

# Synthesis and Characterization of Sucrose-Modified CaO Catalyst Derived from Dolomite for Transesterification of *Reutealis trisperma* Oil

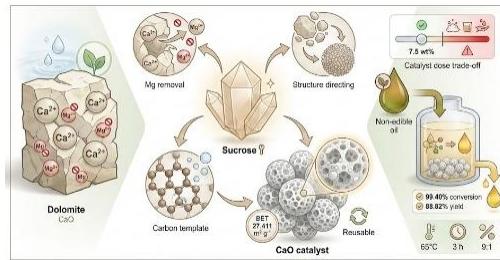
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## ABSTRACT

This study presents a novel method for synthesizing solid base catalysts by modifying calcium oxide (CaO) from dolomite via a sucrose-mediated hydrothermal process. In this approach, sucrose acts as a complexing agent to remove magnesium ions ( $Mg^{2+}$ ), a structure-directing agent, and a carbon-based template. After  $Mg^{2+}$  removal, calcium species were recovered through coprecipitation using sodium carbonate. The synthesized catalysts were characterized to evaluate their structure using X-ray diffraction (XRD), identify functional groups via Fourier-transform infrared spectroscopy (FTIR), observe morphology and elemental composition through scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX), measure particle size distribution by particle size analysis (PSA), and determine surface area through Brunauer-Emmett-Teller (BET) analysis. The catalysts exhibited a surface area of  $27.411 \text{ m}^2/\text{g}$  and reduced crystallite size, both contributing to enhanced catalytic activity. In the transesterification of *Reutealis trisperma* oil under optimal conditions ( $65^\circ\text{C}$ , 3 hours, methanol-to-oil ratio 9:1), the catalyst achieved 99.40% oil conversion and 88.82% biodiesel yield. A catalyst dosage of 7.5 wt% was optimal, while higher amounts caused emulsion and soap formation due to viscosity-related mass transfer limitations. This environmentally friendly synthesis route offers a reusable catalyst system for sustainable biodiesel production from non-edible feedstocks.



**Keywords:** Solid base catalyst; Biodiesel; Calcium oxide; Sucrose, Hydrothermal; Transesterification.

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## INTRODUCTION

Rising global energy demand, driven by industrial development and population growth, is still largely met by nonrenewable sources. The International Energy Agency reports that more than 80% of the world's energy supply continues to come from fossil

fuels, including coal, petroleum, and natural gas [1]. This heavy dependence contributes substantially to environmental degradation, particularly through greenhouse gas emissions that accelerate global warming, drive sea level rise, and disrupt ecosystems [2]. Therefore, the development of environmentally friendly and sustainable

alternative energy sources is urgently needed. Among various alternatives, biodiesel has gained considerable attention due to its biodegradability, lower emission levels, and direct usability in conventional diesel engines without requiring modifications [3].

Despite these advantages, biodiesel production still faces economic challenges, particularly when using edible oils such as soybean or palm oil, which may lead to food-versus-fuel competition and increased production costs [4]. To address this issue, non-edible vegetable oils have been explored as more sustainable alternatives, especially those that can grow on marginal land. One promising feedstock is *Reutealis trisperma* oil, which contains over 55% triglycerides and is abundantly available in Indonesia [5]. However, this oil typically contains high levels of free fatty acids (FFA), often above 5%, which can cause soap formation during transesterification with base catalysts, reducing biodiesel yield [6]. Although acid pretreatment can reduce FFA levels, it adds extra steps and operational costs. Thus, the development of a robust, reusable base catalyst that can tolerate high FFA levels is crucial for simplifying and enhancing biodiesel production from such feedstocks [7].

Calcium oxide (CaO) is one of the most studied solid base catalysts due to its high basic strength, thermal stability, low cost, and ease of recovery and reuse [8]. It can be synthesized from various calcium-rich natural sources such as limestone, eggshells, and dolomite. Among these, dolomite is particularly attractive due to its high content

of both calcium and magnesium carbonates. However, calcination of dolomite produces a mixture of CaO and magnesium oxide (MgO), where the presence of amphoteric MgO may interfere with the active basic sites of the catalyst, thereby reducing its catalytic performance [9], [10].

A conventional method for removing MgO is acid leaching using hydrochloric acid (HCl). While this method is effective, it generates hazardous chemical waste, violating green chemistry principles [11]. As a greener alternative, sucrose has been investigated as a complexing agent capable of selectively binding Mg<sup>2+</sup> ions into soluble complexes, while Ca<sup>2+</sup> ions remain in solution and can precipitate as calcium carbonate [12]. Furthermore, sucrose can serve as a structure-directing agent and soft carbon template in hydrothermal treatment, facilitating the formation of uniform, porous catalyst morphologies [13].

Hydrothermal treatment is known to improve catalyst surface area, morphology, and reduce particle agglomeration, all of which are critical for enhancing catalytic activity in heterogeneous systems [14]. However, most previous studies focus solely on either Mg<sup>2+</sup> removal or morphological improvement, without integrating both aspects in a single synthesis strategy. For instance, Anjani and Suherman [15] utilized untreated dolomite and achieved biodiesel yields below 80%, while Sinaga et al. [16] employed oxalic acid for purification but still reported residual MgO and poor environmental compatibility. Therefore, there remains a clear research gap in developing an integrated, eco-friendly method for

synthesizing high quality CaO catalysts from dolomite.

This study aims to develop a green, integrated synthesis method for CaO catalysts by combining sucrose-assisted  $Mg^{2+}$  separation with hydrothermal treatment. This approach is expected to improve both the purity and morphological properties of CaO without the use of harmful chemicals. The resulting catalysts were characterized to evaluate their crystallinity, surface morphology, and particle size, and their catalytic activity was assessed through the transesterification of *Reutealis trisperma* oil. This research is expected to contribute to the development of sustainable and efficient solid base catalysts for biodiesel production from non-edible oils.

## METHOD

### 1. Materials and Instruments

The materials used in this study included dolomite limestone sourced from Bukit Jaddih, Madura, sucrose ( $C_{12}H_{22}O_{11}$ , Merck), sodium carbonate ( $Na_2CO_3$ , Merck,  $\geq 99\%$ ), methanol ( $CH_3OH$ , Merck,  $\geq 99.8\%$ ), distilled water, and *Reutealis trisperma* (kemiri sunan) oil.

The instruments and equipment included a muffle furnace (Nabertherm LHT 02/17 LB, Germany), hydrothermal autoclave (Berlab, 100 mL), magnetic stirrer with hot plate (IKA C-MAG HS 7, Germany), drying oven (Memmert UN 55, Germany), X-ray diffraction (XRD, PANalytical X'Pert PRO, Netherlands), Fourier transform infrared spectroscopy (FTIR, Shimadzu IRSpirit, Japan), scanning electron microscope

equipped with energy-dispersive X-ray spectroscopy (SEM-EDX, JEOL JSM-6510LA, Japan), particle size analyzer (PSA, Horiba SZ-100, Japan), and gas chromatography with flame ionization detector (GC-FID, Shimadzu GC-2010 Plus, Japan).

## 2. Experimental Procedures

### a. Calcination of Dolomite

Dolomite powder (100 mesh) was calcined at 900 °C for 3 hours to decompose the material into a CaO/MgO mixture [9].

### b. Magnesium Ion Removal

10 grams of calcined powder were mixed with 100 mL of 30% (w/v) sucrose solution at 70 °C under constant stirring for 2 hours. Sucrose selectively complexed  $Mg^{2+}$  ions, allowing separation of calcium as insoluble carbonate [12].

### c. Filtration and Co-precipitation

The mixture was filtered, and the calcium-rich filtrate was titrated with 1 M  $Na_2CO_3$  until pH 12, followed by stirring for 3 hours to co-precipitate calcium as  $CaCO_3$ -sucrate [11].

### d. Hydrothermal Treatment

The resulting precipitate was placed in a Teflon-lined stainless steel autoclave and heated at 100 °C for 24 hours to improve crystallinity and morphology [13].

### e. Drying and Final Calcination

The product was dried at 100 °C and calcined at 800 °C for 3 hours to remove

residual carbonates and organic content, resulting in high-purity CaO catalyst [10].

### 3. Catalyst Characterization

#### a. X-ray Diffraction (XRD)

XRD was performed using Cu-K $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ) across  $2\theta = 10^\circ - 80^\circ$  at  $2^\circ/\text{min}$  to identify crystalline phases and estimate crystallite size [10].

#### b. Fourier Transform Infrared Spectroscopy

Samples were mixed with KBr and pressed into pellets; spectra were recorded from  $400-4000 \text{ cm}^{-1}$  to identify carbonate, hydroxide, and Ca-O bonds [17].

#### c. Scanning Electron Microscopy-EDX

Samples were gold-coated and observed under  $1000-10,000\times$  magnification. Elemental mapping was performed to observe Ca and Mg distribution [6].

#### d. Particle Size Analysis (PSA)

Samples dispersed in ethanol were sonicated and analyzed using laser diffraction to determine particle size distribution [6].

#### e. Brunauer-Emmett-Teller (BET)

Degassed samples were analyzed at 77 K via nitrogen adsorption to determine surface area and porosity [13].

### 4. Catalytic Activity Testing

#### a. Feedstock and Reagent Preparation

*Reutealis trisperma* oil (100 mL) was mixed with methanol and CaO catalyst at varied dosages (2.5%, 5%, 7.5%, and 10% w/w of oil). A 9:1 methanol-to-oil molar ratio was used [6].

#### b. Transesterification Reaction

The reaction was conducted at  $65^\circ\text{C}$  for 3 hours in a reflux setup with magnetic stirring at 600 rpm to ensure homogeneity [6].

#### c. Catalyst Separation

After reaction, the catalyst was filtered with Whatman No. 42 paper, dried, and stored for reuse [10].

#### d. Product Purification

The mixture was transferred to a separating funnel and allowed to settle. The upper biodiesel phase was washed with warm distilled water to neutral pH and dried at  $105^\circ\text{C}$  for 1 hour [18].

#### e. Biodiesel Composition and Yield Analysis

The final biodiesel was analyzed using GC-FID to determine the FAME profile [6]. All experiments were performed in triplicate to ensure accuracy and reproducibility.

## RESULT AND DISCUSSION

### 1. Catalyst Preparation

Calcium oxide (CaO) catalysts were synthesized from dolomite through a multi step route designed to obtain high chemical purity, favorable morphology, and improved catalytic performance. The process combined thermal decomposition, selective magnesium removal through sucrose complexation, coprecipitation, hydrothermal crystallization, and final calcination. This strategy is consistent with sustainable chemistry principles and provides a more environmentally friendly alternative to

conventional acid leaching or simple direct calcination.

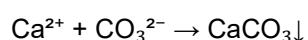
The process began with the calcination of dolomite powder (100 mesh) at 900 °C for 3 hours. The thermal decomposition of dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ) resulted in a mixture of  $\text{CaO}$  and  $\text{MgO}$ , releasing carbon dioxide as described by the reaction:



The resulting grayish powder indicated the presence of both oxides. However,  $\text{MgO}$ , being amphoteric, is considered unfavorable in basic catalysis due to its tendency to neutralize or reduce the overall basicity of the catalyst surface [9]. Therefore, magnesium needed to be selectively removed to obtain a purer  $\text{CaO}$  phase.

To achieve this, the calcined material was treated with a 30% (w/v) sucrose solution at 70 °C under continuous stirring for 2 hours. Sucrose acts as a mild chelating agent capable of forming water-soluble complexes with  $\text{Mg}^{2+}$  ions, while  $\text{Ca}^{2+}$  remains largely unbound under these conditions. This selective complexation effectively separated magnesium from the mixture, leaving behind a calcium-enriched solution [12].

The filtrate was subsequently titrated with 1 M  $\text{Na}_2\text{CO}_3$  solution until the pH reached 12. At this alkaline condition,  $\text{Ca}^{2+}$  ions reacted with  $\text{CO}_3^{2-}$  to form calcium carbonate precipitate, following the reaction:



This white precipitate may also contain residual organic content from sucrose, forming  $\text{CaCO}_3$ -sucrate complexes. These intermediates are known to facilitate

the formation of dispersed and uniform  $\text{CaO}$  particles upon further thermal treatment [11].

The precipitate was subjected to hydrothermal treatment at 100 °C for 24 hours in a Teflon-lined stainless-steel autoclave. This step enhanced the structural integrity and crystallinity of the precursor by enabling controlled nucleation and particle growth under pressure [13]. Hydrothermal processing also improved particle dispersion and reduced agglomeration.

The hydrothermally treated solid was then dried at 100 °C and calcined at 800 °C for 3 hours. This final thermal activation decomposed the carbonate into catalytically active  $\text{CaO}$  while removing residual organic material, according to the reaction:



The final product was a fine white powder, indicative of high-purity  $\text{CaO}$  with improved physicochemical properties. This method offers significant advantages compared to conventional approaches. The sucrose-assisted pathway avoids the use of hazardous mineral acids, reduces corrosive waste, and minimizes environmental impact. Moreover, the combination of selective magnesium removal and hydrothermal processing results in a catalyst with superior surface characteristics, enhanced basicity, and higher activity for transesterification reactions [6].

## 2. Characterization of $\text{CaO-S}$

### a. X-ray Diffraction

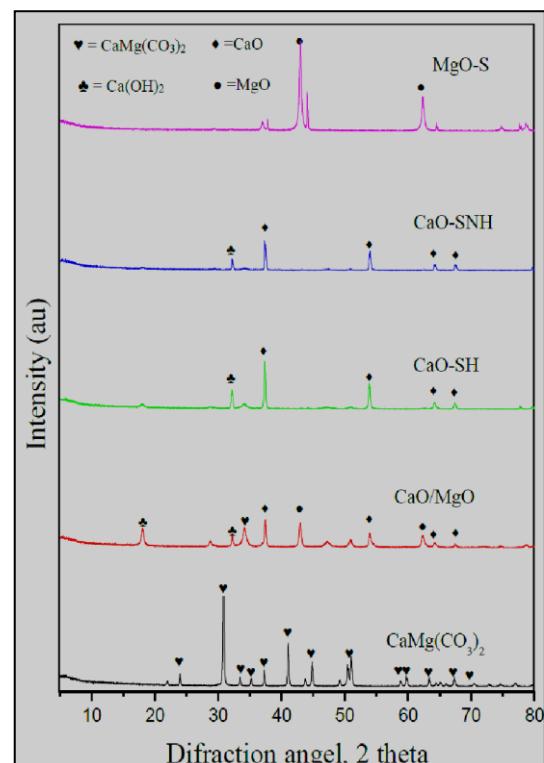
X-ray diffraction (XRD) analysis was performed to investigate the crystalline structure and phase composition of the

synthesized CaO catalyst. As shown in [Figure 1](#), the diffraction pattern displayed sharp and intense peaks at  $2\theta$  values of  $32.2^\circ$ ,  $37.3^\circ$ ,  $53.8^\circ$ , and  $64.1^\circ$ , which correspond to the (111), (200), (220), and (311) planes of calcium oxide (CaO) based on JCPDS No. 37-1497. These peaks clearly indicate that CaO is the dominant crystalline phase in the final catalyst.

The absence of diffraction signals associated with magnesium oxide, calcium hydroxide, or carbonate residues confirms the effectiveness of the sucrose-assisted separation process in removing magnesium [\[12\]](#). Moreover, the absence of calcium hydroxide peaks suggests that the catalyst maintained its structural stability during storage and resisted significant rehydration, despite the inherently hygroscopic nature of CaO [\[17\]](#). Structural stability is essential for maintaining catalytic performance, as moisture and carbon dioxide absorption can deactivate active sites.

The well-defined and narrow peaks also reflect high crystallinity, implying an ordered lattice structure and low defect density. This structural quality is desirable in heterogeneous catalysis since it supports better thermal stability and higher basic strength. According to previous research by Palitsakun and colleagues [\[10\]](#), highly crystalline CaO catalysts tend to exhibit superior activity in biodiesel synthesis due to improved active site accessibility. Similarly, Kurniawan et al. [\[19\]](#) found that applying hydrothermal treatment after magnesium removal enhances the phase purity and crystallinity of CaO derived from dolomite.

These results demonstrate that the combination of calcination, sucrose treatment, and hydrothermal processing successfully yielded a single-phase CaO catalyst with excellent crystallinity and structural purity.



**Figure 1.** XRD Diffraction Patterns of  $\text{CaMg}(\text{CO}_3)_2$ ,  $\text{CaO}/\text{MgO}$ ,  $\text{CaO-SH}$ ,  $\text{CaO-SNH}$ , and  $\text{MgO-S}$

#### b. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectroscopy was carried out to identify surface functional groups and confirm the presence or absence of carbonate and hydroxide residues in the CaO catalyst. The resulting spectrum is presented in [Figure 2](#). A broad absorption band centered at  $3640 \text{ cm}^{-1}$  was observed, which corresponds to O-H stretching vibrations. This signal is typically associated with surface hydroxyl groups

formed due to mild exposure of CaO to atmospheric moisture [17]. This band originates from physically adsorbed water or a very thin hydroxyl layer on the surface [20]

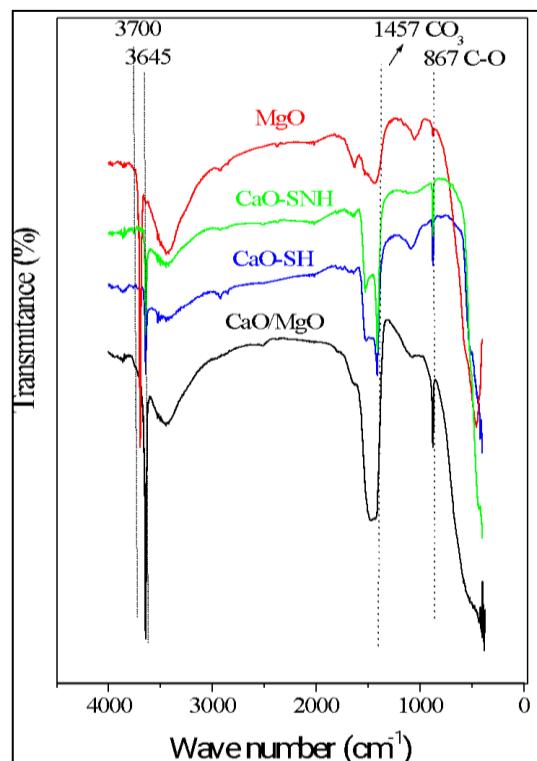
In addition, weak bands at  $1420\text{ cm}^{-1}$  and  $875\text{ cm}^{-1}$  were observed, which are characteristic of carbonate ions through asymmetric stretching and out-of-plane bending modes [21]. The low intensity of these peaks suggests that only a small amount of carbonate remained after calcination, potentially caused by limited reabsorption of carbon dioxide during air exposure [21]. This observation supports the success of the calcination process in decomposing carbonate species.

A prominent absorption band also appeared between  $500$  and  $600\text{ cm}^{-1}$ , corresponding to calcium-oxygen (Ca-O) stretching vibrations [17]. This confirms the presence of CaO as the dominant oxide species in the sample. The FTIR data are in good agreement with the XRD results, providing further evidence of the structural integrity of the catalyst.

Comparable findings were reported by Bedir and Dogan [20], who demonstrated that successful CaO synthesis should result in dominant Ca-O peaks with minimal carbonate or hydroxyl contamination. Sari et al. [21] also emphasized that the presence of residual carbonate must be limited to preserve high basic strength and prevent inhibition of active catalytic sites.

Overall, the FTIR analysis confirms that the synthesized catalyst is composed primarily of CaO with only minor surface hydration and minimal carbonate residues,

making it suitable for transesterification applications.

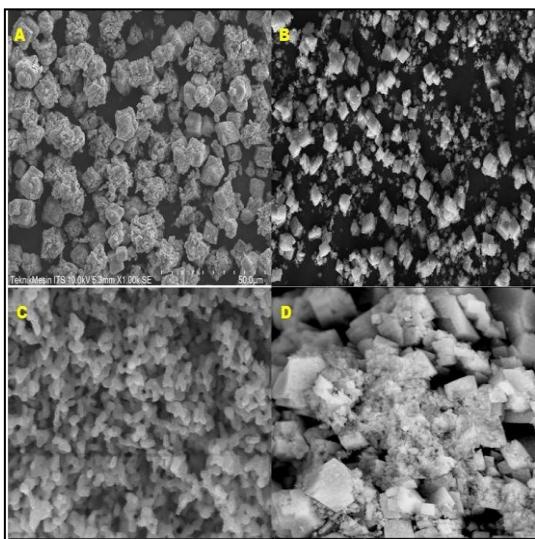


**Figure 2.** FTIR Spectra of MgO, CaO-SNH, CaO-SH, and CaO/MgO

### c. Scanning Electron Microscopy - Energy Dispersive X-ray (SEM-EDX)

The surface morphology of the synthesized CaO catalyst was examined using scanning electron microscopy (SEM), and the results are shown in Figure 3. The micrographs revealed that the catalyst particles had irregular shapes with rough surfaces and visible agglomeration. This morphology is commonly observed in materials subjected to thermal decomposition and hydrothermal post-treatment [14]. The particle sizes varied within the micrometer scale, and the presence of rough textures and voids suggested the development of a porous structure [25].

Agglomeration among particles was attributed to electrostatic interactions and partial sintering during calcination. While excessive agglomeration may reduce overall surface area, the formation of meso- and macropores between fused particles can actually benefit catalytic reactions by facilitating mass transfer and providing space for the diffusion of larger reactant molecules such as triglycerides [25]. The existence of these pores aligns with previous studies, which have reported that porosity contributes significantly to the catalytic efficiency of CaO-based catalysts [22].



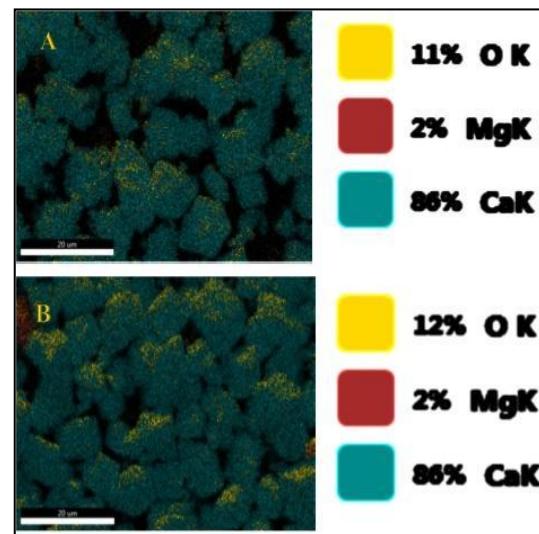
**Figure 3.** Morphology of CaO-SNH (A), CaO-SHD (B), CaO-S (C), and CaO/MgO (D)

Elemental composition analysis was conducted using energy-dispersive X-ray spectroscopy (EDX), as presented in Figure 4. The EDX spectrum showed strong peaks for calcium (Ca) and oxygen (O), confirming CaO as the dominant component of the catalyst. Notably, no magnesium (Mg) peaks were detected, indicating that the sucrose-assisted separation process effectively removed  $Mg^{2+}$  ions from the precursor. This

aligns with the XRD results, which showed no crystalline phases related to MgO.

The absence of Mg is particularly important, as MgO has amphoteric behavior that can reduce the basic strength of the catalyst and introduce unwanted side reactions [16]. The high Ca content, combined with the porous structure observed in SEM, suggests the catalyst possesses a high density of accessible basic sites. These characteristics are favorable for enhancing catalytic activity in the transesterification of *Reutealis trisperma* oil.

Overall, the SEM-EDX analysis validates the success of the synthesis approach in producing a Mg-free, highly basic, and structurally porous CaO catalyst suitable for heterogeneous biodiesel production.



**Figure 4.** EDX Spectrum and Elemental Composition of CaO-S

#### d. Particle Size Analysis (PSA)

Particle size distribution of the synthesized CaO catalyst was analyzed using a laser diffraction particle size analyzer. The results, illustrated in Figure 5, indicate a

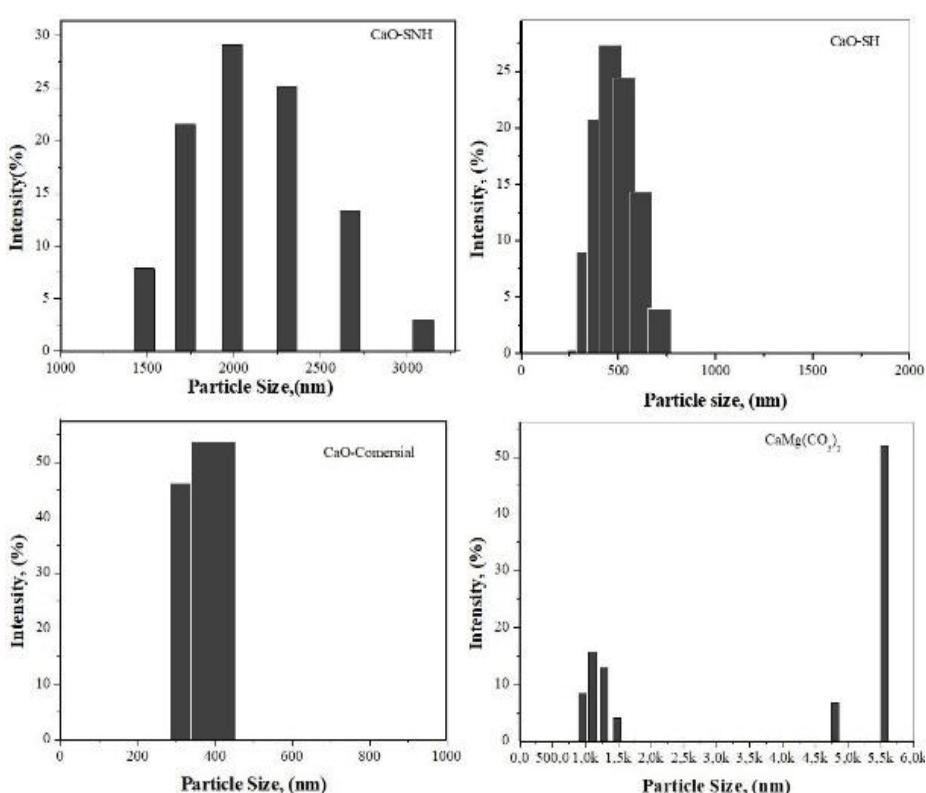
relatively broad distribution with a dominant peak centered around 0.8  $\mu\text{m}$ . This submicron particle size suggests that the synthesis method particularly the coprecipitation followed by hydrothermal treatment was effective in producing finely dispersed catalyst particles [19].

The small particle size is a favorable feature for heterogeneous catalysis, as it typically correlates with higher surface area and more accessible active sites. Although some degree of particle agglomeration was observed in SEM analysis, the PSA data confirms that a significant portion of the material remained in the desired fine range, which is critical for maximizing catalytic contact with reactants [24].

Furthermore, the narrow and consistent particle size distribution implies

good control over the nucleation and growth phases during synthesis [19]. This uniformity in particle dimension not only improves surface area but also contributes to better dispersion in the reaction medium, minimizing mass transfer limitations during the transesterification of *Reutealis trisperma* oil.

Similar studies have shown that CaO catalysts with submicron particle sizes, particularly in the range of 200-1000 nm, offer superior catalytic performance due to enhanced surface exposure and reduced diffusion resistance [23], [24]. The particle size of  $\sim 0.8 \mu\text{m}$  observed in this study falls within this optimal range, aligning with previous findings that link finer dispersion to higher biodiesel yields.



**Figure 5.** Particle Size Distribution of CaO-SNH, CaO-SH, CaO-S, and CaO/MgO

### e. Brunauer-Emmett-Teller (BET) Surface Area Analysis

The surface area, pore volume, and average pore diameter of the synthesized CaO catalysts were evaluated using the BET

method, with the results presented in [Table 1](#). These data clearly demonstrate that the applied synthesis methods had a significant impact on the textural properties of the catalysts.

**Table 1.** BET surface area of CaO catalyst synthesized from dolomite.

Sampel	Method	Surface Area (m <sup>2</sup> /g)	Crystal Size (nm)	Basicity (mmol/g)	CaO (%)	MgO (%)
CaO/MgO	Calcination	9.708	137.45	0.332	44.71	55.29
CaO-SH	Co-P + S + H	27.411	61.9	0.273	86.6	13.4
CaO-SNH	Co-P + S	22.165	112	0.268	91.9	8.1
CaO	Co-P	9.810	30.57	0.175	100	-

Among the samples, CaO-SNH exhibited the highest specific surface area at 11.673 m<sup>2</sup>/g, followed by CaO-SH (10.122 m<sup>2</sup>/g) and CaO-S (9.842 m<sup>2</sup>/g). In contrast, CaO/MgO, which was obtained through direct calcination, showed the lowest surface area at 6.218 m<sup>2</sup>/g. The superior surface area of CaO-SNH is attributed to the combined effect of sucrose-assisted magnesium removal and hydrothermal treatment, which suppressed particle agglomeration and enhanced porosity. A higher surface area increases the availability of basic active sites, thereby improving catalytic performance in transesterification reactions, as also noted in previous studies on CaO-based catalysts [\[25\]](#).

A similar trend was observed in the pore volume measurements. CaO-SNH recorded the highest pore volume at 0.074 cm<sup>3</sup>/g, indicating a well-developed porous structure. In contrast, CaO/MgO displayed the lowest value (0.038 cm<sup>3</sup>/g), likely due to the absence of post-synthesis modification. CaO-S and CaO-SH showed intermediate values. A greater pore volume is

advantageous in catalysis, as it facilitates the diffusion of large reactant molecules, such as triglycerides, through the catalyst matrix.

Regarding average pore diameter, all catalysts exhibited mesoporous characteristics. CaO-SNH had the largest pore diameter (26.13 nm), followed by CaO-SH (23.42 nm), CaO-S (20.81 nm), and CaO/MgO (18.25 nm). Mesopores are ideal for heterogeneous catalytic processes, offering a balance between surface area and effective diffusion pathways. The larger pore diameter of CaO-SNH suggests a more open structure, likely resulting from improved control over crystal growth during hydrothermal treatment. Overall, the BET analysis confirms that the incorporation of sucrose-assisted magnesium removal and hydrothermal processing significantly enhances the surface characteristics of the catalyst. The CaO-SNH sample, in particular, displayed the most favorable properties high surface area, substantial pore volume, and mesoporous structure which are all essential for facilitating efficient mass transfer and maximizing active site exposure. These

attributes are closely linked to the improved catalytic performance observed in the transesterification of *Reutealis trisperma* oil. **Catalytic Activity**

#### a. Effect of Catalyst Type on Transesterification

The catalytic activity of CaO based catalysts including CaO/MgO, CaO-S, CaO-SH, and CaO-SNH was tested through transesterification of *Reutealis trisperma* oil under identical reaction conditions. The results presented in [Table 2](#) show that the catalyst synthesized using the complete sucrose assisted method with hydrothermal treatment and co-precipitation (CaO-SNH) produced the highest biodiesel yield of 87.43%. In contrast, the lowest yield was obtained from CaO/MgO, with a value of 45.32%.

The low yield of CaO/MgO was due to the presence of magnesium, which remained because no separation process was applied. MgO has amphoteric properties that reduce catalytic activity, and the presence of unreacted carbonate also hinders the active basic sites of CaO [\[8\]](#), [\[14\]](#). In the CaO-S sample, sucrose-assisted

separation removed magnesium, but without hydrothermal or precipitation treatment, the catalyst had poor surface characteristics, leading to a moderate biodiesel yield of 61.48% [\[5\]](#).

The CaO-SH catalyst involved hydrothermal treatment after sucrose separation, which improved surface structure, particle uniformity, and crystallinity. As a result, this catalyst gave a higher biodiesel yield (69.57%) compared to CaO-S [\[9\]](#). The CaO-SNH catalyst, synthesized through a complete process of magnesium removal, co-precipitation, hydrothermal treatment, and final calcination, showed optimal performance due to its better surface morphology, purity, and crystal structure, which facilitated higher basicity and catalytic activity [\[18\]](#). These results demonstrate that magnesium removal, followed by controlled precipitation and hydrothermal treatment, plays a critical role in producing an efficient catalyst. The multi-step synthesis route in CaO-SNH yielded a highly active, structurally optimized catalyst, resulting in the highest conversion of triglycerides to biodiesel [\[3\]](#), [\[10\]](#).

**Table 2.** Catalytic Activity of CaO-S in the Transesterification of *Reutealis trisperma* Oil

Catalyst	Conversion of oil (%)	Methyl ester selectivity (%)	FFA selectivity (%)	Methyl ester yield (%)	FFA yield (%)
CaO/MgO	29.66	89.73	10.27	26.62	3.04
CaO-SNH	94.67	91.58	8.42	86.70	7.97
CaO-SH	99.40	91.87	8.13	88.82	8.08

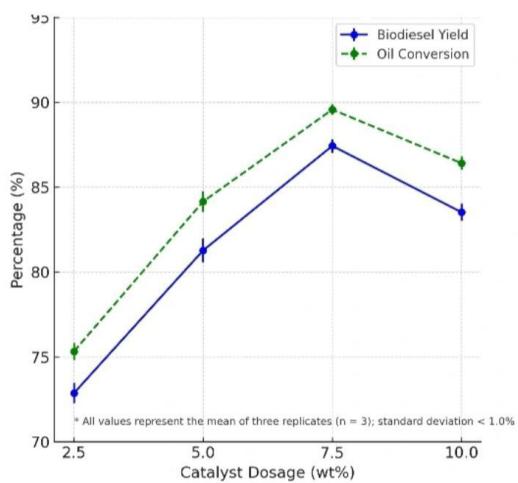
Data are mean  $\pm$  SD from triplicate experiments (n = 3, SD < 1.0%)

#### b. Effect of Catalyst Loading on Transesterification

Optimal catalyst loading was evaluated by conducting transesterification

reactions with the CaO-SH catalyst at dosages of 2.5%, 5%, 7.5%, and 10% (w/w relative to oil). [Figure 6](#) shows that increasing the catalyst dosage from 2.5% to 7.5%

increased biodiesel yield from 72.86% to 87.43%. A further increase to 10% led to a slight decrease in yield to 83.52%, indicating that 7.5% is the most effective dosage under the tested conditions.



### **Figure 6. Effect of CaO-SH Catalyst**

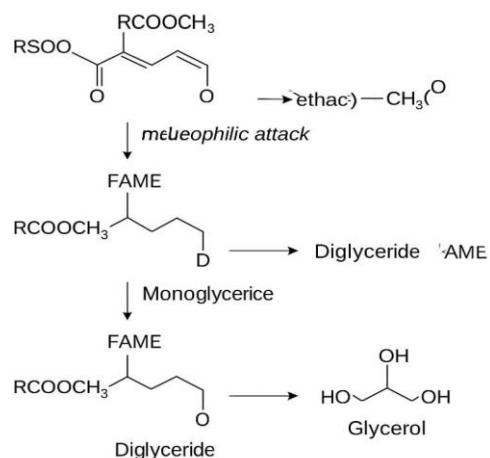
This behavior can be explained by the increased number of active basic sites at higher catalyst concentrations, which accelerates the reaction and enhances triglyceride conversion [13]. However, an excessive catalyst amount increases the viscosity of the reaction mixture, reduces mass transfer efficiency, and may promote catalyst agglomeration, which in turn reduces the availability of active sites [19].

The results indicate that 7.5% w/w is the optimal dosage for CaO-SH, providing the best balance between catalytic activity and reaction efficiency. At this dosage, sufficient active sites are present without significantly increasing system viscosity or blocking surface area through agglomeration [16].

These findings align with literature that suggest there is an optimum catalyst amount beyond which further increases lead to declining efficiency. In this study, the enhanced catalytic properties of CaO-SH,

combined with its fine particle size and mesoporous structure, enabled effective reactant catalyst interaction and high conversion efficiency at the optimal dosage [20].

The transesterification reaction mechanism using a solid base catalyst such as CaO involves the generation of methoxide ions ( $\text{CH}_3\text{O}^-$ ), as illustrated in [Figure 7](#), through the activation of methanol by basic oxide species ( $\text{O}^{2-}$ ) [\[7\]](#), [\[8\]](#). These methoxide ions act as nucleophiles that attack the carbonyl carbon of the triglyceride, leading to the formation of fatty acid methyl esters (FAME) and glycerol. This mechanism occurs in three sequential steps: conversion of triglyceride to diglyceride, then to monoglyceride, and finally to glycerol, with the release of one FAME molecule at each stage.



**Figure 7.** Transesterification Mechanism

This mechanistic pathway supports the experimental findings, where the presence of strong basic sites on the CaO-based catalyst significantly enhanced the methanol activation and facilitated the successive conversion of triglycerides to methyl esters, leading to higher biodiesel yield [23], [24].

## CONCLUSION

The sucrose-assisted hydrothermal synthesis method successfully produced a high-purity calcium oxide catalyst with enhanced crystallinity, particle uniformity, and surface area. The catalyst, CaO-SH, demonstrated outstanding catalytic activity in the transesterification of *Reutealis trisperma* oil, achieving high conversion and biodiesel yield at an optimal catalyst dosage of 7.5 wt%. This research contributes theoretically by validating the effectiveness of green separation and crystal refinement strategies in improving solid base catalyst performance. It also offers practical implications for biodiesel production using sustainable and locally available materials. However, this study is limited to batch-scale reactions without reuse testing. Future research should investigate the catalyst's long-term stability, reusability, and its performance in continuous flow systems or against other non-edible oil feedstocks to assess broader applicability.

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