Sorption-based lithium recovery from Aral Sea brines: process development and experimental validation

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This study presents the development and experimental substantiation of a sorption-based process for selective lithium recovery from highly mineralized brines. The process integrates alkaline precipitation of interfering cations with dynamic sorption using acetophosphate cellulose as a selective sorbent. The static exchange capacity of the developed sorbent was determined as 25.48 mg/g, with lithium desorption efficiency reaching 98.25% and sorbent stability maintained through 10 consecutive sorption-desorption cycles (93% capacity retention). The process demonstrates selective lithium extraction under moderate temperature conditions without toxic organic solvents, providing a scientific foundation for subsequent technological development and pilot-scale implementation. The results demonstrate both scientific significance and technological applicability of the proposed process.

Key words: lithium extraction, brine processing, acetophosphate cellulose, sorption, Mg/Li separation, Aral Sea, hydrometallurgy.

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1. Introduction

The rapid proliferation of electric vehicles, rechargeable battery systems, and grid-scale energy storage infrastructure has precipitated an unprecedented surge in global lithium demand. This market dynamic necessitates urgent diversification of lithium raw material sources and development of efficient extraction methodologies, particularly from low-concentration resources including natural and technogenic brines [1–3].

The primary scientific and technical challenge in lithium hydrometallurgy encompasses the enhancement of lithium ion recovery efficiency, improvement of separation selectivity relative to concomitant cations, maximization of sorbent capacity, and reduction of energy and reagent consumption during processing of low-grade brines. Among contemporary approaches, sorption and membrane methods offer particular promise for selective lithium concentration from complex multicomponent systems [4, 5].

Integrated membrane distillation (MD) and nanofiltration (NF) configurations have demonstrated the capability to produce lithium concentrates with mass fractions up to 4% Li, achieving water recovery efficiencies of 95% [6]. Combined technologies incorporating mechanoactivation, solid-liquid and liquid-liquid extraction have been developed for lithium and cobalt-containing waste processing [7]. Various sorption materials and ion-exchange resins have been reported to enable selective lithium extraction in the presence of Na^+, K^+, $Ca2^{2+}$, and Mg^{2+} while maintaining stability through multiple sorption-desorption cycles [8–10].

For mineral raw materials, including lithium-bearing micaceous ores, combined pyrometallurgical and hydrometallurgical approaches have been established, enabling subsequent sorptive lithium recovery and production of battery-grade lithium carbonate [11, 16]. The critical issue of magnesium/lithium separation has been addressed through ionic distillation, selective electrodialysis, and bipolar membrane electrodialysis, achieving Mg^{2+}/Li^+ ratio reduction below unity [12–14].

Despite these advances, the challenge of selective lithium extraction from highly mineralized brines with low Li^+content remains unresolved and requires further investigation focused on sorbent optimization and experimental validation of process conditions [15–18].

However, existing studies primarily focus on either sorbent development or separation techniques as independent processes, while insufficient attention has been given to the integration of chemical pre-treatment and

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sorption stages for highly mineralized brines with ultra-low lithium concentrations.

In particular, the role of controlled carbonate formation via CO_2 introduction in enhancing pre-treatment efficiency and improving downstream sorption selectivity has not been systematically investigated.

Therefore, there remains a critical need for an integrated process approach that combines feed conditioning with selective lithium capture under mild operating conditions.

The aim of this study is to develop and experimentally validate an integrated lithium recovery process from Aral Sea brines based on alkaline pre-treatment and selective sorption on acetophosphate cellulose.

The novelty of this work lies in the process-level integration of alkaline pre-treatment with controlled CO_2-assisted carbonate formation and selective sorption, enabling enhanced removal of competing ions prior to lithium capture.

In particular, the role of CO_2 introduction during the conditioning stage is investigated as a means of promoting calcium removal via carbonate precipitation and improving overall process selectivity.

2. Process concept and technological approach

The developed lithium recovery technology is designed as an integrated multi-stage process that combines chemically controlled pre-treatment with selective sorption and efficient regeneration. The process is specifically tailored for low-grade, highly mineralized brines with elevated Mg^{2+}/Li^{+} ratios.

Stage I: Pre-treatment via Alkaline Precipitation. This stage targets the removal of interfering multivalent cations (Fe^{3+}, Ni^{2+}, Co^{2+}, Mg^{2+}) through controlled hydroxide precipitation at elevated pH. The process exploits differential solubility products, enabling selective retention of Li^{+}, Na^{+}, K^{+}, and residual alkaline earth elements in solution while precipitating transition metal and magnesium hydroxides.

Stage II: Selective Sorption. The clarified brine undergoes dynamic sorption on acetophosphate cellulose in column configuration. This stage leverages the high selectivity of phosphate-functionalized cellulose for Li^{+} over competing cations, particularly the challenging Mg^{2+}/Li^{+}separation. The acetophosphate groups provide chelating sites with preferential affinity for lithium ions under controlled pH and flow conditions.

Stage III: Desorption and Sorbent Regeneration. Lithium-loaded sorbent is regenerated using dilute hydrochloric acid, producing a concentrated lithium eluate suitable for subsequent purification and lithium salt production. The regenerated sorbent maintains structural integrity and functional capacity for repeated operational cycles.

The process logic emphasizes: (i) upstream removal of major interferents to prevent sorbent poisoning; (ii) selective lithium capture under mild conditions; and (iii) efficient sorbent regeneration to ensure process economics and environmental sustainability.

The overall process flow diagram of the developed lithium recovery technology is presented in **Figure.**

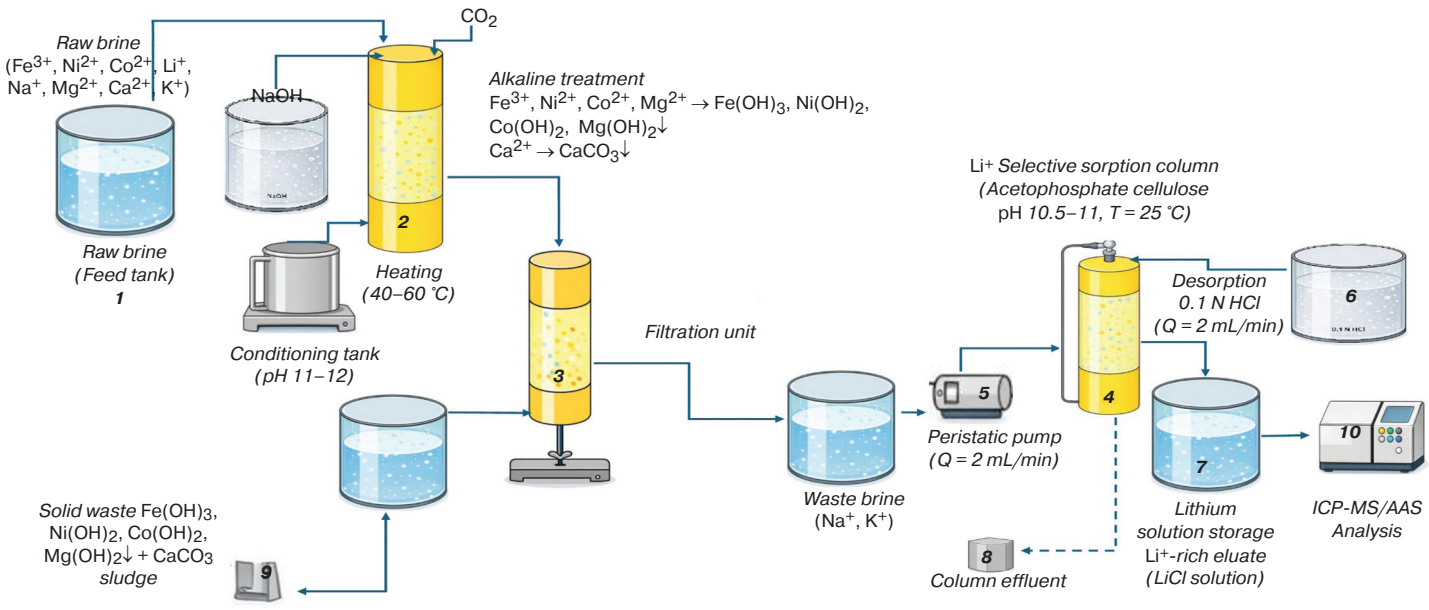

Figure. Schematic diagram of lithium extraction from brine via sorption–desorption process:
1 – raw brine; *2* – conditioning tank (alkaline pre-treatment); *3* – filtration unit; *4* – sorption column (acetophosphate-modified cellulose); *5* – peristaltic pump; *6* – desorption unit (0.1 N HCl); *7* – lithium-rich eluate; *8* – column effluent (waste brine); *9* – solid waste (hydroxide and carbonate sludge); *10* – analytical unit (ICP-MS/AAS)

The scheme represents a conceptual laboratory-scale process intended to illustrate the process logic; industrial flow rates, material balances, and equipment scale are not depicted.

2.1. Lithium Separation and Source of Carbon Dioxide (CO_2)

Role of Carbon Dioxide in the Process

CO_2 dissolution generates carbonate species depending on pH; under strongly alkaline conditions (pH > 10), CO_3^{2-} becomes the dominant species, enabling effective precipitation of Ca^{2+} as $CaCO_3$. This step reduces interfering ions and improves subsequent lithium sorption efficiency.

The process is governed by the following equilibria:

$$CO_2 + H_2O \rightleftharpoons H_2CO_3 \rightleftharpoons HCO_3^- \rightleftharpoons CO_3^{2-}$$

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3\downarrow$$

Selective removal of Ca^{2+} and Mg^{2+} is determined by differences in solubility products. Magnesium precipitates as $Mg(OH)_2$ ($Ksp = 5.6 \times 10^{-12}$), while calcium forms $CaCO_3$ ($Ksp = 3.3 \times 10^{-9}$). In contrast, lithium remains in solution due to the significantly higher solubility of its compounds ($Ksp(Li_2CO_3) \approx 8.2 \times 10^{-4}$). Controlled CO_2 introduction enhances calcium removal and improves overall process selectivity.

3. Materials and methods

3.1. Reagents and equipment

Cellulose acetate (purity 99.98%, Sigma-Aldrich, CAS 9004-35-7) and lithium hydroxide (purity 99.99%, Sigma-Aldrich, CAS 1310-65-2) were used for standard solution preparation. Orthophosphoric acid (95%) was employed without further purification. Elemental analysis was performed using inductively coupled plasma mass spectrometry (ICP-MS).

3.2. Sorbent synthesis

Acetophosphate cellulose was synthesized by reacting 10 g cellulose acetate with 6 mL 85% phosphoric acid in a 200 mL vessel, followed by addition of 100 mL double-distilled water. The mixture was heated to boiling (100 ± 2°C) in an ultrasonic bath (35 kHz), facilitating substitution of acetate groups by phosphate moieties. The product was washed with double-distilled water to remove acetic acid byproducts and excess reagents (verified via molybdate reagent). Final washing to neutral pH and drying at 150 °C to constant mass completed the preparation [19, 20].

3.3. Process implementation

3.3.1. Pre-treatment stage

Brine samples (3 L) were heated to 50 ± 2 °C in a 5.0 L glass vessel equipped with thermostatic control. A NaOH solution (0.5 mol/L) was added to adjust the pH to 11–12, resulting in the precipitation of hydroxides of Fe^{3+}, Ni^{2+}, Co^{2+}, and Mg^{2+} due to their low solubility under strongly alkaline conditions.

The precipitation stage was carried out for 30 min under continuous stirring at 200 rpm. Following pH adjustment, carbon dioxide (CO_2) was introduced into the suspension by bubbling through a gas diffuser at a flow rate of 50 ± 5 mL/min for 20 ± 2 min under the same stirring conditions. The treatment was performed at 50 ± 2 °C, and the pH was continuously monitored and maintained within the range of 10.5–11.5.

Under these conditions, magnesium was predominantly removed via hydroxide precipitation:

$$Mg^{2+} + 2OH^- \rightarrow Mg(OH)_2\downarrow$$

Simultaneously, CO_2 introduction promoted carbonate formation, enhancing calcium removal via precipitation:

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3\downarrow$$

After completion of the treatment, the suspension was allowed to settle for at least 1 h. The precipitate was then separated by glass filtration, and the clarified solution was used for subsequent sorption.

3.3.2. Sorption stage

Dynamic sorption was performed in a 1 L glass column loaded with 50 g of acetophosphate cellulose (pre-activated by washing with distilled water). Lithium recovery was evaluated based on the mass balance between feed, effluent, and eluate streams.

The brine solution was pumped using a peristaltic pump (Watson-Marlow 323) at a flow rate of 2 ± 0.1 mL/min at 25 ± 1 °C. Fractions of 50 mL were collected and analyzed by ICP-MS.

The process parameters were optimized to maximize lithium recovery while minimizing co-extraction of $Mg^{2+}$and Ca^{2+}. Optimal conditions were established at pH 10.5–11.0, sorbent mass of 50 g, and flow rate of 2 mL/min.

Under these conditions, lithium recovery reached up to 93%, while Mg^{2+} and Ca^{2+} concentrations remained essentially unchanged, indicating negligible sorption of these ions.

Flow rates exceeding 3 mL/min resulted in decreased lithium recovery due to insufficient contact time, whereas flow rates below 1 mL/min did not provide a significant improvement but considerably increased the process duration. Sorbent masses below 30 g led to premature lithium breakthrough, while increasing the sorbent mass beyond 50 g did not result in further improvement in recovery efficiency.3.3.3. Desorption and Regeneration.

Lithium-saturated columns were regenerated using 0.1 N HCl at 2 mL/min. Desorption efficiency reached 98.25%. Post-regeneration washing to neutral pH enabled sorbent reuse in subsequent cycles.

3.4. Recovery efficiency calculation

Lithium recovery efficiency was calculated according to:

$$\eta = \frac{C_0 - C}{C_0} \times 100\%,$$

where C_0 represents the initial lithium concentration in the brine (mg/L), and C represents the residual lithium concentration after sorption (mg/L). This formulation quantifies the degree of lithium transfer from solution to solid phase, serving as the primary metric for process performance evaluation. All experiments were performed in triplicate, and average values are reported.

4. Results and discussion

4.1. Brine characterization and process feasibility

Literature indicates lithium concentrations exceeding 0.05 g/L (50 mg/L) represent economically viable thresholds for brine processing [21]. Optimal concentrations are reported for Salar de Atacama (~1.5 g/L), Salar de Uyuni (0.3–0.7 g/L), and Zabuye Salt Lake (~0.5 g/L) [22–26].

The Mg^{2+}/Li^{+} ratio critically determines process selection: ratios <6 permit chemical precipitation, while ratios >10 necessitate advanced technologies including membrane methods, sorption, or extraction. ICP-MS analysis of Aral Sea brine **(Table 1)** revealed lithium concentration of 2.809 mg/L – substantially below economic thresholds, yet subject to seasonal variation potentially reaching 0.05 g/L.

The elevated Mg^{2+}/Li^{+} ratio (~5.6) and low absolute lithium content necessitated the integrated approach combining alkaline precipitation with selective sorption.

4.2. Pre-treatment efficiency

Alkaline precipitation at pH 11–12 and 50 °C achieved substantial removal of Sc, Mg, and Ni **(Table 2)**. Magnesium concentration decreased from 15.667 to 5.667 mg/L; scandium from 30.686 to 0.246 mg/L; nickel from 0.289 to 0.014 mg/L. Calcium showed partial removal (19.411 to 5.251 mg/L) via carbonate precipitation promoted by controlled CO_2 introduction during alkaline treatment. Lithium remained predominantly in solution (2.809 to 2.719 mg/L), confirming the feasibility of alkaline pre-treatment for lithium preservation while removing major interferents.

4.3. Sorption performance and selectivity

Dynamic sorption on acetophosphate cellulose (50 g loading, flow rate 2 mL/min) demonstrated near-complete removal of lithium from the pre-treated brine **(Table 3)**. The residual lithium concentration in the effluent was below the detection limit of the analytical method, corresponding to lithium recovery efficiency (η) of up to 93%.

At the same time, the concentrations of Mg^{2+} and Ca^{2+} remained essentially unchanged (5.667 and 5.251 mg/L, respectively), indicating negligible sorption of these ions under the selected conditions. This observation provides quantitative evidence of high selectivity of the sorption system toward Li^{+}.

The observed selectivity cannot be attributed solely to an intrinsic binding preference of acetophosphate groups for lithium ions. Rather, it arises from the combined effect of process conditions and sorbent chemistry.

First, the alkaline pre-treatment stage significantly reduces the concentration of competing multivalent cations, thereby minimizing competitive interactions during the sorption step. Second, under the working pH conditions (10.5-11.0), acetophosphate groups are predominantly deprotonated, forming negatively charged oxygen-containing functional sites capable of interacting with alkali metal ions.

Third, Mg^{2+} ions are strongly hydrated in aqueous solution and exhibit slow dehydration kinetics, which limits their ability to interact with the sorbent surface. As a result, Mg^{2+} remains largely in the aqueous phase rather than being sorbed.

Table 1
Elemental composition of Aral Sea brine (ICP-MS analysis)

No.	Element	Concentration (mg/L)
1	K	807.428
2	Sc	30.686
3	V	21.401
4	Ca	19.411
5	Mg	15.667
6	Li	2.809
7	Ni	0.289
8	Al	0.101

Table 2
Brine composition following alkaline precipitation (ICP-MS)

No.	Element	Concentration (mg/L)
1	K	807.428
2	Na	500
3	Sc	0.246
4	V	1.155
5	Ca	5.251
6	Mg	5.667
7	Li	2.719
8	Ni	0.014
9	Al	0.101

Table 3
Sorption performance on acetophosphate cellulose (ICP-MS)

No.	Element	Concentration (mg/L)
1	K	807.428
2	Na	500
3	Sc	0.246
4	V	1.155
5	Ca	5.251
6	Mg	5.667
7	Li	<0.1 (below detection limit)
8	Ni	0.014
9	Al	0.101

Similarly, Ca^{2+} shows minimal interaction with the sorbent under the selected conditions, which is consistent with the experimental observation of its nearly unchanged concentration after sorption.

Thus, the high apparent selectivity toward Li^+ in this system is best explained by the synergistic effect of upstream chemical conditioning and the functional properties of acetophosphate cellulose, rather than by a single isolated coordination mechanism.

The static exchange capacity of 25.48 mg/g, combined with 98.25% desorption efficiency and capacity retention of 93% after 10 cycles, substantiates the sorbent's suitability for process implementation. The selectivity coefficient for Li^+ relative to Mg^{2+}, estimated from concentration ratios, exceeds 100, indicating high separation performance under the studied conditions.

4.4. Process integration and scientific implications

The experimental results demonstrate that integration of alkaline precipitation with selective sorption on acetophosphate cellulose enables effective lithium recovery from extremely dilute, highly mineralized brines. The process achieves:

Efficiency: Near-complete lithium capture under moderate conditions (25°C, atmospheric pressure);

Selectivity: negligible sorption of Ca^{2+} and Mg^{2+} was observed;

Sustainability: Biodegradable, renewable-based sorbent with multi-cycle regeneration capability;

Environmental compatibility: Absence of toxic organic solvents; dilute acid regeneration.

These findings provide a scientific basis for subsequent pilot-scale investigations and engineering design.

The integration of pre-treatment and sorption stages reduces reagent consumption and improves process efficiency, indicating potential economic advantages for industrial implementation.

A preliminary economic assessment was performed to evaluate the feasibility of the proposed process. Based on the experimental lithium concentration in Aral Sea brine (2.8 mg/L) and a recovery efficiency of 93%, approximately 2.6 g of lithium can be recovered from 1 m^3 of solution, corresponding to about 13.8 g of Li_2CO_3.

Considering an average market price of 20 USD/kg Li_2CO_3, the estimated product value is approximately 0.28 USD per m^3 of treated brine. The operational costs associated with alkaline precipitation, acid desorption, energy consumption, and sorbent utilization are estimated at approximately 0.18 USD per m^3.

The economic assessment presented in this study is preliminary and limited to a screening-level estimation. Capital expenditures, equipment costs, lab or, and downstream processing were not included. Therefore, the results indicate technical feasibility rather than confirmed economic viability.

5. Conclusions

An integrated process for lithium recovery from highly mineralized Aral Sea brines has been developed and experimentally validated. The approach combines alkaline pre-treatment with selective sorption on acetophosphate cellulose, enabling effective removal of interfering ions prior to lithium capture.

The sorbent demonstrated a static exchange capacity of 25.48 mg/g, desorption efficiency of 98.25%, and stable performance over 10 sorption–desorption cycles with 93% capacity retention. Under optimized conditions (pH 10.5–11.0, flow rate 2 mL/min), lithium recovery reached up to 93%, while Mg^{2+} and Ca^{2+} were not significantly sorbed, confirming high selectivity.

The results indicate that the proposed process is applicable for lithium recovery from low-grade brines (<3 mg/L Li) under mild operating conditions without the use of toxic organic solvents. The integration of pre-treatment and sorption stages improves process efficiency and reduces interference effects.

The developed process can be considered a basis for further scale-up and pilot-level implementation. Additional optimization of solid–liquid separation, column hydrodynamics, and eluate processing is required for industrial application.

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