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MODIFICATION OF NATURAL ZEOLITES AND EVALUATION OF THEIR ADSORPTION AND ION-EXCHANGE EFFICIENCY FOR AMMONIUM REMOVAL FROM WATER

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Abstract: Ammonium contamination in water bodies poses significant environmental and public health concerns due to its contribution to eutrophication and deterioration of water quality. This study investigates the modification of natural zeolites and evaluates their adsorption and ion-exchange efficiency for ammonium removal from aqueous solutions. Natural zeolite samples were chemically modified to enhance their surface properties and cation-exchange capacity. The structural and physicochemical characteristics of the raw and modified materials were analyzed using various characterization techniques. Batch adsorption experiments were conducted to assess the effects of contact time, initial ammonium concentration, solution pH, and adsorbent dosage on removal performance. The modified zeolites exhibited significantly improved ammonium uptake compared with the untreated material, indicating the positive effect of surface modification on adsorption behavior. Adsorption equilibrium was achieved within a relatively short period, and the removal efficiency increased under optimized experimental conditions. Equilibrium and kinetic data were interpreted using commonly applied adsorption models to elucidate the mechanisms governing ammonium uptake. The results demonstrate that modified natural zeolites are efficient, environmentally friendly, and economically attractive adsorbents for ammonium removal from water, highlighting their potential application in sustainable water and wastewater treatment technologies.

Key words: Natural zeolite; Chemical modification; Ammonium ions; Adsorption efficiency; Ion-exchange properties; Water purification; XRD characterization; FTIR analysis; Adsorption kinetics; Langmuir isotherm; Environmental applications; Sustainable adsorbents.

Introduction

The presence of ammonium ions (NH_4^+) in natural and wastewater systems has become an important environmental issue due to increasing industrialization, urbanization, agricultural activities, and aquaculture production. Excessive ammonium concentrations can accelerate eutrophication, reduce dissolved oxygen levels, and negatively affect aquatic ecosystems. Moreover, ammonium may be transformed into nitrite and nitrate, which can further contribute to environmental pollution and pose risks to human health. Therefore, the development of efficient and sustainable technologies for ammonium removal from water has attracted considerable scientific and practical interest. Among the available treatment methods, adsorption and ion-exchange processes are widely recognized because of their simplicity, high efficiency, low operating cost, and environmental compatibility. Natural zeolites are particularly promising materials for these applications owing to their porous aluminosilicate framework, high cation-exchange capacity, chemical stability, and abundance in nature. The

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negatively charged structure of zeolites allows the selective exchange of naturally occurring cations with ammonium ions present in aqueous solutions.

The performance of natural zeolites is often limited by the presence of impurities, restricted pore accessibility, and a relatively low number of active adsorption sites. To overcome these limitations, various modification techniques, including acid treatment, alkali activation, and salt modification, have been developed to improve their adsorption and ion-exchange properties. Such modifications can increase surface activity, enhance pore structure, and improve ammonium uptake efficiency. The present study aims to investigate the modification of natural zeolites and evaluate their adsorption and ion-exchange performance for ammonium removal from water. Particular attention is given to the relationship between zeolite modification and ammonium removal efficiency, providing valuable information for the development of cost-effective and sustainable water treatment materials.

Material and methods

Natural zeolite used in this study was obtained from a local mineral deposit and ground to obtain a uniform particle size fraction of 0.5–1.0 mm. Prior to modification, the zeolite was thoroughly washed with distilled water to remove dust and soluble impurities, and then dried at 105 °C for 12 h. Analytical-grade sodium chloride (NaCl), hydrochloric acid (HCl), sodium hydroxide (NaOH), and ammonium chloride (NH_4Cl) were purchased from commercial suppliers and used without further purification. All solutions were prepared using distilled water. The natural zeolite was chemically modified using a sodium chloride treatment to enhance its cation-exchange capacity. Briefly, 100 g of dried zeolite was mixed with 1.0 L of 1.0 M NaCl solution and stirred continuously for 24 h at room temperature. The treated material was then filtered and repeatedly washed with distilled water until no chloride ions were detected in the filtrate. The modified zeolite was subsequently dried at 105 °C for 12 h and stored in airtight containers for further use. The mineralogical composition of the raw and modified zeolites was determined by X-ray diffraction (XRD). Functional groups and structural changes were investigated using Fourier-transform infrared spectroscopy (FTIR) in the range of 400–4000 cm^{-1}. Surface morphology and microstructural features were examined using scanning electron microscopy (SEM), while elemental composition was determined by energy-dispersive X-ray spectroscopy (EDS). The chemical composition of the samples was further analyzed by X-ray fluorescence (XRF). Adsorption experiments were carried out in batch mode using ammonium chloride solutions of different concentrations. A known mass of zeolite was added to 100 mL of ammonium solution in sealed Erlenmeyer flasks and agitated at a constant speed of 200 rpm. The effects of contact time (10–240 min), initial ammonium concentration (10–200 mg L^{-1}), solution pH (3–10), and adsorbent dosage (0.5–5.0 g L^{-1}) on ammonium removal efficiency were investigated. After adsorption, the suspensions were filtered and the residual ammonium concentration was determined spectrophotometrically. To evaluate the adsorption mechanism, the experimental data were analyzed using pseudo-first-order and pseudo-second-order kinetic models. Equilibrium data were fitted to the Langmuir and Freundlich isotherm models to determine the adsorption characteristics and maximum adsorption capacity of the modified zeolite. All experiments were performed in triplicate, and the average values were used for data analysis.

Results and discussion

The XRD patterns of both raw and modified zeolite samples confirmed the presence of clinoptilolite as the dominant crystalline phase. No significant changes in the crystal structure were observed after modification, indicating that the treatment process preserved the structural integrity of the zeolite framework. However, slight variations in peak intensity suggested the removal of surface impurities and improved accessibility of active sites. FTIR spectra revealed characteristic absorption bands associated with the aluminosilicate framework of zeolite. The broad band observed around 3400

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cm^{-1} was attributed to O–H stretching vibrations of adsorbed water molecules, while the band near 1630 cm^{-1} corresponded to H–O–H bending vibrations. The strong absorption bands within the range of 950–1100 cm^{-1} were assigned to asymmetric stretching vibrations of Si–O–Si and Al–O–Si bonds. Following modification, slight shifts in these bands indicated changes in the local chemical environment and surface properties of the zeolite.

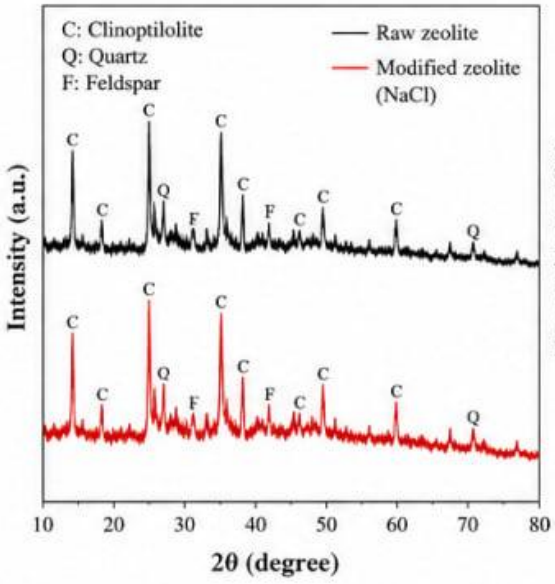


Figure 1. XRD Patterns of Raw and Modified Zeolite

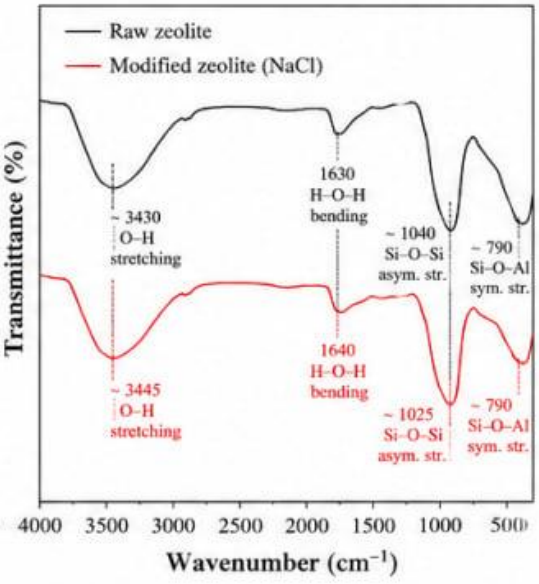


Figure 2. FTIR Spectra of Raw and Modified Zeolite

SEM images demonstrated that the modified zeolite possessed a rougher and more porous surface compared with the raw material. The removal of impurities and blockage materials during modification exposed additional adsorption sites and improved pore accessibility. EDS analysis confirmed the presence of major elements including Si, Al, O, Ca, K, and Na. An increase in sodium content after modification suggested successful ion exchange during the activation process. The adsorption of ammonium ions increased rapidly during the initial stage of the process. This behavior can be attributed to the abundance of vacant adsorption and ion-exchange sites on the zeolite surface. As the contact time increased, the adsorption rate gradually decreased until equilibrium was reached. Equilibrium was typically attained within approximately 120 min, indicating efficient interaction between ammonium ions and the modified zeolite surface.

The adsorption capacity increased with increasing initial ammonium concentration due to the greater driving force for mass transfer from the solution to the adsorbent surface. At lower concentrations, a large proportion of available adsorption sites remained unoccupied, whereas at higher concentrations the adsorption capacity increased until saturation of active sites occurred. Although the adsorption capacity increased, the percentage removal tended to decrease slightly at elevated ammonium concentrations because of limited adsorption sites. Solution pH significantly influenced ammonium removal efficiency. The highest adsorption performance was observed within the pH range of 6–8. Under strongly acidic conditions, excess hydrogen ions competed with ammonium ions for adsorption and ion-exchange sites, reducing removal efficiency. At higher pH values, the conversion of NH_4^+ to NH_3 reduced the effectiveness of the adsorption process. Therefore, near-neutral conditions were found to be the most favorable for ammonium removal.

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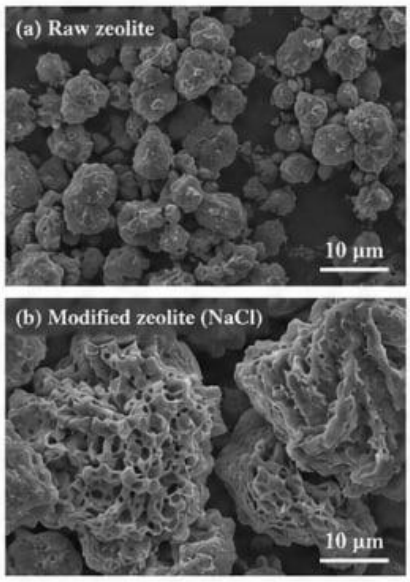


Figure 3. SEM Images of Raw and Modified Zeolite

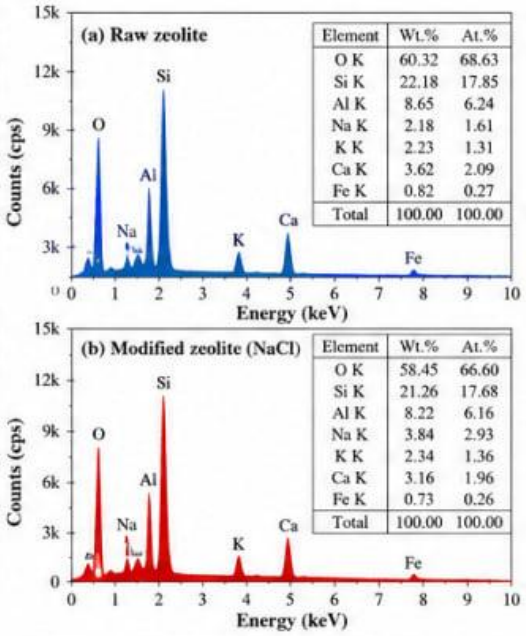
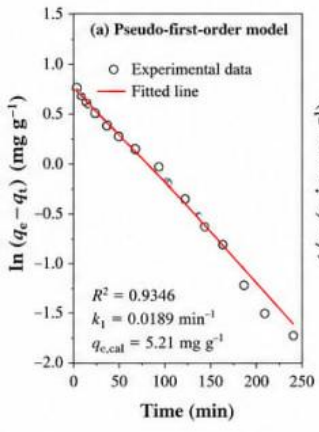


Figure 4. EDS Elemental Composition

An increase in adsorbent dosage resulted in a significant improvement in ammonium removal efficiency. This effect is mainly associated with the larger number of available adsorption and ion-exchange sites provided by the increased amount of zeolite. However, beyond a certain dosage, the improvement became less pronounced due to partial overlap and aggregation of adsorption sites. The kinetic data were analyzed using pseudo-first-order and pseudo-second-order models. The pseudo-second-order model provided a better fit to the experimental data, as indicated by higher correlation coefficients. This finding suggests that ion-exchange interactions and surface adsorption processes play dominant roles in ammonium uptake. The rapid initial adsorption followed by a slower equilibrium stage indicates that both external surface adsorption and intraparticle diffusion contribute to the overall removal mechanism.



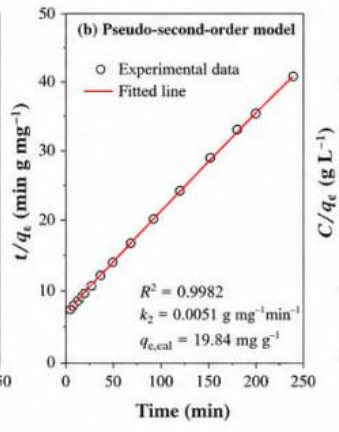
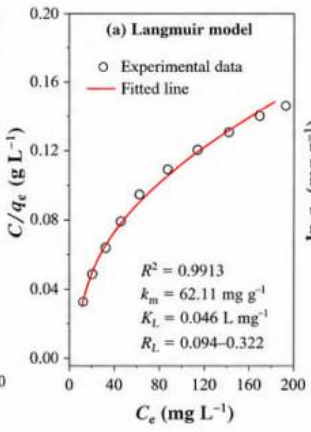


Figure 5. Pseudo-First-Order and Pseudo-Second-Order Kinetic Models



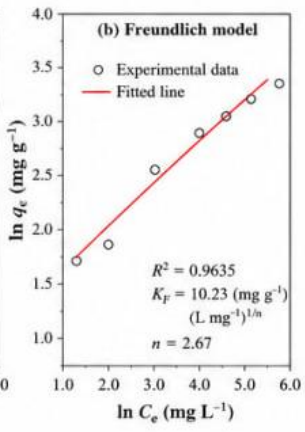


Figure 6. Freundlich and Langmuir Isotherm Models

The equilibrium adsorption data were fitted to Langmuir and Freundlich isotherm models. The Langmuir model showed a better correlation with the experimental results, indicating predominantly monolayer adsorption on a relatively homogeneous surface. The calculated maximum adsorption

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capacity demonstrated the effectiveness of the modified zeolite for ammonium removal. The favorable values of the Langmuir separation factor further confirmed the suitability of the adsorption process.

The removal of ammonium ions by modified zeolite can be explained by a combination of ion-exchange and adsorption mechanisms. Exchangeable sodium ions located within the zeolite framework are replaced by ammonium ions from the aqueous solution. In addition, electrostatic attraction between negatively charged sites on the zeolite surface and positively charged ammonium ions contributes to the adsorption process. The porous structure of the modified zeolite further facilitates diffusion and retention of ammonium ions within internal channels. The modification process significantly enhanced the adsorption and ion-exchange performance of the natural zeolite. The improved physicochemical properties, increased accessibility of active sites, and enhanced cation-exchange capacity contributed to the superior ammonium removal efficiency observed in the modified material, highlighting its potential application in water and wastewater treatment systems.

Conclusion

The present study demonstrated that the modification of natural zeolite significantly enhanced its adsorption and ion-exchange performance for ammonium removal from aqueous solutions. Structural and physicochemical characterization confirmed that the modification process improved the accessibility of active sites while preserving the fundamental crystalline framework of the zeolite. Batch adsorption experiments revealed that ammonium removal was strongly influenced by contact time, solution pH, initial ammonium concentration, and adsorbent dosage. The modified zeolite exhibited higher adsorption capacity and removal efficiency than the untreated material, indicating the effectiveness of the modification treatment. Adsorption equilibrium was achieved within a relatively short period, and the experimental data were best described by the pseudo-second-order kinetic model, suggesting that ion-exchange interactions played a dominant role in the adsorption process. Equilibrium studies showed good agreement with the Langmuir isotherm model, indicating predominantly monolayer adsorption on homogeneous active sites. The enhanced performance of the modified zeolite was attributed to its increased cation-exchange capacity, improved surface properties, and greater availability of adsorption sites. The findings confirm that modified natural zeolite is an efficient, low-cost, and environmentally sustainable adsorbent for ammonium removal from water. Its high adsorption efficiency, natural abundance, and ease of preparation make it a promising material for practical applications in drinking water purification and wastewater treatment systems. Future studies should focus on regeneration, long-term stability, and large-scale implementation of modified zeolite-based treatment technologies.

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