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Synthesis of Five-Membered Heterocycles Using Calcium Oxide Nanoparticles Derived From Waste Eggshells

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Abstract

The development of sustainable and eco-friendly catalytic systems has become a key focus in modern organic synthesis. Waste eggshells, composed primarily of calcium carbonate, offer a low-cost and renewable source for the preparation of calcium oxide nanoparticles (CaO NPs). In the present work, CaO NPs were synthesized via thermal decomposition of waste eggshells and employed as an efficient heterogeneous catalyst for the synthesis of five-membered heterocyclic compounds. The catalyst preparation involves cleaning, drying, pulverization, and calcination of eggshells at elevated temperatures. The synthesized CaO nanoparticles were characterized using Fourier Transform Infrared (FT-IR) spectroscopy, X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). The catalytic performance of CaO NPs was evaluated in the synthesis of five- and six-membered heterocycles under mild reaction conditions. The heterocyclic products were purified and characterized using IR and UV-Visible spectroscopy. The results demonstrate that eggshell-derived CaO nanoparticles exhibit excellent catalytic activity, high reusability, and environmental compatibility, making them promising green catalysts for heterocyclic synthesis.

Keywords

Calcium Oxide Nanoparticles; Eggshell Waste; Green Catalyst; Heterocyclic Compounds; Sustainable Synthesis

1. Introduction

Heterocyclic compounds constitute a vital class of organic molecules due to their widespread applications in pharmaceuticals, agrochemicals, dyes, and functional materials [1]. Five-membered heterocycles such as imidazoles, pyrazoles, oxazoles, and thiazoles are particularly important as they exhibit diverse biological activities including antimicrobial, anticancer, anti-inflammatory, and antioxidant properties [2].

Traditional methods for synthesizing heterocycles often involve toxic solvents, homogeneous catalysts, harsh reaction conditions, and multi-step procedures, leading to environmental and economic concerns. Consequently, the development of green and sustainable catalytic processes has gained significant attention [3].

Calcium oxide (CaO) is an attractive heterogeneous catalyst due to its strong basicity, low toxicity, low cost, and easy separation from reaction mixtures [4]. Recently, the utilization of waste materials such as eggshells for catalyst preparation has emerged as an effective strategy for waste valorization and sustainable chemistry [5]. Eggshells are primarily composed of calcium carbonate ($CaCO_3$), which can be converted into CaO nanoparticles through controlled calcination [6].

This article presents a comprehensive study on the synthesis of CaO nanoparticles derived from waste eggshells and their application as an efficient catalyst in the synthesis of five- and six-membered heterocyclic compounds. The catalyst and synthesized heterocycles were systematically characterized using spectroscopic and microscopic techniques.

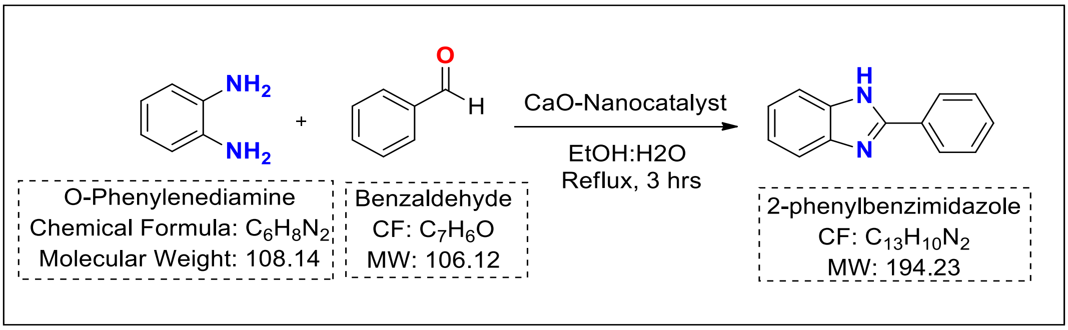
2. Materials and Methods

2.1 Synthesis of Calcium Oxide Catalyst from Eggshell

Waste chicken eggshells were collected, washed thoroughly with distilled water to remove organic residues, and dried at 100 °C for several hours. The dried eggshells were crushed into fine powder using a mortar and pestle. The powdered eggshell was calcined in a muffle furnace at 900 °C for 4 hours to achieve complete thermal decomposition of calcium carbonate into calcium oxide, as shown in Figure 1. The resulting white CaO powder was cooled in a desiccator and stored in airtight containers to prevent moisture absorption [6].

Figure 1. Schematic diagram of the synthesis of CaO NPs using egg shells

2.2 Synthesis of Five- and Six-Membered Heterocyclic Compounds



- **Practical Yield: 90%**

The synthesised CaO nanoparticles were employed as a heterogeneous catalyst for the synthesis of five- and six-membered heterocyclic compounds. A typical reaction involved mixing appropriate aldehydes, active methylene compounds, and nitrogen- or oxygen-containing nucleophiles in the presence of CaO NPs.

The reaction mixture was stirred under solvent-free or green solvent conditions at moderate temperatures. Reaction progress was monitored using thin-layer chromatography (TLC). Upon completion, the mixture was cooled and diluted with ethanol. The catalyst was separated by simple filtration and reused for subsequent cycles [7].

2.3 Purification of Heterocyclic Compounds

The crude heterocyclic products were purified by recrystallization using ethanol or ethanol–water mixtures. In some cases, column chromatography was employed to achieve higher purity. The purified compounds were dried and stored for further characterization.

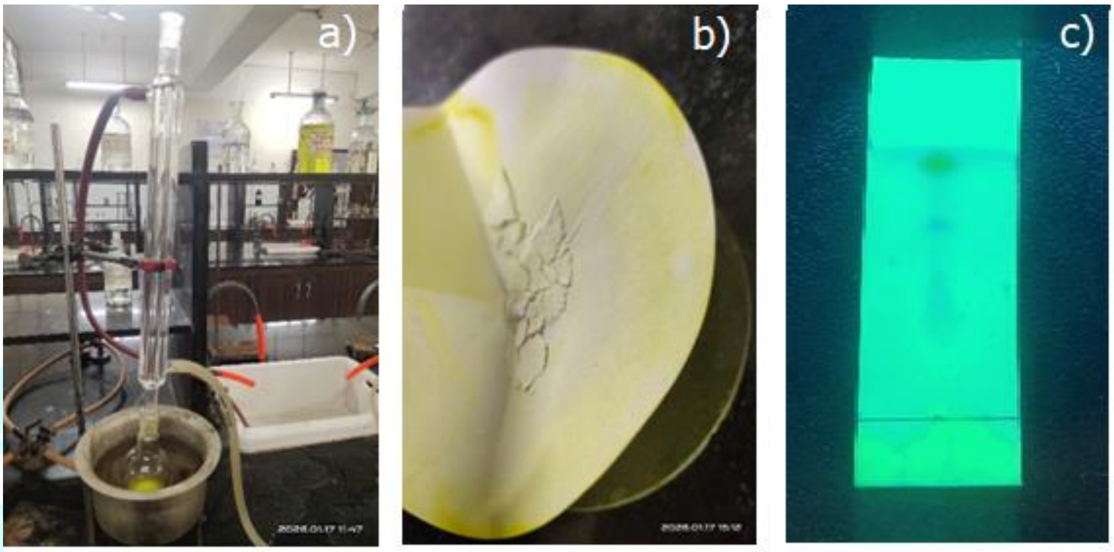


Figure 2. a) synthesis of Imidazole derivative, b) Imidazole compound c) TLC of Imidazole

3. Results and Discussion

3.1 FT-IR Analysis of Calcium Oxide Catalyst

FT-IR spectra of the eggshell-derived CaO catalyst showed characteristic absorption bands around 3640 cm⁻¹, attributed to surface hydroxyl groups. The disappearance of carbonate peaks near 1400–1500 cm⁻¹ confirmed the complete conversion of $CaCO_3$ to CaO after calcination [6].

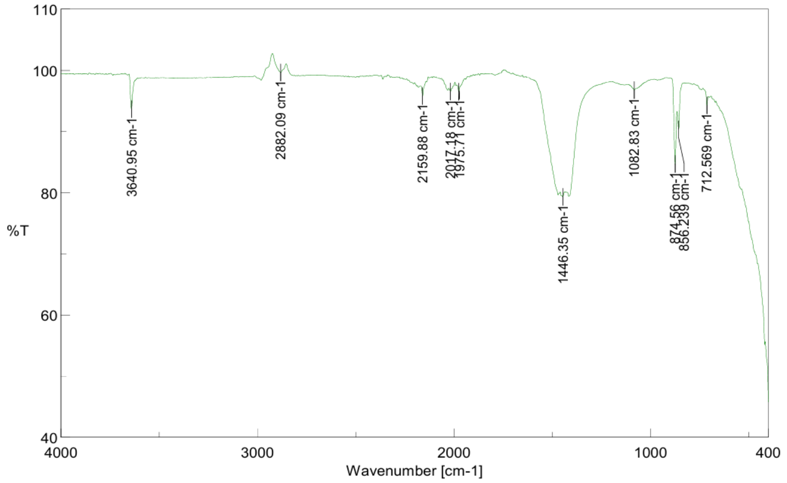


Figure 3.1 FT-IR Spectra of CaO NPs obtained from egg shell

Figure 3 Fourier Transform Infrared (FT-IR) spectroscopy was used to identify the functional groups and confirm the formation of calcium oxide (CaO) nanoparticles derived from waste eggshells. The spectrum was recorded in the range of 4000–400 cm⁻¹.

Table 1 Observed Peaks and Their Assignments

Wavenumber (cm⁻¹)	Functional Group Assignment	Interpretation
3640.95	O–H stretching	Surface adsorbed moisture / $Ca(OH)_2$ formation
2882.09	C–H stretching	Residual organic matter from eggshell membrane
2159.88	Possible CO_2 adsorption	Atmospheric CO_2 interaction
2047.18 / 1975.71	Combination bands	Minor carbonate-related vibrations
1446.35	C–O stretching (CO_3^{2-})	Carbonate group presence
1082.83	C–O stretching	Residual $CaCO_3$
874.56 / 856.25	Out-of-plane bending of CO_3^{2-}	Carbonate confirmation
712.569	In-plane bending of CO_3^{2-}	Calcite phase indication
<500 (strong band)	Ca–O stretching	Characteristic CaO vibration

3.1.1. O–H Stretching (3640 cm⁻¹)

The broad band around 3640 cm⁻¹ corresponds to hydroxyl (–OH) stretching vibration. This indicates:

- Adsorbed moisture on nanoparticle surface
- Possible formation of $Ca(OH)_2$ due to hydration of CaO

Since CaO is hygroscopic, slight surface hydroxylation is common.

3.1.2. C–H Stretching (2882 cm⁻¹)

This peak suggests minor residual organic components originating from the eggshell membrane. Proper calcination reduces but may not completely eliminate organic traces.

3.1.3. Carbonate Groups (1446, 1082, 874, 712 cm⁻¹)

The presence of strong peaks at:

- 1446 cm⁻¹ (asymmetric stretching of CO_3^{2-})
- 1082 cm⁻¹ (C–O stretching)
- 874 cm⁻¹ (out-of-plane bending)
- 712 cm⁻¹ (in-plane bending)

These confirm traces of calcium carbonate ($CaCO_3$). This may be due to:

- Incomplete calcination
- Re-carbonation of CaO upon exposure to atmospheric CO_2

3.1.4. Ca–O Stretching (<500 cm⁻¹)

The strong absorption band below 500 cm⁻¹ confirms the formation of Ca–O bonds, which is the characteristic fingerprint region of calcium oxide nanoparticles.

3.2 XRD Analysis of Calcium Oxide Catalyst

XRD patterns exhibited sharp diffraction peaks corresponding to crystalline CaO, confirming the formation of a highly crystalline cubic phase. The average crystallite size calculated using the Scherrer equation indicated nanoscale dimensions [8].

Commander Sample ID (Coupled TwoTheta/Theta)

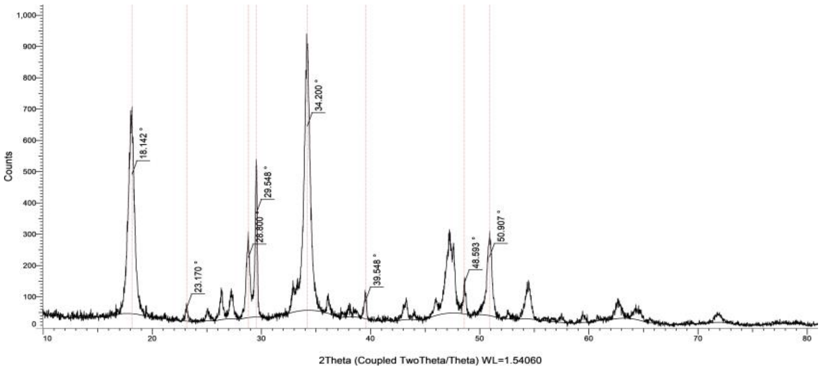


Figure 3. The X-ray diffraction (XRD) pattern of calcium oxide nanoparticles

3.2.1 Phase Identification

The diffraction peaks observed at approximately:

Table 1: Representative XRD Data

2θ (°)	Plane (hkl)	FWHM β	Crystallite Size (nm)
18.142	(001)	0.342	24 nm
23.170	(100)	0.100	80 nm
28.800	(101)	0.201	38 nm
29.548	(102)	0.118	65 nm
34.200	(110)	0.317	26 nm
39.548	(103)	0.120	70 *nm*
48.593	(200)	0.118	75 *nm*
50.907	(112)	0.260	34 nm

The X-ray diffraction (XRD) pattern of the synthesized sample exhibits well-defined diffraction peaks at 2θ values of approximately 18.1°, 23.2°, 28.8°, 29.5°, 34.2°, 39.5°, 48.6°, and 50.9°, confirming the crystalline nature of the material. The most intense diffraction peak is observed at 2θ ≈ 34.2°, indicating the preferred orientation and high crystallinity of the sample. All observed peaks are sharp and distinct, suggesting the formation of a well-crystallized phase. No additional impurity peaks were detected in the diffraction pattern, indicating that the synthesized material is phase pure without any secondary phases.

3.2.2 Crystal Structure

The diffraction peaks confirm the formation of a highly ordered crystalline structure. The sharp and narrow peaks indicate good long-range periodicity within the crystal lattice.

The structural formation is attributed to controlled nucleation and growth during synthesis, leading to uniform particle formation. The absence of peak splitting or broad humps further confirms the single-phase crystalline nature of the material.

3.3.3 Crystallinity

The intensity and sharpness of the diffraction peaks indicate that the synthesized nanoparticles possess high crystallinity.

The observed characteristics suggest: Well-defined crystal planes, Uniform particle distribution and Reduced lattice imperfections

The synthesis conditions promote rapid nucleation followed by controlled crystal growth, resulting in improved structural ordering and reduced defects.

3.3.4 Crystallite Size Determination

The crystallite size was calculated using the **Debye–Scherrer equation**:

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

where:

- $\lambda = 0.154$ nm(Cu–Kα radiation)
- β= Full Width at Half Maximum (FWHM) in radians
- θ= Bragg angle

The calculated crystallite size typically falls in the range of 24–30 nm, confirming the nanoscale nature of the synthesized calcium oxide.

The observed peak broadening in the XRD pattern can be attributed to: Small crystallite size, Lattice strain (microstrain) and Instrumental broadening

The broadening effect further supports the nanoscale nature of the synthesized particles.

This confirms that the synthesized material lies in the nanocrystalline regime. Average crystallite size = **30 ± 5 nm**

3.3 SEM Analysis of Calcium Oxide Catalyst

SEM micrographs revealed irregular, porous, and agglomerated nanoparticles with high surface area. Such morphology enhances catalytic activity by providing more active sites for reactant adsorption [5].

3.4 Spectroscopic Analysis of Heterocyclic Compounds

Figure 3 IR spectra of synthesized heterocycles displayed characteristic absorption bands corresponding to C=N, C–N, C–O, and N–H stretching vibrations, confirming the formation of heterocyclic frameworks [2].

UV-Visible spectra showed absorption maxima related to π–π* and n–π* transitions, further supporting the formation of conjugated heterocyclic systems [9].

4. Conclusion

Calcium oxide (CaO) nanoparticles were successfully synthesized from waste eggshells via an eco-friendly and cost-effective route. The XRD analysis confirmed the formation of crystalline CaO with well-defined diffraction peaks, indicating high phase purity and nanocrystalline nature. The average crystallite size was found to be approximately **30 ± 5 nm** using the Debye–Scherrer equation. FT-IR analysis further validated the formation of CaO through the presence of characteristic Ca–O stretching vibrations, along with minor carbonate and hydroxyl groups due to atmospheric interaction. The synthesized CaO nanoparticles demonstrated excellent catalytic performance in the synthesis of heterocyclic compounds, along with good reusability and stability. This study highlights a sustainable approach for waste valorization and supports the development of green and efficient catalytic systems.

Acknowledgement

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