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Pb-clinoptilolite as novel reactive material for As removal from water

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Our study addresses development and application of a novel reactive mineral-based material for remediation of arsenic-contaminated waters. Pb-modified zeolite was used for efficient arsenate removal, in which As is immobilized via precipitation of the low-solubility mimetite ($Pb_5(AsO_4)_3Cl$). The process is driven by controlled release of Pb^{2+} from zeolite, enabling its reaction with arsenate and chloride ions and resulting in mimetite precipitation on zeolite grains. Lead bound to zeolite exhibits binding strength sufficient to prevent Pb release into water, yet labile enough to sustain reaction.

The material was prepared from natural clinoptilolite, initially in Ca^{2+} form and converted to Na^{+} form. Pb^{2+} was subsequently sorbed onto Na-clinoptilolite and washed to remove excess and weakly bound lead. The resulting Pb-zeolite (70 g Pb/kg) was tested both as a powder and mechanically stable granules (d=2 mm).

XRD confirmed crystalline mimetite formation after reaction of Pb-zeolite with As-containing solutions, while SEM-EDS revealed porous mimetite crusts coating zeolite grains and granules surfaces.

Static batch experiments (0.1-50 mg As/L; pH 2-8; 0.5 g zeolite/40 mL solution) demonstrated rapid and highly efficient arsenate sequestration under optimal conditions (pH 6-8), with >90% removal achieved within minutes and approaching 99% after 1 h (Tab.1). Pb release was negligible, and removal remained high in the presence of competing ions. Granulated Pb-zeolite exhibited performance comparable to powder in static systems.

Dynamic fixed-bed column experiments confirmed suitability of granules for continuous operation. Column experiments were conducted using synthetic arsenate solutions (5 mg As/L) at flow rates of 0.5-1.5 mL/min, and 3.5 g or 6.25 g of Pb-zeolite. Increasing material mass and bed depth significantly delayed breakthrough and improved removal efficiency (Fig.1). Higher flow rates accelerated breakthrough due to reduced residence time, indicating transport-controlled precipitation kinetics. Long-term operation revealed logarithmic breakthrough behaviour governed by intraparticle Pb transport, with only ~10% of the theoretical Pb inventory consumed, indicating substantial remaining reactive capacity.

Performance was further validated using arsenic-contaminated natural waters, achieving >90% removal in static tests and 85-95% removal under dynamic conditions. Overall, Pb-modified zeolite provides robust arsenate removal, highlighting its practical and scalable potential for water treatment.

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Table 1. Time-dependent changes in arsenic concentration (mg/L) and corresponding removal efficiency (%) during static batch treatment using granulated Pb-zeolite.

Time [min]	Solution					
	1 mg As/L		5 mg As/L		Natural water - Złoty Stok	
	Residual As [mg/L]	As removal [%]	Residual As [mg/L]	As removal [%]	Residual As [mg/L]	As removal [%]
0	0.97	0	5.51	0	0.73	0
1	0.08	92	3.77	32	0.32	56
3	0.07	92	0.64	88	0.12	84
8	0.04	96	0.07	99	0.09	87
15	0.04	96	0.09	98	0.08	89
30	0.04	96	0.07	99	0.06	91
60	0.00	100	0.03	99	0.09	87
120	0.02	98	0.03	99	0.03	96
300	0.00	100	0.02	100	0.00	100

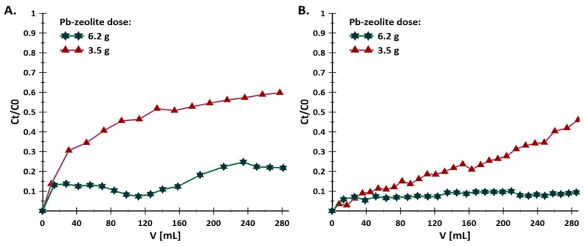


Figure 1. Breakthrough curves for As removal in Pb-zeolite granules fixed-bed columns (5 mg As(V)/L; pH = 8) at different material loadings and flow rates: (A) 1 mL/min and (B) 0.5 mL/min. Comparison illustrates the effect of reactive material mass and flow rate on removal performance.