



Synthesis of Cancrinite Zeolite from Toraja Natural Bentonite for Heavy Metal Removal from Wastewater

Yuli Astuti

1Department of Chemistry, Hasanuddin University, Makassar, Indonesia

Paulina Taba

Department of Chemistry, Hasanuddin University, Makassar, Indonesia, paulinataba@unhas.ac.id

Siti Fauziah

Department of Chemistry, Hasanuddin University, Makassar, Indonesia

Syarifuddin Liong

Department of Chemistry, Hasanuddin University, Makassar, Indonesia,

Yusafir Hala

Department of Chemistry, Hasanuddin University, Makassar, Indonesia

See next page for additional authors

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Keywords

Zeolite, Cancrinite, Adsorption, Heavy metals, Natural bentonite

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Authors

Yuli Astuti, Paulina Taba, Siti Fauziah, Syarifuddin Liong, Yusafir Hala, Nur Umriani Permatasari, Satria Putra Jaya Negara, and Fadliah Fadliah

RESEARCH PAPER

Synthesis of Cancrinite Zeolite From Toraja Natural Bentonite for Heavy Metal Removal From Wastewater

Yuli Astuti^a, Paulina Taba^{a,*}, St. Fauziah^a, Syarifuddin Liong^a, Yusafir Hala^a, Nur U. Permatasari^a, Satria P.J. Negara^b, Fadliah^c

^a Department of Chemistry, Hasanuddin University, Makassar, Indonesia

^b Department of Chemistry, State University of Makassar, Makassar, Indonesia

^c Department of Mining Engineering, Trisakti University, Jakarta, Indonesia

Abstract

This study explores the production of cancrinite (CAN) zeolite from bentonite found in Toraja (a place in South Sulawesi Province, Indonesia) and its effectiveness in removing Pb^{2+} and Fe^{3+} ions from wastewater. The synthesis was carried out using a hydrothermal method with varying NaOH concentrations, where a single phase of zeolite was formed at 5 M, as confirmed through XRD analysis. FTIR results showed the characteristics of CAN zeolite with typical absorption peaks at 676, 622, and 565 cm^{-1} , as well as the presence of carbonate groups (CO_3^{2-}) at 1300-1400 cm^{-1} . SEM analysis revealed thin columnar needle-like crystals characteristic of CAN zeolite. Nitrogen adsorption-desorption tests revealed that CAN zeolite is mesoporous with an average pore size of 5.38 nm and a specific surface area of 51.97 m^2/g . The optimal conditions for Pb^{2+} adsorption were achieved with a contact time of 180 min and a pH of 5, while for Fe^{3+} , the optimal conditions were a contact time of 30 min and a pH of 3. The adsorption isotherm was evaluated using Langmuir, Freundlich, and Sips models, with the best described by the Sips model in non-linear fitting. The maximum adsorption capacities for Pb^{2+} and Fe^{3+} ions were 194.95 mg/g and 196.63 mg/g, respectively. The adsorption kinetics for both ions followed a pseudo-second-order kinetic model. This study reports about natural Toraja bentonite, a rarely explored precursor for CAN zeolite synthesis. It also assesses the zeolite's adsorption capacity for two metal ions of different valences, highlighting its potential in wastewater treatment.

Keywords: Zeolite, Cancrinite, Adsorption, Heavy metals, Natural bentonite

1. Introduction

The concentration of heavy metals in water has risen due to industrial activities, anthropogenic factors, and modern industrialization [1]. Heavy metals are acknowledged to be hazardous and resist natural decomposition, resulting in their accumulation in the environment [2]. Heavy metals in water can negatively impact human health, especially when concentrations exceed permitted limits [3,4]. Heavy metal ions frequently found in industrial effluent include lead (Pb^{2+}) and iron (Fe^{3+}), usually identified in significant concentrations [5]. Pb^{2+} ions are harmful environmental contaminants that have significant toxic effects on

various organs, even at low concentrations [6]. Fe^{3+} ions are essential micronutrients that serve as enzyme cofactors, facilitate oxygen transport, bolster immune system function, and play a role in energy metabolism and cellular growth [7]. The accumulation of Fe^{3+} ions can lead to health issues, including skin and ocular irritation, intestinal wall damage, and respiratory complications. Based on the harm caused by these heavy metal ions, separating and treating of industrial wastewater containing metal ions is necessary to prevent harm to ecological systems and human health.

Various techniques have been used for separation and treatment of wastewater to remove Pb^{2+} and

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* Corresponding author.

E-mail address: Paulinatataba@unhas.ac.id (P. Taba).

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Fe^{3+} ions, such as membrane separation [8], ion exchange [9,10], chemical precipitation [11], coagulation [12], electrolysis [13], and adsorption [14]. Adsorption is a highly beneficial technique for mitigating metal pollution [15–17]. Adsorption techniques can remove contaminants even at low concentrations, using minimal energy and easily accessible raw materials to create adsorbents from various sources [18,19].

Types of adsorbents that are widely used in wastewater treatment include activated carbon [20], chitosan [21], magnetite [22], mesoporous silica [23], and nanoparticles [24]. However, the limited adsorption capacity and selectivity for certain ions limit their effectiveness. Therefore, zeolites are a promising alternative material for treating wastewater containing heavy metal ions [25].

Zeolites are crystalline, hydrated aluminosilicates with well-defined molecular sieving properties. They are characterized by a high adsorption capacity and large specific surface area. The three-dimensional framework of zeolites is composed of SiO_4 and AlO_4 tetrahedra linked by shared oxygen atoms, forming cage-like structures and channels [26,27]. The significant adsorption and elevated cation exchange capacity make zeolites particularly effective for removing environmental pollutants [28,29]. The arrangement of zeolite compounds features numerous channels and voids among its molecules, facilitate the diffusion of species into the inter-particle space [30,31]. The application of zeolites as adsorbents has been extensively studied due to their thermal and chemical stability, low environmental friendliness impact, and excellent binding properties. The method for preparing zeolites is straightforward and cost-effective [32].

Zeolites can be obtained naturally or synthesized [33]. Although natural zeolite has been widely used as an adsorbent, its limited availability and variable properties have prompted the development of synthesized zeolite with high purity [34]. Furthermore, the zeolite synthesis process tends to be expensive when using commercial materials, so further research is needed to find alternative, more economical raw materials, such as natural bentonite [32]. As a natural precursor, bentonite can significantly reduce the cost of zeolite synthesis. Its high aluminosilicate content plays a crucial role in minimizing or even eliminating the need for expensive commercial chemical sources, such as sodium silicate and sodium aluminate [35]. Several zeolites synthesized from natural bentonite have been extensively developed and utilized as adsorbents for metal ions. Huang et al. [36] described Na-A zeolite

synthesized from bentonite for the adsorption of Cu^{2+} ions, whereas [37] produced ZSM-5 zeolite from bentonite as an adsorbent for Pb^{2+} ions. Moreover [38], successfully synthesized nitrate cancrinite zeolite from natural bentonite. Based on the potential of bentonite as a raw material, these investigations prompted an attempt to synthesize zeolite cancrinite from Toraja bentonite, which has not been extensively researched or utilized as an adsorbent.

Zeolites were synthesized using the hydrothermal technique. The procedure is straightforward and does not require commercial sources of silica-alumina, as reported [39]. The synthesized zeolite was characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy (SEM), and surface area analyzer (SAA) techniques. A Fourier Transform Infrared (FTIR) spectrophotometer was used to identify functional groups within the zeolite structure. The zeolites produced from this synthesis procedure were then employed as adsorbents for Pb^{2+} and Fe^{3+} ions in the test solution. The effects of contact time, pH, and initial solution concentration were analyzed. The adsorption selectivity of Pb^{2+} and Fe^{3+} ions was examined in a binary solution system. The Pb^{2+} and Fe^{3+} concentrations are quantified in the solution post-adsorption using an Atomic Absorption Spectroscopy (AAS).

2. Materials and methods

2.1. Materials

The materials used in this study include bentonite, a natural mineral sourced from a mining area in the Toraja district of South Sulawesi, Indonesia (ground to pass through a 200-mesh sieve), NaOH (Merck), $\text{Pb}(\text{NO}_3)_2$ (Merck), FeCl_3 (Merck), HNO_3 (Merck), Na_2EDTA (Merck), double distilled water, Whatman 42 filter paper (Merck), and universal pH paper.

2.2. Synthesis of CAN zeolites

Zeolite synthesis was conducted under hydrothermal conditions, dissolving 1.4401 g of NaOH in 18 g of water to create a 2 M NaOH solution. Subsequently, 0.7776 g of Toraja natural bentonite (4 % w/w of the mixture) was introduced into the NaOH solution. The mixture was agitated until uniformly distributed, then placed in an autoclave, securely sealed, and exposed to thermal treatment at 170 °C for 24 hrs. After the thermal treatment, the synthesized particles were subjected to vacuum filtration

and rinsed until a neutral pH was reached. The synthesized zeolite underwent a drying process at 105 °C for 24 hrs. Varying concentrations of NaOH, namely 2 M, 3 M, and 5 M, were employed to evaluate their effect on the characteristics of the final product. The synthesized zeolite was then analyzed using XRD, FTIR, SEM, and SAA techniques.

2.3. Characterization

The composition of Toraja natural bentonite was analyzed using the XRF method. XRD (Shimadzu XRD-700 with Cu K α radiation) was used to characterize natural mineral samples and CAN zeolite. FTIR spectra were obtained using a Shimadzu IR Prestige 21, covering the 4000 to 400 cm⁻¹ range. The morphology was examined using SEM-EDX (SU-3500). Nitrogen adsorption–desorption isotherms were obtained using a SAA (Altamira micro 200 instrument). The surface area was determined using the Brunauer-Emmett-Teller (BET) method, while the pore size distribution was assessed through the BJH method.

2.4. Adsorption experiment

A total of 0.1 g of prepared CAN zeolite was mixed into 50 mL of solution containing Pb²⁺ and Fe³⁺ metal ions. The mixture underwent agitation at 150 rpm for the specified duration. After shaking, the mixture was filtered using filter paper, and the resulting filtrate was analyzed to determine the concentration of metal ions through AAS. Each experiment was conducted in duplicate. The adsorption capacity of the synthesized CAN zeolite for metal ions was evaluated using the following Equation (1):

$$q_e = \frac{C_0 - C_e}{m} V \quad (1)$$

m (g) represents the weight of zeolite CAN, while V (L) denotes the total volume of the metal ion solution.

The main variables influencing the adsorption process were assessed by examining the effects of contact time (3, 5, 10, 20, 30, 60, 90, 120, 180, 210, and 240 mins), pH (1–7), and the initial concentration of the sample solution (25, 50, 100, 150, 200, and 300 mg/L) using a single-variable experimental design. The competitive interaction between Pb²⁺ and Fe³⁺ ions was examined in a binary metal ion system. Each 50 mL metal ion solution was combined and then stirred under the optimal conditions identified for each metal.

2.5. Adsorption isotherm

The Langmuir isotherm indicates a monolayer adsorption process at specific active sites on the zeolite surface [40,41]. The Freundlich isotherm describes a multilayer adsorption process on heterogeneous surfaces, characterized by interactions between ions adsorbed on surrounding active sites [42,43]. The Sips isotherm model combines the characteristics of both the Langmuir and Freundlich models and principles of the preceding two models. It is explicitly employed for adsorption systems on heterogeneous surfaces [44,45]. Equations (2)–(6) represent the Langmuir, Freundlich, and Sips linear and non-linear isotherm models, respectively:

$$\frac{C_e}{q_e} = \frac{q_m}{q_m} + \frac{1}{K_L q_m} \quad (2)$$

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

The amount of CAN adsorbed is denoted by q_e (mg g⁻¹), while q_m (mg g⁻¹) represents its adsorption capacity. K_L (L mg⁻¹) is the Langmuir adsorption constant, while C_e (mg L⁻¹) is the equilibrium concentration of metal ions.

$$q_e = K_f C_e^{\frac{1}{n}} \quad (4)$$

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (5)$$

K_f (mg g⁻¹) represents the Freundlich adsorption constant, which is related to adsorption capacity, whereas $1/n$ indicates the constant that reflects the surface heterogeneity of the adsorbent.

$$q_e = \frac{q_{ms} K_s C_e^{ns}}{1 + K_s C_e^{ns}} \quad (6)$$

$$\ln \frac{q_e}{q_m - q_e} = \ln K_s + \frac{1}{n} \ln C_e \quad (7)$$

q_m (mg g⁻¹) represents the maximum adsorption capacity, K_s (L mg⁻¹) serves as the Sips equilibrium constant linked to adsorption affinity, and n is the Sips exponent that reflects surface heterogeneity. When $n = 1$, the surface is homogeneous, making the Sips model equivalent to the Langmuir isotherm.

2.6. Adsorption kinetics

Kinetic studies are conducted to assess the dynamics of the adsorption process using rate constants [46]. The adsorption process consists of three

key stages: (i) the movement of the solute from the bulk solution to the boundary layer on the adsorbent surface, (ii) the migration of the solute from the boundary layer onto the adsorbent surface, and (iii) the intraparticle diffusion of the solute from the surface into the internal pore structure of the adsorbent. This investigation examined the adsorption kinetics of Pb^{2+} and Fe^{3+} ions onto synthetic CAN zeolite using linear pseudo-first-order and pseudo-second-order kinetic models. The corresponding mathematical expressions are presented in Equations (8) and (9),

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (8)$$

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q^2} \quad (9)$$

k (min^{-1}) denotes the adsorption rate constant, t (min) represents the adsorption time, and q_t (mg g^{-1}) represents the adsorption capacity at a given time t .

3. Result and discussion

3.1. Material characterization

Cancrinite zeolite was synthesized using the hydrothermal method with bentonite from Toraja as the raw material. XRF analysis (Table 1) confirmed that the bentonite contains 56.55 % SiO_2 and 37.39 % Al_2O_3 , indicating its suitability for zeolite synthesis due to its high silica and alumina content [32].

The concentration of NaOH significantly influences zeolite synthesis by affecting the skeletal structure. Zeolite synthesis was conducted using

Toraja bentonite with NaOH at varying concentrations of 2, 3, and 5 M. Fig. 1a displays data of Toraja bentonite and synthesized zeolite at different NaOH concentrations. Toraja bentonite comprises many minerals, notably quartz, feldspar, and montmorillonite, which serve as its primary constituents. The existence of these compounds is evidenced by the prominent diffraction peaks at angles $2\theta = 19.49^\circ$, 20.97° , 26.53° , 29.46° , 35.04° , 47.35° , 50.00° , 59.97° , 62.31° , 68.47° , and 74.93° [47].

In the synthesis with a 2 M NaOH concentration, distinct diffraction peaks were observed at $2\theta = 15.84^\circ$, 20.16° , and 26.01° , indicating the formation of the analcime (ANA) phase [48]. Additional peaks at $2\theta = 10.81^\circ$, 11.79° , and 30.15° signify the existence of the nepheline hydrate (NHI) phase [49], while peaks at $2\theta = 18.86^\circ$, 24.24° , and 27.42° validate the formation of the cancrinite (CAN) phase [50]. At 2 M NaOH concentration, three zeolite phases-ANA, NHI, and CAN-were formed. Based on the report by Ref. [51], ANA typically forms at low NaOH concentrations. However, 2 M NaOH is not sufficiently low to produce a pure ANA phase, so the NHI and CAN phases also formed. NHI serve as an intermediate in CAN zeolite synthesis [52]. At 3 M NaOH, the characteristic peak of ANA diminishes, while at 5 M NaOH, only the distinctive peak of CAN remains, confirming that the successful formation of pure CAN zeolite. This result is consistent with [53] report, which states that CAN is more likely to form at high alkali concentrations.

Fig. 1b presents the FTIR spectrum of the Toraja natural bentonite (NB) and zeolite CAN synthesized. The materials exhibit characteristic spectral patterns of aluminosilicates [54]. The synthesized CAN zeolite exhibits a distinct absorption at 3600 cm^{-1} . The absorption correlates with hydroxyl groups (O-H) associated with the zeolite structure. A notable absorption shift occurs around 950 cm^{-1} , associated with the asymmetric stretching vibrations of the T-O bond, where T denotes Si or Al.

Table 1. Chemical composition of Toraja natural bentonite.

Wt. % Oxide	SiO_2	Al_2O_3	K_2O	Fe_2O_3	CaO	TiO_2
Natural bentonite	56.55	37.39	2.50	1.97	1.20	0.30

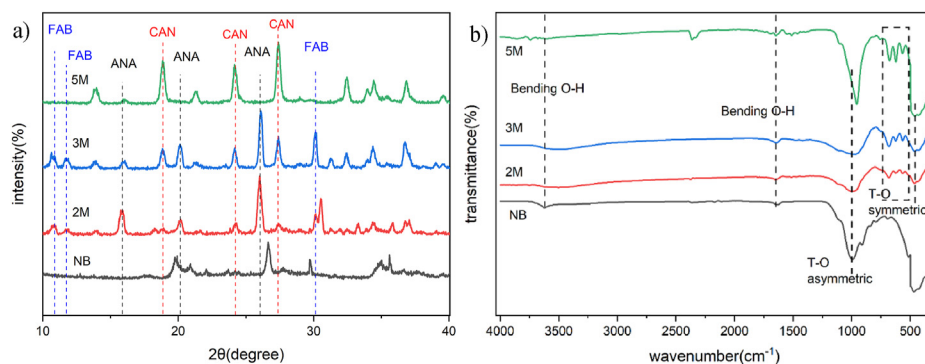


Fig. 1. a) XRD pattern; and b) FTIR spectra of Toraja natural bentonite and samples synthesized with 2 M, 3 M, and 5 M NaOH.

The characteristic pattern of zeolite CAN is evident in the absorption peaks observed at 676, 622, and 565 cm^{-1} . A weak absorption in the 1300–1400 cm^{-1} range signifies the presence of carbonate groups (CO_3^{2-}), characteristic of the structure of CAN zeolites. The presence of CO_3^{2-} groups demonstrates the capacity of CAN zeolites to accommodate large anions within their structure [55,56]. Additionally, the bands observed around 2350 cm^{-1} are attributed to atmospheric CO_2 , which is detected as a result of the ATR-IR measurement [38].

SEM analysis revealed significant disparities in surface morphology between Toraja bentonite and zeolite synthesized with 5 M NaOH (CAN) following hydrothermal treatment (Fig. 2). Toraja bentonite exhibits a textured structure similar to cornflakes, with a smooth surface, as reported in previous research [57]. The zeolite synthesized with 5 M NaOH exhibited thin columnar crystals resembling needles designated as CAN zeolite [58]. These findings confirm that Toraja bentonite can be converted into CAN zeolite using a hydrothermal method that employs suitable alkaline activators.

Alkaline activators promote the dissolution of silica and alumina oxides from the Toraja bentonite matrix, enabling the production of silica and alumina tetrahedral units that define cancrinite [59].

Based on the EDX analysis (Table 2), there is a significant difference in the Si/Al ratio between natural bentonite and the synthesized CAN zeolite. The Si/Al ratio decreased following synthesis, with the synthesized CAN zeolite exhibiting a Si/Al ratio of 1.27. This result is consistent with previous studies by Refs. [38,60], which reported that the Si/Al ratio of CAN zeolite typically ranges around 1.0.

Type IV adsorption isotherms, indicative of mesoporous materials, were identified in Toraja bentonite and cancrinite [61,62], as illustrated in Fig. 3. The BJH desorption branch patterns substantiate the findings, which affirm that both

Table 2. EDX analysis of Toraja natural bentonite and synthesized CAN zeolite (5 M NaOH).

Element	Atomic (%)	
	NB	CAN
O	60.38	55.87
Na	0.02	15.29
Mg	2.77	—
Al	9.27	12.72
Si	22.11	16.13
K	2.47	—
Ti	1.12	—
Fe	1.85	—
Si/Al	2.39	1.27

materials are crystalline mesoporous aluminosilicates with pore sizes ranging from 2 to 50 nm. The synthesis of cancrinite from Toraja bentonite enhanced BET surface area, reaching 51.97 m^2/g , alongside a total pore volume of 0.07 cc/g , as detailed in Table 3.

3.2. Adsorption of Pb^{2+} and Fe^{3+}

Adsorption studies on heavy metals were conducted using Pb^{2+} and Fe^{3+} ions. The adsorption processes were carried out with a contact time of 5–240 mins, a pH range of 1–7, and a concentration of 25–300 mg/L . Fig. 4a shows the effect of contact time on Pb^{2+} and Fe^{3+} ion adsorption using synthetic CAN zeolite. The optimal adsorption time for Pb^{2+} and Fe^{3+} ions was achieved at 30 mins and 180 mins, respectively. This optimal time indicates that the adsorption of Fe^{3+} ions occurs more rapidly with synthesized CAN zeolite than Pb^{2+} ions. This difference is attributed to the higher positive charge of Fe^{3+} , which enhances electrostatic interactions with negatively charged active sites on the zeolite surface, thereby facilitating faster adsorption [63,64]. After this period, adsorption capacity decreased, signifying the saturation of active sites on the CAN zeolite and the weakening of electrostatic

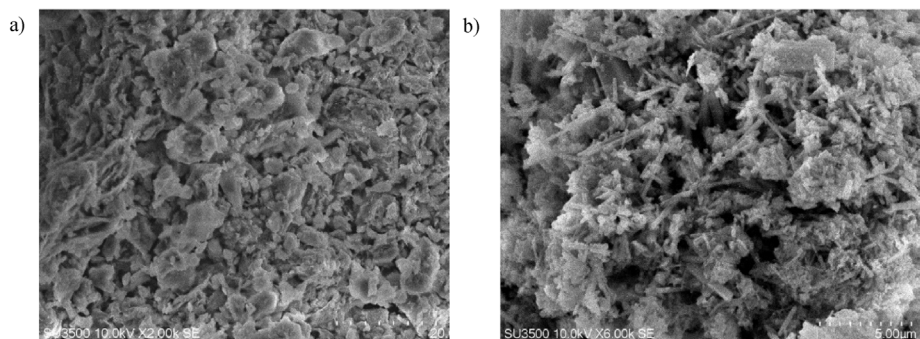


Fig. 2. SEM image of a) Toraja natural bentonite b) synthesized CAN zeolite (5 M NaOH).

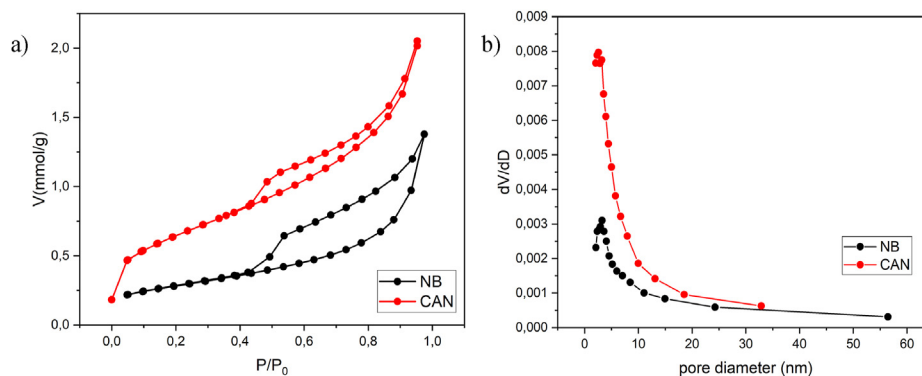


Fig. 3. a) The N₂ isotherm adsorption–desorption characteristics; b) pore size distribution analysis of Toraja natural bentonite and synthesized CAN zeolite.

Table 3. Surface area and porosity of Toraja natural bentonite and synthesized CAN zeolite (5M NaOH).

Materials	BET surface area (m ² /g)	Total pore volume	Average pore size (nm)
NB	22.05	0.05	8.67
CAN	51.97	0.07	5.38

interactions between Pb²⁺ and Fe³⁺ ions and the zeolite surface.

The adsorption of metal ions in solution is significantly impacted by pH because metal cations and H⁺ ions compete for active sites on the

adsorbent surface [65]. The adsorption of Fe³⁺ ions reached its optimal pH at 3, while Pb²⁺ ions achieved theirs at 5 (Fig. 4b). The removal effectiveness of Fe³⁺ ions is greater under acidic conditions than Pb²⁺, which is related to the higher charge on Fe³⁺, making it more prone to hydration in aqueous environments and more stable at low pH. Both Fe³⁺ and Pb²⁺ ions exhibit similar adsorption behaviors, with maximum effectiveness occurring at a pH that is neither excessively acidic nor basic. Under very acidic conditions, competition between H⁺ ions and metal cations reduce metal ion binding on the

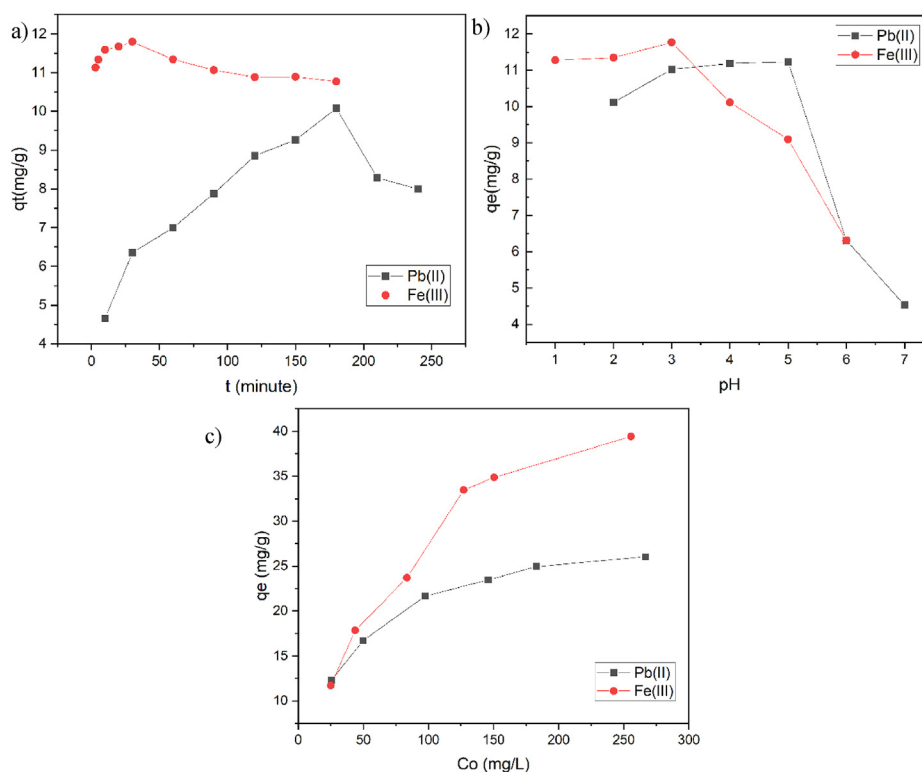
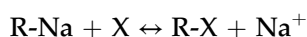


Fig. 4. Graph illustrating the correlation between a) adsorption time; b) pH of solution; c) concentration and the total of adsorbed Pb²⁺ and Fe³⁺ ions by synthesized CAN zeolite.

adsorbent. Excess H^+ ions may interact with the active sites of the material, obstructing metal ion adsorption. Conversely, at near-neutral pH, increased OH^- ion concentration can lead to metal ion precipitation as hydroxides, reducing adsorption efficiency [66].

Adsorption analysis reveals that increasing metal ion concentration enhances adsorption capacity [67]. The elevated concentration facilitates the migration of metal ions from the solution into the zeolite pores. The adsorption process is influenced by the absorbency and pore structure of zeolite. Metal ions penetrate the zeolite pores via interactions with the $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ structures within the zeolite framework. Metal ions confined within the pores and adhered to the zeolite contribute to the solution's decreased metal ion concentration. Fig. 4c shows that CAN synthetic zeolite adsorbs Fe^{3+} more effectively than Pb^{2+} . This difference is influenced by the physicochemical properties of the metal ions, as well as the surface characteristics and pore structure of the zeolite. In this study, the large pore size of the zeolite enables metal ions to penetrate the pore system and interact with active sites. Fe^{3+} ions have a greater hydration diameter and hydration energy than Pb^{2+} ; however, the higher adsorption capacity of the zeolite for Fe^{3+} ions can be attributed to the higher ionic charge, which generates a strong electrostatic attractive force toward negatively charged active sites. Additionally, the sizable pore size of the zeolite and its surface chemical affinity for Fe^{3+} further enhance adsorption efficiency [68].

The adsorption of heavy metals onto zeolites occurs through several processes, including ion exchange, diffusion, and interactions with hydroxyl groups [66,69]. Ion exchange transpires between heavy metal ions and the exchangeable cations within the zeolite framework. For instance, Na^+ ions participate in the following reaction:



Besides ion exchange, the adsorption mechanism involves interfacial interactions occurring on the zeolite surface. Interactions occur between hydroxyl groups and heavy metal ions, including hydrogen bonding and van der Waals forces. These interactions influence the stability of metal ions on the adsorbent surface. Moreover, diffusion mechanisms significantly contribute to the adsorption process, particularly with Pb^{2+} and Fe^{3+} ions. Metal ions traverse the channels and cavities of zeolite micropores according to the principle of

selective screening, referred to as molecular sieving events [69].

3.3. Adsorption isotherm

Fig. 5 illustrates the linear adsorption isotherm curves derived from the Langmuir, Freundlich, and Sips models for the adsorption of Pb^{2+} and Fe^{3+} ions by the synthesized CAN zeolite. Table 4 presents the derived linear isotherm parameters. The study shows that the Langmuir isotherm model offers the best fit for the adsorption of Pb^{2+} and Fe^{3+} ions by synthesized CAN zeolite, with R^2 values closest to 1.

The Langmuir adsorption model outlines the process of a single saturated layer of solute molecules adhering to the adsorbent surface while disregarding any lateral interactions between adsorbed molecules. Adsorption occurs on a structurally homogenous adsorbent, forming a monolayer on the particle surface [41,70]. The separation factor (R_L) for metal ion adsorption by synthesized CAN zeolite, derived from Fig. 5a, ranged from 0.02 to 0.1, indicating values less than 1. The R_L values suggest that the Langmuir model accurately characterizes the adsorption [71] of Pb^{2+} and Fe^{3+} ions by the synthesized CAN zeolite. The maximum adsorption capacity for both metal ions was determined using the Langmuir equation. The adsorption capacity for Pb^{2+} was 26.52 mg/g, whereas Fe^{3+} had an adsorption capacity of 42.02 mg/g, exceeding that of Pb^{2+} . The ionic radii of Pb^{2+} and Fe^{3+} are 0.119 nm and 0.065 nm, respectively.

The difference in radius affects the hydration diameter of the ions, where Pb^{2+} ions have a smaller hydration diameter than Fe^{3+} ions. In theory, ions with smaller hydration diameters are more easily adsorbed because they experience lower diffusion resistance. However, the results of this study did not fully follow this trend. Zeolite CAN exhibits a higher adsorption capacity for Fe^{3+} than for Pb^{2+} . These findings align with previous studies suggesting that ions with larger radii and higher molecular masses, such as Pb^{2+} , may saturate adsorbent pores or active sites more rapidly, thereby reducing adsorption capacity [70,72].

Fig. 5b and Table 4 illustrate the adsorption effects of Pb^{2+} and Fe^{3+} ions using the synthesized CAN zeolite, integrating with the Freundlich isotherm model. The Freundlich isotherm describes the adsorption process on heterogeneous surfaces [43,73]. The heterogeneity factor ($n = 1/n_f$) correlates with the adsorption intensity, which depends on the material's heterogeneity. According to Fig. 5b and Table 4, the value of $1/n_f$ for both ions is less than 1.

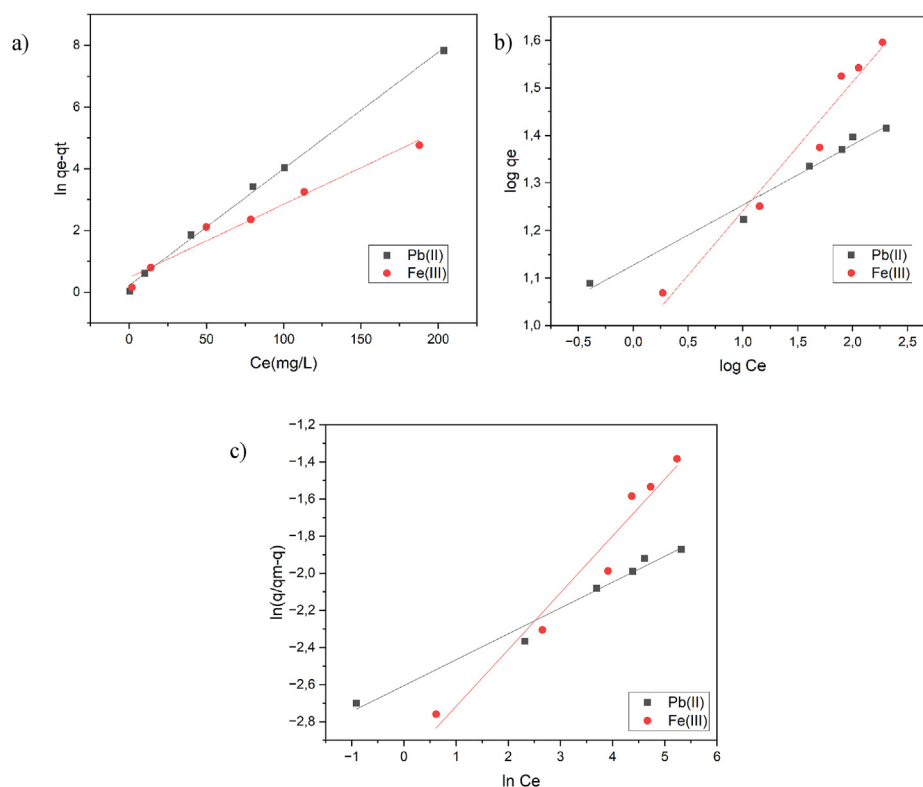


Fig. 5. Linear adsorption isotherms of a) Langmuir, b) Freundlich, and c) Sips for the adsorption of Pb^{2+} and Fe^{3+} metal ions by synthesized CAN zeolite.

Table 4. Specifications of the linear adsorption isotherm for Pb^{2+} and Fe^{3+} ions adsorbed by synthesized CAN zeolite.

Model	Parameters	Metal ions	
		Pb^{2+}	Fe^{3+}
Langmuir	K_L	0.1712	0.0503
	q_m	26.5252	42.0168
	R^2	0.9978	0.9734
Freundlich	K_f	13.4153	9.3584
	n_f	7.9177	3.6928
	R^2	0.9821	0.9674
Sips	K_s	0.0965	0.0366
	n_s	1,1787	3.2669
	q_m	194.9483	196.626
	R^2	0.9803	0.9615

The $1/n_f$ values suggest that Pb^{2+} and Fe^{3+} adsorption on synthesized CAN zeolite conforms to the Freundlich isotherm model, indicating favorable adsorption [74].

The Sips isotherm model (Fig. 5c) integrates the Langmuir and Freundlich equations to enhance understanding of the behavior of homogeneous and heterogeneous adsorption systems [75]. Furthermore, the Sips model addresses the shortcomings of the Freundlich isotherm model at elevated adsorbate concentrations. The Sips model resembles the Freundlich isotherm at low adsorbate concentrations.

Nevertheless, the Langmuir isotherm [42] suggests that single-layer adsorption is likely when a substantial amount of adsorbate is present. The q_{\max} values for Pb^{2+} and Fe^{3+} ions derived from the Sips model exceeded those obtained from the Langmuir model (refer to Table 4).

The adsorption isotherms were modeled using nonlinear equations for the Langmuir, Freundlich, and Sips models to improve result accuracy. Fig. 6a and b illustrate the three adsorption models for Pb^{2+} and Fe^{3+} ions on synthesized CAN zeolite, with adsorption details provided in Table 5. Modelling adsorption isotherms with non-linear equations primarily evaluates the Sum of Squared Residuals (SSR). SSR measures the model's accuracy in comparison to the experimental data. A lower SSR value signifies enhanced consistency between the model and the empirical data.

The examination of SSR values (see Table 5) suggests that the Sips isotherm model best describes the adsorption of Pb^{2+} and Fe^{3+} ions by synthesized CAN zeolite, as it presents the lowest SSR value relative to the Langmuir and Freundlich models. The Sips isotherm model quantifies heterogeneity in the adsorption system. This heterogeneity can arise from the properties of the solid (adsorbent), the adsorbate, or a combination of both, as shown by the

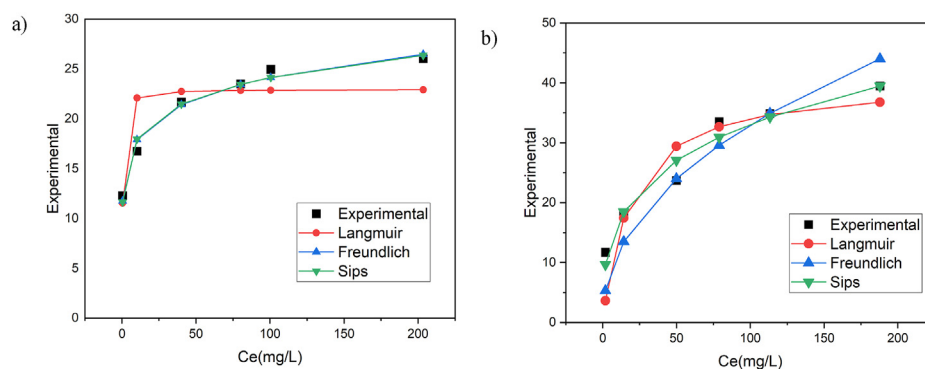


Fig. 6. Non-linear adsorption isotherms of Langmuir, Freundlich, and Sips for the adsorption of, a) Pb^{2+} and b) Fe^{3+} metal ions by synthesized CAN zeolite.

Table 5. Specifications of the non-linear adsorption isotherm for Pb^{2+} and Fe^{3+} ions adsorbed by synthesized CAN zeolite.

Model	Parameter	Metal ions	
		Pb^{2+}	Fe^{3+}
Langmuir	K_L	2.5138	0.0533
	q_m	22.9545	40.4532
	R_L	0.0056	0.1858
	R^2	0.9993	0.9734
	SSR	44.6591	105.9124
Freundlich	K_f	13.2424	4.0098
	n_f	7.6810	2.1855
	R^2	0.9971	0.9830
	SSR	2.5228	96.0983
	SSR	2.5228	96.0983
Sips	K_s	0.0725	9.39×10^{-5}
	n_s	0.1449	0.3422
	q_m	194.9483	196.626
	R^2	0.9949	0.9914
	SSR	2.1990	22.9527

heterogeneity exponent n_s . If n_s is close to 1, it suggests that the solid has relatively homogeneous adsorption sites. If $n_s < 1$, the system exhibits greater heterogeneity and corresponds more closely with the Freundlich isotherm model. The n_s values for Pb^{2+} and Fe^{3+} ions indicate that the adsorption system aligns more closely with the Freundlich isotherm model, reflecting the characteristics of a heterogeneous adsorbent surface. Modeling results revealed that the Sips isotherm model has a maximum adsorption capacity of 194.9 mg/g for Pb^{2+} and 196.6 mg/g for Fe^{3+} . The comparison of maximum adsorbed amount of Pb^{2+} and Fe^{3+} ions on examined materials surface with others zeolites and natural minerals is presented in Table 6.

3.4. Adsorption kinetics

Fig. 7 illustrates the experimental and predicted outcomes using linear first-order and second-order pseudo-kinetic models for the adsorption of Pb^{2+} and Fe^{3+} ions by synthesized CAN zeolite. The parameters of these kinetic models are outlined in

Table 6. Comparison of adsorption capacity of metal ions on the examined solids surface with others zeolite and natural mineral.

Adsorbent	Adsorbate	Adsorption capacity	References
CAN zeolite from Kaolinite	Pb^{2+}	192	[76]
K-type zeolite from Fly ash	Pb^{2+}	55.53	[77]
Azide CAN from zeolite K	Pb^{2+}	52.63	[78]
Zeolite A from waste LD-slag	Fe^{3+}	27.55	[79]
Zeolite 4A waste	Fe^{3+}	95.18	[80]
Natural zeolite	Fe^{3+}	1.18	[81]
Brown bentonite	Fe^{3+}	16.65	[82]
Natural bentonite from Slovakia	Pb^{2+}	32.68	[83]
Na-type zeolite from Coal Gastification slag	Pb^{2+}	10.00	[66]
CAN zeolite from Toraja Bentonite	Pb^{2+}	194.95	This study
CAN zeolite from Toraja Bentonite	Fe^{3+}	196.63	This study

Table 7. The pseudo-second-order kinetic model demonstrated a better fit in describing the adsorption of Pb^{2+} and Fe^{3+} ions, as indicated by higher R^2 values compared to the pseudo-first-order kinetic model. Moreover, the q_e values predicted by this model closely aligned with the q_e values derived from experimental observations, confirming that the pseudo-second-order model was the best fit [66,84]. These findings confirm that the removal of Pb^{2+} and Fe^{3+} ions using synthesized CAN zeolite occurs through a chemisorption mechanism. This mechanism results from a combination of electrostatic forces and chemical interactions, including covalent bonding, ion exchange, and complexation between the metal ions and the zeolite surface.

3.5. Adsorption selectivity

The adsorption selectivity was assessed to determine the capacity of synthesized CAN zeolite to

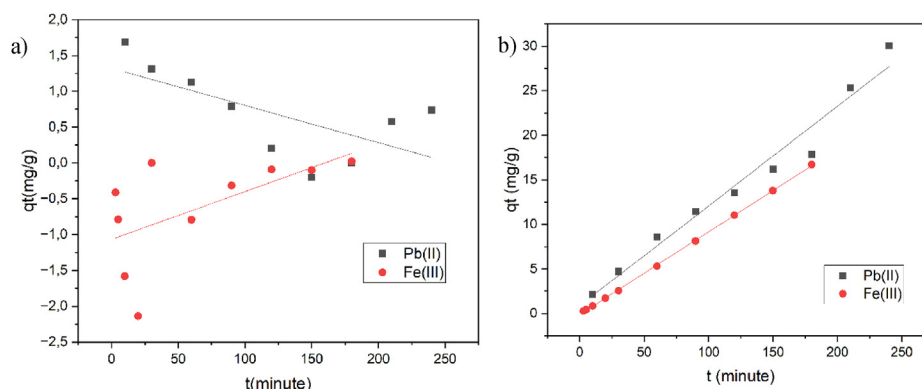


Fig. 7. Kinetics of Pb^{2+} and Fe^{3+} metal ion adsorption by synthesized CAN zeolite. a) pseudo-first-order; b) pseudo-second-order.

Table 7. Specifications of the adsorption kinetics for Pb^{2+} and Fe^{3+} ions adsorbed by synthesized CAN zeolite.

Metal ions	Pseudo-first order			Pseudo-second order		
	K_1	q_e	R^2	K_2	q_e	R^2
Pb^{2+}	0.0052	3.7449	0.4452	0.0110	8.9445	0.9694
Fe^{3+}	0.0067	0.3445	0.3522	0.0010	10.7757	0.9997

adsorb Pb^{2+} and Fe^{3+} ions in both single-ion and binary systems. In the binary system, solutions containing Pb^{2+} and Fe^{3+} ion concentrations of 25 mg/L each in 50 mL of solution were prepared and processed under the optimal conditions for each metal ion.

Fig. 8a illustrates the adsorption selectivity of Pb^{2+} and Fe^{3+} ions in a single system, presented as the relationship between pH and adsorption capacity. The optimum pH of each ion indicates that pH influences the adsorption selectivity [85] of Pb^{2+} and Fe^{3+} ions. Fe^{3+} ions exhibit greater selectivity at acidic pH compared to Pb^{2+} ions due to Fe^{3+} 's elevated hydration potential, which facilitates the formation of aqua complexes in the presence of sufficient H^+ ions [86]. This condition also inhibits the hydrolysis of Fe^{3+} ions, which typically hydrolyze at pH levels around neutrality, allowing Fe^{3+} to interact effectively with zeolite active sites [87].

Conversely, Pb^{2+} ions exhibits greater stability in its ionic form near neutral pH, making it more accessible for adsorption by zeolites. The interaction between zeolite and metal with metal ions is more favorable at near-neutral pH than at acidic pH, when the protonation of zeolite's active groups occurs [88,89]. In addition, the hydration diameter of metal ions significantly influences the adsorption selectivity of Pb^{2+} and Fe^{3+} ions by CAN zeolites [90]. Compared to the zeolite pore size (5.38 nm), the hydration diameter of Pb^{2+} ions is approximately 0.40 nm, while that Fe^{3+} ions is roughly 0.90 nm. Heavy metal ions can readily penetrate the zeolite pore and interact with the cations within, depending on zeolite pore size and the metal hydration radius.

Adsorption selectivity is also affected by the electronegativity of the metal [91]. Pb^{2+} ions exhibit a higher electronegativity (2.33) than Fe^{3+} (1.83), which presumably enhances their interaction with the active sites of the adsorbent. A higher electronegativity of an ion correlates with stronger interaction with the zeolite surface [92].

Although Pb^{2+} has a smaller hydration diameter and higher electronegativity than Fe^{3+} ions, experimental results show that Fe^{3+} is more selectively adsorbed than Pb^{2+} in the single system (see Fig. 8a). Fe^{3+} is more selective than Pb^{2+} , which may

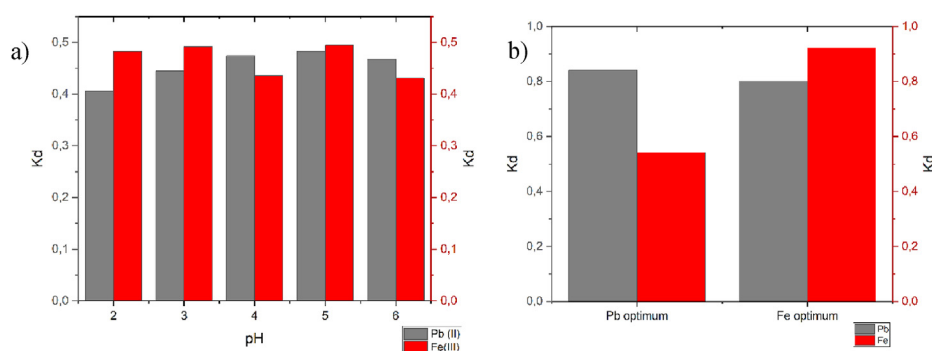


Fig. 8. Adsorption selectivity of metal ions in (a) single metal systems; (b) binary systems.

be due to the size mismatch between Pb^{2+} hydration and zeolite pores, allowing ions with larger diameters to be more easily adsorbed [68]. In addition, the larger charge of Fe^{3+} ions lead to stronger electrostatic interactions with the zeolite active groups, increasing the adsorption efficiency.

The difference in adsorption selectivity in the binary system is shown in Fig. 8b. In the Fe^{3+} - Pb^{2+} system, Fe^{3+} acts as the target ion, while Pb^{2+} is the competitor ion. The adsorption selectivity of Pb^{2+} is 0.80, and Fe^{3+} is 0.92, showing a high value at Fe^{3+} 's optimum condition. CAN zeolite more effectively adsorb Pb^{2+} due to its lower hydration energy and higher electronegativity than Fe^{3+} [93]. In contrast, in the Pb^{2+} - Fe^{3+} system, Pb^{2+} acts as the target ion, while Fe^{3+} is the competitor ion. The adsorption selectivity of Fe^{3+} (0.54) is lower than that of Pb^{2+} (0.84), indicating that Fe^{3+} is less competitive with Pb^{2+} . CAN zeolite only adsorbs Fe^{3+} if the Pb^{2+} concentration is insufficient to achieve adsorption saturation. Based on the selectivity of the binary system, it can be concluded that Pb^{2+} has a higher adsorption selectivity than Fe^{3+} in the presence of competitor ions.

3.6. Desorption and regeneration

The reuse of adsorbents is an essential factor in assessing their potential applications. This study conducted adsorption–desorption experiments to evaluate recovery efficiency and assess the reusability of the synthesized zeolite. The desorption performance of the synthesized CAN zeolite was evaluated using 0.1 M HNO_3 , 0.1 M Na_2EDTA , and H_2O solutions. The desorption behavior of Pb^{2+} and Fe^{3+} ions from the synthesized CAN zeolite is illustrated in Fig. 9a.

According to the desorption data in Fig. 9a, the 0.1 M HNO_3 solution demonstrated the highest efficacy in desorbing Pb^{2+} and Fe^{3+} ions, achieving 85.09 % and 77.18 %, respectively. The acidic condition causes protonation of the adsorbent surface, further facilitating the release of positively charged

metal ions [94]. Sari et al. [95] indicated that HNO_3 serves as an effective desorption agent due to the high-affinity NO_3^- ions, which promote the formation of stable complexes with Pb^{2+} and Fe^{3+} ions. During adsorption, cations on the CAN zeolite are substituted by Pb^{2+} and Fe^{3+} ions. Conversely, in the desorption phase, Pb^{2+} and Fe^{3+} ions are released from zeolite surface and re-enter the solution.

Na_2EDTA desorption agent effectively releases Pb^{2+} and Fe^{3+} ions, achieving 77.20 % and 66.37 % desorption, respectively. The desorption process occurs as ligands in the Na_2EDTA solution donate electrons through two oxygen and four nitrogen atoms, forming a stable six-ringed chelate complex. The process makes Na_2EDTA an effective desorption agent for metal ions [96,97]. In contrast, desorption using H_2O shows a lower percentage because it cannot participate in competitive ion exchange. As a neutral solvent, H_2O does not provide sufficient H^+ ions to protonate the adsorbent surface, causing metal ions to remain bound to the zeolite. These results align with research conducted by Ref. [98], which reported that desorption of metal ions is more effective in acidic conditions compared to neutral conditions.

The reuse study of CAN zeolite was conducted twice using HNO_3 , identified as the most suitable desorption agent from the previous experiment, as illustrated in Fig. 9b. The adsorption efficiency of Pb^{2+} and Fe^{3+} metal ions by CAN zeolite decreased significantly from the first to the second cycle, by approximately 25 %. After two regeneration cycles, efficiency declined considerably, possibly due to structural damage caused by exposure to acidic media [99]. These results are in line with research conducted [100], which examined the regeneration of Pb^{2+} metal ions on zeolites from coal fly ash using HNO_3 solution. The study reported a decreased in adsorbent mass, indicating physical changes and structural damage due to acid exposure.

In addition, the lower desorption efficiency compared to the adsorption efficiency in the

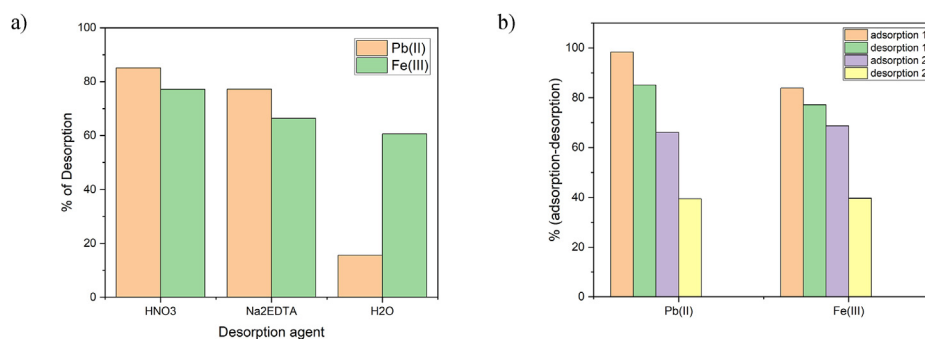


Fig. 9. (a) Desorption of Pb^{2+} dan Fe^{3+} ion using desorption agent; (b) Reuse cycle of zeolite CAN on Pb^{2+} and Fe^{3+} adsorption–desorption.

adsorption–desorption cycle indicates heterogeneity on the zeolite surface [101,102]. Some active sites on zeolites exhibit higher binding energy, allowing ions to form strong bonds that are difficult to desorb [103]. These findings suggest that the synthesized CAN zeolite still requires further modification to improve its reusability. A similar study [104] also tested the regeneration of CFAZ magnetite composites for Pb^{2+} metal ions over 3–4 regeneration cycles.

4. Conclusion

Cancrinite zeolite was effectively synthesized from Toraja natural bentonite via hydrothermal process with the incorporation of sodium hydroxide. This zeolite has proven effective as an adsorbent for removing Pb^{2+} and Fe^{3+} ions from wastewater. The optimization of process parameters, such as contact time, pH level, and initial concentration of metal ions, enhanced adsorption capacity until equilibrium was attained. Adsorption adhered to the Sips isotherm model, whereas the kinetics of adsorption conformed to a pseudo-second-order model. Selective experiments indicated that the zeolite exhibited more excellent selectivity for Fe^{3+} compared to Pb^{2+} in a single system but was more selective for Pb^{2+} than Fe^{3+} in a binary system. Consequently, CAN zeolite exhibits potential as an effective adsorbent for treating wastewater containing Pb^{2+} and Fe^{3+} ions. For future research, it is recommended to conduct a detailed investigation the mechanism of metal ion adsorption by zeolites, particularly in relation to pH conditions, as this factor has a significant impact on adsorption. To assess the adsorbent ability of CAN zeolite in alignment with eco-friendly and cost-effective principles, it is crucial to further examine the regeneration and reutilization of synthetic zeolite. Additionally, the production of composites with synthetic zeolites may present an opportunity to enhance adsorption ability.

Ethics information

This study involved no human or animal subjects. All experiments focused on synthesizing and characterizing zeolites from natural minerals and evaluating their adsorption of $\text{Pb}(\text{II})$ and $\text{Fe}(\text{III})$ ions in aqueous solutions. All procedures complied with institutional safety and environmental standards.

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Conflicts of interest

The authors assert that they possess no recognized competing financial interests or personal relationships that may have seemingly influenced the work presented in this study.

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