Facile Synthesis of Zeolite A from Molybdenum Mine Tailings and Its Potential Application in Mine Reclamation

Hyeseong Kim[1], Hyunjin In[1], Jiyun Jeong[1], Sang Yun Lee[1], Uikyu Choi[2], Youn Soo Oh[2]

[1]*Sanha Engineering & Construction Co., cometk94@naver.com*

[2]*Korea Mine Rehabilitation & Mine Resources Co., ukchoi@komir.or.kr*

Abstract

This study presents a facile and scalable synthesis of zeolite A from molybdenum mine tailings using simple alkaline fusion (550 °C) and low-temperature crystallization (30–90 °C), avoiding energy-intensive hydrothermal process. Pilot-scale production achieved a ≈90% yield. The synthesized zeolite A demonstrated a high cation exchange capacity (292 meq/100 g). In batch tests, it removed ≥99.9% of aqueous cadmium and 13.5% of fluoride, and when mixed with limestone, completely suppressed cadmium and reduced arsenic leaching in contaminated soils. This approach sustainably converts potentially problematic mine waste into functional materials for environmental remediation.

Keywords: Synthetic zeolite, mine waste, mine reclamation, adsorbent, soil stabilizer

Introduction

The global mining industry inadvertently generates massive volumes of mine tailings and waste rock. Typically stockpiled near extraction sites, these by-products pose severe environmental risks. In the Republic of Korea, exposed mining waste in abandoned areas has caused significant soil and water pollution, often involving toxic inorganic contaminants like arsenic (As), cadmium (Cd), and fluoride (F) (Lim *et al.* 2015).

Historically, mining waste management relied on physical containment such as capping. However, these methods require large land areas, do not fundamentally treat contaminants, and are vulnerable to extreme weather, leading to potential re-release of contaminants. Consequently, a transition toward fundamental solution like recycling is imperative.

Because mine waste is rich in silicon (Si) or aluminium (Al), they could be excellent precursors for synthesizing functional materials like zeolites or ceramic filters (Huang *et al.* 2024). Zeolites, with their porous structure and exceptional cation exchange capacity (CEC), are highly effective for environmental remediation. Converting these liabilities into secondary resources offers a dual benefit: mitigating environmental threats while generating functional materials.

However, conventional zeolite synthesis often relies on energy-intensive or unscalable steps, such as high-temperature hydrothermal treatment or centrifugation (Park *et al.* 2000; Jeon *et al.* 2025), hindering commercialization. Therefore, developing highly scalable methods is crucial.

In this study, we propose a facile and scalable recycling technology to convert molybdenum (Mo) mine tailings into synthetic zeolite A via a simplified alkaline fusion (550 °C) and low-temperature crystallization (30–90 °C) process. This research aims to provide a functional environmental material capable of efficiently adsorbing contaminants and stabilizing soils, offering a scalable pathway for sustainable mine reclamation.

Methods

Raw Materials and Synthesis Procedure of Zeolite A

Mo mine tailings were dried and milled to ensure a uniform particle size. The raw tailings were classified as non-hazardous general waste, with trace metal and leaching

concentrations strictly satisfying the Korean Soil Contamination Warning Standards for Area 1 and Waste Control Act thresholds, respectively (MCEE 2025a, 2025b). Zeolite A was synthesized via an alkaline fusion methods. The tailings were homogeneously mixed with NaOH using either a mortar (lab-scale) or a ribbon mixer (pilot scale), followed by heating at 550 °C for 2 h to decompose mineral phases. Sodium aluminate ($NaAlO_2$) was added to optimize the SiO_2/Al_2O_3 molar ratio. The fused mixture was hydrated and subjected to a two-stage reaction: aging at 30 °C for 3 h and crystallization at 90 °C for 3 h. The final product was washed, filtered and dried. Following lab-scale optimization of the SiO_2/Al_2O_3 and NaOH/tailings ratio (Fig. 1), the process was successfully scaled up in a pilot system, producing 10–20 kg per batch with a ≈90% yield (Fig. 2).

Characterization of Synthesized Materials

The samples were characterized using X-ray diffraction (XRD) for crystalline phases, X-ray fluorescence (XRF) for chemical composition, and scanning electron microscopy (SEM) for surface morphology. Cation exchange capacity (CEC) was measured using the standard 1 N ammonium acetate method (detection limit: 0.1 mg/L; error margin: ±20%) to assess ion-exchange potential. Commercial synthetic zeolite A was also analysed under identical conditions for comparison.

Environmental Performance Evaluation

Duplicate batch experiments were conducted to evaluate the environmental remediation performance (Fig. 3):

Batch Adsorption: To evaluate the adsorption capacity for both cationic and anionic species, Cd and F were selected as target contaminants, respectively. Separate 10 mg/L solutions with a 1 g/L adsorbent dosage were agitated at 300 rpm for 2 h before measuring residual ion concentrations.

Soil Stabilization: Batch leaching tests followed KOMIR protocols to assess As and Cd, selected for their severe toxicity, high mobility, and strict regulatory limits (KOMIR 2022). Artificially contaminated soil (120.3 mg/kg As, 23.5 mg/kg Cd) was prepared using high-purity standard

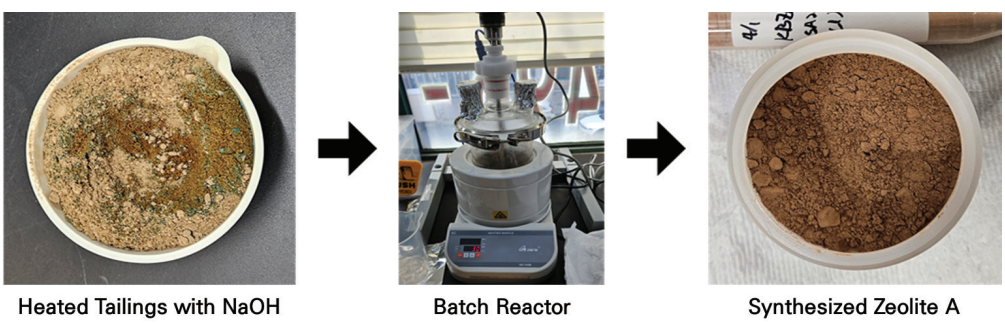

Figure 1 *Synthesis process of zeolite A by batch reactor.*

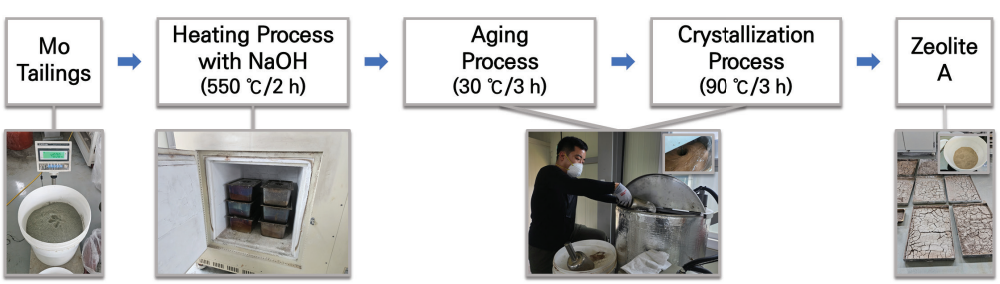

Figure 2 *Pilot production with a 200 L reactor.*

Figure 3 *Batch-scale experiment of product.*

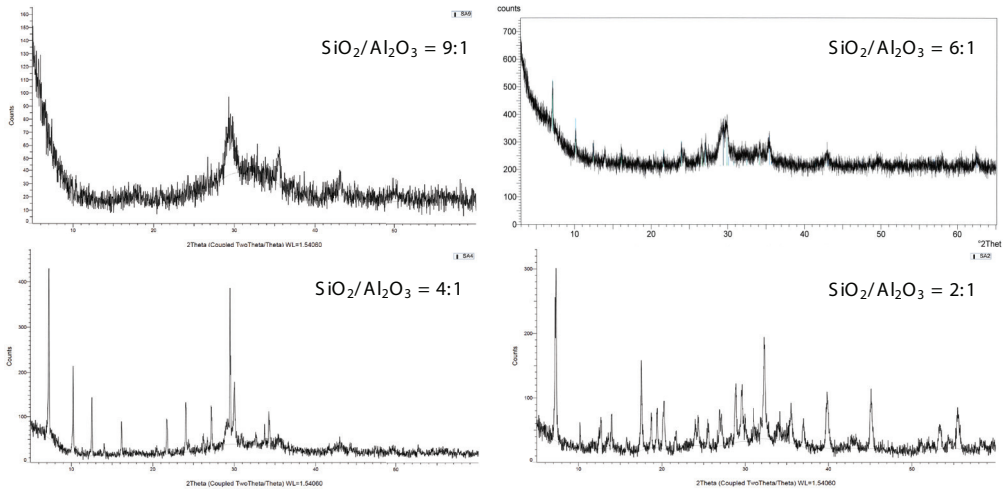

Figure 4 *XRD patters of the synthesized zeolites according to SiO_2/Al_2O_3 molar ratio.*

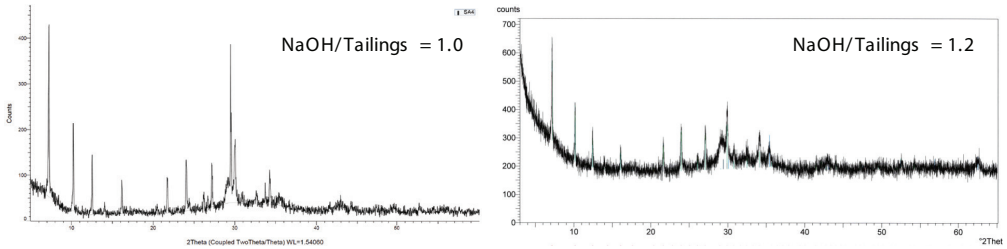

Figure 5 *XRD patterns of synthesized zeolite A at different NaOH/Tailings mass ratio.*

solutions and equilibrated for 24 h. Three experimental groups were compared: (1) an untreated control, (2) a limestone-zeolite A mixture (60:40 wt%), and (3) a limestone-steel slag mixture (60:40 wt%) as a reference. Stabilizers were applied at 5 wt% of the soil. Samples were periodically collected during the 36 h agitation.

Results and Discussion

XRD analysis determined the optimal SiO_2/Al_2O_3 molar ratio for zeolite A synthesis to be 4:1 (Fig. 4). Ratios ≥ 5:1 lacked sufficient aluminium for framework formation, while a ratio of 2:1 exhibited decreased peak clarity, indicating excessive alumina hindered crystallization. With the SiO_2/Al_2O_3 ratio fixed at

4:1, increasing the NaOH/tailings mass ratio to 1.2 deteriorated synthesis, likely because excessive OH- ions interfere with structure formation (Fig. 5).

Regarding the pilot-produced material, XRD confirmed the primary (7.2°) and characteristic peaks (10.2°, 12.5°, 16.1°, 21.7°, 24.0°, 27.1°, 30.0°, 34.2°) of standard zeolite A (Treacy and Higgins 2001), and SEM revealed the typical cubic morphology with sharp edges (Pan *et al.* 2025), verifying the successful pilot-scale synthesis (Fig. 6).

XRF analysis revealed residual impurities such as Fe_2O_3, CaO, and MgO from the tailings in the synthesized product (Table 1). The SiO_2/Al_2O_3 molar ratio (4.2–4.5) slightly exceeded the theoretical value of commercial zeolite A, likely due to unreacted silicate phases, residual tailings impurities, and inherent XRD instrumental variations (Pan *et al.* 2025).

To assess ion-exchange reliability, CEC was measured across three separate pilot batches, yielding an average of 292 meq/100 g (Table 2). While individual values exhibited variability (258.11-325.43 meq/100 g), this deviation falls within the analytical error margin (±20%) of the CEC measurement method, alongside minor contributions from the raw tailings' inherent mineralogical heterogeneity and pilot-scale process fluctuations. Despite these variations, the synthesized zeolite's average CEC surpassed that of commercial synthetic zeolite A (283 meq/100 g) evaluated under identical conditions.

In batch adsorption test, the product exhibited Cd removal (≈100% at 10 mg/L for 2 h) comparable to commercial zeolite (Fig. 7a). Notably, it demonstrated a 5.6-fold higher F removal efficiency (13.5% vs. 2.4%) despite the zeolite framework's inherent electrostatic repulsion toward anions (Wang and Peng 2010) (Fig. 7b). For soil stabilization, untreated soil leachates averaged 2.70 mg/L for As and 0.37 mg/L for Cd, exceeding Korean Waste Control Act limits (1.5 and 0.3 mg/L, respectively) (MCEE 2025b). The limestone-zeolite A mixture suppressed As leaching to 0.90 mg/L (Fig. 8a), and completely immobilized Cd over 36 h (Fig. 8b), matching the conventional limestone-steel slag combination. While adding limestone slightly increased soil pH from 6.3 to 7.1, pH differences between zeolite and steel slag treatments were negligible (Table 3). This confirms the immobilization efficiency is not merely due to pH-induced precipitation, highlighting the product's potential for environmental remediation.

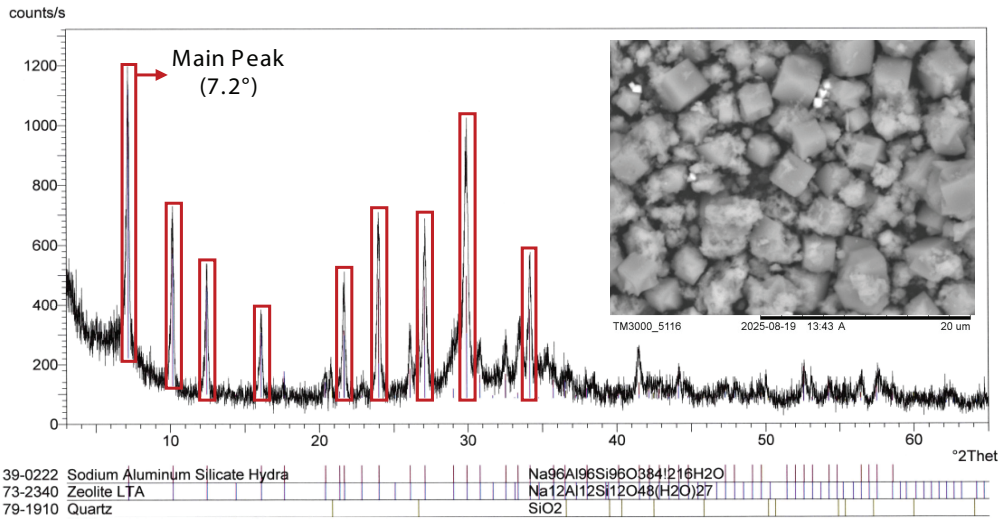

Figure 6 *XRD patterns and SEM image (inset) of synthesized zeolite A from pilot-scale production.*

Table 1 XRF analysis of fabricated zeolite A from pilot production (Unit: %, by mass).

Contents	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	MnO	P_2O_5
Tailings	41.80	7.83	11.99	25.47	3.08	0.78	0.23	0.30	1.02	0.08
Product(1)	37.77	14.18	8.02	12.21	1.57	0.30	11.74	0.18	0.68	0.03
Product(2)	33.18	13.36	7.99	13.04	1.45	0.30	15.88	0.18	0.68	0.04

- Minor elements were not detected as their concentrations were below the detection limit of the fusion method employed for major oxide quantification.
- Analysed from two separate pilot batches.

Table 2 CEC measurement of fabricated zeolite A from pilot production (unit: meq/100 g).

Contents	Product(1)	Product(2)	Product(3)	Average
Exchangeable-Mg	1.04	0.10	0.53	0.56
Exchangeable-Ca	5.51	0.20	1.49	2.40
Exchangeable-K	2.71	1.40	1.80	1.97
Exchangeable-Na	316.17	292.04	254.29	287.50
Total	325.43	293.74	258.11	292.43

- Measured from three separate pilot batches.
- Commercial synthetic zeolite A CEC: ~283 meq/100 g

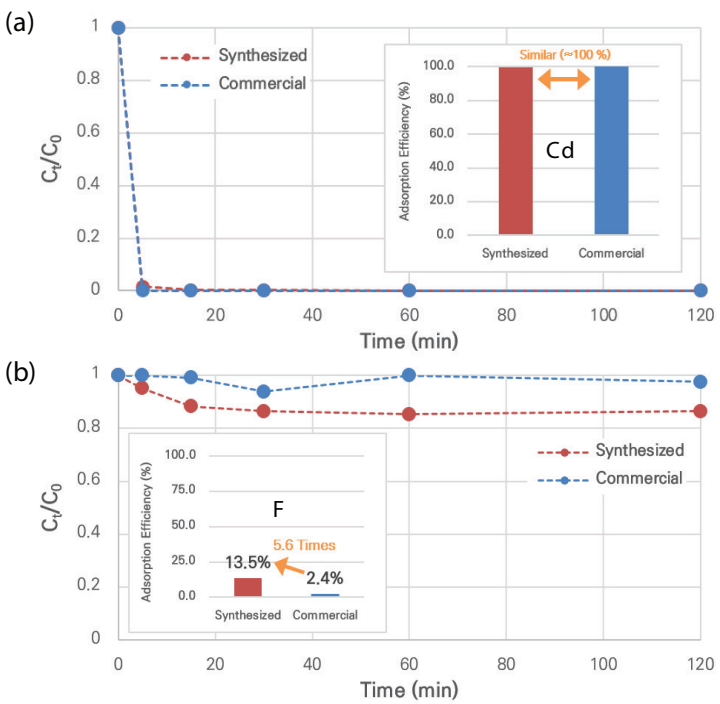

Figure 7 Batch-scale adsorption performance of the synthesized product for (a) Cd and (b) F.

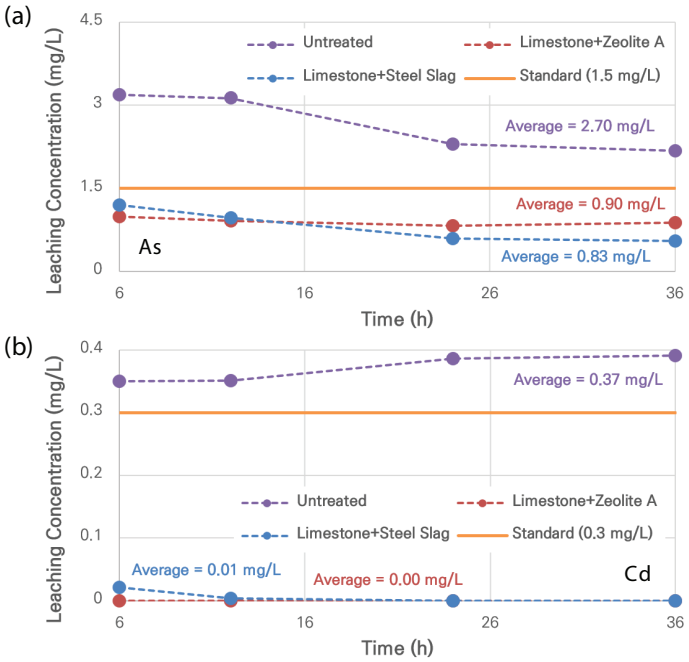

Figure 8 *Stabilization performance for (a) As and (b) Cd in soil.*

Table 3 *Changes in pH of soil leachates during the soil stabilization batch test.*

Retention Time (h)	Untreated	Limestone+Product	Limestone+Steel slag
6	6.3	6.9	6.8
12	6.4	7.0	6.9
24	6.3	7.1	7.3
36	6.1	7.2	7.3
Average	6.3	7.1	7.1

Conclusions

This study demonstrated a facile and scalable method for synthesizing zeolite A from Mo mine tailings without high-pressure hydrothermal treatment or centrifugation. By optimizing synthesis conditions (NaOH/tailings mass ratio of 1.0; SiO_2/Al_2O_3 molar ratio of 4:1), the process was successfully scaled up in a 200 L pilot reactor. This system produced 10–20 kg/batch of zeolite A with a ≈90% yield. Physicochemical analyses confirmed the crystal structure and a high CEC (292 meq/100 g), comparable to commercial product. Finally, batch-scale experiments validated the product's excellent performance and potential as an environmental remediation material for both trace metal adsorption and soil stabilization.

Acknowledgements

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