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Adsorption-membrane filtration hybrid method for separation of lithium from geothermal water and its reverse osmosis concentrate by spinel type λ-MnO_2

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The adsorption-membrane filtration hybrid process can be considered as an alternative method to classical fixed-bed adsorption for lithium recovery from aqueous media. This technique integrates adsorption and membrane filtration by combining advantages of both processes. It is possible to use fine particles having high surface area and faster kinetics in the adsorption-membrane filtration integrated method as adsorbent. Thus, less amount of adsorbent can be employed in the process and the cost of the process can be reduced. In this study, adsorption-membrane filtration process was utilized for lithium separation from geothermal water and its reverse osmosis concentrate by means of lithium selective adsorbent spinel type λ-MnO_2. During hybrid tests, effect of some operational parameters as adsorbent concentration, particle size of adsorbent, replacement speeds of saturated and fresh adsorbents, flow rate of permeate on lithium separation was investigated. In hybrid tests, the optimal operating conditions with powder λ-MnO_2 adsorbent were defined as 2 g/L adsorbent concentration, 5 mL/min as permeate/feed flow rate, and 6 mL/min as adsorbent replacement rate. It was determined that by reducing permeate/feed flow rate and increasing saturated/fresh adsorbent replacement rate, the lithium separation could be accomplished more successfully.

KEYWORDS
adsorption, concentrate management, geothermal water, hybrid process, ion exchange, lithium, reverse osmosis, ultrafiltration

1 Introduction

Lithium is recognized as a strategic metal for global energy generation and technological advancement due to its exceptionally high redox potential, considerable specific heat capacity, pronounced electrochemical reactivity, and low coefficient of thermal expansion (Swain, 2016; Gu et al., 2024). Lithium and its salts, including lithium carbonate, lithium hydroxide, and lithium chloride, exhibit distinctive physical,

chemical, and electrochemical properties that render them essential in applications such as battery production, heat-resistant glass and ceramics, grease lubricants, and continuous casting mold flux powders. Furthermore, lithium salts are utilized in the pharmaceutical field as effective agents for the treatment of bipolar disorder (Machado-Vieira et al., 2009; Swain, 2017; Craddock, 2018; Murphy and Haji, 2022; Nikkhah et al., 2024; Ali et al., 2025).

Across various industrial sectors, the rechargeable battery industry has become the primary driver of lithium demand, which has risen sharply in recent years. This trend is expected to continue, driven by the global shift toward low-carbon energy systems and the rapid electrification of transportation. In this context, reliable energy storage solutions have become indispensable for mitigating pollution, reducing greenhouse gas emissions, and addressing the limitations of fossil fuel resources. As a result, lithium-ion batteries have emerged as the key enabling technology for large-scale renewable energy integration and the electrification of the transportation sector (Tarascon, 2010; Durmuş and Kaymaz, 2020; Disu et al., 2024; Nikkhah et al., 2024).

Given these favorable properties, global lithium demand across all these sectors is escalating rapidly (Nikkhah et al., 2024). Traditional lithium production methods such as hard-rock mining of spodumene or lepidolite, solar evaporation of continental or salar brines, and recycling of spent lithium batteries have been the primary sources (Kipouros and Sadoway, 1998; Vikström et al., 2013; Zhang et al., 2019; Murphy and Haji, 2022; Kölbel et al., 2023; Disu et al., 2024; Gu et al., 2024; Ali et al., 2025). However, lithium is a finite resource with surging demand, and at the current rate of consumption, terrestrial lithium reserves are anticipated to face significant depletion in the foreseeable future, which is increasingly driving the search for alternative lithium recovery routes (Murphy and Haji, 2022; Kölbel et al., 2023; Gu et al., 2024).

Despite the absence of a universal extraction method for lithium from brines, a variety of techniques, including adsorption, precipitation, electrodialysis, ion exchange, and solvent extraction, have been proposed. More recently, emerging approaches such as direct lithium extraction from geothermal brines, electrochemical leaching of lithium-bearing clays, biological leaching using microorganisms, and mechano-chemical recovery from low-grade ores or waste streams have attracted growing attention in the literature (Barbosa et al., 2014; Meshram et al., 2014; Harvianto et al., 2016; Flexer et al., 2018; Kaya et al., 2022; Gu et al., 2024; Nikkhah et al., 2024).

Geothermal fluids are becoming acknowledged as a viable unconventional source of lithium owing to their passage through lithium-rich rocks and elevated geochemical reactivity at high temperatures. These fluids frequently extract lithium from subterranean volcanic or granitic deposits, facilitating its concentration in brines over extended underground residency periods. The geothermal environment promotes lithium mobilization by augmenting mineral dissolution and ion exchange processes, especially at increased temperatures and pressures (Sanjuan et al., 2016; Cao et al., 2025).

The lithium concentration in geothermal waters can range from a few milligrams to over 400 mg/L depending on geological settings, water-rock interactions, and the depth of circulation (Sanjuan et al., 2016). Notably, brines with higher salinity and temperature often show elevated lithium content. Geothermal reservoirs in Western Anatolia have been identified as potential lithium sources, with studies reporting concentrations in selected geothermal brines ranging from a few milligrams per liter up to tens of milligrams per liter, offering potential for resource recovery (Cetiner et al., 2015; Sengun Cetin et al., 2024; Warren, 2021). Lithium concentrations in geothermal waters range from 1 to 100 mg/L (Flexer et al., 2018), which is significantly higher than seawater (~0.17 mg/L), In addition to greater lithium concentrations, geothermal fluids have lower ion content than seawater, making lithium separation easier.

The reverse osmosis (RO) concentrates derived from geothermal fluids represent a significant secondary lithium source, which remains underutilized and poses environmental challenges due to the volume and chemical composition of the concentrate stream. In this framework, spinel-type λ-MnO_2 adsorbents in both powder and granular forms were investigated for the selective recovery of lithium from geothermal reverse osmosis concentrates. The results demonstrated that the powder-form adsorbent exhibited significantly faster adsorption kinetics and higher lithium uptake capacity compared to the granular form (Sever et al., 2025).

When evaluating geothermal brines, it is essential to account for the other ions that coexist with lithium. This diverse array of ions includes potassium, sodium, boron, calcium, magnesium, arsenic, chloride, sulfur, nitrate, and bicarbonate. Recently, researchers have focused on the presence of Mg^{2+} ions, which have a comparable ionic hydration radius and chemical properties to those of Li^+ ions. These similarities complicate the separation process (Gunerhan et al., 2001; Paranthaman et al., 2017; Ping, 2022).

Owing to economic and environmental considerations, adsorption using selective adsorbents is widely regarded as a promising approach for lithium removal from brines with high Mg^{2+}/Li^+ ratios. Conventional methods such as precipitation often face economic limitations under such conditions due to the relatively high solubility of Li_2CO_3, which leads to low recovery efficiency. In contrast, selective inorganic adsorbents can achieve high Li^+ selectivity even in the presence of excess competing ions, reducing the need for extensive pre-treatment. In addition, their chemical stability and reusability contribute to improved process economics over multiple cycles. These characteristics suggest that adsorption-based systems offer a cost-effective alternative, although a detailed economic evaluation is beyond the scope of this study (Tian et al., 2010; Abe and Chitrakar, 1987).

A multitude of inorganic adsorbents has been extensively examined, with manganese-based lithium ion sieves (LIS) being regarded as the most promising materials for the extraction of lithium from geothermal brines (Chitrakar et al., 2012; Yu et al., 2013; Zandevakili et al., 2014; Stringfellow and Dobson, 2021). Manganese-based LIS with diverse crystal structures have been synthesized and evaluated as ion sieve precursors. The diverse valence states of manganese and the varying Li/Mn mole ratios employed in ion sieve synthesis have facilitated the development of manganese-based LIS with distinct crystal structures (Xu et al., 2016). The λ-MnO_2 adsorbent is a manganese-based LIS adsorbent produced through the topotactic extraction of lithium ions from spinel-type lithium dimanganese tetraoxide

TABLE 1 Compositions of geothermal waters, RO concentrate of geothermal water.

Parameters	Unit	Geothermal water Aydın–Sultanhisar region	Geothermal water Manisa–Alaşehir region	Geothermal water Izmir Yenikale (adapted from Jarma et al., 2021)	RO concentrate of geothermal water	Li-spiked RO concentrate of geothermal water
TDS	g/L	3.11	3.09	1.23	2.74	3.04
Conductivity	mS/cm (25 °C)	4.35	3.81	1.81	5.74	5.86
pH	-	7.40	9.28	8.52	8.70	8.70
Salinity	‰	2.4	2.1	0.9	3.10	3.10
Li	mg/L	4.20	5.70	1.40	4.80	12.0
As	μg/L	480	2,200	170	429	429
B	mg/L	48	108	12.0	16.0	16.0

($Li_{1.5}Mn_2O_4$) (Hunter, 1981; Yoshizuka et al., 2006; Oshima et al., 2025). During acid treatment, Li^+ ions in the spinel structure are replaced by H^+ ions while preserving the manganese oxide framework, creating lithium-selective vacant sites. During adsorption, Li^+ions are selectively reinserted into these sites through an ion-exchange mechanism, which is generally referred to as the ion-sieve effect (Sever, 2022). In addition, previous studies have shown that the adsorption performance of lithium ion sieves can be significantly influenced by particle morphology and size. Smaller particles generally provide shorter diffusion pathways, improved accessibility of lithium-selective sites, and enhanced adsorption kinetics (Sever, 2022; Sever et al., 2025). Consequently, powdered λ-MnO_2 is often reported to exhibit superior lithium uptake performance compared to granulated forms, which is consistent with the results obtained in the present study.

Yoshizuka et al. (2002) developed and evaluated λ-MnO_2 adsorbent for the selective separation of lithium from an artificial seawater solution containing Li^+ ions and elevated concentrations of Na^+ ions (>10,000 mg/L) (Yoshizuka et al., 2002). It was demonstrated that the molar selectivity of Li^+ was approximately 2,280 times higher than that of Na^+ ions. Moreover, an increase in solution pH enhanced the lithium separation capacity of the adsorbent. These results indicate that λ-MnO_2 can effectively adsorb Li^+ ions even under high Na^+/Li^+ molar ratios. In a subsequent study, Yoshizuka et al. (2006) reported a lithium recovery rate of 33.3% from seawater containing various alkali and alkaline-earth elements (Yoshizuka et al., 2006). Furthermore, Nishihama et al. (2011) successfully purified lithium and synthesized Li_2CO_3 with a purity exceeding 99.9% (Nishihama et al., 2011).

Despite the growing interest in lithium recovery from alternative resources, studies conducted using real geothermal fluids remain limited, as many investigations in the literature rely on synthetically prepared geothermal water solutions.

TABLE 2 Characteristics of the membrane module used in the hybrid adsorption–membrane filtration system.

Membrane process	Ultrafiltration (UF)
Model	ZeeWeed-1 (ZW-1), submersible module
General type	Supported, non-ionic,hydrophilic
Outside diameter (mm)	2
Effective membrane surface area (m^2)	0.047
Nominal pore diameter (μm)	0.04
Hold up volume (mL)	10
Maximum permeation pressure (bar)	0.6
Operating transmembrane pressure (bar)	0.07–0.55
Permeate flow range (mL/min)	5–25
Maximum operating (cleaning) temperature (°C)	40
Operating pH range	5–9
Cleaning pH range	2–10.5

Similarly, research addressing the utilization of RO concentrates of geothermal waters as a potential lithium source is still scarce. In addition, studies focusing on the comparative performance of powdered and granulated lithium manganese dioxide (λ-MnO_2) adsorbents and their integration into hybrid membrane–adsorption systems are also limited in the literature.

This study aims to investigate lithium recovery from different geothermal fluid and the RO concentrate of geothermal water using integrated ion exchange based methods. In this context, spinel type lithium manganese dioxide (λ-MnO_2) adsorbent was employed,

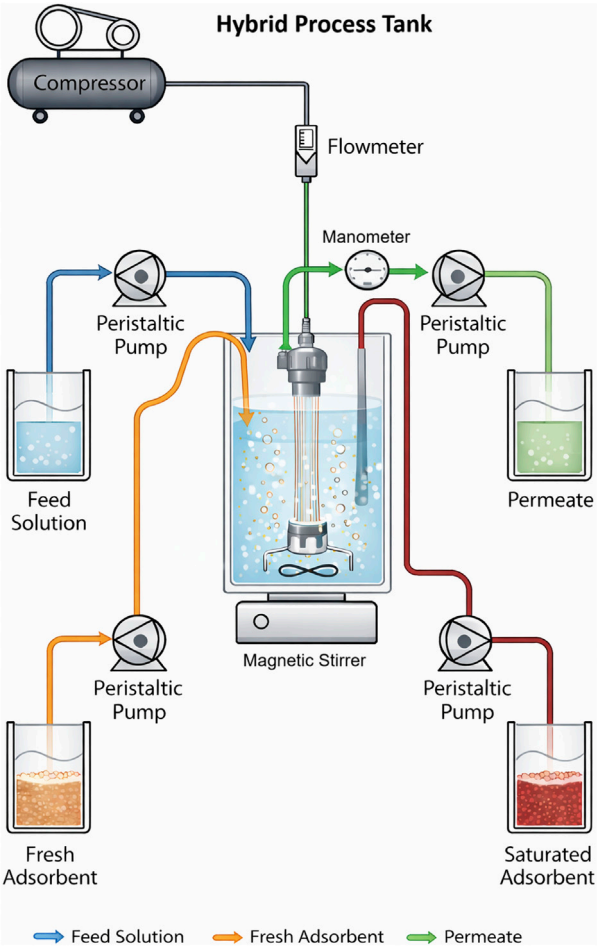


FIGURE 1
Schematic representation of hybrid system experimental set-up.

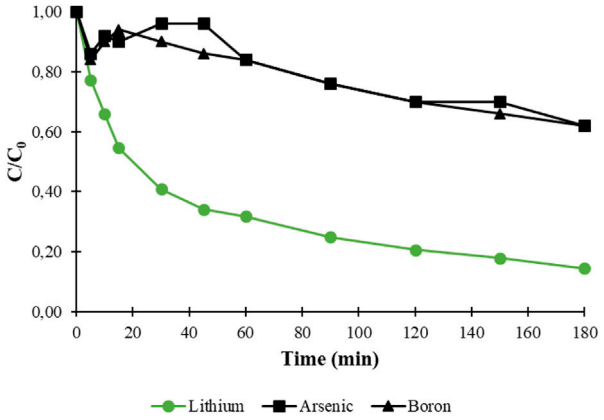


FIGURE 2
C/C_0 versus time plot for lithium, arsenic and boron adsorption in geothermal fluid from the Aydın–Sultanhisar region (Qs: 6 mL/min, Qp: 5 mL/min).

using a hybrid system combining the adsorption process with membrane filtration was applied to separate lithium.

2 Experimental

2.1 Materials and methods

2.1.1 Preparation of spinel type λ-MnO_2

The spinel type λ-MnO_2 adsorbent was synthesized from the precursor $Li_{1.5}Mn_2O_4$ using a method analogous to that described in the existing literature (Yoshizuka et al., 2021). The resultant material was granulated with an alumina-based binder (Cataloid AP-1), provided by Catalysts and Chemicals Ind. Co. Ltd (Kanagawa, Japan). The granulated adsorbent's density, determined using a pycnometer, was 3.42 g/cm^3.

A certain amount of precursor was contacted with 1.0 M HCl solution overnight with continuous agitation to facilitate ion exchange between Li^+ ions in the precursor and H^+ ions in the HCl solution, resulting in the formation of λ-MnO_2 adsorbent. Upon completion of the acid treatment (after the conversion of $Li_{1.5}Mn_2O_4$ to λ-MnO_2), the resulting spinel type λ-MnO_2 adsorbent was rinsed with deionized water until the excess acid was removed. Subsequently, the vacuum filtration was carried out to filter the λ-MnO_2 adsorbent using a membrane filter with a pore size of 0.45 microns. The adsorbent was then air-dried and subsequently put in a vacuum oven until a constant weight is reached.

2.1.2 Geothermal water samples

During initial studies, geothermal waters collected from production wells operated for electricity generation at two different geothermal sites (Aydın–Sultanhisar and Manisa–Alaşehir regions) were used as representative feed solutions.

The RO concentrate samples of geothermal water were collected via membrane experiments conducted utilizing a mini-pilot scale RO plant at the Yenikale Geothermal Heating Center of Izmir Geothermal Co. Initially, the geothermal water discharged from the Geothermal Heating Center was treated with prefiltration using sand and cartridge filters. The geothermal fluid was then subsequently processed using a mini pilot-scale RO treatment system utilizing spiral wound BW30 RO membrane to collect the RO concentrate samples. The RO treatment process was conducted at a pressure of 15 bar, achieving a permeate recovery rate of 70%.

The RO concentrate was subsequently spiked with LiCl to get an initial Li^+ concentration of 10.5 ± 1.5 mg/L in the sample. The characteristics of the geothermal waters and the RO concentrate samples utilized in the hybrid tests were depicted in Table 1.

2.1.3 Adsorption-membrane filtration hybrid studies

A ZeeWeed®-1 (ZW-1) GE-type hollow-fiber ultrafiltration membrane module was employed in the adsorption–membrane filtration experiments, and its main characteristics are summarized in Table 2. Figure 1 present the flow diagram of the hybrid system and the experimental setup.

The ultrafiltration (UF) hollow-fiber membrane module was submerged in a vessel for filtration purpose during the adsorption-

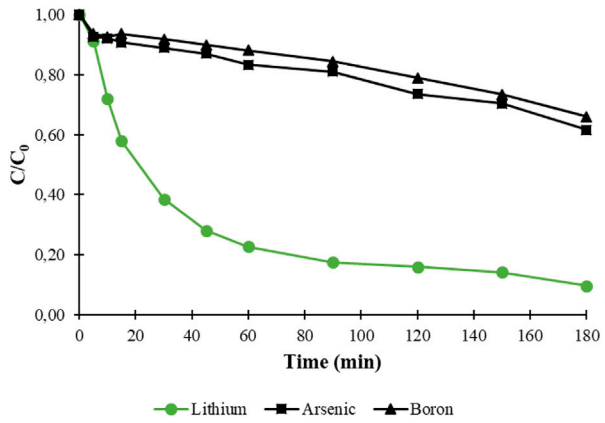


FIGURE 3
C/C_0 versus time plot for lithium, arsenic and boron adsorption in geothermal fluid from the Manisa–Alaşehir region (Qs: 6 mL/min, Qp: 5 mL/min).

membrane filtration hybrid study. Geothermal fluids and Li-spiked RO concentrate of the geothermal brine was fed into the vessel along with suspensions of fresh λ-MnO_2 adsorbent in deionized water simultaneously while the saturated λ-MnO_2 adsorbent and permeate were also simultaneously withdrawn from the vessel. The fresh adsorbent tank and the vessel were continuously stirred to maintain a homogeneous suspension. The submerged UF membrane retained the adsorbent particles while allowing permeate collection from the vessel. To mitigate membrane fouling, air was continuously supplied to the membrane module at a flow rate of 4 mL/min.

To mitigate membrane fouling, continuous air supply was applied during the experiments at a flow rate of 4 mL/min. In addition, the submerged hollow-fiber UF system operates within a relatively low transmembrane pressure range (0.07–0.55 bar), which may further limit severe foulant accumulation and compact cake layer formation on the membrane surface during short-term operation. Feed and permeate streams were delivered and withdrawn, respectively, using a multichannel peristaltic pump (Masterflex L/S Easy-Load II), whereas fresh and saturated adsorbent solutions were pumped into the vessel and taken from the vessel, respectively, through another multichannel pump.

The feed solutions were introduced into the vessel containing the submerged UF module. After system start-up, fresh λ-MnO_2 adsorbent was added into the vessel as required during operation. The concentrations of powdered and granulated λ-MnO_2 were predetermined based on preliminary optimization studies. The feed and permeate flow rates were defined as 5 and 10 mL/min and adjusted to maintain a constant liquid level in the hybrid vessel. The flow rates of the fresh and saturated adsorbent suspensions were maintained at 3 and 6 mL/min throughout the operation. Each study lasted 3 h, during which permeate samples were collected at predetermined time intervals (0, 5, 10, 15, 30, 45, 60, 90, 120, 150, and 180 min). Blank experiments were performed by using either geothermal water or Li-spiked RO concentrate of geothermal water in the hybrid vessel and deionized water in the fresh adsorbent tank without feeding any adsorbent in order to evaluate the dilution effect of deionized water because during all sets, the adsorbent was supplied into vessel as a suspension in deionized water.

For the hybrid tests run with geothermal water, 1.5 L of geothermal water collected from the Aydın–Sultanhisar and Manisa–Alaşehir regions was introduced into the vessel containing the submerged UF module. The concentrations of powdered λ-MnO_2 used in hybrid experiments were 0.5 g/L. The feed and permeate flow rates were set at 5 mL/min and regulated to maintain a constant liquid level in the hybrid vessel. The flow rates of the fresh and saturated adsorbent suspensions were maintained at 6 mL/min throughout the operation.

The hybrid tests using the RO concentrate of geothermal fluid, which was collected from a field study, 1.5 L of the RO concentrate spiked with lithium was employed. The concentrations of powdered λ-MnO_2 used in hybrid experiments were 1.0 g/L and 2.0 g/L, while granulated λ-MnO_2 was tested at 2.0 g/L. The flow rates of Li-spiked RO concentrate (feed) and permeate were maintained at 5 and 10 mL/min (Qp), respectively, ensuring a constant liquid level in the hybrid process vessel. The flow rates of fresh and saturated adsorbent solutions were kept equal at 3 and 6 mL/min.

2.2 Analyses

The chemical analyses of geothermal waters from the Aydın–Sultanhisar, Manisa–Alaşehir regions, the RO concentrate of Izmir Yenikale geothermal water, and Li-spiked RO concentrate solutions were performed using an ICP-OES system (Perkin Elmer Avio 200). Lithium concentrations were determined using an atomic absorption spectrometer (Shimadzu AA-7000). Additional water quality parameters, including salinity, pH, and conductivity, were measured using a Hach Lange HQ40d and WTW 3420 multimeter, whereas total dissolved solids (TDS) were determined gravimetrically.

3 Results and discussion

3.1 Adsorption-membrane filtration hybrid studies using geothermal fluids

The application of small-sized adsorbents in fixed-bed column operations is limited due to substantial pressure drops. Koltuniewicz et al. (2004) reported that operating costs will increase in direct proportion to the amount of adsorbent employed (Koltuniewicz et al., 2004). As particle size increases, the specific surface area decreases, thereby requiring a larger quantity of adsorbent to achieve comparable solute adsorption, which subsequently elevates operational costs. These limitations have prompted researchers to explore the development and optimization of powdered adsorbents. Yoshizuka et al. (2002) and Kitajou et al. (2003) investigated strategies to mitigate significant pressure drops in chromatographic operations through the use of powdered λ-MnO_2. To address this issue, they granulated the adsorbent to enhance its performance in continuous lithium separation processes (Yoshizuka et al., 2002; Kitajou et al., 2003). Recepoğlu et al. (2018) proposed integrating complementary processes to

TABLE 3 Summary of comparison study in hybrid system (effect of adsorbent type).

Adsorbent	C (g/L)	Qp (mL/min)	Qs (mL/min)	[Li]o (mg/L)	$[Li]_f$ (mg/L)	R (%)
Powder	2	5	6	10.85	~0.00	100
Granulated				8.79	0.13	99

C: adsorbent concentration; Q_s: Fresh and saturated adsorbents replacement rate; Q_P: Feed and Permeate flowrate. $[Li]_0$: Lithium concentration in permeate at t = 0 min; $[Li]_f$: Lithium concentration in permeate at t = 180 min. R: lithium separation efficiency.

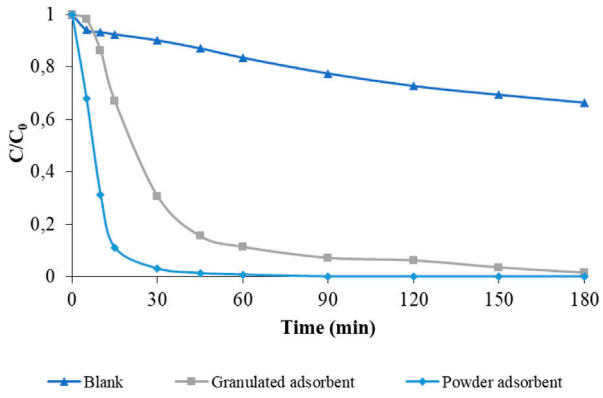


FIGURE 4
C/C_o vs. time plots of adsorbent type (C = 2 g/L, Q_s = 6 mL/min, Q_p = 5 mL/min).

improve the efficiency of fixed-bed adsorption. They investigated a hybrid system combining UF membrane technology with adsorption to overcome the challenge of separating powdered adsorbents from solution, while also leveraging the synergistic effects occurring on the membrane surface. The parameters examined included adsorbent type, adsorbent concentration, saturated adsorbent replacement rate, and permeate flow rate for lithium recovery from geothermal water (Recepoğlu et al., 2018). Building upon these findings, a hybrid membrane–adsorption approach was employed for lithium recovery from geothermal fluids collected from two different geothermal power plants in Türkiye.

Lithium, arsenic, and boron separations from geothermal fluids collected from the Aydın–Sultanhisar and Manisa–Alaşehir regions were investigated using the adsorption–membrane filtration hybrid method. The results were evaluated by plotting the ratio of element concentrations in the geothermal fluid at a given time to their initial concentrations (C/C_o) versus time. The lithium, arsenic, and boron results of the experiments conducted using geothermal fluids from the Aydın–Sultanhisar and Manisa–Alaşehir regions are presented in Figures 2, 3, respectively.

Since the fresh adsorbent was introduced into the system as a suspension prepared in deionized water, dilution effects could influence the concentrations of lithium, arsenic, and boron during operation. Therefore, additional blank experiments were conducted under identical experimental conditions without λ-MnO_2 adsorbent in order to evaluate the contribution of dilution to concentration changes.

As shown in Figure 2, lithium concentration in the Aydın–Sultanhisar geothermal fluid decreased significantly throughout the hybrid process, whereas arsenic and boron concentrations exhibited comparatively minor changes. After 180 min, lithium separation reached 85%, demonstrating the strong lithium selectivity of λ-MnO_2 under hybrid operating conditions. In contrast, arsenic and boron concentrations remained relatively stable during the experiments, indicating that λ-MnO_2 did not exhibit significant affinity toward these species.

After 180 min, lithium concentration decreased from 4.4 mg/L to 0.64 mg/L in the presence of the adsorbent, whereas it decreased to 2.6 mg/L in the blank system. Arsenic concentration decreased from 0.50 mg/L to 0.31 mg/L in the presence of the adsorbent and to 0.34 mg/L in the blank system. Similarly, boron concentration decreased from 50 mg/L to 31 mg/L with the adsorbent and to 32 mg/L in the blank system. These findings indicate that the observed concentration decreases for arsenic and boron were mainly associated with dilution effects rather than selective adsorption onto λ-MnO_2.

A similar trend was observed for the geothermal fluid collected from the Manisa–Alaşehir region, as presented in Figure 3. Lithium concentration decreased continuously during the hybrid process, reaching 90% separation after 180 min. Meanwhile, arsenic and boron concentrations exhibited only limited variation throughout the experiments. These findings confirm the lithium ion-sieve behavior of λ-MnO_2 and its high selectivity toward lithium in complex geothermal matrices.

Lithium concentration decreased from 5.7 mg/L to 0.55 mg/L in the presence of λ-MnO_2, whereas it decreased to 3.2 mg/L in the blank system after 180 min. Arsenic concentration decreased from 2.2 mg/L to 1.3 mg/L with the adsorbent and from 2.1 mg/L to 1.2 mg/L in the blank system. Similarly, boron concentration decreased from 109 mg/L to 72 mg/L in the presence of the adsorbent and from 107 mg/L to 61 mg/L in the blank system. These results indicate that the observed concentration decreases for arsenic and boron were mainly associated with dilution effects rather than selective adsorption onto λ-MnO_2. The slightly higher boron concentration decrease observed in the blank experiment suggests that boron removal was not associated with selective adsorption onto λ-MnO_2. The observed variations are considered to be related to dilution and experimental fluctuations within the hybrid system.

Overall, the results obtained from both geothermal fluids using the adsorption–membrane filtration hybrid system demonstrate that λ-MnO_2 exhibits high selectivity toward lithium. In contrast, arsenic and boron concentrations showed only limited variation during the experiments, indicating that λ-MnO_2 did not exhibit significant affinity toward these species under the investigated conditions. The findings confirm the lithium ion-sieve behavior of λ-MnO_2 under hybrid system operation and highlight its effectiveness for selective lithium separation from geothermal fluids.

TABLE 4 Summary of comparison study in hybrid system (effect of adsorbent concentration as a function of feed and permeate flow rate).

Adsorbent	C (g/L)	Q_p (mL/min)	Q_s (mL/min)	$[Li]_0$ (mg/L)	$[Li]_f$ (mg/L)	R (%)
Powder λ-MnO_2	1	5	6	8.87	0.13	98.5
	2			10.85	0.00	100.0
	1	10	6	9.06	0.71	92.1
	2			9.10	0.50	94.5
Blank	0	5	6	8.71	5.78	33.7
		10	6	8.94	6.41	28.3

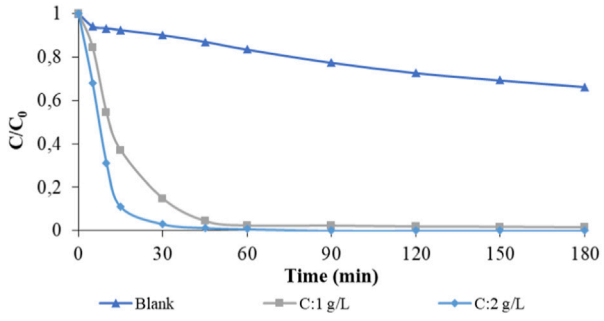


FIGURE 5
Effect of powder adsorbent concentration on C/C_o vs. time plot (Q_s: 6 mL/min, Q_p: 5 mL/min).

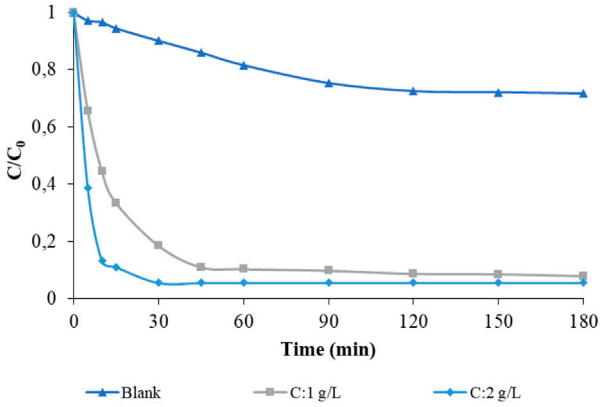


FIGURE 6
Effect of powder adsorbent concentration on C/Co vs. time plot (Qs = 6 mL/min, Qp = 10 mL/min).

3.2 Evaluation of the adsorption–membrane filtration hybrid method using Li-spiked RO concentrate of geothermal water

RO concentrate used in the hybrid experiments was obtained from a mini-pilot scale RO system operated at the Yenikale Geothermal Heating Center. Compared with the original geothermal water, the concentrate exhibited a higher Li^+ concentration, and it was further spiked with LiCl (lithium chloride) to achieve an initial Li^+ concentration of 10.5 ± 1.5 mg/L. The main characteristics of the geothermal water and RO concentrate are presented in Table 1.

Geothermal water and RO concentrates have significant quantities of competing ions (Na^+, Mg^{2+}, etc.), which may have adverse effects on lithium selectivity and adsorption effectiveness. In particular, Mg^{2+} is identified as one of the most competitive ions because of its comparable chemical behavior and hydrated properties with respect to Li^+. Still, the λ-MnO_2 adsorbent also showed selective lithium absorption under the studied circumstances, demonstrating the lithium-ion sieve behavior of the adsorbent in complicated geothermal matrices. Moreover, the hybrid system was still able to realize the effective separation of lithium despite the much larger concentrations of Na^+ than Li^+ (Yoshizuka et al., 2002).

3.2.1 Effect of adsorbent type

At the outset of the hybrid experiments, the performance of powdered and granulated adsorbents in extracting lithium from Li-spiked RO concentrate of geothermal water was evaluated. The operating parameters of the hybrid system were adjusted to a feed/permeate flow rate (Qp) of 5 mL/min and adsorbent replacement rate (Qs) of 6 mL/min, based on preliminary trials that identified these values as optimal for lithium separation from the geothermal water (Recepoğlu et al., 2018).

During the experiments, fresh adsorbents were introduced into the system as an adsorbent–deionized water suspension, and the corresponding percent of dilution was determined. The operating parameters for the hybrid system were set as follows: concentration of adsorbent (C): 2 g/L, flow rate of fresh and saturated adsorbents (Qs: 6 mL/min), and flow rate of feed and permeate solutions (Qp: 5 mL/min).

The "Blank" values presented in the figures represent the concentration decreases observed during blank experiments conducted under identical operating conditions without λ-MnO_2 adsorbent. These values were used to evaluate the contribution of dilution effects within the hybrid adsorption–membrane filtration system.

The adsorption performance of the tested adsorbents is presented in Table 3. The normalized concentrations (C/Co) versus time illustrated in Figure 4. Although the final concentrations of lithium after 180 min were nearly identical for both adsorbent forms, the powdered λ-MnO_2 exhibited significantly faster lithium separation and higher initial removal efficiency

TABLE 5 Summary of comparison study in hybrid system (effect of flow rate of feed and permeate solutions as a function of flow rate of fresh and saturated adsorbents).

Adsorbent	C (g/L)	Qp (mL/min)	Qs (mL/min)	$[Li]_o$ (mg/L)	$[Li]_f$ (mg/L)	R (%)
Powder λ-MnO_2	2	5	6	10.85	0.00	100.0
		10		9.10	0.50	94.5
		5	3	11.23	0.50	95.6
		10		9.10	2.79	77.5
Blank	0	5	6	8.71	5.78	33.7
		10		8.94	6.41	28.3
		5	3	9.44	7.23	23.4
		10		9.57	7.99	16.5

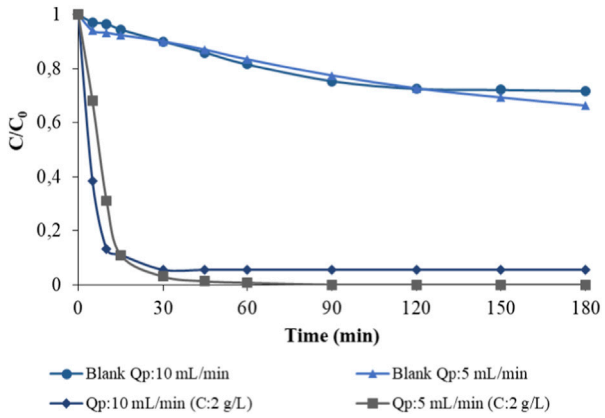


FIGURE 7
Effect of permeate flow rate on C/C_0 vs. time plots (Qs = 6 mL/min).

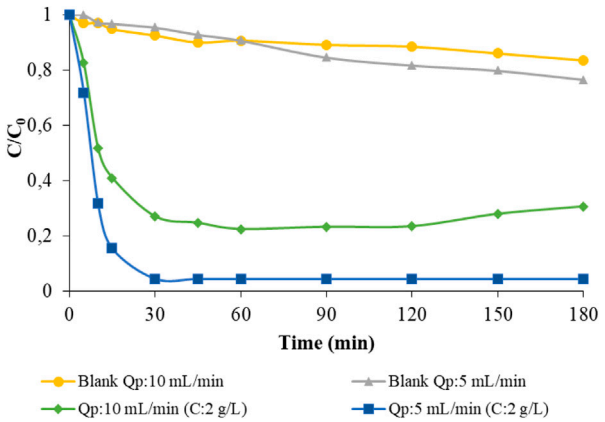


FIGURE 8
Effect of permeate flow rate on C/C_0 vs. time plot (Qs: 3 mL/min).

compared to the granulated form, which required a longer contact time to reach similar removal level. The dilution percentage, indicated as "blank" in the graphs, was calculated to be 33.7%. The higher surface area of powdered λ-MnO_2 resulted in enhanced lithium uptake and improved adsorption kinetics, supporting previously reported findings (Koltuniewicz et al., 2004).

Given that superior results were achieved using powder λ-MnO_2 adsorbent, subsequent investigations to assess the influence of other operational parameters were conducted exclusively using powder λ-MnO_2 adsorbent.

3.2.2 Effect of adsorbent concentration as a function of flow rates of feed and permeate

Following the comparative investigations of adsorbent types, the impact of powder adsorbent concentration on lithium separation from the Li-spiked RO concentrate of geothermal water was investigated.

For this objective, the hybrid system operating parameters were established as follows: adsorbent concentration (C): 1 and 2 g/L, feed and permeate flow rate (Qp): 5 and 10 mL/min, with a constant fresh and saturated adsorbents replacement rate (Qs) of 6 mL/min. The experimental data collected were compiled in Table 4 and depicted in Figures 5, 6. Initially, test data from each trial indicated that an

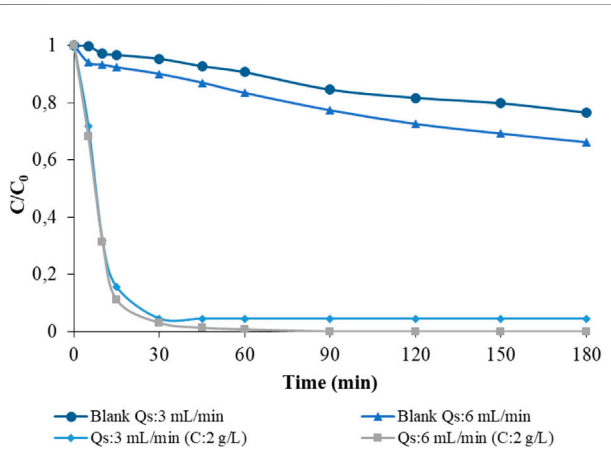


FIGURE 9
Effect of adsorbent replacement rate on C/C_0 vs. time plot (Q_p: 5 mL/min).

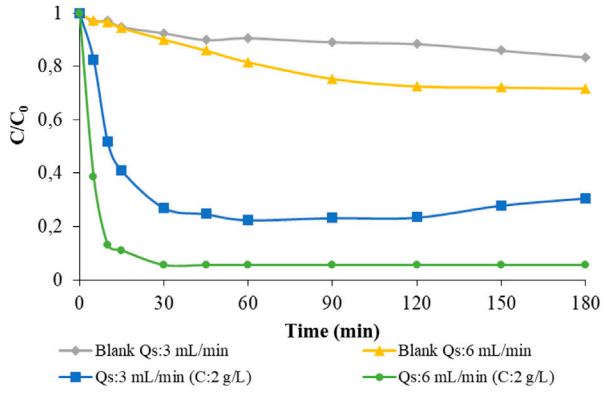


FIGURE 10
Effect of adsorbent replacement rate on C/C0 vs. time plot (Qp: 10 mL/min).

increase in adsorbent dose resulted in enhanced efficiency for lithium separation.

3.2.3 Effect of flow rate of feed and permeate solutions as a function of flow rate of fresh and saturated adsorbents

Following the evaluation of the effect of λ-MnO_2 powder concentration on lithium separation using a powder adsorbent in the hybrid system, the study was extended to investigate the influence of the flow rate of the feed and permeate solutions on lithium separation from Li-spiked RO concentrate of the geothermal water as a function of the flow rate of fresh and saturated adsorbents on lithium separation. For this purpose, the operational parameters of the hybrid system were established as follows: C: 2 g/L, Qp: 5 and 10 mL/min, and Qs: 3 and 6 mL/min.

Table 5 summarizes the experimental results and corresponding dilution rates, which are illustrated in Figure 7.

The effect of the permeate flow rate on lithium separation was analyzed at an adsorbent replacement rate (Q_s) of 3 mL/min, and the results are presented in Figure 8.

As seen in Figure 8, when Q_s was reduced to 3 mL/min, a marked decrease in lithium separation was observed at the higher permeate flow rate of 10 mL/min, highlighting the combined impact of reduced adsorbent renewal and limited contact time on the system's performance.

At an adsorbent replacement rate (Q_s) of 6 mL/min, increasing the permeate flow rate (Q_p) from 5 to 10 mL/min reduced the maximum lithium separation from 100% to 94.5%.

When Qs was reduced to 3 mL/min, raising Qp from 5 to 10 mL/min led to a sharper decline in lithium separation, dropping from 95.5% to 69.3%. This combined effect of lower adsorbent replacement and higher feed and permeate flow rate substantially shortened the contact time between the adsorbent and the solution, resulting in nearly a 30% decrease in lithium separation, emphasizing the importance of balancing flow rate and adsorbent replacement rate to minimize the kinetic limitations.

Ultimately, the effect of the adsorbent replacement rate on the hybrid system performance for lithium separation from Li-enriched RO concentrate of geothermal water was analyzed. Two distinct adsorbent replacement rates (Q_s) of 3 and 6 mL/min were tested under conditions of a permeate flow rate of 5 mL/min and a powder adsorbent concentration of 2 g/L. The experimental findings are presented in Figure 9.

TABLE 6 Summary of experimental data.

Test no.	C (g/L)	Q_p (mL/min)	Q_s (mL/min)	$[Li]_0$	$[Li]_f$	R (%)
1	0	5	6	8.71	5.78	33.7
2	0	10	6	8.94	6.41	28.3
3	0	5	3	9.44	7.23	23.4
4	0	10	3	9.57	7.99	16.5
5*	2	5	6	8.79	0.13	98.5
6	2	5	6	10.85	0	100.0
7	1	5	6	8.87	0.13	98.5
8	2	5	3	11.23	0.5	95.6
9	1	5	3	9.39	2.30	83.9
10	2	10	6	9.10	0.5	94.5
11	1	10	6	9.06	0.71	92.1
12	2	10	3	9.10	2.79	77.5
13	1	10	3	9.06	3.44	71.7

*Study with granulated type adsorbent. The others are represent the studies with powder adsorbent.
Tests 1, 2, 3 and 4 belong to the blanks and show the dilution percentage.

Enhanced lithium separation was achieved as the adsorbent replacement rate increased from 3 to 6 mL/min due to the higher number of available active sites by an increase in supply of fresh adsorbent and removal of saturated adsorbent.

The lithium content in permeate decreased to 0.33 mg/L after 30 min at an adsorbent replacement rate of 6 mL/min and continued to decline thereafter. At a rate of 3 mL/min, the minimum lithium concentration in permeate was measured at 0.5 mg/L after 30 min and remained stable until the end of the experiment. The dilution rates (control trials) for adsorbent replacement rates of 3 and 6 mL/min were determined to be 23.4% and 33.7%, respectively.

During the inspection, the effect of the adsorbent replacement rate was assessed as the permeate flow rate increased from 5 to 10 mL/min. The data are shown in Figure 10. When the flow rate (Q_p) was 10 mL/min and the concentration (C) was 2 g/L, raising the adsorbent replacement rate from 3 to 6 mL/min resulted in an improved lithium separation percentage.

All experimental results obtained in adsorption membrane filtration hybrid method studies were summarized in Table 6.

4 Conclusion

This study demonstrated the effectiveness of a hybrid ultrafiltration–adsorption system for lithium recovery from geothermal fluids and their RO concentrates. Among the tested materials, powdered λ-MnO_2 showed superior performance compared to its granulated form due to enhanced surface area and mass transfer characteristics.

The results confirmed that lithium separation is strongly influenced by operating conditions, particularly adsorbent concentration, flow rate, and adsorbent replacement rate. Higher adsorbent concentrations and lower flow rates improved lithium recovery, while increased flow rates reduced performance due to limited contact time. Continuous adsorbent renewal was found to partially compensate for these kinetic limitations.

Despite dilution effects observed in the system, λ-MnO_2 maintained high lithium selectivity across different geothermal sources. Under optimized conditions, high separation efficiencies were achieved.

The suggested hybrid adsorption-membrane filtering technique allows the utilization of fine powder adsorbents without the constraints of high pressure drop suffered by conventional fixed-bed adsorption systems. In addition, the membrane enables optimal retention of suspended λ-MnO_2 particles during operation, which reduces the adsorbent loss but ensures an efficient solid-liquid separation. The submerged UF arrangement runs under comparatively low pressure settings compared to pressure-driven membrane systems.

Overall, the findings highlight the importance of balancing hydrodynamics and adsorbent management in hybrid systems and demonstrate the potential of this approach for lithium recovery from geothermal resources (Dupont, 2022; Koseoglu et al., 2011; Recepoğlu et al., 2017).

Data availability statement

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

Author contributions

KB: Data curation, Formal Analysis, Investigation, Validation, Writing – original draft. BS: Formal Analysis, Investigation, Writing – original draft. EA: Conceptualization, Methodology, Supervision, Writing – review and editing. YJ: Conceptualization, Investigation, Methodology, Supervision, Writing – review and editing. NK: Conceptualization, Funding acquisition, Methodology, Project administration, Resources, Supervision, Writing – review and editing. MA: Formal Analysis, Funding acquisition, Project administration, Resources, Supervision, Writing – review and editing. SN: Conceptualization, Investigation, Methodology, Supervision, Writing – review and editing. KY: Conceptualization, Investigation, Methodology, Supervision, Writing – review and editing.

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