This approach not only supports waste valorization but also aligns with sustainable energy development goals. Ultimately, the findings of this study could provide a practical pathway for developing efficient and eco-friendly catalysts.

Although calcium oxide (CaO) is widely used as a heterogeneous base catalyst due to its strong basicity, low cost, and abundance, it suffers from several inherent limitations, such as poor thermal stability, high sensitivity to moisture, and rapid deactivation caused by carbonation when exposed to atmospheric CO_2. These drawbacks can significantly reduce catalytic activity and reusability during biodiesel synthesis, particularly under prolonged reaction times and elevated temperatures.

To address these limitations, modification of CaO with magnesium oxide (MgO) has been proposed. MgO exhibits higher thermal stability and better resistance to moisture compared to CaO. When incorporated into the CaO structure, MgO acts as a structural promoter that suppresses CaO sintering, enhances catalyst durability, and maintains strong basic active sites. The synergistic interaction between CaO and MgO improves catalytic performance, making the modified CaO.MgO catalyst more suitable for biodiesel synthesis.

2. EXPERIMENTAL SECTION

2.1. Materials and Instrumentations

The materials used in this study included chicken eggshell waste, crude palm oil (CPO), methanol (≥99.8%, Merck), methyl acetate (≥99%, Sigma-Aldrich), magnesium oxide (MgO, ≥99%, analytical grade, Merck), sulfuric acid (H_2SO_4, 95–97%, Merck), sodium hydroxide (NaOH, ≥98%, Merck), sodium carbonate (Na_2CO_3, ≥99%, Merck), urea (≥99%, Merck), ethanol (≥99.5%, Merck), phosphoric acid, bleaching earth, chloroform, Wijs solution, potassium iodide, sodium thiosulfate, phenolphthalein indicator, bromothymol blue indicator, starch indicator, and deionized water. All chemicals were used as received without further purification.

Fourier Transform Infrared (FTIR) analysis was conducted using a Shimadzu FTIR-8400S spectrometer in the wavenumber range of 400–4000 cm^{-1}. X-ray diffraction (XRD) analysis was performed using a PANalytical X'Pert PRO diffractometer with Cu Kα radiation (λ = 1.5406 Å), operating over a 2θ range of 10°–90°.

2.2. Synthesis of CaO Catalyst from Chicken Eggshells

The initial step involved washing chicken eggshells using running water to remove dirt and separate the inner membrane. After that, the eggshells were weighed and dried in an oven at 100°C for two hours. The dried shells are then crushed into a fine powder. The final stage involves calcination using a muffle furnace at a temperature of 1000°C for 5 h.

2.3. CaO Modification by MgO

In this modification process, CaO derived from calcined chicken eggshells was used as the primary catalyst, while MgO acted as a modifier to improve catalytic stability and performance. CaO and MgO were mixed at a fixed molar ratio of 1:1 using a coprecipitation method. This approach was designed to enhance the physicochemical properties of CaO without altering its dominant catalytic role. Calcium oxide calcined from chicken eggshells and magnesium oxide with a molar ratio of 1:1 are dissolved in 100mL of deionized water. The solution is then stirred for 30 minutes. Six percent urea is then added to the solution. The mixture was added with 1M NaOH to achieve a pH of 9-10 in the solution and 1M Na_2CO_3 as a precipitating agent. The solution was stirred for 12 hours at a temperature of 60˚C. The mixture was filtered and washed using deionized water. Subsequently, drying is carried out at a temperature of 80˚C, followed by a calcination process at a temperature of 800˚C for 6 h.

2.4. Modification of Sulfonated CaO

The sulfonation stage of the CaO catalyst began with reacting CaO into a 3 M H_2SO_4 solution. The solution was stirred using a stirrer for 2 hours at room temperature. The solution was then filtered and washed using distilled water. Next, drying was carried out at a temperature of 200°C for 4 h.

2.5. Interesterification

This stage is carried out to synthesize palm oil/DBPO into methyl ester. The interesterification process is carried out with a molar ratio of oil and methyl acetate of 1:9. The palm oil is then heated to a temperature of 65°C in a three-neck flask equipped with a condenser. Next, it is mixed with methyl acetate and CaO catalyst with a variable of 2.5% of the total weight of oil and methyl acetate. Then, the heat of the mixture is maintained at a reaction temperature of 65˚C using a hot plate equipped with a magnetic stirrer. The mixture is stirred for 3 hours. After that, the mixture is then separated by centrifugation to obtain a layer of methyl ester and glycerin triacetate.

3. RESULT AND DISCUSSION

3.1. Synthesis of CaO Catalyst from Eggshells

In this study, the synthesis of calcium oxide (CaO) was successfully carried out. This was evidenced by the reduction in the mass of chicken eggshells after the calcination process by 47.59% of the initial mass. This result indicates the release of carbon dioxide from calcium carbonate in chicken eggshells. The chemical reaction equation for this process is as follows equation (1).

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

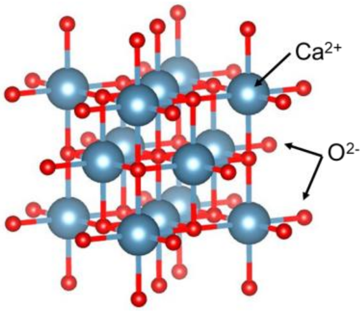

Figure 1. CaO crystal structure

CaO is an alkaline earth metal oxide with an ionic crystal structure, also known as a rock salt structure. The calcium cation is a very weak acid. Therefore, the conjugated oxygen anion exhibits strong basic properties. CaO also demonstrates good catalytic efficiency due to its high basic strength.

In this structure, Ca^{2+} and O^{2-} ions are arranged in a face-centered cubic (FCC) crystal system. As shown in **Figure 1,** calcium ions (Ca^{2+}) occupy lattice points at the corners and centers of each side of the cube, while oxide ions (O^{2-}) occupy interstitial positions that form high cubic symmetry. Each Ca^{2+} ion is octahedrally coordinated with six O^{2-} ions around it, and vice versa. The strength of this structure comes from the strong electrostatic attraction between oppositely charged ions, making CaO a thermally and mechanically very stable ionic solid.

3.2. Modification of CaO using MgO

The method used in this modification is coprecipitation, with the precursors used being CaO from the calcination of chicken eggshells and MgO. The crystal structure of the CaO·MgO mixture is a solid solution formed from two basic oxides, namely CaO and MgO. Both have the same crystal structure, namely FCC. In this structure, metal ions (Ca^{2+} , and Mg^{2+}) randomly occupy the cation lattice positions, while O^{2-} ions occupy the anion positions with preserved symmetry.

3.3. Sulfated CaO Modification

The results tend to have a more moist texture compared to pure CaO and CaO.MgO catalysts. This is because sulfonated CaO catalysts are more hygroscopic than pure CaO and CaO.MgO catalysts. The sulfonate group ($-SO_3H$) content in the catalyst increases polarity, making it more capable of attracting and retaining water molecules through hydrogen bonds. The crystal structure of anhydrous $CaSO_4$ is arranged in an orthorhombic crystal system, where calcium ions (Ca^{2+}) bond with sulfate ions (SO_4^{2-}) in a dense and regular formation. Each sulfate ion forms a tetrahedron with four oxygen ions, and calcium ions are located between these sulfate groups.

3.4. Characterization of CaO and CaO.MgO Catalysts

FTIR analysis was performed to compare the functional group spectra of three types of catalysts, namely CaO, CaO.MgO, and sulfonated CaO. Based on the FTIR test data, the samples can be identified their functional groups. FTIR characterization was carried out in the wavelength range of 500 nm to 4000 nm [8].

The FTIR spectra of CaO, CaO.MgO, and sulfonated CaO catalysts are presented in **Figure 2**. The absorption bands observed below 600 cm^{-1} correspond to metal–oxygen (M–O) stretching vibrations, specifically Ca–O and Mg–O bonds, which are characteristic of alkaline earth metal oxides. In the CaO.MgO catalyst, an increase in band intensity and broadening in this region was observed, indicating stronger metal–oxygen interactions and successful incorporation of MgO into the CaO lattice.

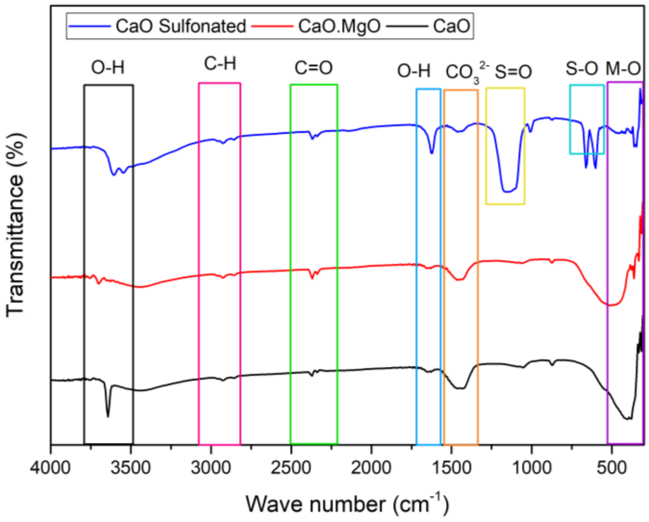

Figure 4. FTIR Spectrum of CaO, CaO.MgO, and Sulfonated CaO Catalysts

The presence of absorption peaks around 1010 cm^{-1}, 683 cm^{-1}, and 601 cm^{-1} in the sulfonated CaO catalyst confirms the presence of sulfate (SO_4^{2-}) groups, indicating successful sulfonation. Additionally, bands observed in the range of 1400–1550 cm^{-1} are attributed to carbonate (C=O) species formed due to CO_2 adsorption from the atmosphere, while the broad band around 3400 cm^{-1} corresponds to O–H stretching vibrations associated with surface hydroxyl groups or adsorbed moisture [9-11]. These spectral features collectively confirm the successful modification of CaO through MgO incorporation and sulfonation [12].

The results of X-ray diffraction characterization of the catalyst samples were used to identify the crystal structure and crystal lattice size of the samples. The test was conducted with X-rays in a wide angle range, specifically from 10° to 90° with a wavelength of 1.5406 Å. The diffractogram is shown in **Figure 3.**

The X-ray diffraction pattern of the CaO catalyst in **Figure 5** shows sharp and well-defined peaks at $2\theta \approx$ 18.07°; 28.80°; 34.29°; 47.14°; 50.96°, and 54.46°, which are consistent with the cubic crystal structure of CaO and correspond well with JCPDS card No. 37-1497 [13]. This confirms the successful formation of crystalline CaO from eggshell-derived calcium carbonate.

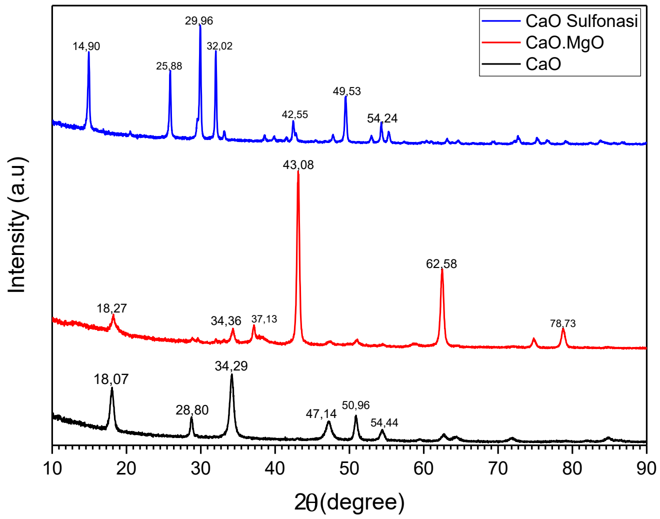

Figure 3. XRD diffractograms of CaO, CaO.MgO and sulfonated CaO under open vs closed system at different temperatures

Furthermore, the results of the CaO.MgO catalyst diffractogram show dominant peaks that still reflect the presence of CaO, but there are additional new peaks at 2θ ≈ 18.27°; 34.36°; 37.13°; 43.08° and 62.58°. The peaks at 43.08° and 62.58° are characteristic of MgO (JCPDS No. 45-0946) [14], indicating that the modification process successfully added a new phase to the CaO structure. No new crystalline compound was detected; however, the coexistence of CaO and MgO phases confirms successful modification rather than simple physical mixing. This resulted in peak shifts or the addition of a new phase that is thermodynamically stable. This modification has the potential to enhance the basic properties and thermal stability of the catalyst. In the sulfonated CaO sample, this peak is no longer as sharp as in the previous sample. This indicates that the crystal structure has changed significantly. This is an indication that modification with sulfonate groups causes a decrease in crystallinity and the possible formation of a semi-crystalline or amorphous phase.

The smallest crystal size of CaO compared to CaO.MgO and sulfonated CaO is 12.96 nm. A smaller size means a larger surface area, providing more active sites, but small crystals tend to sinter (agglomerate) more easily when heated. CaO is generally a strong base, good for basic reactions such as transesterification, but it is susceptible to moisture and carbonation. CaO.MgO has a larger average crystal size than CaO, at 22.53 nm. Larger crystals tend to have a smaller specific surface area, but they have better thermal stability and crystal structure. This can increase resistance to sintering and is suitable for high-temperature reactions. The combination of CaO and MgO also provides a synergistic effect that can increase the base activity of the catalyst. Meanwhile, sulfonated CaO has the largest crystal size of the two catalysts, at 38.34 nm, but the data also shows peaks with very large FWHM, indicating a local amorphous structure due to sulfonation. Sulfonation enhances acidic properties, making the catalyst amphiprotic (possessing both acidic and basic sites), which is highly beneficial for complex reactions such as simultaneous transesterification and esterification. However, if crystallinity is overly disrupted, it can reduce the catalyst's stability and durability.

3.5. Biodiesel Characterization

The fatty acid methyl esters (FAME) produced in this study correspond to biodiesel, as they consist predominantly of methyl esters derived from triglycerides in accordance with international biodiesel definitions. Biodiesel is synthesized using the transesterification method, which is a non-alcoholic biodiesel synthesis that produces methyl ester and triacetin products. Transesterification is a chemical process in which ester groups in two or more different molecules exchange places, producing a new ester mixture [3].

Based on the chemical reaction shown in **Figure 4**, it is known that the interesterification reaction of triglycerides with methyl acetate occurs through a nucleophilic attack mechanism initiated by the heterogeneous base catalyst CaO. The oxide ion produced from catalyst activation causes the formation of a reactive methoxide anion, which then attacks the carbonyl group in the triglyceride molecule. This process produces an unstable tetrahedral intermediate, which further decomposes to form methyl ester and reduces the glyceride level from triglyceride to diglyceride.

The reaction proceeds gradually until monoglycerides and finally glycerol are formed, with each stage producing one molecule of methyl ester. The catalyst regeneration cycle allows CaO to remain active throughout the reaction without undergoing permanent structural changes. This mechanism proves that the use of heterogeneous base catalysts, particularly modified CaO, can provide high efficiency in biodiesel synthesis and facilitate product separation due to the catalyst's insolubility in the reaction phase.

Several indicators of successful biodiesel synthesis are percentage yield, flame test, density, viscosity, acid number, and iodine number. The values of each indicator are shown in **Table 1**.

The physicochemical properties of the produced FAME were compared with biodiesel quality standards, namely SNI 7182:2015 and ASTM D6751. The viscosity values of biodiesel produced using CaO (2.46 cSt), CaO.MgO (2.42 cSt), and sulfonated CaO (2.32 cSt) catalysts fall within the acceptable range specified by SNI (2.3–6.0 cSt), indicating good fuel flow characteristics [15].

The density values obtained (924–962 kg/m^3) are consistent with the typical density range for biodiesel fuels and are comparable with values reported in SNI and ASTM standards. The acid number of biodiesel

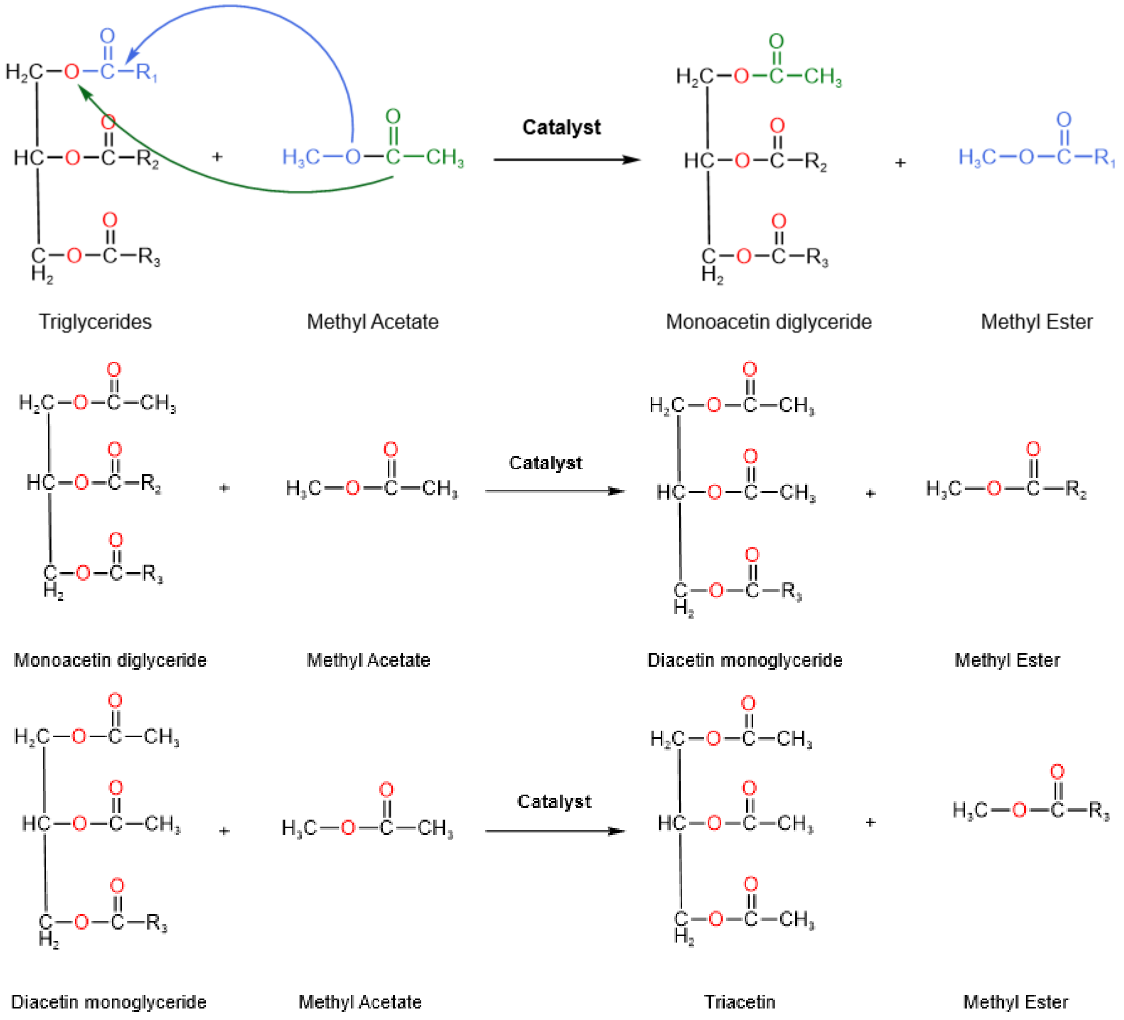

Figure 6. Chemical reaction equation for interesterification

Table 1. Characterization of biodiesel using CaO, CaO.MgO, and sulfonated CaO catalysts

Indicators	Catalyst			Without Catalyst	Unit
	CaO	CaO. MgO	CaO Sulfo-nation		
Yield	78,71	89,63	83,02	81,59	%
Flame Test	33.00	55.00	45.00	0.00	s
Density	924	958	962	971	Kg/m^3
Viscosity	2,46	2,42	2,32	1,50	cSt
Acid Number	0,84	2,24	2,86	2,84	mg/KOH/g
Iodine Number	15,23	9,64	27,16	48,71	mass %

produced using the CaO.MgO catalyst (2.24 mg KOH/g) is lower than that of sulfonated CaO and remains within the acceptable limit, indicating reduced risk of corrosion during storage and engine operation.

Furthermore, the iodine value obtained for the CaO.MgO catalyst (9.64 g I_2/100 g) is well below the maximum limit specified by SNI (<115 g I_2/100 g), indicating good oxidative stability. These results confirm that the biodiesel produced using the CaO.MgO catalyst meets the essential quality requirements of SNI 7182:2015 and ASTM D6751 standards.

4. CONCLUSION

Calcium oxide was obtained from the calcination of chicken eggshell waste, which is rich in calcium carbonate. The extracted calcium oxide was then combined with magnesium oxide and sulfuric acid through a coprecipitation method, producing a heterogeneous catalyst with good thermal stability. This catalyst was characterized using FTIR, showing absorption peaks consistent with the calcium oxide database. Both combinations using magnesium oxide and sulfuric acid were further characterized by XRD to determine the crystallinity of each type of catalyst. The results showed that magnesium oxide modification caused a peak shift or the addition of a new phase that was thermodynamically stable. Meanwhile, modification using sulfate resulted in a decrease in intensity and peak broadening, causing a decrease in crystallinity and the possible formation of a semi-crystalline or amorphous phase.

The biodiesel properties obtained using CaO, CaO.MgO, and sulfonated CaO catalysts were compared with SNI 7182:2015 and ASTM D6751 standards. The results indicate that biodiesel produced using the CaO.MgO catalyst meets the required specifications in terms of viscosity, density, acid number, and iodine value, confirming its suitability as biodiesel fuel. The higher biodiesel yield obtained using the CaO.MgO catalyst (89.63%) can be attributed to the synergistic interaction between CaO and MgO. MgO enhances the thermal stability of CaO, reduces catalyst deactivation due to moisture and carbonation, and provides additional basic active sites, leading to improved catalytic efficiency and higher conversion rates.

SUPPROTING INFORMATION

There is no supporting information in this paper. Data supporting the findings of this study are available upon request from the authors (DAS).

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CONFLICT OF INTEREST

There was no conflict of interest in this study.

AUTHOR CONTRIBUTIONS

DAS, MF, RB, and G performed the conceptualization, manager of the characterization, review and editing. FH, AKA, AKHS, AKN, HSN, and ZAF conducted the experiment, calculations, and wrote the manuscript. All authors agreed to the final version of this manuscript.

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