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Review Article

Lithium Recovery from Water Resources by Membrane and Adsorption Methods

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Abstract *- Due to the rapid development of portable electronic devices, hybrid and electric vehicles, there is an increasing demand for Lithium and its compounds in the form of the carbonate, lithium hydroxide, and mineral concentrates, which account for 80% of the world market. Lithium demand is projected to rise by 60% in the coming years, driven by electric vehicles. This requires efficient and fast methods of exploration and discovery of new deposits and cost-effective high-resolution exploration technologies. Hyperspectral imaging allows you to quickly map minerals and obtain data on the amount and spatial arrangement of ore and fossil minerals. The reserves of lithium resources in salt lake water, sea, and geothermal water are 70-80% of the total, which are excellent raw materials for lithium extraction. In this regard, more and more research is aimed at being involved in the industrial production of Lithium from water resources. An alternative method to increase lithium production is the recycling of lithium-ion batteries. Lithium concentrations in geothermal waters are deficient compared to brines, and not all methods are acceptable. The methods used to extract Lithium from liquid media include evaporation, solvent extraction, membrane usage, nanofiltration, and adsorption. The most effective and promising use of selective adsorbents with high functionality, low energy consumption, and environmental safety. The most studied are ionic sieves based on manganese LMO. More chemically stable lithium-ion sieves are based on titanium LTO.*

Keywords *- Lithium recovery, Adsorption, ion exchange, Geothermal water, Bittern.*

1. Introduction

Lithium ranks third in the table of the periodic system of chemical elements after hydrogen and helium, is the lightest alkali metal with an atomic weight of 6.939 [1], a density of 0.534 g / cm^3, has a high electrode potential of 3.05 in and has the highest specific heat capacity among all solid elements [2-4]. These properties of lithium compounds make them attractive in various industries. Lithium and its compounds are used not only in the production of glass, ceramics, refrigerants, and batteries [5] but also to increase the resistance of greases to extreme temperatures, in the production of aluminum and catalysts for the pharmaceutical and rubber industries, as well as in air conditioning systems and drainage [1].

Lithium is a rare element; its content in the earth's crust is 0.007% [5-7]. Lithium resources are divided into two main types: liquid and solid. Liquid waters include seawater, brines of salt lakes, and geothermal waters, while solid waters include deposits of mineral ores and secondary raw materials in the form of waste from lithium-ion batteries and the electronics industry [4, 8].

The world resources of Lithium are estimated at 14–15 million tons [8, 9]. The main deposits of minerals containing lithium compounds are located in Chile, China, Canada, Russia, Serbia, Congo, and Afghanistan [10, 11]. Lithium does not occur in nature in a free state and contains more than 150 minerals and clays [12].

Huge amounts of Lithium are concentrated in the sea, ocean water, salt lakes, and geothermal waters, which reach 70-80% of its total resources. [5, 10].

Sea and ocean waters are not yet of interest for the industrial production of Lithium due to the low content of 0.1–0.2 ppm, although its content is about 2600 billion tons [13–18].

Lithium in geothermal waters ranges from 1 to 100 ppm [9, 19]. The content of various impurities in geothermal waters with a high concentration of other metals complicates their processing and production of Lithium [20].

Saline lakes are more concentrated in Lithium and contain hundreds to thousands of parts per million. The difficulty of processing brines lies in the high concentration of impurities, especially magnesium [21]. In sum, all this complicates the problem of lithium isolation from natural waters, especially with high concentrations of alkaline and alkaline earth elements [22]. One of the main inorganic salt materials (lithium borate) has many excellent physical properties, such as diversified structure and a wide transparency range. Also, chemical properties such as good heat resistance and chemical stability [105].

Currently, the main products based on Lithium are lithium carbonate, mineral concentrates, and lithium hydroxide, which accounts for 80% of the market [23]. Lithium carbonate is obtained by extracting and processing spodumene rows and brines from salt lakes [24]. Lithium is used to produce batteries, and it takes more importance in developing electric mobility due to the necessity of having a system that uses sustainable energy. Also, it allows us to increase the displacement in relation to transportation expenses [104].

Spodumene is the first raw material used for industrial production. This ore is found in a rock called pegmatite. In these rocks, the lithium content is 1–4%, and the degree of extraction reaches 60–70% [24].

In addition to spodumene, lithium carbonate can be obtained from other ores in pegmatite rocks. The lithium content in such rocks reaches 6% and is called amblygonite, lepidolite, nepidalite, netallite, or zinivaldite [24].

With the depletion of lithium-containing ores, it becomes necessary to involve aqueous solutions containing Lithium in industrial processing. Lithium's water resources include brines from salt lakes, geothermal waters, and water from the seas and oceans.

Despite the low concentration of Lithium in aqueous solutions, this direction is more promising due to their wide availability and ease of processing [12].
The review is devoted to the extraction of Lithium from aqueous solutions by various methods and to the establishment of the further development of the industry for the production of lithium compounds characterized by environmental friendliness, high selectivity, cost-effectiveness, and simplicity of technical solutions.

2. Geology, Mineralogy

In recent years, the demand for Lithium has increased significantly due to its widespread use in batteries for portable electronic devices and hybrid electric vehicles. In connection with the gradual transition to economically clean energy fuels, the extraction of Lithium from ores is of great economic importance. Therefore, studying the behavior of Lithium and related minerals in some ore rocks is given special attention [25].

So, effective and fast methods are needed to explore and discover new deposits. Mineral resource mapping requires cost-effective exploration technologies. Satellite and aerial photographs provide large-scale regional quartering [26].

To do this, it is better to use hyperspectral imaging (HIS). This rapidly developing technology allows you to map minerals quickly, facilitating the study of the earth's surface at various scales [27]. The sensors used for this make it possible to collect data in a wide spectral range. This makes obtaining data on the quantity and spatial arrangement of ore and fossil minerals in the core, hand samples, and outcrops with an accuracy of millimeters to centimeters.

Precise housing is a complex problem in geology, especially in hard-to-reach regions. A multi-scale approach to hyperspectral imaging for the quartering of lithium-containing minerals and their spatial variability in pegmatites at three scales [27] created a spatially continuous three-dimensional map of quarry minerals. It showed the possibility of accurate processing in three-dimensional space and mapping lithium-containing minerals. The possibility of using hyperspectral images to explore and optimise production and other types of raw materials is indicated [107].

3. Reserves, Resources, Economy, Need for Lithium Compounds

The increased demand for Lithium and its compounds in the last decade is associated with its growing consumption in the electronics industry, lithium-ion batteries, and car batteries. Therefore it is called "new gold" or "white oil" [29].

Recently, the global consumption of Lithium has increased several times due to its use in various industries [30].

Lithium is used mainly in the production of batteries - 39%, ceramics and glass - 30%, greases - 8%, polymer products - 5%, air treatment - 3%, and others 10% [31].

The demand for Lithium in 2019 increased by 18% compared to 2017 and amounted to 58 thousand tons [33]. Lithium demand is projected to increase by 60% in the coming years [34]. It is due to increased demand for the production and use of electric vehicles. According to forecasts, by 2025, sales of electric vehicles will increase to 10 million, in 2030 to 28 million, and by 2040 to 56 million, which water will account for more than 50% of cars produced in 2040 [35].

According to forecasts, by 2027, the global demand for Lithium will be about 1.5 million tons in lithium carbonate equivalent [36]. At the same time, approximately 62% of Lithium is mined from aqueous brines and 38% from mineral rocks [24].

Even though Lithium is currently obtained from brine and spodumene ores, more and more research is directed toward the involvement of geothermal waters in the lithium production process. According to forecasts, the extraction and supply of Lithium from these sources can reach from 4% to 8% of the lithium supply in the United States [37].

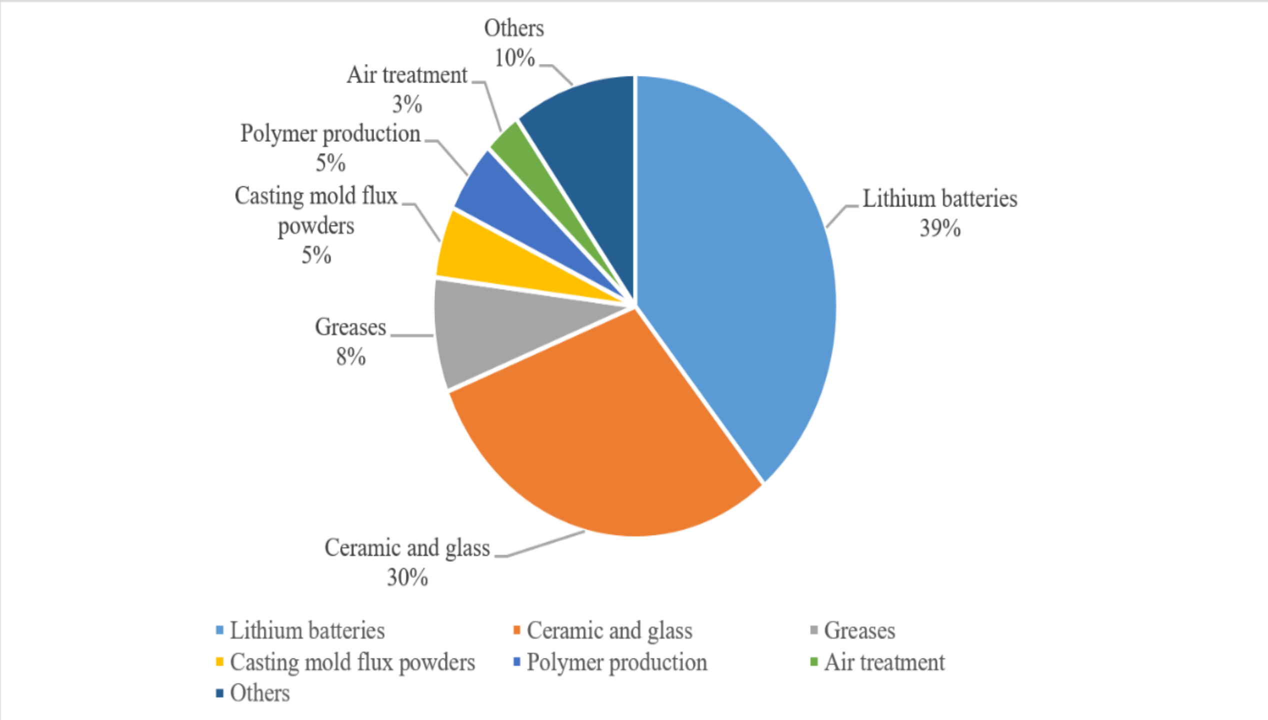

Fig. 1 Main Lithium uses according to Jaskula (2017) in relative percentages of Li metal equivalent used [32]

According to estimates, the cost of extracting Lithium from rocks is about two times higher than from brines [38]. It encourages the search for new and alternative ways to isolate Lithium from known and recycled feedstock sources to meet projected lithium demand.

An alternative method for obtaining Lithium is the recycling of lithium-ion batteries. However, this method of obtaining Lithium from rechargeable batteries is constrained by economic reasons, the difficulty of separating Lithium, and poor recycling, which is about 5% in the EU and the USA [37].

In order to at least partially solve the problem of lithium resources and production, it was proposed that a strategy be developed to conserve lithium resources, including intensive mining and a more efficient recycling system [7].

According to the US Geological Survey, the estimated reserves of Lithium are more than 14 million tons. The resources are about 34 million tons. The geography of the main lithium reserves includes Chile, Bolivia, Argentina, South and North America (USA, Canada), China, Australia, and European countries. About 60% of lithium reserves are concentrated in South America, 4.4 million tons in North America, 5.4 million tons in China, 0.85 million tons in Australia, and about 1.2 million tons in European countries [39]. There are also deposits of Lithium in Zimbabwe, Russia, and Afghanistan. Lithium deposits are located in European countries such as Germany, the Czech Republic, and Serbia.

Table 1. Li resources/reserves in Europe.

Country	Resource Tonnage (t)	Resource Li_2O (t)	Resource Li metal (t)	Reference
Czech Republic	695900000.00	2921631.77	1355.637	[40]
England	26666666700.00	16000000.02	7424.000	[41]
Austria	10980000.00	109800.00	50.947	[42]
Spain	112000000.00	683200.00	317.005	[43]
France	8500000.00	66300.00	30.763	[40]
Portugal	10300000.00	103000.00	47.792	[44]
Finland	4429000.00	50047.70	23.222	[45]
Germany	35510000.00	124959.69	57.981	[46]

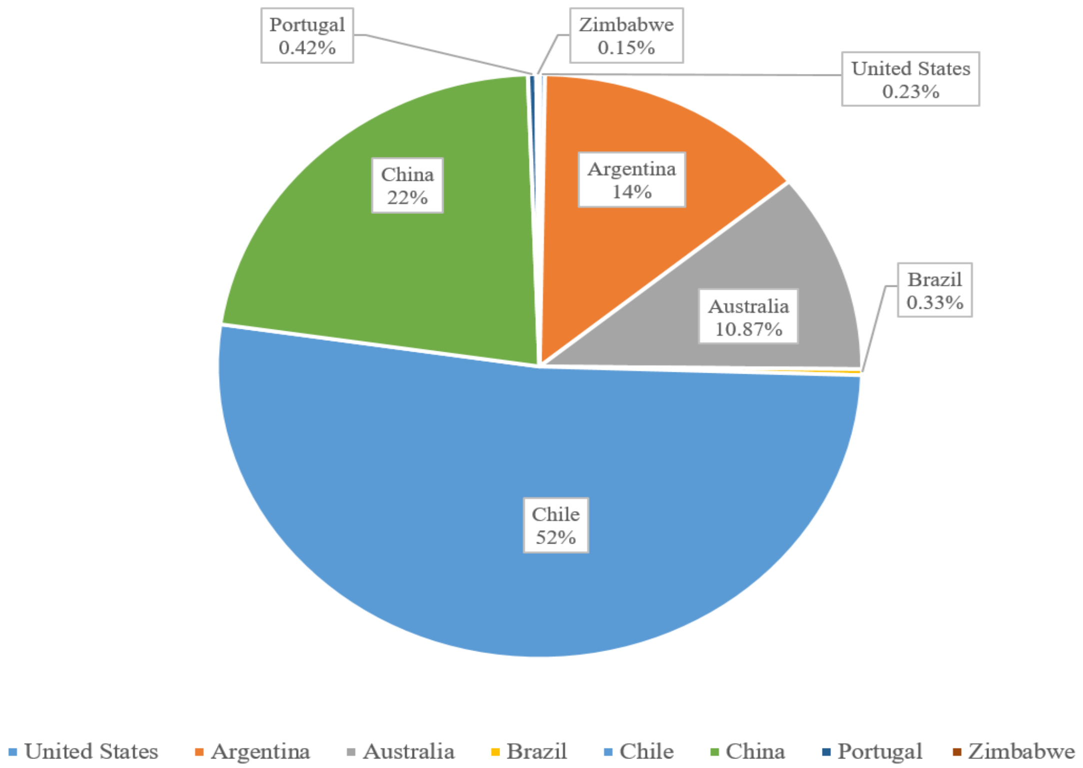

Fig. 2 World reserves of Lithium [32].

The global consumption of Lithium in 2019 was 58 thousand tons, 18% more than in 2017 [33]. However, global lithium production decreased by 19%, from 95 thousand tons in 2018 to 77 thousand tons in 2019, excluding US data. This is primarily due to a decrease in lithium prices due to a decrease in its demand, which can be explained by a decrease in sales of electric vehicles.

4. Isolation of Lithium

Currently, the main raw materials for lithium production are brining and solid minerals. The main reserves of Lithium are in Chile, Australia, Argentina, and China [32]. Lithium minerals make up only 25% of the available reserves, while brines are readily available and account for 65% [47]. Lithium production from continental brines is 30–50% cheaper than from minerals and is based on evaporation technology [4]. The process proceeds in artificial ponds due to solar energy and wind until the optimal concentration is obtained [48]. The availability of Lithium from brines and the need for their long-term evaporation for 1–2 years limit the production capacity of production facilities [1]. According to the European Commission, Lithium's demand will increase 18 times by 2030 and 60 times by 2050 [49]. Therefore, there is an acute problem with the selective extraction of Lithium from all existing primary and secondary sources of raw materials.

This section provides information on obtaining Lithium from its water-salt solutions.

5. Membrane Compartment of Lithium

The use of membranes for separating Lithium is under investigation. Studies of the processes of lithium separation under pressure, in particular, nanofiltration, have become widespread [50–52]. Nanofiltration makes it possible to separate the monovalent lithium cation from divalent calcium, especially magnesium cantons [53–55]. It has been established that nanofiltration rejects 26% of Lithium and more than 85% of magnesium from seawater [56].

The effect of monovalent potassium and sodium cations on the nanofiltration process has not yet been thoroughly studied [57]. The solutions obtained by this method are very dilute and require an additional concentration step before further processing [50].

Membrane distillation is also used to concentrate Lithium from brines. This method is used to extract salts from brines [56]. This method was practically not used for enrichment [50-51]. Currently, research on the extraction of Lithium while obtaining clean water is limited. Research is underway on a combination of nanofiltration and membrane distillation processes, which will allow both to separate lithium and multivalent cations [57]. Studies on the extraction of Lithium from brines of a salt lake using preliminary nanofiltration and membrane distillation to reduce the magnesium content showed the possibility of obtaining Lithium at an accessible concentration. Membrane filtration separated more than 90% of calcium and magnesium cations and from 42% to 60% of potassium, sodium, and lithium cations. The nano distillation method makes it possible to reduce the mass ratio of magnesium to Lithium to less than 6.

A study on lithium stripping from a loaded organic phase is done with an aqueous hydrochloric acid solution (0.5 mol L^{-1}). Aqueous feed phase pH of 12 was selected to favor lithium extraction while avoiding the degradation of the PVDF membrane, which could undergo dehydro-fluorination under alkaline conditions.

Also, the study mentioned that Lithium is extracted from ammoniacal solutions at the feed/liquid membrane interface. The extraction of this occurs through cation exchange with the proton of the β-diketone according to the following equation:

$$(Li^{+})_{feed}+x(HFDOD)_{SLM}+y(TOPO)_{SLM}\leftrightarrow(Li(FDOD)_{x}(TOPO)_{y})\ SLM^{+}\ (H^{+})_{feed}$$

Where the subscript "feed" and "SLM" refers to the aqueous feed phase and the supported liquid membrane phase, respectively. Heptafluoro-dimethyloctanedione (HFDOD) and tri-octyl phosphine oxide (TOPO), i.e. x=1 and y=1 or 2 depending on the TOPO concentration in the solvent phase.

Also, some researchers found that the PVC-based locally fabricated electrodialysis stack was used to assess the suitability of composite membranes for the recovery of lithium ions. This setup is mainly composed of 3 compartments (concentrated compartment (CC), diluted compartment (DC) and electrode wash (EW) compartment) separated by alternatively arranged cation and anion exchange membranes with an effective area of 66 cm^2 per membrane [103].

6. Extraction of Lithium with the use of Lithium-Ion Sieve Adsorbents

Lithium and its compounds are strategic resources of this century and are widely used in many industries due to their physicochemical characteristics [58-61]. Every year the demand for Lithium increases dramatically. With the depletion of natural solid deposits of minerals and high energy costs for the extraction of Lithium, much attention has been paid in recent years to the extraction of Lithium from brines of salt lakes, geothermal waters, and waters of the seas and oceans [47].

Due to the low concentration of Lithium in seawater and geothermal waters and the high concentration of associated ions in the brines of saline lakes, new solutions for extracting Lithium from such solutions are needed. One such method for obtaining concentrated lithium solutions is lithium-ion sieve adsorbents [106].

To extract Lithium from liquid media, depending on the lithium concentration in the solution, evaporation processes, solvent extraction, adsorption, and nanofiltration [4, 62–68], the use of membranes [69–71], and electrochemistry [72–74] are used.

Lithium-ion sieves can absorb lithium, separate it from complex aqueous systems, and are selective adsorbents with high ion screening functionality [75-77]. Lithium-ion sieves are divided by origin - based on manganese oxide (LMO) and titanium oxide (LTO) [78].

Among various technologies for the extraction of Lithium from solutions, adsorption of ion sieves is considered one of the promising methods due to its low energy consumption and environmental safety [79].

Due to their high adsorption capacity, ionic sieves based on manganese oxide LMO are Lithium's most studied selective adsorbents.

Several LMOs with a high adsorption capacity for Lithium have been synthesized using MnO_2, $MnO_2\cdot0.3H_2O$, and $MnO_2\cdot0.5H_2O$ [80–81]. The adsorption capacity of adsorbents depends significantly on the molar ratio of Li and Mn. As the Li: Mn molar ratio increases, the adsorption capacity improves. $MnO_2\cdot0.5H_2O$ has the highest adsorption capacity at the Li: Mn molar ratio [79].

Lithium-ion adsorbents of the LMO type are attributed to the spinel structure. In spinel $LiMn_2O_4$, the ratio of Li and Mn cations is 1:2, but it can be violated under certain conditions [82]. Spinel stability structure is affected by manganese dissolution during processing, reducing lithium sieves' adsorption capacity during recycling [79]. Since the adsorption capacity strongly depends on the material's morphology, porosity, and crystal structure, studies are underway to improve the performance using nanomaterials with an increased surface area and accessible extraction-desorption centers for the extraction of Lithium [75, 83].

It has been shown that the degradation of single-crystal LMO nanotubes can be overcome by using $(NH_4)_2S_2O_8$ as an eluent, reducing the dissolution of manganese, and maintaining capacity during adsorption-desorption processes [84]. The studies were carried out on pure solutions of LiCl, and the degree of lithium extraction was 89.73% of the theoretical capacity of the sorbent.

It was proposed to use environmentally friendly chitosan for lithium adsorption from its aqueous solutions [85]. It has been shown that the hydroxyl and amino groups of chitosan react with the epoxy group and alkyl chloride in epichlorohydrin under acidic and alkaline conditions. The material showed high thermal and chemical stability when coated with an ultrafine-grained hydrogen-type ion sieve at a mass ratio of chitosan:H4Mn5O12 = 1:1. Dissolution of manganese was not more than 1.15%. With the use of Lithium to extract from geothermal water with a temperature of 433 K and a lithium content of 25.78 mg/l, the adsorption capacity has reached a degree of extraction of 88.42% [85].

The high efficiency and selectivity for lithium ions from aqueous solutions of lithium-ion LMO sieves, their industrial use is limited due to difficult separation and a decrease in adsorption capacity due to the dissolution of manganese. To improve the properties of sieves with ionic lithium manganese oxide, magnetically recyclable iron-doped sieves with a spinel structure and made of Li $Mn_{2-x}Fe_xO_4$, synthesized by the solid-phase reaction method, were proposed by the authors [86]. The effect of temperature, calcination time, and the amount of alloying iron on the phase composition, dissolution losses, and adsorption characteristics, as well as the effect of the pH value of the solution, the initial lithium concentration, and the adsorption temperature, were studied. It is shown that the adsorption capacity of lithium-ion sieves can reach 34.8 mg.

The dissolution loss of Mn is reduced to 0.51%, which is much lower than that of undoped lithium-ion sieves, which reach 2.48%. It is explained by inhibiting the disproportionation reaction with an increase in the proportion of manganese in the skeleton. Comparing the results of the reuse of non-doped and iron-doped sieves on adsorption properties, it was found that the adsorption capacity of unalloyed sieves decreases by about 50% after the fourth cycle, which is higher than that of alloyed sieves, which are about 32%. This means that iron-doped lithium-ion sieves have good adsorption stability in long-term repeated use.

Due to the simplicity of the process and high selectivity, adsorption is recognized as an ideal method for extracting Lithium from solutions with low lithium content. Lithium-ion sieves based on lithium-titanium are more chemically stable than sieves based on manganese. [87]. However, the ultra-fine morphology results in low liquidity, low permeability, and low processing efficiency under commercial conditions, leading to serious post-separation complications. To eliminate these problems, an effective method is immobilising Ti-LiS powders with binding materials - chitosan, polyvinyl alcohol, polyacrylonitrile, polyvinyl chloride, etc. [88-93].

Some researchers found that adsorption is one of the most promising methods for extracting Lithium from geothermal waters [94]. The application is constrained due to the difficulty of synthesizing adsorbents with high adsorption characteristics and stability. Also, they found that the developed adsorbents are mainly powder form. Also, it is difficult to use in industrial conditions. Various forms of composite adsorbents have been developed to involve powdered adsorbents. [89, 93, 95-100]. It was not enough to improve the adsorption characteristics and stability. Polystyrene binder, polyacrylonitrile, polyvinyl chloride, and polysulfone are good coatings materials and have excellent chemical stability and mechanical strength [101]. Despite this, such composite materials' adsorption rate and capacity are much lower than that of powdered sieves. In this regard, filamentous materials based on polymer fibers are more promising, have a large specific surface area, and improve adsorption capacity [76, 102]. However, their stability was unsatisfactory without coating with polymeric binder materials and a high adsorption capacity.

Deng et al. [94] used Li_2TiO_3 to prepare a porous composite adsorbent with a fiber-based lithium-ion sieve for large-scale synthesis. They used 4 polymer binders, such as polystyrene, polyacrylonitrile, polyvinyl chloride, and polysulfone, to improve adsorption characteristics and stability. They used Li_2TiO_3 with tiny particle sizes and synthesized using a modified solid-state method to maintain structural stability. [94].

Prepared fiber composite adsorbent using a spinning device in combination with wet spinning technology and polysulfone (PSF) as auxiliary material. The fiber showed high adsorption performance and stability close to powder. The stability and adsorption capacity was increased by using ultrafine Ni_2TiO_3 synthesized by the modified solid-state method. The maximum equilibrium adsorption capacity for Lithium reaches 30.51 mg/g. During the cyclic tests, the average dissolution loss of Ti did not exceed 0.6%. The excellent properties of the developed PSF/HTO fiber point to a wide range of applications for the extraction of Lithium from geothermal waters and other aqueous solutions.

7. Conclusion

Lithium is one of the most demanded, rare elements with sufficient content in raw materials. It does not occur in nature in a free state and is found in more than 150 minerals. The demand for Lithium is growing every year and is explained by the increase in demand for electric vehicles.

Currently, more than 60% of Lithium is mined from water sources, which include brines, seawater, and geothermal waters and contain 70-80% of the total world lithium reserves.

Therefore, searching for ways to obtain Lithium from water sources suitable for producing lithium compounds is a serious and very urgent task.

Many methods have been developed to extract lithium compounds from water resources, including precipitation, solvent extraction, selective membrane separation, liquid extraction, ion exchange adsorption, electrodialysis, etc.

The most promising of them is the adsorption of ion sieves since it allows the extraction of Lithium from low-concentration lithium solutions and high concentrations of other elements. The method is energy-saving and environmentally friendly. Lithium-ion sieves are selective adsorbents with high ion screening functionality.

However, ion sieves have a relatively low ion exchange capacity, poor stability, and loss of sorbent.

To solve this problem, a lot of scientific work is being done to improve the stability of sorbents, increase the absorption capacity and selectivity, and accelerate the sorption time. Many methods are used for this, including organo-chemical, synergistic, binding, and composites. But none of them allows industrialization of the lithium adsorption method. Therefore, the search for ways to improve the lithium adsorption method remains a difficult task.

Lithium adsorption recovery may be an alternative to meet future demand, energy sustainability, environmental protection, and circular economy.

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