

# Removal of Fe(III) from Wastewater Using Calcium Oxide Prepared from Duck Eggshells

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**Abstract**—The adsorption of Fe<sup>3+</sup> on CaO-standard and CaO prepared from duck eggshells was investigated. Duck eggshell CaO was prepared by calcination at 900 °C for 3 h and characterized using X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Fourier-Transform Infrared Spectroscopy (FT-IR), FT-Raman and Brunauer-Emmett-Teller (BET) techniques. The adsorption experiments were conducted in a batch process, with the parameters affecting adsorption capacity, i.e., contact time and initial concentration, were studied. Characterization methods revealed that duck eggshell CaO exhibited similar properties to CaO-standard but had a slightly lower surface area. Equilibrium adsorption was achieved within 60 min, and the adsorption behavior of Fe<sup>3+</sup> on both CaO materials was better described by the Langmuir isotherm model. The maximum adsorption capacity of duck eggshell CaO was approximately 89% of that of CaO-standard, demonstrating its potential as an alternative and effective adsorbent for Fe<sup>3+</sup> removal.

**Keywords**—adsorption, calcium oxide, duck eggshell, wastewater

## I. INTRODUCTION

Wastewater pollution is a significant global issue, particularly when it involves contamination with heavy metal ions. The consumption of water contaminated with heavy metals poses severe health risks, potentially leading to conditions such as hemochromatosis, liver and kidney cancers, and cardiovascular diseases [1–3]. Among the various heavy metals, iron is considered one of the most hazardous due to its detrimental effects on aquatic ecosystems and associated health risks [4–5]. Iron contamination commonly occurs in groundwater and is associated with industrial processes such as spent pickling, foundries, smelting, chemical milling, and wire drawing. Additionally, diffuse sources including combustion byproducts, corroded piping materials, and other industrial activities contribute to its presence. Iron predominantly exists in its Fe<sup>2+</sup> and Fe<sup>3+</sup> forms, with Fe<sup>3+</sup> being particularly highly stable and persistent in the environment [6]. Consequently, the removal or reduction of iron concentrations in wastewater is crucial before its discharge into the environment.

Among various removal methods, adsorption has emerged as a particularly effective approach due to its low energy requirements, cost-efficiency, compact operational footprint, and the regenerability of adsorbents. In recent years, several studies have explored the use of agricultural wastes and natural materials as low-cost and abundant adsorbents for wastewater treatment. One such material is calcium oxide (CaO), which can be derived from natural sources such as

duck eggshells, chicken eggshells, and oyster shells through calcination at high temperatures (approximately 700–900 °C). Previous studies have demonstrated the effectiveness of CaO in adsorbing heavy metals such as Pb<sup>2+</sup> [3], Cu<sup>2+</sup> [7], and Cd<sup>2+</sup> [8]. However, information regarding the removal of Fe<sup>3+</sup> using CaO derived from natural materials is still limited.

In this study, the adsorption of Fe<sup>3+</sup> using CaO prepared from duck eggshells (CaO-DE) was investigated and compared with the adsorption performance of commercially available CaO. Parameters such as initial Fe<sup>3+</sup> concentration and contact time were evaluated to understand their effects on the adsorption process. The findings of this study aim to promote the use of CaO-DE as a cost-effective and sustainable adsorbent for the pretreatment of heavy metal-contaminated wastewater.

## II. MATERIALS AND METHODS

### A. Materials

Duck eggshells were obtained from a local fresh market in Bangkok, Thailand. CaO standard (purity 90%) was purchased from LobaChemie (India) and FeCl<sub>3</sub>·6H<sub>2</sub>O (purity 99%) was supplied by QRec (New Zealand).

### B. Preparation of Calcium Oxide from Duck Eggshells

The method for preparation of CaO-DE was adjusted from the procedure presented by Razali *et al.* [9]. Briefly, 10 g of duck eggshell was washed by de-ionized water and dried in an oven (OF-01E, JEIO TECH) at 100 °C for 24 h. It was then grounded to fine particles and burned in a furnace (Thermo Scientific FB1415M, TEquipment) at 900 °C for 3 h, to calcine calcium carbonate (CaCO<sub>3</sub>) to CaO. The CaO was then characterized by XRF (S8 Tiger, Bruker) to determine percent of composition, XRD (D8 Discover, Bruker) to determine phase and crystallographic parameters, FT-IR (INVENIO S, Bruker) and FT-Raman (XploRA PLUS, Horiba) to investigate functional groups on surface area and BET (Tristar 3000, Micromeritics Instrument Crop) to determine total surface area, pore volume, and pore diameters.

### C. Adsorption Experiments

Batch adsorption experiments were conducted to investigate the kinetics and equilibrium behavior of Fe<sup>3+</sup> ion adsorption onto the prepared adsorbent. A known amount of the adsorbent, approximately 0.25 g, was introduced into 50 mL of Fe<sup>3+</sup> solution with a known initial concentration. The mixture was agitated using an incubator rotary shaker (ZHWHY-100B, Zhicheng) at 200 rpm and a temperature of

35 °C for predetermined contact times: 5, 10, 15, 20, 30, 45, 60, 90, 120, 180, and 240 min. At the end of each contact time, aliquots were withdrawn from the solution and the adsorbent was separated by centrifugation (LC-6S, Joanlab) at 5000 rpm for 5 min. The concentration of  $\text{Fe}^{3+}$  ions in the supernatant was measured using a UV-Visible spectrophotometer (T60 V, PG Instruments) at a wavelength of 520 nm. The amount of  $\text{Fe}^{3+}$  adsorbed at time  $t$  ( $q_t$ ) was calculated using Eq. (1):

$$q_t = \frac{(C_0 - C_t)V}{W} \quad (1)$$

where  $C_0$  and  $C_t$  are the initial concentration and the concentration of  $\text{Fe}^{3+}$  ions at time  $t$  (mg/L),  $V$  is the volume of solution (mL) and  $W$  is the weight of adsorbent (g).

To determine the equilibrium time, the initial concentration of  $\text{Fe}^{3+}$  ions was set at 200 mg/L. Samples were collected at the predetermined time intervals and  $C_t$  and  $q_t$  were calculated using the procedure outlined above. For adsorption isotherm studies, the initial concentration of  $\text{Fe}^{3+}$  ions was varied between 10 and 200 mg/L. The solutions were shaken under the same experimental conditions until equilibrium was achieved. The equilibrium concentration ( $C_e$ ) and the equilibrium adsorption capacity ( $q_e$ ) were determined using the same analytical procedures as described for kinetic studies.

### III. RESULTS AND DISCUSSION

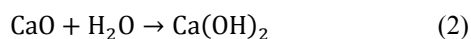
#### A. Characterization of CaO-DE and CaO Standard

XRF analysis was performed to determine the composition of the calcined duck eggshell. As summarized in Table 1, CaO is the primary component, with a high purity of approximately 98.1%, comparable to that of the commercial CaO standard. Trace amounts of other oxides, including  $\text{P}_2\text{O}_5$ , MgO, and  $\text{Na}_2\text{O}$ , were also detected, each contributing less than 1% to the overall composition.

Table 1. XRF Characterization of calcined duck eggshell

Component	Percent
CaO	98.1
$\text{P}_2\text{O}_5$	0.55
MgO	0.48
$\text{SO}_3$	0.37
$\text{Na}_2\text{O}$	0.31
Other	0.19

The XRD analysis was conducted to investigate the crystal structure and crystallinity of CaO standard and CaO-DE. As shown in Fig. 1, the XRD pattern of CaO-DE closely aligns with that of the CaO standard. Peaks observed at  $2\theta = 32^\circ$ ,  $37^\circ$ ,  $54^\circ$ ,  $67^\circ$ ,  $80^\circ$ , and  $89^\circ$  correspond to CaO, the main phase present in both samples. However, additional peaks at  $2\theta = 18^\circ$ ,  $29^\circ$ ,  $34^\circ$ ,  $47^\circ$ ,  $51^\circ$ ,  $63^\circ$ , and  $72^\circ$  indicate the presence of calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) in CaO-DE. Notably, the peaks at  $2\theta = 18^\circ$  and  $34^\circ$  are more prominent, suggesting a slightly higher  $\text{Ca}(\text{OH})_2$  content compared to the standard. This is likely due to the reaction of CaO with atmospheric moisture during preparation, as described in Eq. (2) [10].



FT-IR analysis was conducted to examine the surface functional groups present on the adsorbents, with the results shown in Fig. 2. The FT-IR spectrum of CaO-DE closely

resembles that of a standard CaO sample. A prominent peak observed at  $3641 \text{ cm}^{-1}$  corresponds to the hydroxyl functional group, indicating the presence of  $\text{Ca}(\text{OH})_2$  in the sample [11, 12]. This finding aligns well with the results obtained from XRD characterization. Additionally, peaks observed in the range of  $875\text{--}1460 \text{ cm}^{-1}$  are associated with carbonyl functional groups, likely due to a small residual amount of  $\text{CaCO}_3$  in the sample [13].

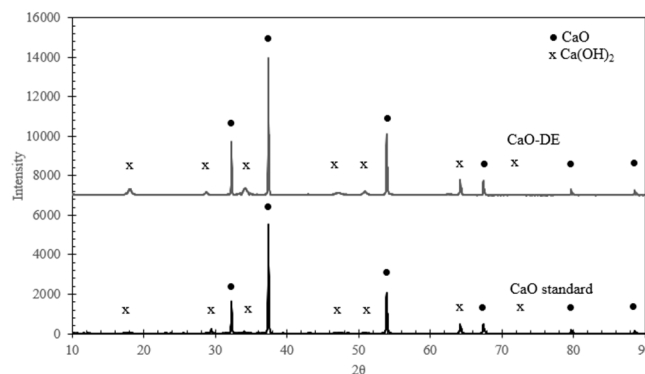


Fig. 1. XRD patterns of CaO standard and CaO-DE.

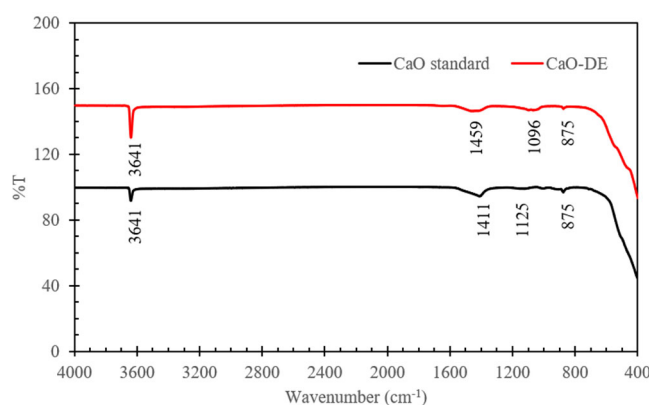


Fig. 2. FT-IR spectrum of CaO standard and CaO-DE.

The FT-Raman spectra, shown in Fig. 3, provide further confirmation of the presence of CaO. Three major peaks observed in the ranges of  $340\text{--}350 \text{ cm}^{-1}$ ,  $665\text{--}675 \text{ cm}^{-1}$ , and  $1070\text{--}1080 \text{ cm}^{-1}$  are attributed to the Ca–O bond, which is characteristic of CaO [14]. Overall, the FT-IR and FT-Raman results are consistent with those reported in previous studies [15–17], further supporting the successful synthesis of CaO-DE, with minor residual impurities such as  $\text{Ca}(\text{OH})_2$  and  $\text{CaCO}_3$ .

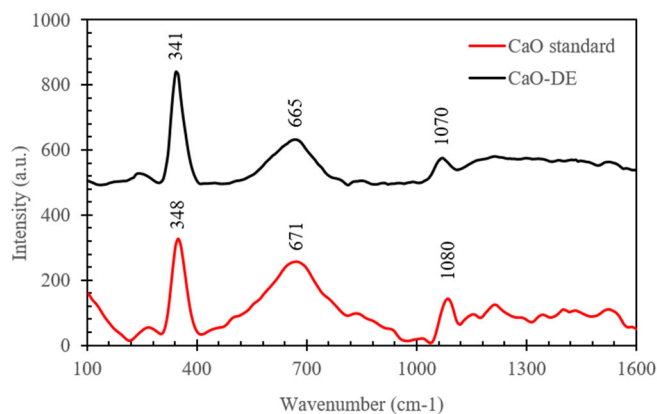


Fig. 3. FT-Raman spectrum of CaO standard and CaO-DE.

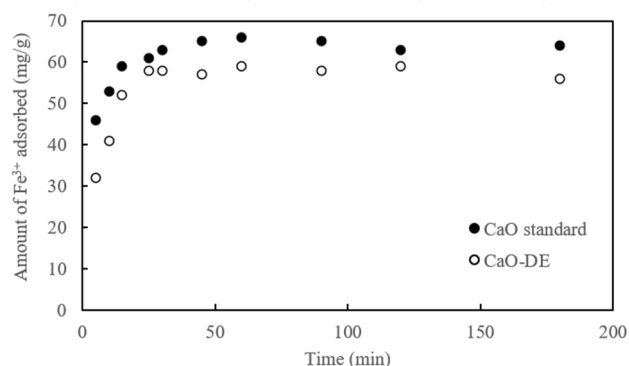
BET analysis was conducted to determine the total surface area, total pore volume, and average pore diameter of the CaO standard and CaO-DE, as shown in Table 2. The results indicate that the average pore diameter of CaO-DE is comparable to that of the CaO standard. However, the total surface area and total pore volume of CaO-DE are approximately 80% of those of the CaO standard. Despite these differences, the characteristics of CaO-DE align well with those of CaO synthesized from other materials reported in the literature [11, 15, 18].

Table 2. BET characterization of CaO standard and CaO-DE

Adsorbent	Total surface area (m <sup>2</sup> /g)	Total pore volume (cm <sup>3</sup> /g)	Average pore diameter (nm)
CaO-DE	8.09	0.0101	7.12
CaO standard	10.56	0.0126	7.36

### B. Adsorption Experiment Results

A preliminary test was conducted to determine the equilibrium time for the adsorption of Fe<sup>3+</sup> onto CaO standard and CaO-DE. The results, as presented in Fig. 4, indicate that the adsorption process exhibited a rapid initial phase for both adsorbents, with the amount of Fe<sup>3+</sup> adsorbed increasing sharply during the first 20 min. This phase can be attributed to the abundant availability of active sites on the adsorbents' surfaces at the beginning of the process. After 20 min, the adsorption rate slowed and equilibrium was achieved at approximately 60 min for both CaO standard and CaO-DE. To ensure complete adsorption during subsequent studies, a contact time of 120 min was selected, exceeding the observed equilibrium time.

Fig. 4. Amount of Fe<sup>3+</sup> adsorbed on CaO standard and CaO-DE at various times (initial concentration 400 ppm).

The experiments for the determination of the isotherm of Fe<sup>3+</sup> on CaO-DE and CaO standard were carried out using batch adsorption with initial Fe<sup>3+</sup> concentrations ranging from 10 to 500 mg/L. The results in Fig. 5 show that both CaO-DE and CaO standard exhibit similar trends. Specifically, the amount of Fe<sup>3+</sup> adsorbed increases linearly with the equilibrium concentration up to approximately 100 mg/L. Beyond this point, the equilibrium adsorption capacity tends to plateau, indicating that the adsorbent sites are approaching their maximum capacity.

The equilibrium adsorption capacity of Fe<sup>3+</sup> on both adsorbents was modeled using two isotherms, the Langmuir and Freundlich models. The corresponding equations are represented in Eqs. (3) and (4), respectively, while the parameters obtained from the experimental data and their correlation coefficients (R<sup>2</sup>) are summarized in Table 3.

$$q_e = \frac{q_{\max} K_L C_e}{1 + K_L C_e} \quad (3)$$

$$q_e = K_F C_e^{1/n} \quad (4)$$

where  $q_{\max}$  is the maximum adsorption capacity,  $K_L$  is the Langmuir isotherm constant, and  $K_F$  and  $n$  are the Freundlich isotherm constants.

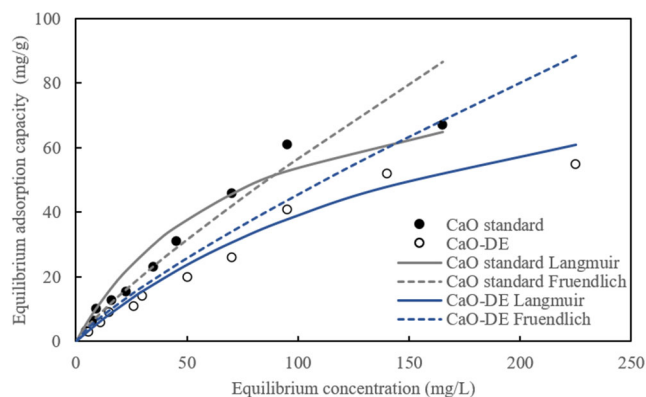
Fig. 5. Adsorption isotherms of Fe<sup>3+</sup> onto CaO-DE and CaO standard.

Table 3. Langmuir and Freundlich isotherm parameters

Model	Parameter	CaO standard	CaO-DE
Langmuir	$q_{\max}$ (mg/g)	91.41	80.89
	$K_L$ (L/mg)	0.0134	0.0155
	$R^2$	0.9705	0.9930
Freundlich	$K_F$ (mg/g)	1.16	1.05
	$n$	1.18	1.31
	$R^2$	0.9587	0.9664

A comparison of the two models for both adsorbents reveals that the Langmuir model better describes the adsorption behavior of Fe<sup>3+</sup> on both CaO-DE and CaO standard, as evidenced by the higher R<sup>2</sup> values. This indicates that the adsorption process follows a monolayer physical adsorption mechanism. These results align with reports on the adsorption of heavy metals on CaO in the literature [3, 8, 19, 20].

In terms of maximum adsorption capacity ( $q_{\max}$ ), CaO-DE demonstrates slightly lower efficiency compared to the CaO standard, achieving about 89% of the CaO standard's performance. The slight reduction in adsorption capacity can be attributed to the lower surface area of CaO-DE, as confirmed by BET characterization. Nevertheless, CaO-DE offers significant advantages: it is derived from duck eggshells, a food waste material, and its preparation process is environmentally friendly, requiring no chemical usage. To further enhance the adsorption efficiency of CaO-DE, additional experiments focusing on increasing its surface area are recommended.

## IV. CONCLUSION

In this study, CaO-DE for Fe<sup>3+</sup> adsorption has been successfully prepared. Characterization revealed that CaO-DE had similar properties to CaO standard but exhibited a relatively lower total surface area and total pore volume, at approximately 80% of those of CaO standard. The equilibrium time for Fe<sup>3+</sup> adsorption on both adsorbents was 60 min. The adsorption behavior of Fe<sup>3+</sup> was better described by the Langmuir model than the Freundlich model. The maximum adsorption capacity of Fe<sup>3+</sup> on CaO-DE is slightly lower than that of CaO standard. These results suggest that

while CaO-DE has slightly lower adsorption efficiency, it could be an alternative adsorbent for Fe<sup>3+</sup> removal.

#### CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

#### AUTHOR CONTRIBUTIONS

Wirada Artsamat and Arnancha Intawaree designed, conducted the experiments, and analyzed data regarding the adsorption of Fe<sup>3+</sup> on the adsorbents; Weerawat Clowutimon and Pornsawan Assawasaengrat characterized XRF, XRD, FT-IR, FT-Raman and BET of the adsorbents and organized the manuscript. All authors had approved the final version.

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