Lithium Recovery from Lithium-Ion Battery

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Lithium-ion batteries are becoming an integral part of renewable-based energy systems that helps to provide an efficient and greener solution for energy storage. LIBs have found their use in a variety of applications ranging from portable electronic devices to energy grid systems. Owing to the reduction in carbon dioxide emission and improved energy to fuel weight ratio, LIBs have also been widely used in electronic vehicles. LIBs have been especially desirable in this case due to their high charge to mass potential in comparison to other battery types. In the recent decade, the extensive use of LIBs has posed not only a great threat to the world's lithium resource depletion but also the prevailing problem concerning the consumed and non-recycled LIBs. Hence, immediate attention to alleviate any danger to the ecosystems due to the release of harmful chemicals is required.

lithium lithium recovery and recycling membrane technologies lithium ion battery

1. Conventional Methods

Currently, the commercial processes used for recycling and refining of lithium and other metals (including nickel, copper, cobalt, and aluminium) from LIBs can be divided into two major categories: (i) pre-treatment processes and (ii) metal-extraction processes [1].

1.1. Pretreatment Process

In a typical pre-treatment process, the spent LIBs are firstly discharged using saturated-salt solutions (e.g., NaCl and Na2SO4 salt solution) to prevent short-circuiting or self-ignition caused by combustion [2]. Furthermore, it is recommended to recycle the electrolyte before the discharging stage. This is achieved by using organic solvent extraction or supercritical carbon dioxide to prevent the formation of hazardous vapors from electrolyte (LiPF6) and salt contact [3][4]. The use of supercritical carbon dioxide has proven to be more effective as it does not contaminate the electrolyte and the electrolyte recovery is significantly simplified [5][6][7]. Then, the obtained batteries are disassembled manually to separate the cathode from the anode to facilitate metal extraction and further processing [8]. Different solvents are in use to dissolve the organic binder to effectively separate the cathode from aluminium foil using the solvent dissolution method [9][10][11][12]. Zhou et al. have found 60 °C as an optimum temperature for effective removal of polyvinylidene fluoride (PVDF) binder through dissolution in dimethylformamide (DMF) [13]. Elsewhere, Zhang et al. used 15 vol% of trifluoroacetate (TFA) for dismantling the cathode from the aluminium foil through a solid-state reaction at relatively mild conditions of 40 °C for 180 min. The optimized liquid to solid (L/S) ratio was found to be 8 mL g^{-1} [14].

Another pre-treatment technique being used for the effective removal of strongly bonded PVDF from aluminium foil and the cathode material is ultrasonic-assisted separation [15][16][17]. This technique utilizes the combined effect of ultrasonic waves and agitation to induce a cavitation effect. Li et al. found that the separation efficiency increased significantly when agitation was coupled with ultrasonic treatment [17]. He et al. achieved a 99% separation using n-methyl pyrrolidone (NMP) as a solvent in conjunction with ultrasound waves [16]. Thermal treatment methods are also widely used for effective detachment of cathode from aluminium foil by high-temperature degradation of organic binder [18][19][20][21]. The temperature range for effective pyrolysis was recorded as 500–600 °C, however, a vacuum was applied to avoid high-temperature aluminium brittleness [21]. Although the thermal treatment method has proven to be highly productive in terms of operational efficiency and high throughput, it has a disadvantage in producing hazardous gases due to high-temperature decomposition reactions. To avoid high energy consumption, facile mechanical separation methods including crushing, grinding, sieving, and magnetic separation have been reported [22]. Shin et al. concluded that the separation efficiency of targeted metals can be enhanced by integrating mechanical methods before the metal-leaching process [23].

Both mechanical and thermal treatment methods have the advantages of being straightforward and convenient, however, suffer from producing hazardous chemicals (**Table 1**) [24]. Even though most pre-treatment processes have successfully been applied in different industries across the world, there are still great developments to be researched to improve the process. Such research should include methods that are not only economically feasible but simultaneously reduce the environmental footprints.

Table 1. Summary of strengths and weaknesses of using old conventional methodologies for Li harvesting from Sea-water brines and LIBs.

Techniques/Processes	Strengths	Weaknesses
Conventional technologies for Lithium extraction from Brines/Seawater		
Precipitation	Simple Process, Green energy source (solar evaporation)	Time-consuming, A high volume of waste
Solvent Extraction	simple, adaptable, and continuous operation	A high volume of waste, expensive co-agents, highly corrosive solvents, Toxic material formations
Adsorption	Simple operation, low energy consumption. Adaptable	Time-consuming, adsorbents are expensive, powdery and easily degrade in acid-driven desorption
Electrodialysis	Tailorable for Li production	Time-consuming, hazardous and corrosive materials
Pre-treatment technologies for Lithium recycling from spent Lithium-Ion Batteries		
Solvent dissolution	High separation	High cost of solvent, environmental

Techniques/Processes	Strengths	Weaknesses
	efficiency	hazards
Ultrasonic-assisted separation	Simple operation, almost no exhaust emission	Noise pollution, high device investment
Thermal Treatment	Simple operation, high throughput	High energy consumption, high device investment, poisonous gas emission
Conventional technologies for Lithium recycling from Lithium-Ion Batteries		
Pyro-metallurgy, e.g., High-temperature alloy reduction followed by Li extraction	Great capacity, simple operation	High temperature, high energy consumption, low metal recovery rate
Hydro-metallurgy, e.g., leaching and solvent extraction.	Low energy consumption, high metal recovery rate	A long recovery process, high chemical reagents consumption
Bio-metallurgy, e.g., microorganism cultivation.	Low energy consumption, mild operating conditions	Long reaction period, bacteria are difficult to cultivate

1.2. Metal Extraction Process

Metal extraction is the most significant part of the LIBs recycling process. In the recent decade, hydro-metallurgy, pyro-metallurgy, bio-metallurgy, and hybrid processes are widely used in industries not only for the recycling and refining of lithium but also for the extraction of other metals including nickel, copper, cobalt, and aluminium. In this section, the above-mentioned metal-extraction techniques are reviewed in terms of their strengths and weaknesses within current recycling processes.

Pyro-Metallurgy Processes

Pyro-metallurgical processes work on the principle of high-temperature smelting, typically in the presence of a reducing agent (e.g., coke) [24]. Normally, these processes do not require pre-treatment and the spent LIBs are directly added to the smelting furnace where they are heated beyond their melting point. Consequently, reducing the amount of carbon by converting it into alloys. The majority of the energy for the burning is provided by the combustion of the carbonaceous compounds, plastics and other volatile matter already present inside the spent LIBs. This high-temperature reductive alloy formation is followed by a secondary recovery stage through leaching. This is typically achieved with various reagents such as water or various acids (e.g., sulphuric acid (H2SO4)) [25]. Finally, solvent extraction is employed to obtain the products containing Ni, Fe, Co, and Mn. The drawback of this recovery process is the loss of lithium due to slag formation [1]. Georgi-Maschler et al. improved lithium recovery from slag by applying secondary leaching using sulphuric acid (H2SO4) [26]. In another study, Hu et al. proposed a series of steps for enhanced lithium recovery from LIBs. The method starts with the roasting of LIBs under an argon environment followed by a water leaching process to extract Li_2CO_3 alongside other metal components. The mixture is then subjected to CO_2 to convert Li_2CO_3 to $LiHCO_3$. Finally, the lithium is recovered through evaporation crystallization [27]. Träger et al. studied lithium recovery through evaporation at a temperature beyond 1400 °C, however, it proved to be economically inviable due to the high demand for energy consumption [28].

Although lithium recovery from LIBs using pyro-metallurgical processes is simple, they have obvious disadvantages such as high operational cost, lithium losses, and risk of secondary pollution [29]. To mitigate the operational hazards, current research has been focused on either process refinement or hybrid methodologies, e.g., pyro-metallurgy coupled with hydro-metallurgy [30].

Hydro-Metallurgy Processes

Similar to pyro-metallurgy, hydro-metallurgical processes typically initiate with LIB pre-treatment followed by leaching, precipitation, and solvent extraction. The effectiveness of a leaching process mainly depends upon the process parameters, including the type and concentration of the leaching reagent, process temperature, time duration, solid/liquid ratio and type of reducing agent [1][29]. The most commonly used leaching reagents include organic acids (ascorbic acid [31][32][33], acetic acid [34][35], oxalic acid [36][37], citric acid [33][34][38][39], tartaric acid [40] and succinic acid [41]), inorganic acids (sulphuric acid (H_2SO_4) [42][43][44], hydrochloric acid (HCl) [45][46][47][48], phosphoric acid (H_3PO_4) [49][50], and nitric acid (HNO_3) [51]), and/or alkaline solutions to leach the desired component out for further purification [52].

Joulié et al. studied different inorganic acids including HCl, H_2SO_4, and HNO_3 for lithium-nickel-cobalt-aluminium oxide (NCA) cathodes and compared their leaching performance [47]. They found that the rate of leaching was significantly higher for HCl due to the formation of chloride ions as a result of the reaction between HCl and $LiCoO_2$. 4 mol L^{-1} of acidic concentration, 4 h of leaching time, and 50 g L^{-1} of S/L ratio were found to be the optimum leaching conditions, obtaining almost 100% of dissolution for desired elemental recovery. In a study involving HNO_3, Lee and Rhee et al. observed a lithium recovery rate as high as 99% when introducing H_2O_2 as a reducing agent [50]. Despite the high lithium leaching rate using inorganic acids, one of the major drawbacks is the production of hazardous waste (such as wastewater, Cl_2, NO_x, and SO_2) that causes serious threats to environmental regulations.

In recent years, organic acids which are degradable and more environmentally friendly have been extensively studied. Such materials have shown a great potential to maintain promising Li recovery rates in hydro-metallurgical methods. Therefore, they have been widely used as alternatives to replace traditional inorganic acids. For example, Li et al. found ascorbic acid was quite effective in Li recycling from LIBs, and a lithium recovery rate of 98.5% was readily obtained [15]. Chen et al. studied the effect of citric acid in a similar process and achieved a Li recovery rate of ~99% [38]. In another study, Zhang et al. combined the biodegradable trichloroacetic acid (TCA) with a reducing agent (H_2O_2) and observed a Li recovery rate as high as 99.7% [53].

Irrespective of process complexity, hydro-metallurgical processes are considered to be the most favorable processes owing to their high metal recovery rate and good product quality [54].

Bio-Metallurgy Processes

In comparison to pyro-metallurgy and hydro-metallurgy, bio-metallurgy processes have proven to be more efficient in terms of equipment and operating costs [29]. These processes mainly rely on the in-situ production of organic and inorganic acids from microbial activities [55]. Xin et al. found that the rate of release of H2SO4 from micro-organisms significantly influenced the rate of lithium recovery [56]. Mishra et al. explored the significance of ferrous ions and elemental sulphur-oxidizing bacteria in yielding metabolites such as ferric ions and sulphuric acid inside the leaching medium, respectively. These metabolites later helped in dissolving the metal ions from the solution, including Li and Co [54]. In another study, Xin et al. found that the Li-ions can be extracted through a non-contact mechanism with a maximum extraction efficiency achieved at a lower system pH [57].

Compared to other Li extraction methods, bio-metallurgical processes favor mild reaction conditions and are very cost-effective and simple in recovery procedures. However, the whole recovery process is time-consuming and cultivation of the desired batch of micro-organisms is difficult (**Table 1**) [29].

Other Processes for Lithium Recovery from LIBs

With the aim to develop environmentally friendly recovery processes, mechanochemical method, a hybrid process that utilizes mechanical energy to influence the physicochemical and structural properties of the metal component, has been reported [58][59][60][61]. Saeki et al. studied the effect of grinding on lithium recovery. In this method, polyvinyl chloride (PVC) was mixed with lithium-containing LIB waste (LiCoO2) and ground in a ball mill [58]. LiCoO2 decomposed in the presence of externally applied mechanical energy and converted to lithium and cobalt chlorides, while chlorine in PVC converted to its inorganic chlorides. In a later phase, these lithium and cobalt chlorides were leached out using water at an overall recovery efficiency of 100% and 90%, respectively. In a similar study reported by Wang et al., zero-valent Fe was added as a third component inside the ball mill along with PVC and LiCoO2. Their research achieved a Li and organic Cl recovery up to 100% and 96.4%, respectively [61]. Maschler et al. reported a hybrid process for efficient recovery of both lithium and cobalt by incorporating pyro- and hydro-metallurgy with a mechanical pretreatment process [26]. Whereas Gupta et al. introduced a ‘chemical extraction technique’ that utilized the oxidizing properties of –Cl2, I2, and Br2 for fast lithium recovery from LiCoO2, although this method requires harsher recycling conditions [62].

Overall, conventional techniques for extracting lithium from lithium-ion batteries have many advantages. Despite this, such techniques have exhibited disadvantages such as high energy consumption, large waste production, and excessive operational requirements (**Table 1**). Overcoming these challenges and achieving equivalent purity is crucial for future research in this field, with some research previously investigated regarding membrane technologies (**Table 2**).

Table 2. A comparison of the process efficiency and percentage lithium recovery in lithium-ion battery-based extractions.

Lithium Extraction Technologies	Process Efficiency	Percentage Lithium Removal	References
Pyro-metallurgy	>95	85–96	[1][25][27][28]
Hydro-metallurgy	>90	90–99.7	[31][32][33][34][35][36][37][38][39][40][41][42][43][44][45][46][47][48][49][50][51][53]
Bio-metallurgy	>95	~98	[56][57]
Membranes	>90	80–99.99	[63][64][65][66][67]

2. Membrane Processes

Supported liquid membranes (SLM) have been studied for liquid-phase metal ion extraction/separation [63][64]. It has been considered as an alternative to conventional solvent extraction due to its advantages such as operational simplicity, low solvent demand, low energy consumption, zero effluent discharge, and high selectivity [64]. Furthermore, the process is considered “green” as few chemicals are involved. In contrast with the traditional solvent extraction method, it requires much less organic solvent solely as a molecule carrier.

Swain et al. studied the separation factor of Co(II) and Li(I) from dilute aqueous sulphate media using SLM, a hydrophobic PVDF membrane with a 0.45 µm pore size was used as the solid support [64]. The liquid phase was a mixture of Cyanex 272 and DP-8R, which acted as mobile carriers. Parameters such as pH, extractant concentration, feed concentration, and stirring speed were studied. The group found optimal performance was achieved at pH = 5, a mixture of Cyanex 272 and DP-8R at a concentration of 750 mol m^{-3} and 350 rpm stirring speed. The resultant conditions allowed for a separation factor of Co(II)/Li(I) = 497:1. Using similar conditions, hollow fiber (HF) supported liquid membranes can be combined with non-dispersive solvent extraction (NDSX). The best condition for separation using this technique was achieved in aqueous feed at pH 6 and 750 mol m^{-3} of Cyanex 272 in the membrane. Complete separation of Co(II) and Li(I) with a 99.99% purity was achieved using the HF-supported liquid membrane process with Cyanex 272 as an extractant [65]. Recently, a novel type of liquid membrane—polymer inclusion membrane (PIM) has attracted much attention due to its obvious advantages such as smaller quantities of the extractant and reduced environmental impact. PIMs have also been found to maintain high selectivity and separation efficiency compared with solvent extraction [66]. Further studies suggest that Co(II) ions were effectively removed from the source phase through the PIM containing 32 wt.% Triisooctylamine, 22 wt.% cellulose triacetate, and 46 wt.% o-nitrophenyl octyl ether, with deionized water as the receiving phase [67]. Other PIM systems containing both thenoyltrifluoroacetone and trioctylphosphine oxide as the carrier and cellulose triacetate as the base polymer exhibited high selectivity of Li(I) over Na(I) and K(I) with a separation factor of 54.25 and 50.60, respectively [65].

These liquid membranes combine the benefits of both solvent extraction and membrane technologies as well as low energy consumption and low waste discharge. However, the liquid membrane typically has low stability and faces some scaling-up challenges.

Membrane technologies are considered novel methods for aqueous phase lithium recovery and have been widely studied in the recent decade. However, only a few processes, such as NF and membrane distillation crystallization, have been applied at an industrial scale. Though membrane technologies have been facing some drawbacks such as membrane fouling, defects, and industrial scaling-up challenges, they provide great solutions for highly efficient and environmentally friendly lithium recovery from the liquid phase.

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