

## Biomass energy production from sunflower plant

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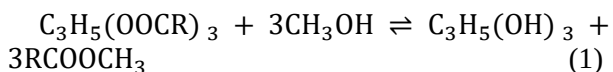
Concerns about fossil fuel depletion and the environmental costs of petroleum combustion have steadily pushed biodiesel toward the center of renewable energy research. This study reports on the production of biodiesel from sunflower oil via base-catalyzed transesterification, using a nano-scale calcium oxide (n-CaO) catalyst prepared by the calcination of calcium carbonate (CaCO<sub>3</sub>) at 900 °C. The synthesized catalyst was characterized by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) analyses. XRD patterns confirmed the formation of predominantly crystalline cubic CaO with minor carbonate and hydroxide traces, with crystallite sizes ranging from 27 to 31 nm and a mean of approximately 29 nm calculated from the Debye–Scherrer equation. FTIR data showed Ca–O stretching as the primary surface feature, with weak carbonate and hydroxyl signals attributable to limited atmospheric contact. SEM images revealed an irregular, porous, and substantially agglomerated particle morphology consistent with a high accessible surface area. The optimal conditions for transesterification were a methanol-to-oil molar ratio of 12:1, a catalyst loading of 3 wt%, a reaction temperature of 60–65 °C, and a reaction duration of 2 hours, yielding 91.0 ± 1.2% biodiesel. ASTM D6751 test methods were employed to determine significant fuel properties of the biodiesel, revealing an acid value of 0.95 mg KOH/g, the sole property exceeding the standard limit. These findings confirm that n-CaO prepared by simple thermal decomposition of an inexpensive, readily available CaCO<sub>3</sub> precursor is an easily separable heterogeneous catalyst for biodiesel production.

**Keywords:** nano-CaO, biodiesel, transesterification, sunflower oil, heterogeneous catalyst, acid value.

### INTRODUCTION

Global energy systems are increasingly transitioning toward renewable alternatives, driven by the finite nature of fossil fuel reserves, the environmental consequences of combustion emissions on global climate, and the geopolitical vulnerabilities associated with concentrated petroleum supply chains [1-3]. Among the fuels capable of integrating into existing infrastructure without requiring wholesale engine redesign, biodiesel occupies a well-studied and practically viable position [2].

Biodiesel is produced through the transesterification of triglycerides- plant oils, animal fats, or used cooking oil- with a short-chain alcohol, typically methanol. The overall reaction, shown in Eqn. (1), yields three moles of fatty acid methyl esters (FAME) per mole of triglyceride, along with glycerol as a co-product [4, 5]:



The chemistry is sensitive in practice: the nature and loading of the catalyst, the free fatty acid (FFA) content of the feedstock, the alcohol excess, the reaction temperature, and the contact time all affect

the usable yield obtained. High FFA content is a particular complication in base-catalyzed systems, where it promotes saponification—the formation of soap rather than fuel—which reduces yield and makes subsequent separation more difficult [5].

Heterogeneous solid base catalysts, particularly calcium oxide (CaO), have attracted considerable attention for biodiesel production due to their ease of separation, recyclability, and avoidance of alkaline wastewater generation [2, 6]. Among solid bases, CaO stands out for its strong basicity, low cost, and wide availability; it can be sourced from calcium-rich waste streams such as eggshells, marine shells, and marble dust with no meaningful performance penalty [7, 8]. Calcination at approximately 900 °C is generally considered sufficient for complete CaCO<sub>3</sub>-to-CaO conversion [7, 9, 10].

Nanoscale CaO particles possess higher surface-area-to-volume ratios and basic site densities, enhancing catalytic activity at lower loadings [2, 5]. Thermal decomposition is the simplest synthesis route, routinely producing crystallite sizes in the 20–31 nm range depending on calcination temperature. Degfie *et al.* [5] confirmed that nano-CaO (~29 nm, 500 °C calcination) delivers 96% yield at only 1 wt% loading and 50 °C using waste cooking oil, while Erchamo *et al.* [53] achieved 94% with eggshell-

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derived nano-CaO, showing that waste precursors match reagent-grade performance at lower cost [10].

Despite the substantial body of literature on nano-CaO-catalyzed biodiesel production, several gaps remain. First, the majority of nano-CaO studies use waste cooking oil as feedstock, while fewer systematically investigate refined sunflower oil—which, owing to its lower viscosity and negligible FFA content, provides a cleaner baseline for isolating catalyst effects from feedstock variability. Second, most comparative studies either vary feedstock or vary catalyst source, but rarely hold both constant against a nano-scale versus bulk-scale CaO comparison. Third, to the best of the authors' knowledge, limited studies have combined the 900 °C thermal decomposition route for nano-CaO synthesis with a full ASTM D6751 characterization of the resulting biodiesel from sunflower oil—including a dedicated acid value determination—while simultaneously providing a mechanistic discussion of the mass-transfer constraints that arise in the absence of a cosolvent.

This study synthesizes n-CaO from CaCO<sub>3</sub> at 900 °C, characterizes it by XRD, FTIR, and SEM, optimizes transesterification conditions for sunflower oil, evaluates fuel properties against ASTM D6751, and benchmarks yield against fifteen related CaO-based systems.

## EXPERIMENTAL

### Catalyst preparation

The nano-CaO catalyst was prepared by heating calcium carbonate (CaCO<sub>3</sub>) powder in a muffle furnace at 900 °C for three hours. Calcination was carried out at 900 °C, which achieves complete phase conversion and peak BET surface area for carbonate-derived CaO [10]. The freshly calcined CaO was immediately transferred to a desiccator to avoid rapid atmospheric deactivation [2]. When the catalyst was to be reused, the spent material was re-calcined at 900 °C; this step thermally decomposes any accumulated Ca(OH)<sub>2</sub> and CaCO<sub>3</sub> back to the active oxide [30,31].

### Feedstock characterization

Before starting the transesterification runs, the sunflower oil was characterized by acid value titration to quantify its free fatty acid content. A weighed oil sample was dissolved in a 1:1 ethanol-diethyl ether mixture and titrated with 0.1 N KOH using phenolphthalein as indicator, continuing until the endpoint color persisted for at least 30 seconds. The measured acid value was 1.40 mg KOH/g oil—low enough to proceed directly to base-catalyzed

transesterification without a preliminary esterification step.

### Transesterification procedure

All transesterification reactions were run in triplicate ( $n = 3$ ) in a 250 mL round-bottom flask fitted with a reflux condenser to contain methanol vapor [3, 8]. In each run, the catalyst and methanol were combined first and stirred for 30 minutes at room temperature before the oil was added. This pre-activation step encourages the formation of surface methoxide species—the proximal catalytic intermediates—before the triglyceride substrate is introduced [17, 28].

The optimized reaction conditions are summarized in Table 1. The 60–65 °C range was chosen because it sits just below methanol's atmospheric boiling point of 64.7 °C, keeping the system at a kinetically favorable temperature while the reflux condenser recaptures any vapor that forms [5, 24]. In the absence of a cosolvent, agitation at 800 rpm was relied upon to maintain contact between the oil and methanol phases [3]. After two hours the flask was removed from the heating plate and allowed to cool before the product was worked up. Biodiesel yield was calculated gravimetrically as the mass of purified, dried biodiesel obtained relative to the initial oil mass, as shown in Eqn. (2) [5, 8]:

$$\text{Yield (\%)} = \frac{m_b}{m_o} \times 100 \quad (2)$$

where:  $m^b$  = mass of purified, dried biodiesel (g);  $m_o$  = initial oil mass (g).

**Table 1.** Optimized transesterification reaction conditions

| Parameter                     | Value |
|-------------------------------|-------|
| Oil amount (g)                | 100   |
| Methanol volume (mL)          | 55    |
| Methanol-to-oil molar ratio   | 12:1  |
| Catalyst loading, n-CaO (wt%) | 3     |
| Reaction temperature (°C)     | 60–65 |
| Reaction time (h)             | 2     |
| Stirring speed (rpm)          | 800   |

### Product separation and purification

The cooled reaction mixture was poured into a separatory funnel and left undisturbed for 12 h [3, 29]. Two layers developed: a lighter, FAME-rich biodiesel phase on top and a denser phase below containing glycerol, unreacted methanol, and dissolved impurities [3, 8].

### Catalyst characterization

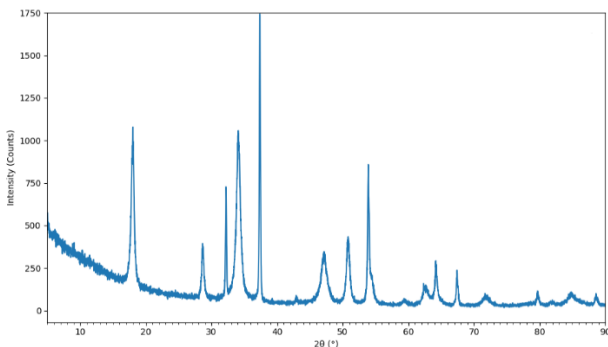
Phase composition and crystallite size were determined using a Malvern PANalytical X'Pert

PRO diffractometer with Cu K $\alpha$  radiation ( $\lambda = 1.54059 \text{ \AA}$ ) [10, 24]. Crystallite size was calculated by applying the Debye–Scherrer equation to the five most prominent reflections [17, 24]. Surface functional groups were identified with a PerkinElmer Spectrum 100 FT-IR spectrometer, scanning from 4000 to 400  $\text{cm}^{-1}$  [2, 17]. Particle morphology was examined using a Thermo Scientific Apreo 2S field-emission SEM; images were collected at four magnifications of  $\times 5,000$ ,  $\times 20,000$ ,  $\times 30,000$ , and  $\times 60,000$  [5, 7].

## RESULTS AND DISCUSSION

### Catalyst characterization results

XRD spectra of the synthesized nano-CaO are presented in Fig. 1. Sharp peaks at  $2\theta = 32.2^\circ$ ,  $37.3^\circ$ ,  $47.3^\circ$ ,  $53.8^\circ$ ,  $64.1^\circ$ ,  $67.3^\circ$ ,  $82.2^\circ$ , and  $86.5^\circ$  index to the cubic rock-salt structure of CaO (JCPDS No. 37-1497), confirming that 900  $^\circ\text{C}$  calcination converted  $\text{CaCO}_3$  predominantly to CaO [10, 17]. Two lower-intensity peaks near  $29.4^\circ$  and  $18.0^\circ$  are assignable to trace  $\text{CaCO}_3$  and  $\text{Ca(OH)}_2$ , characteristic surface species formed during handling [2, 17]. Applying the Debye–Scherrer equation to the five strongest reflections gave crystallite sizes of 27.02–31.21 nm (mean: 29.41 nm), consistent with the  $\sim 29 \text{ nm}$  reported by Degfie *et al.* [5] for nano-CaO prepared by thermal decomposition.

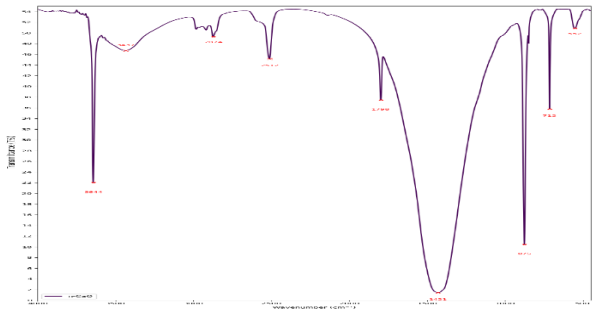


**Fig. 1.** XRD pattern of nano-CaO catalyst.

The FTIR spectrum (Fig. 2) shows a prominent Ca–O stretching absorption at 500–600  $\text{cm}^{-1}$ ,

confirming CaO as the dominant phase [2, 17]. A band at 3640  $\text{cm}^{-1}$  indicates O–H stretching in surface  $\text{Ca(OH)}_2$ , while features at 1400–1470  $\text{cm}^{-1}$  and 870  $\text{cm}^{-1}$  arise from carbonate species. The relative weakness of these secondary bands confirms that surface carbonation and hydroxylation are superficial and do not compromise bulk catalytic activity [2, 17]. These FTIR features are consistent with published spectra of nano-CaO prepared by thermal decomposition [5, 10].

SEM images at  $\times 5,000$ ,  $\times 20,000$ ,  $\times 30,000$ , and  $\times 60,000$  (Fig. 3) revealed an irregular, porous, and substantially agglomerated particle morphology, typical of thermally synthesized nano-scale oxide powders [8, 21]. The rough, textured surface and visible porosity support high accessible surface area for contact with the oil–methanol mixture. The agglomerated morphology also explains why 3 wt% catalyst loading is required to achieve maximum yield, as the true accessible surface area is lower than what the mean crystallite size alone would suggest [5, 24].

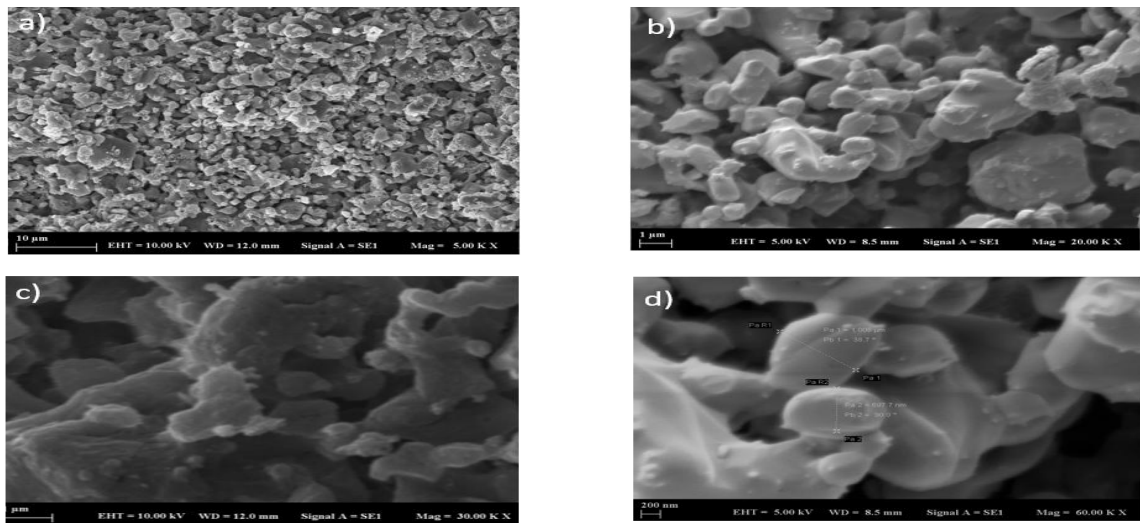


**Fig. 2.** FTIR spectrum of the nano-CaO catalyst.

Table 2 summarizes the catalytic performance of CaO derived from diverse calcium-rich sources, as reported across the literature. As shown in Table 2, yields consistently exceed 90% across a wide range of precursor types and feedstocks when calcination is carried out at or near 900  $^\circ\text{C}$ , confirming the robustness of the thermal decomposition route [36, 37].

**Table 2.** Catalytic performance of CaO derived from diverse calcium-rich sources

| CaO source                                | Feedstock         | MeOH:Oil | Cat. (wt%) | Temp ( $^\circ\text{C}$ ) | Time (h) | Yield (%) | Ref. |
|-------------------------------------------|-------------------|----------|------------|---------------------------|----------|-----------|------|
| Egg/mollusk shell (800 $^\circ\text{C}$ ) | Palm olein        | 9:1      | 3          | 65                        | 2        | >90       | [7]  |
| Chicken eggshell (900 $^\circ\text{C}$ )  | Sunflower         | 9:1      | 3          | 60                        | 3        | 94.73     | [10] |
| Quail eggshell (900 $^\circ\text{C}$ )    | Sunflower         | 10.5:1   | 2          | 60                        | 2        | 99.0      | [38] |
| River snail shell (900 $^\circ\text{C}$ ) | Palm olein        | 12:1     | 5          | 65                        | 1.5      | 98.5      | [40] |
| Cockle shell (900 $^\circ\text{C}$ )      | Palm olein        | 9:1      | 10         | 65                        | 4        | 94.47     | [42] |
| Chicken bone (900 $^\circ\text{C}$ )      | WCO               | 15:1     | 5          | 65                        | 4        | 89.33     | [31] |
| Oyster shell (combusted)                  | Soybean           | 24:1     | 2          | 65                        | 5        | 98.0      | [43] |
| n-CaO – 29 nm                             | Waste cooking oil | 8:1      | 1          | 50                        | 1.5      | 96        | [5]  |



**Fig. 3.** (a–d) SEM images of synthesized nano-CaO catalyst at magnifications of  $\times 5,000$ ,  $\times 20,000$ ,  $\times 30,000$  and  $\times 60,000$ , respectively.

**Table 3.** Biodiesel yield: this work alongside selected CaO-based literature systems

| Feedstock / catalyst                        | MeOH:oil | Catalyst load (wt%) | Temperature (°C) | Time (h) | Yield (%)      | Ref.      |
|---------------------------------------------|----------|---------------------|------------------|----------|----------------|-----------|
| Sunflower oil / n-CaO (29 nm, 900°C)        | 12:1     | 3                   | 60–65            | 2        | 91.0 $\pm$ 1.2 | This work |
| Sunflower oil / CaO (eggshell, 900°C)       | 9:1      | 3                   | 60               | 3        | 97.75          | [10]      |
| Sunflower oil / CaO (quail eggshell, 900°C) | 10.5:1   | 2                   | 60               | 2        | 99.0           | [19]      |
| Waste cooking oil / n-CaO (29 nm)           | 8:1      | 1                   | 50               | 1.5      | 96             | [5]       |
| WCO / lime-based Zn-CaO                     | 14:1     | 5                   | 57.5             | 2        | 96.5           | [33]      |
| Argemone mexicana / eggshell CaO            | 9.7:1    | 3.05                | 60               | 3        | 99.07          | [50]      |
| Sunflower oil / CaO (marble dust)           | 15:1     | 5                   | 60               | 2        | 89.14          | [12]      |
| Sunflower oil / CaO (eggshell)              | 15:1     | 5                   | 60               | 2        | 73             | [12]      |
| Sunflower oil / CaO (sugar industry)        | 15:1     | 5                   | 60               | 2        | 87.64          | [12]      |
| Sunflower oil, NaOH                         | 6:1      | 1                   | 65               | 1        | 90.9           | [14]      |
| Soybean oil / CaO (snail shell)             | 6:1      | 3                   | 28               | 7        | 98             | [51]      |
| WCO / CaO (chicken manure)                  | 15:1     | 7.5                 | 65               | 5        | 90             | [29]      |
| Canola oil / CaO (ultrasound)               | 7.48:1   | 5.35                | 60               | 2.5      | 99.4           | [4]       |
| WCO / nano CaO+MgO                          | 7:1      | 1.5                 | 75               | 6        | 94.4           | [24]      |
| Rapeseed oil / Eggshell CaO                 | 9:1      | 4                   | 60               | 1        | 95.12          | [52]      |
| WCO / eggshell nano-CaO (MeOH/EtOH)         | 12:1     | 2.5                 | 60               | 2        | 94             | [53]      |
| WCO / white bivalve clam shell CaO          | 12:1     | 7                   | 65               | 1        | 94.25          | [53]      |

*Effect of reaction parameters on biodiesel yield*

- **Catalyst loading.** Yield increased with catalyst loading up to 3 wt%, beyond which excess solid raised mixture viscosity and promoted saponification, both reducing yield. The 3 wt% optimum is notably lower than the 5 wt% required by bulk CaO systems [7, 33], reflecting the higher surface area per gram of the nano-scale material [5, 24].

- **Methanol-to-oil molar ratio.** Yield increased with methanol-to-oil ratio up to 12:1, beyond which excess methanol dissolved glycerol into the biodiesel phase and impeded separation. The 12:1 optimum exceeds the 8:1 reported by Degfie *et al.* [5] for waste cooking oil, which is attributed to the absence of a cosolvent here: without a phase-bridging agent the oil–methanol boundary imposes a mass-transfer penalty that a larger methanol excess compensates for [40, 51].

- **Reaction temperature.** Yield improved up to 60–65 °C, where reduced oil viscosity improves mass transfer and thermal energy accelerates the reaction rate. The reflux condenser retained methanol vapor, keeping the effective molar ratio at the optimum. The higher optimum compared with the 50 °C of Degfie *et al.* [5] reflects sunflower oil's lower viscosity requiring more thermal driving force [4, 46].

- **Reaction time.** Yield increased over the first 2 h as the reaction approached equilibrium; extending beyond this added no conversion and risked reverse reaction. The 2 h optimum falls within the 1.5–2.5 h window reported for comparable CaO systems [5, 48].

- **Acid value of the produced biodiesel.** The acid value of the biodiesel produced under the optimized transesterification conditions (Table 1) was determined according to BS EN 14104:2021, the international standard method for the determination of acid value in fatty acid methyl esters. A weighed biodiesel sample was dissolved in a neutralized 1:1 (v/v) mixture of ethanol and diethyl ether and titrated against 0.1 N potassium hydroxide solution using phenolphthalein as the visual endpoint indicator. The acid value was calculated using the standard relationship given in Equation (3):

$$AV \text{ (mg KOH/g)} = \frac{V \times N \times 56.1}{m} \quad (3)$$

where:  $V$  = volume of KOH solution consumed (mL);  $N$  = normality of the KOH solution (0.1 N); 56.1 = molar mass of KOH (g/mol);  $m$  = mass of the biodiesel sample (g).

However, the measured value of 0.95 mg KOH/g remains slightly above the maximum limit of 0.50

mg KOH/g specified by both ASTM D6751 and EN 14214 for finished biodiesel. This result suggests the presence of residual free fatty acids or minor acidic by-products remaining in the final product. In addition, process variables such as reaction efficiency, phase separation behavior, and the extent of impurity removal may also contribute to the observed acid number.

The acid value result is consistent with that of Bedir and Doğan [7], who also reported finished fuel properties within EN 14214 and ASTM D6751 limits only after additional purification optimization; and with Degfie *et al.* [5], who reported an acid number below 0.50 mg KOH/g for nano-CaO-catalyzed biodiesel from waste cooking oil after an extended purification protocol.

## CONCLUSION

This study synthesized and characterized a nano-CaO catalyst by thermal decomposition of  $\text{CaCO}_3$  at 900 °C and evaluated its catalytic performance in the transesterification of sunflower oil. XRD confirmed predominantly crystalline cubic CaO with a mean crystallite size of 29.41 nm; FTIR and SEM analyses showed a porous, agglomerated morphology consistent with high accessible surface area. Under optimized conditions (12:1 methanol-to-oil, 3 wt% catalyst, 60–65 °C, 2 h), a biodiesel yield of  $91.0 \pm 1.2\%$  was achieved. The acid value of 0.95 mg KOH/g, determined by BS EN 14104:2021, was marginally above the ASTM D6751 limit of 0.50 mg KOH/g, indicating that downstream purification remains the primary optimization target. The 3 wt% optimum catalyst loading—below the 5 wt% typically required for bulk CaO—confirms the surface-area advantage of the nano-scale morphology. Future work should focus on targeted purification steps, systematic reusability testing, and the use of waste-derived  $\text{CaCO}_3$  precursors to further reduce production cost.

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personal relationships that could have appeared to influence the work reported in this paper.

**Declaration of generative ai and ai-assisted technologies in the manuscript preparation:** During the preparation of this work the authors used ChatGPT to improve readability and language. The analysis data was visualized by generating Python code using Google Colab support.

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