



# **Review** Separation of Critical Metals by Membrane Technology under a Circular Economy Framework: A Review of the State-of-the-Art

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Abstract: The demand for critical metals for net-zero technologies, including electric vehicles and wind/solar energy, puts pressure on extraction and recycling processes. As the treatment of solutions is becoming more and more complex and associated with the decreasing concentration of critical metals and the concentration of contaminants increasing, the development of separation techniques is required. Among them, membrane separation has been evaluated for hydrometallurgical processes with similar results to traditional techniques. This work aimed at reviewing the literature on membrane applications to obtain critical metals—lithium (Li), cobalt (Co), and rare earth elements (scandium—Sc, yttrium—Y, lanthanum—La, and neodymium—Nd). The main novelty is that this literature review focuses on the application of membrane techniques in industrial processes, not only water and wastewater treatment. For this, we searched a scientific database for different keywords, and the bibliometric analysis demonstrated a strong linkage between membrane separation and critical metals. The application of membranes to obtain critical metals from primary and secondary sources, acid mine drainage (AMD), industrial wastes, and the recycling of electronic wastes (e-wastes) and brine was revised. Among these traditional technologies, no relation was found with reverse osmosis. The outstanding use of membranes included combinations of solvent extraction techniques, including supported liquid membranes and polymer inclusion membranes.

Keywords: membrane separation; electrodialysis; supported liquid; polymer inclusion; nanofiltration

# 1. Introduction

Due to the economic importance and potential risk of the supply chain both in the short and medium-term, several countries worldwide have classifications of the most critical and strategic raw materials, including metals. These metals are crucial to the production of key technologies in three strategic sectors—renewable energy, e-mobility, defense, and aerospace—including Li-ion batteries, fuel cells, clean energy generation (solar and wind), robotics and drones, and digital technologies [1]. In addition, these metals are essential for technologies to achieve a low-carbon society [2]. For instance, the European Union classifies rare earth elements (Sc, Y, and lanthanides) as very high risk in the supply chain [3].

Several metals are included in different lists, including rare earth elements, Co, and Li. This has occurred due to primary production being controlled by a few countries associated with low recycling rates. For example, the production of rare earth elements is controlled by China, which is responsible for 70% of global production [4]. In the case of Co, about 68% of global production is provided by the Democratic Republic of Congo [5], and Australia and Chile [6] provide 82% of Li's global supply

Due to the limitation of primary sources associated with the social and environmental impacts of extractive metallurgy [7,8], several stakeholders worldwide have led the search for new sources of critical metals. Studies and processes have been developed to obtain critical metals from mining waste, for instance [9,10]. For example, Sc may be obtained from bauxite residue, the primary residue generated in the Bayer process to obtain alumina [11]. Another example is the recycling of electronic equipment (e-waste). For example, recovering Li and Co from Li-ion batteries is critical for the promotion of a circular economy and



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**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). reusing these elements to produce new batteries. For instance, replacing the internal combustion engine with electric vehicles requires more of both elements than the current production [12]. In both cases (the recovery of metals from mining waste and recycling of e-waste), the dependence and impact of extractive processes from primary sources decline.

There are two main industrial processes to obtain metals from secondary sources: pyrometallurgy (thermal process) and hydrometallurgy (aqueous processing). The pyrometallurgical process has drawbacks regarding the emission of greenhouse gases and energy consumption. In addition, it is possible to obtain high-pure products [13]. For example, in the recycling of Li-ion batteries, the recovery percentage for Li is lower than 80% in highly acidic conditions (when possible) since the element is presented in the slag phase after thermal treatment [14]. Moreover, the hydrometallurgical process may achieve more Sustainable Development Goals [15–17].

The hydrometallurgical route involves leaching and separation/purification steps. First, after pretreatment as comminution and physical separations, the source of critical material is leached to transform the metals into an ionic form. In most cases, leaching occurs in acid conditions, as most metals are leached in a pH lower than 2 [18]. Furthermore, the solution contains the target metals, and contaminations may be purified to obtain high-pure products. The most common techniques are solvent extraction [19], ion exchange resins [20], and precipitation [21].

Although separation and purification techniques meet industrial processes, technological challenges are changing, such as obtaining metals from sources with ever lower levels, contaminants in very high concentrations, increasingly complex solutions to work with, and the use of greener technologies. In this case, membrane separation technology has received attention in hydrometallurgical routes for separation and purification with less reagent consumption. Recent developments have aimed at using membranes beyond water and wastewater treatment but also in the separation and concentration of metals in hydrometallurgical processes [22–25].

The present review of the literature aimed at the analysis of membrane technologies for the separation of critical metals. This review focused on rare earth elements such as scandium, yttrium, lanthanum and neodymium, cobalt, and lithium. A database search was carried out for the leading membrane technologies: microfiltration, nanofiltration, ultrafiltration, reverse osmosis, and electrodialysis. Through this search, the manuscripts were divided by source and discussed. In addition, a bibliometric analysis was carried out to observe leading relations among the keywords.

#### 2. Membrane Technology

The most traditional membrane technologies are microfiltration, ultrafiltration, nanofiltration, and reverse osmosis. These techniques are usually involved in water and wastewater treatment for water reuse, both urban, agricultural, and industrial wastewater; however, they have also been explored for industrial purposes [26]. Microfiltration is used to separate particles with an average particle size of between 0.1 and 10  $\mu$ m and pressure applied around the 2 bar, with membranes capable of high temperature, pressure, and corrosive resistance. Among the chemical composts that may be separated, this includes bacteria, yeast cells, pain pigments, clay, silts, and dust [27,28]. In the case of ultrafiltration, the technique removes particles in the size range of 0.001–0.02  $\mu$ m and the pressure 1–10 bar to separate macromolecular solutes and colloidal materials [29]. Nanofiltration and reverse osmosis are similar in application, where nanofiltration operates at a lower pressure (5–35 bar) than reverse osmosis (15–150 bar). Nanofiltration is used for Na, Mg, and Ca rejection. Reverse osmosis is usually applied to separate all chemical composts, including ions, and obtain high-pure water [29].

In addition to such techniques, electrodialysis has been explored. This technique uses membrane ion-selective, which separates ions according to their charge. These membranes have fixed charged groups bound into the polymer matrix to which mobile ions have opposite charge–cationic and anionic membranes. The separation process occurs through an electrical current [29].

In addition to such techniques, other membrane configurations have attracted attention for industrial applications to improve acid/alkali resistance and selective separation and antifouling. The metal-organic framework is one of these techniques that have been explored, which comprises metal ions or metal ion clusters linked with organic ligands via coordination. These membrane types have milder synthesis methods, a higher porosity, and surface area. The metal-organic framework has been classified as a potentially new generation of membranes for the selective separation of metals [25].

Supported liquid membranes and polymer inclusion membranes have similarities in their application and selectivity. Supported liquid membranes use simple membranes (PVDF or PTFE) impregnated with an organic extractant (as a carrier): the same used in the traditional solvent extraction process. Therefore, it sums up the advantages of the membrane technique with the selectivity of a solvent extraction separation [25,30]. On the other hand, polymer inclusion membranes are liquid membranes with a base polymer (such as PVC or cellulose triacetate), a carrier immobilized into the polymer, and a plasticizer achieving high stability, selectivity, efficiency, and durability [31]. Most recently, there have been studies using ionic liquids as a carrier in supported ionic liquid membranes since these compounds are more viscous than traditional organic extractants, which is a great advantage in reducing carrier losses [32,33].

## 3. Review Methodology

In the present study, a systematic review was carried out to evaluate the literature on the use of membrane technology when separating critical metals. This study focused on rare earth elements (specific in Sc, Y, La, and Nd) due to market criticality [9,34] and Li and Co due to the demand for electric vehicles to meet SDGs [12]. The review strategy was focused on manuscripts published in scientific journals using the Scopus database. Therefore, only manuscripts published in journals were considered, excluding books, book chapters, patents, and conference papers. The literature was revised from 2018 to 2022 [9,35,36].

For this, different keyword combinations were used to find the manuscripts related to the topic of this review of the literature. Figure 1 shows the keyword combinations, which were searched for each topic to obtain publications for each membrane technique. For instance, "rare earth elements" and "microfiltration", "rare earth elements" and "nanofiltration", and "rare earth elements" and "ultrafiltration" combinations were used. The same was applied to all critical metals. The choice to use rare earth elements and Sc, Y, La, and Nd was related to the search for specific publications on the use of membrane technology in relation to them.

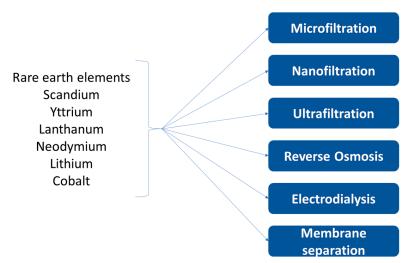


Figure 1. Keyword combinations used in the review of the literature in the Scopus database.

As depicted in Figure 2, after the search, it was found that 1458 articles adopted the following criteria: language (English), type of article (research or review), and date of publication (2018–2022). Further, the manuscripts were organized (in an Excel spreadsheet) and filtered according to the title/abstract, agreed topic, and repeated publications (360 articles). The manuscripts were separated into three main classifications: separation, purification, and industrial applications. Then, the manuscripts were evaluated.

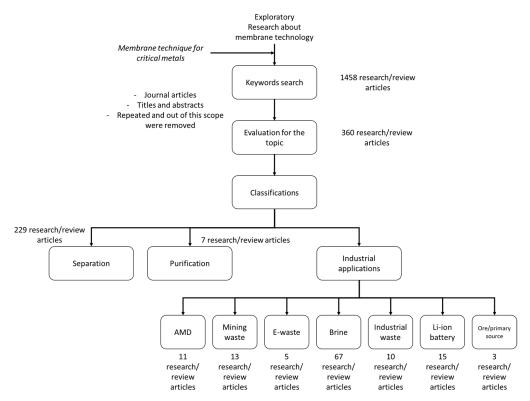
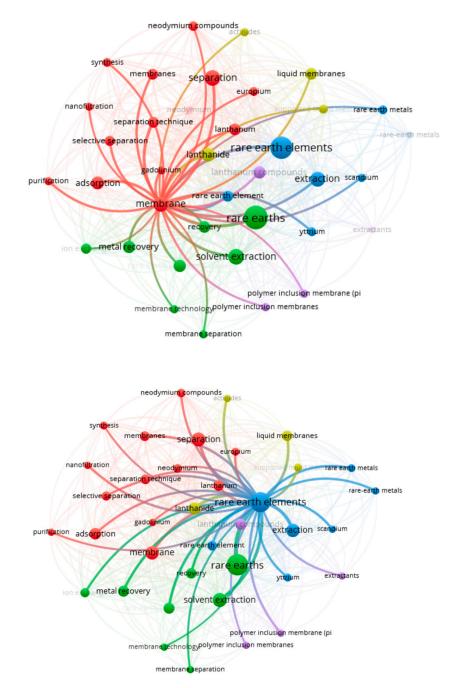


Figure 2. Flowchart of the literature review.

#### 4. Results and Discussion

4.1. Bibliometric Analysis between Keywords and "Membrane Separation"

Figure 3 presents the bibliometric analysis between "rare earth elements" and "membrane separation". Five clusters were found, differentiated by colors, where a strong connection was found in the separation/recovery process of rare earth elements by membranes. The main cluster (fourteen items in red) showed the highest occurrence of research interest in the adsorption process and the synthesis of membranes using rare earth elements. These studies were not considered for further chapters since they are out of the scope of this literature review. Cluster 2 (eight items in green) relates the focus on ion exchange separation and solvent extraction process to recover these elements. In the case of Cluster 3 (seven items in blue), the interest in the extraction process to obtain rare earth elements, scandium, and yttrium was highlighted due to their economic importance and high applications, respectively. Cluster 4 (four items in yellow) and Cluster 5 (four items in purple) are related to membrane technologies for the selective separation of metals, including liquid membranes and polymer inclusion membranes. Figure 3 highlights the connections between the membrane (in red) and rare earth elements (in blue) according to the literature review.

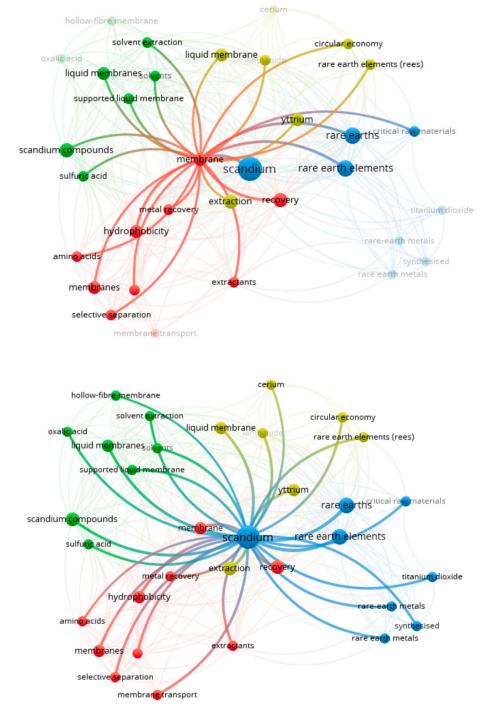


**Figure 3.** Keyword relation between rare earth elements and membrane separation elaborated using VOSviewer Software 1.6.19.

Figure 4 depicts the relationship between scandium and membrane separation. A strong connection was observed in the separation techniques involving membranes (hollow-fiber membrane and solvent extraction), metal recovery, rare earth elements, and a circular economy. It might have occurred since scandium is associated with other rare earth elements, the main yttrium, and obtaining it is associated with a circular economy, as mainly scandium is obtained from secondary sources. In addition, the search for new sources of scandium to supply the demand (which is scarce) makes this element one of the main and most critical ones [9].

The literature map shows co-occurrences between the keywords grouped in four clusters. The first one (ten items in red) relates aspects of the membrane in the separation process (hydrophobicity and extractants, for instance). Cluster 2 (eight items in green) highlights membrane technologies for scandium separation, including a hollow-fiber mem-

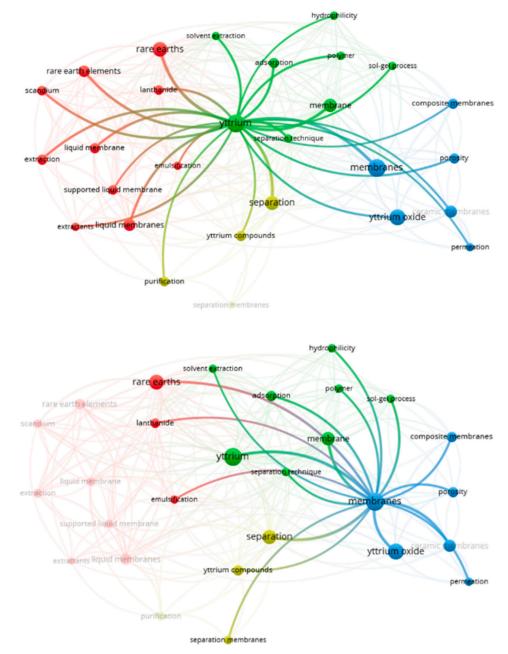
brane, liquid membrane, and supported liquid membrane. Cluster 3 (eight items in blue) is related to the use of critical metals, which are rare earth elements, for membrane synthesis. Finally, Cluster 4 (seven items in yellow) is strictly related to the promotion of a circular economy using membranes, as corroborated by the keyword "liquid membrane" linked to all clusters.



**Figure 4.** Keyword relations between scandium and membrane separation elaborated using VOSviewer Software 1.6.19.

Figure 5 presents the keywords relation between yttrium and membrane separation. It could be observed that there was a strong connection between yttrium and membrane technologies. The literature review observed that the element was used for materials synthesis in water treatment. For instance, Jiang the al. (2021) used the  $Y_3Al_5O_{12}-Al_2O_3$ 

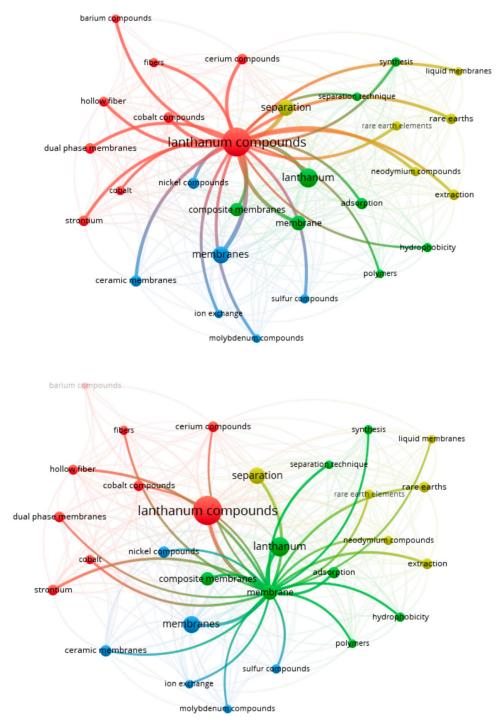
membrane to obtain ultra-high pure water by ultrafiltration [37]. Despite that, it was found that papers linked to the goal of this literature review. Cluster 1 (ten items in red) is related to the extraction of rare earth elements using liquid membranes, where yttrium is involved. Within Cluster 2 (eight items in green), the adsorption of the synthesized membrane (by the sol-gel process) is highlighted. Cluster 3 (six items in blue) relates the ceramic membranes prepared by yttrium oxide, and Cluster 4 (four items in yellow) links the separation and purification techniques by membrane using yttrium compounds.



**Figure 5.** Keyword relations between yttrium and membrane separation elaborated using VOSviewer Software 1.6.19.

For lanthanum, it was observed that, as occurred for yttrium, this element may be used to improve the efficiency of the membrane technique. Koh et al. (2021) manufactured a nanofiltration membrane using lanthanum incorporated into polyethersulfone/sulfonated polyphenylenesulfone for phosphorous treatment. According to the authors, this membrane achieved a performance 20 times higher than commercial membranes [38]. Cluster 1 (nine items in red) depicts a high connection to barium, cerium, and cobalt compounds

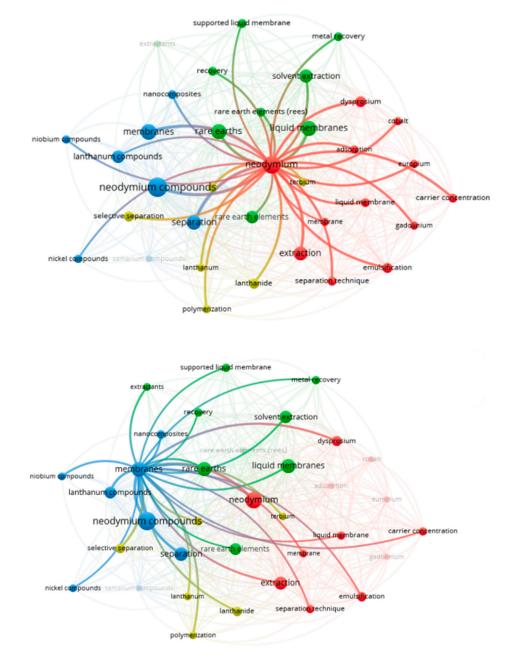
related to the synthesis of membranes. The adsorption and separation techniques are reported in Cluster 2 (eight items in green), while Cluster 3 (six items in blue) links ceramic membranes using nickel and sulfur compounds. Cluster 4 (six items in yellow) connects the extraction processes used to obtain rare earth elements through liquid membranes. Figure 6 shows a connection between lanthanum in the extraction process using membranes and the synthesis of materials for separation.



**Figure 6.** Keyword relations between lanthanum and membrane separation elaborated using VOSviewer Software 1.6.19.

Figure 7 depicts the connection between neodymium and membrane separation. As reported by other rare earth elements, neodymium could improve the separation efficiency when used in membrane synthesis. For example, Wu et al. (2021) reported

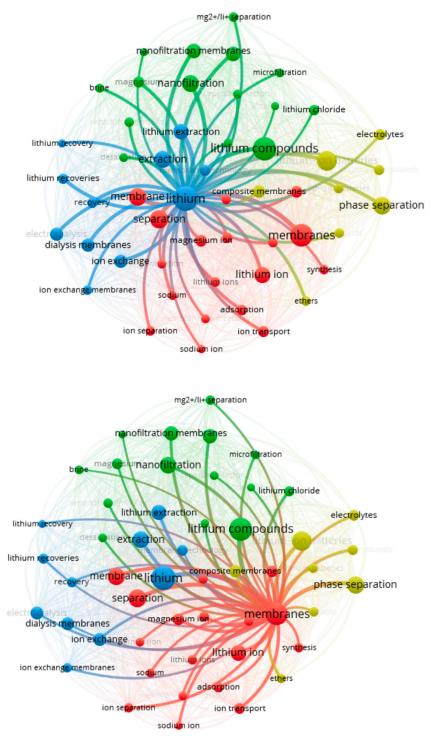
using a neodymium-imprinted nanocomposite membrane in a natural wood [39]. Cluster 1 (twelve items in red) relates the separation of metals (such as cobalt and dysprosium) using liquid membrane and adsorption, and Cluster 2 (nine items in green) depicts the separation of rare earth elements using membranes associated with the solvent extraction process (supported liquid membranes). Cluster 3 (eight items in blue) and Cluster 4 (six items in yellow) link the synthesis of membranes using metals for separation.



**Figure 7.** Keyword relations between neodymium and membrane separation elaborated using VOSviewer Software 1.6.19.

In this review of the literature, the search using lithium and membrane separation keywords found the highest number of manuscripts. For this reason, Figure 8 presents several connections. Indeed, there are several studies involving membrane separation to obtain lithium due to several reasons, one of them being the demand to supply the Li-ion battery market. First, about 59% of lithium resources are salt-lake brines; the primary contaminant is magnesium (Mg<sup>+2</sup>). Due to the difference in the electric charge of the electrons, where lithium is presented in solution as Li<sup>+</sup>, monovalent ion selective

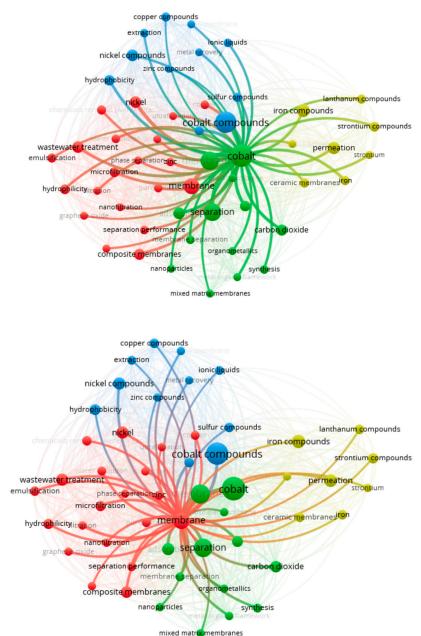
membranes have been largely studied to obtain a lithium-concentrated solution. It is advantageous to separate lithium from a multi-elementary solution under low-pressure or low-voltage environments [40].



**Figure 8.** Keyword relations between lithium and membrane separation elaborated using VOSviewer Software 1.6.19.

Cluster 1 (eighteen items in red) is related to membrane separation to obtain lithium and the effect of other ions in separation, which include magnesium and sodium as the main ones. Cluster 2 (fifteen items in blue) and Cluster 3 (eleven items in blue) depict the use of membrane technology (nanofiltration and ultrafiltration) to obtain lithium from brine and Li/Mg separation. Finally, Cluster 4 (eight items in yellow) reports using different membranes (ceramic and ionic liquids) in Li-ion batteries as separators.

As occurred in the literature review for lithium, many keywords were found for cobalt related to Li-ion batteries, as both elements are crucial to the largest types of batteries (around 95%) [12]. Furthermore, the presence of nickel is linked to cobalt sources, where the element is obtained in nickel and copper sources [41,42]. Cluster 1 (20 items in red) depicted in Figure 9 relates to the use of membrane technologies for water treatment, as cobalt may be used for membrane synthesis [43]. As occurred in Cluster 1, the use of cobalt in membranes for separation is depicted in Cluster 2 (13 items in green), but in addition, showed the use of gas separation, where the main focus is on  $CO_2$  separation [44]. Cluster 3 (11 items in blue) relates the extraction of cobalt and the presence of copper and nickel by novel membrane technology (ionic liquid and polymer inclusion membrane), and Cluster 4 (eight items in yellow) has less interaction. It is related to ceramic membranes synthesized using cobalt, strontium, and lanthanum.



**Figure 9.** Keyword relations between cobalt and membrane separation elaborated using VOSviewer Software 1.6.19.

#### 4.2. Application of Membranes for Ores/Primary Sources

The main sources of Li are brine and spodumene. The extraction of Li from brines and the use of membrane technologies are discussed in Section 4.6. Spodumene ores mainly contain about 8% of Li<sub>2</sub>O as a lithium-aluminum silicate (Li<sub>2</sub>O.Al<sub>2</sub>O<sub>3.4</sub>SiO<sub>2</sub> or LiAlSi<sub>2</sub>O<sub>6</sub>), which is closely associated with quartz, feldspar, and micas. The hydrometallurgical route is commonly used to obtain Li after thermal treatment [45]. The literature review follows the methodology depicted in Section 4 reports the possible Li resource from lepidolite ore using membrane technology. It is considered a hard-rock ore with the chemical formula KLi<sub>1.5</sub>Al<sub>1.5</sub>AlSi<sub>3</sub>O<sub>10</sub>F<sub>2</sub> but lower Li concentration than spodumene. Despite that, the urgency to find sources of Li to supply the battery market may result in lepidolite or a potential Li source [46,47].

As reported by the studies found in the literature review [46,48,49], the main goal is the selective separation of Li and Al after lepidolite leaching. Gao et al. (2020) leached the lepidolite material with sulfuric acid—60 wt% H<sub>2</sub>SO<sub>4</sub>, solid–liquid ratio 1/2.5 at 160 °C for 4 h—with 97% of Li extraction. The authors tested nanofiltration to separate Al/Ca and Li using a DK membrane (150–300 Dalton). In the separation process, the authors used Ca(OH)<sub>2</sub> to neutralize the excess acid (until pH 2.2). According to the authors, the nanofiltration process exhibited high retention for SO<sub>4</sub><sup>2–</sup> ions. In the case of Li separation, the retention rate followed the order: Al > Ca > K > Na > Li. Al and Ca retentions were over 96% and 94%, respectively, while Li was 52% at pH 2.2 [46].

In this process, to obtain Li form lepidolite, in another study, Guo et al. (2020) evaluated different commercial membranes for nanofiltration (commercial membranes: DK, DL, NF270, and Duracid NF). According to the authors, the Li/Al separation rate was 471.3 using the DK membrane due to the pore size, smooth membrane surface, and appropriate zeta potential. In addition, the Ca retention rate was also high due to a similar pore size between the ions and the membrane. The order of the separation factor of Al and Li was as follows: DK > Duracid NF > DL > NF270 [48]. The Al/Li separation in different conditions represented an excellent opportunity to use nanofiltration to obtain Li from the processing of lepidolite or other sources. In addition, it may represent an essential source of Li shortly [49].

#### 4.3. Application of Membranes for AMD and Industrial Wastes

Acid mine drainage (AMD) is a global problem causing negative impacts on the environment and living beings. It is formed by the oxidation of pyrite (or materials-bearing pyrite) or other sulfate compounds as waste piles, coal mine piles, tailing pits and dams, and mines underground upon exposure to air, microbial activities (as a catalyst), and water. As a result,  $H_2SO_4$ , Fe(II), and Fe(III) are released, and the acid solution reacts with the material as it is percolated, leaching other metals. Therefore, if not properly controlled, it may be released into the environment, causing several negative impacts on living beings, such as mortality, growth disorders, lower reproduction rates, deformities, and injuries. In addition, adverse economic impacts are also observed due to the AMD treatment [50,51].

The issues caused by AMD raise the search for remediation approaches for treatment and management [50]. Conventional techniques are used for AMD treatment, which includes precipitation using gypsum, lime, and limestone, adsorption, or alternative methods such as biological treatment (as wetlands), the use of waste materials and by-products from other industries, and membrane technology [52].

Among membrane technologies, nanofiltration is the main one used for AMD treatment—about 80% of the publications are related to nanofiltration. In addition, the literature reports the potential use of reverse osmosis as well, owing to the good quality of water produced for reuse [23,50,52,53].

López et al. evaluated nanofiltration as a treatment technology for AMD containing rare earth elements. According to their work, an acid solution (H<sub>2</sub>SO<sub>4</sub>) may be recovered from a pH lower than 3.0 using semi-aromatic polyamide, poly-pyperazinamide/proprietary polyamide, and sulfonated polyethersulphone membranes. The solution containing about

2200 mg/L of Al, 9500 mg/L of SO<sub>4</sub>, and 10 mg/L of rare earth elements (La, Pr, Nd, Sm, Dy, and Yb) can be concentrated almost 20 times, where after nanofiltration may be sent to the precipitation step to obtain the phosphate of rare earth elements [54,55].

As observed, the high acid concentration in AMD damages the membrane [56]. For this reason, an acid-resistant ceramic and polymeric nanofiltration membrane was prepared and evaluated for acid recovery and to concentrate rare earth elements. The membrane, when synthesized, allows the transport of  $H^+$ . The metal rejections achieved around 80% with high resistance again acid action keeping their selectivity [57]. Another problem to be solved with AMD treatment using membrane technology is fouling and scaling events due to the presence of microorganisms and high concentrations of Al and Ca [58].

In addition, water may be recovered from AMD using nanofiltration [59,60]. Fonseka et al. (2022) evaluated a polyamide membrane (NF90) with positively charged functional groups on its surface, where the rejection rates of Na were 90% and for other elements were over 98%. The authors also observed biofouling due to organic matter, which declined the flux [59]. Although most of these publications are related to acid or water recovery from AMD, rare earth elements may be concentrated for further steps, as described by López et al. (2019). The authors stated that rare earth elements might be recovered after the nanofiltration step, in which  $H_2SO_4$  can be recovered as phosphate salt [55].

Regarding the application of membrane technology from industrial wastes, a central focus was given to nuclear power and electroplating industries. Kim et al. (2020) compared reverse osmosis and nanofiltration for the separation of radioactive nuclides—Sr, Co, and Cs. Nanofiltration showed a 64% rejection, while reverse osmosis achieved 80% rejection using commercial membranes (NF90, NF270, TW-2540, and XLE-2540). According to the authors, the concentration of nuclides increased while water was recovered [61]. Chen et al. (2020) synthesized PEI membranes for reverse osmosis. Although the focus was on water recovery, this process concentrated Co ions almost 3500 times [62], which may be used for other processes to obtain Co. Lazarev et al. (2021) evaluated electro-nanofiltration for the treatment of electroplating wastewater containing Cu, Zn, Co, and Ni. In this case, a current density was applied and associated with nanofiltration separation, which may be an alternative for ions migration on a membrane–solution interface [63].

Electrodialysis has been applied for wastewater treatment due to the possibility of separating cationic and anionic ions in different streams. For instance, in a nuclear plant, cationic membranes can be used to separate cations (Co, Li, and Cs) and obtain water for reuse, where about 98% of cationic ions are separated [64]. Moreover, the electrodialysis process can use the organic phase with a recovery efficiency of over 95% [65].

Despite the traditional technologies, studies have focused on membrane preparation for the selective separation of metals and cationic ions from complex wastewater. For example, Kedari et al. (2020) studied wastewater containing Am, La, Ce, Nd, Sm, Sr, Pr, and Y in a lactic acid solution from nuclear waste to separate Am. The authors used a hollow fiber liquid membrane with diethylene triamine pentacaetic acid (DTPA), and the process selectively separated Am with lactic acid from the other metals, where the emulsion phase contained di(2-ethylhexyl) phosphoric acid (HDEHP), achieving a decontamination factor of 412 [66].

The search for new sources of critical metals promotes the technological development of separation processes from wastes [9,67,68]. In this review of the literature, an interest was observed in the recovery of rare earth elements from coal fly ashes, which are generated during coal combustion. This residue may contain about 30% of rare earth elements, higher than ores. It results in the development of the separation of these elements after leaching [69]. Mutlu et al. (2018) evaluated nanofiltration to concentrate rare earth elements [70]. The application of electrodialysis was studied by Couto et al. (2020), where the cationic membrane was used to concentrate the catholyte solution, achieving a recovery efficiency of 70% [71]. The combination of membrane technology and the solvent extraction technique was explored by Smith et al. (2019) using a liquid membrane and supported liquid membrane with D2EHPA. The separation of rare earth elements was higher using a supported liquid membrane, achieving 70%, and was more selective than common elements such as Na, Al (less than 5%), Ca, and Fe (less than 10%). The extraction of Y achieved 100% using a supported liquid membrane [72].

The search for new sources of Sc is important due to its critical and high market price [9]. The interest in extracting Sc from red mud could be related to the amount of residue stored worldwide—about 4 billion tons [73–76]. However, it is not the only potential Sc source to be explored. Hedwig et al. (2022) evaluated polymer inclusion membranes combined with an organic extractant to separate scandium from titanium dioxide pigment production waste. The authors combined the PVDF membrane with D2EHPA, where a high separation rate was observed even in a solution containing 30 other elements [77]. Similarly, in another study, Hedwig et al. (2022) combined nanofiltration with solvent extraction, which removed Sc from Fe, where D2EHPA and Cyanex 923 were selective for Sc [78].

In addition to using membrane technology to obtain critical metals from mining, Feijoo et al. (2021) demonstrated that electrodialysis can be used to concentrate Co from nickel laterite waste processing. The use of cationic and anionic membranes concentrated the Co from a stream, and  $H_2SO_4$  was recovered from being recycled in the process. Extraction efficiencies achieved about 98% within 136 h [79].

## 4.4. Application of Membranes for the Recycling of e-Waste and Li-ion Battery

The search for new sources of critical metals (primary or secondary) leads to studies and process design from urban mining [80]. It occurs due to the amount of waste generated in the cities and growth rates. For instance, e-waste generation worldwide increased by 38% from 2010 to 2019, representing about 53.6 Mt of residue. In addition, the e-waste growth rate is three times higher than any other waste (3–5% per year) [81]. These residues contain critical metals such as Li, Co, and rare earth elements in concentrations 20–100 times higher than primary sources [16,82–84]. Moreover, it may be a source of critical metals in countries with no primary source. For instance, there is no primary source of Co in Europe [85], and spent Li-ion batteries may represent a source for the continent [12].

Korkmaz et al. (2018) evaluated the nanofiltration process when recycling Ni-metal hydrate batteries. The authors leached anode and cathode materials with HCl, and the leach solutions were treated by nanofiltration to recover the HCl solution to be reused in the leaching step. The process was carried out at 20 bar and 19  $L/m^2 \cdot h$ , where the permeate stream contained a concentration of rare earth elements lower than 8 mg/L and 47 mg/L of Ni [86]. Therefore, this process may be important to concentrate the leach solutions for further treatments.

In addition, several studies in the literature report the use of membrane technology to separate the critical metals selectively after leaching. For example, Yuksekdag et al. (2022) studied supported liquid membranes to separate rare earth elements. The authors tested polyvinylidene fluoride (PVDF) supported with organic extractants, from which bis-2-Ethylhexyl phosphoric acid (D2EHPA) and di-2,4,4,-trimethylpentyl phosphinic acid (Cyanex 272) were evaluated. As a result, Sc was separated selectively using a membrane supported by Cyanex 272, while D2EHPA separated Ce, Dy, Er, Eu, Gd, Ho, Nd, Pr, Sm, Tb, Y, and Sc. For this reason, the use of the PVDF membrane supported with Cyanex 272 can be used to separate Sc from rare earth elements (with small quantities of Dy, Er, Ho, Tb, and Y) and can be supported with D2EHPA to separate the remaining rare earth elements from common elements (as Fe, Cu, Al, Ca, Mn, Cr, Cd and Co) [87].

It occurs due to a strong connection between rare earth elements and organic extractants with P ions in functional groups [11]. Furthermore, Ni'am et al. (2020) tested hollow fiber membranes supported with D2EHPA and Cyanex 272 separate rare earth elements from magnet recycling. The traditional solvent extraction process did not separate the elements from Fe and was not extracted using membrane support. The extraction of Nd, Dy, and Pr were 63.13 %, 15.21 %, and 56.29 %, respectively, without iron co-extraction [88]. Islam et al. (2022) evaluated a membrane supported with a neutral extractant to separate rare elements—tetraoctyl diglycolamide (TODGA)—from scrap magnets. Instead, the authors used a hollow fiber membrane. As a result, the concentration of rare earth elements increased over time in the permeated stream, where the purity of the final product was 99.5% in rare earth elements [89]. In addition, Ni'Am et al. (2020) evaluated hollow fiber membranes supported with D2EHPA in recycling scrap magnets. In contrast to the results obtained by Islam et al. (2022), which used a neutral extractant, the use of organophosphorus extractants, such as D2EHPA, achieved the best separation rate for Nd against Dy and Pr, where 90.82% of Nd was permeated through the membrane, while Dy and Pr were 11.89% and 1.50%, respectively [90]. As aforementioned, this occurred due to a strong connection between rare earth elements and phosphorus organic extractants.

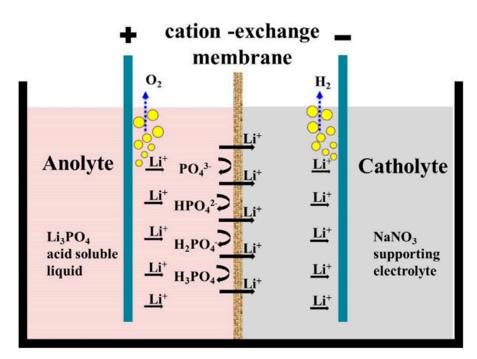
Yadav et al. (2019) also studied the separation of rare earth elements from scrap magnet recycling using a hollow fiber membrane, where the authors supported the membrane with 2-ethylhexyl 2-ethyhexylphosphonic acid (EHEHPA). In this case, the process was more selective for Dy than Nd and Pr, even owing to the concentration difference between the elements, in which Nd is the main component in permanent magnets. As a result, Dy was obtained with 97% purity [91].

Due to the growing demand for net zero technologies, the market of electric vehicles has grown worldwide, mainly in Europe, North America, and BRICS countries [12]. As a result, the demand for metals, mainly Li, to supply this market is over current production [92]. Along with searching for sources of Li from a primary source, recycling these batteries is important to promote the circularity of materials. Membrane technology comes as an alternative for selective separation to obtain pure or high-pure Li solution. According to Swain et al. (2018), reverse osmosis is a cost-effective alternative for Li concentration from battery recycling. It may also overcome technical limitations from traditional separation technologies [93].

Gao et al. (2019) studied the ultrafiltration technique to separate Li from a simulated leach solution. After Al and Fe removal by precipitation at pH 7, the solution containing Li, Ni, Co, Mn, and Cu was mixed with polyacrylate sodium (PAAS). As a result, PAAS reacted with all the metallic ions except Li, and in the ultrafiltration process, Li ions permeated the membrane, which rejected the complex compounds. Therefore, about 94.6% of complexed ions (Ni, Co, Mn, and Cu) were separated, while the Li separation rate was 96.1% [94].

Kumar et al. (2022) evaluated the nanofiltration process for Li concentration and separation from Ni, Co, and Mn. First, spent Li-ion batteries were leached by  $H_2SO_4$  and followed by precipitation to remove Fe and Al as impurities. Then, ultrafiltration was used to concentrate Li, where 92.5% of Ni, 94.6% of Co, and 95.8% of Mn, were rejected, while 89.6% of Li was permeated throughout the membrane with 7.5 L/(m<sup>2</sup>·h) and 10 bar [95]. According to Swain (2018), reverse osmosis may also be used for Li concentration from recycling Li-ion batteries [93].

Electrodialysis has been studied for the selective separation of metals from Li-battery recycling. The process can be carried out using a monovalent cation-exchange membrane to separate Li from other metallic ions or complexing agents (such as EDTA) to form negatively charged compounds and apply cationic membranes for selective separation [96]. Figure 10 presents a schematic diagram for separating Li ions from  $PO_4^{3-}$  and  $HPO_4^{2-}$  ions, which are present in the recycling of LFP batteries (LiFePO<sub>4</sub>). First, the electrodialysis device is fed with the solution, and an electrical potential difference is applied; as a result, the Li ions are permeated to the cationic membrane, and the phosphate ions are not. Song and Zhao (2018) described how the Li ions were separated from other elements by precipitation at pH 12 using a phosphate salt, where Li remained in the solution. Then, the membrane Nagion-117 (monovalent membrane) was used to separate Li from  $PO_4^{3-}$  and  $HPO_4^{2-}$  ions. After electrodialysis separation, the final product contained 99.3% purity (Li<sub>2</sub>CO<sub>3</sub>) [97].



**Figure 10.** Schematic diagram of an electrodialysis device used to separate Li from  $PO_4^{3-}$  and  $HPO_4^{2-}$  ions (image obtained after authorization by [97].)

For recycling LFP batteries, electrodialysis can be used as a separation technique. After leaching, He et al. (2020) obtained a high-pure LiOH using a monovalent cation-exchange membrane. In this case, the authors evaluated a slurry electrodialysis, as described by Li et al. (2020), where the cathode was mixed with water in an electrodialysis cell, and Li was released and separated by the membrane under an electrical potential difference. Cationic and monovalent membranes may be used for ion separation [98,99].

Cerrillo-Gonzalez et al. (2020) demonstrated that electrodialysis might separate Li and Co from recycling LCO batteries (LiCoO<sub>2</sub>). The authors tested the cation-exchange membrane after leaching with HCl, and the recovery rates were 62% of Li and 33% of Co. Furthermore, it was observed that cobalt tended to accumulate within membranes due to their being a divalent cation [100]. In addition, compared to the results reported by He et al. (2020), using a monovalent membrane is crucial to the separation of Li ions against divalent and trivalent ions selectively.

Gmar et al. (2022) demonstrated that it is possible to separate Li from the recycling of the NMC battery (LiNiMnCoO<sub>2</sub>) using a monovalent membrane. The authors observed that the current applied in electrodialysis must be lower than the limiting current to avoid the precipitation of metallic ions, such as hydroxide, into the membrane porosity. Additionally, in the process, as exemplified in Figure 10, H<sub>2</sub>SO<sub>4</sub> may be recovered and reused for leaching [101]. On the opposite side, Chan et al. (2022) used different cationic membranes to separate the ions from NMC battery recycling. However, in order to perform a selective separation, the authors mixed EDTA in the solution to complex the metallic ions. So, the first step was Ni complexing to separate from the Co-Mn-Li solution. Then, Co was complexed with EDTA; after that, Mn reacted, and Li was separated from the solution. Finally, different NMC batteries were evaluated (NMC111, NMC532, and NMC622), which changed the proportion of Ni, Mn, and Co, and the results were similar [102].

In addition to traditional membrane technologies, the search of the literature reported new potential applications. For example, Keller et al. (2022) treated a hydrophobic PTFE membrane with D2EHPA to separate Mn from Co. This process demonstrated a high selection for Mn, which occurred in only one reactor against the solvent extraction process that required several steps (extraction, stripping, and regeneration) [103]. Regarding the recovery of metals from Li-ion batteries, Li et al. (2019) synthesized a stable metal–organic

framework, ZIF-8, to form a membrane composite to separate Li from Co. The synthesized membrane exhibited a large surface area, a high channel density, and a short channel length, with a Li/Co separation factor of 8.29 [104].

## 4.5. Application of Membranes for Brines Processing

About 53% of the literature review focused on industrial applications of membrane technology to obtain critical metals from brine. In this case, it is an important source of Li due to its abundance, which is crucial for market supply. In this view, membrane technology may provide technical and economic feasibility for Li when obtaining it from brine selectively [105,106]. In addition, rare earth elements may be found but in extremely low concentrations (ppb). For this reason, metal–organic framework nanoparticles have been explored as an alternative. For example, Liu et al. (2021) synthesized nanoparticles with diethylenetriamine coated on the outside of the Fe<sub>3</sub>O<sub>4</sub> and further modified with polystyrene sulfonate, which can be used to capture rare earth elements [107].

Due to the number of manuscripts in the literature review, a few examples are further presented and discussed. For example, Baudino et al. (2022) recovered Li from brine using a graphene oxide membrane. Such material was used due to the functionalization in which sub-nanometric hydrophilic channels are rich in oxygen groups, making them appealing for separation with antifouling properties. As a result, the authors achieved up to 70% of Li recovery [108].

Nanofiltration has been used to separate Li from salt lake brine due to the selectivity of rejecting divalent/multivalent ions. As a result, Li ions (monovalent) permeate the membrane. Using a commercial membrane (DK membrane), Li et al. (2019) observed that the presence of Mg is the main contaminant in Li separation from brine, and the authors achieved 97% of Li recovery and a separation factor of 13 [109]. To improve the separation rate of Mg and Li ions, Li et al. (2022) synthesized a nanofiltration membrane with polyethyleneimine bonded with a 15-crown-5 ether through hydrogen interaction, where the Mg concentration declined from 1500 mg/L to 2.7 mg/L while the Li concentration remained similar [110]. For the same reason, Soyekwo et al. (2022) synthesized a membrane with an ionic liquid to improve the Mg/Li separation factor, which achieved 26.5 [111]. All these studies demonstrate the high efficiency of nanofiltration to separate Li from brine, with Mg ions being the main contaminant of the process, reaching a 50 times higher concentration than Li ions [109].

Electrodialysis was explored for Li obtained from brine. Zhou et al. (2018) used electrodialysis to concentrate a Li sulfate solution in the brining process after acid leaching. In this case, the authors used cationic and anionic membranes to increase the Li concentration from 6 wt% to 17 wt%. According to the authors, the cost of the process was considered competitive with conventional methods [112]. On the other hand, Ying et al. (2020) evaluated the Li extraction directly from brine with Li and Mg concentrations equal to 7.99 g/L and 78.67 g/L (Mg/Li equal to 9.85), respectively, using cationic and anionic membranes. The process achieved a 90% Li recovery and reduced the Mg/Li ratio from 9.85 to 0.57 [113].

The electrodialysis process could be used to obtain material for Li-ion battery production directly from brine with 98.8% purity and 118 g/L of Li nitrate solution [114]. Zhang et al. (2021) evaluated the use of monovalent membranes to separate Li from brine, obtaining an 83.5% recovery percentage with 99.68% of purity [115]. As observed, nanofiltration and electrodialysis can be used to obtain Li from brine as a source for different markets.

#### 4.6. Application of Membranes for Separation and Purification Processing

The literature reports on the use of membrane technology for the separation and concentration of metals in a solution. This was found in studies from different sources of critical metals. The focus on Li, Co, and rare earth elements (Sc, Y, La, and Nd) was related to their application in green technologies with low greenhouse gas emissions. Although membrane technology is widely (and traditionally) used for water and wastewater treatment, the bibliometric analysis (Section 4.1) demonstrated a strong link between these critical metals and separation techniques. Regarding the technologies, evaluations of nanofiltration, microfiltration, and ultrafiltration concentrate leach solutions and reuse water and acid after leaching was observed. Electrodialysis was evaluated as an ion-exchange technique for the selective separation of ions. It was observed that manuscripts regarding reverse osmosis were not found for any critical metals.

The membrane technique may be used for purification to remove impurities. For example, Khaless et al. (2022) purified industrial phosphoric acid by nanofiltration for sulfate, fluorine, and arsenic removal. The authors evaluated the MPS36 membrane, which operated at 1700 L/h and 40 bar. In addition to anion removal, nanofiltration reduced the Cd, Al, Fe, and rare earth elements concentration by over 95% [116]. Similarly, Mans et al. (2020) evaluated hollow fiber membranes to purify the Ni-Co leach solution [117].

The literature review reported an increased interest in membrane technology for rare earth element separation owing to higher selectivity, lower energy requirements, and the possibility of zero liquid discharge [118,119]. To achieve the goals for selectivity, there were two main membrane-based techniques: supported liquid membranes and polymer liquid membranes. Both combine the high selectivity of the solvent extraction technique with a smaller area and a smaller volume. The organic extractants studied were usually applied in the traditional solvent extraction of rare earth elements, such as D2EHPA, Cyanex 272, TBP, and TOPO [120,121].

The extraction of Sc has been evaluated by comparing solvent extraction techniques and supported liquid membranes. As observed by Rout and Sarangi (2022), the recovery of Sc achieved 99.9% using solvent extraction and 91% using a supported liquid membrane, and Cyanex 272 was used as a carrier. According to the authors, the separation of Sc was higher than Ni, Mn, Co, Zn, Fe, and Cu [122]: the most common contaminants of nickel laterite waste processing to obtain Sc [123]. Polymer inclusion membranes with organic extractants, such as N-[N,N-di(2-ethylhexyl)aminocarbonylmethyl]-glycine (D2EHAG), and N-[N,N-di(2-ethylhexyl)-aminocarbonylmethyl]phenylalanine (D2EHAF) also achieved high extraction rates for Sc than Ni, Al, Co, Mn, Cr, Mg and Ca [124]. The literature also reported the use of Nd and La using mono-(2-ethylhexyl) phosphoric acid (EHEHPA, or P507) as a carrier in supported liquid membranes [125]; this included Dy, Ga, and Nd using Cyanex 572 (organophosphorus-based extractant) as a carrier in emulsion liquid membrane [126,127]; 2-thenoyltrifluoroacetone as a carrier in Y extraction [128]; and 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester as a carrier for Y and Eu separation [129].

Membrane technology is an important alternative for Li extraction from different sources. From primary sources, the main challenge to be overcome is the selective separation from alkali and alkaline earth metals, including Na, K, Ca, and Mg. Electromembrane extraction using a membrane supported with D2EHPA, TBP, heptafluorodimethyloctanedione (HFDOD), and tri-n-octylphosphine oxide (TOPO) [130,131], a polymer inclusion membrane supported with ionic liquids [132,133], metal–organic frameworks [134], and graphene oxide membranes [135] have been studied, and the results demonstrated a high separation rate. Electrodialysis has been demonstrated to be an important technique for Li separation from battery recycling, as monovalent membranes can separate up to 99.4% of Li without Co [136]. In the literature review, the interest in developing new membranes to support acid and solvent solutions was observed, which is important for designing hydrometallurgical processes [137].

Regarding the sources discussed in the present manuscript (Sections 4.2–4.5), increased interest was observed in obtaining critical metals from secondary sources (such as AMD, industrial wastes, and e-waste recycling). Moreover, the Li demand puts pressure on its extraction from primary sources, which includes brines. Therefore, the present review established a strong connection between the use of membrane technologies and the promotion of a circular economy.

# 5. Conclusions

The present study aimed at a literature review of membrane technology applications for separating critical metals. It focused on Li, Co, and rare earth elements (Sc, Y, La, and Nd) due to their growing demand for net zero technologies and their risk in the short and medium-term supply chain. The bibliometric analysis demonstrated a high link between critical metals and membrane separation. Among the most traditional technologies were microfiltration, ultrafiltration, nanofiltration, and electrodialysis. Studies about reverse osmosis were not found to separate critical metals since this technique is mainly applied to obtain high-pure water. Compared to nanofiltration, reverse osmosis requires more energy, and in hydrometallurgical processes, the main goal is to obtain metal or acid recovery. Despite these traditional techniques, new membranes are being developed to improve selectivity, including supported liquid membranes and polymer inclusion membranes. Both techniques combine the high selectivity of the solvent extraction technique with a smaller area and a smaller volume, but future developments are necessary to achieve a pilot and industrial scale. This review of the literature demonstrates the development of membrane technology and the potential to obtain critical metals from several sources. This work acknowledges the potential use of several types of membranes to obtain metals from primary and secondary sources, AMD, industrial wastes, recycling of e-wastes, and brines.

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