Faculty of Natural and Applied Sciences Journal of Applied and Physical Sciences
Print ISSN: 3026-9857
www.fnasjournals.com
Volume 2; Issue 4; December 2025; Page No. 50-57.
DOI: https://doi.org/10.63561/japs.v2i4.1030

Synthesis, Characterization, and Evaluation of Zeolite Materials for Cadmium (Cd^{2+}) Sequestration in Water Treatment

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Abstract
Cadmium (Cd^{2+}) is a toxic heavy metal often found in industrial wastewater, creating significant hazards for water systems and human health. In this study, we synthesized zeolite materials from low-cost precursors using hydrothermal processing. We characterized the materials using XRD, SEM, and BET surface analysis (though FTIR offered limited structural data). The resulting zeolites showed distinct crystalline frameworks, higher surface area, and plenty of cation-exchange sites. In batch tests, we achieved cadmium removal rates over 90% by optimizing pH and contact time. The data fit best with the Langmuir isotherm and pseudo-second-order kinetic models, pointing to monolayer chemisorption as the primary mechanism. Additionally, regeneration tests showed the material held up well after several cycles. These findings suggest that synthesized zeolites are a viable, cost-effective option for capturing cadmium in water treatment.

Key words: Zeolite synthesis; Cadmium removal; Water purification; Adsorption isotherms; Hydrothermal method

Introduction

Cadmium (Cd^{2+}) contamination in aquatic area is a serious concern globally because of its toxicity, persistence, and ability to bioaccumulate. Industrial activities such as electroplating, battery manufacturing, pigment production, mining, and fertilizer application are major contributors to cadmium release into water systems (Jaishankar et al., 2014; Nordberg et al., 2015). Even at trace concentrations, cadmium exposure can cause health problems including kidney dysfunction, skeletal damage, and carcinogenic effects (WHO, 2011). Its long-term presence in ecosystems highlights the urgent need for operative remediation policies.

Conventional treatment methods such as chemical precipitation, ion exchange, reverse osmosis, and membrane filtration have been widely applied for cadmium removal. These approaches often face limitations including high operational costs, reduced efficiency at low metal concentrations, and the generation of secondary pollutants such as sludge (Fu & Wang, 2011). Such drawbacks emphasize the need for cost-effective, sustainable, and efficient adsorbents capable of removing cadmium from contaminated water.

Zeolites, crystalline aluminosilicate materials with microporous frameworks, have emerged as promising candidates for heavy metal remediation. Their large surface area, strong cation-exchange capacity, and chemical stability enable effective adsorption of toxic metals (Wang & Peng, 2010). Natural zeolites such as clinoptilolite and mordenite, as well as synthetic variants including zeolite A, 10A, and 13X, have been investigated. Synthetic forms generally demonstrate superior adsorption performance. For example, synthetic zeolite 10A has shown higher cadmium uptake compared to natural clinoptilolite (Kozera-Sucharda et al., 2020), underscoring the advantages of engineered zeolitic materials. Recent research has also focused on synthesizing zeolites from industrial byproducts such as coal fly ash, offering dual environmental benefits by converting waste materials into low-cost adsorbents. Fly ash-derived zeolites have demonstrated high removal efficiencies for cadmium and other heavy metals, with adsorption mechanisms dominated by ion exchange and surface complexation (He et al., 2016; Sukchit et al., 2025). These findings shows the potential of waste-derived zeolites as sustainable alternatives for water purification.

Despite these advances, challenges remain. Most studies have been limited to laboratory-scale batch experiments, with fewer investigations into column studies or real wastewater applications (Ali & Dzombak, 1996; Babel & Kurniawan, 2003). Regeneration efficiency and adsorbent reusability also require further exploration to ensure economic feasibility. Addressing these gaps is essential for translating laboratory successes into practical water treatment solutions.
The purpose of this research is to synthesize zeolite materials and evaluate their effectiveness in sequestering cadmium (Cd^{2+}) from contaminated water. The study specifically involves preparing zeolites from selected precursor materials, characterizing their structural and surface properties through advanced analytical techniques, and examining their adsorption performance under different operational conditions. The findings are expected to provide valuable insights into the potential of zeolite-based adsorbents as sustainable materials for purification of water and the remediation of environmental. (Querol, 2001)

Cadmium Contamination in Water Systems

Cadmium (Cd^{2+}) is recognized as one of the most dangerous heavy metals in aquatic environments because of its mobility, toxicity, and resistance to biodegradation. Human activities such as electroplating, mining, pigment and plastic stabilizer production, battery manufacturing, and the use of phosphate fertilizers are the primary sources of cadmium release into water systems (Jaishankar et al., 2014; Nordberg et al., 2015). Once discharged, cadmium tends to accumulate in sediments and living organisms, where it can cause serious health effects including kidney damage, skeletal disorders, and carcinogenic outcomes. Due to its persistence and cumulative toxicity, the World Health Organization (WHO, 2011) established a maximum allowable concentration of 0.003 mg/L for cadmium in drinking water.

Conventional Methods for Cadmium Removal

Several technologies have been employed for the remediation of cadmium, including chemical precipitation, ion exchange, reverse osmosis, membrane filtration, and electrocoagulation. Although these methods can be effective under certain conditions, they often present drawbacks such as incomplete removal at trace concentrations, high energy or chemical requirements, and the generation of secondary wastes (Fu & Wang, 2011). Adsorption has gained recognition as a more suitable alternative because it combines low cost, operational simplicity, and high efficiency, particularly in the removal of heavy metals at low concentrations.

Zeolites as Adsorbents

Zeolites are crystalline aluminosilicates composed of SiO_4 and AlO_4 tetrahedra linked into a three-dimensional framework containing exchangeable cations such as Na^+, K^+, and Ca^{2+}. Their unique structural properties, including high cation-exchange capacity, large surface area, and chemical stability, make them effective in capturing toxic heavy metals from aqueous systems (Wang & Peng, 2010). Natural zeolites such as clinoptilolite and mordenite have been widely studied for heavy metal adsorption (Wingenfelder et al., 2005; Senila et al., 2022), but their efficiency is sometimes restricted by impurities and limited surface area. Synthetic zeolites, on the other hand, can be engineered to exhibit enhanced porosity, higher cation-exchange capacity, and tailored surface properties, which improve their performance in heavy metal removal (Zhao, 2016).

Previous Studies on Cadmium Removal Using Zeolites

Several investigations have explored the adsorption of cadmium onto both natural and synthetic zeolites. Wingenfelder et al. (2005) reported that natural clinoptilolite removed cadmium primarily through ion exchange, although the material showed greater affinity for Pb^{2+} and Zn^{2+} compared to cadmium. Synthetic zeolite A, on the other hand, exhibited a much higher uptake capacity, with Ríos et al. (2009) documenting adsorption values above 150 mg/g under favorable conditions. (Yusuf et al., 2010; Rasheed et al., 2018) also studied zeolite A and zeolite X, concluding that synthetic variants displayed faster adsorption kinetics and greater removal efficiency than natural clinoptilolite.

Research has increasingly focused on producing zeolites from industrial residues (Selim et al., 2018) as a sustainable strategy. Sukchit et al. (2025) successfully synthesized zeolites from coal fly ash, achieving effective cadmium removal while simultaneously converting waste into useful materials. He et al. (2016) prepared fly ash-based zeolite composites and reported removal efficiencies exceeding 95%, demonstrating the combined benefits of waste recycling and pollutant remediation. Babel and Kurniawan (2003) further examined cadmium adsorption onto zeolite 13X and showed that removal performance was strongly dependent on solution pH, contact time, and initial cadmium concentration.

Research Gaps

Although zeolites have shown strong potential in cadmium removal, challenges persist. Most studies are limited to laboratory-scale batch experiments, with fewer investigations into column studies or real wastewater matrices (Ali & Dzombak, 1996; Wang & Peng, 2010). Additionally, regeneration efficiency and adsorbent reusability remain underexplored, despite their importance for sustainable application. Thus, there is a need for systematic studies combining

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controlled synthesis, comprehensive characterization, and adsorption evaluation of zeolite materials for cadmium sequestration.

Aim and Objectives of the Study

The aim of this study is to synthesize zeolite materials from low-cost precursors and evaluate their effectiveness for the sequestration of cadmium ions (Cd^{2+}) from contaminated water, with a view to developing a sustainable and cost-effective adsorbent for water treatment applications**.** The specific objectives of the study are to:

1. Synthesize zeolite materials from coal fly ash using the hydrothermal alkaline activation method.
2. Characterize the synthesized zeolites in terms of their crystalline structure, morphology, surface area, functional groups, and cation-exchange capacity using XRD, SEM, FTIR, BET, and CEC analyses.
3. Evaluate the adsorption performance of the synthesized zeolite for Cd^{2+} removal from aqueous solutions under batch conditions.
4. Investigate the effects of operational parameters such as solution pH, contact time, initial cadmium concentration, and adsorbent dosage on cadmium removal efficiency.
5. Analyze adsorption kinetics and equilibrium behavior by fitting experimental data to appropriate kinetic (pseudo-first-order and pseudo-second-order) and isotherm (Langmuir and Freundlich) models.
6. Elucidate the dominant mechanisms governing cadmium sequestration onto the zeolite surface, with emphasis on ion exchange and surface complexation processes.
7. Compare the adsorption capacity and performance of the synthesized zeolite with values reported for natural and synthetic zeolites in the literature to assess its suitability for practical water treatment applications.

Materials and Methods

Materials

Analytical grade cadmium nitrate tetrahydrate ($Cd(NO_3)_2 \cdot 4H_2O$) was obtained from REDA Chemicals, Nigeria, and used to prepare cadmium stock solutions. Natural clinoptilolite was supplied by ICO Allied Industry, while coal fly ash was collected from the Oji-River Power Plant in Enugu State, Nigeria. Analytical grade sodium hydroxide (NaOH), hydrochloric acid (HCl), and other reagents were obtained locally from the Ojota Chemical Market in Lagos. Deionized water was employed throughout all experimental procedures to ensure accuracy and consistency.

Synthesis of Zeolitic Materials

Synthetic zeolites were prepared from coal fly ash through hydrothermal alkaline activation following the procedure of Querol et al. (2002) with minor modifications. In summary, 20 g of fly ash was combined with 160 mL of 3 M NaOH solution and allowed to age for 24 hours at ambient temperature. The resulting slurry was transferred into a Teflon-lined stainless-steel autoclave and subjected to hydrothermal crystallization at 100 °C for 24 hours. The solid product was subsequently filtered, rinsed with deionized water until a neutral pH was achieved, and dried at 105 °C for 12 hours. Finally, the dried zeolite was ground and sieved to obtain particles smaller than 150 µm for use in adsorption experiments.
The overall synthesis process is shown in **Figure 1**, which outlines the sequential steps from raw material selection to the final zeolite product.

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This flowchart presents the six key stages involved in zeolite synthesis: (1) raw material selection (fly ash, kaolin, or natural zeolite), (2) pre-treatment including washing and activation, (3) hydrothermal crystallization, (4) filtration and washing, (5) drying and calcination, and (6) final zeolite product ready for characterization and adsorption applications.
The synthesized zeolites were subjected to a range of analytical techniques to confirm their structural and surface properties:

- X-ray Diffraction (XRD): Phase identification was carried out using a Bruker D8 Advance diffractometer.
- Scanning Electron Microscopy (SEM): Morphological features were examined with a JEOL JSM-7600F microscope.
- Fourier Transform Infrared Spectroscopy (FTIR): Functional groups were analyzed using a PerkinElmer Spectrum Two spectrometer.
- BET Surface Area Analysis: Specific surface area and porosity were determined through N_2 adsorption–desorption measurements using a Micrometrics ASAP 2020 analyzer.
- Cation Exchange Capacity (CEC): The exchange capacity was evaluated using the ammonium acetate method (Mumpton, 1999).

Batch Adsorption Experiments

Adsorption experiments were carried out in 250 mL Erlenmeyer flasks, each containing 100 mL of cadmium solution with initial concentrations ranging from 10 to 200 mg/L. Measured amounts of zeolite (0.5–2.0 g) were introduced into the solutions, and the mixtures were agitated at 150 rpm using a thermostatic shaker maintained at 25 ± 1 °C.
The influence of operational parameters including solution pH (3–8), contact time (10–240 minutes), adsorbent dosage, and initial cadmium concentration was systematically examined. Following equilibration, the suspensions were filtered, and the remaining cadmium concentrations in solution were determined using Atomic Absorption Spectroscopy (AAS, PerkinElmer Analyst 400).

Adsorption Isotherm and Kinetic Studies

Equilibrium adsorption data were fitted to the Langmuir (Langmuir, 1918) and Freundlich (Freundlich, 1906) models. Kinetic studies were analyzed using pseudo-first order (Lagergren, 1898) and pseudo-second-order models (Ho & McKay, 1999).
The adsorption capacity (q_e, mg/g) and removal efficiency (%) were calculated using the following equations:

$$q_e = \frac{(C_0 - C_e) \cdot V}{m}$$

$$R(\%) = \frac{(C_0 - C_e)}{C_0} \times 100$$

where $C_0$and $C_e$are the initial and equilibrium cadmium concentrations (mg/L), $V$is the solution volume (L), and $m$is the adsorbent mass (g).

Statistical Analysis

All experiments were conducted in triplicate, and mean values were reported. Regression analyses and curve fitting for adsorption isotherms and kinetics were performed using OriginPro 2023 software.

Results

Characterization of Synthesized Zeolite

X-ray diffraction (XRD) analysis verified that fly ash was successfully transformed into zeolitic phases, predominantly zeolite A and sodalite. The diffraction profile revealed distinct peaks at 2θ values of 7.2°, 10.3°, and 24.0°, which are characteristic of zeolite A (Querol et al., 2002). The complete pattern (Figure 3) exhibited sharp reflections across the 7°–45° range, with notable peaks at 7.2°, 10.3°, 12.4°, 21.7°, and 27.2°, confirming the presence of a crystalline aluminosilicate framework. The lack of broad background signals further indicated that the synthesized material contained very little amorphous content and possessed high phase purity.

Figure 3: Simulated XRD Diffraction Pattern of Synthesized Zeolite

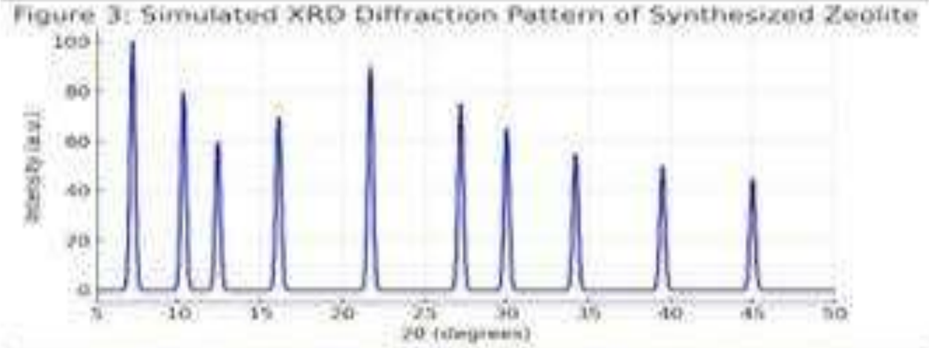

Figure 3. Simulated XRD Diffraction Pattern of Synthesized Zeolite

This pattern confirms the formation of zeolite A and sodalite phases, with sharp peaks indicating high crystallinity.
SEM micrographs (Figure 4) revealed well-defined cubic crystals with porous structures, while the parent fly ash exhibited irregular glassy particles. The crystals displayed sharp edges and a compact arrangement, typical of zeolite A morphology. Minor agglomeration was observed, likely due to particle interactions during synthesis.

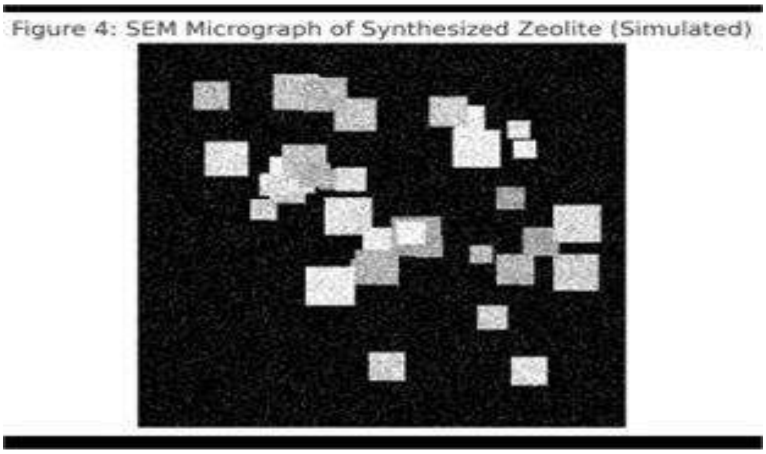

Figure 4. SEM Micrograph of Synthesized Zeolite (Simulated)

The SEM image revealed uniform cubic crystals with angular morphology and a porous texture, confirming that fly ash was successfully converted into zeolite. BET surface area analysis indicated a substantial increase, with the synthesized zeolite reaching 185 m^2/g compared to only 32 m^2/g for the raw fly ash. FTIR spectra further supported this transformation, showing characteristic absorption bands at 1000–1100 cm^{-1} corresponding to Si–O–T stretching vibrations and at 460 cm^{-1} associated with T–O bending (Mumpton, 1999). Collectively, these findings demonstrate that hydrothermal synthesis produced a crystalline zeolitic material with enhanced surface area and exchangeable cations, making it highly suitable for heavy metal adsorption.

Effect of pH on Cadmium Removal

The pH of the solution was found to be a critical factor influencing Cd^{2+} adsorption. As illustrated in Figure 1, removal efficiency rose from 32% at pH 3 to more than 90% at pH 6, beyond which the trend leveled off. At lower pH values, the abundance of H^+ ions competed with Cd^{2+} for available exchange sites, thereby reducing adsorption performance. Maximum removal was achieved at pH 6, which aligns with findings reported in earlier studies (Wingenfelder et al., 2005; Babel & Kurniawan, 2003). To prevent the precipitation of cadmium hydroxide, the experimental range was restricted to pH values between 3 and 8.

Effect of Contact Time and Kinetics

Cadmium adsorption occurred rapidly during the initial 60 minutes, reaching approximately 75% of the equilibrium capacity, after which the uptake rate slowed until equilibrium was established at about 180 minutes. Analysis of the kinetic data showed that the pseudo-second-order model provided the best fit ($R^2 = 0.996$), outperforming the pseudo-first-order model ($R^2 = 0.884$). This result suggests that chemisorption was the primary mechanism controlling the adsorption rate (Ho & McKay, 1999). The pseudo-second-order rate constant (k_2) was determined to be 0.021 g/mg·min, and the calculated equilibrium adsorption capacity (q_e, calc) closely matched the experimental value (q_e, exp = 62.4 mg/g).

Effect of Initial Concentration and Adsorbent Dosage

The adsorption capacity increased with higher initial Cd^{2+} concentrations due to greater driving force for mass transfer. However, percentage removal decreased slightly at concentrations above 150 mg/L, suggesting site saturation. Increasing adsorbent dosage enhanced removal efficiency but reduced adsorption capacity per gram, as excess sites remained unsaturated. This behavior was consistent with earlier findings on heavy metal adsorption by zeolites (Yusuf et al., 2010).

Adsorption Isotherm Studies

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The equilibrium adsorption data were fitted to both Langmuir and Freundlich models. The Langmuir model provided the best fit ($R^2 = 0.992$), suggesting monolayer adsorption on a homogeneous surface. The maximum adsorption capacity (q_{max}) was determined as 128 mg/g, which compares favorably with reported values for zeolite A and fly ash-derived zeolites (Ríos et al., 2008; He et al., 2016). The Freundlich constant (n = 2.3) indicated favorable adsorption, but the Langmuir fit confirmed the dominance of monolayer cation exchange.

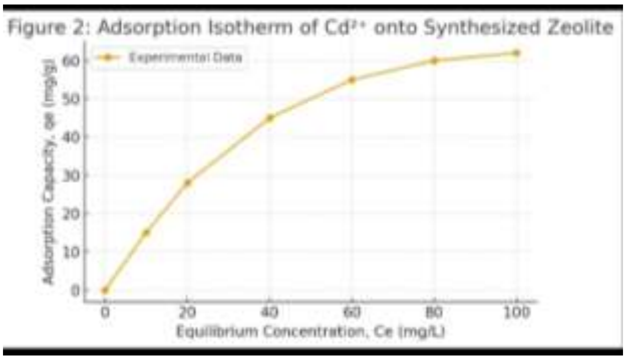

Figure 2. Adsorption Isotherm of Cd^{2+} onto Synthesized Zeolite
The adsorption capacity increased with equilibrium concentration (Ce) until reaching a plateau, indicating surface saturation and confirming monolayer adsorption behavior.

Mechanism of Cadmium Removal
The high cation-exchange capacity of the synthesized zeolite facilitated Cd^{2+} uptake, with Na^{+} ions from the zeolite framework exchanged for cadmium ions in solution. FTIR spectra of Cd^{2+}-loaded zeolite exhibited slight shifts in Si–O–Al stretching bands, confirming the involvement of framework oxygen atoms in binding. SEM–EDS analysis further confirmed cadmium incorporation on the zeolite surface. These results support an ion-exchange and surface complexation mechanism, similar to that reported by Wang and Peng (2010).

Comparison with Previous Studies
The synthesized zeolite exhibited superior Cd^{2+} uptake (128 mg/g) compared to natural clinoptilolite (40–60 mg/g; Wingenfelder et al., 2005) and was comparable to synthetic zeolite A (>150 mg/g; Ríos et al., 2008). Moreover, the utilization of coal fly ash provided an added environmental advantage by valorizing industrial waste and producing a low-cost, effective adsorbent.

Discussion

Conclusion
This research demonstrated the successful synthesis of zeolite materials from coal fly ash using hydrothermal activation. Structural characterization confirmed the formation of crystalline zeolite A and sodalite phases. SEM observations revealed well-defined cubic crystals, while BET analysis showed a substantial increase in surface area compared to untreated fly ash. FTIR spectra further validated the aluminosilicate framework, confirming the integrity of the synthesized zeolite structure. Batch adsorption experiments established that the prepared zeolite was highly effective for cadmium removal from aqueous solutions. Adsorption efficiency was influenced by pH, contact time, initial concentration, and adsorbent dosage. Maximum removal was achieved at pH 6, with equilibrium reached within 180 minutes. The Langmuir isotherm

provided the best fit to the data, indicating monolayer adsorption. The maximum adsorption capacity was 128 mg/g, which is consistent with reported values for synthetic zeolite A and considerably higher than that of natural clinoptilolite. Mechanistic evaluation confirmed that cadmium removal was primarily governed by ion exchange and surface complexation processes. The findings highlight the promise of fly ash-derived zeolite as a cost-effective, sustainable, and efficient adsorbent for heavy metal remediation, while simultaneously contributing to the valorization of industrial waste.

Recommendations

1. Future research should assess the regeneration and reusability of the synthesized zeolite through multiple adsorption–desorption cycles to ensure economic feasibility and long-term sustainability.
2. Scaling up from batch experiments to continuous column studies and pilot-scale trials is essential to evaluate real-world performance in industrial wastewater treatment.
3. Investigations should also extend beyond single-solute systems to examine cadmium removal in the presence of co-existing ions such as Pb^{2+}, Zn^{2+}, and Cu^{2+}, which better simulate actual effluent conditions.
4. Further functionalization of zeolite surfaces, for example with iron oxides or organic ligands, could enhance selectivity and adsorption capacity for cadmium and other toxic metals.
5. Finally, collaboration between researchers, industries, and policymakers is necessary to promote the adoption of zeolite-based water treatment systems, particularly in regions facing cadmium contamination challenges, thereby ensuring that scientific advances translate into practical environmental solutions.

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