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Review

Sustainable Extraction of Critical Minerals from Waste Batteries: A Green Solvent Approach in Resource Recovery

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Abstract: This strategic review examines the pivotal role of sustainable methodologies in battery recycling and the recovery of critical minerals from waste batteries, emphasizing the need to address existing technical and environmental challenges. Through a systematic analysis, it explores the application of green organic solvents in mineral processing, advocating for establishing eco-friendly techniques aimed at clipping waste and boosting resource utilization. The escalating demand for and shortage of essential minerals including copper, cobalt, lithium, and nickel are comprehensively analyzed and forecasted for 2023, 2030, and 2040. Traditional extraction techniques, including hydrometallurgical, pyrometallurgical, and bio-metallurgical processes, are efficient but pose substantial environmental hazards and contribute to resource scarcity. The concept of green extraction arises as a crucial step towards ecological conservation, integrating sustainable practices to lessen the environmental footprint of mineral extraction. The advancement of green organic solvents, notably ionic liquids and deep eutectic solvents, is examined, highlighting their attributes of minimal toxicity, biodegradability, and superior efficacy, thus presenting great potential in transforming the sector. The emergence of organic solvents such as palm oil, 1-octanol, and Span 80 is recognized, with advantageous low solubility and adaptability to varying temperatures. Kinetic (mainly temperature) data of different deep eutectic solvents are extracted from previous studies and computed with machine learning techniques. The coefficient of determination and mean squared error reveal the accuracy of experimental and computed data. In essence, this study seeks to inspire ongoing efforts to navigate impediments, embrace technological advancements including artificial intelligence, and foster an ethos of environmental stewardship in the sustainable extraction and recycling of critical metals from waste batteries.

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1. Introduction

Sustainable development and the environment are intrinsically interconnected, serving as the cornerstone of global initiatives aimed at fostering a harmonious coexistence between industry and nature [1,2]. Notably, the investigation of sustainable methods in battery recycling and mineral extraction is of utmost importance. Both are essential components of contemporary technology, but their traditional approaches are often tainted by environmental deterioration and the exhaustion of resources [3]. The practice of recycling batteries is becoming more popular due to the growing demand for electric vehicles (EVs) and portable electronic gadgets [4]. The battery industry had significant growth from USD 62.00 billion in 2014 to USD 120.00 billion in 2019 [5]. The estimated size of the worldwide rechargeable battery market in 2022 was USD 110.44 billion [6]. It is projected to reach around USD 195.55 billion by 2032, with a compound annual growth rate of 5.88% over the forecast period from 2023 to 2032, as shown in Figure 1A. The expansion of Pb-acid batteries is expected to be driven by their cost-effectiveness and dependable performance, as shown in Figure 1B [7].

However, conventional recycling techniques, which depend on strong chemicals and energy-intensive procedures, cause notable environmental hazards [8]. In addition, recycling technologies for rechargeable batteries cannot only contaminate (in the event of their leakage) the environment with heavy metals but also with organic compounds and highly acidic or alkaline electrolytes [9].

The U.S. Geological Survey (USGS) defines key minerals as those that are vital to national security and the economy yet susceptible to supply chain disruptions because of limited domestic production and geopolitical considerations. These are essential for electronics, defense applications, and renewable energy technologies [10]. The process of extracting minerals from ores is often demanding in terms of energy. It is necessary to employ high temperatures during the smelting or leaching process, as well as significant amounts of strong acids or bases, both of which demand energy to create. It is necessary to treat the gas, liquid, or solid waste that is typically generated by these processes before it can be properly disposed of. Extra energy is needed for this waste treatment process as well. The traditional reagents used in these approaches often exhibit potential risks to human health, the environment, and safety, and the cause other associated concerns [11]. To enhance the safety and cleanliness of these approaches, it is essential to investigate the use of green and more eco-friendly reagents. It is estimated that around 20 million metric tons (MT) of organic solvents are produced annually at the industrial scale [12]. Overuse of hazardous, non-renewable solvents is a prime example of unsustainable behavior and unhygienic for the environment [13].

Solvation offers several significant benefits, including enhanced handling and safety, by acting as a heat sink that absorbs excess heat generated during chemical reactions, thereby preventing overheating, protecting solvents from thermal degradation, and maintaining optimal conditions for selective recovery. For example, green solvents such as choline chloride–urea effectively serve as heat sinks in battery-recycling processes [14]. Over the past two decades, researchers have introduced and investigated a new class of solvents known as “master solvents”, which include ionic liquids, supercritical carbon dioxide, deep eutectic solvents (DESs), thermomorphic solvents, and fluorous solvents. These solvents, often referred to as green or designer solvents, have the potential to expand the capabilities of conventional solvents by enhancing solubility, stability of excited states, and overall process efficiency [15]. For a solvent to be considered green, it should be non-volatile, non-toxic, recyclable, and biodegradable and require minimal energy for its synthesis [16,17].

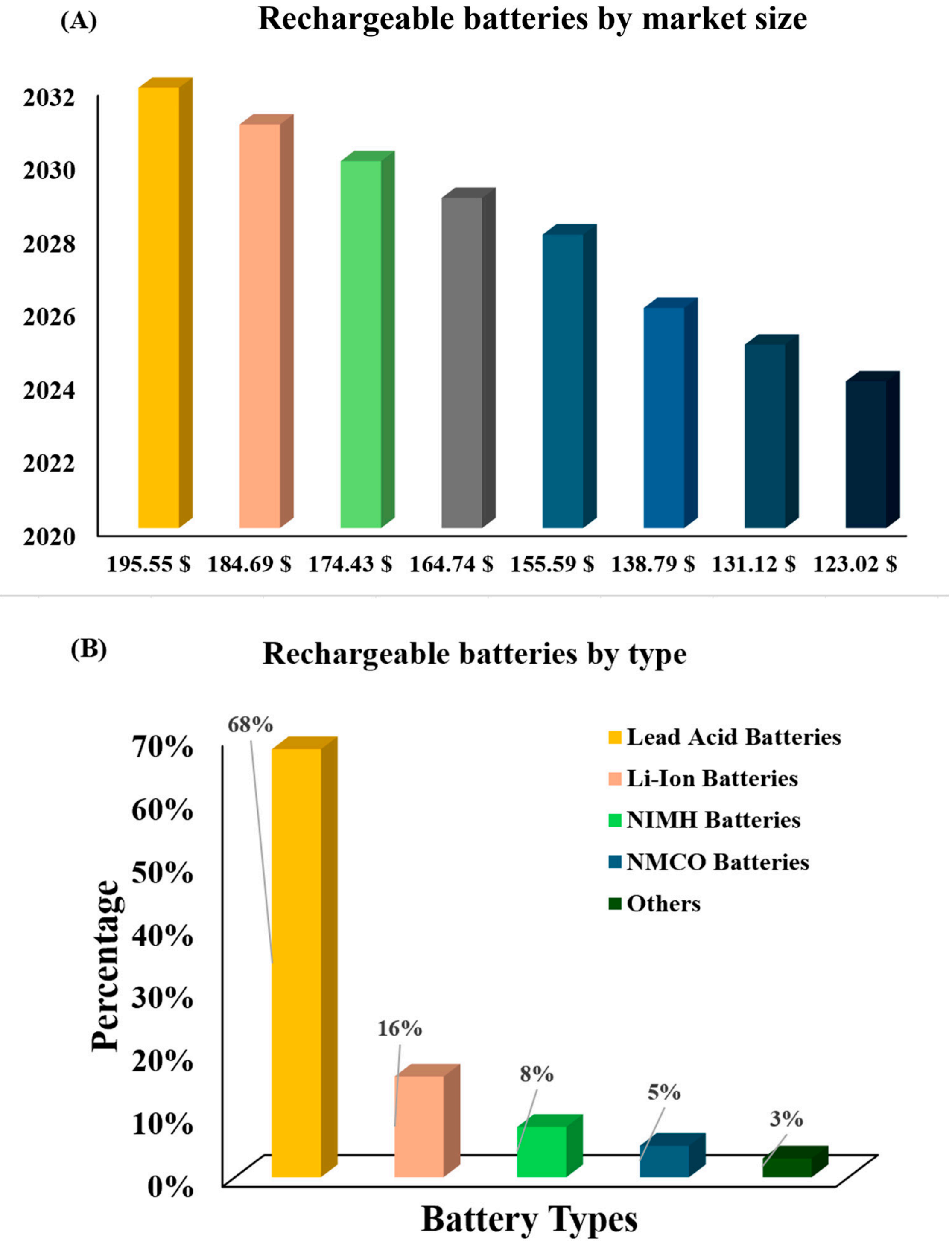

Figure 1. (**A**) Rechargeable batteries by market size, 2022–2023 (billion USD), and (**B**) rechargeable batteries by battery type, 2022.

Ionic liquids, supercritical CO_2, DESs, thermosmorphic, and fluorous solvents comprise majority of the components. These solvents and their applications are well-documented and widely appreciated [18]. Utilizing ionic liquids (ILs) and DESs as agents in mineral flotation and extraction of important metals offers innovative alternatives to conventional technologies, enabling environmentally friendly and sustainable production [19]. A number of minerals, including carbonate asphalt, quartz, quartz hematite, and rare earth minerals, have been successfully floated in DESs and ILs in recent years [20].

The scalability of existing recycling technologies remains limited due to technical and economic constraints, presenting a significant challenge, particularly in the case of direct positive-electrode recycling. To enable scaling, it will be crucial to recover feedstock directly from end-of-life (EoL) LIBs to verify its feasibility at scale [21]. While direct positive-electrode recycling is recognized for its potential to recover valuable materials with a lower environmental impact compared to conventional methods, several challenges must be addressed before it can be adopted on an industrial scale. For example, the handling of fluorine impurities generates toxic byproducts, hindering the recycling process [21]. Additionally, the precise chemical selection of waste LIBs is essential, further increasing the complexity and cost of recycling.

The rapid advancement of positive-electrode chemistries suggests that older recycling techniques may not be compatible with the cutting-edge materials used in next-generation batteries. This disparity necessitates continuous adaptation of recycling methods to align with ongoing developments in battery design, potentially hindering the widespread adoption of direct positive-electrode recycling [22]. To address these challenges and enhance the environmental sustainability of LIB production and recycling, the development of eco-friendly methods is critical. New binders can replace traditional polymeric binders like PVDF, which rely on solvents such as N-methyl-2-pyrrolidone (NMP), known for its environmental drawbacks.

Considering the present challenges, it is essential to develop sustainable strategies to address future challenges and opportunities in battery recycling and mineral extraction. The novelty of this study lies in the comprehensive determination of the gaps and limitations in current recycling methods, such as, for example, massive cross contamination of black mass and traditional inefficiencies of hydrometallurgical and pyrometallurgical methods. This study highlights the urgent need to develop green organic solvents, binders, and more efficient recycling methods.

Additionally, it also explores the radical potential of ILs and DESs to overcome the challenges faced, as innovative solutions that could markedly lower environmental impacts and raise the viability of large-scale battery recycling. The emerging role of machine learning and its application may be useful in this industry. A focus on sustainable, green solvents allows this study to make a significant contribution toward advancing environmentally responsible and economically feasible recycling technologies.

2. Critical Minerals in Batteries

2.1. The Role of Critical Minerals in Battery Technology

The most crucial minerals in rechargeable batteries are cobalt (Co), lithium (Li), nickel (Ni), copper (Cu), and lead (Pb) [21]. Further details are given in Table 1. A key element is a valuable component of a mineral commodity, based on the state of demand, which faces the risk of supply disruption [22].

Table 1. Critical minerals in battery chemistry.

Critical Mineral	Main Global Producer	Uses	Global Demand	References
Cobalt	Republic of Congo (73%), Russia (4.3%) and Australia (4%)	Batteries (50%), super alloys (17%), hard metals (8%), magnets (3%), catalysts (5%), and ceramics/pigments (4%)	It is forecast that in 2030, e-mobility batteries alone will account for an estimated 176,000 metric tons of cobalt demand worldwide. In contrast, non-battery applications of cobalt are only expected to amount to 65,000 metric tons of cobalt demand globally by that year.	[23,24]
Lithium	Australia (53%), Chile (32%), and China (13%)	Lithium-ion batteries (LIBs) (59%), ceramics and glass (9%), lubricating greases (8%), casting and alloy industries (3%), air conditioning (3%), and primary aluminum manufacturing (1%)	In 2030, the global demand for lithium is expected to be more than quadruple the demand in 2022, having increased from 720,000 metric tons to a forecasted 3.1 million metric tons.	[25,26]
Graphite	China (62%)	Almost 90% of the spherical negative-electrode-grade graphite used in LIBs	Global graphite consumption by LIBs is anticipated to grow to just below 447,000 metric tons in 2021 and increase four-fold thereafter by 2030.	[27,28]
Nickel	Indonesia (40.2%)	Stainless steel (69%), batteries (11%), nonferrous alloy (7%), plating (6%), alloy steel (3%), and foundry (1%)	According to smart new energy (SNE) research, the global demand for nickel batteries is predicted to double from 385,000 tons in 2022 to 841,000 tons in 2025 and increase six-fold to 2.37 million tons in 2030. The market will rise from USD 7.7 billion in 2022 to USD 18.511 billion in 2025 and USD 71.088 billion in 2030.	[29]
Copper	Chile (27%)	Transport (36%), infrastructure (18%), and appliances (20%)	Recently, Co has been projected to increase to 44.4 Mt in 2050 compared to 24.3 Mt in 2015. The need for copper in transportation infrastructure and automobiles will increase fourfold. A higher collection rate can reduce the primary copper output (from copper ore) by 2.2 and 2.3 Mt, respectively, by 2050.	[30]

Table 1. *Cont.*

Critical Mineral	Main Global Producer	Uses	Global Demand	References
Manganese	South Africa (36%)	Solar, wind power, and LIB	The global market for Mn will be increased to USD 3.9 billion in 2038.	[31,32]
Aluminum	Guinea, Australia, Vietnam	Electrolysis, positive electrode manufacturing, alumina smelting, and bauxite mining and Al scrap	By 2050, the demand for aluminum products is expected to increase by 81%.	[33]
Titanium	China (30%)	Titanium dioxide (TiO_2), a white pigment in paints, paper, and plastics	By 2028, the titanium market is expected to expand by USD 8.78 billion at a compound annual growth rate (CAGR) of 5.89% due to its use in the automotive, aerospace, and medical sectors.	[34,35]
Iron	Australia (37.6%)	Metallurgical industry	In 2024, the worldwide iron ore market was estimated to be worth USD 297.82 billion. It is anticipated to increase at a CAGR of 2.7% from 2024 to 2032, reaching USD 392.11 billion.	[36,37]

2.2. Challenges and Limitations in Traditional Extraction Methods

The ubiquitous demand for critical minerals is supported by the growing need for renewable energy and low-carbon technology, which are prerequisites for a future energy system that uses less carbon [38]. Important minerals for low-carbon technology include Co, Ni, Ag, Mn, Cu, Li, and rare earth elements. Numerous products that benefit the environment make use of these eco-friendly minerals. Those include solar photovoltaic panels, wind turbines, electrical vehicle (EVs), and energy storage devices. A considerable portion of the world's supply of different minerals is processed in China [39]. Extraction corporations persist in extracting minerals at a rate that is not sustainable, resulting in the depletion of resources.

2.2.1. Extraction Practices and Environmental Deterioration

Environmental deterioration is a significant problem that is strongly connected to the exhaustion of both natural and mineral resources [40]. Annually, a significant amount of mine waste, amounting to several gigatons, is generated worldwide. The rapidly growing worldwide demand for natural resources is driving an increase in the extraction of larger-scale and lower-grade deposits [41]. Mine waste is typically kept on-site indefinitely when there is no technical or financial means to recycle or otherwise increase its value. Even with proper management in designed storage facilities, extraction waste may still have far-reaching consequences. Among them are climatic, environmental, hydrological, geotechnical, and ecological factors pertaining to the state of the natural environment, including air, water, and soil [42].

Excavation activities have a significant impact on the environment by altering land usage, causing deforestation, generating waste [43] ,and resulting in other disturbances in the ecological system, such as air and water contamination [44]. The release of greenhouse gases resulting from extraction and beneficiation operations, as well as fuel and electricity use, contributes to climate change [45]. The adverse effects on population displacement, human health, and safety and the creation of conflicts over land usage are also widely recognized [46]. Excavation and extraction activities can affect regions extending up to 50 km from mine sites [47]. Because of their detrimental effects on biodiversity, extraction-related activities close to or inside protected areas have occasionally been prohibited [48].

The necessity of extraction will persistently rise in the future due to the significant contribution of mineral resources in fostering socio-economic progress and facilitating the shift towards a more environmentally friendly future [49]. The World Bank has determined that the output of some minerals may see roughly a 500% rise by the year 2050 in order to satisfy the rising need for renewable energy technology [50].

2.2.2. Environmentally Friendly Extraction Practices

Due to the substantial dependence of sustainable development on minerals, it is essential to participate in meticulous planning and decision-making procedures to ensure that extraction operations provide advantages for all stakeholders. Eco-friendly and climate-smart extraction are novel methodologies that effectively address the environmental and sustainability concerns linked to mineral extraction activities, while also mitigating the impacts of climate change [46].

Implementing recycling technologies and procedures is the optimal and primary approach to ensuring the preservation of mineral waste [51]. An increasing number of nations recognize the significance of metal recovery in parallel with the exploitation of natural resources and have implemented a governance framework for the metal-recycling sector [52]. For example, European mineral extraction companies prioritize the utilization of valuable secondary minerals by using the circular economy concept. This approach has significant potential to address the scarcity of minerals, generate profit, and simultaneously reduce energy consumption, environmental risks, and overall environmental impact [53]. In South Africa, these forms of mineral waste are commonly deposited in landfills, resulting in significant environmental and health issues for local people. The adverse impacts of excavation and metallurgical and metal production processes are being addressed by implementing recycling techniques [54]. A general illustration of the mineral extraction impact is given in Figure 2.

2.3. Potential of Using Recycled Critical Minerals in Battery Production

Critical minerals are the fundamental aspects of battery technology. Li and Co, in particular, are critical and primary pillars of major economies including China, the US, Japan, and the EU. Because of their geopolitical supply chain risk, better understanding and importance of energy evolution [55].

In this study, we performed a comprehensive analysis of recent reports and statistics, as mentioned in Figure 3 [56]. According to the analysis and reports, Cu is high in demand, with its extraction reaching 31,131 KT in 2023, and mining is not enough to fulfill the demand. This scenario will worsen by 2040 with a 60% shortage of Cu, Co (50.37%), Li (69.11%), and Ni (31.74%), as described in Figure 3b. Therefore, it is necessary to unlock the potential of critical recycled minerals (Figure 3c) for battery production, to achieve a low-carbon and green sustainable future.

Figure 2. Impact of mineral extraction and environmental deterioration, emission of gases during mining and battery production, and ubiquitous consumption of electronic appliances.

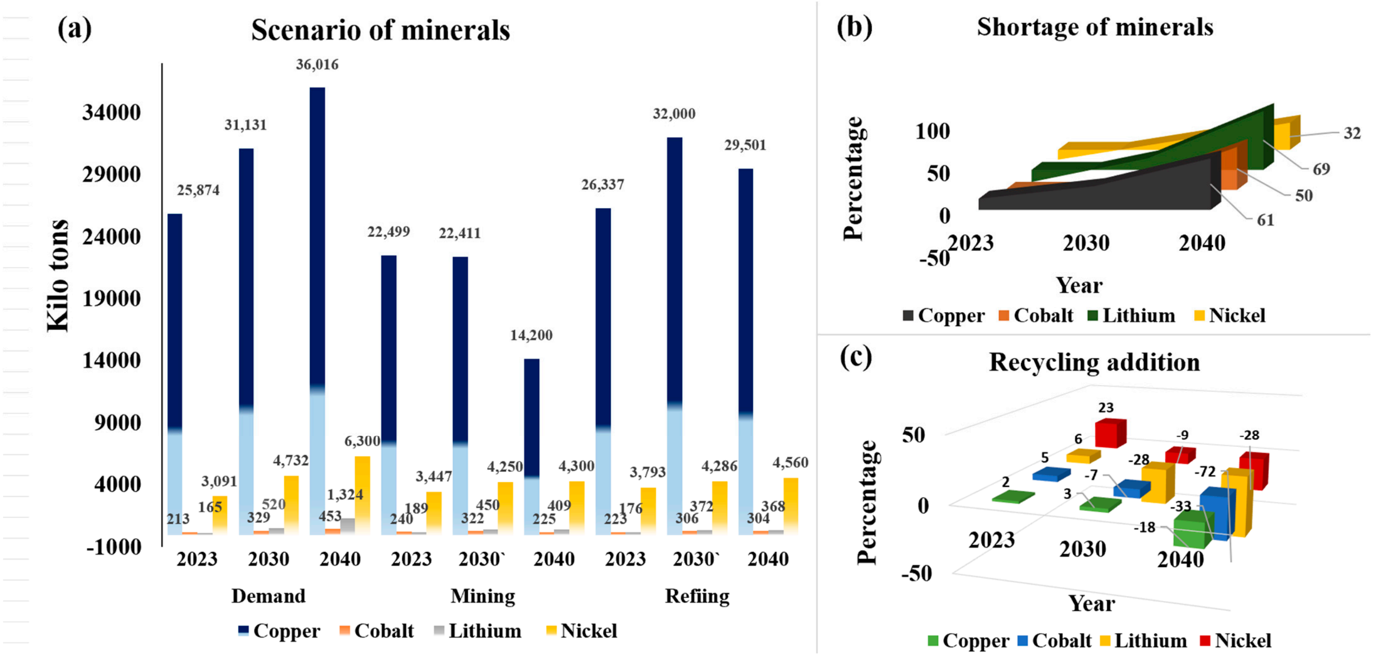

Figure 3. (**a**) Critical minerals in different aspects including demand, mining, and refining minerals in 2023, 2030, and 2040, (**b**) shortage of minerals, which is calculated by demand and mining parameters, and (**c**) refinery addition of minerals to fulfill the demand.

Developments in battery recycling and battery-manufacturing technology are directly linked and poorly discussed in the previous literature [57]. For instance, the stress in the DRC mining industry was reflected in a threefold increase in Co prices between 2016 and 2018. Because these resources are essential for satisfying the expanding worldwide demand brought on by advancements in the battery-manufacturing industry, it is imperative that DR Congo industrializes and diversifies its Co production [58,59]. If we examine Figure 3c, then we can see that recycling facilities are increasing and this is combating the total need for minerals (2023), but if we further look at the recycling facilities, we can see that production will not be enough to fulfill mineral demand; for instance, in the case of Li, the recycling facilities will offer −39% the demand. This scenario reveals a need to develop novel methods to improve the efficiency of recycling facilities.

Moreover, another analysis indicates that by recycling end-of-life batteries efficiently, the global EV material demand may be reduced by 55% for freshly mined Cu, 25% for Li, and 35% for Co and Ni by 2040. Recent analysis reflects that there is significant potential in recycling minerals, for example, recycling lithium cobalt oxide (LCO, 43%) and nickel manganese cobalt (NMC: NMC333 27%, NMC811 16%), hydro-recycling them (LCO 41%; NMC333 13%, and pyro-recycling them (LCO 38%; NMC333 6%, NMC811 5%). Furthermore, it is projected that by 2050, positive electrodes will reach 22 million metric tons (MMT) with an 80% recovery efficiency, cobalt will reach 2.7 MMT with an 80% recovery rate, and nickel will reach 8 MMT with 6.5 MMT recovery [60]. This presents a substantial potential to minimize the requirement for new mineral extraction industries to achieve sustainable development goals. It will also be crucial to explore additional methods for recycling, such as regulations that discourage private automobile ownership and increase accessibility to public and active transportation options, given the rate at which the demand for EVs is growing.

3. Current Recycling Techniques

The use of rechargeable batteries is increasing in direct proportion to the substantial expansion in the worldwide demand for these batteries. In light of the current situation, the remaining capacity of end-of-life rechargeable batteries presents a compelling possibility for new enterprises via battery reuse and remanufacturing for second-life applications [61]. Recycling used batteries to reduce energy consumption and CO_2 emissions conserves natural resources by removing the need to extract new materials and arrange imports, mitigates environmental damage, provides economic advantages, minimizes waste, and addresses safety issues [62]. An earlier investigation reported that metal recycling might produce a 13% reduction in the cost of LIBs per kilowatt-hour. However, at present, the worldwide recycling rate for LIBs is at less than 3%, which is alarming [63].

3.1. Metallurgical Processes and Study Gaps

3.1.1. Hydrometallurgy

The hydrometallurgical process has gained substantial attention in both research circles and industry due to its remarkable recovery rates of Co, Ni, and Li, exceeding 98%, along with its low energy requirements, excellent selectivity, minimal pollutant generation, and relatively low cost, particularly at lower production volumes [64]. This approach involves a systematic sequence of four phases, preceded by pretreatment. During pretreatment, the sophisticated recycling process typically comprises five steps [65,66]. Initially, a sorting process eliminates non-battery waste and categorizes battery chemistries, sizes, and conditions according to recycling facility standards [67]. Stabilization follows to mitigate thermal runaway risks and potential product losses from fires, before disassembly and comminution facilitate outer shell removal, enabling potential future advancements [68]. Subsequent separation yields highly concentrated materials for further reclamation processing, with resulting byproducts including various components like metals, plastics, and separators [69]. After implementing all this preprocessing, the primary recycling technologies of pyrometallurgy and hydrometallurgy are used to recover essential components, as shown in Figure 4.

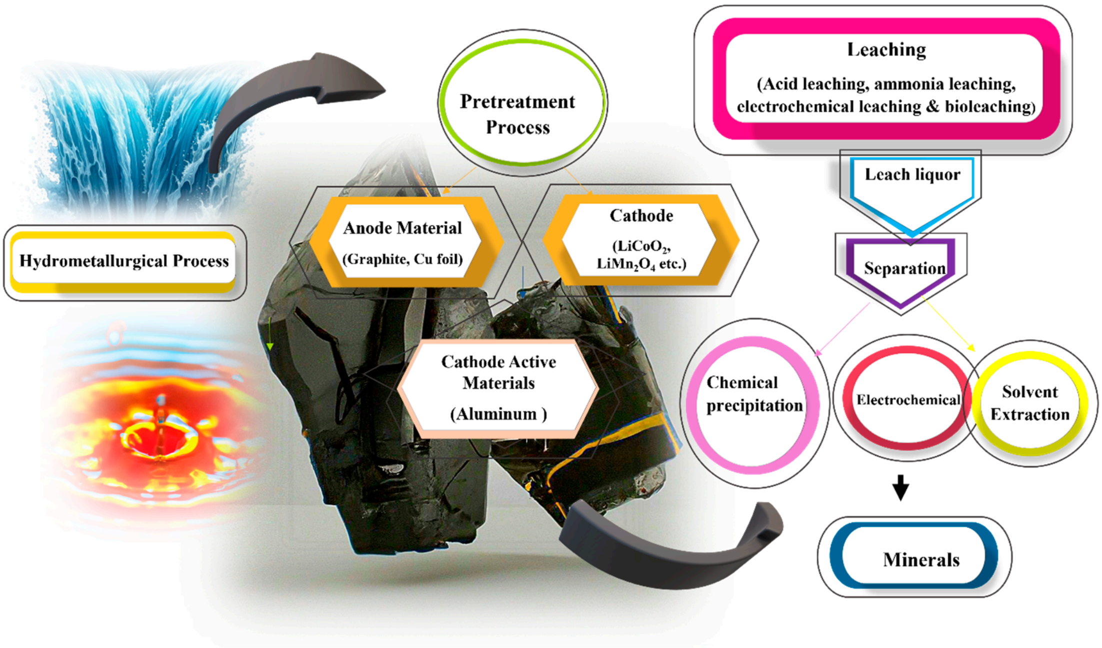

Figure 4. Hydrometallurgical process comprising the pretreatment of negative-electrode and positive-electrode materials followed by potential leaching and mineral extraction.

Leaching serves as the subsequent stage in hydrometallurgy post-pretreatment. Here, the objective is to convert metals from the positive-electrode material into ionic solutions crucial for metal recovery [64]. Leaching techniques encompass acid (inorganic) [70] or organic [71]), ammonia [72], electrochemical [73], and bioleaching methods [74], often supplemented with reductants to optimize effectiveness [73,75]. Acid leaching has particularly proven efficient in extracting Li, Ni, Co, and Mn in the solution, with impurities subsequently extracted [76]. This necessitates purification and separation procedures for successful recovery. Separation becomes critical due to the complex composition of leaching solutions [77], aiming to recover targeted minerals effectively and selectively while preventing the leaching of unwanted elements like Cu and Al [78], such as $MeSO_4$ (where Me represents Ni, Co, or Mn) [79], Co_3O_4 [76], MnO_2, Li_2CO_3, or Li_3PO_4 [80].

Solvent extraction, chemical precipitation, and electrochemical deposition are employed to retrieve metals from unpolluted solutions. Extracting different minerals from heterogeneous solutions can be complicated, even though it is simple for some components like Li and transition minerals [80]. Techniques for isolation range from progressive segregation to systematic categorization followed by gradual separation. With the new EU battery directive emphasizing high standards for precursor materials and circular economy goals, hydrometallurgy emerges as a favorable approach [81]. Its ability to produce high-purity metal salts and recover greater amounts of battery ingredients aligns with these requirements [82].

However, challenges persist, including cross-contamination during mechanical pretreatment, control of manganese's oxidative state, fluorine recovery from electrolytes, and commercially viable extraction of Li, Ni, Co, and graphite [83]. Additionally, ensuring the recovery of suitable battery salt remains a hurdle, with only half of the overall battery material, including crucial constituents like graphite, being recovered. Resolving these technological issues is imperative to advance hydrometallurgical recycling operations, enhancing their sustainability and competitiveness in the transition towards a circular economy approach to LIB recycling [64].

One significant limitation of hydrometallurgical recycling is the reliance on physical disassembly and separation methods, which often lead to cross-contamination of the black mass with organic materials, such as the fluorine-containing polyvinylidene fluoride (PVDF) binder, as well as impurities like Al, Cu, and Ti [63]. Moreover, hydrometallurgical processes face technological challenges, including controlling the oxidative state of Mn, recovering fluorine from electrolytes, and efficiently extracting Li, Ni, Co, and graphite in a commercially viable form suitable for battery reuse [59,73]. Subsequently, it is difficult to guarantee the recovery of Li, Ni, and Co salts suitable for batteries in a way that satisfies market requirements. Merely 50% of the overall battery material is comprised of the positive-electrode materials that have been recovered [59]. Important constituents are lost, including graphite, which accounts for 12.5% (0.978 kg kW^{-1} h^{-1}) of the total energy in an nickel manganese cobalt (NMC) 111 battery, as well as Cu and Al, which make up 3.11 kg kW^{-1} h^{-1} and 0.677 kg kW^{-1} h^{-1}, respectively [64]. To advance hydrometallurgical recycling operations and improve their sustainability and competitiveness as we move toward a circular economic approach to LIB recycling, it is imperative that these technological issues be resolved.

3.1.2. Pyrometallurgy

A conventional method for recycling rechargeable batteries typically involves the steps in Figure 5 [84]. The process begins with adding the batteries and reductants, such as charcoal, to the smelter. To roast the mixture, the furnace is then brought to a temperature of more than 1400 °C. Notably, the organic electrolyte, Al current collector, and negative-electrode graphite may be used as fuel or reductant on the spot, which can lead to savings in both money and energy [85]. Subsequently, the smelter is supplemented with slag-forming agents such as (CaO) and SiO_2 to separate the non-reducible components such as Li, Mn, and Al from the alloy portion. Mostly pyrometallurgical techniques are often used for the industrial recycling of LIBs [52]. The recovery of transition metals such as Ni, Co, and Cu is efficient, but elements like Li and Al are lost in the slag [86]. From an ecological perspective, this procedure is not favorable since it requires a significant amount of energy and specialized equipment.

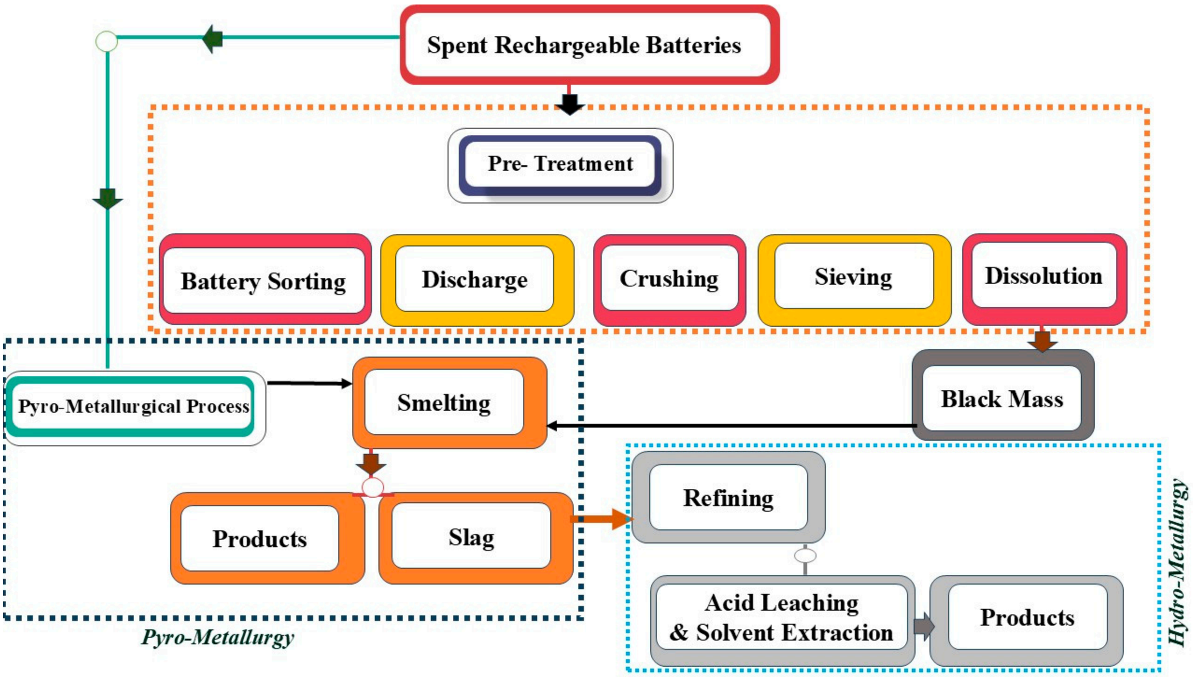

Figure 5. Pyrometallurgical recycling layout to recover the minerals.

Hydrometallurgical procedures such as leaching, solvent extraction, and precipitation are used for metal extraction [87]. Pyrometallurgical methods have limited flexibility since they demand significant economic expenditures and include complicated procedures for extracting metals [88]. Additional drawbacks of these methods include restricted storage capabilities, massive energy consumption, and suboptimal recycling efficiency [89]. One benefit of these methods is their resilience, as they need very little preprocessing and conditioning of the input material, while avoiding several possible issues such as gas emission and loss of minerals in slag formation, etc. [89,90].

One significant disadvantage of pyrolysis operations, which are carried out at temperatures ranging from 300 °C to 900 °C in inert atmospheres, is the absence of selective recovery. Although pyrolysis can break down organic materials, it is unable to selectively extract high-value metals from LIBs, leaving behind a blend of low-value elements [91]. In addition, pyrolysis is not an environmentally or economically viable process because expensive equipment is required, processes are energy-intensive, and dangerous gases are produced.

Pyrolysis is ineffective in the initial stages and raises operating costs as treatment times are extended. Li recovery presents similar difficulties during smelting, which takes place in air atmospheres at temperatures ranging from 1400 °C to 1700 °C. Even though Li can reach high temperatures that are favorable for metal extraction, smelting procedures frequently result in low Li recovery rates, requiring the inclusion of metal alloys to speed up the recovery process [91]. The smelting and roasting process is made more difficult and expensive by this additional step, which reduces its overall viability for large-scale metal recovery from LIBs and Li_2CO_3 as a common compound. The advancement of the effective and sustainable recycling of valuable metals from electronic waste streams, such as wasted LIBs, depends on pyrometallurgical processes overcoming these limitations [92].

3.1.3. Bio-Metallurgy

Electrode materials may be bio-metallurgically extracted by utilizing the oxidative and reductive capabilities of microbes [85]. In the past decades, microbial leaching as a method for removing metal-rich sulfides or low-grade ores has gained a lot of traction [93]. Bioleaching is an innovative technique used to extract metals from various solid materials. The capacity of certain microbes to transform solid elements into metals that can be extracted is a determining factor in this process [94]. The most common types of microbes that may dissolve metals include fungi, as well as bacteria like *Aspergillus*, *Thiobacillus ferrooxidans*, and *T. thiooxidans*, and yeasts like *Penicillium* and *Thiobacillus* [85]. In contrast to bacterial leaching, fungal leaching is more resistant to potentially dangerous chemicals. Recently, research was conducted into the bioleaching of *Aspergillus niger* from spent LIBs in a variety of settings. The aim was to recycle metals, and the findings demonstrated that Ni (38%), Co (45%), Al (65%), Mn (70%), Li (95%), and Cu (100%) were successfully retrieved [95,96]. Bioleaching techniques that use microorganisms as leaching agents have several benefits. For example, bioleaching demonstrates high extraction efficiency and low operating expenses, and it is particularly successful in extracting low amounts of Li from used batteries [97].

The management of environmental risks and regulatory compliance are two major obstacles that bio-metallurgy faces, despite its potential benefits. Concerns over the release of genetically modified organisms (GMOs) into the environment and the possible ecological effects of introducing non-native microbial species into natural ecosystems are raised on the topic of using microorganisms in bioleaching procedures. If waste and byproducts from bioleaching processes are not adequately managed, they may spread infections or harmful compounds that endanger human health and the environment [98]. Bio-metallurgical processes are also difficult to adopt commercially because different regions have different regulatory frameworks for the use of GMOs and the disposal of bioleaching waste. To guarantee the sustainable and ethical application of bioleaching technology, it is imperative to tackle these environmental and regulatory obstacles.

4. Green Organic Solvents: A Paradigm Shift in Mineral Extraction

Environmentally friendly solvents are sometimes referred to as "green organic solvents" due to their exceptional biodegradability, renewability, and recyclability [13]. According to several studies, these solvents are very stable, easily soluble, non-flammable, and non-toxic [99]. Green organic sorbents often originate from renewable sources like biomass, bio-based feedstock, or agricultural waste. As a result, we rely less on fossil fuels and may utilize solvents in a more environmentally friendly way [100]. Furthermore, green solvents have lower vapor pressures and cause fewer air pollutants, which lower ground-level air pollution and ozone depletion [101]. Table 2 provides the physiochemical characteristics of certain green organic solvents.

Table 2. Properties of green organic solvents including chemical formulas, molecular weights, densities, melting points, and levels of solubility in water.

Properties of Some Green Organic Solvents					
Green Organic Solvent	**Chemical Formula**	**Molecular Weight (g/mol)**	**Density at 20 °C (g/cm^3)**	**Melting Point at 1 atm (°C)**	**Solubility in Water (g/L)**
Palm Oil	$C_{16}H_{32}O_2$	846.1	0.892 (50 °C)	35	0.23
PFAD	$C_{12}H_{24}O_2$	265.5	0.861 (60 °C)	43	0.104
1-Octanol	$C_8H_{18}O$	130.23	0.827	−15	0.096
Span 80	$C_{24}H_{44}O_6$	428.61	1.068	11	Insoluble
Ethyl Acetate	$C_4H_8O_2$	88.10	0.902	−83	1:10 Less soluble
d-Limonene	$C_{10}H_{16}$	136.24	0.856	−74	Insoluble
Methyl Oleate	$C_{19}H_{36}O_2$	296.49	0.880	−3	0.01
Ethylene Glycol	$C_2H_6O_2$	62.07	1.113	−13	Miscible
N-Methyl-2-Pyrrolidone	C_5H_9NO	99.1	1.02	−2	Miscible

Source: [99,102,103].

Green solvents are highly valued for their environmental friendliness and sustainability, encompassing a range of materials, such as water, bio-based solvents, DESs, and supercritical fluids, and their combinations [99]. DESs have emerged as promising alternatives to conventional solvents due to their low vapor pressure, non-flammability, superior chemical and thermal stability, tunability, ease of preparation, cost-effectiveness, and potential for applications like metal recovery [102].

However, some DES formulations have raised concerns regarding poor biodegradability and potential toxicity, attributed to specific cationic groups, side-chain lengths, and anions [100,101]. To overcome these limitations, ongoing research is focused on developing new DES formulations that enhance biodegradability and minimize toxicity while retaining their beneficial properties. These advancements aim to improve DES safety and sustainability, particularly for applications in metal recovery, supporting green chemistry and circular economy initiatives.

Incorporating environmentally acceptable solvents in mineral extraction provides a sustainable and eco-friendly approach to obtaining critical minerals from natural sources. Green solvents can enhance the efficiency of critical mineral recovery while reducing reliance on hazardous chemicals, fostering a circular economy for these essential resources [103]. For example, DESs containing iodine have proven effective for dissolving sulfide minerals [104]. Ethylene glycol (EG) is considered a preferred environmentally friendly solvent, as noted in the CHEM21 selection guide and the Global Harmonized System (GHS) [105]. Additionally, in a green solvometallurgical process for extracting copper from sulfidic ores, $FeCl_3$ combined with ethyl glycerol has been identified as an effective oxidizing agent [106]. DESs have also demonstrated significant potential in recycling LIBs, offering a more environmentally friendly alternative to traditional mineral acids [107,108]. Although there are different types of green solvents, here, we focus on acids, functional ionic solvents, and DESs for mineral extraction. Table 3 demonstrates different types of green solvents and their properties and applications.

Table 3. Classification of green solvents based on their properties, applications, and roles in mineral extraction.

Green Solvents	**Description**	**Properties**	**Role in Mineral Extraction**	**Other Applications**	**References**
Bio-based Solvents	Derived from renewable biomass sources, such as plants, algae, or microorganisms	Non-toxic, biodegradable, and with a low VOC content. They offer favorable health and environmental profiles compared to petroleum-based solvents	Extraction of critical minerals from spent batteries through bioleaching	Applications in various industries, including extraction processes, cleaning agents, coatings, and personal care products	[104]
Water-based Solvents	Water as the primary component, often supplemented with other solvents or additives	Non-toxic, non-flammable, and have low VOC emissions. They are readily available, inexpensive, and have high heat capacity	Extraction of mineral matter from organic compounds	Extensively used in industries such as paints, coatings, adhesives, cleaning products, and textile manufacturing	[105,106]
Deep Eutectic Solvents	Combination of hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA); these can be either bio-based or non-bio-based compounds	Low toxicity, low volatility, and high thermal stability. Specific characteristics (tunable polarity, viscosity, and solubility)	Direct extraction of copper from copper sulfide minerals, recovery of mineral from spent batteries and e-waste, and green leaching of chalcopyrite, sulfide ore, gold, and silver ore	Applications across various industries and processes such as catalysis and electrochemistry, and as reaction media for organic synthesis	[107,108]
Supercritical Fluids	Pressure and temperature are above their respective critical values	Typical thermophysical properties of SCFs are low viscosity, high diffusivity, and density and a dielectric constant that can easily be changed by varying the operating pressure and/or temperature	Mineral recovery from spirulina microalgae and rare earth elements	Many applications in various fields, including pharmaceuticals, food processing, and materials science	[109,110]
Ionic Liquids	Organic or inorganic cations (like imidazolium or pyridinium) and anions (e.g., nitrate, acetate) that are liquid at or below 100 °C	Low vapor pressure, high thermal stability, and tunable polarity	Green leaching of chalcopyrite, sulfide ore, gold, and silver ore. Extractions of gold (Au (III)), silver (Ag (I)), palladium (Pd (II)), and platinum (Pt (IV))	ILs can be used in various areas from chemistry to engineering through the medical and the pharmaceutical fields	[111,112]

4.1. Harnessing Organic Acids for Sustainable Extraction Practices

Acids play a crucial role in extracting minerals from different substances, and most of the environmentally conscious research on sustainable mineral recovery methods centers around leaching agents. The acid utilized for leaching can consist of either organic acid (such as citric acid or succinic acid) or inorganic acid (such as sulfuric acid or nitric acid) [113]. Inorganic sulfuric acid is a commonly used chemical reagent for leaching different minerals, including non-ferrous metals from polymetallic tailings [114].

Organic acids, such as oxalic acid, combined with sulfuric acid solutions have been used to extract manganese from low-grade ores. These organic acids have shown effectiveness in selectively extracting calcareous elements from low-grade phosphate ores, suggesting potential for increasing ore value [115]. The choice of acid significantly impacts the leaching process. For example, the dissolution kinetics of smithsonite were examined in sulfamic acid solution, highlighting the importance of the specific acid used in the leaching process. Different acids, including sulfuric, acetic, and oxalic acids, have varying effects on iron mobilization from dust source materials, emphasizing the importance of acid selection in mineral extraction processes [116].

While inorganic acids are effective in facilitating metal recovery, their usage has significant environmental consequences. This is mostly due to the substantial water consumption, emission of gases such as Cl_2, SO_3, and NOx, and generation of secondary pollutants during the treatment process. Their utilization further involves corrosion-resistant equipment, as well as expenditure of financial resources and energy [117]. On the other hand, microorganisms synthesize organic acids, which pose a lower risk to operators and equipment. These acids also enable the targeted extraction of metals. Additionally, they are easily broken down in both aerobic and anaerobic conditions and do not produce gases, making their use in leaching processes environmentally sustainable. Although their use is more expensive, it is cost-effective since it circumvents the issues and constraints associated with inorganic acids [118]. In this review, our emphasis lies solely on organic acids for mineral extraction due to their superior eco-friendliness compared to inorganic acids.

4.1.1. Organic Acids and Mineral Extraction

Organic acid is an organic compound exhibiting an acidic pH due to the presence of functional groups, including carboxyl (-COOH) and hydroxyl (-OH), which determines the strength of its effects. The four main features that categorize organic acids are their carbon chain type (aromatic, aliphatic, alicyclic, or heterocyclic), saturation level (high, low, or unsaturated), substitution status, and number of functional groups (mono-, di-, or tri-carboxylic) [119]. One advantage of organic acids is that they break down naturally, leading to less run-off in the environment. In addition, they are recyclable and show excellent metal selectivity [120]. In contrast, organic molecules have shown remarkable reductive capabilities, especially in the processing of ores, and they are renewable, cheap, and ecologically benign [121].

Leaching, chelating, and precipitation are all roles they may play due to their capacity to contribute hydrogen ions and form metal complexes. When it comes to binding lanthanides from different sources including coal ash, neodymium magnets, and red mud, citric acid has been shown to be the most successful of the known acids. This is because its structure has three carboxylic acid groups and one hydroxyl group [122]. The use of organic acids, such as leachates, in mineral and metal recovery is on the rise. These acids include citric, tartaric, succinic, and malic acids [123,124]. Some examples of waste products that may be used to recover valuable minerals include used catalysts, old batteries, and low-grade mineral ores. Oxalic acid has been shown to be effective in this regard [125]. Using a solution containing 30 g/L of oxalic acid in a 0.75 molar (M) sulfuric acid (H_2SO_4) medium has been shown to achieve a leaching efficiency of around 90.49% for Mn and 6.78% for iron (Fe) when leaching manganic-ferrous ore [126].

Similarly, a study indicated that 65% of light rare earth elements (LREEs) and 19% of heavy rare earth elements (HREEs) could be extracted in a single stage using a 5% tartaric acid concentration, all without the use of mineral acids. The most effective method for extracting rare earth element (REE) minerals from phosphor-gypsum is to use organic acids generated from *A. niger* [127]. These acids include citric acid, gluconic acid, and oxalic acid. Under perfect leaching circumstances, they may attain a maximum leaching rate of 74% [128]. One typical chemical leaching method is the extraction of low-grade rare earth minerals via the use of inorganic acids such as sulfuric acid. However, an environmentally friendly alternative to this method is available, which involves little investment and low energy usage [129]. Table 4 shows the different organic acids and their efficiency in the leaching process.

Table 4. Organic acids and their efficiency in leaching mineral resources.

Material	Inorganic Acids	Side Effects of Inorganic Acids	Alternative Organic Acid	Leaching Efficiency	References
Ion-adsorption-type Rare Earth Ores (IAREOs)	($(NH_4)_2SO_4$), ($MgSO_4$)	- Residues in the soil of mining areas (ranging from 200 to 1000 mg/L). - Groundwater contamination. - Soil acidification. - $MgSO_4$ lead to soil salinization, compaction, and Mg^{2+} accumulation.	Citric acid	96%	[27,130]
Saprolitic Ores	H_2SO_4, HNO_3, HCl	- High acid consumption. - Leaching toxicity.	Citric acid	72%	[131]
Lateritic Nickel Ore	H_3PO_4	- Release toxic gases (Cl_2, SO_3, and NOx). - Waste acid solution.	Oxalic acid	Ni (89.63%), Co (82.89%), Fe (69.63%)	[132]
Spent Lithium-Ion Batteries	H_2SO_4, HCl, HNO_3	- Release toxic gases like Cl_2, SO_3, and NOx. - Waste acid solutions are harmful to the environment.	Malic acid, citric acid, tartaric acid, succinic acid	Co and Li (99%)	[133]

4.1.2. Organic Acid Leaching Mechanisms

Organic acid–mineral resource interactions may be understood via acid hydrolysis, organic acid chelation, and the participation of other organic ligands [134]. Various processes may lead to the dissolution of mineral resources, including complex-lysis, acidolysis, and redox reactions, among others [135]. The protonation of organic acid oxygen atoms is the primary mechanism by which acids undergo acidolysis. When the acid dissociates, the oxygen in the metal complex may be protonated with relative ease. Extraction of metal ions from metal ore surfaces is made possible when protons connect to and react with the ore's surface, weakening the bonds and demonstrating this phenomenon. The trace mineral separates from the solid's surface as a result of the reaction between water and the protons and oxygen [136].

In organic acid chelation, elements that have been leached in the past are complexed with organic acids to produce soluble metal complexes [137]. Instead of metal chelates, organic acids may dissolve the bond between surface metals and the leached material [138]. Furthermore, organic acids can supply protons during the process of leaching and decrease the concentration of ore in a solution by engaging in ligand reactions. Over time, additional minerals are discharged into the solution and then retrieved by diverse techniques [139].

4.2. Advancements in Functional Ionic Solvents for Eco-Friendly Extraction

The use of ILs as diluents and/or extractants instead of volatile organic compounds is a new and fast-growing technique for separation [69]. A class of organic salts known as ILs contain both inorganic and organic anions, as well as organic cations like tetraalkylammonium and tetra-alkyl-phosphonium, along with inorganic or organic tetrafluoroborate, trifluoromethyl sulphonate, acetate, nitrate, and halide. The melting points of these compounds are lower than 100 °C [140]. Since ILs do not release acid, traditional extractants would have to be neutralized afterward since the raffinate would include hydrogen ions

(H+). This is why ILs are better [141]. As a result of their low vapor pressure, wide range of operating temperatures in the liquid state, high solubility of both inorganic and organic substances, and high chemical, thermal, and electrochemical stability, which reduces potential health risks, ILs have long been categorized as environmentally friendly solvents. They can also be mixed with water to the desired levels [142]. Recent studies have clearly reported that certain ILs have significant harmful effects on various organisms and stages of the food chain [143].

Extraction of Minerals with Ionic Liquids

Several operations in the mineral- and metal-processing industry have been exploring the use of ILs, including solvent extraction, froth flotation, and mineral leaching [144]. A new approach of collecting quartz from hematite via froth flotation has been investigated; it involves the use of ILs comprising ammonium, imidazolium, and pyridinium [145]. ILs demonstrated a significant ability to selectively dissolve metals and metal oxides, which can create novel possibilities for ion metallurgy and mineral processing [146]. Minerals and metal oxides may be dissolved in ILs due to their coordinating-ion functionalities or oxide-binding species [144]. Ore and waste mineral processing has also made use of ionic liquids as leaching agents to extract certain metals from their native matrix [147].

It is worth mentioning that bifunctional ILs have been shown to be more effective than conventional extractants and other ILs. This is because the extraction process employs both cations and anions from the ILs [148].

(A) Mineral flotation with ionic liquids

Flotation of minerals is an important and extensively utilized process in the mineral-processing industry. The advent of this technique has greatly improved mineral resource exploitation and productivity, leading to a dramatic decrease in the minimum grade needed for mineral resource development. As a result, the world's economy is better able to rely on the availability of natural resources [149]. An essential aspect of achieving successful mineral flotation is the careful selection of highly effective and discriminating flotation reagents [150].

Over the past few years, numerous effective flotation chemicals have been created and advanced for various mineral-processing purposes. Activators, foaming agents, collectors, inhibitors, dispersants, and modifiers are some of the reagents used in the process of extracting valuable minerals from ores [151]. Significant progress has been made in the processing of minerals that are difficult to float by using ILs, a class of green solvents characterized by a variable structure, lack of flammability, low vapor pressure, and almost nonvolatile characteristics [152]. One can utilize 1-butyl-3-methyl imidazolium bis (trifluoromethyl-sulfonyl)-imide ([Bmim]Ntf2) and acidic N-dimethylacetamide bis(trifluoromethyl-sulfonyl)imide ([DMAH]Ntf2) ILs for the process of separating the most concentrated material and producing minerals similar to Bast ($RECO_3F$) as well as RE_2O_3 and $RE_2(CO_3)_3$ compounds [153].

In an aqueous solution, the tetrabutyl bis(2-ethylhexyl)ammonium phosphate ([N4444][DEHP]) ionic liquid was used to test the flotation behavior of model monazite and bastnäsite minerals. The collectors used in the experiments were Aero-6493 (containing 6–10 carbon alkyl chains) and Aero-9849 (containing 10–18 carbon alkyl chains) [154]. A combination of micro-flotation methods, zeta potential measurements, Fourier-transform infrared spectroscopy (FTIR), and X-ray photoelectron spectroscopy (XPS) was used to analyze the flotation test findings. The micro-flotation tests on the two minerals reveal that [N4444][DEHP] exhibits a superior ability to collect minerals compared to the typical hydroxamic acid collector. Similarly, 1-propyl-3-methylimidazolium bromide (propyIMIMBr) ionic liquid shows efficiency in the recovery of barite minerals. Therefore, the propyIMIMBr ionic liquid can enhance the recovery of valuable materials from drilling waste and has the potential for reuse in other applications [26,155].

In addition to earlier research, there is an emerging polymerization trend of ionic acid monomers, for example, imidazolium-SO_3H^- heteropolyanion, poly-4-vinlpyridine, divinylbenzene, and resorcinol formaldehyde, as given in Figure 6a. Then, Figure 6b presents the general synthesis method, where -SO_3H functionalized heteropolyanion is prepared via polymerization of a zwitterionic monomer [156]. Grafting the imidazolium polysulfonic acid—Brönsted acidic, ionic liquids (BAILs)—on a silica top surface using chlorophyll silica also makes it easier to access the acidic function [157], as given in two steps in Figure 6c. In the preceding research works, the use of ILs in the flotation of various minerals has also been researched, and outstanding results have been achieved.

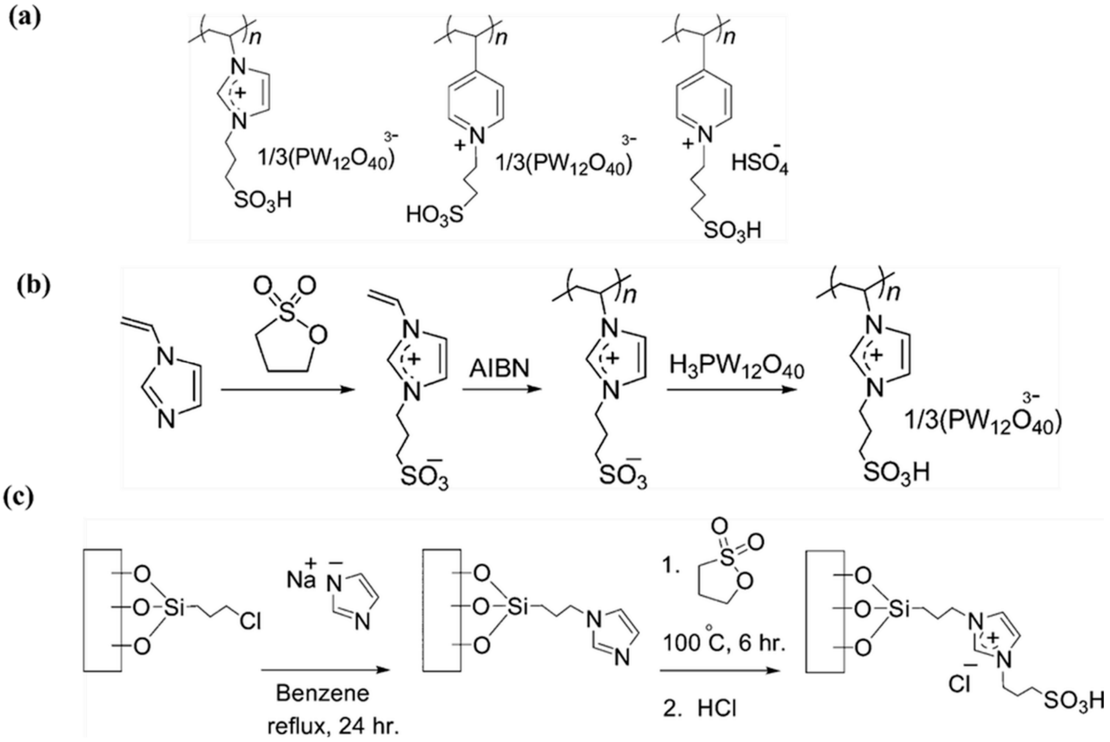

Figure 6. Innovations in ionic liquid: (**a**) common polymeric, acidic, ionic liquids, (**b**) polymeric, acidic, ionic liquid synthesis process [156], and (**c**) sulfonic-acid-functionalized acidic, ionic liquid synthesis by grafting [157,158]. Reprinted with permission from reference [158]. Copyright 2016, American Chemical Society.

(B) Mineral leaching with Ionic Liquids

These eco-friendly chemicals are being used in pretreatment and leaching, which are the primary steps of processing mineral ore. The significant acidifying properties of some inorganic compounds (including ILs) have prompted an investigation into leaching [159]. Mineral leaching has emerged as a significant technique for separating and extracting several precious elements due to its benefits of being environmentally friendly, easy to perform, energy-efficient, and cost-effective [20]. The utilization of ILs in the process of leaching offers numerous benefits in comparison to traditional hydrometallurgical leaching methods. ILs can be used instead of mineral acids to dissolve metals, offering more selectivity for the desired metals compared to the secondary impurities present in the solid materials. This typically enables a decrease in the requirements for future separation and purification processes, resulting in reduced consumption of reagents [160].

Chalcopyrite is a prevalent kind of copper sulfide ore, known for being particularly challenging to extract Cu from, details are given in Table 5. The advent of ILs offers a novel potential approach to accomplish this objective [161]. Using two acidic ILs based on imidazoles, namely [Bmim]HSO_4 and [Hmim]HSO_4 (1-hexyl-3-methyl-imidazolium hydrogen sulphate), speeds up the leaching of chalcopyrite. After 60 °C, the Cu recovery rate may reach 70%, which is a significant improvement [142].

Similarly, industrial brass ash was leached using the ionic liquid [Bmim]HSO_4 to recover Cu and Zn. With the temperature maintained at 70 °C, the Cu dissolution reached 25% and the zinc dissolution reached nearly 99% [162,163]. The synthesized imidazolyl cyanate ionic liquids exhibited a notable capacity to dissolve gold, with [C_2MIM][OCN] demonstrating a superior efficacy in extracting gold from mineral deposits due to its low viscosity and minimal steric hindrance [164].

Table 5. Leaching with ILs for critical minerals.

Material	Used IL	Conditions			Leaching Yields (%)	References
		Temperature	Time (h)	L/S (L/kg)		
Bastnasite ore	$[Hbet]^+ [NTf_2]^-$ with 30–50 v% water	50–90 °C	24	5–20	40–100% HREE and 65–100% LREE	[165]
Pyrite ore	$[C_4MIM]^+ [HSO_4]^-$ with Thiourea (as complexant, 20 g/kg ore) and ferric sulfate (as oxidant, 0.5 g/kg ore)	25 °C	48	4	85% Au and 60% Ag	[166]
Chalcopyrite	([HMIm]HSO_4) with 20% H_2O_2	45 °C	2	10	98.3%	[167]
Brass waste	$[C_2MIM]^+ [HSO_4]^-$ 20–80 v% in water with H_2O_2 (as oxidant) 20 v%	40–80 °C	2	10	100% Zn 40–50% Cu	[168]

4.3. Deep Eutectic Solvents, Kinetics, Mechanism, and Machine Learning Intrusion

A new class of eco-friendly solvents called DESs was introduced by Abbott in 2003 [169]. Along with ILs, they have remarkable attributes, such as cost-effectiveness, an environmentally friendly nature, low volatility, biodegradability, and ease of synthesis [170]. Metal electrodeposition, metal recovery, extraction, biocatalytic activity, organic synthesis, and related uses are among their primary applications [171]. More recently, DESs have shown potential as replacements for inorganic and organic acids and as a leaching agent to remove metals from old batteries. They find widespread use in metal separation and recovery processes because of their high solubility in various metal materials [172].

Mineral recovery with DESs is driven by their capacity to improve separation and extraction efficiency as well as environmental issues. Strong coordination with metal ions and dissolution of metal–oxygen connections are made possible by the fact that DESs have a separate hydrogen bond donor and acceptor. This allows for the complete dissolution of a number of metal oxides that are poorly soluble in water. Altering the system's acidity and alkalinity may improve its solubility. In addition, DESs have unique reducibility, which means that additional reducing agents are not needed during leaching. The addition of oxidants enables the catalytic dissolution of metals in DESs. An important advantage of utilizing DESs in this research field is avoiding the need for an additional step, such as a lowering agent [173].

4.3.1. Types of Deep Eutectic Solvents

There are several types of DESs, such as Cat+, which may be any cation of the ammonium, phosphonium, or sulphonium type; X, which is a Lewis base, and Y, which is a Lewis acid; this is the conventional way of representing the DES composition. X and Y form a complex, with z being the number of Y molecules interacting with an anion [174]. A wide variety of DESs based on complex chemicals have been created.

Figure 7 shows the five types separated into their component parts. Similar to composites of metal halides and imidazolium salts, Type I DESs are produced by mixing quaternary ammonium salts with metal chlorides [175]. Metal chloride hydrates and quaternary ammonium salts make up the second category of DESs. Choline chloride (ChCl) with hydrogen HBDs including alcohols, amides, polysaccharides, and others makes up Type III DESs. Because of their adaptability and capacity to dissolve transition metal species, these solvents have attracted a lot of interest from researchers. Depending on the HBD used in the combination, the physical properties of these DESs may be altered. HBD and metal chlorides are the building blocks of Type IV DESs. HBD and HBA, which are non-ionic molecular entities, are forming a new class of DES called Type V. This family of DESs is characterized by the abundance of hydrogen bonds [176,177].

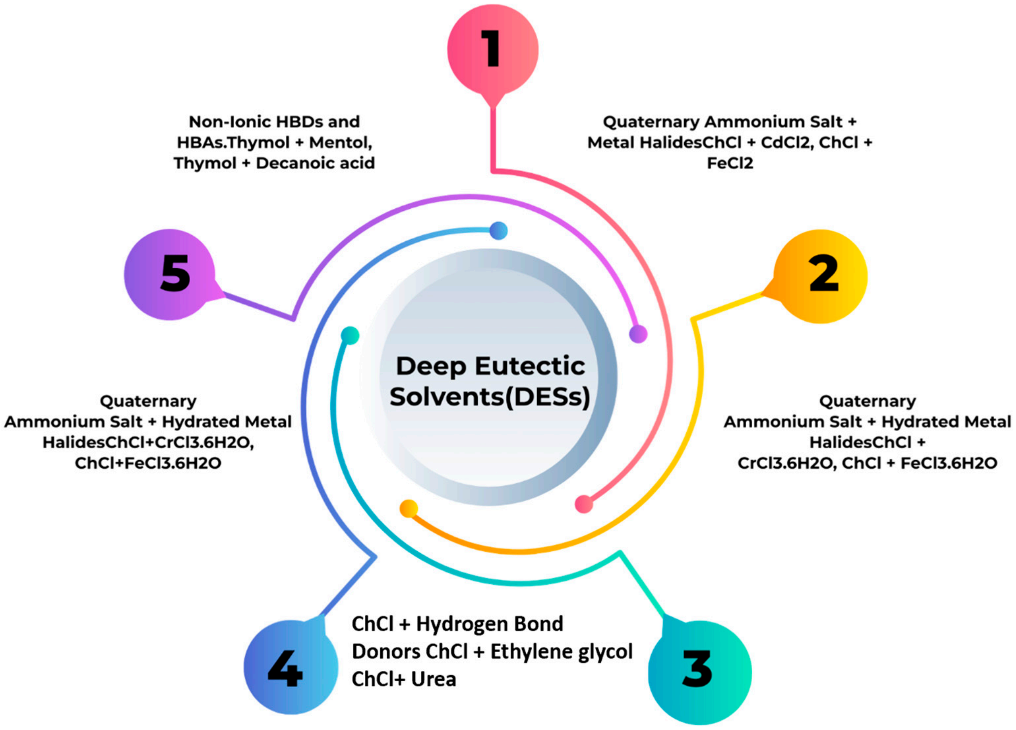

Figure 7. Deep eutectic solvents with their five different types.

4.3.2. Preparation of Deep Eutectic Solvents and Mechanism of Mineral Recovery

In many cases, DES synthesis is straightforward, whereby HBDs and HBAs are combined at temperatures usually between 50 and 100 °C during the synthesis of these compounds. After allowing enough time for the hydrogen bonds to develop, the temperature is lowered. Two distinct approaches to combining the parts have been considered. To melt the component with the lowest melting point first and then add the component with the highest melting point is one way to work it. Alternatively, the mixing and initiating processes of two substances with similar high melting points occur concurrently [178].

Recently, research has been conducted on the mechanics involved in the separation of minerals using DESs. DESs interact with the mineral surface through a combination of physical and chemical processes via hydrogen bonding [179]. When DESs interact with a mineral surface, the process of dissolving the metal oxide of the minerals can be considered as two steps [180]. First, the carboxyl and hydroxyl groups of organic acids and alcohols, as well as the protons from HBDs, react with the active OH sites on the hydrated metal oxide.

Protonated oxide-containing intermediate species are formed because of this reaction. The following chemical equations (Equations (1) and (2)) describe this procedure:

$$\mathrm{Bulk} \equiv \mathrm{Me} - \mathrm{OH} + \mathrm{HX} \leftrightarrow \mathrm{bulk} \equiv \mathrm{Me} - \mathrm{OH_2} + \cdots \mathrm{X^{n-}} \tag{1}$$

where "Bulk $\equiv$ Me $-$ OH" stands for the hydrated metal oxide with active OH sites, and HX stands for the HBDs of DESs.

In the second step, protons will break the bonds between the metal and oxide if the complexes with ligands are more stable than the ones with OH. The OH active sites will be replaced by the deprotonated HBD ligand via metal–ligand complexation. After that, in the bulk DESs, the ligand will be swapped out for an anion, such as chloride, $\mathrm{Cl^-}$:

$$\mathrm{Bulk} \equiv \mathrm{Me} - \mathrm{OH_2} + \cdots \mathrm{X^{n-}} \leftrightarrow \mathrm{Me} - \mathrm{X_{(DES)^{n-}}} + \cdots \mathrm{H_2O} \tag{2}$$

4.3.3. Mineral Extraction with Deep-Eutectic-Solvent-Based Leaching

In leaching, a powdered material is subjected to acid or basic solutions in order to dissolve the metals, while some impurities remain insoluble. Two new subfields of hydrometallurgy, solvo-metallurgy and iono-metallurgy, have emerged due to DESs. Non-aqueous solutions combining Lewis and Bronsted acids and bases are used in solvo-metallurgy, which is different from hydrometallurgy. These mixtures are ideal for processing ores of lower grades and provide novel opportunities for natural resource extraction. Processing lower-quality ores is very necessary due to the depletion of high-quality raw materials that contain greater quantities of minerals [141]. DES-based leaching has eco-friendly features that make it safer for the environment and ensure that natural resources are handled responsibly [181].

The mining ores are put through DES leaching to recover minerals. The solvent is usually a mixture of an HBD and a hydrogen bond acceptor (HBA) [173]. The raw material is crushed and ground into mineral grains to a precise size appropriate for leaching, a process known as comminution. The leaching procedure may not have extracted enough of the critical mineral (antimony) from the source material if the grains were too big. It was also undesirable for the grains to be too small since this would have increased viscosity. Therefore, the ideal grain size was very important during leaching [83]. The crushed ores are then mixed with the DES solution, allowing the solvent to penetrate the mineral matrix and initiate the solvation process.

During the reaction of a DES, the HBD component solvates metal ions from the mineral surface, forming metal complexes, while the HBA component may also participate in complexation through coordination with the metal ions. This contact facilitates the release of metal ions from the mineral surface into the DES phase [182]. DES's special qualities, namely, its low volatility and high viscosity, help with mass transfer processes. The sample was filtered after the leaching procedures and then rinsed with distilled water at a particular ratio to the liquid eutectic mixture, as given in Figure 8 [183].

4.3.4. Machine Learning and Intrusion with Kinetics of Deep Eutectic Solvents

DESs have been used to extract a variety of minerals, such as bastnasite, lead, Cu, zinc sulfide, telluride, iron sulfide, and arsenide [184,185]. Recent studies explored the different types of DESs fabricated with ChCl, urea, EG, and a combination of those to recover Li, Ni, Mn, and Co at different temperatures. Li was efficiently recovered at 100 °C with ChCl and urea. The kinetics of chemical reactions are regulated by a thin ash/product layer when ChCl:EG and ChCl:urea are present. Conversely, kinetics highlight the leaching efficiency increase as temperature rises. Thus, the ChCl:EG DES reaction is under the influence of a mixed control effect. Additionally, NiO production on the surface is likely indicated by

Ni peaks in the EDS analysis, as in Figure 9. The particle surface is coated by NiO, which stops reagent diffusion across the surface.

Figure 8. Mineral extraction with DES-based leaching, initiating with choline chloride as the hydrogen bond acceptor and urea as the hydrogen bond donor.

In addition, DESs were applied for metal recovery and reclamation from various sources [172]. For example, using saprolite ore as an example, the direct mineral refining method first used a hydrophobic DES. By lowering the leaching of Mn, the DES proved to be more efficient than traditional sulfuric acid leaching in recovering Mn, Ni, and Co [186]. DESs were utilized for the extraction of gold-bearing ores and other minerals. By applying electrolysis to the DES, scientists have shown that selective dissolving is possible. The achieved dissolution rates met expectations, and the method was much less harmful to the environment than the cyanidation methods previously used [83].

In recent studies, it was reported that quantum and machine learning techniques were employed to estimate the phase diagrams of deep eutectic solvents (DESs) across an extensive chemical space. By pinpointing the eutectic point coordinates, one study highlighted the impact of melting properties and mole fractions on eutectic temperature (ET) [187]. Furthermore, molecular dynamics simulations at the eutectic points emphasized the role of hydrogen bonding in elucidating the contributions of individual components within the mixture. The findings underscore that computer-aided estimation of eutectic diagrams for novel extraction systems deserves special attention, offering a promising pathway for designing efficient and sustainable solvent systems [187].

In addition, three distinct DES types were tested using a sample taken from extraction waste produced during antimonite ore flotation. The leaching process was optimized by changing the quantities of the solid and liquid components and increased oxidized efficiency [63].

Temperature is crucial element that can influence the leaching percentage concentration of metals. In a recent study, different types of DESs with a combination of ChCl were used. The results revealed that, in most cases, a maximum concentration was observed at 100 °C and that ChCl with a combination of urea and EG was best for Li, Ni, and Mn concentration, as given in Figure 10.

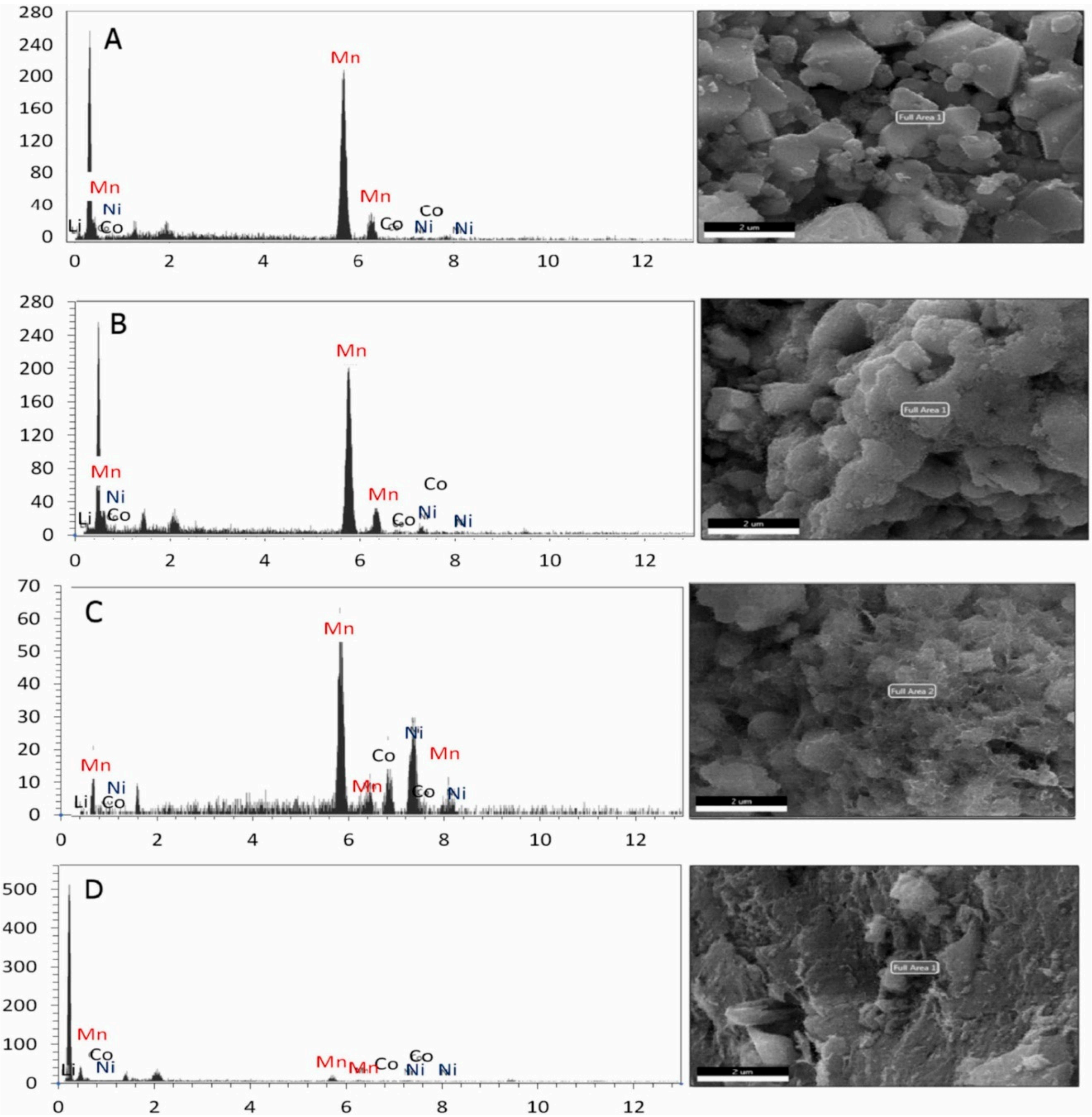

Figure 9. EDS analysis and SEM images from before (**A**) and after (**B**–**D**) the leaching process: (**A**) ChCl + urea, (**B**) ChCl + EG, (**C**) ChCl + urea + EG, (**D**) DES: (ChCl:urea 1:2, ChCl:EG 1:2, and ChCl:urea:EG 1:2:1 at 100 °C) [188]. Reprinted with permission from reference [188], Copyright 2022, Elsevier.

The graphs in Figure 11 show the actual concentration data for different metals at various temperatures, which were extracted from previous studies [92,189], along with predictions from linear and quadratic regression models. Each subplot represents a different metal (Li, Co, Ni, Mn), and within each subplot, the actual data points and the predictions from both regression models are plotted.

To enhance the credibility of the extracted data, a machine learning model was employed. The coefficient of determination (R^2) and mean squared error were determined by the following equation:

$$MSE = 1/\mathrm{n}\sum_{i=1}^{n}\left(Y_i - \hat{Y}_i\right)^2 \tag{3}$$

where n is the number of data points, Y_i is the observed value, and Y is the predicted value. According to the data, most of the DESs have an R^2 near 1, which highlights the accuracy of data, and similarly, the *MSE*, which is the average square difference between calculated and estimated values, elucidates the variability in different leaching extraction efficiencies. After predictions are made from the linear, quadratic model, the quantifying measures are calculated, i.e., *MSE* and R^2. This assesses the performance of the model for each respective metal, as given in Table 6.

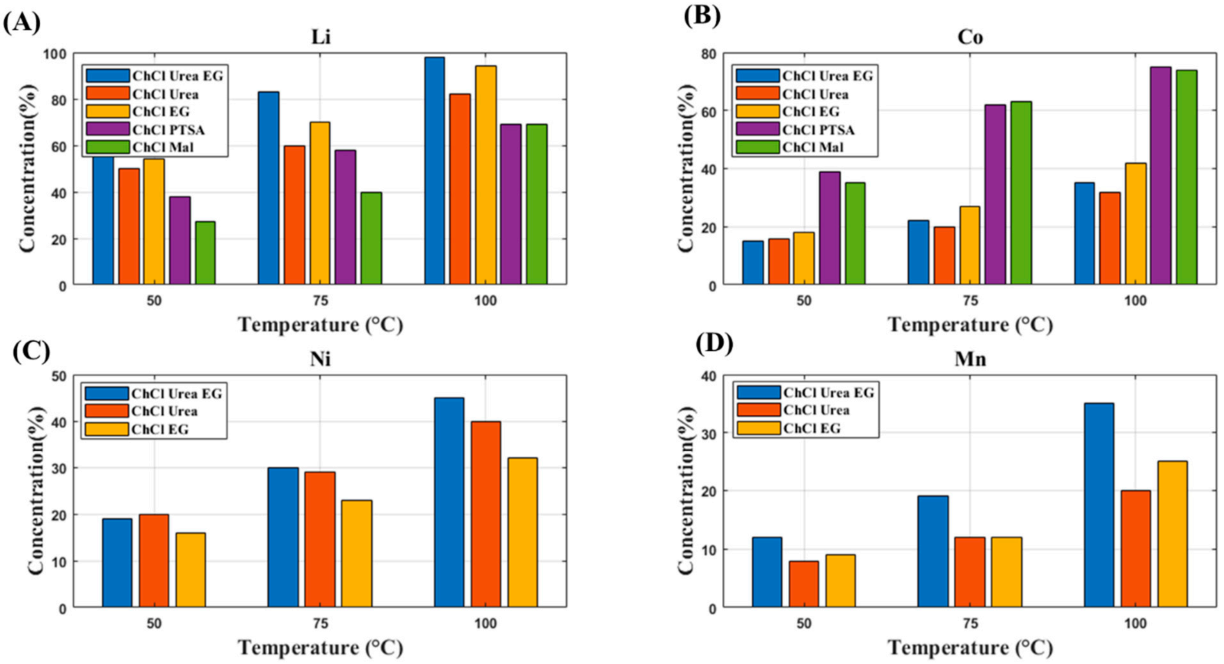

Figure 10. Effect of leaching temperature on mineral efficiency of three different types of DESs—ChCl, urea, and EG—and combination of urea and EG. (**A**) Percentage Li recovery, (**B**) percentage Co recovery, (**C**) percentage Ni recovery, and (**D**) percentage manganese recovery. Source: [99,102,103].

Table 6. Coefficients of determination and mean squared errors of DESs with their respective metals.

DES	Metal	MSE	R^2
ChCl + Urea + EG	Li	2.00	0.99
ChCl + Urea + EG	Mn	4.5	0.955
ChCl + Urea	Li	8.00	0.95
ChCl + Urea	Co	3.566	0.923
ChCl + Urea	Ni	0.22	0.99
ChCl + Urea	Mn	0.88	0.96
ChCl + EG	Li	3.55	0.98
ChCl + EG	Co	2.00	0.97
ChCl + EG	Ni	0.22	0.99
ChCl + EG	Mn	5.55	0.88
ChCl + PSTA	Li	4.50	0.97
ChCl + PSTA	Co	5.55	0.97
ChCl + Mal	Li	14.22	0.95
ChCl + Mal	Co	16.05	0.94

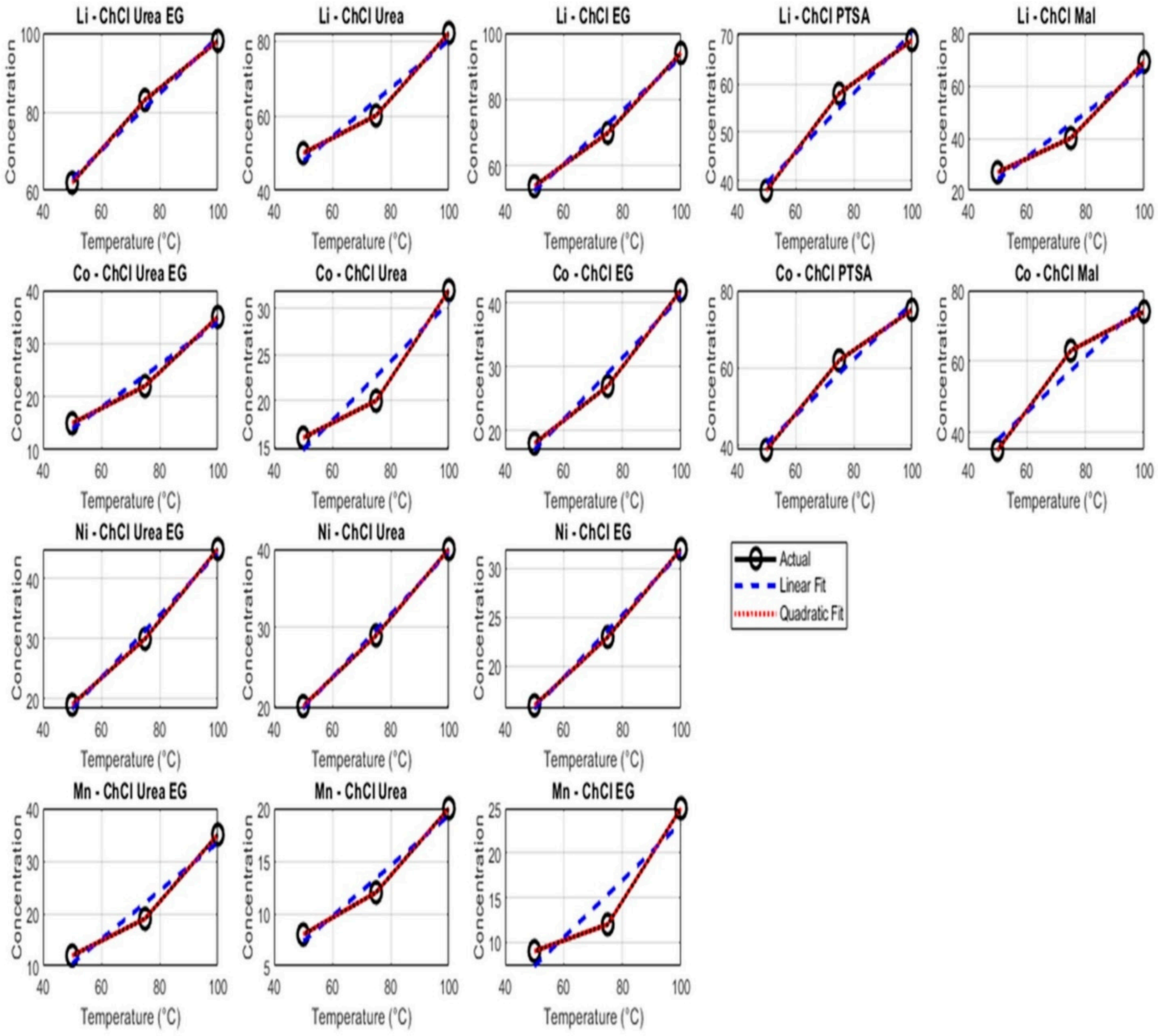

Figure 11. Percentage concentrations of respective metal combinations of DESs with fluctuating temperature (50, 75, and 100 °C), where black, blue, and red represent actual, linear fit, and quadratic fit values, respectively.

5. Innovations and Future Directions in Sustainable Mineral Extraction

5.1. Sustainable Development in Mineral Extraction

The contemporary world cannot function without sustainable development, which takes a comprehensive view of the interconnected problems of social, economic, and environmental welfare [190]. As a major factor impacting development, the mineral extraction industry is vital in the fight for sustainable development goals [191]. Given the mineral extraction industry's interconnectedness with global supply networks and resource exploitation, its practices greatly impact environmental preservation, social welfare, and economic progress.

Sustainable mineral extraction refers to the implementation of practices during excavation operations that lead to positive environmental and social outcomes compared to conventional resource development methods. It also prioritizes the well-being and safety of workers, as well as the concerns of stakeholders and affected communities [192]. Environmental education has a significant role in promoting broad change in the quest for sustainability. By integrating ecological education into curricula across all levels of education, ranging from elementary to higher education, society may cultivate a cohort of ecologically aware individuals who possess the requisite knowledge and abilities to address complex issues [193].

5.2. Technological Innovations in Extraction Efficiency

Currently, research is responsible for promoting the creative growth of the mineral extraction industry in nations that possess abundant natural resources [194]. Technology enables the use of Geographic Information System (GIS) operations to provide guidance to miners during exploratory activities and to assist in the assessment of the value of a specific ore. Moreover, the use of automated procedures, such as the utilization of trucks, ventilation systems, and site monitoring, has resulted in enhanced efficiency within the enterprise [195].

The mineral extraction sector has historically used technology to tackle challenges related to workplace efficiency and environmental difficulties [196]. The implementation and use of innovative extraction technologies have significantly augmented productivity, leading to an overall improvement in working conditions within the extraction and recycling sector. Technological advancements play a crucial role in driving the expansion of the recycling sector [197].

5.3. Advancements in Battery Recycling with Artificial Intelligence

The field of AI offers remarkable methods for accomplishing intelligent assembly to disassembly. AI is reviving and growing quickly with recent advancements in computer technology and algorithms, especially in machine learning (ML) tools [198,199]. Numerous intelligence techniques inspired by nature, including evolutionary computation and neural networks, endow machines with improved learning capabilities to handle complicated and nonlinear situations. AI is starting to show promise as a sustainability booster for two reasons.

The mineral extraction and battery industry has greatly benefited from artificial intelligence. To enable a sustainable closed-loop supply chain, recovery operations can be optimized with the use of intelligent robotics tools and equipment, networking, cloud services, and learning-based decision support systems [200,201].

In recent work, researchers computed a dataset of 1510 groups that can elicit symbolic visual and functional expressions using the genetic programming model (GPM). The findings showed that, because of various initial applied stresses, the GPM can be associated with better fitting (accuracy) for capacity prediction as a function of real-time stress. As a result, this GPM can be used to classify battery waste according to its residual energy [202,203]. To determine the solubility of Co ions, fourteen parameters and properties were analyzed, including viscosity, water content, molecular weight, and the hydrogen bond acceptor to hydrogen bond donor (HBA:HBD) ratio. The dissolution of the positive electrode can be facilitated and coordinated with an organic acid ion and Cl^-. In addition, the acidity of the solution, reducibility, and strength of coordination are directly correlated with the reaction potential and solubility of Co. Therefore, the predictive performance with Shapley additive explanation (SHAP) of different models is represented as R^2XGBoost > R^2others, and MSEXGBoost < MSEothers, revealing the effectiveness of XGBOOST [204].

Battery technology and mineral extraction are parallel processes. Recently, the Pacific Northwest National Laboratory (PNNL) and Microsoft Azure Quantum Elements developed a methodology and system where 32.6 million electrolytes are used in battery processing into eighteen usable electrolytes in just nine months. This paradigm shift converts 20 years of work into a short period [205,206]. Similarly, AI facilitates the process of material design; sixteen solid-state electrolytes with ionic conductivity greater than

10^{-4} S cm^{-1} were identified by ML training from 3000 compounds [207]. The study further used an artificial neural network model to estimate and predict the dielectric constant of complex and complicated electrolytes and concluded that there is a specific volcano pattern with an increasing Li ion content. These findings attest to the remarkable innovation in electrochemical and battery technology [208]. AI not only assists in the recovery of minerals from waste batteries but also aids in exploring and identifying new mineral resources. Based on its integration and AI projections, KoBold Metals has found and claimed 800 km of metal reserves in North Quebec, Canada, including Ni and Co [204]. Increased equipment utilization via continuous operation; automation, prediction, and precision; and exploration of new resources are positive aspects for the mineral extraction industry [209]. In addition, deep generative models can be developed on large, verifiable datasets to incorporate physics-driven restrictions in materials structures at both the atomic and continuum capacities by analyzing the underlying probability distributions in the input space [210]. Then, novel and creative yet still feasible designs can be created by employing trained models as potential battery materials [211].

6. Concluding Remarks

To summarize, this research emphasizes the need to switch from conventional to synergistic methods for mineral extraction, as well as the noteworthy developments and new trends in the creation of green organic solvents. Effective resource management and environmental problems can only be addressed by incorporating sustainable approaches into mineral extraction and battery recycling. Critical minerals like Ni, Cu, and Co play crucial roles in battery technology, and their extraction and recycling provide inherent obstacles that are highlighted in this manuscript. Global demand for cobalt is anticipated to reach 176,000 metric tons by 2030; in comparison, it is only anticipated that cobalt demand for non-battery uses will reach 65,000 metric tons worldwide by that year. Meanwhile, it is projected that LIB battery production will reach slightly less than 447,000 metric tons in 2021 and then quadruple by 2030. In a recent projection of 2050, the positive electrode, Co, and Ni are forecast to reach 22 MMT, 2.7 MMT, and 6.5 MMT, respectively, with an 80% recovery efficiency.

Conventional techniques, such as hydrometallurgy, pyrometallurgy, and bio-metallurgy cause substantial environmental hazards and have technological constraints despite their relative effectiveness. For example, hydrometallurgy typically involves a physical disassembly and separation approach that often results in black mass cross-contamination with organics such as the fluorine-containing PVDF binder and impure metals such as Al, Cu, and Ti. Meanwhile, in pyrometallurgy, Li has the capacity to achieve high temperatures that are advantageous for the extraction of metals; however, smelting processes usually lead to low rates of Li recovery, necessitating the addition of metal alloys to accelerate the recovery process. This extra stage increases the complexity and cost of the smelting process, decreasing its overall feasibility for large-scale metal recovery from LIBs.

The use of green solvents, like DESs and ionic liquids (ILs), is changing the paradigm and offering viable environmentally acceptable substitutes. Emerging organic solvents include palm oil, PFAD, 1-Octanol, and Span 80 due to their low solubility and temperature flexibility. The combination of different solvents such as 30 g/L of oxalic acid in 0.75 molar (M) sulfuric acid (H_2SO_4) can achieve the maximum leaching efficiency (approx. 91% for Mn). Organic acid obtained from *A. niger* can be used to extract rare earth minerals from phosphorus–gypsum ores. Recent studies confirm that reaction kinetics can influence the extraction and leaching process, e.g., four hours of leaching at 100 °C can achieve almost 100% iodine removal or uptake, which cannot be achieved with a low temperature and less time. Thus, the entry of machine learning into the mining industry may revolutionize mineral recovery, as demonstrated by data extracted from previous studies and computed values, which have coherence and significant R^2 and MSE results.

Sustainable extraction methods can benefit from these solvents' low toxicity, biodegradability, and minimal environmental impact. Essentially, the use of green organic solvents in mineral extraction not only helps the industry become more robust and ecologically sensitive but also supports global sustainability goals. Research is not just limited to rechargeable batteries; this method can also facilitate mineral extraction from other energy wastes. By moving forward with this, we can pave the way for a future that is resilient and equitable for coming generations. That hinges on us cooperating, encouraging creativity, and exhibiting a commitment to environmental stewardship.

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