



# “Green Synthesis of CaO Nanocatalysts Using *Musa paradisiaca* Leaves and Its Efficient Application in the Synthesis of N-Morpholinobenzothioamide

Jayesh T Patil<sup>1\*</sup>, Sandesh V Jaybhaye<sup>2</sup>

1- “Research Scholar at the Nanotechnology Research Laboratory, Department of Chemistry, B.K. Birla College, Kalyan, affiliated with the University of Mumbai, Maharashtra – 421301” India.

2- “Professor and Research Guide, Department of Chemistry, B.K. Birla College, Kalyan, Maharashtra – 421301” India.

## Abstract:

The development of sustainable and eco-friendly catalytic systems has emerged as a central focus in modern synthetic chemistry. In this study, a green and cost-effective approach is reported for the synthesis of calcium carbonate (CaO) nanocatalysts using *Musa paradisiaca* (banana) leaf extract as a natural reducing and stabilizing agent. The biogenic route eliminates the need for hazardous chemicals, offering an environmentally benign alternative while enhancing the physicochemical properties of the nanomaterial. The synthesized CaO nanocatalysts were systematically characterized using standard analytical techniques, confirming their nanoscale morphology, high surface area, and crystalline nature. Exploiting these features, the prepared nanocatalyst was successfully employed as an efficient heterogeneous catalyst for the synthesis of N-Morpholinobenzothioamide under mild and sustainable conditions. The catalytic system demonstrated excellent activity, affording high yield, reduced reaction time, and easy product isolation. Furthermore, the catalyst exhibited good reusability and stability, highlighting its practical applicability in green organic synthesis. This work not only provides a novel utilization of plant-mediated nanotechnology but also establishes a greener synthetic pathway for thioamide derivatives. The integration of renewable biological resources with nanocatalysis underscores a promising strategy toward environmentally responsible chemical processes and sustainable development in organic synthesis.

**Keywords:-** Green synthesis, Calcium oxide nanoparticles (CaO), *Musa paradisiaca*, Nanocatalyst, Heterogeneous catalysis, N-Morpholinobenzothioamide, Thioamide synthesis, Sustainable chemistry

## 1. Introduction

The imperative to develop environmentally benign and sustainable synthetic methodologies has driven extensive research in green chemistry and nanotechnology. Green chemistry principles emphasize the reduction of hazardous substances, energy efficiency, and the use of renewable materials throughout chemical processes [1]. Nanomaterials, with their high surface-to-volume ratios and tunable physicochemical properties, present unique opportunities in catalysis and organic synthesis [2,3]. They have been widely explored for applications ranging from environmental remediation to catalysis due to enhanced reactivity relative to their bulk counterparts. Among nanomaterials, calcium carbonate (CaO) nanoparticles represent an environmentally friendly and cost-effective class with significant potential in sustainable catalysis. CaO is widely available in nature and exists in multiple polymorphs, such as calcite, aragonite, and vaterite, which influence particle morphology and reactivity [4]. Nanoscale CaO exhibits improved surface area, porosity, and catalytic properties, which have been leveraged in controlled drug delivery, composite materials, and catalysis [5,6]. Conventional synthesis of CaO nanoparticles using chemical precipitation, microemulsion, or hydrothermal methods often involves toxic reagents, high energy input, or complex processing, limiting sustainability [7,8]. These challenges have catalyzed interest in plant-mediated or “green” synthesis routes that rely on natural biomolecules as reducing, stabilizing, and templating agents. Plant extracts contain diverse phytochemicals, such as polyphenols, flavonoids, terpenoids, and carbohydrates, which can act as eco-friendly agents for nanoparticle formation. These biomolecules facilitate nucleation and growth while preventing particle agglomeration, resulting in well-defined nanostructures without the need for harsh chemicals [9,10]. Bio-mediated synthesis has been widely demonstrated for metallic and metal oxide nanoparticles; for example, Ag and Au nanoparticles synthesized using *Ocimum tenuiflorum* (Tulsi) leaf extract exhibited excellent stability and catalytic activity [11], while ZnO nanoparticles synthesized with *Azadirachta indica* extract showed enhanced photocatalytic performance [12]. The use of agricultural waste and plant biomass aligns with the principles of sustainability and circular economy. *Musa paradisiaca* (banana) leaves are produced in vast quantities as agricultural residues and are rich in flavonoids, phenolics, and cellulose [13,14]. These phytochemicals have been shown to be effective in the biogenic synthesis of nanoparticles, including silver and zinc nanoparticles, indicating their potential as green reagents [15,16]. However, the application of *Musa paradisiaca* leaf extract for the synthesis of CaO nanocatalysts has not been extensively explored, representing an untapped opportunity for sustainable nanomaterial fabrication. The catalytic potential of CaO nanoparticles is associated with their basic surface sites and high surface area, which promote organic transformations under mild conditions [12,17]. Nanocatalysis using heterogeneous catalysts like CaO allows for easy catalyst recovery, recyclability, and reduced waste generation, which are key advantages in green synthesis [18,19].

For organic transformations requiring nucleophilic activation or surface adsorption of reactants, nanostructured CaO offers enhanced active sites that improve reaction efficiency and selectivity.

Thioamides are an important class of sulfur-containing compounds with broad applications in pharmaceuticals, agrochemicals, and materials science due to their unique chemical reactivity and biological properties [20,21]. Among these, N-Morpholinobenzothioamide has emerged as a valuable molecule for potential medicinal applications and as a versatile intermediate in heterocyclic synthesis [22]. Traditional synthetic routes for thioamides typically involve toxic phosphorus-based reagents such as Lawesson's reagent or phosphorus pentasulfide in non-green conditions, limiting scalability and environmental compatibility [23,24]. Recent efforts in green organic synthesis have focused on developing heterogeneous catalytic systems that minimize hazardous reagents and operate under milder conditions [25,26]. Nanocatalysts such as metal oxides, carbon-based materials, and mixed composites have demonstrated improved efficiencies in C–S bond formation, an essential step in thioamide synthesis [27,28]. Yet, studies leveraging green-synthesized CaO nanocatalysts for thioamide formation remain limited. In this study, we report a green synthesis approach for CaO nanocatalysts using *Musa paradisiaca* leaf extract, followed by their application in the efficient synthesis of N-Morpholinobenzothioamide. The developed nanocatalyst was systematically characterized to confirm morphology, crystallinity, and surface properties. Its catalytic performance was evaluated under sustainable conditions, demonstrating high efficiency, reusability, and reduced environmental impact. This work bridges green nanotechnology with organic synthesis, presenting a sustainable strategy for the fabrication and application of plant-derived nanocatalysts.

## 2. Experimental Work:

### 2.1 Synthesis of CaCO<sub>3</sub> nanoparticles using green synthesis method

Calcium nitrate tetrahydrate [Ca(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O] and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) were used as precursors, and fresh *Musa paradisiaca* leaves were collected locally for extract preparation. The leaves were thoroughly washed, chopped, and boiled in distilled water (10 g in 100 mL) at 80 °C for 15 min. The extract was cooled, filtered, and stored at 4 °C for further use as a biogenic stabilizing agent. For the synthesis, an aqueous solution of calcium nitrate (0.1 M) was mixed with 20 mL of the leaf extract under continuous stirring. Subsequently, 0.1 M sodium carbonate solution was added dropwise until complete precipitation of calcium carbonate (CaCO<sub>3</sub>) occurred. The reaction mixture was stirred for 2 h and aged for 24 h to ensure complete formation of the precursor. The precipitate was then collected by centrifugation, washed several times with distilled water and ethanol to remove impurities, and dried at 70 °C for 6–8 h. The obtained dried CaO powder was finely ground and subjected to calcination at 700–900 °C for 2–4 h in a muffle furnace. During this process, CaCO<sub>3</sub>

undergoes thermal decomposition to form calcium oxide (CaO) nanoparticles with the release of CO<sub>2</sub> gas. The resulting CaO nanoparticles were cooled to room temperature and stored in an airtight container for further use. [29,30].

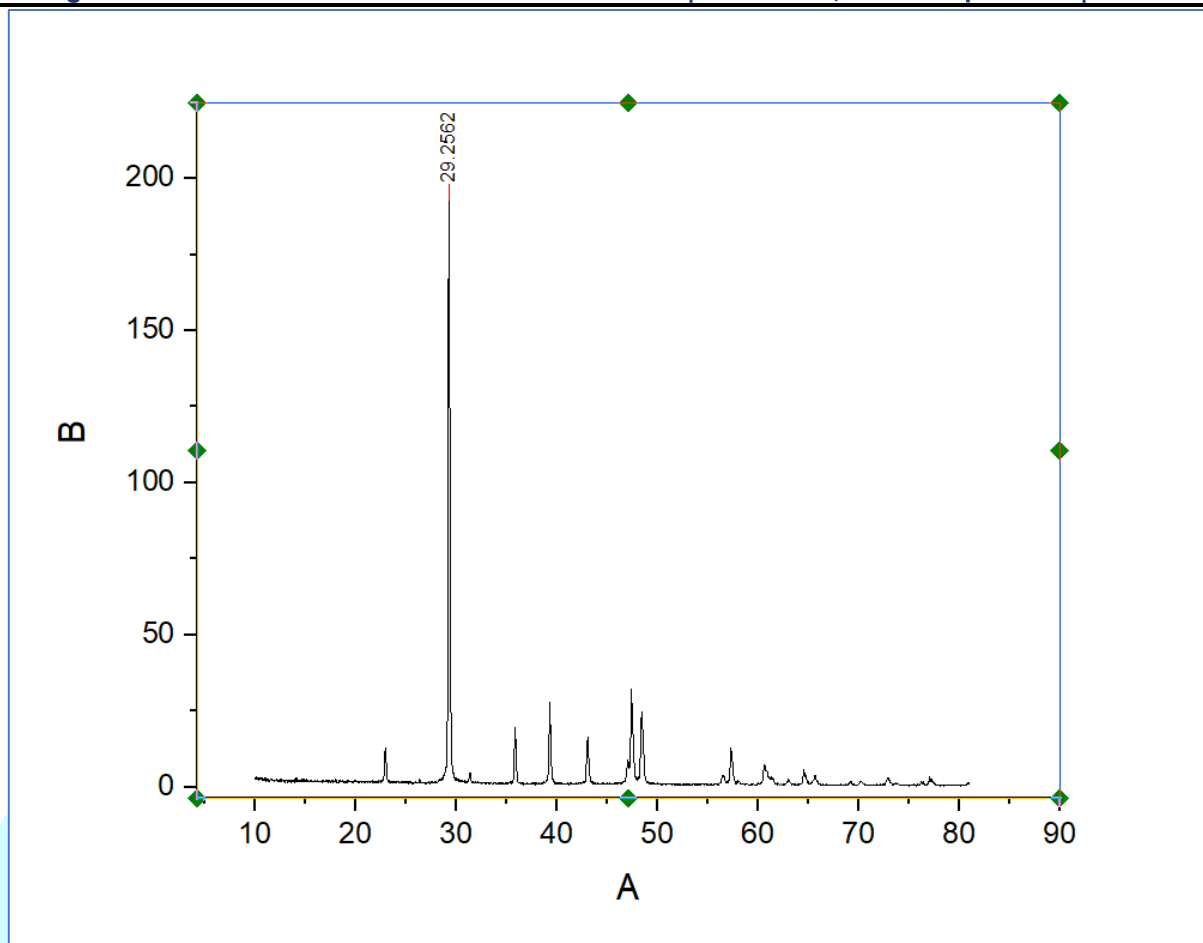
## 2.2 Structural Characterization:

The synthesized CaO NPs were characterized by XRD, FESEM, and EDAX to study the structural properties, surface morphology, and elemental composition analysis respectively.

## 2.3. Results and Discussion:

### 2.3.1 X-Ray Diffraction (XRD)

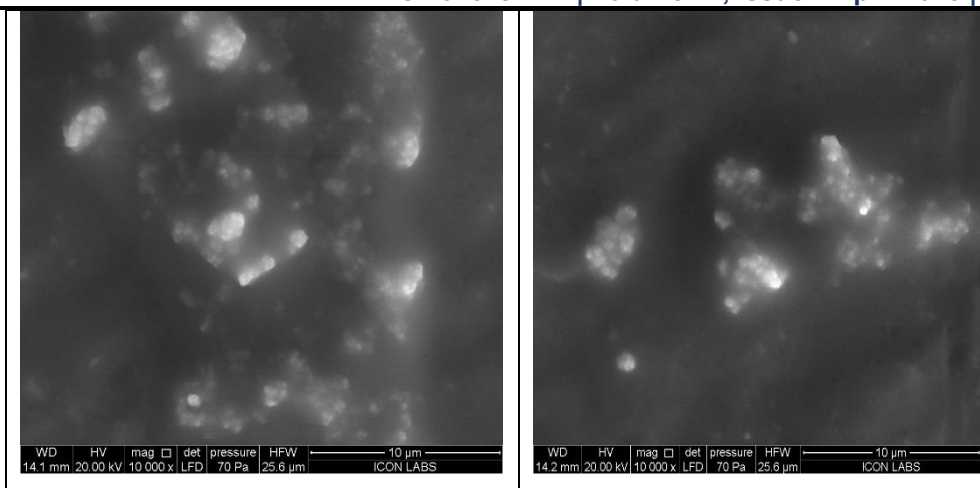
The X-ray diffraction (XRD) pattern of natural CaO nanocatalyst reveals a highly crystalline calcite phase (This pattern clearly matches the phase of standard (JCPDS card No 01-071-3699, 00-066-0867), with characteristic peaks at  $2\theta = 29.299^\circ$  (104 plane, 100% intensity,  $d = 3.046 \text{ \AA}$ ),  $39.309^\circ$  (21.9%),  $35.877^\circ$  (12.4%),  $47.445^\circ$  (17.8%), and others, figure-1, confirming rhombohedral structure without vaterite or aragonite impurities. Sharp peaks (FWHM  $\sim 0.10^\circ$ ) indicate nanoscale domains; using the Scherrer equation  $D = \frac{K\lambda}{\beta \cos \theta}$  ( $K=0.9$ ,  $\lambda=1.5406 \text{ \AA}$ ,  $\beta=0.00175 \text{ rad}$  at  $2\theta=29.299^\circ$ ), the average crystallite size is calculated as 82 nm. This morphology ensures high surface area and stability, ideal for heterogeneous catalysis in organic synthesis. The structural properties of CaO nanocatalyst were confirmed by X-ray diffraction (XRD); Bruker D2 PHASER (Open designated in 2<sup>nd</sup> generation as A26-X1-A2B0B2B).



**Figure 1: XRD pattern of CaO nanocatalysts**

### 2.3.2 Scanning Electron Microscopy (SEM)

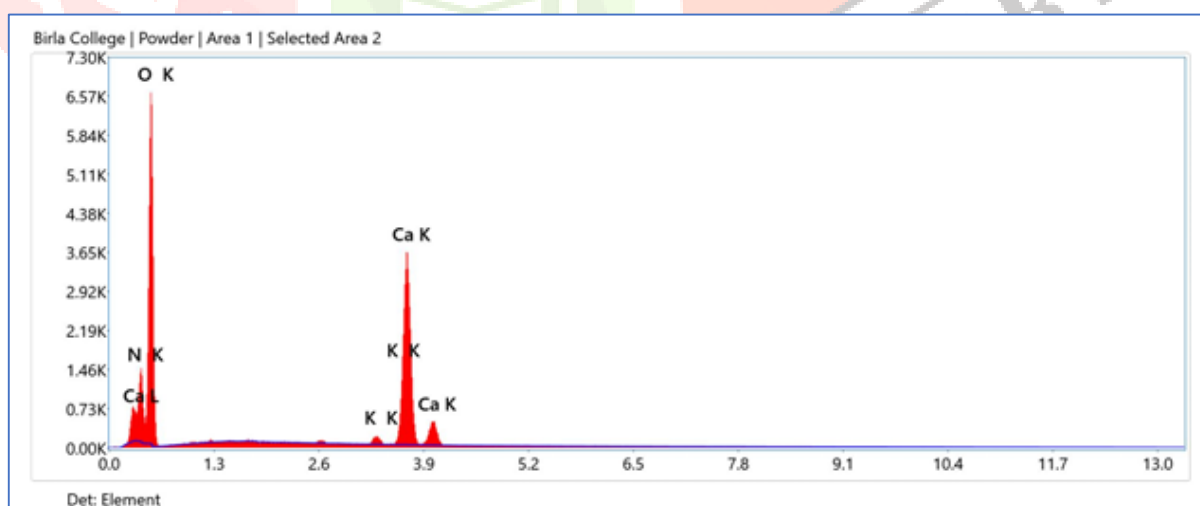
Figure 2 shows a SEM micrograph of the synthesised CaO NPs. The formation of spherical nanoparticles with large agglomeration was observed in SEM images of ZnO NPs obtained using the green synthesis method. The SEM images reveal that the CaO particles are irregularly shaped aggregates composed of nanosized primary particles. The brighter regions correspond to dense agglomerates, suggesting partially fused or clustered nanoparticles. The particle size distribution appears non-uniform, with average particle diameters in the range of approximately 80-100 nm, although smaller primary particles can also be distinguished in some areas. The observed agglomeration is typical for CaO nanoparticles, likely due to strong van der Waals and electrostatic interactions among the fine particles. The morphological properties of CaO nanocatalyst were confirmed by SEM Qanta 200 manufacture FEI. The surface morphology shows rough and porous features, which can enhance the specific surface area and thereby improve the catalytic activity of the nanocatalyst.[32-33] Figure-2



**Figure 2: FESEM micrograph of CaO nanoparticles**

### 2.3.3 Energy Dispersive X-Ray Analysis (EDAX)

The proportional elemental composition of CaO nanoparticles was confirmed using the energy dispersive X-rays Analysis (EDAX) tool, as illustrated in Figure 3, by measuring the intensity of the characteristic emitted x-rays. In synthesised calcium oxide nanoparticles, EDAX revealed major the presence of two elements: calcium and oxygen. The spectrum exhibits prominent peaks for Oxygen (O) and Calcium (Ca), which are the primary constituents of calcium oxide (CaO). Minor peaks corresponding to Nitrogen (N), Chlorine (Cl), and Potassium (K) are also observed, suggesting the presence of impurities or residual elements from the synthesis process. The high intensity of the O and Ca peaks indicates their high concentration in the sample. The Energy Dispersive X-Ray Analysis of CaO nanocatalyst were confirmed by instrument EDAX AMEPEK.



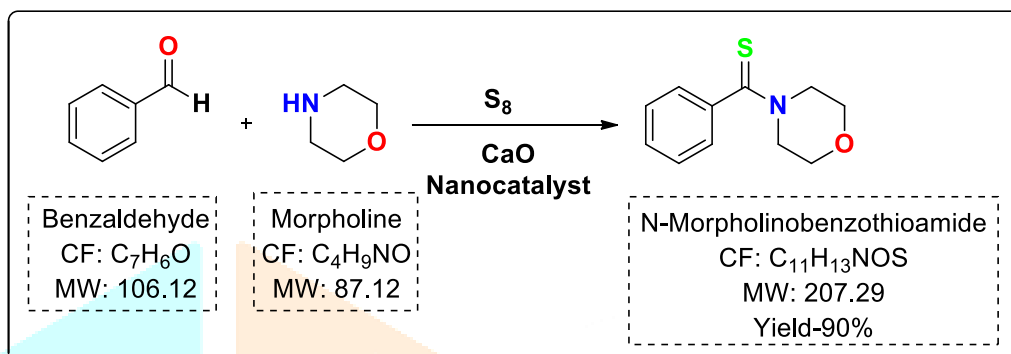
**Figure 2: Energy Dispersive X-Ray Analysis (EDAX)**

### 3. Application of synthesized CaO Nanocatalysts in the Synthesis of N-Morpholinobenzothioamide.

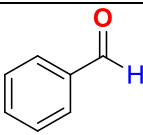
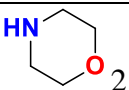
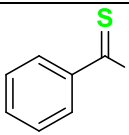
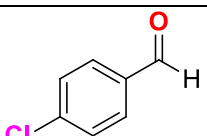
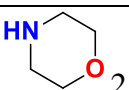
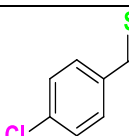
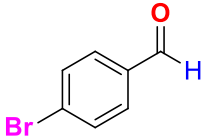
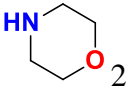
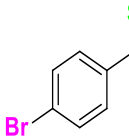
The synthesized CaO nanoparticles were efficiently employed as a heterogeneous nanocatalyst for the synthesis of N-morpholinobenzothioamide from an aromatic aldehyde, morpholine, and a sulfur source under

mild conditions. The high surface area and basic active sites of CaO facilitate carbonyl activation and subsequent thioamide formation, leading to good to excellent yields without the need for harsh reagents or chromatographic purification. The porous and agglomerated morphology further enhances catalytic performance.[34-35]

### 3.1 Methodology for the synthesis of N-Morpholinobenzothioamide using calcium oxide nanocatalysts.



N-Morpholinobenzothioamide was synthesized via a one-pot multicomponent reaction using calcium oxide (CaO) nanoparticles as a heterogeneous base catalyst. In a typical procedure, an equimolar mixture of an aromatic aldehyde (1 mmol), morpholine (1 mmol), and a suitable sulfur source such as elemental sulfur (S<sub>8</sub>) (1 mmol) was taken in a round-bottom flask. A catalytic amount of CaO<sub>3</sub> nanoparticles (10–15 mol%) was added to the reaction mixture, and the contents were stirred under solvent-free conditions (or in ethanol, if required) at 70–80 °C. The progress of the reaction was monitored by thin-layer chromatography (TLC). Upon completion, the reaction mixture was cooled to room temperature, and the solid catalyst was separated by simple filtration. The crude product was washed with cold water and crystallized from ethanol to afford pure N-morpholinobenzothioamide in 90% yield. Table-1

Aldehyde	Morpholine	Yield	Product
 <b>1-a</b>	 2	90%	 <b>3-a</b>
 <b>1-b</b>	 2	88%	 <b>3-b</b>
 <b>1-c</b>	 2	88%	 <b>3-c</b>

**Table-1: Synthesize derivative of N-Morpholinobenzothioamide.****3.2: Characterization of N-Morpholinobenzothioamide derivative by <sup>1</sup>H NMR Spectroscopy.**

The structure of N-morpholinobenzothioamide was confirmed by <sup>1</sup>H NMR spectroscopy, which showed characteristic signals corresponding to aromatic protons in the  $\delta$  7.0–8.0 ppm region. The morpholine ring protons appeared as distinct multiplets in the  $\delta$  2.5–4.0 ppm range, while the thioamide (–NH) proton was observed as a downfield singlet due to hydrogen bonding. These spectral features are consistent with the proposed molecular structure.

**3-a Molecule-<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.75 (d,  $J \approx 8.4$  Hz, 2H), 7.32 (d,  $J \approx 8.4$  Hz, 2H), 3.98 (t,  $J \approx 4.8$  Hz, 4H), 3.72 (t,  $J \approx 4.8$  Hz, 4H).

**3-b Molecule-<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.62 (d,  $J = 8.4$  Hz, 2H), 7.28 (d,  $J = 8.4$  Hz, 2H), 3.78–3.72 (m, 4H), 3.62–3.55 (m, 4H).

**3-c Molecule-<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.78 (d,  $J = 8.4$  Hz, 2H), 7.34 (d,  $J = 8.4$  Hz, 2H), 3.98 (t,  $J = 4.8$  Hz, 4H), 3.72 (t,  $J = 4.8$  Hz, 4H).

**4. Conclusion and Discussion:**

In the present study, a green and sustainable approach was successfully developed for the synthesis of CaO nanocatalysts using *Musa paradisiaca* leaf extract. The biogenic method provided an eco-friendly alternative to conventional synthesis routes, yielding highly crystalline CaO nanoparticles with nanoscale dimensions (~80–100 nm), as confirmed by XRD, SEM, and EDAX analyses. The observed porous and agglomerated morphology contributed to enhanced surface area and catalytic activity. The synthesized CaO nanocatalyst demonstrated excellent efficiency in the one-pot synthesis of N-morpholinobenzothioamide under mild conditions, affording high yields and eliminating the need for harsh reagents or complex purification techniques. The catalytic performance can be attributed to the basic surface sites and high reactivity of the nanomaterial. Furthermore, the catalyst exhibited good stability and reusability, highlighting its practical applicability. Overall, this work establishes a simple, cost-effective, and environmentally benign strategy that integrates green nanotechnology with organic synthesis, offering a promising pathway for sustainable catalytic processes and future applications in medicinal and synthetic chemistry.

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