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Recent advances in electrodialysis technologies for recovering critical minerals from unconventional sources

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ABSTRACT

The global challenge of climate change and the pursuit of carbon neutrality drive the demand for clean energy technologies, increasing the need for critical minerals. However, diminishing ore quality and supply chain vulnerabilities highlight the necessity of exploring unconventional mineral sources. This paper assesses the potential of sources such as low-grade ores, brines, and secondary byproducts for extracting critical minerals, focusing on lithium, nickel, cobalt, and rare earth elements. Conventional refining techniques struggle with the complexity and low concentrations found in unconventional sources, prompting the exploration of innovative methods. Electrically driven membrane technologies, particularly electrodialysis (ED), show promise for selectively transporting metals through ion exchange membranes. The combination of functionalized membranes and advanced ED technologies offers high selectivity for specific elements, and renewable energy-based ED operations can minimize environmental impact. Recent advancements in ED, including selective ED and liquid membrane ED, demonstrate efficient recovery of critical minerals. This comprehensive review explores the principles and recent progress in ED technologies, with a focus on recovering critical minerals from unconventional sources. The paper is structured into three main sections: categorizing the properties of unconventional sources of critical minerals based on the latest literature, exploring the fundamental principles underpinning modern ED technologies, and investigating recent breakthroughs in the separation and recovery of critical minerals from unconventional sources using ED technologies. This framework highlights the potential of advanced ED technologies in efficiently processing diverse unconventional sources and adeptly retrieving critical minerals.

1. Introduction

The global transition towards carbon neutrality is driving the phasing out of conventional fossil fuel-based energy systems in favor of cleaner alternatives like renewable energy, electric grids, rechargeable batteries, and electric vehicles [1]. While these technologies significantly reduce CO_2 emissions by decreasing reliance on fossil fuels, their production requires a substantial amount of minerals for infrastructure and manufacturing [2]. According to the International Energy Agency (IEA), achieving carbon neutrality by 2050 will demand over six times the current metal consumption [3]. However, there has been a consistent decline in ore quality, exemplified by the reduction in copper (Cu) ore grade from 0.9 % in 2005 to 0.7 % in 2019 [3]. Similarly, research by Mudd et al. indicates a general downward trend in ore grades for various metals, including Cu, lead (Pb), zinc (Zn), and nickel (Ni).

This surge in demand coupled with declining ore quality raises concerns about potential supply chain disruptions and their impact on the energy transition. Consequently, many regions and nations are now designating certain minerals and materials as ’critical’ due to their pivotal role in future energy sectors and high-tech applications (Fig. 1)

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[4–7]. Although the terminology may differ across countries and regions, critical minerals are generally recognized as chemical elements and minerals crucial for economic and national security, which are encountering substantial supply challenges due to physical scarcity, intricate refining processes, and geopolitical risks [8].

The number and types of critical minerals designated vary by country, but they commonly include several elements closely related to the clean energy transition, such as lithium (Li), cobalt (Co), Ni, and rare earth elements (REEs) [9]. In response to the mineral supply crisis, efforts are increasingly focused on exploring unconventional sources such as low-grade ores, brines, and secondary sources, which have previously received less attention [10–14].

Unconventional sources refer to geological deposits such as coal underclays, marine phosphates, low-grade ores, and brines, as well as secondary byproducts from human activities, including mining waste, coal fly ash, and steel-making slags [8]. These unconventional sources differ from conventional, well-established deposits, presenting significant challenges in minerals refining due to factors such as low metal concentrations, complex intermixing with other materials (such as alkali or alkaline earth metals, gangue minerals, plastics, ceramics, etc.), and inconsistent composition [15–17].

Addressing these challenges requires highly sophisticated and tailored processing technologies [18]. Traditional metallurgical techniques for refining unconventional sources often require more energy and solvent usage, leading to increased overall CO_2 emissions and waste generation, thus raising significant environmental concerns. To mitigate these issues and minimize energy and solvent usage in refining low-concentration or complex matrices of critical minerals, the development of advanced technologies surpassing conventional methods is essential [17,19]. This advancement involves exploring innovative metallurgical processes such as novel hydrometallurgy, pyrometallurgy, solvometallurgy, and electrometallurgy.

In this context, membrane technology, an emerging field in metal separation and recovery, is attracting attention. Historically, membrane technologies have been utilized in water and wastewater treatment and desalination, leveraging the selective permeation properties of membranes [20,21]. Recently, electrically driven membrane technology, particularly electrodialysis (ED) coupled with ion exchange membranes (IEMs), has received significant interest for its potential in the selective separation of critical minerals [22]. The development of IEMs through structural modifications, pore size adjustments, and functionalization enhances the selectivity for specific ions, enabling the efficient separation of critical minerals even at low concentrations [22–27]. The synergistic combination of recently developed ED configurations and functionalized membranes offers advantages for targeting various feedstocks and critical minerals [28].

Several inherent advantages of ED systems include (i) mild operational conditions of temperature and pressure, (ii) reduced consumption of chemicals such as solvent extractants and precipitants, and (iii) decreased discharge of wastewater, all of which contribute to minimizing the environmental footprint of critical mineral extraction [29–32]. Furthermore, integrating electrically driven membrane technologies with renewable energy sources can potentially reduce the carbon footprint of ED systems [29].

Several recent papers have reviewed the application of ED technologies for metal recovery. Wu et al. conducted an in-depth analysis of electrosorption and ED technologies for Li recovery from salt-lake or brine, focusing on the principles, membrane and electrode materials, and operational modes [33]. Juve et al. reviewed the current state of ED technologies for the removal and recovery of various metals from acidic metallic effluents, providing insights into the effects of operational parameters and proposing strategies to overcome associated challenges [34]. Gurreri et al. conducted a comprehensive review of studies on the removal and recovery of heavy metals and toxic metals from wastewater using ED technologies [35]. Their research provided experimental results, process performance, and the advantages and disadvantages of ED technologies, aiming to highlight prospects in this field. Kabir et al. reviewed the application of ED technologies in the water industry, such as municipal and industrial wastewater treatment plants (WWTPs) and metallurgical industries [36]. Their research focused on resource

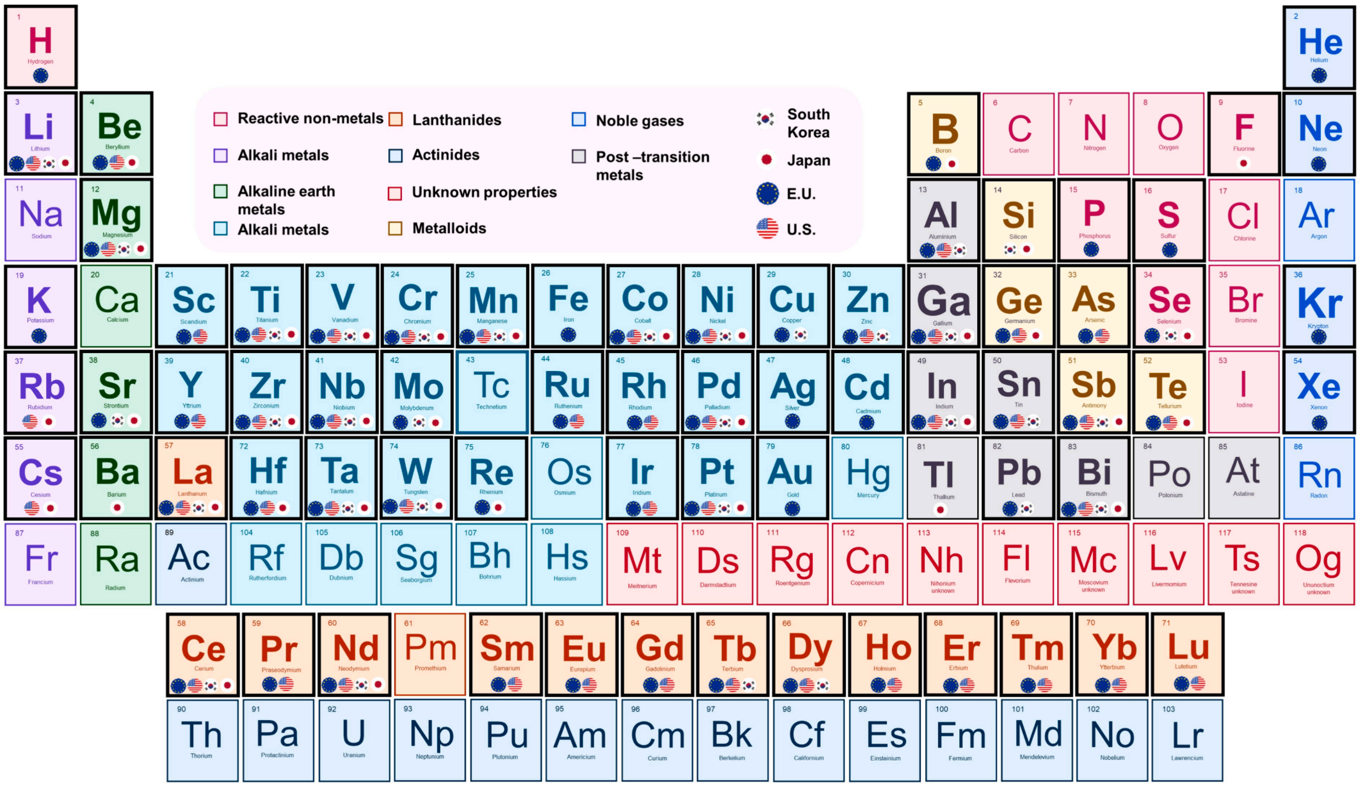

Fig. 1. The periodic table is highlighted as crucial minerals across the United States, European Union, Japan, and South Korea.

recovery from wastewater, encompassing nutrients, metals, water, and energy, from a circular economy perspective. These papers have offered insights into ED technologies applied for metal removal and recovery from aqueous phase feedstocks, such as Li brine and wastewater. However, the potential of ED technologies as a tool for metal refining and critical mineral recovery from various sources, such as end-of-life products, solid-phase slag, Li brine, and low-concentration geological minerals, has yet to be extensively explored.

To consider ED technologies as a viable method for recovering critical minerals from unconventional sources, it is crucial to have a comprehensive understanding of both recent advancements in ED technologies and the condition of critical minerals in the feedstocks, such as metal concentration, impurities, and leachate properties.

Recent advancements in ED technologies for metal recovery encompass a range of methods, including selective electrodialysis (SED), bipolar membrane electrodialysis (BMED), liquid membrane electrodialysis (LMED), and chelation-assisted electrodialysis (CAED) systems [37–40]. The applicability of these ED technologies varies depending on the properties of the target metals, such as ionic size, charge density, and metal groups [26,40–42]. Additionally, the effectiveness of ED technologies is influenced by the type of feedstock (e.g., brine, wastewater, or industrial wastes), each with distinct characteristics in terms of pH, alkalinity, and metal content [40,42,43]. Hence, there is a crucial need to develop IEMs and ED systems tailored specifically to the target feedstock and metals.

Therefore, this review endeavors to explore the principles and recent advancements of ED technologies and IEMs, with a particular emphasis on recovering critical minerals from unconventional sources. Specifically, this review explores ED technologies, focusing not on the traditional removal and recovery of heavy metals from wastewater, but on separating and recovering energy-related critical minerals (i.e., Li, Ni, Co, and REEs) from various sources.

The paper is organized into three primary sections. Firstly, it categorizes and delineates the properties of unconventional sources of critical minerals based on the latest reports. Secondly, it focuses on comprehending the fundamental principles underpinning modern ED technologies. Lastly, it examines recent breakthroughs in separating and recuperating critical minerals from unconventional sources through ED technologies. This framework underscores the potential of advanced ED technologies in efficiently processing diverse unconventional sources and adeptly retrieving critical minerals.

2. Unconventional sources of critical minerals

Several critical minerals are essential for the clean energy transition. In the battery sector, Li, Co, Ni, manganese (Mn), and graphite play crucial roles. Additionally, REEs, particularly neodymium (Nd) and dysprosium (Dy), are vital for magnets in wind turbines, electric vehicles (EVs), batteries and other clean energy technologies. The IEA predicts a significant demand surge for these minerals by 2040: Li (41.9 times), Co (21.3 times), Ni (19.4 times), Mn (8.1 times), REEs (7.3 times), and Cu (2.6 times) compared to the demand in 2020 [3].

An investigation by the International Renewable Energy Agency (IRENA) analyzed the frequency of ’critical’ designations for materials across various lists [9]. Among 51 materials, Li, Co, Cu, Ni, Nd, Dy, indium (In), gallium (Ga), tellurium (Te), and praseodymium (Pr) were consistently identified as critical. These minerals are particularly important for renewable energy, batteries, and permanent magnets, highlighting their indispensable role in clean energy technologies. Notably, the recycling rate of Li and REEs from end-of-life products remains below 1 % [3].

This section focuses on battery minerals (i.e., Li, Co, and Ni) and REEs due to their pivotal role in the energy transition. To understand the potential of unconventional sources, this review examines recent interests in feedstocks like brine, industrial wastes, and low-concentration ore. Tables 1 and 2 summarize the metal concentrations, associated impurities, and challenges for each resource.

2.1. Li-bearing feedstock

The supply of Li primarily relies on spodumene ore and Li brines, with significant unconventional sources including spent Li ion batteries (LIBs) and low-concentration brines [67–69]. Spent LIBs, a substantial waste stream in the Li production lifecycle, not only contain Li but also various critical minerals such as Ni, Co, and Mn within their cathode materials. The concentration of Li in cathode materials typically falls within the range of 3.4 to 8 %, although it may vary depending on battery type or cathode composition [45–48].

The electrochemical recovery process from spent LIBs involves multiple stages, including shredding, grinding, and mechanical sorting,

Table 1
Categorization of unconventional sources of Li and Ni: concentrations, impurities, and extraction issues.

Metal	Feedstock	Concentration		Impurities	Extraction Issues	References
Lithium	Brine	Li brine Geothermal brine	60–1,570 ppm 40–180 ppm	Na, Mg, Ca, K, B, SO_4^{2-}, Cl^-	High concentrations of salts (e.g., Na and Mg) more abundant than Li	[44]
	Spent lithium ion batteries (LIBs)	3.4–8 % (Depends on the type of cathode material)		Ni, Mn, Co, Fe, C, Binder, etc.	Multi-stage treatment (physical beneficiation, leaching, extraction, etc.) and feedstock-dependent tailored process required	[45–48]
Nickel	Spent battery(LIBs, NiMHs or Ni-Cd Battery)	LIBs	11.8–32.5 % (Depends on the type of cathode material)	Li, Mn, Co, Al, C, Binder, etc.	– Multi-stage treatment (physical beneficiation, leaching, extraction, etc.) and feedstock-dependent tailored process required	[48–50]
		Ni Metal Hydride (NiMH) Battery	55–65 %	La, Ce, Nd, Co, Mn, etc.	– Difficulties in Ni and Co separation due to similar physicochemical properties of transition metals	[51]
	Electroplating waste	Wastewater Sludge	20–200 ppm ~31.6 %	Cr, Zn, Ca, Cu, Fe	– Separation of heavy metals (i.e., Cu and Zn) required – Zn tends to remain with Ni	[52–54]
	Unconventional ore and mine waste(Olivine, Cu-slag)	Cu-Slag	~ 0.5 %	Ca, Mg, Co, Cu	– Lower concentration of Ni compared to traditional ore require more energy and solvent consumption – Base elements (i.e., Ca, Mg, Cu, etc.) separation required	[55]
		Olivine	~ 0.27 %	Mg, Si, Fe	– Lower concentration of Ni compared to traditional ore require more energy and solvent consumption – Silicate structure ($(Mg,Fe)_2SiO_4$) binding with Ni and energy-intensive process required	[56]

Table 2
Categorization of unconventional sources of Co and REEs: concentrations, impurities, and extraction issues.

Metal	Feedstock	Concentration		Impurities	Extraction Issues	References
Cobalt	Magnet (Samarium–Cobalt Magnet)	50–60 %		Sm, Cu, Fe, etc.	High concentration of solvent (H_2SO_4) required for leaching	[57]
	Spent battery(LIBs or NiMHs Battery)	LIBs	9–60 % (Depends on the type of cathode material)	Li, Mn, Co, Al, C, Binder, etc.	Multi-stage treatment (physical beneficiation, leaching, extraction, etc.) and feedstock-dependent tailored process required	[58]
		NiMH Battery	5.5–7 %	La, Ce, Nd, Ni, Mn, etc.		[51]
	Unconventional ore and mine waste(Olivine, Cu-slag, Ni-slag)	Olivine	~0.005 %	Mg, Si, Fe	– Lower concentration of Co compared to traditional ore required more energy and solvent consumption – Silicate structure ($(Mg,Fe)_2SiO_4$) binding with Co and energy-intensive process required	[56]
		Cu-Slag	~0.16 %	Ca, Mg, Ni, Cu	– Base elements (i.e., Ca, Mg, Cu, etc.) separation required – Low concentration of critical minerals (e.g., Ni and Co) and variance of feedstock composition	[55]
		Ni-Slag	~7.46 %	Li, Co	High concentration of solvent (e.g., H_2SO_4) required for leaching	[59]
REEs	Pre-Manufacturing(scraps, residues)	Magnet swarf	REEs: 30–40 %	Nd, Pr,Dy, Tb, Sm	Multi-stage treatment (physical beneficiation, leaching, extraction, etc.) and feedstock-dependent tailored process required	[60,61]
		Polishing powders	REEs: 50–99 %	La, Ce		[60,61]
	Post-Manufacturing(E-waste, etc.)	NdFeBMagnet	REEs: 30–40 %	Fe_2O_3, MnO	– High concentration of solvent (e.g., H_2SO_4) required for leaching – Separation of REEs from high concentration of Fe_2O_3 (68 %)	[60,61]
		NiMH battery	REEs: 8–10 %	Mn, Zn, Ni, Co	Multi-stage treatment (physical beneficiation, leaching, extraction, etc.) and feedstock-dependent tailored process required	[62]
	Landfilled waste	Coal fly ash	0.035–0.07 %	Ca, Fe, Al, Mg	– Lower concentration of REEs compared to traditional ore required much energy and solvent consumption – Aluminosilicate structure binding with REEs and energy-intensive process required	[60,63–66]
		Phosphogypsum	0.3–0.6 %	SO_3,CaO,SiO_2	– Large amount of toxic solvent (acid and base) required – Selective separation and concentration of low-concentrated REEs required	
		Red mud	0.04–0.045 %	Fe. Al. Si, Ca, Na		
		Steel-making slag	0.03–0.05 %	Ca, Si, Al. Mg		

followed by metal leaching in acidic solutions [12,70,71]. Generally, substantial amounts of solvents (e.g., sulfuric acid (H_2SO_4) and sodium hydroxide (NaOH)) are employed for metal recovery of spent LIBs, resulting in the generation of large volumes of wastewater including sodium sulfate (Na_2SO_4). The resulting leachate, typically acidic, contains a mixture of critical minerals (e.g., Li, Co, Ni, Mn, etc.), necessitating comprehensive multi-element recovery and separation processes to maximize the extraction of valuable metals [72].

For Li brines, the conventional process primarily involves evaporation, followed by the precipitation of salts from continental brines [73,74]. Recent advancements have led to the exploration of low-concentration brines such as geothermal and oilfield brines. Additionally, there is ongoing development of direct lithium extraction (DLE) technologies, which allows for more efficient Li recovery directly from various brine sources [75]. Recent studies have indicated that brines contain Li concentrations ranging from 60 to 1,570 ppm, with geothermal brines containing lower concentrations (i.e., 40 to 180 ppm) [44].

A significant challenge in the electrochemical extraction process is the separation of Li from alkali or alkaline earth metals, such as sodium (Na), potassium (K), calcium (Ca), and magnesium (Mg), which exhibit similar behavior to Li [12,76–79]. Specifically, the similar ionic radii of Li^+ (i.e., 0.069 nm) and Mg^{2+} (i.e., 0.072 nm) ions present a major challenge for their separation in ED technologies [80].

2.2. Ni-bearing feedstock

Ni is primarily obtained from oxide ores such as laterite and sulfide ores like pentlandite [81,82]. Recent studies have focused on various unconventional sources containing Ni, including (i) spent batteries (LIBs or nickel-metal hydride (NiMH) batteries), (ii) electroplating waste, and (iii) unconventional ores (e.g., olivine) and mine waste [48–51,83]. In waste batteries, $Li(Ni_xCo_yMn_{1-x-y})O_2$ (NCM) cathode materials typically contain Ni concentrations ranging from 11.8 % to 32.5 %, while NiMH batteries contain approximately 55 % to 65 % Ni [48–51]. These waste batteries comprise a variety of critical minerals; NCM cathode materials contain Li, Co, and Mn, while NiMH batteries include REEs such as lanthanum (La), cerium (Ce), Nd, and Pr. Achieving high selectivity for Ni in the electrochemical separation process remains challenging due to the similar physicochemical properties of transition metals such as Ni and Co [84,85].

Ni finds extensive usage in the electroplating industry due to its exceptional corrosion resistance. The Ni plating process typically generates significant wastewater volumes, often containing Ni concentrations ranging from 20 to 200 ppm [52,53]. This wastewater is commonly treated using chemical precipitation, leading to the production of a substantial amount of Ni-rich sludge, which comprises approximately 31.6 % of its composition [54].

Several recent studies have investigated various methods for Ni recovery from electroplating waste, including membrane separation processes (e.g., reverse osmosis (RO) and nanofiltration (NF)) and several electrochemical technologies (e.g., ED, electrolysis, and electrodeionization) [86,87]. Direct treatment of these complexes or their dissociation by reducing the pH with concentrated acid solvents becomes imperative. Additionally, the presence of other heavy metals such as chromium (Cr), Cu, Zn, and Ni in sludge and wastewater underscores the necessity for an integrated approach to maximize the extraction of valuable metals.

Various types of mine waste containing Ni (such as pyrolusite tailings, Cu-Ni slag, and Ni-slag) in concentrations ranging from 0.3 % to 3.2 % are gaining attention as novel sources of Ni [11,88,89]. In 2022, the U.S. Department of Energy (DOE) initiated several projects focused on extracting critical minerals from extensively piled mine waste and unconventional sources using carbon–neutral pathways [90]. Recent studies have also focused on recovering low concentrations of Ni (approximately 0.22 %) from olivine, which is considered the origin of laterite [83,91]. Given that Ni is present in low concentrations and intricately mixed with various base elements (such as Ca, Mg, aluminum (Al), silicon (Si), iron (Fe), and Cu), a multistage process involving leaching, separation, and concentration becomes necessary.

2.3. Co-bearing feedstock

Co is primarily obtained as a byproduct in the refining processes of Cu-Co ore, Ni-Co ore, and laterite, rather than from primary Co ore sources (e.g., cobaltite, erythrite, and glaucodot) [88,92,93]. Due to the challenges in increasing its supply, Co is widely acknowledged as a critical mineral in numerous countries [9]. Unconventional sources of Co include samarium-cobalt (SmCo) magnets, waste batteries, and unconventional ores and mine wastes like olivine, copper- and Ni-slags [51,55–59]. In SmCo magnets, Co comprises approximately 50–60 % and is accompanied by valuable Sm, with both metals being targeted for recovery [57]. Recycling of SmCo magnets has been achieved through hydrometallurgical or pyrometallurgical pathways. In the electrochemical recovery process, acid leaching is necessary to ionize Sm and Co, and subsequent elemental separation is crucial due to their relatively high concentrations.

In spent batteries, Co concentration typically ranges from 9 % to 60 % in various cathode materials such as NCM and $LiCoO_2$ (LCO), while it constitutes about 5.5–7 % in NiMH batteries [51,58]. Due to the high cost of Co, its recovery is essential for maximizing recycling value. In batteries, Co typically exists in an oxidized state (Co^{3+}), requiring the use of reducing agents like hydrogen peroxide (H_2O_2) in the hydrometallurgical recovery process [94,95]. The concentration and characteristics of Co vary depending on battery type and cathode material, necessitating an appropriate approach to recovery. Additionally, the similar physicochemical properties of Co and Ni present challenges for their separation in electrochemical recovery methods [84,85].

Moreover, Co is also found in mine wastes such as Cu-slag (0.5 %) and unconventional ore such as olivine (55 ppm) [55,56]. Similar to Ni, recent studies have been exploring methods to recover lower concentrations of Co from unconventional sources [55,56]. Given the low concentration of Co in unconventional sources, combining the Co recovery process with other technologies, such as CO_2 storage, could be advantageous [83,91]. Typically, mine wastes and unconventional sources contain a mix of elements including Ca, Mg, Fe, Si, Al, and Cu. Accordingly, selective recovery processes are essential for extracting low concentrations of Co from such complex matrices.

2.4. Rees-bearing feedstock

REEs are extensively utilized in renewable energy, batteries, catalysts, and various high-tech applications, resulting in a significant surge in demand [61,96]. Presently, REEs are primarily extracted from minerals such as bastnasite, laterite clay, monazite, and loparite [16,17,97,98]. The extraction of high-demand REEs such as Nd and Dy often involves the co-extraction of lower-demand REEs, including La and Ce, due to fluctuating demands driven by evolving technologies in various applications [99]. Adjusting production to optimize the operational margins results in surpluses of some REEs and shortages of others [100]. The scarcity of less common REEs causes significant price spikes due to their highly inelastic demand, creating an imbalance among these elements [96,99]. This imbalance presents a challenge for producers, who must align market demand with the natural distribution of REEs in mining operations to meet critical application needs [99,101].

Jones et al. categorized REE-bearing unconventional sources into three groups: pre-consumer REEs (e.g., scrap and residue), end-of-life products (e.g., phosphors, batteries, and magnets), and landfilled REEs (e.g., industrial waste, coal fly ash, and phosphogypsum) [102]. Pre-consumer REEs typically refer to manufacturing scraps from neodymium-iron-boron (NdFeB) magnet production or residues from polishing processes. Manufacturing scraps of NdFeB magnets usually contain magnetic REEs such as Nd, Dy, Pr, terbium (Tb), and Sm, with concentrations around 30–40 % [60,61]. In polishing powders, Ce and La are present in high concentrations, between 50–99 % [60,61]. Direct recycling is widely regarded as an appropriate method for REE recycling due to their substantial REE content.

Post-consumer REEs, referred to as end-of-life products, include a variety of items such as waste phosphors, light-emitting diodes (LEDs), spent batteries, etc. NdFeB magnets contain high concentrations of REEs such as Nd, Pr, Dy, Tb, and Sm, typically in the range of 30 % to 40 % [60,61]. Waste phosphors contain substantial REEs (44–50 %), including yttrium (Y), europium (Eu), La, Ce, and Tb, and LEDs contain around 5 % REE-based dyes like Ce, Y, and Eu [60,61,103].

Separation of single REEs is challenging due to their similar physicochemical properties, including ionic radii. Additionally, these end-of-life products often require multi-stage processing due to the complex mixture of REEs with other materials like ceramics and plastics. The variance of metal concentration and composition in different waste streams necessitates feedstock-tailored processing methods to effectively recover REEs. Landfilled waste contains considerable amounts of REEs in various feedstocks such as phosphogypsum (0.3–0.6 %), red mud (400–450 ppm), fly ash (350–700 ppm), and steelmaking slag (300–500 ppm) [60,63–66]. The U.S. DOE has undertaken projects to concentrate REEs above 2 % from coal waste that contains over 300 ppm REEs, transforming them into alternative REE sources [104,105]. Recent studies have also explored CO_2 storage in base elements of landfilled waste, such as Ca and Mg, while concurrently concentrating REEs [66,106]. Given the low concentration of REEs intermixed with major elements (e.g., Ca, Mg, Si, Fe, and Al), landfilled waste necessitates highly efficient and multistage processing (e.g., beneficiation, leaching, extraction, etc.).

2.5. Cut-off-grade of minerals and challenge of metal refinery

The concept of cut-off grade holds significance in mining, denoting the minimum concentration of minerals necessary for economical mining operations [107,108]. This threshold varies depending on factors such as the metallic characteristics of the deposit and the feasible amount of metal extraction. For critical minerals, typical cut-off grades range around 0.5 % of Li_2O in spodumene, 150–300 ppm of Li in brine, 1–3 % of Ni in laterite or sulfide ore, 200–300 ppm of Co in sulfides or byproducts, and 2.5 % REO in rare earth ores [109–113]. It's essential to recognize that a metal concentration exceeding the typical cut-off grade in unconventional sources doesn't always ensure economic viability. However, comparing metal concentrations across unconventional sources can offer indirect indications of the presence and abundance of critical minerals. Table 3 outlines the cut-off grades for selected critical minerals.

For aqueous-phase feedstocks, direct extraction methods are commonly employed for metal recovery, leading to the development of various electrochemical techniques tailored for these specific feedstocks, such as DLE and Ni recovery processes via electrochemical technologies [44,114]. Conversely, many unconventional sources exist in solid forms, including materials like plastics, polymers, and ceramics. Although end-of-life products such as spent batteries and electronic scrap often contain higher concentrations of critical minerals compared to conventional ores, they typically necessitate complex, multi-stage processing steps (such as physical beneficiation, leaching, and extraction) [115–119]. Furthermore, the composition and properties of feedstocks vary

Table 3
Cut-off-grade of critical minerals and classification of unconventional sources.

Metal	Traditional ore	Cut-off-grade of traditional ore	Unconventional sources (relatively high concentration)	Unconventional sources (relatively low concentration)	References
Lithium	Spodumene	Li_2O: ~0.5 %	Waste LIBs and Li brine for DLE	Low-concentrated brine (geothermal brine)	[109]
	Brine	150–300 ppm			[110]
Nickel	Laterite and sulfide ore	1–3 %	Waste battery (LIBs, NiMHs), electroplating sludge	Olivine, Cu-slag	[111]
Cobalt	Co-bearing sulfides or byproducts	0.02–0.03 %	SmCo magnet, waste battery (LIBs, NiMHs),	Olivine, Cu-slag, Ni-slag	[112]
REEs	Bastnasite and monazite	REO: ~2.5 %	Pre-manufacturing waste (scrap, residue), post-manufacturing waste (waste battery, E-waste)	Coal fly ash, phosphogypsum	[113]

significantly depending on their origin, requiring the development of feedstock-tailored processes to ensure efficient metal recovery [119,120].

In unconventional ores and mining wastes, low concentrations of metals are typically encountered, necessitating increased use of solvents and energy for extraction [15,121]. The presence of critical minerals with various base metals like Ca, Mg, Al, Si, and Fe adds complexity to designing the refining process. Therefore, a sophisticated, multi-stage separation process is required, consuming significant amounts of solvents and energy. Recent research has explored the potential of base elements (such as alkali and alkaline earth metals) as CO_2 storage media during critical mineral recovery [66,83,91,106]. These alkali and alkaline earth metals can react with CO_2 in thermodynamically favorable ways, suggesting integrated processes that simultaneously enable CO_2 storage and mineral recovery [122]. This innovative approach not only facilitates resource recovery but also operates in carbon capture, utilization, and storage (CCUS). However, addressing the heterogeneity of these metals remains a challenge, as critical minerals and alkali or alkaline earth metals require different treatment processes.

3. Fundamentals of separation and recovery mechanisms of critical minerals in electrically driven membrane technologies

The functionalities and performance requirements for membranes and ED systems depend heavily on the composition and characteristics of the feedstock. For instance, extracting Li from low-concentration Li brines requires high selectivity to separate Li^+ ions from alkali metals (e.g., Na and K) and alkaline earth metals (e.g., Ca and Mg). Similarly, recovering valuable metals from cathode materials in spent batteries, such as Ni, Co, Mn, and Li, requires high selectivity for transition metals like Ni and Co, which is challenging due to their similar properties. Additionally, sources such as slag or mine waste contain critical minerals in low concentrations with a diverse range of base elements (e.g., Ca, Mg, Al, Si, Fe, and Cu), necessitating the ability to target and recover these low-concentration critical metals effectively. Therefore, achieving optimal selectivity for the target metals and feedstocks is essential for the successful application of ED technology.

The selectivity observed in electrically driven membrane processes is fundamentally determined by the ability of membranes to separate specific substances. In the context of achieving selectivity for targeted ions, membranes designed for electrochemical applications are fabricated by exploiting the intrinsic properties of ions along with the principles underlying ion separation mechanisms. The mechanisms that enable the selective permeation of target ions through these membranes primarily cover size exclusion, electrostatic interactions, and chemical affinity. In ED membrane processes, the application of an electrical potential serves as the driving force, facilitating the movement of ions through the membrane [123,124]. ED is recognized as an example of such electrically-driven membrane processes, which can be specifically designed in various forms to separate particular ions or to accommodate different types of membranes [123]. These systems include SED, BMED, LMED, and CAED each designed to optimize the separation process for specific target ions or to utilize distinct membrane properties [37,124–126]. Fig. 2 provides an overview of ion separation mechanisms in electrically driven membrane processes. These mechanisms are described in detail in Section 3.1 and 3.2.

3.1. *Ion separation mechanism of membrane*

3.1.1. *Size exclusion*

The underlying principle of the size exclusion approach in electrically driven membrane processes involves controlling the sizes of membrane pores to enable selective permeation of desired ions [127,128]. In this strategy, the membrane's pore size is meticulously tailored to surpass the diameter of the hydrated ions targeted for transport, facilitating their passage through the membrane [129]. Concurrently, the engineered pore serves as a barrier to ions with larger dimensions, preventing their passage (Fig. 2 **(a)**) [129–131]. This selective permeability is attained by utilizing the disparity in size between the desired ions and other coexisting species, ensuring that only ions meeting specific size criteria can traverse, thereby enabling a highly selective and efficient separation process [132–134].

To achieve effective ion sieving, it is crucial that the nanometer-scale pores within membranes are accurately manufactured and evenly distributed. Significant research efforts have focused on designing membrane structures or pores tailored for ion separation in electrically driven processes. Notably, two-dimensional (2D) materials such as graphene oxide (GO) [135–138], molybdenum disulfide (MoS_2) [139,140], MXene nanosheets [141,142], metal–organic frameworks (MOFs) [143,144], and covalent organic frameworks (COFs) [145,146] have been extensively investigated.

GO, MoS_2, and MXene nanosheets form a lamellar structure by stacking layers, characterized by sub-nanometer-scale 2D channels between the lamellar layers [147–149]. Theoretically, only ions with a hydrated diameter smaller than the interlayer spacing can penetrate the membrane, while larger hydrated ions are excluded [150]. Porous COFs and MOFs offer specific and uniform pore sizes within the dense structure of polymeric membranes. The transport of ions in their hydrated form through these membranes in aqueous solutions is governed by size-exclusion principles [151,152]. These materials offer structural and pore size adjustability at the nanoscale, presenting significant potential for modifying membrane properties to achieve specific ion separation objectives.

Although well-defined nano-pores are crucial for facilitating effective ion sieving by selectively permitting ions of specific sizes to pass through, this selectivity can inherently decrease ion permeability [153,154]. Recent research has also been dedicated to addressing the trade-off between selectivity and permeability [144,155,156]. This entails devising strategies and materials to uphold high selectivity for target ions while minimizing the impact on ion transport rates, with the goal of optimizing membrane performance for electrically driven ion separation processes.

3.1.2. *Electrostatic repulsion*

Ion separation through electrostatic repulsion operates on the fundamental principle that ions with opposite charges attract each other, while ions with identical charges repel each other [157]. Membranes can be engineered to carry either a positive or negative charge by

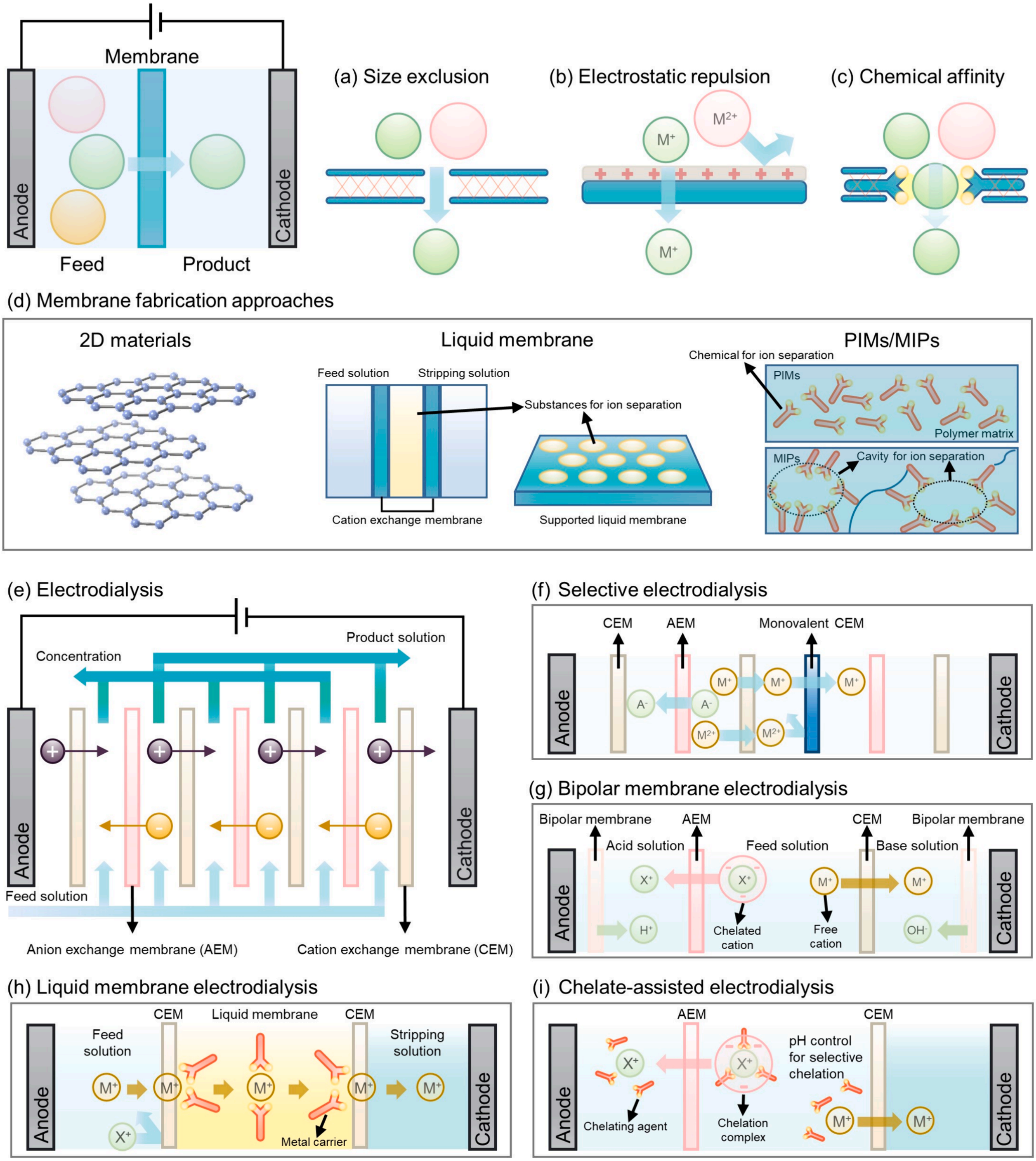

Fig. 2. An overview of electrically driven membrane processes for the recovery and separation of critical minerals, focusing on key ion separation mechanisms such as (a) size exclusion, (b) electrostatic repulsion, and (c) chemical affinity. Additionally, it covers (d) membrane fabrication techniques including 2D materials, liquid membranes, polymer inclusion membranes (PIMs), and molecularly imprinted polymers (MIPs). Principles of operation encompass (e) ED, (f) SED, (g) BMED, (h) LMED, and (i) CAED.

integrating charged polymers during the polymerization process or by applying a charged polymer coating to the membrane surface [158–162]. This modification allows for the selective transport or rejection of ions based on their charge [158,159].

As illustrated in Fig. 2 (b), negatively charged membranes facilitate the passage of cations, whereas positively charged membranes permit the passage of anions. Moreover, this setup facilitates the differentiation between monovalent ions and multivalent ions; for instance, monovalent cations can permeate positively charged membranes, while multivalent cations are repelled [163]. This mechanism is grounded in the principles of Donnan equilibrium, which underpin the functionality of common ion exchange membranes [163].

A commonly employed approach to impart electrostatic properties involves surface modification, where a charged layer is applied to the surface of porous polymeric membranes. For instance, various substances containing amine groups, such as polyethyleneimine and piperazine, can be coated onto the surface of porous polymeric membranes to selectively extract Li from brines with a high Mg/Li ratio by inducing a positively charged layer [164–167]. In electrically driven membrane processes, it is imperative to minimize the thickness of these charged layers to reduce electrical resistance and enhance conductivity [168]. Although electrostatic repulsion facilitates the effective separation of monovalent ions from multivalent ones, achieving the targeted separation and extraction of ions with similar valence, such as Li^+ and Na^+ ions, remains challenging [25,169].

3.1.3. *Chemical affinity*

Selective extraction of targeted ions relies on their chemical affinity for distinct functional groups present in specific substances. Ions with a stronger ability to chemically bond with these functional groups are preferentially permitted to permeate through membranes. Conversely, ions with a lower affinity for such interactions are either impeded in their transport across the membrane or retained on the membrane surface in electrically driven membrane processes (Fig. 2 **(c)**) [170]. The extent of chemical affinity, quantified by parameters like binding energy, serves as a criterion for evaluating the feasibility of separating targeted ions from a mixture of ionic species. Various studies utilize computational methods, including density functional theory (DFT) calculations, to identify suitable functional groups or elucidate the interaction mechanisms between ion species and the functional groups of potential substances [171–174].

Liquid membrane stands out as a separation method that utilizes chemical affinity for metal ion separation. Specific substances are positioned between cation exchange membranes or physically supported within the pores of porous polymeric membranes (i.e., supported liquid membrane (SLM)) to enable selective ion transport [175–177]. Within the liquid membrane, metal ions establish chemical or physical bonds with the functional groups of these substances. Subsequently, the metal ions are dissociated from the functional groups through an exchange process with protons present in the stripping solution (Fig. 2 **(d)**) [178,179].

A diverse array of chelating agents, including various ligands, has been investigated for their effectiveness in metal ion separation within liquid membrane systems [176,179,180]. However, liquid membranes present inherent challenges, notably their limited stability and the acidic conditions required in the stripping solution, typically involving hydrochloric acid. These conditions can result in the emission of chlorine gas during the electrolysis process, posing a significant obstacle to the environmental and operational safety of liquid membrane systems [181,182].

Recent advancements in the application of chemical affinity focus on integrating substances with specific functional groups into the polymer matrix, either physically or chemically, for targeted ion separation [183]. This has led to the development of PIMs and molecularly imprinted polymers (MIPs), offering enhanced stability and selectivity in metal ion separation, thereby addressing the limitations of traditional liquid membrane approaches [183,184].

3.2. *Principles of electrically driven membrane technologies*

3.2.1. *ED*

ED is extensively employed in desalination, wastewater treatment, and, more recently, in the recovery of critical minerals [123–125]. In an ED system, an electric field is applied, causing cations to migrate towards negatively charged electrodes (cathodes), while anions move towards positively charged electrodes (anodes) (Fig. 2 **(e)**). Typically, an ED process consists of multiple stacks, where cation and anion exchange membranes are arranged alternately. This arrangement facilitates the selective concentration of cations through cation exchange membranes and likewise for anions through anion exchange membranes [123]. However, the conventional application of ED systems has faced challenges in selectively extracting specific metal ions. To harness ED effectively for the separation or recovery of critical minerals, it is imperative to integrate membranes capable of precise ion separation into the ED stacks [126,185].

Crucially, the selection of membranes should account for both selectivity and electrical characteristics, as the electrical resistance of membranes directly impacts the overall electrical resistance of the ED system and consequently, the operational efficiency requiring the number of stacks [123]. Recent research endeavors have focused on assessing the compatibility of various membranes within ED setups and evaluating their performance concerning ion separation efficiency [167,186].

3.2.2. *Recent advances in ED techniques*

The application of ED has expanded through strategic modifications tailored to specific objectives, with the functionality and potential uses determined by the properties and selection of membranes within the system. SED facilitates the separation of monovalent from divalent ions [124]. This separation is achieved by integrating one or more selective membranes into the conventional ED stack, positioned between the anion and cation exchange membranes (Fig. 2 **(f)**) [124].

BMED incorporates a bipolar membrane [187,188] into ED. BMED is used for extracting ions from aqueous solutions and recovering them into their respective acids and bases [189]. Unlike traditional IEMs, the primary function of a BPM is to induce a disproportionation reaction where water is electrochemically dissociated into protons (H^+) and hydroxide ions (OH^-) at the bipolar junction, notably without generating gaseous byproducts [38,190].

When an electrical current is applied across a BPM, the ionic current conduction is driven exclusively by H^+ and OH^- generated by the electrochemical dissociation of water at the membrane interface. This mechanism results in the generation of acidic and basic solutions on opposite sides of the membrane, effectively creating a pH gradient across the BPM [38]. As illustrated in Fig. 2(g), free cations are separated through a cation exchange membrane (CEM) and subsequently recovered in the base compartment. Conversely, chelated cations, which form anions in the feed solution, are separated through an anion exchange membrane (AEM) and then recovered in the acid compartment [189]. Based on the electro-dissociation mechanisms in BPMs, extensive research has been conducted to explore industrial applications, including pH adjustment processes, resource recovery from waste streams, and chemical synthesis [38].

Incorporating ED technology with liquid membranes enables a synergistic combination of carrier-mediated transport and electrical forces, significantly enhancing the efficiency of metal ion extraction [181,191,192]. This integration involves the use of selective ionic liquids (ILs) like tributyl phosphate (TBP), Di(2-ethylhexyl) phosphoric acid (D2EHPA), and Tricaprylmethylammonium chloride (Aliquat 336), enhancing the membrane's chemical affinity and thereby improving selectivity for specific metals. The electric field gradient acts as the primary driving force in ED, expediting ion movement across liquid membranes and boosting metal recovery efficiency [193]. Several studies, including early ones by Purin et al., have focused on applying this method to refine metals such as rhenium (Re) from industrial solutions [194]. Subsequent research expanded to extract and separate various metals like Ni, Cu, and cadmium (Cd) [195].

LMED provides distinct advantages, including high selectivity and efficient simultaneous extraction and stripping of metals. When targeting metals with similar physicochemical properties, such as ionic radii and charge, LMED could provide new opportunities for separating challenging metal mixtures (Fig. 2 **(h)**).

CAED is a novel approach that effectively combines ED technologies and chelating agents [40,196,197]. Since improving selectivity in membranes presents several challenges, the CAED technology suggests the addition of metal selectivity via the formation of stable anion or cation metal complexes. By utilizing the inherent stability constant of various metals, controlling the amount of chelating agents could produce selective formation of metal complexes, obtaining new physicochemical properties (e.g., complex size, complex charge, mobility, etc.) [198]. For example, various aminocarboxylic acids (such as ethylenediamine tetraacetic acid (EDTA), nitrilotriacetic acid (NTA), etc.) preferentially form stable anion complexes with several transition metals and REEs [199]. These anion complexes could transport through AEMs and be separated from other cation metals. As an equivalent amount of chelating agents to the target metal is sufficient to form complexes, this approach might be beneficial for low concentrations of critical minerals separation (Fig. 2 **(i)**).

4. Advanced ED technologies for separation and recovery of critical minerals

ED-based processes have shown significant success in separating valuable minerals from brine, waste streams, and the leachate of hydrometallurgical processes, crucial for various industries. While ED excels in separating cations and anions, it encounters challenges when dealing with diverse ionic compositions with different or identical valences in leachates and brines. Additionally, the performance of ED is heavily influenced by the perm-selectivity of the membranes, and conventional IEMs often lack the necessary selectivity towards critical metal ions. Consequently, the conventional ED process struggles to achieve high selectivity for critical minerals when separating them from competing ions in these complex solutions.

For instance, Li et al. investigated the effectiveness of ED in separating scandium (Sc) from synthetic wastewater, both with and without impurities including Fe, Al, K, Na, Ca, Mg, and Zn [42]. They achieved Sc removal rates of 65.3 % without impurities and 95.5 % under optimum conditions. However, in the presence of impurities, conventional ED lacked the necessary selectivity to remove Sc from these impurities, as high concentrations of Al (76 %) and Fe (63 %) were also detected in the product stream alongside Sc. To address these challenges, recent developments in ED technologies have focused on integrating ED with selective IEMs, BPMs, liquid membranes, chelating agents, and hybridization with other separation processes, each tailored to specific needs. These recent advancements will be discussed in more detail in this section.

4.1. SED

Efficient separation and recovery of critical minerals through ED can be significantly enhanced by developing selective IEMs. Numerous studies have demonstrated the potential of selective IEMs to effectively and economically address the challenges associated with ion competitions in brines and leachates. SED has been employed to separate and recover critical minerals, particularly Li^+ ion, by utilizing monovalent anion exchange membranes (MAEMs) and monovalent cation exchange membranes (MCEMs) instead of conventional IEMs [77].

In this process, when a potential difference is applied, monovalent ions such as Cl^-, Li^+, Na^+, and K^+ selectively migrate through MAEMs and MCEMs. Divalent ions (e.g., SO_4^{2-}, Mg^{2+}, and Ca^{2+}) are efficiently rejected by these membranes, leading to increased concentrations of monovalent ions in the concentrate chamber and decreased concentration in the dilute chamber.

In the context of Li separation, monovalent selective membranes can overcome the challenge of properly separating Mg^{2+} and Li^+ ions, which have similar ionic radii, enabling efficient separation in systems with a high Mg/Li ratio. The functionality of these membranes relies on charge rejection and the creation of a crosslinked layer to hinder the transport of divalent ions [200]. Examples of commercially available monovalent-selective membranes, such as NEOSEPTA CIMS (ASTOM) and Selemion CSO (AGC), have been utilized in ED systems for efficient Li recovery [201].

Designing and fabricating MCEMs and MAEMs with high permselectivity are crucial for the SED process. These enhancements stem from separation mechanisms like partial dehydration, pore-size sieving, and electrostatic repulsion [202]. In terms of partial dehydration, Pang et al. developed a surface-modified MCEM through in-situ polymerization and polypyrrole (PPy) layer deposition, adjusting hydrophobicity by quarternizing with iodoalkane featuring various chain lengths [203]. They found that membrane surface resistance was due to the steric hindrance from the PPy layer rather than the charging effect of quaternization. Their studies showed that increasing surface hydrophobicity significantly enhanced Li/Mg selectivity (Li/Mg = 1.71) by over 50 % compared to the commercial MCEM (Neosepta CIMS) (Li/Mg = 1.07), likely due to a higher energy barrier for ion dehydration, highlighting the dehydration effect's importance in MIEMs separation efficiency (Fig. 3 **(a)**).

In addition to the hydrophobicity effect, electrostatic repulsion is another prominent separation factor that can enhance monovalent selectivity by coating positively charged layers or sites in the inner channels of the membranes. Ding et al. fabricated a novel design of Nafion membranes coated with multilayers of protonated poly (allylamine)/poly(4-styrenesulfonate) through layer-by-layer assembly (Fig. 3 **(b)**). This membrane showed 99.5 % monovalent separation efficiency when treated with similar molar concentrations of K, Mg, and Li. They also demonstrated that SED, with implementing these modified Nafion membranes, can achieve a high Li/Mg selectivity of 1000 and Li recovery of 65 % with 70 % current efficiency. The effect of polyelectrolyte layers was demonstrated by enhancing the selectivity when a higher polyelectrolyte concentration was used during membrane-coating deposition [204].

To enhance the feasibility of SED for larger-scale operation, a continuous mode was developed with the batch mode. While the batch mode suits smaller setups with its high separation efficiency and lower energy consumption at a constant voltage, it may encounter limiting current density effects. This issue prompted the development of the continuous mode, enabling higher current densities and improved Li^+ ion separation with higher purity in a more economical manner compared to the batch mode [205].

Additionally, Xu et al. introduced a unique "ion distillation" technique through electro-membrane stacking for Li extraction from brine, achieving an unprecedented selectivity of 26,177 between Li and Mg in a four-stage process [201]. This technique, enhancing Li selectivity via an electro-stripping mechanism, resulted in LiCl product purity exceeding 99.95 %, surpassing battery-grade standards and providing an efficient method for ion separation with high selectivity.

The studies towards selective IEMs are not limited to Li recoveries from brines, and several investigations have been conducted to separate Co from waste batteries by designing new IEMs. Siekierka et al. focused on Co-selective cation exchange membranes coupled with ED systems, aiming for the separation of Co from LIBs [206–208]. This group engineered Co-selective IEMs modified with 5-chloro-8-hydroxyquinoline (5C8Q). The 5C8Q modified membrane created selective binding sites that complex with Co, enabling separation from Ni and Li due to the enhanced ionic diffusion through preferential complexation (Fig. 4 **(a)**).

The 5C8Q modified membrane can be obtained using a two-stage chemical grafting method, starting with the hydrolysis of the polyacrylonitrile (PAN) surface followed by the incorporation of 5-chloro-8-hydroxyquinoline (5C8Q) [208]. The performance of the PAN-5C8Q membrane in ED experiments exhibited promising selectivity, achieving a Co removal efficiency of 91 % and Co/Li and Co/Ni separation factors of 5.6 and 16.1, respectively, within just 3 h under a constant voltage of 5 V. Mechanistic investigations using Nernst-Plank modeling revealed that Co form aqua-complexes with

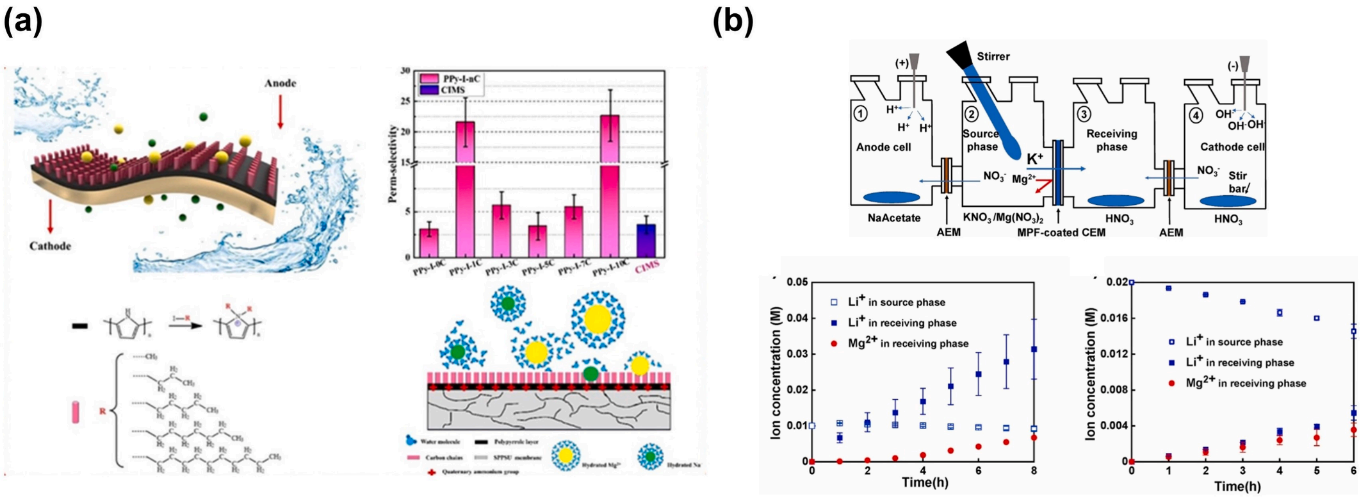

Fig. 3. (a) Permeable membranes selectively designed for monovalent cation separation, targeting Mg^{2+}/Na^{+} and Mg^{2+}/Li^{+} ions, and (b) Nafion membranes enhanced with polyelectrolyte multilayers for effective separation and selectivity of Mg^{2+}/K^{+} and Mg^{2+}/Li^{+} ions. [(a) Reproduced with permission from Ref. [203], Copyright 2021, Elsevier. (b) Reproduced with permission from Ref. [204], Copyright 2022, Elsevier.].

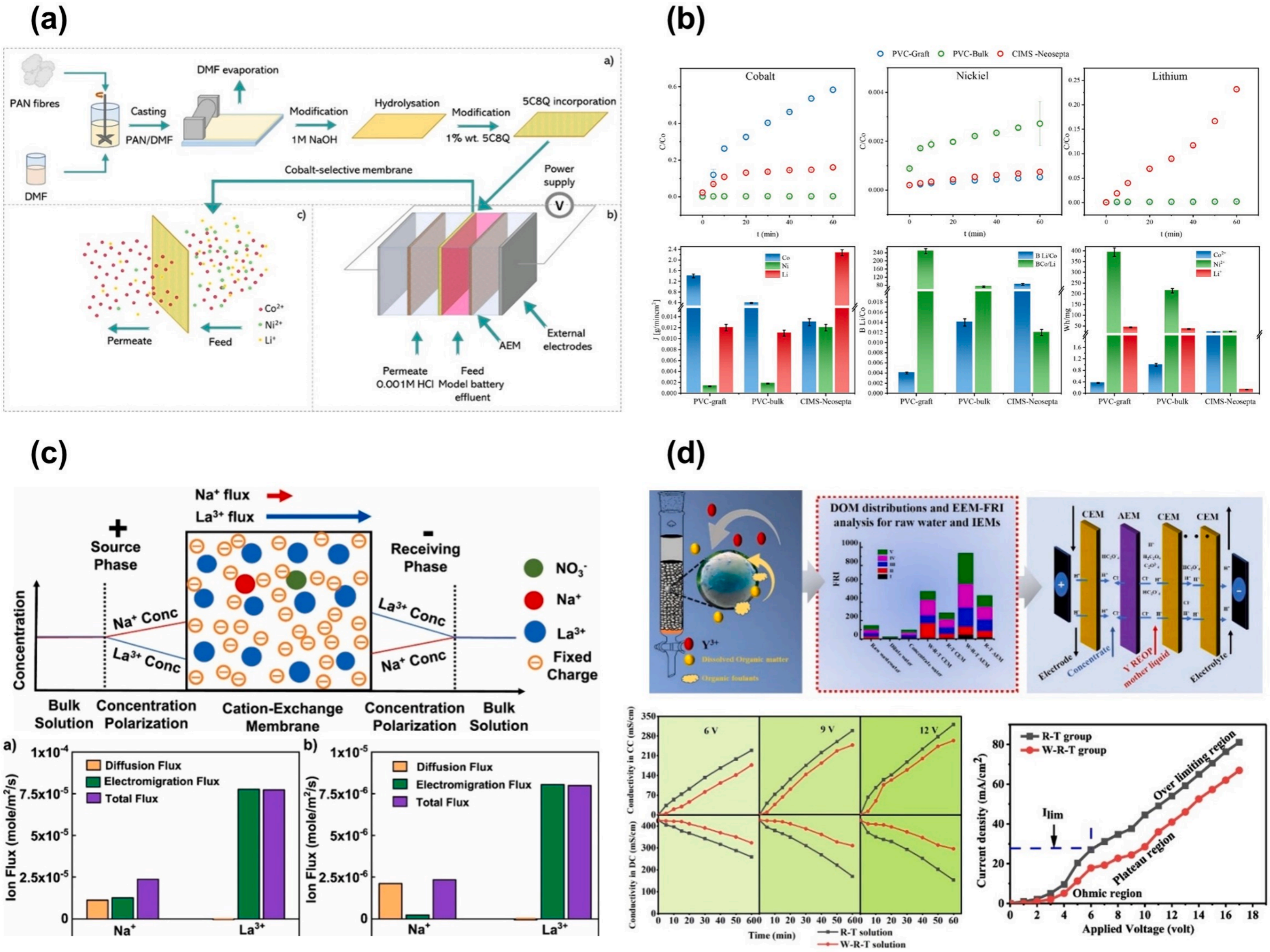

Fig. 4. (a) PAN-5C8Q membranes for the separation of Co from waste LIBs, (b) PVC-EDA-5C8Q membranes for the separation of Co from waste LIBs, (c) schematic diagram of Nafion-based SED for the separation of Na^{+}/La^{3+} ions and the fluxes of metal species in ED system, and (d) ion-exchange process combined with SED for the recovery of Y and HCl. [(a) Reproduced with permission from Ref. [208], Copyright 2022, Elsevier. (b) Reproduced with permission from Ref. [206], Copyright 2023, Elsevier. (c) Reproduced with permission from Ref. [209], Copyright 2023, Elsevier. (d) Reproduced with permission from Ref. [211], Copyright 2023, Elsevier.].

hydroxyquinoline on the membrane surfaces, facilitating their selective transport. This selective Co transport was driven by an electro-diffusive process, stemming from a moderately specific interaction between the chelating agent and Co. Alternatively, the Co-selective membrane can be obtained using poly(vinyl chloride) (PVC) with ethylene diamine (EDA) and 5C8Q chelating agents [206].

The membranes were synthesized through surface and bulk grafting methods, benchmarked against commercial CIMS Neosepta membranes. In the ED system, the surface-grafted membrane demonstrated superior performance, successfully recovering up to 58 % in multi-component solutions, with Co/Li separation factors as high as 247. Moreover, the surface-grafted membranes exhibited the lowest energy of sorption compared to bulk membranes, being ten times lower than commercial CIMS Neosepta reference membranes (Fig. 4 **(b)**).

In the case of REEs, the larger ionic radii of REEs relative to more prevalent metals (i.e., Li, Na, K, Ca, and Mg) present a challenge in designing SED systems. Ding et al. developed an innovative approach to separate REEs in the SED system by controlling the flux of metal ions or integrating with other processes [209]. This group focused on trivalent La^{3+} ion separation from Na^+ or Mg^{2+} ions using a commercial CEM, Nafion N117.

The selectivity of the ED system was influenced by the densities of fixed charges and ion diffusion coefficients. The diffusion coefficient of Na is 15 times higher than that of La, which is unfavorable for La selective penetration [210]. Nevertheless, the presence of trivalent ions rather than monovalent ions can decrease the Donnan potential, suggesting that La^{3+} may become the compensatory ion for the fixed charges in Nafion. It utilized the Donnan partitioning of metal species across CEM, which allows ions into the membrane to reduce the Donan potential between the feed solution and the membrane. Through simulating the transport of La^{3+} and Na^+ ions, the Donnan selectivity was calculated as 6 ± 1 within 0.2 M equimolar $NaNO_3$ and $LaNO_3$ solution.

Remarkably, the calculated Donnan selectivity appeared to escalate at reduced ionic strengths, achieving an estimated 170 ± 30 in a 0.001 M solution. Despite the negligible diffusion flux of La, the superiority of La electromigration flux over that of Na played a pivotal role in enhancing selectivity. Interestingly, the selectivity in actual ED experiments represented 4.7 ± 1.0 in 0.001 M equimolar $NaNO_3$ and $LaNO_3$ solution within a 0.8 mA/cm^2 condition, which implies that other factors such as ionic mobility inside the membrane also control metal selectivity. Collectively, this study demonstrated the selective transport of La in the ED system, especially effective in low ionic strength (Fig. 4 **(c)**).

Unconventional sources or industrial waste leachates often contain precipitates, contaminants, and dissolved organic matter (DOM) in the feed phase, which can lead to membrane fouling. Zhou et al. proposed a novel process integrating ion exchange resin and SED for the recovery of low-concentration REEs and the separation of HCl, significantly reducing membrane fouling [211]. Ion exchange resins (CH-93 and D001) were applied to the Y rare-earth oxalic precipitation (Y REOP) prior to SED, absorbing over 94 % of Y. After REEs recovery and contaminants removal, the resin-treated (R-T) and untreated (W-R-T) solutions were employed in the SED chamber for the recovery of HCl from the mother liquors.

The R-T solution demonstrated a 68.91 % HCl recovery efficiency at 12 V, significantly higher than the 47.9 % efficiency of the untreated solution, also achieving more than 99 % HCl purity. The R-T solution showed higher current density and lower energy consumption across a range of voltages (0–18 V) compared to the W-R-T solution. This enhanced performance was attributed to the resin ability to remove contaminants from the Y REOP, thus effectively preventing membrane fouling. This study suggests the advantage of integrating ED with other metal recovery pretreatment, which could improve the ED efficiency (Fig. 4 **(d)**).

In summary, SED technology is advancing by enhancing membrane functionalities such as size exclusion, electrostatic repulsion, and chemical affinity to improve the selective separation of critical minerals. SED is effective for the separation of monovalent, divalent, and trivalent ions. For instance, SED can effectively separate monovalent Li^+ ions from divalent ions (e.g., Ca^{2+} and Mg^{2+}) in Li brine, or separate trivalent REEs^{3+} ions from monovalent Na^+ ions in fly ash leachate. Additionally, membranes with high selectivity for specific metals like Co can facilitate the separation of Ni and Co from waste batteries. Given the complex mixtures of metals in unconventional sources, developing highly selective membranes can significantly broaden the applicability of SED technology to various feedstocks.

4.2. BMED

BMED, an innovative integration of BPM and IEMs within an ED stack, represents a significant advancement in membrane processes. This pioneering design allows the BMED process to utilize a direct current electric field with a potential higher than the electrolysis of water (1.2 V), leading to the separation of water molecules in the hydrophilic layer into H^+ and OH^-, a characteristic unique to bipolar membranes [188]. BMED technology offers several remarkable benefits, including high separation efficiency, exceptional perm-selectivity, and low electrical resistance. Moreover, it exhibits impressive chemical and mechanical stability, minimal voltage drops, and superior current efficiency [77].

These advantages underscore the potential of BMED to significantly enhance processing capacities by incorporating membranes while reducing the usage of lime and other chemical substances. Additionally, BMED is a highly versatile technology capable of effectively separating aqueous solutions into distinct acidic and basic streams. Furthermore, it can recover critical minerals from brines and waste streams. The dual functionality of BMED, enabling critical minerals separation and simultaneous production of useful acids and alkalis, positions it as a promising and eco-friendly solution for various industrial applications.

The feasibility and implementation of BMED for critical mineral recovery were initially investigated through systematic studies on Li separation from various sources, including spent LIBs, brines, and simulated geothermal brine solutions containing both boron (B) and Li. One such systematic study was conducted by İpekçi et al., demonstrating the effectiveness of BMED in separating coexisting B and Li [126,212]. They examined various parameters, such as the types of acid and base solutions, the effects of B and Li concentrations, coexisting ions, and pH levels. Their findings illustrated that BMED can operate efficiently with high separation recovery and efficiency at reasonably low operating voltages and current densities, resulting in modest specific power consumption.

Further investigation into the efficiency of B and Li separation was conducted by Kabay et al. [213]. They implemented two different ED stacks (ED-1 and ED-2) with different IEMs and BPMs, evaluating the removal and separation recoveries at voltages of 15 and 20 V and varying concentrations of boric acid (H_3BO_3) and LiOH. The ED-2 stack exhibited the best performance, achieving over 99 % of Li removal and 72 % of B removal, with the highest Li recovery of 64 % achieved at 20 V when using 0.05 mol/L of H_3BO_3 in the acid chamber and 0.05 mol/L of LiOH in the base chamber.

In addition to geothermal brine containing B and Li, various alternative sources have been explored for Li recovery. Wei et al. emphasized the effectiveness of BMED in converting low-value lithium phosphate (Li_3PO_4) into high-value LiOH and phosphoric acid (H_3PO_4) [214]. They investigated the solubility of Li_3PO_4 in H_3PO_4 and its role in the salt chamber, facilitating the transfer of Li^+ and PO_4^{3-} to the acid and base chambers through IEMs (Fig. 5 **(a)**).

Through the optimization of current density and phosphate concentration, they achieved a remarkable Li recovery rate of up to 99 %. Additionally, the process was fine-tuned to an energy consumption of 10.54 kWh/kg and an estimated cost of $2.941 per kg of LiOH at a current density of 20 mA/cm^2, thus confirming BMED as an economical, efficient, and environmentally sustainable method for processing

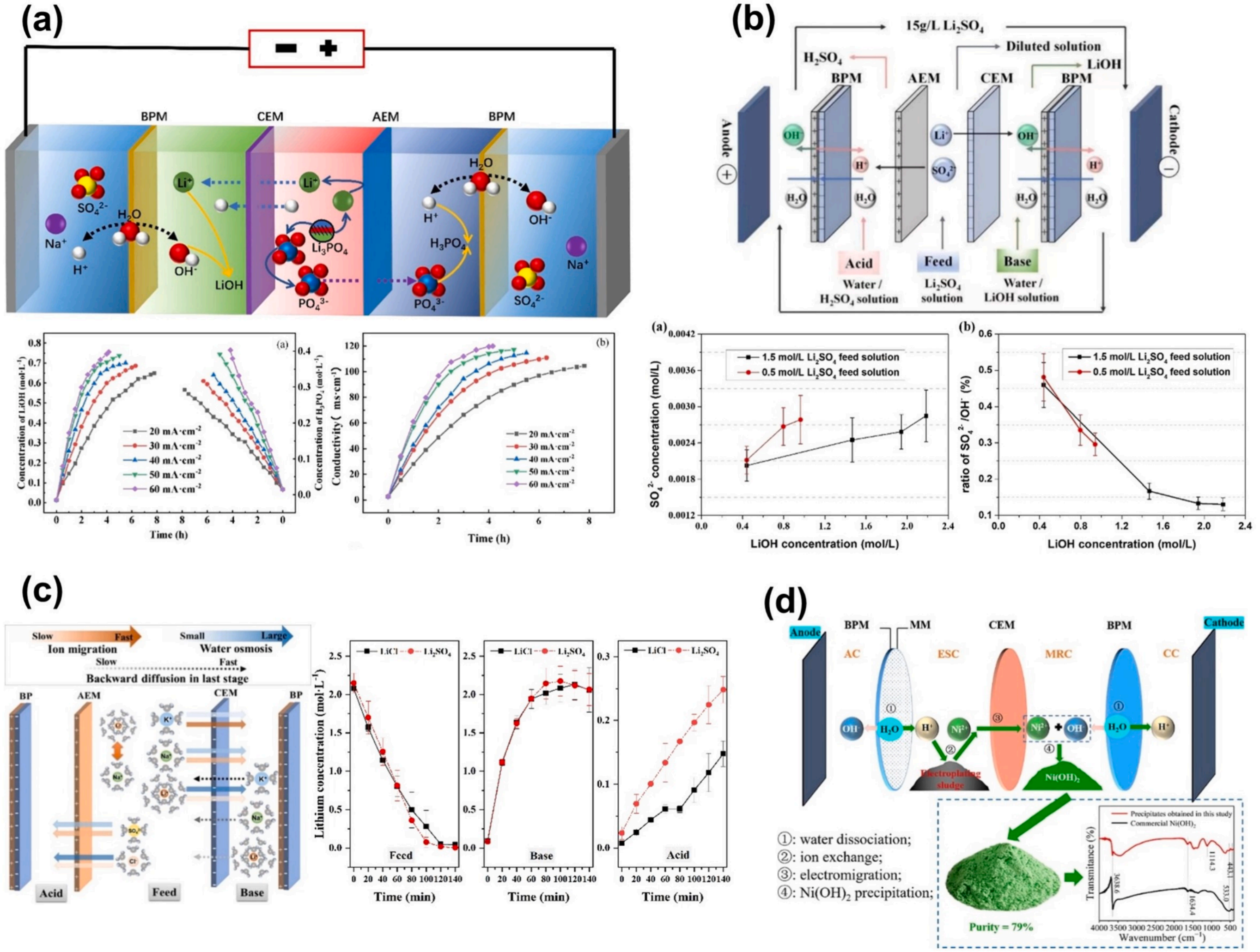

Fig. 5. (a) Schematic diagram of the BMED to produce LiOH from Li_3PO_4, (b) systematic representation of BMED process for generating LiOH from Li_2SO_4, (c) impact of concurrent ions and competition between Na^+ and Li^+ ions for preparation of LiOH with Li_2SO_4 as the feed solution using BMED process, and (d) schematic illustration for efficient $Ni(OH)_2$ production and Ni recovery from electroplating sludge utilizing BMED process. [(a) Reproduced with permission from Ref. [214], Copyright 2022, Elsevier. (b) Reproduced with permission from Ref. [215], Copyright 2021, Elsevier. (c) Reproduced with permission from Ref. [216], Copyright 2021, Elsevier. (d) Reproduced with permission from Ref. [54], Copyright 2023, Elsevier.].

Li_3PO_4.

Lithium sulfate (Li_2SO_4), an intermediate in conventional Li extraction processes, serves as another significant Li recovery source. The feasibility of BMED for producing valuable LiOH and H_2SO_4 from Li_2SO_4 was explored, with Li^+ and SO_4^{2-} ions transferring through CEM and AEM to the base and acid chambers, respectively [215]. This process utilizes Li_2SO_4 as the feed and electrode rinse solutions flowing through IEMs and BP membranes (Fig. 5 **(b)**).

The study evaluated two homogeneous (TRJBM and Neosepta BP-1E) and one heterogeneous BP membrane (EU-M) to assess the effect of BP membranes on preventing co-ion diffusion, which could lead to contamination and reduced current efficiency. The BP-1E membrane effectively blocked SO_4^{2-} ion leakage, while the TRJBM allowed most SO_4^{2-} ion to diffuse through the membrane. The heterogeneous EU-M BP membrane resulted in the highest applied voltage and the lowest LiOH production rate compared to homogeneous ones. Optimizing the feed conditions using the BP-1E membrane could result in obtaining LiOH purity of over 99 % with low energy consumption of 7 kWh/kg LiOH. This demonstrates the potential of BMED as an efficient and economical method for extracting LiOH and Li_2SO_4.

Another study by Zhao et al. assessed the efficiency of BMED in generating LiOH from various Li-containing solutions, as shown in Fig. 5 **(c)** [216]. They emphasized that the feasibility of BMED can be significantly influenced by the ion characteristics and composition of the feed solution. Specifically, cations with higher hydration numbers may slow ion migration and increase water flux, affecting the mass transfer from the feed to the base compartment. On the other hand, anions with higher valence could lead to increased Li leakage into the acid compartment due to their slower migration rates.

The presence of Na^+ and K^+ ions in the feed solution was found to decrease Li migration and the concentration of Li in the base solution, with K having a more pronounced effect on increasing the energy consumption for LiOH production. The ratio of Na to Li in the feed solution was identified as crucial since a higher ratio resulted in decreased Li migration and increased energy consumption due to competitive migration and backward diffusion of Na.

Moreover, the properties of the CEM, such as ion exchange capacity and resistivity, were highlighted as critical factors affecting Li migration, cation backward diffusion, and overall BMED process efficiency. Optimal CEM characteristics could lead to higher current efficiency and lower energy consumption, underscoring the interconnectedness of ion characteristics, feed solution composition, and membrane properties in optimizing BMED for Li recovery.

Recently, Liu et al. introduced a novel BMED-based process aimed at recovering Ni in the form of nickel hydroxide ($Ni(OH)_2$) from electroplating waste, as illustrated in Fig. 5 **(d)** [54]. In this process, the proton generated in BMED was utilized to lower the pH of the feed phase, facilitating the extraction of Ni from electroplating waste. The extracted Ni^+ ions were then transferred to the metal recovery compartment where they reacted with OH^- to form stable $Ni(OH)_2$. For electroplating sludge, the pH of the feed phase was reduced to below 1.0 under a current density of 20 mA/cm^2, resulting in a remarkable 93.5 % recovery of Ni after 28 h. Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy analyses confirmed that the precipitate obtained in the study closely resembled commercial $Ni(OH)_2$, exhibiting a purity of 79 %.

Similarly, a comparable approach was applied to electroplating wastewater containing Ni-EDTA, which is commonly used to achieve uniform plating [114]. The proton generated during BMED reduced the pH of the feed phase, causing Ni-EDTA to dissociate into Ni^{2+} ions. These dissociated Ni^{2+} ions were then transferred to the metal recovery compartment and recovered as $Ni(OH)_2$. This experiment demonstrated a high Ni-EDTA removal rate of 97.1 % and a Ni recovery rate of 95.2 % for actual plating wastewater under a current density of 16 mA/cm^2 after 32 h. The recovered $Ni(OH)_2$ from actual wastewater exhibited a purity of 75.2 %.

In summary, BMED technology enables the simultaneous recovery of metals and the reuse of solvents (such as acids or bases), as well as the reclamation of specific compounds like H_3PO_4 and EDTA. This process involves the transport of metals, H^+, OH^-, and ligands through the BME to the cathode or anode. When applied to Li-containing feedstocks, BMED can recover Li as LiOH while producing H_2SO_4 or H_3PO_4. For Ni-containing feedstocks, BMED allows for the recovery of Ni as $Ni(OH)_2$ while simultaneously reclaiming acid. Additionally, the extraction of metals from solid-phase feedstocks typically involves an acid-consuming leaching step that requires a considerable amount of protons. The advantage of BMED technology lies in its ability to separate and recover metals while recycling acids or chemical compounds, thereby enhancing the efficiency and sustainability of the metal refining process.

4.3. LMED

LMED represents a relatively recent advancement that utilizes the distinctive attributes of various liquid membranes, including SLM and PIMs, in conjunction with ED, to accomplish targeted ion separation. Its effective utilization in the extraction and retrieval of diverse metal ions has opened avenues for scientists to investigate its key variables in the critical mineral recovery domain, with a particular emphasis on Li.

For instance, the successful utilization of PIMs in conjunction with ED for Cr (VI) separation, demonstrating enhanced ion permeability and operation at low voltage [39,217,218], served as inspiration for Wang et al. [192] to explore LMED for the recovery of Li and Co from spent LIBs. They developed a novel PIM using poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) as the base polymer and incorporating four different ILs (Aliquat 336, Cyphos IL101, and TOA) through a solvent evaporation casting technique.

By situating the PIMs between commercial CEM and IEM and introducing NH_4SCN into the feed solution, they enhanced the selectivity for Co over Li by forming a $Co(SCN)_4^{2-}$ complex. Among the ILs tested, Aliquat 336 exhibited superior performance in terms of extraction degree and product purities for Li and Co due to its lower area resistance, larger effective area, and hydrophilic properties, which facilitated metal ion diffusion.

They investigated the primary mechanism of Co and Li transfer across PIM in the PMED process, uncovering that the PIMs structure domain, with fixed-site jumping, provides a continuous ion transport pathway even at low current densities. The optimized membrane achieved over 98.5 % purity for Li^+ and 99.4 % for Co^{2+} ions from real spent LIBs leachate at a limited voltage of 3.9 V.

Scaling up the process with two ED stacks doubled the initial flux of Co (288 $\mu mol/m^2$), while achieving 97 % stripping degree and over 99 % purity. Their economic analysis yielded promising results in specific energy consumption (4.4 kWh/kg) and GHG emissions (3.1 kg-CO_2/kg), comparing favorably with other electro-driven membrane processes [192] (Fig. 6 **(a)**).

In a recent study, Cui et al. investigated the feasibility of using LMED for Li separation from solid wastes, particularly focusing on Li extraction from simulated coal fly ash leachate [182]. They developed an SLM utilizing TBP as the Li^+ ion carrier and integrated it with three different ILs: [C_2mim][NTf_2], [C_4mim][NTf_2], and [HOEmim][NTf_2]. These ILs have varying conductivities that can directly affect the separation performance. The LMED configuration involved placing the liquid membrane between two CEMs (Fig. 6 **(b)**).

The feed solution, composed of various inorganic ions such as Li^+, Mg^{2+}, Ca^{2+}, and Al^{3+} dissolved in deionized water, was designed to mimic the composition of coal fly ash. Since Al was the predominant cation in coal fly ash, the study evaluated the performance across different Al/Li ratios. Additionally, three commercial CEMs (i.e., CIMS, Grion0011, and GCCM-KS) were tested with the liquid membrane to identify the most effective combination.

Cui et al. demonstrated that the extraction using TBP in addition to ILs primarily involves cation exchange process, where a more hydrophilic cation enhances Li separation [182]. Their findings revealed that a 5:5 (v/v) combination of [HOEmim][NTf_2], which has a more hydrophilic cation compared to the other two ILs, with the TBP system as the optimum SLM showed superior performance in releasing Li under various stripping solution acidities. The introduction of ILs led to an improved voltage response of the liquid membrane under an electric field, thereby enhancing Li separation. LMED demonstrated the capability to achieve a separation factor of up to 200 for an Al/Li ratio of 100 at an optimal voltage of 3 V.

In summary, LMED technology exploits the chemical affinity of membranes to enhance selectivity for difficult-to-separate metals. The well-established database of metal selectivity for solvent extractants and ILs enable the precise selection of solvents tailored for targeted metal recovery in liquid membrane fabrication. Consequently, membranes could be designed to achieve high selectivity for specific critical minerals within the complex metal matrices found in unconventional sources. For example, solvents such as Aliquat 336 and Cyphos IL 101, which are effective in separating Co from waste batteries, can be employed for membrane fabrication. However, issues such as solvent loss from the membrane, which reduces recyclability, and the relatively high membrane resistance compared to commercial membranes, present challenges to the efficiency of ED systems. Therefore, further technological advancements are necessary to address these limitations.

4.4. CAED

CAED is getting attention to enhance the selectivity of critical minerals from complex solutions. As previously mentioned, one of the main challenges in ED, especially for REE separation, lies in the low selectivity of IEMs towards ions with identical valence or ions with different valences. This makes it challenging to differentiate between heavy and light REEs, as well as Li and Co in leachates from spent LIBs.

To overcome this challenge, chemicals such as EDTA, citric acid, malic acid, and lactic acid have been employed. These agents form complex anions with critical minerals, leading to the formation of complexes that are less likely to permeate through the IEMs [189,197,206]. This enables the effective separation of critical minerals from other ions with similar valences. The stability constant, K_{ABS}, plays a crucial role in this process by determining the equilibrium between chelated and ionized forms in the solution. Higher constants indicate a stronger affinity, resulting in more stable chelate formation [196].

Takahashi et al.'s work in utilizing CAED for REE separation is among the early and notable contributions to this field [198,219,220]. Their pioneering studies, which began in the early 1990 s, were

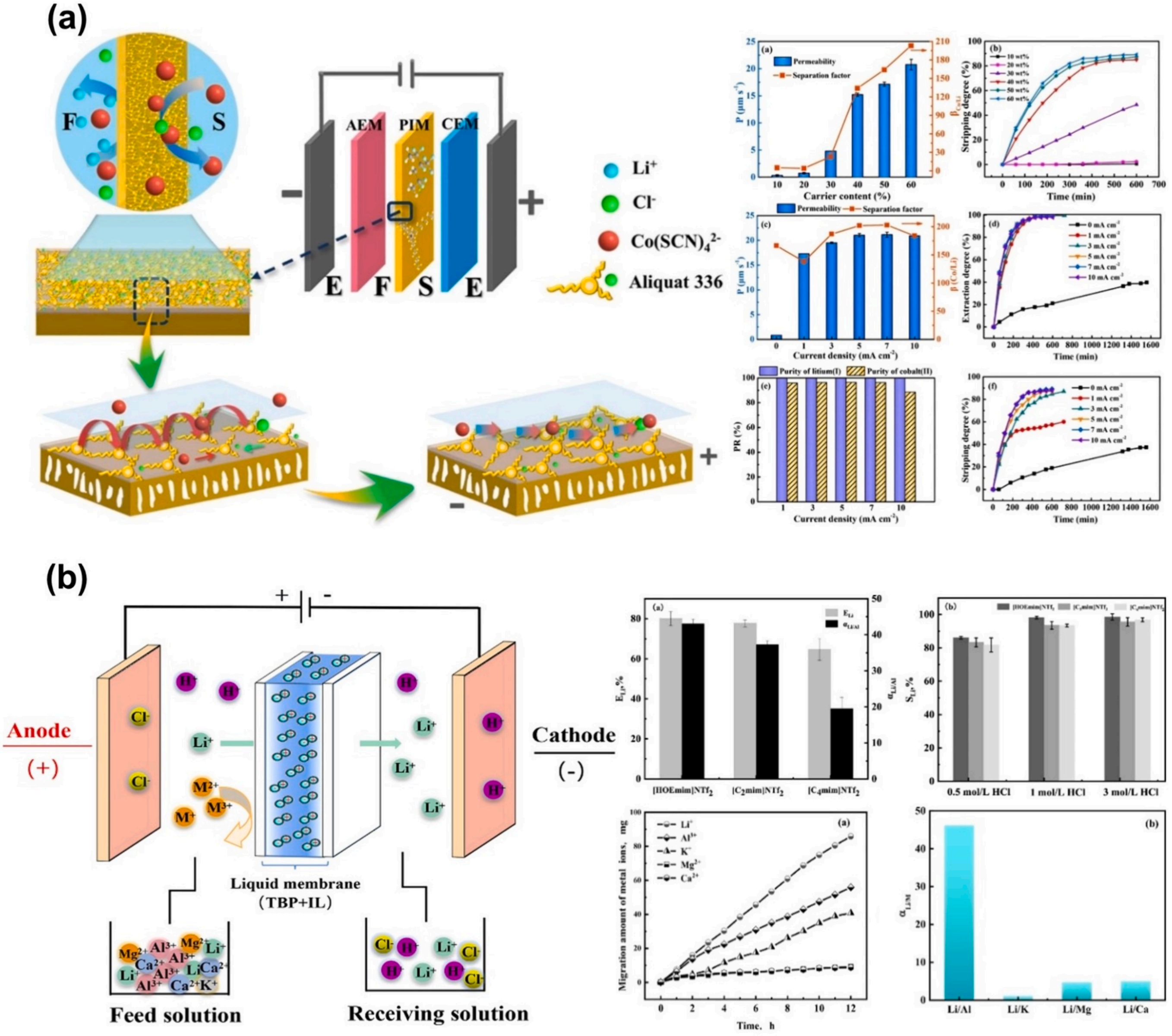

Fig. 6. (a) Illustration of the Co and Li separation in an ED stack employing the Aliquat 336 PIMs-ED system with F, S, E denoting feed, stripping, and electrode rinse solutions, respectively, and (b) general illustration of LMED for effective Li recovery with high Li/Al selectivity from fly ash acid leaching solution. [(a) Reproduced with permission from Ref. [192], Copyright 2022, Elsevier. (b) Reproduced with permission from Ref. [182], Copyright 2024, Elsevier.].

primarily focused on separating Ce from mixtures containing Ce-Gd-Y and Ce-La-Pr-Nd, employing ED with Na_2EDTA as the chelating agent. They achieved successful concentration of Ce or Ce-La in the concentrate compartments, attributing the efficacy of separation to the differential mobility of chelated versus non-chelated ions across the membrane. To support their experimental findings, they developed a model based on the Nernst-Planck equation, considering factors such as solution selectivity, membrane selectivity, and ion migration.

Expanding upon their initial work, Takahashi et al. further investigated the use of both Na_2EDTA and lactic acid to further enhance the separation efficiency, particularly focusing on La separation from Y, and Nd separation from Gd-Y mixtures. They found that increasing concentrations of EDTA in the solution significantly improved the separation factor of La and Y, achieving remarkable separation at specific EDTA concentrations and pH levels. Similarly, the separation factor of Nd relative to Gd and Y varied with the Nd concentration fraction in the solution, demonstrating the potential of ED to effectively concentrate Nd across a wide concentration range.

Recent studies focusing on Sc recovery using ED without chelating agents have revealed poor selectivity, with optimization mainly limited to Sc concentration. This highlights the difficulty in adjusting concentrations for waste streams containing REEs. Furthermore, uncertainties remained regarding the efficacy of chelating agents other than EDTA for REE separation. Considering these challenges, Mosadeghsedghi et al. sought to address gaps in REE separation research by incorporating various chelating agents at concentrations similar to those found in waste streams. They also aimed to optimize operational parameters and membrane configuration to enhance the separation process [197].

Their ED setup comprised five cell pairs of commercial CEMs and AEMs (CMX and AMX Neosepta®), and they employed four different aminocarboxylic acid chelating agents: EDTA, diaminocyclohexanetetraacetic acid (DCTA), N-(2-hydroxyethyl) ethylenediaminetriacetic acid (HEDTA), and diethylenetriamine-pentaacetic acid (DTPA) for separating light and medium REEs from binary and tertiary solutions. The feed solutions consisted of various REE chlorides and chelating agents in water, while 0.01 M HCl was used on the concentrate side, with

0.05 M HCl and H_2SO_4 for the cathode and anode sides, respectively.

Experimental results revealed a lack of selectivity in the absence of chelating agents, with HEDTA demonstrating the highest selectivity and a 10-fold increase in separation factor for La and Nd-Pr. The order of selectivity among the chelating agents was HEDTA>DCTA->EDTA>DTPA, with higher separation factors observed at increased voltages due to enhanced ionic mobility of non-chelated REEs. Through optimization of pH (3.0–4.5), electrolyte type, and cell configuration, they overcame precipitation challenges, achieving a separation factor of 42, surpassing those obtained from conventional solvent extraction processes (Fig. 7 **(a)**).

Ding et al. demonstrated the successful separation of heavy and light REEs using EDTA as a chelating agent [221]. They employed Na_2EDTA-assisted ED to separate Dy from Pr and Nd, two light REEs, based on the concept of K_{ABS} differences. This method relies on light REEs remaining as free cations while heavy REEs form a chelating complex ([Dy-EDTA]), facilitating efficient separation in the ED process. The feed solution consisted of sulfate salts of REEs and chelating agents, with pH adjustment for improved separation. They devised a specific system utilizing one CEM (CMX) and AEMs (CMX and PC-400D), with PC-400D targeting efficient separation of large organic molecules like [Dy-EDTA].

They investigated the effects of feed pH, electric potential, and rinse solution concentration with Na_2SO_4, noting that a higher rinse solution concentration is crucial for stabilizing charge neutrality and maintaining low resistance, albeit with minimal impact on separation efficiency. Higher voltages increased ion mobility and separation, with 12 and 14 V demonstrating similar separation efficiency, though 12 V was preferred to avoid excessive water electrolysis at higher voltages. The optimal pH range was identified as 3 to 5, with a 1:1 EDTA to Dy molar ratio yielding the best separation performance. At this similar molar ratio, the chelation effect was maximum for Dy and minimum for Nd and Pr, achieving a separation factor of 125, highlighting the enhanced selectivity between heavy and light REEs. Energy consumption was estimated at 45 GJ per ton of REEs separated, higher than solvent extraction processes but offset by reduced consumption of organic solvent. Further studies are necessary to lower energy consumption through alternative chelating agents, electrode rinse solutions, and more selective IEMs to enhance separation efficiency at lower voltages.

Another recent study by this group introduced the first comprehensive modeling based on the Nernst-Planck equation for the separation of

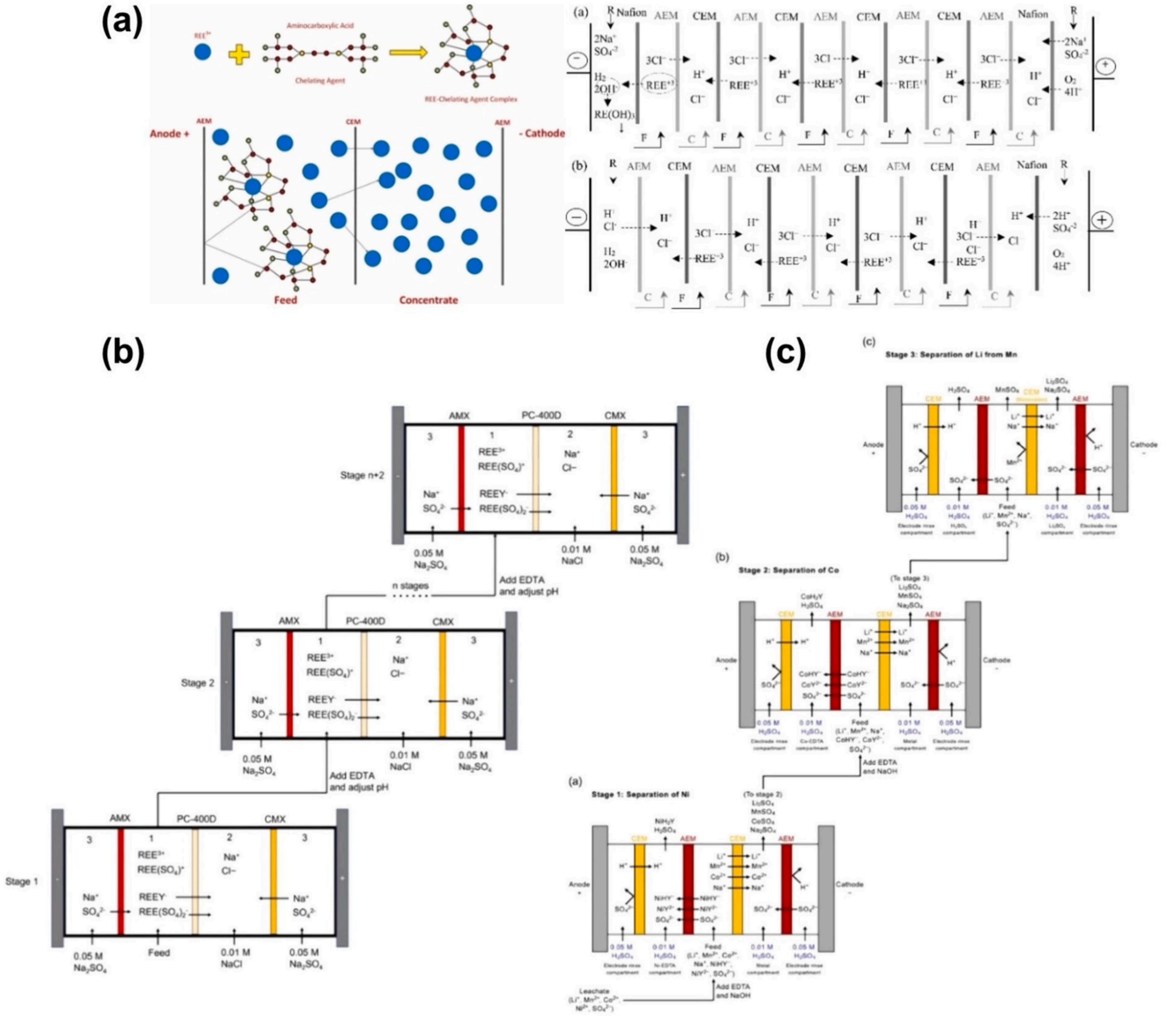

Fig. 7. (a) Systematic illustration and mechanism of aminocarboxylic acid chelated ED for efficient REEs separation/recovery, (b) representation of efficient three stage CAED process for selective Dy separation from REEs, and (c) general illustration of three-stage CAED method with feed compositions for the separation of Li, Ni, Mn, and Co from NMC111. [(a) Reproduced with permission from Ref. [197], Copyright 2023, Elsevier. (b) Reproduced with permission from Ref. [221], Copyright 2023, Elsevier. (c) Reproduced with permission from Ref. [40], Copyright 2022, Elsevier.].

Nd and Pr from mixtures with Ce and La using CAED and HEDTA as the chelating agent [196]. The model optimized the chelating agent concentration and pH for improved separation efficiency, advocating a multistage approach with HEDTA concentrations below the stoichiometric ratio. Predictions demonstrated up to 91.2 % purity at specified HEDTA/Nd ratios and pH levels, showcasing the models effectiveness in designing CAED experiments for REE separation. They also discussed that due to limited research in this area, successful implementation of this type of phenomenological model can provide insight into separation efficiency and purity in the feed and concentrate compartments, greatly benefiting REE separation processes.

Research on CAED expands beyond REE recovery, with successful applications in processing spent batteries. Iizuka et al. demonstrated a 99 % selectivity for Li and Co from mixed solutions simulating waste batteries using EDTA in a three-cell-type BMED process with two IEMs (Selemion CMV and Selemion AMV) and one BP membrane (BM-1E) [189].

Similarly, Afifah et al. coupled the SED process with EDTA to achieve a 99.4 % separation efficiency for Li and Co [222]. However, these studies primarily focused on Li and Co separation, leading Chan et al. to investigate the $LiNi_xMn_yCo_{1-x-y}O_2$ chemistry, which includes four critical minerals. They demonstrated high recovery and purity for Li, Co, Ni, and Mn using an EDTA-assisted ED process [40].

They simulated the leachate of NMC11 cathode material as the feed with a carefully adjusted concentration of the metals' sulfate salts dissolved in H_2SO_4 and water. The separation was conducted in three stages, achieving 93 % Ni separation in the first stage, 87.3 % Co in the second, and 99 % Li from Mn in the third, by altering one of the CEMs to a monovalent CEM in the third stage (Fig. 7 **(c)**). The process resulted in all recovered metals exhibiting purities above 99 %.

Xing et. al. recently explored the feasibility and application of CAED for processing leachate of waste battery, addressing a gap in earlier studies that focused on synthetic feeds with high Li content and overlooked the presence of various metal ions that could hinder the chelation and separation processes [223]. They employed the BPED process, utilizing four different chelating agents: EDTA, DTPA, HEDTA, and L-glutamic acid (GLDA), in a three-compartment BPED setup arranged sequentially with BPM, CEM, AEM, and another BPM (Fig. 8). This setup processed real leachates from LIBs black mass, categorized into lithium-cobalt (LCO), Ni-rich, and NMC leachates, targeting the recovery of valuable elements for the electric vehicle industry.

DTPA, chosen as the optimal chelating agent at a 1:15 M ratio of metal to DTPA, enabled the recovery of 63.9 % Li with over 99 % purity. High recoveries were noted for Ni-rich and LCO black masses, with limited effectiveness for NMC leachates due to the high presence of transition metals, suggesting that additional chelating agents could enhance separation.

In summary, CAED technology enhances metal selectivity by introducing chelating agents that form complexes with specific metals. This approach utilizes the mobility and charge differences of the resulting metal complexes to improve separation efficiency. Lanthanide metals, which are typically challenging to separate due to their similar ionic sizes and charges, can be selectively recovered by using chelating agents to form stable complexes. Additionally, the formation constants between metals and chelating agents in databases allow for the precise selection of chelating agents for specific metals, even in the presence of multiple base elements (e.g., Ca, Mg, Al, Fe, etc.). Unconventional sources often contain critical minerals at very low concentrations along with various impurities. In such scenarios, the differences in formation constants between critical minerals and other metals can be used to selectively separate specific low-concentration metals.

4.5. *NF membrane combined ED*

Various processes, in addition to the major ED-based methods mentioned previously, have been combined with ED to enhance selectivity and practicality, especially considering the complex composition of leachates and brine streams containing critical minerals. NF membranes have been integrated with ED processes to improve selectivity and efficiency in the separation and recovery of critical minerals. In the context of Li separation from brines, standalone conventional ED and NF processes presence challenges in achieving high-purity Li separation, especially when dealing with brines with high Mg/Li ratios. However, it has been demonstrated that integrating two common pressure-driven membrane processes with BMED can yield high-purity LiOH at high concentrations [224].

Specifically, by employing dual stages of NF and RO prior to BMED, it became possible to feed BMED with a solution rich in Li (>14 g/L) and low in Mg (<0.5 g/L) from salt-lake brine containing an Mg/Li ratio higher than 30. Adding a single stage of NF has been shown to significantly improve Li recovery, increasing from 16.67 % to 41.67 %. However, coupling these pressure-driven membrane processes with different ED techniques can lead to increased energy consumption, potential challenges with membrane performance, and varying current efficiencies, which may complicate the process.

Nevertheless, these challenges can be mitigated by developing and

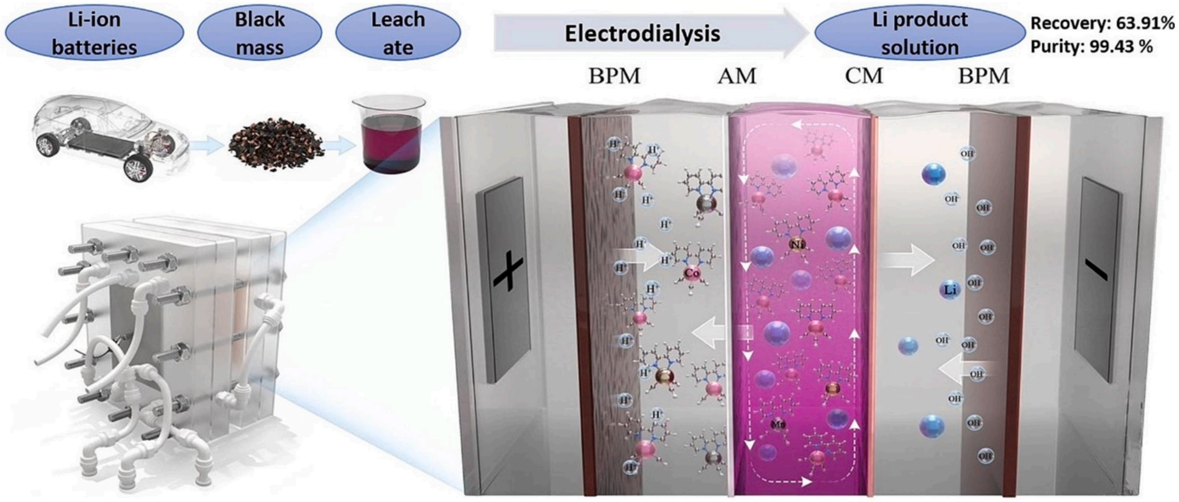

Fig. 8. General illustration of Li recovery from black mass leachate using chelation-assisted BMED. [Reproduced with permission from Ref. [223], Copyright 2023, Elsevier.].

implementing efficient and selective membranes and IEMs, particularly multicomponent exchange membranes (MCEMs) with high separation efficiency for Li separation and recovery. For instance, Wang et al. developed a gallic acid/polyethyleneimine assembled membrane (M−GA/PEI) inspired by mussel adhesion via layer-by-layer assembly on a commercial CEM [225].

This fabricated membrane exhibited improved performance over CEM and commercial MCEMs, utilized in two stages of SED to obtain a Li-rich solution from a one-stage NF process (Fig. 9 **(a)**). The feed water, initially containing a high Mg/Li ratio, underwent significant reduction after the first stage of SED, resulting in enhanced Li purity from 50 % for commercial CEM to 85.5 % for M−GA/PEI. This improvement was attributed to the introduction of dense cationic exchange groups providing high cation flux and the thin GA/PEI monovalent selective layer with high selectivity towards Li and Mg.

Although no significant improvement in Li purity was observed compared to a commercial MCEM (CSO), the results after the first stage of SED indicated a reduction in energy consumption by over 20 %, and significantly improved current efficiency of M−GA/PEI. The second stage of SED using the M−GA/PEI membrane demonstrated enhanced energy efficiency over commercial MCEM even compared to the first stage, achieving energy consumption of 0.011–0.014 kWh/mol Li versus 0.040–3.795 kWh/mol Li with commercial membranes. This highlights the effectiveness of specialized MCEMs in multistage NF-SED processes for optimizing Li recovery from complex brine solutions, offering a promising solution to the challenges associated with critical mineral extraction.

Research on combining NF systems with ED extends beyond simple pretreatment before the ED process. NF membranes are also employed under an electric field in a technique known as electro-nanofiltration, an advanced form of ED aimed at enhancing the selectivity and separation of Li and Mg from brine. This technique capitalizes on the electrostatic repulsion and size sieving capabilities of NF, offering nanoscale separation precision.

Xu et al. explored the use of NF membranes within an ED system [226]. They developed a membrane featuring an ultrathin polyamide layer that demonstrated improvements over the commercial MCEM, CSO (Selemion), by effectively rejecting ions with a larger hydrated

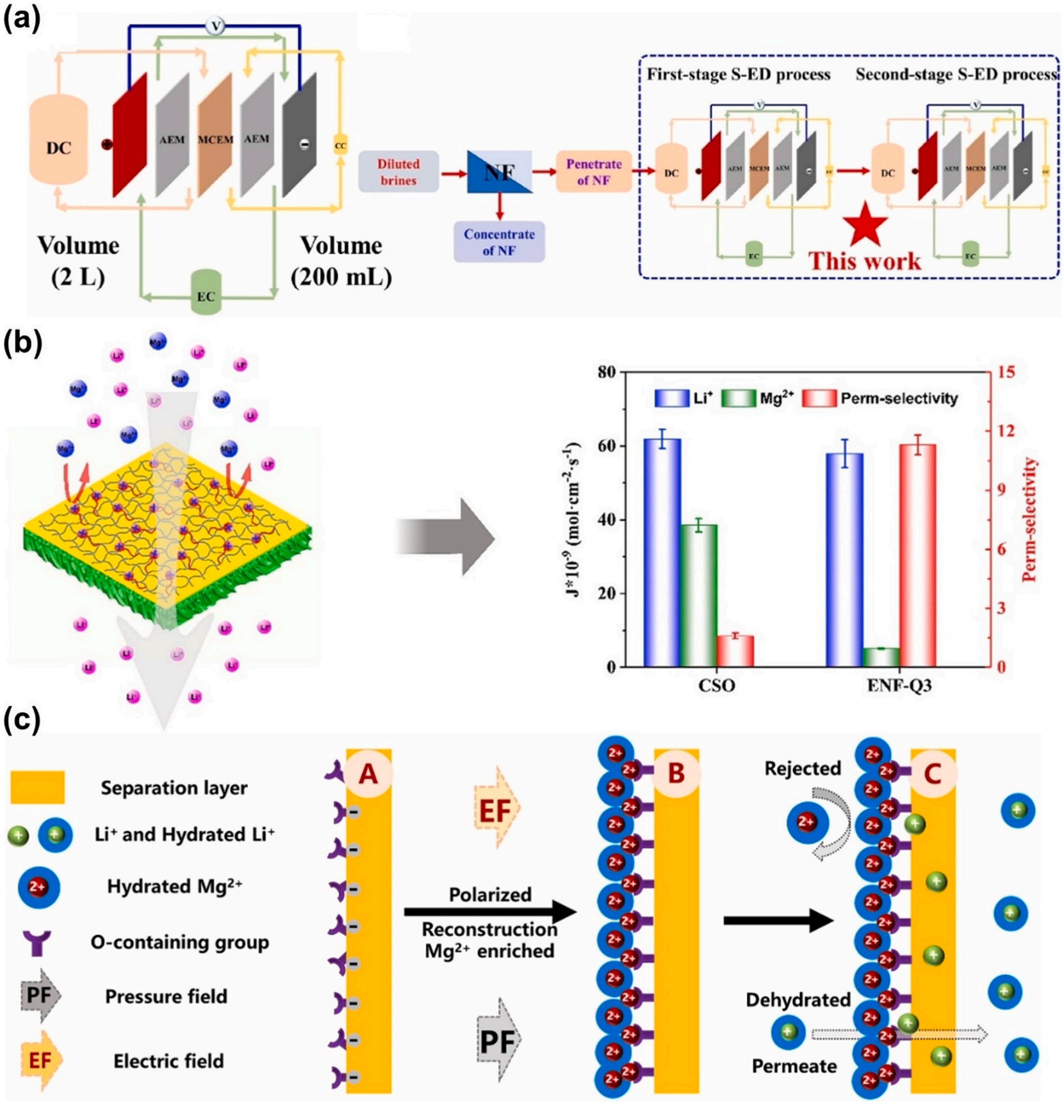

Fig. 9. (a) General representation of multistage coupled membrane process diagram showing hybridization of NF to SED, (b) illustration of NF membrane structure for Li and Mg separation as well as the effectiveness of the membrane in terms of perm-selectivity, and (c) schematic representation of reconstruction of the positive charges on NF membrane surface and the enhancement mechanism of membrane separation performance. [(a) Reproduced with permission from Ref. [225], Copyright 2023, Elsevier. (b) Reproduced with permission from Ref. [227], Copyright 2020, Elsevier. (c) Reproduced with permission from Ref. [228], Copyright 2022, Elsevier.].

radius, thereby reducing area resistance, and improving ion flux. Building on this success, they further refined NF membranes with a densely charged cationic polyamide layer, which significantly enhanced the perm-selectivity between monovalent and divalent cations compared to the CSO MCEM, owing to pore size sieving and enhanced electrostatic repulsion [227]. The selectivity, particularly for Li and Mg separation, was optimized by adjusting the quaternization degree in the polyamide layer, leading to a Li/Mg ratio of 11.3 for the optimized membrane (ENF-Q3), seven times greater than that associated with the CSO MCEM. (Fig. 9 **(b)**).

A recent study conducted by Li et al. presented an additional effective utilization of electro-nanofiltration [228]. By tuning the positively charged layer on the negatively charged NF membrane, they enhanced the current density, resulting in nearly complete rejection of Mg^{2+} ion in a Li-Mg binary system, capable of producing highly pure Li salt. They extensively discussed how Li^+ ion permeation is improved under an electric field due to its lower hydration energy and charge, facilitating easier dehydration and reduced electrostatic repulsion by positively charged membranes. Additionally, increased current density boosts Li^+ ion permeability and transport (Fig. 9 **(c)**).

In summary, combining NF and ED can enhance the separation of valuable metals, particularly Li in high Mg-to-Li ratio brine streams. These advancements are achieved by integrating NF with efficient membrane design, which minimizes ion leakage and improves selectivity, thus enabling more efficient separation. However, the separation of more complex streams beyond brine could lead to severe fouling issues and challenges in selectivity for NF membranes. For instance, the separation of transition metals (e.g., Ni, Co, Mn, etc.) or REEs by NF membrane requires further investigation. Designing monovalent, divalent, and trivalent selective IEMs with enhanced ionic conductivity and separation efficiency, along with NF membranes capable of distinguishing transition metals or REEs, could be a potential approach to address these challenges.

4.6. *Electro-electrodialysis (EED)*

Electrolysis combined with ED has emerged as a promising method for the separation of critical minerals. During electrolysis, an electric current is passed through the stream, causing metal ions to either plate out onto an electrode or form soluble complexes that can be selectively removed. This combination can lead to more efficient separation of targeted metals in mixed stream solutions, providing streams enriched streams of targeted metal ions. Cifuentes et al. successfully employed the EED process to deposit uranium tetrafluoride (UF_4) through reduction on the cathode. This was achieved by dissolving uranium (VI) oxide (UO_3) in hydrofluoric acid (HF) as the catholyte, with sulfuric acid serving as the anolyte in an EED cell, as shown in Fig. 10 **(a)** [229].

Various cathode materials, including Al and stainless steel, were tested, along with a sulfonated polystyrene CEM and a graphite anode, to effectively separate valuable UF_4 materials suitable for use as a reagent in nuclear power plants. The study primarily focused on evaluating the performance of the cathode as the primary depositing agent. Al was identified as a suitable choice, exhibiting a high current efficiency of 68.29 % and an energy consumption of 675 kWh/kg U. Moreover, it was capable of depositing U with a concentration of 25 g/L in the catholyte.

The EED process has emerged as an effective method for Ni recovery from wastewater and Ni-plating baths. Wang et al. successfully extracted Ni from electroplating wastewater with a low concentration of 1 mg/L by employing a titanium plate as the anode, stainless steel as the cathode, and a commercially available CEM (TRJCM) [230]. The commercial TRJCM CEM with sulfonic acid functional groups played a crucial role in the integrated process of ED and electrodeposition by enabling the migration of H^+ and Ni^{2+} ions from the anolyte to the catholyte due to the electrochemical driving force.

Maintaining a low initial pH value was crucial for several reasons: Firstly, it reduced the formation of $Ni(OH)_2$ near the anode, thereby maintained higher Ni concentrations. Secondly, it ensured higher anolyte conductivity, minimizing energy consumption. Thirdly, it controlled pH changes in the electrolytes, and promoted efficient Ni electrodeposition on the cathode by minimizing hydrogen evolution, which would otherwise impede the process and increase energy consumption. The optimized voltage was 2.2 V; lower values resulted in reduced Ni deposition, while higher values were hindered by the concurrent hydrogen evolution. The Ni concentration was successfully decreased to 0.015 mg/L and 0.085 mg/L on the anolyte and catholyte sides, respectively, demonstrating the efficacy of the EED process for Ni recovery.

In another recent study by the Wang et al., efficient recovery of Ni from spent electrolysis Ni plating baths was demonstrated [231]. Their EED process also proved useful for removing harmful phosphorus-

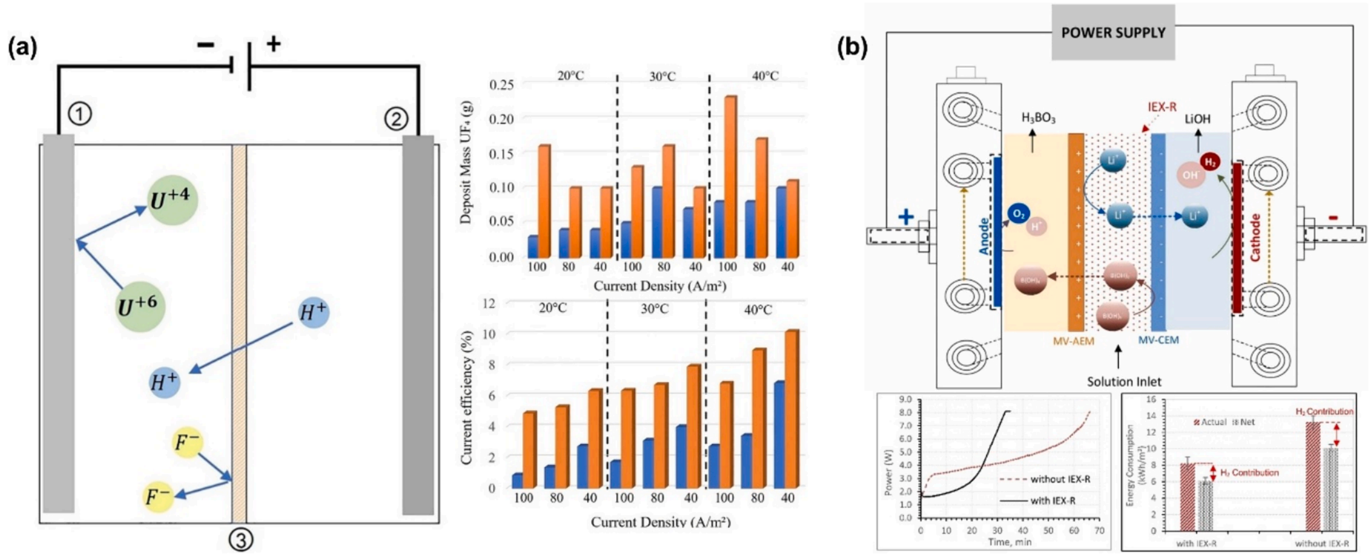

Fig. 10. (a) Schematic illustration of EED and deposited mass of UF_4 obtained from feed compositions of 15 g/L (orange) and 25 g/L (blue) UO_3 in different current densities [229], and (b) schematic representation of the EED process with ion-exchange resins for efficient B and Li recovery and hydrogen production [233]. [(a) Reproduced with permission from Ref. [229], Copyright 2022, Elsevier. (b) Reproduced with permission from Ref. [233], Copyright 2023, Elsevier.]. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

containing substances (HPO_3^{2-}) from the spent bath. The anolyte solution comprised Na_2SO_4, while the catholyte was the spent bath solution with a native pH of 4.5, deliberately diluted tenfold. An AEM was chosen over the CEM to effectively separate the negatively charged HPO_3^{2-} ion from the catholyte side to the anolyte side. Efficient simultaneous recovery of Ni (83.3 %) by deposition and removal of HPO_3^{2-} ion (51.6 %) was achieved with an efficient current density of 3.5 mA/cm^2 and energy consumption of 8.28 Wh/kg.

To enhance the practicality of the EED process, several research studies have explored the simultaneous production of valuable gases or the capture of CO_2 while separating and recovering critical minerals [232–234]. For instance, Alshebli et al. applied the EED process to separate B and Li from simulated wastewater from the mining industry, utilizing ion exchange resin to fill the space between MAEM and MCEM (Fig. 10 **(b)**) [233]. This approach demonstrated the ability to remove over 95 % of Li and B through ion exchange resins while producing hydrogen gas at a rate of 15 mM/h. These findings suggest that this process could become more practical and energy-efficient, with an energy consumption of 6.1 kWh/m^3 when treating synthetic wastewater.

Overall, EED technology has demonstrated efficient performance in recovering metals from waste streams such as Ni-plating baths and Li wastewater. For optimal EED operation, factors such as pH optimization, feed flow rates, and the design of efficient electrodes and membranes are critical. However, the applicability of EED for metal separation from various feedstocks, including leachate of REEs and waste batteries, requires further investigation.

5. Prospects and challenge

ED technology has been extensively researched for applications such as desalination and heavy metal removal from wastewater. Recent advancements in membranes and ED technology have highlighted its potential for recovering valuable metals. In the metal refinery industry, techniques like solvolmetallurgy and electrochemical metal recovery are being explored to recover critical minerals from unconventional sources in a more environmentally friendly approach [17,19]. This comprehensively review covers recent advances in ED technology and IMEs as a methodology for critical mineral recovery.

As reviewed in Section 2, energy-related critical minerals can be obtained from various sources, including aqueous phases such as low-concentrated Li brine or Ni electroplating wastewater, and solid-state feedstocks such as waste batteries, mine waste, slag, and low-concentration geological ores. Processing solid-state feedstocks typically involves a metal leaching process using lixiviants, which consume large amounts of acid. The resulting leachate is generally acidic and may contain various anions (e.g., Cl^-, NO_3^-, and SO_4^{2-}) from the lixiviant [13]. Therefore, employing ED technology for metal separation and simultaneous solvent regeneration is expected to reduce the processing costs associated with handling solid-state feedstocks.

Additionally, unconventional sources of critical minerals often contain various impurities and include metals that are challenging to separate. To effectively utilize ED technology, it is essential to clearly define the goals: (i) separating entire groups of metal species, such as transition metals or groups of REEs, from base elements; (ii) separating specific metals from base metals alongside similar characteristics; or (iii) concentrating specific critical minerals. Establishing clear objectives for employing ED technology and selecting the appropriate IEMs and techniques are expected to enhance metal recovery efficiency.

From a technical perspective, achieving selectivity for specific metals and metal groups is crucial for effective metal recovery using ED. The recent ED technologies discussed in this review (e.g., SED, BMED, LMED, and CAED) each presents unique advantages and disadvantages, and their effectiveness in separating metals varies. Furthermore, the target mineral sources differ, leading to slight variations in technological development trends.

Research on SED technology is actively progressing in various fields, particularly in the recovery of critical minerals from Li brine and waste batteries [26,208]. Li brine, along with geothermal and oilfield brines, contains not only Li but also alkali and alkaline earth metals such as Na, K, Ca, and Mg. Although there is considerable research on developing IEM materials to separate Li from these metals, achieving high selectivity for separating Na and K from Li remains challenging. Furthermore, while membranes for SED applications are being developed to extract specific metals from waste batteries, increasing selectivity typically results in higher electric resistance of the membranes. Thus, to effectively employ SED technology for metal separation, further research is needed to enhance selectivity while minimizing electric resistance of the membranes.

Research related to BMED technology focuses on the separation and recovery of metals such as Li and Ni from various feedstocks, while also recovering solvents [54,124]. The ability to electrochemically regenerate solvents while recovering metals makes this technology highly attractive for metal refineries, as it can significantly reduce process costs. This benefit is particularly crucial in the battery industry, where large amounts of Na_2SO_4 are discharged in the final wastewater [235]. However, BMED technology encounters challenges when processing feedstocks with complex mixtures of metals, and its application to REEs has not yet been extensively studied. Therefore, it is anticipated that developing advanced BPM materials and improving ED systems to function in more diverse environments could significantly expand the applicability and versatility of BMED technology.

In LMED, membranes are fabricated with solvents commonly employed in solvent extraction, which enhances the chemical affinity of the metal separation mechanism [181,192]. The extractants used in forming liquid membranes often have databases on metal selectivity, making it easier to synthesize membranes tailored for specific target metals. Consequently, liquid membranes exhibit high selectivity, even for metals that are difficult to separate, and represent high metal permeability when integrated with ED technology. However, liquid membranes typically have high electrical resistance, resulting in increased electrical energy consumption. Additionally, the extractant could be lost from the membrane, complicating the maintenance of long-term cyclability. Therefore, developing membranes with lower electrical resistance and greater stability could lead to systems that are both highly selective and durable for extended use.

In CAED, both membranes and chelating agents are utilized to enhance metal separation through complexation, resulting in high selectivity for specific metals [189,196]. This technique demonstrates excellent performance in separating transition metals (e.g., Ni, Co, and Mn) from waste batteries, and REEs from mine wastes. However, further research is needed to develop methods for effectively reusing the chelating agents used during the operation and to reduce the resistance of the ED system.

In summary, effectively separating critical minerals using ED technology requires enhancing metal selectivity and reducing membrane resistance through material development or system improvement. To maximize the advantages of each technology, it is crucial to develop feedstock-tailored ED systems that align with the characteristics of the targeted critical minerals and unconventional sources. Furthermore, addressing fouling issues necessitates further investigation by employing not only model chemicals produced in laboratory settings but also real wastewater, low-concentrated brine, and leachate from solid-phase feedstocks containing various impurities and anions into ED systems.

Advancing these developments could enable the practical application of ED technology in the critical mineral recovery industry, offering numerous environmental benefits such as reduction of toxic solvents, solvent regeneration, and decreased wastewater generation. Notably, ED technology's direct use of electrical energy for metal separation and recovery makes it particularly advantageous. By integrating renewable energy with ED technology, the carbon footprint of the refining process could be significantly reduced. These characteristics of ED technology are expected to provide substantial advantages in meeting the current demand for environmentally friendly resource recovery in the metal refining industry.

6. Conclusions

The global drive towards achieving carbon neutrality in the energy sector and addressing climate change is expected to significantly increase the demand for critical minerals essential for deploying clean energy technologies. However, obtaining these minerals through conventional mining methods presents challenges in meeting this rising demand and raises concerns about environmental pollution associated with extraction.

Consequently, many countries worldwide are eager to explore sustainable strategies for sourcing these critical minerals from unconventional outlets such as brine, wastewater, e-waste, and industrial waste. These unconventional sources often contain a diverse array of elements, typically with lower concentrations of the desired critical minerals compared to conventional sources and are characterized by complex combinations and interactions of elements. Moreover, variations in factors such as pH, alkalinity, and ionic strength from traditional reservoirs require technological considerations for extracting these critical minerals from unconventional sources.

Considering these challenges, this critical review focuses on critical minerals such as Li, Co, Ni, and REEs, presenting the latest electrically driven membrane technologies (i.e., ED) for extracting these minerals from unconventional sources. These ED technologies offer distinct advantages over traditional metallurgical methods, requiring less solvent and energy. Specifically, advanced ED technologies such as SED, BMED, LMED, CAED, NF integrated ED, and EED are expected to be effectively utilized for recovering Li, Co, Ni, and REEs from unconventional sources.

To efficiently harness these technologies, research is essential not only on the necessary materials but also on the environmental factors associated with these unconventional sources. Considering that these processes rely on electrical energy generated from renewable sources, it is crucial to determine how to integrate them efficiently with renewable energy. Additionally, given the diverse elements present in unconventional sources, it is vital to consider scaling-up approaches that can integrate with existing technologies such as hydrometallurgy if necessary. This review aims to enhance understanding of the latest technologies for effectively recovering critical minerals from unconventional sources, providing an opportunity to advance the implementation of a circular economy.

CRediT authorship contribution statement

Gyudae Sim: Writing – original draft, Conceptualization. **Mohammad Pishnamazi:** Writing – original draft, Visualization, Conceptualization. **Dongju Seo:** Writing – original draft, Visualization. **Shik Rou Kong:** Writing – original draft, Visualization. **Jiwoo Lee:** Writing – original draft, Visualization. **Youngjune Park:** Writing – review & editing. **Soryong R. Chae:** Writing – review & editing.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Youngjune Park reports financial support was provided by National Research Foundation of Korea. Soryong R. Chae reports financial support was provided by National Science Foundation. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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