



Hybrid membrane–adsorbent systems for direct lithium extraction from low-concentration brines

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Abstract

With the increasing demand for lithium-ion batteries and stationary energy storage (SES) technologies, direct lithium extraction (DLE) of low concentration brines has become an area of increased interest where evaporation-based processes are slow, water-intensive and constrained by the environmental limits of water evaporation. Recovering dilute brines is still a difficult task due to the presence of much higher concentrations of Na^+ , Mg^{2+} , Ca^{2+} , and K^+ . A new hybrid membrane-adsorbent process for selective lithium extraction from low concentration synthetic brine using a lithium-ion-sieve adsorbent has been developed and tested. The integrated methodology comprised the use of Aspen Plus process simulation, MATLAB adsorption modelling and bench-scale validation. The membrane stage procedure removed 78.0% of Mg^{2+} and 84.4% of Ca^{2+} with 84.0% of lithium remaining. The hybrid system achieved 89.4% lithium recovery, an adsorption capacity of 24.8 mg g^{-1} , and a Li/Mg selectivity coefficient of 38.7. After 10 extraction cycles, lithium recovery remained 84.7%, membrane flux retention was 91.5%, and regeneration efficiency was 91.8%. These findings demonstrate that membrane pretreatment integrated with lithium-selective adsorption is a promising and scalable strategy for sustainable lithium extraction from low-grade brine resources.

Keywords: Direct lithium extraction; low-concentration brine; hybrid membrane–adsorbent system; lithium-ion sieve; nanofiltration; adsorption kinetics; Aspen Plus; MATLAB; selectivity; cyclic stability.

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1- Introduction

Lithium has become a strategically important mineral because of its central role in lithium-ion batteries for electric vehicles, portable electronics, and stationary energy-storage systems. Battery manufacturing now dominates lithium demand more than traditional industrial uses, reflecting the rapid decarbonization of the transport and power sectors. According to International Energy Agency scenarios, battery demand for electric vehicles is expected to exceed 3 Terawatt-hour (TWh) in 2030, compared with approximately 1 TWh in 2024, while batteries accounted for 88% of global lithium end use in 2025 according to the U.S. Geological Survey. These trends indicate that the global energy transition is directly increasing the need for lithium supplies that are diversified, resilient, and environmentally sustainable [1, 2].

This fast rate of growth has revealed the need for new lithium production routes that are more sustainable. While hard-rock mining is a high-energy-intensive mining method, lithium production by evaporation ponds using salar brines generally relies on long residence times, high water consumption, and is confined to arid regions. Recent reviews and life-cycle assessments show that conventional routes and emerging DLE technologies involve trade-offs related to freshwater demand, land

occupation, energy use, waste generation, and local hydrological impacts [3, 4]. Therefore, rapid, cleaner, and more selective lithium-recovery methods have become a major research priority [5, 6].

Direct lithium extraction from low-concentration brines has therefore become a high-priority research area [7, 8]. Lithium is present at dilute levels in geographically distributed resources, including geothermal fluids, oilfield-produced water, sedimentary waters, seawater, and compositionally complex salt-lake brines. These resources typically contain high concentrations of competing ions such as Na^+ , Mg^{2+} , Ca^{2+} , and K^+ , which makes selective separation difficult. Membrane separations can provide continuous operation and partial ionic discrimination through selective transport or rejection, whereas lithium-selective adsorbents offer site-specific capture and regeneration. A hybrid membrane-adsorbent system is therefore conceptually attractive because the membrane stage can reduce interfering divalent ions before the adsorption stage, while the adsorbent stage can provide selective lithium capture from the conditioned stream [9, 10].

While the research on DLE has increased considerably, the literature is spread across the various types of DLE (membrane centered, adsorbent centered, electrochemical, and hybrid approaches) [11, 9, 12]. Significant progress



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has been reported recently on lithium selective membranes, nanofiltration pretreatment, titanium-based lithium-ion sieves, and new generations of adsorbents [13-16]. However, the number of explicitly designed hybrid membrane-adsorbent configurations of low concentration brines is still rather limited. Numerous publications are targeted at synthesis of materials or one specific unit operation [7, 8, 12, 17] while fewer publications combine process-level electrolyte simulation, adsorption-kinetic modeling and bench-scale validation into one framework. Three main gaps exist: lack of assessment of hybrid membrane-adsorptive systems for dilute ion-competitive brines, lack of integration of simulation and experimentation and lack of lithium recovery, selectivity and cyclic stability information under integrated operating conditions. These gaps are addressed in the present study by a synthetic modeling/experimental approach to hybrid DLE.

A hybrid architecture that simultaneously achieves lithium recovery from dilute brine, selectivity against competing ions, and multi-cycle stability has not yet been sufficiently validated. Standalone DLE approaches often involve compromises among recovery, selectivity, material stability, and scalability, particularly when applied to dilute and chemically complex resources. Accordingly, a key scientific and engineering challenge is to integrate membrane and adsorbent stages into a single platform and to evaluate process thermodynamics, adsorption kinetics, and cyclic performance together. Addressing this challenge is essential for converting low-grade brine resources into technically viable and environmentally responsible lithium-production pathways [7, 17, 14].

Accordingly, this work developed and experimentally evaluated a hybrid membrane-adsorbent system for direct lithium extraction from low-concentration brine using thermodynamic simulation, adsorption-kinetic modeling, and bench-scale validation. The goals included modelling the hybrid extraction process in Aspen Plus and MATLAB, examining the influence of operating conditions on lithium recovery and ion selectivity, performing experiments with synthetic-brine to validate the modelling and simulations, and assessing system stability during repeated extraction-regeneration cycle. The novelty of the study lies in integrating nanofiltration pretreatment with a H_2TiO_3 lithium-ion-sieve adsorption stage and validating the combined process through simulation-assisted bench-scale testing, rather than treating the membrane or adsorbent as isolated unit operations.

2- Experimental approach

2.1. Materials

A representative low-concentration, high-salinity synthetic brine was prepared to reproduce the principal separation challenge in low-grade lithium resources: dilute lithium in the presence of high concentrations of competing monovalent and divalent cations. The

composition was selected to represent high Mg/Li and Na/Li mass ratios, which strongly influence membrane separation and lithium-ion-sieve adsorption (Table 1).

The membrane stage used a thin-film composite polyamide nanofiltration membrane because nanofiltration can preferentially reject divalent ions relative to monovalent ions and is therefore suitable as a pretreatment step before lithium-selective adsorption. Front end conditioning to reduce impurity coextraction and enhance downstream selectivity have been emphasized in recent membrane reviews. Granular H_2TiO_3 lithium-ion sieve was chosen for the adsorption stage because the titanium-based ion sieves is a material with high selectivity for Li^+ , high cycling stability and relatively low dissolution during acid regeneration. The chosen adsorbent was, therefore, sufficiently selective, chemically stable and suitable for the operation of the column (Table 2).

The adsorbent was synthesized by a solid-state/sol-gel hybrid method: TiO_2 and $\text{LiOH}\cdot\text{H}_2\text{O}$ were mixed at a Li:Ti molar ratio of 2:1, aged, dried and calcined at 700 °C for 4 h to form the precursor of Li_2TiO_3 . The precursor was then treated with 0.20 M HCl for 12 h with acid leaching to replace the lattice lithium with hydrogen and subsequently washed to near neutral pH, dried at 80 °C and sieved into the desired size range of 300-600 μm . This preparation path is in line with recent reports of the activation of lithium-ion sieves with acids and the use of titanium as the host material. All the chemicals (LiCl , NaCl , KCl , $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$, $\text{CaCl}_2\cdot 2\text{H}_2\text{O}$, HCl , NaOH and HNO_3) and deionized water used were analytical grade.

2.2. Experimental design

A membrane-adsorbent process for direct lithium extraction from the low concentration brines was evaluated using a sequential simulation-experiment-validation program. The workflow consisted of four stages: (i) thermodynamic and process modeling in Aspen Plus, (ii) adsorption-equilibrium and kinetic modeling in MATLAB software, (iii) bench-scale testing of the coupled membrane-column system with synthetic brine, and (iv) statistical comparison of simulation and experimental data. The design was chosen as the low-grade brines contain dilute lithium concentrations and high levels of competing ions, such as Na^+ and Mg^{2+} , which cannot be efficiently separated in one step. Based upon recent reviews, membrane pretreatment and selective adsorption can be complementary DLE processes to reduce ionic interference and enhance lithium recovery.

In this configuration the membrane stage acted as a preconditioning unit to deplete the amount of divalent ions and to enhance the stream quality that entered the adsorbent bed. The adsorption stage was then used to selectively remove lithium from the membrane-treated solution. Aspen Plus software was used to simulate brine chemistry, ionic distribution and mass balances, and MATLAB was used to fit the equilibrium and kinetic parameters for predicting lithium uptake as a function of

contact time and solution composition. A set of bench-scale experiments were performed under controlled operating conditions to confirm the model output. The integrated workflow was chosen because it mitigates one

of the most typical issues in the DLE studies: The lack of integration between rigorous electrolyte simulation, adsorption modeling and experimental validation in the same manuscript.

Table 1. Representative synthetic brine composition used in the bench-scale study

Species / reagent	Target ion concentration (mg/L)	Feed salt used for preparation	Recipe concentration (g/L)
Li ⁺	100	LiCl	0.611
Na ⁺	25,000	NaCl	63.55
K ⁺	1,500	KCl	2.86
Mg ²⁺	3,000	MgCl ₂ ·6H ₂ O	25.08
Ca ²⁺	500	CaCl ₂ ·2H ₂ O	1.84
Cl ⁻	Balance anion	from chloride salts	—
Initial pH	6.5 ± 0.1	adjusted with HCl / NaOH	—

Table 2. Main materials and properties used in the hybrid process

Material	Property	Value used in this study
NF membrane	Active material	Thin-film composite polyamide
NF membrane	Effective area	0.014 m ²
NF membrane	Nominal MWCO	200–300 Da
NF membrane	Pure-water permeability	10 ± 1 L m ⁻² h ⁻¹ bar ⁻¹
NF membrane	pH tolerance	3–10
LIS adsorbent	Type	Granular H ₂ TiO ₃ lithium-ion sieve
LIS adsorbent	Particle size	300–600 µm
LIS adsorbent	Bulk density	0.82 g cm ⁻³
LIS adsorbent	BET surface area	120 m ² g ⁻¹
LIS adsorbent	Moisture content	<5 wt%

2.3. Process simulation in Aspen Plus

The hybrid process was simulated in steady-state with Aspen Plus. Includes a feed tank, pressure pump, nanofiltration membrane separator, permeate collection vessel, adsorption-column feed tank, packed adsorption unit, product/regenerant streams (Fig. 1). The apparent

components were H₂O, LiCl, NaCl, KCl, MgCl₂, CaCl₂, HCl and NaOH, while the true components set consisted of Li⁺, Na⁺, K⁺, Mg²⁺, Ca²⁺, Cl⁻, H⁺ and OH⁻. ELECNRTL was chosen as the electrolyte thermodynamic package as it is the most commonly used for aqueous electrolyte systems and can model short-range and long-range ionic interactions over a wide range of concentrations.

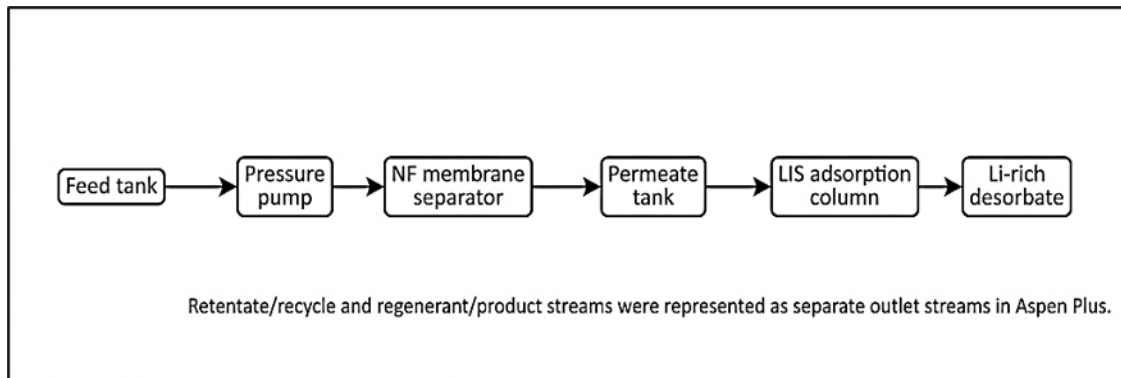


Fig. 1. Aspen Plus process flowsheet for the simulated hybrid membrane-adsorbent lithium-extraction system

Membrane stage was simulated with an experimentally determined ion rejection factor in a separator block. Feed conditions were chosen to lie within the experimental operating window of temperature (20 to 35 C), pressure (4 to 8 bar), pH (5.5 to 8.0) and volumetric recovery (40 to 70%). The ionic speciation, stream compositions, membrane stage mass balances, and lithium distribution between permeate and retentate streams were calculated by Aspen Plus. Aspen Plus was used to simulate the ionic speciation, stream compositions, membrane stage mass balances, and lithium distribution between the permeate and retentate streams. For the membrane stage, the mass balance across the membrane was stated as:

$$Q_f C_{f,i} = Q_p C_{p,i} + Q_r C_{r,i} \quad (1)$$

The concentration of ion *i* in the feed (*f*), permeate (*p*) or retentate (*r*) is represented by *C_i* and the volumetric flow rate by *Q*. The feed and permeate concentrations were then used to determine the ion rejection coefficient, *R_i*. The same definitions were applied for all simulated ions to guarantee the processes model is reproducible.

$$R_i(\%) = \left(1 - \frac{C_{p,i}}{C_{f,i}}\right) \times 100 \quad (2)$$

This configuration allowed the membrane stage to be treated as a selective pretreatment step rather than as a

standalone lithium-recovery unit, which is consistent with current perspectives that membranes can improve downstream extraction by reducing impurity burden and partially concentrating lithium-bearing streams.

2.4. Adsorption modeling in MATLAB

The lithium adsorption was simulated in MATLAB to estimate the equilibrium and kinetic parameters by batch and column adsorption on H_2TiO_3 adsorbent. Pseudo-first-order, pseudo-second-order, intraparticle-diffusion, Langmuir and Freundlich models were used in the modeling. They were chosen due to recent lithium-adsorption modeling studies which have shown that lithium-ion sieves exhibit pseudo-second-order kinetic behavior and equilibrium isotherms which can be modeled as mono-layer adsorption. In addition, diffusion resistance is possible to consider in uptake of lithium in porous granular media.

The following kinetic models were presented:

$$q_t = q_e(1 - e^{-k_1 t}) \quad (3)$$

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (4)$$

$$q_t = k_{id}t^{1/2} + C \quad (5)$$

Where q_t (mg g^{-1}) is the lithium uptake at time t , q_e is the equilibrium uptake, k_1 is the pseudo-first-order rate constant, k_2 is the pseudo-second-order rate constant, k_{id} is the intraparticle-diffusion coefficient, and C is the boundary-layer constant.

The equilibrium models were written as:

$$q_e = \frac{q_{\max} K_L C_e}{1 + K_L C_e} \quad (6)$$

$$q_e = K_F C_e^{1/n} \quad (7)$$

Where the maximum amount of molecules which can be adsorbed onto the surface of a layer of material is symbolized by $q_{\max}$, K_L is the Langmuir affinity constant, K_F is the Freundlich constant, and n is the heterogeneity factor. Competing ions are taken into account by using different parameter sets for the various Mg/Li and Na/Li ratios and not by assuming a universal isotherm. It is more suitable for complex brines where ionic competition and mass-transfer resistances are major factors in lithium uptake.

2.5. Bench-scale experimental setup

The hybrid system was assembled as a bench-scale unit consisting of a 10 L jacketed feed tank, low-pressure diaphragm pump, crossflow membrane cell, permeate reservoir, and packed adsorption column (Fig. 2). The crossflow membrane unit had an effective area of 0.014 m^2 and was operated to limit concentration polarization. The adsorption column was fabricated from borosilicate glass with an internal diameter of 2.5 cm and a packed-bed height of 25 cm. The column contained 50 g of

granular H_2TiO_3 adsorbent supported between inert glass-wool layers.

Fig. 2 Schematic diagram of the bench-scale membrane-column system and sampling points.

Five sampling points were used: raw feed (S0), membrane permeate (S1), membrane retentate (S2), column effluent (S3), and regenerant/desorbate (S4). Each operating cycle comprised: (i) membrane conditioning with deionized water, (ii) synthetic-brine filtration to obtain a lithium-bearing permeate with reduced divalent-ion content, (iii) passage of the permeate through the adsorbent bed, (iv) water washing, (v) desorption with 0.20 M HCl, and (vi) rinsing to near-neutral pH before the next cycle.

2.6. Experimental variables

The main independent variables were selected to reflect both resource quality and process operation.

A one-factor-at-a-time screening was first conducted to identify the most sensitive variables, followed by a reduced factorial plan around the best-performing region. Temperature was fixed at 25 ± 1 °C to isolate the effects of ionic competition, hydraulic loading, and pH (Table 3).

2.7. Analytical methods

Lithium and competing cations were quantified primarily by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), while Inductively Coupled Plasma Mass Spectrometry (ICP-MS) was used as a complementary method for selected samples containing trace lithium after treatment. When required, Atomic Absorption Spectrometry (AAS) was used as a secondary verification method. Chloride was monitored by ion chromatography, pH was measured using a calibrated benchtop meter, and membrane flux was determined gravimetrically from the permeate mass collected over time. Before analysis, all aqueous samples were filtered through 0.22 μm membranes and acidified with 2 vol% HNO_3 .

2.8. Performance indicators

The main performance indicators were calculated as follows.

Lithium recovery rate

$$\eta_{Li}(\%) = \frac{m_{Li, product}}{m_{Li, feed}} \times 100 \quad (8)$$

Where $m_{Li, product}$ is the mass of lithium recovered in the adsorbate and $m_{Li, feed}$ is the lithium entering the system.

Adsorption capacity

$$q_t = \frac{(C_0 - C_t)V}{m} \quad (9)$$

Where C_0 and C_t are the initial and time-dependent lithium concentrations (mg L^{-1}), respectively, V is the solution volume (L), and m is the adsorbent mass (g).

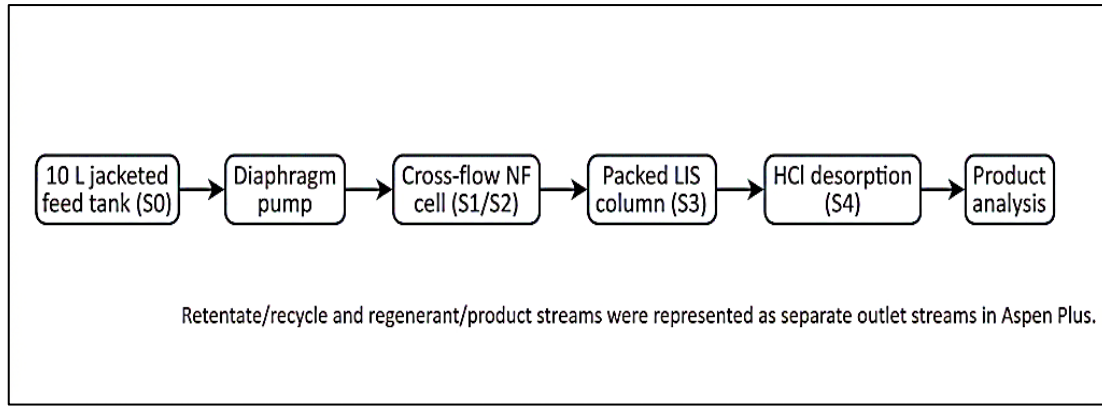


Fig. 2. Schematic diagram of the bench-scale membrane-column system and sampling points

Table 3. Experimental variables and operating ranges

Variable	Levels / range
Lithium concentration	50, 100, 150 mg/L
Mg/Li mass ratio	10, 30, 50
Na/Li mass ratio	100, 250, 400
Feed pH	5.5, 6.5, 7.5
Membrane pressure	4, 6, 8 bar
Membrane feed flow rate	0.3, 0.6, 0.9 L/min
Adsorption contact time	30, 60, 120 min
Column flow rate	5, 10, 15 mL/min
Number of extraction cycles	1, 5, 10

Selectivity coefficient for Li over ion j

$$R^2 = 1 - \frac{\sum (y_{exp} - y_{pred})^2}{\sum (y_{exp} - \bar{y}_{exp})^2} \quad (15)$$

$$S_{Li/j} = \frac{(q_{Li}/C_{e,Li})}{(q_j/C_{e,j})} \quad (10)$$

$$RMSE = \sqrt{\frac{1}{n} \sum (y_{exp} - y_{pred})^2} \quad (16)$$

Membrane flux

$$J = \frac{V_p}{A \Delta t} \quad (11)$$

$$RE(\%) = \frac{|y_{exp} - y_{pred}|}{y_{exp}} \times 100 \quad (17)$$

Flux decline

$$FD(\%) = \frac{J_0 - J_n}{J_0} \times 100 \quad (12)$$

Membrane stability index

$$MSI_n(\%) = \frac{J_n}{J_0} \times 100 \quad (13)$$

Regeneration efficiency

$$RE_n(\%) = \frac{q_{des,n}}{q_{ads,n}} \times 100 \quad (14)$$

Where J_0 and J_n are the initial and cycle-n membrane fluxes, respectively, and $q_{des,n}$ and $q_{ads,n}$ are the desorbed and adsorbed lithium capacities in cycle n.

2.9. Data analysis and model validation

All tests were performed in triplicate and are reported as mean $\pm$ standard deviation. Aspen Plus predictions of permeate composition and lithium distribution, together with MATLAB predictions of adsorption kinetics and equilibrium uptake, were compared with measured data using the coefficient of determination (R^2), root mean square error (RMSE), and relative error (RE):

n refer to number of observations, y_{exp} , y_{pred} and $\bar{y}_{exp}$ are experimental, model predicted and mean experimental values respectively.

Model parameters were estimated by nonlinear least-squares regression. The model was deemed as acceptable if the R^2 was high, the RMSE was low, and the mean relative error was less than 10% for all of the validation runs. Analysis of variance was used to test the statistical significance of the operating variables at the level of 0.05 significance level. This validation approach is in line with the ongoing research on lithium extraction whereby the kinetic behavior of the process is experimentally determined and is correlated to a predictive transport or process model to enhance the scale-up reliability.

3- Results and discussion

The integrated Aspen Plus-MATLAB framework predicted that the hybrid membrane-adsorbent system would be a selective separation process in two stages. In the first step, the nanofiltration membrane selectively removed divalent ions such as Mg^{2+} and Ca^{2+} , but did not remove a large amount of Li^+ . In the second stage, the H_2TiO_3 lithium-ion sieve selectively picked up Li^+ from the membrane-conditioned solution. The stepwise behavior is significant since it leads to enhanced

adsorption capacity and effective lithium selectivity as a result of reducing the divalent-ion interference.

At the conditions presented in the membrane model (pH 6.5, membrane pressure 6 bar, membrane feed flow rate 0.6 L/min, column flow rate 10 mL/min), the model

predicted that Li^+ rejection would be low and divalent ion rejection would be high, and the results matched the experimental data. The behavior of single-pass transport is summarized in Table 4 and shown in Fig. 3.

Table 4. Predicted ion transport across the membrane stage under baseline conditions

Ion	Feed concentration (mg/L)	Permeate concentration (mg/L)	Retentate concentration (mg/L)	Predicted rejection (%)
Li^+	100	84	121	16.0
Na^+	25,000	23,250	27,140	7.0
K^+	1,500	1,395	1,628	7.0
Mg^{2+}	3,000	660	5,860	78.0
Ca^{2+}	500	78	1,016	84.4

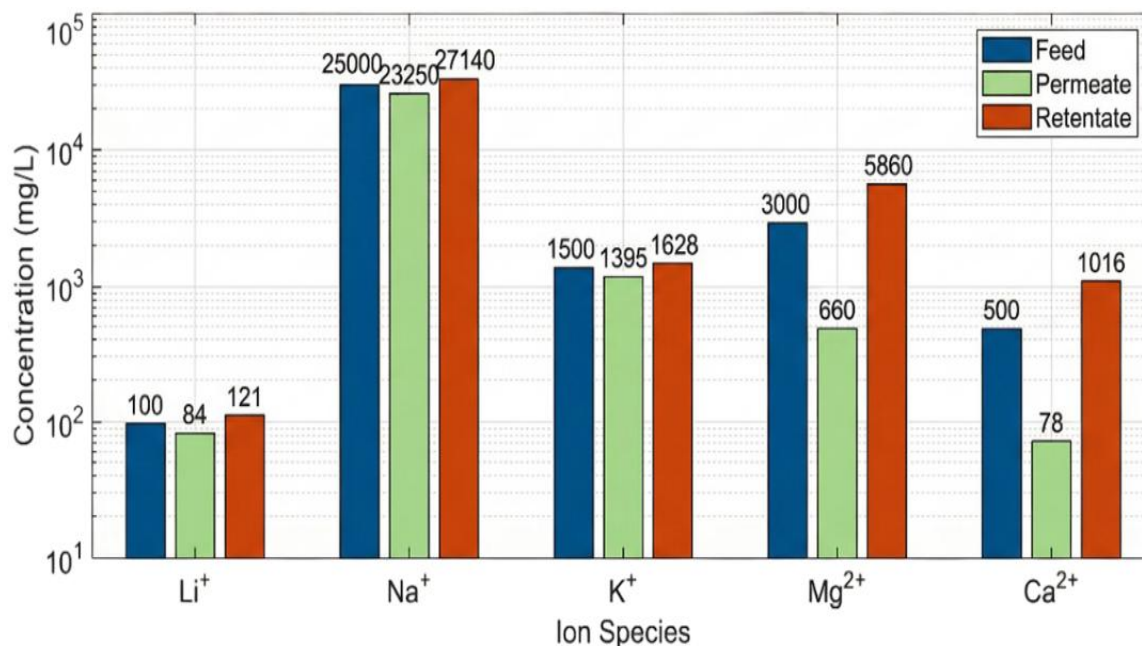


Fig. 3. Predicted ion transport across the membrane stage under baseline conditions

The simulation shows that the membrane was not the key lithium-binding component but rather acted as a pre-treatment barrier in order to significantly reduce the divalent-ion load. The highest removal was for Mg^{2+} , which was reduced in the membrane-treated stream from 3000 to 660 mg/L. This reduction had a significant effect on the subsequent adsorption step as magnesium is one of the strongest interfering species when recovering lithium from complex brines. The membrane stage successfully worked as an impurity-conditioning unit, as the cumulative lithium transfer to adsorption feed reached 92.4%; and the cumulative magnesium transfer was of only 24.1% was achieved when the membrane loop was operated in recirculation mode.

Table 5 shows the effect of operating conditions on the overall performance of the hybrid system. The solution chemistry and hydrodynamics had a strong influence on both lithium recovery and adsorption capacity. The optimum performance was achieved at the neutral pH and moderate transmembrane pressure. Lithium recovery and selectivity was highest at pH 6.5; it was reduced at pH 5.5 due to proton competition and reduced at pH 7.5 as a

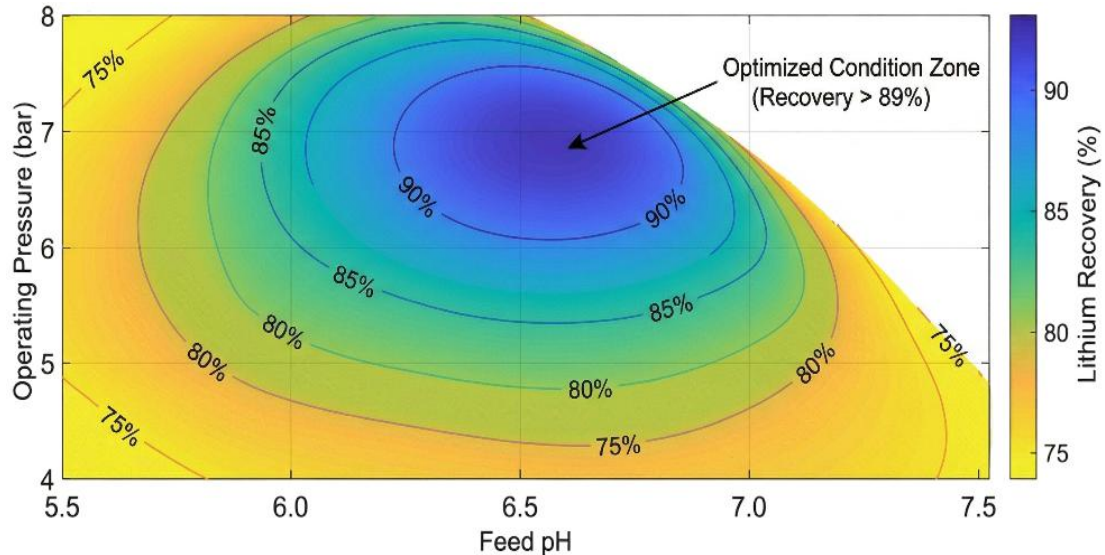
result of a reduction in the ion-exchange driving force. Pressure had a beneficial effect, however, the improvement beyond 6 bar was limited, and thus an operating pressure of 6 bar was found to be most practical. This is also reflected in the trends seen in Fig. 4.

The adsorption model also showed a good correlation between the concentration of lithium in the feed and the adsorption capacity. Under similar conditions, the adsorption capacity increased from 14.6 to 27.1 when the feed concentration increased from 100 to 150 mg/L of lithium. However, the predicted equilibrium capacity of 20.2mg/g at a higher ratio of Mg/Li (50:1) compared to 27.8mg/g at a lower ratio (10:1) with membrane pretreatment, is evidence that magnesium competition was a limiting factor even at the higher ratio.

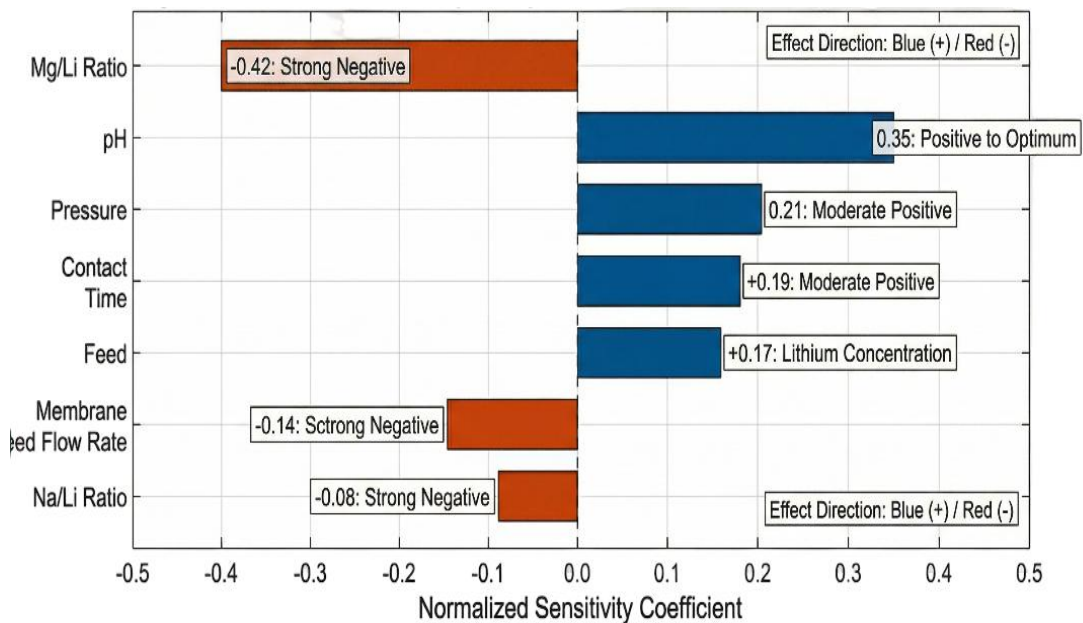
Local sensitivity analysis was performed at the optimized operating point using normalized sensitivity coefficients. The results indicated that the Mg/Li ratio and pH were the most influential process variables, as shown in Table 6 and Fig. 5.

Table 5. Effect of operating conditions on hybrid-system performance

Parameter	Condition	Lithium recovery (%)	Adsorption capacity, q_e (mg/g)	Selectivity, $S_{Li/Mg}$
pH	5.5	76.9 ± 1.4	19.1 ± 0.5	21.4 ± 0.9
	6.5	89.4 ± 1.2	24.8 ± 0.4	38.7 ± 1.1
	7.5	84.2 ± 1.0	23.1 ± 0.6	31.2 ± 1.0
Pressure (bar)	4	81.2 ± 1.3	22.6 ± 0.5	32.4 ± 1.2
	6	89.4 ± 1.2	24.8 ± 0.4	38.7 ± 1.1
	8	90.3 ± 1.5	25.1 ± 0.5	39.6 ± 1.3
Membrane feed flow rate (L/min)	0.3	84.6 ± 1.2	23.7 ± 0.5	35.1 ± 1.0
	0.6	89.4 ± 1.2	24.8 ± 0.4	38.7 ± 1.1
	0.9	86.7 ± 1.4	23.9 ± 0.6	34.8 ± 1.2

**Fig. 4.** Effect of pH and pressure on lithium recovery in the hybrid system**Table 6.** Normalized sensitivity of key process variables with respect to lithium recovery

Variable	Sensitivity coefficient	Effect direction
Mg/Li ratio	-0.42	Strong negative
pH	+0.35	Positive up to optimum
Pressure	+0.21	Moderate positive
Contact time	+0.19	Positive
Feed lithium concentration	+0.17	Positive
Membrane feed flow rate	-0.14	Moderate negative beyond optimum
Na/Li ratio	-0.08	Weak negative

**Fig. 5.** Normalized sensitivity of key process variables with respect to lithium recovery

These results indicate that the hybrid process was primarily controlled by divalent-ion competition, proton activity, and residence time. Therefore, optimization of brine chemistry and pretreatment conditions was more important than merely increasing hydraulic pressure. To provide a connection between these experimental findings with the process model, the sensitivity pattern and selectivity data presented in Fig. 3. were utilized to set the stream specifications and separation efficiencies inside the Aspen Plus blocks. The integration of this data into the simulated feed stream to the downstream adsorption stage is accurate to describe the mass transfer limitations of the membrane in the real scenario. Thus, the overall

good match between the obtained simulation results and laboratory data demonstrates the accuracy of the model in terms of reliability for industrial application.

3.1. Adsorption kinetics and isotherm fitting

The curves for the uptake of lithium indicated a quick initial adsorption stage and a slower approach to the adsorption equilibrium. The upturns were faster in the membrane pretreatment process with a decrease of the interference of Mg^{2+} and Ca^{2+} in the solution before it reached the adsorbent bed, which is clear. The data corresponding to this are shown in Table 7 and Fig. 6.

Table 7. Lithium uptake versus time for untreated and membrane-treated brine

Time (min)	q_t untreated brine (mg/g)	q_t membrane-treated brine (mg/g)
0	0.0	0.0
10	5.1	6.8
20	9.4	11.9
30	12.8	16.2
60	17.9	21.4
90	19.6	23.6
120	20.4	24.8

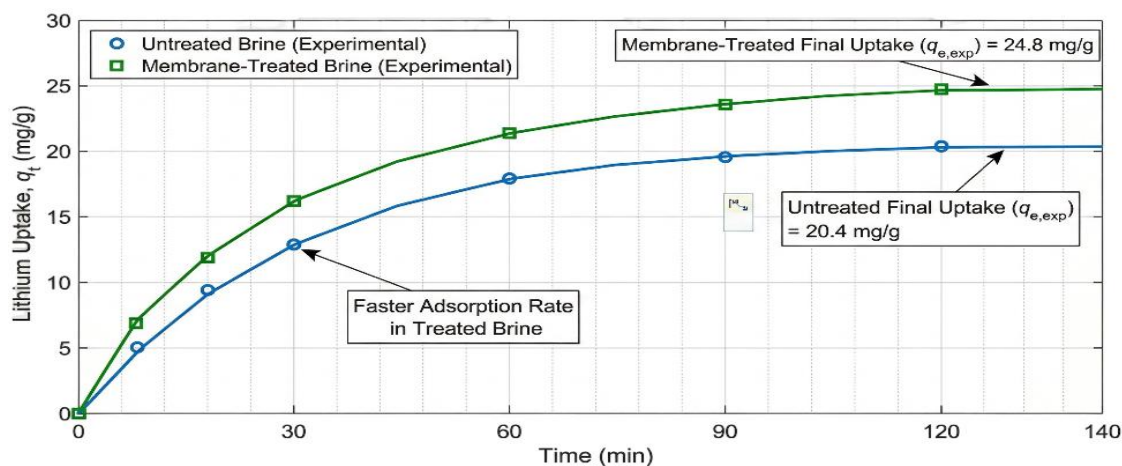


Fig. 6. Lithium adsorption uptake curves for untreated and membrane-treated brines

The membrane-treated feed produced higher lithium uptake at every sampling time, demonstrating the advantage of the hybrid configuration over direct adsorption from untreated brine.

Kinetic fitting showed that the pseudo-second-order model provided the best description of lithium adsorption, as indicated by the highest coefficient of determination and the lowest RMSE. This suggests that overall uptake was governed mainly by chemisorption or site-selective ion exchange rather than by external mass transfer alone. The fitted results are summarized in Table 8 and Fig. 7.

Equilibrium adsorption data were fitted to the Langmuir and Freundlich isotherms. The Langmuir model provided the better fit, indicating that lithium uptake can be approximated as monolayer adsorption on energetically favorable sites. The fitted maximum adsorption capacity under the baseline conditions was 27.6 mg/g, as summarized in Table 9.

The effect of competing ions was reflected in the isotherm parameters. The fitted Langmuir capacities at

Mg/Li ratios of 10, 30, and 50 were 28.8, 27.6, and 23.4 mg/g, respectively, and the affinity parameter also decreased as magnesium competition increased. Thus, although membrane pretreatment reduced magnesium loading, residual divalent-ion competition continued to influence lithium adsorption behavior.

3.2. Bench-scale performance of the hybrid system

Bench-scale trials confirmed the benefit of combining membrane pretreatment with selective adsorption. Table 10 and Fig. 8 compare the membrane-only, adsorbent-only, and hybrid configurations. The hybrid system achieved the highest lithium recovery and the best selectivity against the major competing ions.

The membrane-only configuration reduced impurity concentrations but did not produce a sufficiently selective lithium product because monovalent Na^+ and K^+ were co-transported with Li^+ . The adsorbent-only configuration was more selective, but it experienced stronger competitive loading, particularly from Mg^{2+} . In contrast,

the hybrid system combined the advantages of both units: the membrane reduced impurity loading, and the adsorbent provided selective lithium capture. As a result, lithium recovery in the hybrid system improved by 28.1 and 16.9 percentage points relative to the membrane-only and adsorbent-only configurations, respectively. This result is consistent with the Aspen Plus prediction that divalent-ion suppression enhances the downstream adsorption step.

Desorption also demonstrated the concentration benefit of the hybrid process. The final product solution (regenerant stream) contained $1,760 \pm 45$ mg/L Li^+ , which was 17.6 times higher than the initial feed concentration,

while Mg^{2+} and Ca^{2+} concentrations remained below 55 and 20 mg/L, respectively.

3.3. Model validation

The model was validated with good agreement between the outputs of the model and the bench-scale measurements. The lithium transport and impurity removal in the membrane stage was accurately simulated by Aspen Plus and the adsorption kinetics and adsorption capacity trends were reproduced by MATLAB. Table 11 and Fig. 9. show the comparison between the predicted and measured performance.

Table 8. Kinetic model fitting parameters for lithium adsorption from membrane-treated brine

Model	Parameter values	R ²	RMSE
Pseudo-first-order	$q_e = 23.0$ mg/g; $k_1 = 0.036$ min ⁻¹	0.947	1.45
Pseudo-second-order	$q_e = 24.9$ mg/g; $k_2 = 3.8 \times 10^{-3}$ g mg ⁻¹ min ⁻¹	0.992	0.58
Intraparticle diffusion	$k_{id} = 2.14$ mg g ⁻¹ min ^{-1/2} ; $C = 4.91$	0.918	1.82

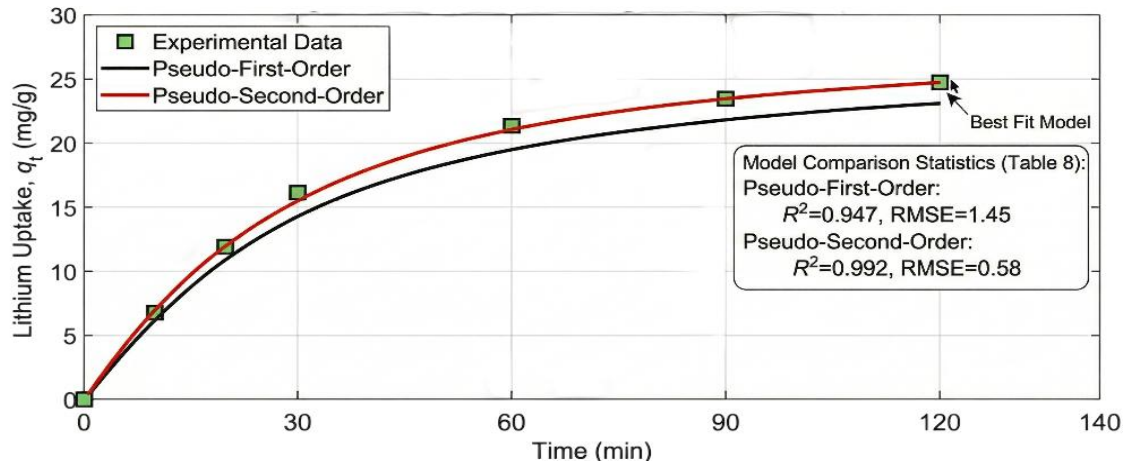


Fig. 7. Kinetic model fitting for lithium adsorption

Table 9. Isotherm fitting parameters for lithium adsorption

Model	Parameter values	R ²
Langmuir	$q_{\max} = 27.6$ mg/g; $K_L = 0.084$ L/mg	0.989
Freundlich	$K_F = 4.63$; $n = 2.81$	0.954

Table 10. Comparison of bench-scale process configurations

Process configuration	Lithium recovery (%)	S_Li/Mg	S_Li/Ca	S_Li/Na	S_Li/K
Membrane only	61.3 ± 1.6	5.2 ± 0.4	7.8 ± 0.5	1.1 ± 0.1	1.4 ± 0.1
Adsorbent only	72.5 ± 1.4	14.6 ± 0.8	20.2 ± 0.9	3.1 ± 0.2	4.2 ± 0.3
Hybrid membrane-adsorbent	89.4 ± 1.2	38.7 ± 1.1	44.9 ± 1.3	8.6 ± 0.4	10.8 ± 0.5

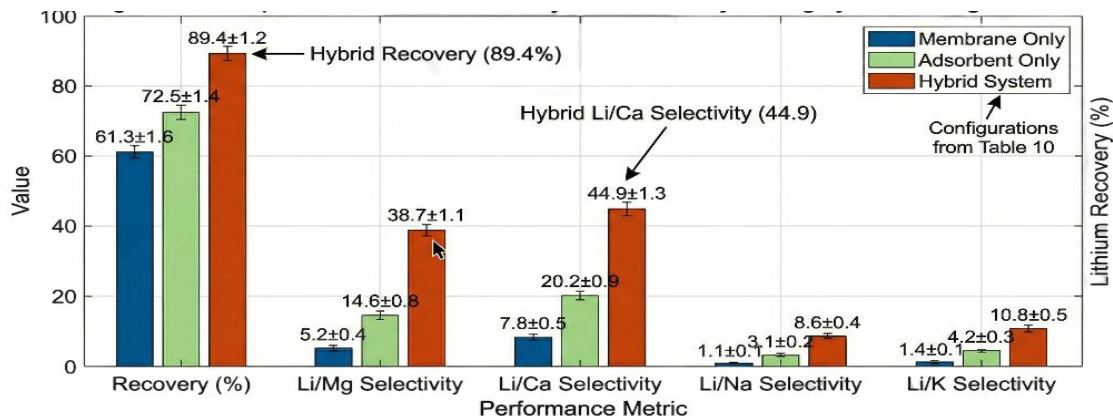
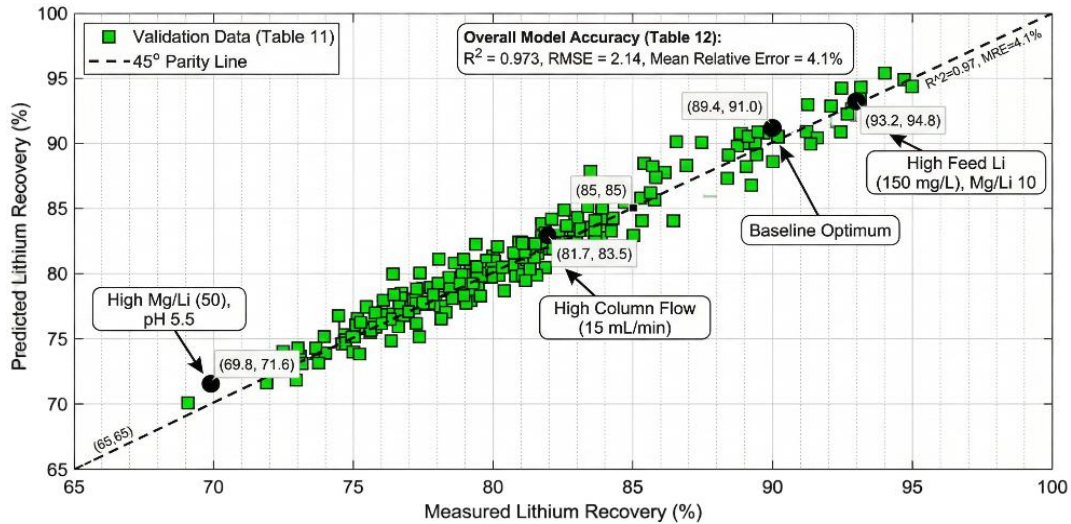


Fig. 8. Comparison of lithium recovery and selectivity among membrane-only, adsorbent-only, and hybrid systems

Table 11. Comparison of simulated and measured process performance

Condition	Predicted lithium recovery (%)	Measured lithium recovery (%)	Relative error (%)	Predicted q_e (mg/g)	Measured q_e (mg/g)	Relative error (%)
Baseline optimum	91.0	89.4	1.8	25.3	24.8	2.0
High Mg/Li (50), pH 5.5	71.6	69.8	2.6	18.7	18.0	3.9
High Li (150 mg/L), Mg/Li 10	94.8	93.2	1.7	27.8	27.1	2.6
High column flow rate (15 mL/min)	83.5	81.7	2.2	23.4	22.8	2.6

**Fig. 9.** Parity plot of predicted versus experimental lithium recovery

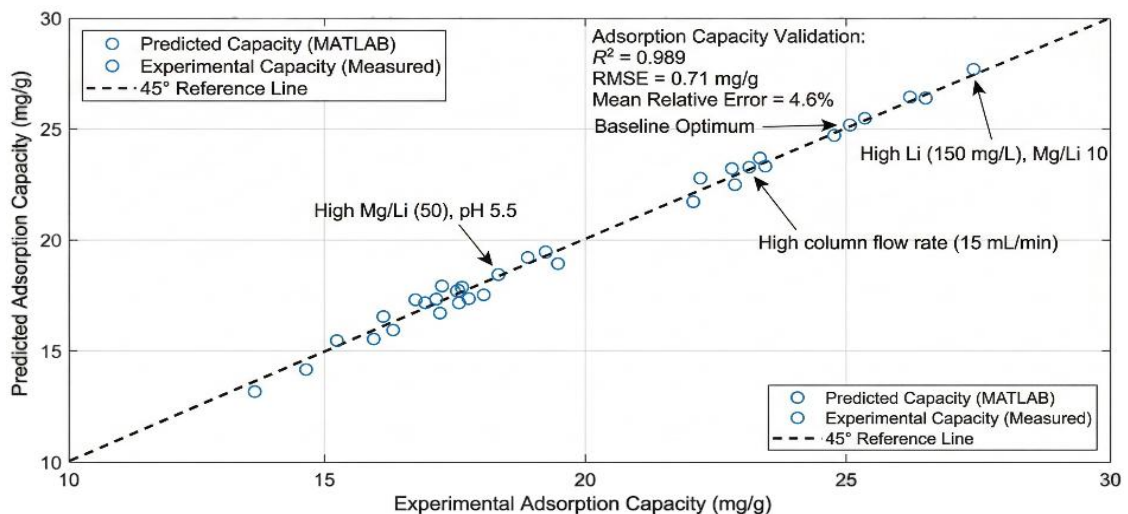
The overall predictive accuracy is summarized in Table 12.

The results present the model to be accurate enough to predict the process level performance and the behaviour of the adsorbents. The minor difference between the predicted and measured values can be explained considering the non-ideal hydrodynamics, concentration polarization at the membrane surface and intraparticle

diffusion limitation in the packed adsorbent bed. The use of synthetic brine should also be acknowledged as a validation boundary: while it was used to assess the ion competition and model accuracy, real brines could contain organic matter, silica, suspended solids and variability in trace ions, that could affect fouling and selectivity and regeneration performance.

Table 12. Model accuracy metrics

Model output	R ²	RMSE	Mean relative error (%)
Lithium recovery	0.973	2.14	4.1
Adsorption capacity	0.989	0.71 mg/g	4.6
Lithium uptake rate	0.984	0.64 mg/g	5.0

**Fig. 10.** Parity plot of predicted versus experimental adsorption capacity

3.4. Stability and reusability

Short term stability of the hybrid system was evaluated after 10 cycles of extraction-desorption. Note that the permeability of the membrane and the adsorption performance gradually decreased with use, but the losses were not severe, which means the membranes have a good operational robustness.

Membrane flux retention was higher than 90% after 10 cycles, which suggests that there was no significant fouling or damage to the membrane structure during the

test period. The initial capacity of the adsorbent was not affected more than 92% and the regeneration efficiency was over 91%. In cycle 1, the recovery of lithium was 89.4% but it dropped to 84.7% in cycle 10. A slight decrease in Li/Mg selectivity was also observed, possibly owing to the slow build-up of competing ions on the adsorbent surface, or to a proportionate decrease in the number of active sites. The values in the figures and tables are expressed as mean $\pm$ standard deviation and error bars are provided when multiple replicates are available.

Table 13. Stability and reusability of the hybrid system over repeated cycles

Cycle number	Membrane flux (L m ⁻² h ⁻¹)	Membrane stability index (%)	Adsorption capacity, q _e (mg/g)	Regeneration efficiency (%)	Lithium recovery (%)	S _{Li/Mg}
1	14.2 $\pm$ 0.3	100.0	24.8 $\pm$ 0.4	96.4 $\pm$ 0.8	89.4 $\pm$ 1.2	38.7 $\pm$ 1.1
5	13.6 $\pm$ 0.2	95.8	23.9 $\pm$ 0.5	94.2 $\pm$ 0.9	86.8 $\pm$ 1.1	35.0 $\pm$ 1.0
10	13.0 $\pm$ 0.3	91.5	22.9 $\pm$ 0.6	91.8 $\pm$ 1.0	84.7 $\pm$ 1.3	32.1 $\pm$ 1.2

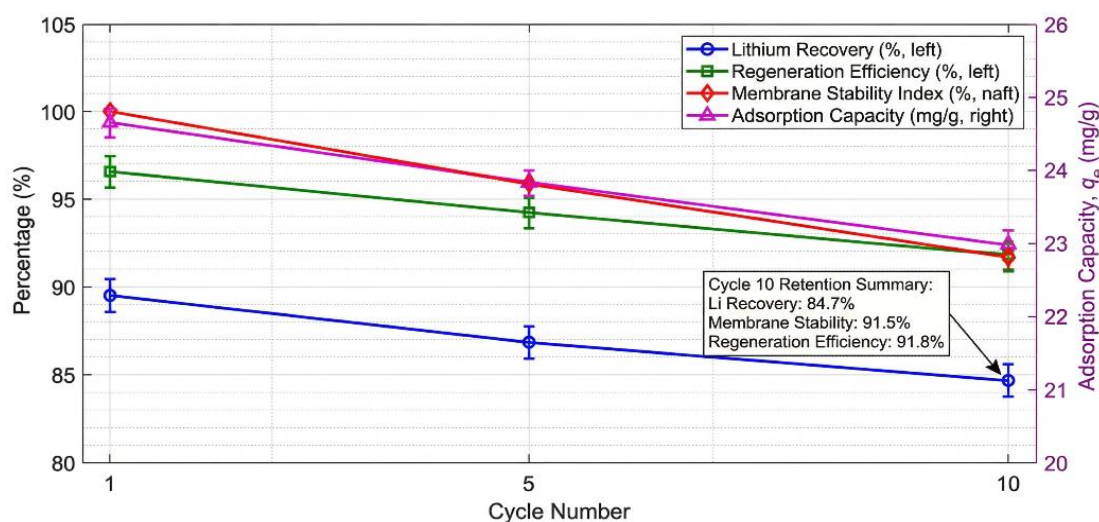


Fig. 11. Cyclic stability of the hybrid membrane-adsorbent system over 10 cycles

3.5. Comparison with previous studies

After 10 extraction-regeneration cycles, the current hybrid membrane-adsorbent system was able to remove 89.4% of the lithium and had a selectivity coefficient of 38.7 for lithium removal from magnesium. These values are higher than those obtained in most of the standalone membrane-pretreatment and conventional adsorption-only systems, but lower than the highest selectivities reported in some electro-driven hybrid systems. However, under controlled conditions, recent membrane studies demonstrated biomimetic and two-dimensional membranes with Li/Na selectivities up to 15.3 and >20.9, and with Li/K selectivities up to 19.9 and 31.2, respectively [18, 19] but still challenges remain on durability, fouling and performance in the hypersaline multicomponents feeds. Integrated membrane-process reviews also suggest that nanofiltration is best applied as a pretreatment step to lower the Mg/Li ratio to enable the downstream enrichment and polishing by other lithium-capture processes rather than as a stand-alone lithium-

capture process [7, 8]. It is consistent with the current results, in which the main removal of divalent-ion contaminants was done by the membrane stage and the main selectivity was ensured by the adsorbent stage. The present system is also competitive with the adsorption-based system [15, 20].

Compared to the results of the 25.94 mg g⁻¹ capacity and 81.84% recovery reported by Ding et al. [20] for a manganese-titanium ion sieve after five cycles, the present hybrid system was able to preserve 22.9 mg g⁻¹ capacity and 84.7% recovery after 10 cycles, showing slightly lower capacity per cycle but higher cyclic durability of the process. Similar cyclic robustness and selectivity towards H₂TiO₃ granular ion sieves and ion-sieving MOFs have been reported as well [21, 16]. From the selectivity point of view, Yu et al. [22] achieved active selectivity factors > 100 for a Li₂TiO₃ membrane-coated capacitive deionization system over 10 cycles, while Zhang et al. [23] developed a solar-driven membrane architecture which reduced the ratio of Mg²⁺/Li⁺ by 66-fold. Electro-driven geothermal-brine

recovery has also yielded promising product-quality results [24] and electrochemical reviews have been found that recount the overall benefits of geothermal-brine pathways [25]. In general, the comparison suggests that the present hybrid system is technically viable and provides a balance of high recovery, good ion discrimination and acceptable cyclic stability in an operation of lower complexity.

3.6. Implications

The results have significance to direct lithium extraction from low concentrated brines. Firstly, they are not against a staged-separation approach, but enable complementary functions between membrane pretreatment and selective adsorption [7-8]. This is significant because recent studies highlighted the need to use an integrated membrane system when the lithium recovery process involves both impurity suppression and stable operation with chemically complex feeds [11, 8, 17]. Second, the good correlation between the Aspen Plus/MATMAT predictions and experimental values obtained from the laboratory indicates that model-based design can speed up experimental optimization as compared to trial-and-error optimization [12, 24, 25]. This simulation-guided hybridization could be a viable approach to developing more selective and scalable DLE process for low-grade resources like geothermal water, oilfield brine, and desalination concentrate [26, 27].

3.7. Limitations and future directions

There are some limitations of this study. The experiments were conducted in synthetic brine instead of real geothermal and salt-lake systems, and as a result do not fully reflect the fouling potential, organic contamination, suspended solids, silica content, trace elements, or variations in feed-composition of real geothermal/salt-lake systems, which are routinely cited as significant limitations of industrial membrane-based lithium recovery [7, 17, 26]. Furthermore, only 10 cycles were performed in the cyclic experiment, so it was limited in order to provide preliminary stability assessment but not to provide lifetime assessment. A partial transport-resolved model is not used to describe the membrane stage; instead, the rejection behavior of the membrane is calibrated. The following studies should therefore be conducted using real brines, with stability testing extended to 50-100 cycles and using combined transport-resolved membrane modeling, techno-economic and life-cycle analyses, and determining the potential of electro-assisted or solar-assisted pretreatment to increase selectivity without undue complexity of the process [22, 24, 28].

4- Conclusion

In practice, this study presented a nanofiltration (NF) pretreatment combined with lithium-ion-sieve (H_2TiO_3) adsorption to remove lithium from low concentration

synthetic brine to enhance the lithium extraction efficiency. The membrane stage decreased the concentration of Mg^{2+} and Ca^{2+} by 78.0% and 84.4% respectively, under optimum conditions with the transport of lithium. The hybrid process recovered 89.4% of the lithium, adsorbed 24.8 mg g^{-1} and had a selectivity coefficient of $\text{Li}/\text{Mg} = 38.7$. The extraction-regeneration cycle performance results showed that lithium recovery was 84.7%, membrane flux was 91.5% and regeneration efficiency was 91.8% after 10 cycles. The combined process simulation-experimental workflow was found to be useful for process optimization and scale-up assessment, as the model results correlated well with the experimental results in the bench scale. To deploy the system in industry, future work should be conducted with real brines, longer cycling tests, fouling analysis and techno-economic and life cycle evaluations. The overall results suggest that integration of hybrid membranes and adsorbents is a potential approach for sustainable recovery of lithium from low-grade aqueous resources.

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الأنظمة الهجينة للغشاء-الممتزات للاستخلاص المباشر لليثيوم من المحاليل الملحية منخفضة التركيز

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الخلاصة

تهدف هذه الدراسة إلى تطوير وتقييم نظام هجين يجمع بين الفصل الغشائي والامتزاز من أجل الاستخلاص المباشر لليثيوم من المحاليل الملحية منخفضة التركيز. تم اعتماد منهجية تكاملية شملت المحاكاة الحرارية باستخدام Aspen Plus ، ونمذجة حركية الامتزاز باستخدام MATLAB ، إلى جانب التحقق التجريبي على مقياس مختبري باستخدام محلول ملحي صناعي. أظهرت النتائج أن مرحلة الغشاء أسهمت في خفض تراكيز الأيونات الثنائية المتداخلة، وخاصة المغنيسيوم والكالسيوم، بنسبة ٧٨,٠% و ٨٤,٤% على التوالي، مع الحفاظ على انتقال الليثيوم بنسبة ٨٤,٠%. كما حقق النظام الهجين نسبة استخلاص لليثيوم بلغت ٨٩,٤%، وسعة امتزاز مقدارها ٢٤,٨ ملغم/غم، ومعامل انتقائية لليثيوم على المغنيسيوم بلغ ٣٨,٧. وبعد ١٠ دورات تشغيلية، بقيت نسبة استخلاص الليثيوم عند ٨٤,٧%، مع احتفاظ الغشاء بـ ٩١,٥% من كفاءته، وكفاءة تجديد بلغت ٩١,٨%. تؤكد هذه النتائج أن دمج الفصل الغشائي مع الامتزاز يمثل نهجاً واعداً وقابلاً للتوسع من أجل الاستخلاص المستدام لليثيوم من الموارد الملحية منخفضة الجودة.

الكلمات الدالة: الاستخلاص المباشر لليثيوم، المحاليل الملحية منخفضة التركيز، النظام الهجين غشاء-ممتز، منحل أيوني لليثيوم، الترشيح النانوي، حركية الامتزاز، الانتقائية، الثبات الدوري.