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Synthesis and Characterization of Calcium Peroxide from Eggshell Waste as a Citric Acid Potential Adsorbent Material

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Abstract. This study presents a sustainable approach for synthesizing calcium peroxide (CaO_2) nanoparticles from biogenic eggshell waste as a potential adsorbent for wastewater treatment. Increasing water pollution caused by industrial and domestic activities has driven the demand for low-cost and environmentally friendly adsorbents. In this study, eggshells from various poultry sources were calcined at 900°C to produce CaO, followed by hydration and chemical precipitation using H_2O_2 to synthesize CaO_2. The resulting materials were dried at different temperatures (60–100°C) to evaluate the effect of thermal treatment on structural properties and adsorption performance. X-ray diffraction (XRD) analysis confirmed the formation of CaO_2 with nanoscale crystallite sizes ranging from 15.3 to 28.85 nm. Brunauer–Emmett–Teller (BET) analysis showed specific surface areas between 10.18 and 19.67 m²/g. Adsorption experiments using acetic acid revealed that performance was significantly influenced by precursor type and drying temperature. The highest adsorption capacity (347.54 mg/g) and removal efficiency (34.75%) were achieved using CaO_2 derived from free-range chicken eggshells at 80°C. This enhanced performance is attributed to the natural biogenic structure, mineral composition, and pore distribution. Overall, eggshell waste is a promising, sustainable, and cost-effective precursor for producing high-performance CaO_2 nanoparticles for wastewater treatment.

Introduction

The scarcity of clean water has become an increasingly critical global environmental challenge, particularly in urban areas and industrial regions with intensive activities. In Indonesia, surface water such as rivers, lakes, and reservoirs remains the main source of raw water for various sectors, including domestic, industrial, and agricultural uses. In the agricultural sector, clean water is essential for irrigation of food crops, horticulture, and livestock, which support national food security. However, the quality of surface water continues to decline due to the high pollution load from industrial and domestic wastewater. According to the *Indonesia Central Bureau of Statistics* (BPS, 2022) , 60% of rivers in Indonesia are polluted by domestic and industrial waste, making surface water unsafe for direct use without treatment.

This condition exacerbates water scarcity, especially in regions with limited groundwater reserves, particularly during prolonged dry seasons.

Indonesia, as an agrarian and maritime country, has great potential in providing natural biomineral raw materials for inorganic material synthesis. One abundant domestic waste is eggshells, which are often discarded without proper processing. On a global scale, eggshell waste reaches approximately 2.3 million tons per year and mainly consists of calcium carbonate ($CaCO_3$) at around 95%, offering a promising solution. The utilization of this waste to produce calcium precursors such as CaO through calcination not only reduces environmental and economic problems caused by landfill disposal but also provides a sustainable alternative to commercial calcium sources in the synthesis of adsorbents such as

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CaO_2 (Ahmed et al., 2021).

Adsorption techniques offer a potential solution for the separation and recovery of pollutants. In this context, nanoparticles, especially nanocalcium peroxide ($nCaO_2$), have emerged as highly promising adsorbents. This material has advantages such as high specific surface area, large adsorption capacity, relatively simple and economical synthesis methods, and environmentally friendly properties due to controlled oxygen release (Amerhaider Nuar et al., 2022). In addition to being known for pollutant remediation through its oxidative properties, CaO_2 has also been proven effective as an adsorbent for various contaminants, including heavy metals (Olyaie et al., 2012), as well as for the recovery of acrylic acid from aqueous solutions (De, 2018), and maleic acid with a very high adsorption capacity of up to 597 $mg{\cdot}g^{-1}$, outperforming commercial adsorbents such as Amberlite IRA 617 resin (345 $mg{\cdot}g^{-1}$), activated carbon (130 $mg{\cdot}g^{-1}$), and layered double hydroxide (1 $mg{\cdot}g^{-1}$) for carboxylic acid adsorption (Dedecan et al., 2022).

The synthesis of CaO_2 is generally carried out through chemical precipitation, where a calcium source such as $CaCl_2$ or $Ca(OH)_2$ is reacted with H_2O_2 under controlled pH conditions (Dedecan et al., 2022). $Ca(OH)_2$ derived from eggshell calcination can serve as an environmentally friendly precursor for this reaction. Synthesis parameters, especially the drying temperature of the CaO_2 precipitate, are crucial because they affect the morphological structure, porosity, and specific surface area of the final material—factors that directly determine adsorption performance (Heo et al., 2024). Therefore, characterization of morphology and surface area is essential to understand the structure–performance relationship (De, 2018).

Based on the great potential of nanocalcium peroxide (CaO_2) as an effective adsorbent, this study aims to address the existing research gap. By utilizing eggshell waste as a sustainable calcium source, this research focuses on developing an environmentally friendly method for synthesizing CaO_2 nanoparticles using $Ca(OH)_2$ derived from eggshell waste and evaluating its performance as an adsorbent for carboxylic acid compounds. The variables studied include the effect of drying temperature during CaO_2 synthesis on morphological structure, surface area, and adsorption capacity. Thus, this study is expected to provide a significant contribution to the development of innovative waste-based adsorbents for water purification and resource recovery applications.

Experimental

Material and Methods

The main raw materials used in this study include eggshells from chicken, duck, and quail, which serve as natural calcium sources due to their high calcium carbonate content. The chemicals used in the synthesis process include hydrogen peroxide (H_2O_2) with concentrations ranging from 30% to 50% as an oxygen donor, and demineralized water for hydration and washing processes.

Procedures

Preparation of Calcium Precursors

The preparation process began with cleaning the eggshells by soaking them in water to remove impurities. After cleaning, the eggshells were dried under sunlight. A total of 20 grams of dried eggshells were then calcined in a furnace at 900°C for 1 hour. This treatment was used to decompose calcium carbonate ($CaCO_3$) into calcium oxide (CaO) and carbon dioxide (CO_2) according to the following reaction:

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

The resulting CaO exhibited high porosity and served as the precursor material for the next synthesis stage.

Synthesis of Calcium Peroxide (CaO_2) Nanoparticles

The synthesis was carried out using a chemical precipitation method, starting with the hydration of CaO in 30 mL of demineralized water. The mixture was stirred at 300 rpm for 15 minutes to produce $Ca(OH)_2$. The precipitation stage involved the gradual addition of 30 mL of 30% H_2O_2 into the solution over 30 minutes until a white CaO_2 precipitate was formed.

The mixture was then centrifuged at 3500 rpm for 5 minutes to separate the solid product from the liquid phase. After thorough washing to remove residual reagents, the precipitate was dried for 2 hours at various temperatures to study the effect of thermal conditions on material structure.

$$CaO_{(s)} + H_2O_{(l)} \longrightarrow Ca(OH)_{2\,(aq)} \quad (2)$$

$$Ca(OH)_{2\,(aq)} + H_2O_{2\,(l)} \longrightarrow CaO_{2\,(s)} + 2H_2O_{(l)} \quad (3)$$

Adsorption Test

The synthesis was carried out using a chemical precipitation method, starting with the hydration of CaO in 30 mL of demineralized water. The mixture was stirred at 300 rpm for 15 minutes to produce $Ca(OH)_2$. The precipitation stage involved the gradual addition of 30 mL of 30% H_2O_2 into the solution over 30 minutes until a white CaO_2 precipitate was formed.

Characterization

The synthesized CaO_2 adsorbent was characterized to determine its physical and chemical properties. Crystal structure analysis was performed using X-ray diffraction (XRD) to identify crystalline phases. Additionally, the specific surface area was measured using the Brunauer–Emmett–Teller (BET) method.

Result and Discussion

Synthesis of Calcium Peroxide from Eggshells

The synthesis of calcium peroxide (CaO_2) in this study was conducted through a two-step process, namely thermal decomposition (calcination) and chemical precipitation using hydrogen peroxide (H_2O_2). Eggshells were selected as a biogenic precursor due to their high calcium carbonate ($CaCO_3$) content, ranging from 94% to 98% (Ahmed et al., 2021).

Initially, the eggshells (duck, free-range chicken, broiler chicken, and quail) were calcined at 900°C for 1 hour to convert $CaCO_3$ into calcium oxide (CaO), as represented in the following reaction:

$$CaCO_{3\,(s)} \longrightarrow CaO_{(s)} + CO_{2\,(g)} \quad (4)$$

This temperature was selected based on previous studies indicating complete transformation of calcite into highly reactive CaO with an open pore structure (Zhuang et al., 2025) The CaO was subsequently hydrated into $Ca(OH)_2$ before reacting with H_2O_2 to form CaO_2.

Based in **Table 1** Yield analysis revealed a strong dependence on drying temperature and precursor type. At lower temperatures (60°C), most samples exhibited yields exceeding 100%, which can be attributed to the formation of hydrated CaO_2 or residual moisture trapped within the porous structure (Dedecan et al., 2022). However, higher temperatures (80–100°C) improved stability, particularly for natural eggshell-derived samples.

Interestingly, pure CaO showed a significant decrease in yield, whereas eggshell-derived materials demonstrated higher thermal stability. This suggests that trace elements such as magnesium and strontium present in natural eggshells enhance structural stability (Gladysheva et al., 2016; Kalaycı et al., 2025).

Refers to **Figure 1**. X-ray diffraction (XRD) analysis confirmed the successful phase transformation from $CaCO_3$ to CaO and subsequently to CaO_2. The CaO phase exhibited cubic crystal structure, while CaO_2 showed a tetragonal structure consistent with JCPDS No. 00-003-0865.

The broiler chicken eggshell sample demonstrated the highest crystallinity and phase purity, consistent with findings by (Heo et al., 2024). However, residual $CaCO_3$ peaks observed at $2\theta \approx 29.3°$ indicated partial recarbonation due to exposure to atmospheric CO_2.

Crystallite size was calculated using the Debye-Scherrer in **Equation 4**, According to **Table 2.** nanoscale dimensions ranging from 15.3 nm to 28.85 nm. These values are significantly smaller than those reported for chemically synthesized CaO_2 (34.77 nm) (Dedecan et al., 2022).

$$D= \frac{k\lambda}{\beta \cos \theta} \quad (5)$$

The smaller crystallite size in biogenic samples is attributed to the presence of organic matrix proteins that act as grain growth inhibitors during calcination. This results in higher defect density and increased reactive surface sites.

Brunauer–Emmett–Teller (BET) analysis showed that the specific surface area ranged from 10.18 to 19.67 m^2/g. The result obtained is match according to research comply by (Dedecan et al., 2022). The highest surface area was observed in the free-range chicken eggshell sample.

Despite the nanoscale crystallite size, the relatively low surface area is attributed to particle agglomeration caused by high-temperature calcination (900°C). Additionally, incomplete degassing at 80°C may have limited nitrogen adsorption due to residual moisture.

Based on **Figure 2.** The adsorption capacity (q_e) results demonstrated that all eggshell-derived CaO_2 samples outperformed pure CaO. The highest adsorption capacity was observed in the free-range chicken eggshell sample, reaching 347.54 mg/g at 80°C.

This superior performance is linked to the natural porous structure of eggshells, which contain 7,000–20,000 micropores (Mukadam, 2020). Additionally, the mineral composition and protein matrix provides a reactive calcium-rich surface and releases Ca^{2+} during decomposition, while associated protein or polymer matrices can modulate ion accessibility and influence transient adsorption or complexation of acetate species (Ma et al., 2024).

Temperature variation indicated that adsorption increased from 60°C to 80°C, suggesting a chemisorption mechanism. However, at temperatures above 80°C, adsorption decreased due to desorption effects, consistent with the exothermic nature of adsorption (Madan et al., 2016).

Removal efficiency analysis showed in **Figure 3.** that eggshell-based adsorbents consistently outperformed pure CaO. The highest efficiency (34.75%) was achieved by free-range chicken eggshell CaO_2 at 80°C, compared to only 15.75% for pure CaO.

Differences among eggshell types are attributed to variations in pore structure and mineral composition. Free-range chicken eggshells exhibited more uniform pore distribution, enhancing adsorption efficiency. In contrast, duck eggshells, although more porous, likely have larger pore sizes that reduce interaction strength with adsorbate molecules (Praipipat et al., 2023).

The efficiency trend further supports a chemisorption mechanism, where thermal energy facilitates bond formation between Ca^{2+} ions and acetate ions. At higher temperatures, efficiency declines due to desorption and reduced stability of peroxide bonds (Dedecan et al., 2022).

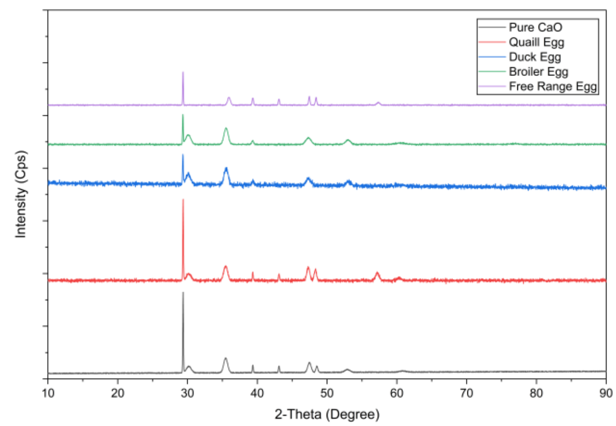

Figure 1. XRD Pattern For Each Egsheell Sample.

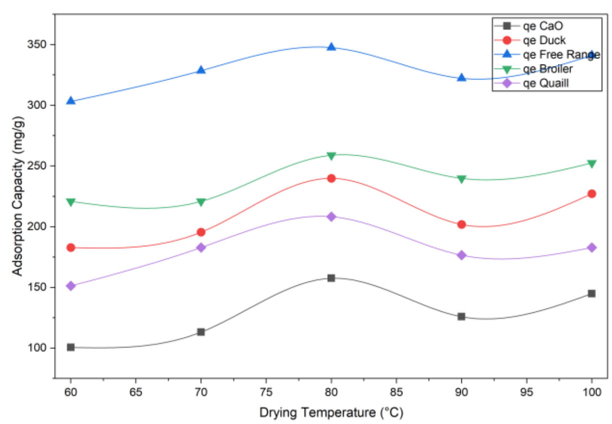

Figure 2. Relationship Between Adsorption Capacity and Drying Temperature in Each Sample

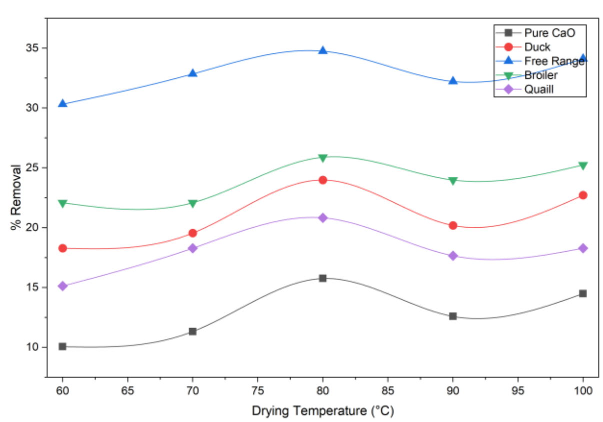

Figure 3. Relationship Between % Removal and Drying Temperature in Each Sample.

Table 1. Products Yield (%) in Each Sample.

Sampel	%Yield				
	60°C	70°C	80°C	90°C	100°C
Pure CaO	119,979	79,087	57,995	75,676	72,690
Duck Egg	106,534	91,581	103,202	99,282	96,112
Broiler Chiken Egg	102,572	100,390	103,973	71,146	92,968
Free range Chiken Egg	106,167	84,853	98,390	96,384	98,075
Quail Egg	98,4939	87,468	93,553	96,650	98,483

Conclusion

This study successfully demonstrates the synthesis of calcium peroxide (CaO_2) nanoparticles from biogenic eggshell waste through a combined calcination–precipitation method. The results confirm that eggshell-derived CaO_2 exhibits superior physicochemical properties compared to pure CaO, including smaller crystallite size, enhanced structural stability, and improved adsorption performance.

Among the tested precursors, free-range chicken eggshells showed the highest adsorption capacity (347.54 mg/g) and removal efficiency (34.75%) at the optimal temperature of 80°C. These findings highlight the critical role of natural biogenic structures, including mineral composition and pore distribution, in enhancing the functional performance of the material.

The study also reveals that adsorption behavior is strongly influenced by temperature, with optimal performance occurring at moderate thermal conditions due to a chemisorption-dominated mechanism. At higher temperatures, performance decreases due to desorption and reduced peroxide stability.

Overall, this research demonstrates that eggshell waste is a highly promising, sustainable, and low-cost precursor for the production of functional nanomaterials. The findings are particularly relevant for environmental applications, especially in wastewater treatment, where efficient and eco-friendly adsorbents are urgently needed.

Conflict of Interest

The authors declare that there is no conflict of interest.

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