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Review

Review of upstream processes for Li-ion batteries recycling: Safety and economic concerns

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ABSTRACT

Recycling of end-of-life Lithium-Ion Batteries (LIBs) is receiving increasing attention due to the economic and environmental costs associated with battery production and the criticality of its material supply chain. However, research has focused on recycling processes, with less emphasis on the pre-treatments involving the battery packs. This review analyses state-of-the-art literature on pre-treatments for LIBs recycling, considering four main issues: residual voltage discharge, cells dismantling, organic components removal, and electrodes active material liberation and concentration into material streams destined to specific recycling treatments. The literature review revealed that current technologies applied in upstream recycling processes ensure efficient materials recovery, but there are still significant challenges in terms of safety and economic viability. Safety issues arise from inadequate voltage discharge and the release of potentially toxic emissions from organic components. Economic challenges are primarily due to the high labor costs of manual cell dismantling, which is not fully met by automated disassembly lines. In conclusion, further research should be devoted to optimizing the economic viability of the above-mentioned operations and their safety, and also to improve the recycling of anodic materials.

1. Introduction

The global market for lithium-ion batteries (LIBs) is experiencing rapid growth, driven by the push to decarbonize transportation and energy sectors. Projections indicate that the demand for LIBs will surpass 90 billion US dollars by 2026 (Statista, 2023). In Europe alone, LIBs sales are expected to range between 250 and 1100 GWh by 2026, with forecasts suggesting a rise to between 600 and 4000 GWh by 2040 (Tsiropoulos et al., 2018). However, LIBs degrade over time, leading to capacity loss and power fading (Kabir and Demirocak, 2017). This is primarily due to the formation of the solid electrolyte interface on the electrodes' surfaces, which causes morphological changes and cell volume alterations, resulting in energy loss (Kim et al., 2020). Once the energy storage capacity of LIBs drops to 70–80 % of initial value, they are deemed unsuitable for electric vehicles. Nevertheless, these batteries can still be recycled or refurbished for stationary grid energy storage, thereby reducing the overall environmental and economic costs (Energy Storage Association, 2020).

Currently, the main LIBs recycling technologies include pyrometallurgical, hydrometallurgical, and direct recycling treatments. Pyrometallurgy involves high temperature smelting to reduce cobalt, nickel, and copper into alloys (Mansur et al., 2021), and burns off carbon-based components and leaves other elements like lithium, aluminum, silicon, calcium, and iron in the slag (Assefi et al., 2020). Despite being a mature technology, pyrometallurgy has significant drawbacks, such as high energy demand, emissions from burning plastic components and electrolytes, and limitations in material recovery (Wang et al., 2020). These limitations hinder material circularity, as promoted by the European legislation, which aims for recycling rates of 65 % of battery weight by 2025 and 70 % by 2030, with specific targets for critical materials like cobalt and lithium (European Commission, 2020). Alternatively, hydrometallurgy selectively recovers elements like cobalt, nickel, manganese, and lithium using solvent extraction. This requires less energy and has a lower environmental footprint compared to pyrometallurgy. However, its high operating costs have limited its full-scale development (Larouche et al., 2020). Recently, direct recycling processes have emerged, reconditioning the electrode active materials without morphological alterations. This approach is promising, especially for batteries with low cobalt content, which have lower economic value and may not justify the costs of traditional recycling

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methods (Harper et al., 2019).

The recovery of materials through LIBs recycling is crucial for meeting the supply demands driven by increasing production (Mayyas et al., 2019). Repurposing LIBs for secondary applications can pose bottlenecks in material supply, making effective recycling strategies essential (Bobba et al., 2019). Consequently, research is focused on optimizing end-of-life (EoL) LIBs recycling strategies to maximize the quantity and quality of recovered materials while reducing the environmental and economic impacts of recycling operations (Asadi Dalini et al., 2020; Heelan et al., 2016; Kader et al., 2021). Upstream processes, which are often overlooked, have the potential to significantly improve the efficiency of the overall recycling process and address environmental and safety issues. However, research on upstream processes is still limited, with existing literature focusing on narrow aspects like physical processes (Sommerville et al., 2020), disassembly (Wu et al., 2023) or emerging technologies developed in recent years (He et al., 2021; Kim et al., 2021). Thereby this review study provides an overview of different technologies applied at full scale and currently under research for the upstream stages of LIBs recycling. Technical performance of different technologies and their efficiency are assessed and compared, and concerns about safety and economic viability are also discussed.

2. Methodology

The literature review was conducted on Scopus using the keywords “li-ion battery recycling” AND “pre-treatment”, “discharging”, “electrolyte removal”, “binder removal”, “mechanical treatment”, “electrode liberation”, “abrasion”, “milling”, “disassembly”, “dismantling”, “froth flotation”, “ultrasound washing” and “physical separation”, in various combinations. The resulting articles have been pre-screened based on the abstract and then clustered into the four main topics addressed in this review (Fig. 1 and Figure A1), corresponding to the main sequential steps of upstream treatments for LIBs recycling processes: (A) discharge, (B) disassembly, (C) electrolyte and binder removal, and (D) liberation and concentration of electrode active materials.

3. Results of literature review

Upstream processes for LIBs recycling involve sequential steps for material preparation and concentration for pyrometallurgical, hydrometallurgical, or direct recycling. Initially, batteries are fully discharged to eliminate residual voltage and then opened. After removing the cell cases, electrodes are either sent to pyrometallurgical treatment to recover metals like cobalt and nickel or undergo mechanical "liberation" and "concentration" processes. Liberation detaches active materials from metallic current collectors, while concentration generates enriched material streams for efficient recovery in hydrometallurgical recycling (Granata et al., 2012). These steps improve downstream recycling efficiency, enhance safety, and increase economic returns. (Träger et al., 2015).

3.1. Discharge

Discharging LIBs is crucial for removing residual potential in EoL LIBs, ensuring safety during subsequent recycling processes. Accurate estimation of residual charge and thermal behavior in LIBs has led to the development of various experimental models (Garg et al., 2019). The two discharging methods applied at full-scale are electrolysis (Kim et al., 2021; Shaw-Stewart et al., 2019; Xiao et al., 2020) and resistance discharge (Langner et al., 2021). Resistance discharge involves connecting the battery to an electrical circuit with load resistors, with discharge currents up to 200 A (Langner et al., 2021). Electrolysis involves immersing battery cells in ionic solutions. LIBs can be submerged in distilled water for up to one day, though the process can be accelerated with ion solutions (Xiao et al., 2020). NaCl solutions are commonly used, with concentrations of 20 % for 24 h achieving high discharge rates, particularly when combined with ultrasound treatment (Torabian et al., 2022). Despite its effectiveness, NaCl poses drawbacks such as potential chlorine gas release and galvanic corrosion of cell casings (Rouhi et al., 2022). Alternatives include NaOH, K_3PO_4, and $MnSO_4$, which, while reducing corrosion and organic leakage, offer only mild discharge rates (Shaw-Stewart et al., 2019; Xiao et al., 2020). Salts like $FeSO_4$ or $ZnSO_4$ improve conductivity and discharge efficiency, with FeSO4 being the most environmentally friendly option (Ojanen et al., 2018). Zinc acetate also limits corrosion effects (Fang et al., 2022). Voltage reduction can reach down to 70 % of starting voltage for $ZnSO_4$ (Ojanen et al., 2018), 60 % for $NaSO_4$ (Ojanen et al., 2018; Wang et al., 2022), 50 % for $MnSO_4$ and 40 % for $MgSO_4$ (Torabian et al., 2022), though they fall short compared to NaCl’s near-total discharge (Ojanen et al., 2018; Torabian et al., 2022). However, they offer reduced corrosion and lower hazardous gas emissions, making them potential "greener" alternatives (Yao et al., 2020). Novel discharge methods include replacing conductive solutions with conductive powders like flake-graphite could mitigate issues such as false voltage readings and potential voltage rebound (Wang et al., 2022). The performance of conventional NaCl, alternative non-corrosive solutions and conductive powders for LIBs discharge is shown in Fig. 2.

In conclusion, discharge is a key phase in LIBs recycling, crucial for preventing short-circuits and fire hazards. Discharging methods include electrochemical discharge with solutions (commonly 5–10 % NaCl), resistance discharge, and passivation. Balancing efficiency, safety, and environmental impact remains a key challenge in advancing these technologies.

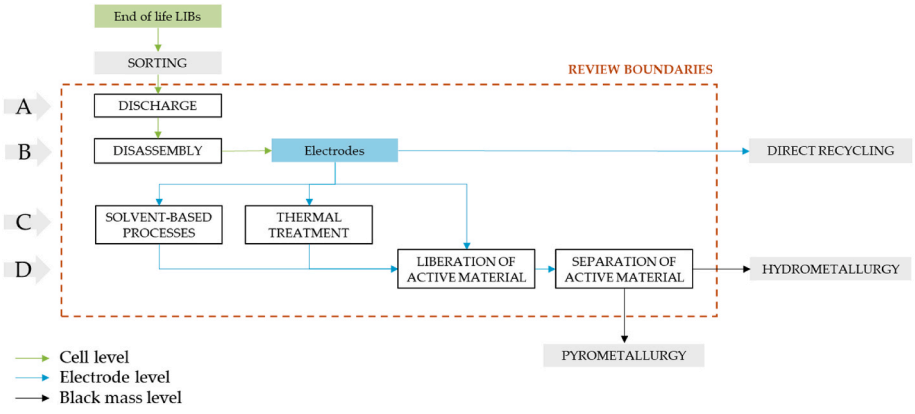

Fig. 1. Sequence of upstream processes for LIBs recycling and boundaries of this review study.

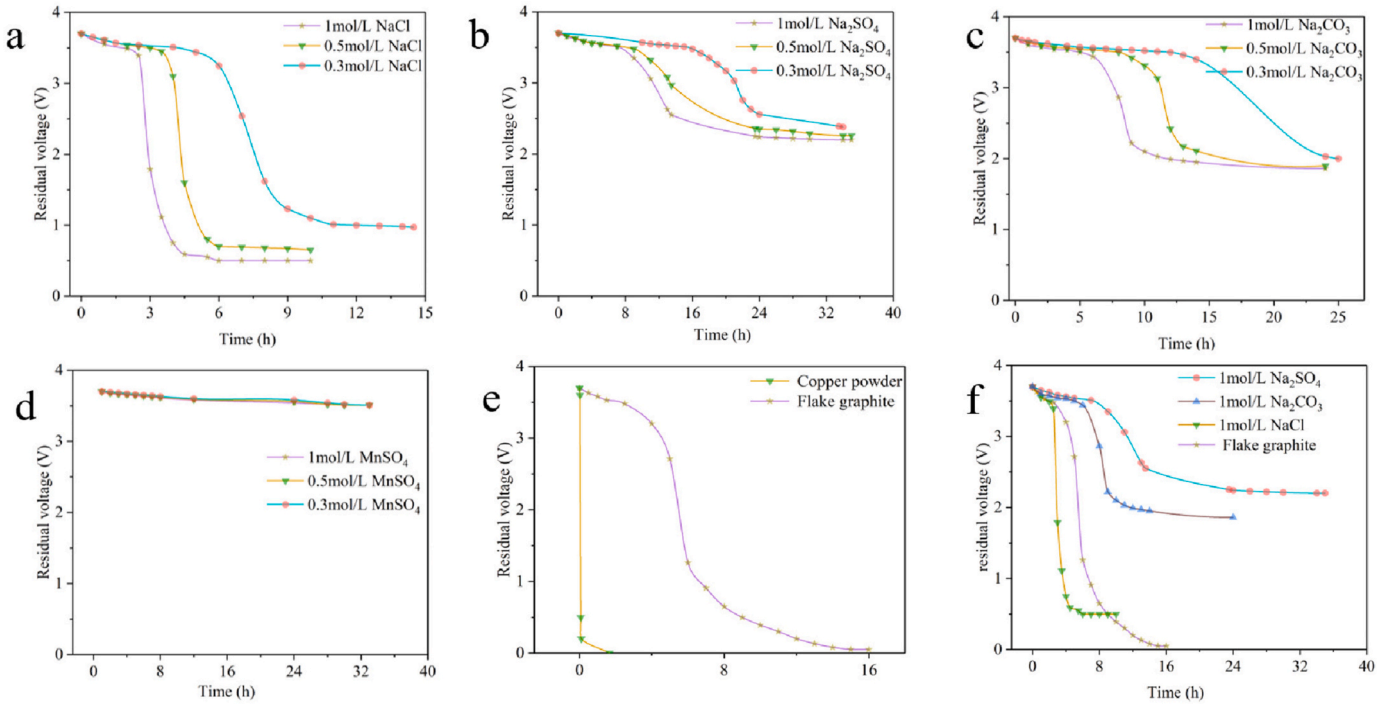

Fig. 2. Comparison of discharge efficiency at the following discharge media: (a) three concentrations of NaCl solution; (b) three concentrations of Na_2SO_4 solution; (c) three concentrations of Na_2CO_3 solution; (d) three concentrations of $MnSO_4$ solution; (e) copper powder and flake graphite; (f) Comparison of optimal discharge efficiency of different discharge media (Wang et al., 2022).

3.2. Disassembly

After discharge, battery cells are disassembled to remove casings and recover electrodes and, occasionally, electrolytes. Disassembly can be manual or mechanical. Manual dismantling is common in large-scale facilities and is well-documented (Takahashi et al., 2020; Wang et al., 2018). Electrohydraulic force is a promising new method for opening cells (Kurz et al., 2021). Automated dismantling usually involves crushing (Wuschke et al., 2019) or cutting mills (Pinegar and Smith, 2019; Zhan et al., 2020) and requires about 4.5 kWh/t of mechanical energy (Wuschke et al., 2019). Hybrid systems combining human labor and automated disassembly have been proposed (Fleischer et al., 2021; Li et al., 2019; Werner et al., 2022). In these, humans handle complex tasks while machines perform repetitive ones, improving ergonomic comfort for workers (Jiao et al., 2024). However, the diversity and complexity of battery designs pose challenges to automation, requiring flexible dismantling systems (Herrmann et al., 2014). Innovations like artificial intelligence and better labelling of batteries aim to address these challenges and enhance automation (Asif et al., 2024; Li et al., 2024; Zorn et al., 2022).

Research on battery disassembly focuses on optimizing economic benefits and reducing environmental impacts while ensuring worker safety (Alfaro-Algaba and Ramirez, 2020; Lu et al., 2022). Economic analyses reveal that cell opening is the priciest step at up to 76 €/kWh, compared to module disassembly (60 €/kWh) and battery dismantling (32 €/kWh) (Rallo et al., 2020). Manual disassembly generally yields purer material streams than shredding, improving efficiency and revenues (Marshall et al., 2020), and is widely used at full-scale (Holzer et al., 2023). However, automated disassembly can cut costs by up to 20 % due to reduced processing time (Kay et al., 2022). Despite higher initial investments, automated systems offer lower operational costs in the long run (Gerbers et al., 2018).

In conclusion, while manual dismantling ensures higher precision in separating components, it is labour-intensive and costly. Future research should aim to integrate manual and automated methods to combine the precision of manual labor with the efficiency of automation, optimizing the overall process.

3.3. Electrolyte and binder removal

LIBs contain organic components like electrolytes and binders. Electrolytes are typically $LiPF_6$ salts in a mixture of Ethylene Carbonate (EC) and linear carbonate (Dimethyl Carbonates DMC, Ethyl Methyl Carbonate EMC, Diethyl Carbonate DEC). Binders, as Polyvinylidene Fluoride (PVDF) and Carboxymethyl cellulose help adhere active materials to current collectors. Recycling processes remove electrolytes through thermal treatment (Zhang et al., 2019a; Zhong et al., 2020), solvent extraction (Grützke et al., 2015a; Haas et al., 2018), or supercritical CO_2 extraction (Nowak and Winter 2017). Thermal treatments achieve up to 99.32 % removal of linear carbonates and 99.93 % of $LiPF_6$ (Zhong et al., 2020). Binders are removed via incineration, pyrolysis, or solvent dissolution, with PVDF removal efficiency nearly 100 % after pyrolysis and 98.5 % with supercritical CO_2 extraction (Fu et al., 2021).

3.3.1. Solvent-based processes

Electrolyte removal is often performed with the use of organic solvents, such as DMC (Buken et al., 2021; Zhu et al., 2020), EC, acetonitrile, propylene carbonate (Grützke et al., 2015a) ethanol or acetone, or supercritical CO_2 (Nowak and Winter 2017). Besides, electrolyte solvents are potentially recoverable via thermal treatment with temperature below 150 °C to collect HF and POF_3 gasses and recover them as acid solutions (Zachmann et al., 2023).

Dissolution of PVDF binder can also happen applying organic polar solvents, like N-Methyl-2-Pyrrolidone (NMP) at temperature between 60 °C and 80 °C (Zhao et al., 2021). A greener alternative to NMP dissolution has been found in DMI (dimethyl isosorbide, which has shown the ability to form H bonds with PVDF and facilitate its removal (Buken et al., 2021; Zhu et al., 2020) or and Dimethylformamide (DMF), which is an organic solvent effective at room temperature with limited operative costs (Xu et al., 2014). Moreover, high temperature leaching with acid solutions has been found effective towards organic binder, e.g. citric acid leaching at 90 °C (Pant and Dolker, 2017) or sulfuric acid,

which simultaneously leaches Co, Mn and Ni from the cathodes and can partially decompose PVDF (Fan et al., 2021; Li et al., 2015). Novel solvents have proposed, such as alkaline solutions of NaOH, which completely dissolve (Han et al., 2024; Senćanski et al., 2017) or ethanol, which does not affect the Al cathode collector. Moreover, extraction with supercritical CO_2 has been presented as an environmentally friendly alternative to remove PVDF without altering its chemical properties to recover the binder solution (Fu et al., 2021), with higher recovery efficiency and minimal presence of Al impurities compared with traditional dissolution methods (Xu et al., 2014). Eventually, advanced oxidation processes based on the use of SO^{4-} radicals were developed to degrade PVDF binders and recover over 99 % of LFP powder from current collectors (Ou et al., 2023)

In conclusion, electrolytes and binders can be removed via solvents extraction or supercritical CO_2. Common solvents for electrolyte extraction as NMP and DMF are commonly applied also for binder dissolution.

3.3.2. *Thermal pre-treatment*

Thermal pre-treatments can either be simple drying to volatize the residual electrolytes on the electrodes surface or high temperature pyrolysis. Electrolyte volatilization through drying is carried out at low temperatures between 60 °C (Takahashi et al., 2020) and 80 °C (Silveira et al., 2017; Widijatmoko et al., 2020a, 2020b), or up to 120 °C (Zhong et al., 2020). Binder's degradation instead requires higher temperature compared to electrolyte solutions. Alternatively, residual electrolyte could be removed together with binders via pyrolysis (Han et al., 2021; Zhang et al., 2022), at temperatures between 500 °C (Han et al., 2021; Zhang et al., 2020) and 650 °C (Jung et al., 2021), and in reducing atmosphere with N_2 (Zhang et al., 2020) or CO_2 (Deng et al., 2022; Jung et al., 2021) .

Thermal treatment for PVDF removal is also often carried out in air (i.e., incineration) applying temperatures between 400 °C and 700 °C (Lombardo et al., 2020; Vieceli et al., 2021), and most commonly around 500–550 °C (Hanisch et al., 2015; Jafari et al., 2020) thus improving separation efficiency due to PVDF decomposition and removal of impurities (Han et al., 2021). Lithium iron phosphate (LFP) cathodes in particular exhibited improved separation rate of active material after thermal treatment at lower temperature, between 240 °C and 300 °C (Bi et al., 2020; Bruno and Fiore, 2024). However, different cathodes' chemistries require at least 500 °C, which is slightly above PVDF decomposition (Jiang et al., 2023). Temperature above 600 °C are not recommended for direct recycling processes since it compromises structural integrity of the electrode material generating holes and cracks and hindering recycling yield (Zhao et al., 2021). Though, incineration at temperature between 200 °C and 400 °C has resulted in PVDF melting and rearrangement, decreasing separation efficiency (Hanisch et al., 2015).

Despite thermal treatments to degrade organic binders have shown great promises in improving active materials separation from current collectors (Makuza et al., 2021a; Yan et al., 2020), concerns arise due to the potential release of toxic gaseous compounds from the decomposition of PVDF and $LiFP_6$ at high temperature (X. Hu et al., 2022). However, pyrolysis vaporizes binders as pyrolytic products (He et al., 2021) and electrolyte as pyrolytic syngas (Jung et al., 2021), and generates lower amounts of off-gas emissions compared to combustion, especially for nickel manganese cobalt (NMC) and lithium iron phosphate (LFP) (Diaz et al., 2019). Nonetheless, 32 % of F and P are released by volatilization of $LiPF_6$ and are easily collected through bag filters (Murakami et al., 2020). Thermal processes to remove electrolyte and binders are bound to the characteristic degradation temperature of these organic components. Therefore, thermal treatment at temperature below 300 °C is favourable to limit Al oxidation and generation of fluoride gas (Kang et al., 2025). The thermogravimetric analysis of a standard black mass and common binders (PVDF and CMC) and the temperature ranges required for their removal through thermal treatment are summarized in Fig. 3.

In conclusion, thermal treatments to remove organic electrolyte can be performed as low-temperature (30°C–100 °C) drying processes or alongside thermal degradation of binders at higher temperature (400°C–800 °C). The latter treatments can be performed in different atmosphere (air, N_2, H_2 and under vacuum). Thermal pre-treatments for

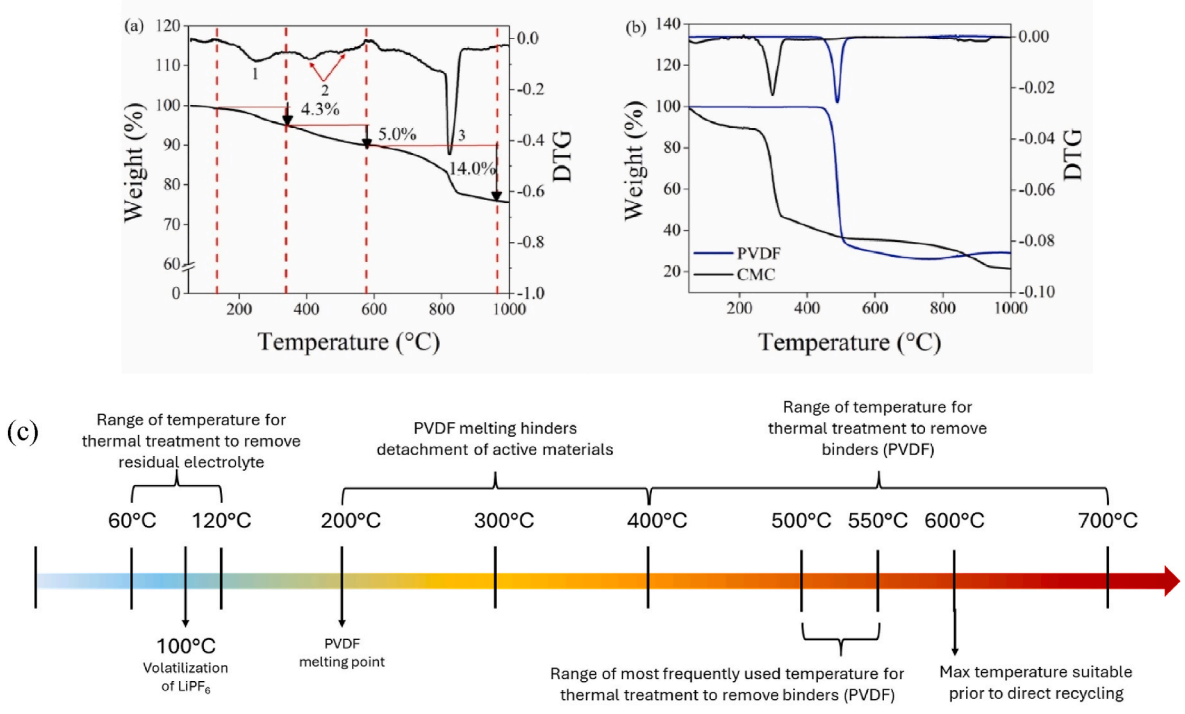

Fig. 3. Derivative thermogravimetric (DTG) analysis of (a) black mass (Golmohammadzadeh et al., 2023) and (b) neat PVDF and CMC (Golmohammadzadeh et al., 2023). (c) Summary of the range of temperatures applied during thermal treatment for electrolyte and binder removal.

organic components removal need still to be optimized, since degradation of organic binders aids the detachment of electrode powders from current collectors, however the presence of electrolyte residues has shown a positive effect in separations based on froth floatation.

3.4. Liberation and concentration of electrode active materials

State-of-the-art LIBs recycling technologies liberate active materials from current collectors via mechanical processes like crushing, shredding, attrition, or ultrasound washing (Lei et al., 2021; Vanderbruggen et al., 2021; Xiao et al., 2017). These processes happen after binder removal (Jiang et al., 2023; Wilke et al., 2023). Concentration exploits differences in specific mass, particle size, hydrophobicity, and conductivity between active materials and current collectors (Chen et al., 2020).

3.4.1. Liberation of electrode active materials

Mechanical pre-treatments consist in a sequence of crushing, milling and sieving steps (Pagnanelli et al., 2017; Xiao et al., 2017), and the overall recycling efficiency can achieve 75–80 % (Diekmann et al., 2018a). Various milling devices have been applied for mechanical crushing of spent LIBs, from impact crushers (Aravindan et al., 2019; Zhou et al., 2010) to knife mills (Granata et al., 2012; Takahashi et al., 2020), with ball milling being the most applied method (Shi et al., 2020; Wang et al., 2016, 2019). Typical operating parameters for ball milling include an agitation frequency of around 600 rpm. Additionally, milling performance improves with the application of cryogenic processes (Mu et al., 2022), which can reduce treatment time up to 5 min (Wang et al., 2019). Cryogenic processes happen around −40 °C, where PVDF physical state transition into a glass-like solid (Wang et al., 2019). Crushing in a wet environment prevents dust generation, however, dry crushing had higher selectivity in concentrating Al, Cu, and casings in the coarser fractions (Zhang et al., 2013). Pulverization at fine particles size is effective for the recovery of graphite and copper from anodes due to their weak chemical bonding (Zhou et al., 2010). Indeed, Cu recovery rate can reach 95.9 % in the fraction with particle size above 0.3 mm (Zhou et al., 2010). The granulometric fractions produced by mechanical crushing are an ultra-fine fraction enriched in electrode powder below 0.5 mm, a fine fraction containing metals from current collector foils between 0.5 mm and 2.0 mm and a coarse fraction containing metallic and plastic casing and separators above 2.0 mm (Pinegar and Smith, 2020). The optimal cut off size that represents the better trade-off between electrode powder recovery rate and grade has been identifies as 0.85 mm, though binder residues may increase this size(Widijatmoko et al., 2020a).

Delamination processes, instead, apply shearing forces to physically detach active materials from current collectors (Vanderbruggen et al., 2022). Exfoliation of active materials from electrode sheets can be performed in water with a silica sand flow with an impeller velocity of 1000 rpm, yielding a separation rate of 89.8 % of active material (Widijatmoko et al., 2020b) or in an agitation rotor with an inert exfoliation media, with a separation rate of 96.88 % (Zhang et al., 2023). Notably, high-speed crushing at 32000 rpm has demonstrated the possibility to separate between 80 % and 85 % of active material from LFP cathodes (Wu et al., 2021). Delamination of current collectors could be aided by the presence of water that weakens mechanical strength of Al foils and facilitates separation (He et al., 2020). The main drawback of delamination processes is the presence of impurities in the separated active materials, where Al and Cu content can reach 14 %mass (Widijatmoko et al., 2020b). Novel strategies, such as, water pressure-washing (Ji et al., 2022; Kurz et al., 2021) or pneumatic abrasion, have been recently proposed to limit the presence of impurities in the separated materials below 1 %.

The liberation of electrodes active material could also rely on the cavitation effect generated by ultrasound washing (Lei et al., 2021). The effects of ultrasound waves, between 20 kHz (Lei et al., 2021) and 40 kHz (He et al., 2015; Li et al., 2014), can mechanically separate active materials from current collectors, by breaking the bonds between them (Takahashi et al., 2020). Electrode powder separation through ultrasound washing is highly effective, indeed 96.88 % of active material can be recovered with a purity grade of 93.89 % (G. Zhang et al., 2018) and the removal of almost 100 % of cathode active materials (Jafari et al., 2020). However, electrode delamination through ultrasound washing can be aided by the addition of the organic solvents, such as NMP (He et al., 2015; Yang et al., 2015), reaching a recovery efficiency of almost 99 % active material (He et al., 2015). Despite ultrasound washing is a promising technique to detach active material from current collectors in EoL cathodes, it is inadequate to treat cathodes production scraps (Bruno and Fiore, 2024). Eventually, high voltage fragmentation has been proposed to prevent material overgrinding and increase separation efficiency (Kikuchi et al., 2021), however the scale-up of this technology is severely hindered by the high energy demand and economic costs entailed (Leiβner et al., 2018). The working mechanisms of novel technologies for detachment of active materials from LIBs electrodes are shown in Fig. 4.

3.4.2. Mechanochemical activation of electrode active materials

Mechanochemical activation combines mechanical forces to weaken chemical bonds and reduce particle sizes, increasing surface area and reaction efficiency (Fan et al., 2018; Makuza et al., 2021b) and reducing the oxidation state of the metals in the active materials (Dolotko et al., 2023). As a results, both liberation and leaching efficiencies improve (Dolotko et al., 2024; Fan et al., 2018). Leaching efficiency of Li in water after mechanochemical activation increases from 30 % to 70 % (Dolotko et al., 2023).

Mechanical forces are provided by ball milling or ultrasound washing in presence of reducing agents. Particularly, the mechanical energy from milling is used to activate the chemical reactions necessary to break the crystal structure of the cathode material and transform it in a more reactive form (Jiang et al., 2022). Planetary ball mills in zirconia (Fan et al., 2018) or stainless steel (Dolotko et al., 2024) with milling frequency around 200–550 rpm (Fan et al., 2018; Guan et al., 2017; Meng et al., 2019) up to 15 min (Xie et al., 2021). Potential reducing agents are dry ice (M. Wang et al., 2021), acidic solutions (Wang et al., 2016) or metallic powders, e.g. Zn (Xie et al., 2021), Fe (Guan et al., 2017).Mechanochemical processes increases the efficiency of downstream hydrometallurgical recycling, thus limiting the need for corrosive acids (Yang et al., 2019), and high temperatures (Jiang et al., 2022). Consequently, mechanochemical recycling limits acid wastewater generation and greenhouse gasses emissions, aligning with the principles of green chemistry (Wang et al., 2021).

3.4.3. Concentration of electrode active materials

The concentration of active materials from EoL LIBs exploits the physical differences between active materials and current collectors, and is based on technologies like magnetic separation, eddy current separation, electrostatic separation, pneumatic separation, and gravity separation. These achieve concentration efficiencies ranging from 50 % to 99 % (Diekmann et al., 2017).

Magnetic separation is widely used to extract active materials from spent LIBs electrodes, recovering from 64.2 % to 90 % of Co and from 14 % to 35.8 % of Mn (Z. Hu et al., 2022; Pindar and Dhawan, 2020a) in the magnetic fraction. The non-magnetic fraction primarily contains graphite and lithium carbonates (Sunil et al., 2019; Sunil and Dhawan, 2019), along with minor impurities like Mn, Cu and Al (Vishvakarma and Dhawan, 2019). Pre-treatments such as mechanical crushing, thermal treatments, or microwave reduction typically enhance the liberation of magnetic particles, improving the concentration efficiency. Recovery efficiency for Co in the magnetic fraction can reach up to 85–90 % (Vishvakarma and Dhawan, 2019), with grades reaching 73 %(Pindar and Dhawan, 2020b). Further processing, such as ball milling followed by a second magnetic separation, can increase Co grade to 83 % (Sunil

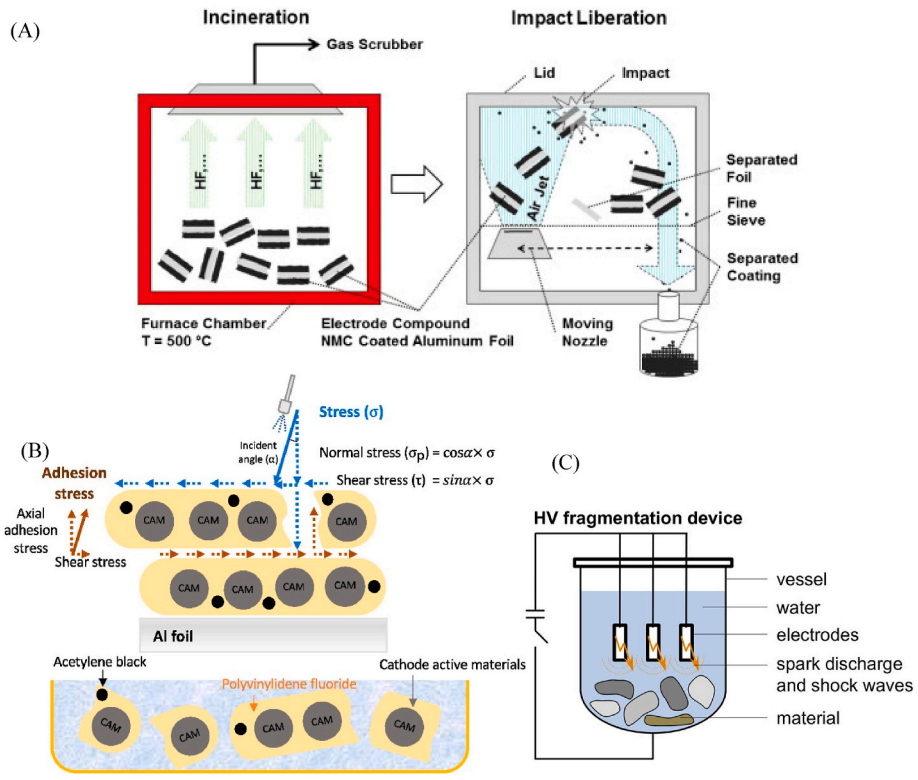

Fig. 4. (A) Working mechanism for impact liberation through ANVIIL method (Hanisch et al., 2015); (B) pressure washing system (Ji et al., 2022); and (C) high voltage fragmentation (Leißner et al., 2018).

and Dhawan, 2019).

Eddy current separation is also used to separate ferrous and non-ferrous metals like Al and Cu, with recoveries reaching up to 19 % (Granata et al., 2012). Eddy current separation is often employed after crushing and thermal treatment to remove residual electrolyte, typically at 300 °C for 2 h (Bi et al., 2021). This method achieves high concentration grades, with 98.98 % for metals and 99.6 % for plastics (Silveira et al., 2017).

Density-based separation, using Stokes' law, is employed to separate cathodic active materials based on density differences. This method includes multi-step flotation processes with liquids of varying densities to achieve high purity. For instance, high purity NMC and lithium manganese oxide (LMO) cathodes have been recovered with grades of 95 % (Folayan et al., 2021). Pneumatic separation exploits differences in particle size and density. For example, air-sifting and pneumatic separation can effectively split Cu and Al from electrode current collectors. Optimal air speeds of 0.34 m/s for fine fractions and 12.85–14.23 m/s for coarser fractions are used, with recovery rates of 92.08 %–97 % for Cu and about 96 % for Al (Bi et al., 2019). Additionally, spouted bed elutriation has been proposed as a simple method for separating plastics from metals (Bertuol et al., 2015). Using a Wilfley shaking table has increased the Cu grade in the non-magnetic fraction from 54 % to 66 % (Da Costa et al., 2015).

Froth flotation is a process based on hydrophobicity differences. It involves creating an aqueous slurry with a pulp density of about 40 g/l and aerating it to float hydrophobic components attached to air bubbles. Chemicals such as Methyl Isobutyl Carbinol (MIBC) and n-Dodecane are added to enhance flotation. MIBC concentrations typically range from 150 to 200 g/t (Wang et al., 2018; Zhang et al., 2019b), and n-Dodecane is added at 40 g/l. Air flows during flotation are between 0.75 l/min and 2 l/min (Yu et al., 2018).

Froth flotation is more effective for concentrating graphite than lithium metal oxides (Verdugo et al., 2022), and is also effective for concentrating metallic fractions like Al and Cu (Traore and Kelebek, 2022). Moreover, froth floatation has shown effective in concentration of metallic rich fractions, mainly Al and Cu (Rinne et al., 2021; Saneie et al., 2022). Pre-treatments such as pulverization or crushing improve flotation efficiency, and fine grinding of electrode materials enhances the process (Zhan et al., 2018). The presence of reducing agents like graphite from anodes has increased Co flotation efficiency to 81.3 % (Ruismäki et al., 2020). Thermal treatments have a variable effect on separation rate through froth flotation since the presence of residual electrolyte increases particles hydrophobicity and, therefore, flotation efficiency (Zhan et al., 2021), whereas residual binder instead increases the agglomeration of active materials hindering concentration through froth floatation (Vanderbruggen et al., 2022).

In conclusion, the concentration of active materials from LIBs involves several key techniques offering specific advantages in terms of efficiency and material purity. Magnetic separation effectively recovers valuable metals like Co and Fe, while eddy current and pneumatic separation target non-ferrous metals and plastics. Froth flotation and density-based separation provide high purity levels for specific materials, with advancements in pre-treatments and process optimizations enhancing overall efficiency. The separation rates and grades of cathode and anode active materials, as well as Al and Cu from current collectors after different liberation and concentration technologies are reported in Table 1, see Table A1. Differences in experimental conditions and analytical protocols can impact the reliability of results and explain variations in separation rates achieved by the same technologies across different studies.

4. Discussion on safety concerns

Upstream processes for LIBs recycling are fundamental to ensure safety for further recycling processes. Firstly, guaranteeing that batteries are safely discharged before recycling, makes the whole process safer, more efficient and environmentally responsible. EoL LIBs can retain a significant charge even after they are no longer useful in their original

Table 1
Separation rates (%) and separation grades (%) of different pre-treatment for cathode and anode active materials and Al and Cu from current collector.

Pre-treatment	Seaparation rate (%) - Cathode	Seaparation grade (%) - Cathode	Seaparation rate (%) - Anode	Seaparation grade (%) - Anode	Separation rate (%) - Al	Separation rate (%) - Cu	Reference
Pyrolysis, colour sorting, high-pressure water cleaning and flotation	49.67 %				99.34 %	96.25 %	Zhong et al. (2019)
Pyrolysis	99.78 %		99.6 %				Jiang et al. (2023)
Supercritical CO_2	98.86 %						Mu et al. (2022)
Alkali leaching (NaOH)	97.8 %						Han et al. (2024)
Pyrolysis and flotation	96.88 %	93.89 %					(G. Zhang et al., 2018)
Pyrolysis and flotation	98.23 %	93.89 %	98.99 %				Zhang et al. (2019b)
Flotation	90 %	83 %	75 %	77 %			Qiu et al. (2022)
Flotation	80 %		98.8 %				(Rinne et al., 2021)
Flotation			90 %				Zhan et al. (2018)
Flotation		97.13 %		73.56 %			Yu et al. (2018)
Flotation	89.4 %		94.4 %				Vanderbruggen et al. (2022).
Flotation	92.86 %		83.21 %				Zhu et al. (2021)
Pneumatic separation					96.68 %	92.08 %	Zhu et al. (2021)
Magnetic separation						54 %	Da Costa et al. (2015)
Gravimetric separation (Wilfley table)						66 %	Da Costa et al. (2015)
Magnetic separation and induced roll magnetic separation		96.6 %	74.54 %				(Z. Hu et al., 2022)
Electrostatic separation							Silveira et al. (2017)
Pneumatic separation	97.1 %						Hanisch et al. (2015)
Falcon Ultra-Fine (UF) centrifugal gravity concentration	90 %	99 %					Zhan and Pan (2022)
Pulverization and sieving						93.10 %	Zhou et al. (2010)
Pulverization and sieving						95.9 %	Zhou et al. (2010)
Abrasion	80 %						Widijatmoko et al. (2020b)
Abrasion	85 %						Wu et al. (2021)
Cryogenic binder	87.29 %						Wang et al. (2019)
Electrical pulsed-discharge	98.8 %						Teruya et al. (2022)
Ultrasound washing		28 %					Li et al. (2009)
Centrifuge	83.14 %	84.87 %					(Y. Zhang et al., 2018)

application. This can pose serious safety risks during LIBs handling and processing in recycling plants, including the potential for electrical shocks, short circuits, fires, and explosions. During recycling, LIBs can undergo mechanical stress, which can lead to unintended chemical reactions and thermal runaways (Hauck and Kurrat, 2018) leading to fires or explosions and eventually to the release of toxic substances (Werner et al., 2022), such as hydrogen fluoride (Zachmann et al., 2023).

Electrochemical discharging processes can corrode the casing of batteries cells (Kim et al., 2021; Rouhi et al., 2021), thus releasing chorine and alkane gaseous emissions (Wang et al., 2022), which are harmful to humans and the environment (Liu and Gao, 2021). Non-electrochemical discharge avoids corrosion of the battery casing (Mondal et al., 2024), however, it may lead to over discharge of the residual voltage, which causes release of gaseous product inside the cell, bulging the casings and creating an explosion hazard (Lee et al., 2023). Moreover, the presence of residual electrolytes and binders should be properly addressed to minimize safety risks. Electrolytes in LIBs typically contain flammable solvents, such as EC and DMC which can pose fire hazards if not properly handled during recycling (Gentilini et al., 2020). Some binders may contain toxic components or additives that can pose environmental and health risks if not properly managed during recycling (Brückner et al., 2020). Residual binder in the recycled materials may require additional treatment or disposal steps to ensure compliance with environmental regulations and to protect human health. Besides, standard solvents, such as NMP, for binder removal add environmental concerns due to their slow biodegradability and toxic effect on human health and the environment (Bai et al., 2021).

Besides, safer and environmentally friendly pre-treatments should avoid the use of acidic solutions. Mechanochemical activation is aided by acids, e.g. EDTA or H_2SO_4, however, recently more diluted organic acids, e.g. oxalic acid (Fan et al., 2018) were proposed. Besides, the use of acids could be avoided altogether by introducing only reducing agents, such as metals: Zn (Xie et al., 2021), Fe (Guan et al., 2017). Analogously, the use of flotation agents constitutes the main driver in the environmental impact among pre-treatments for chemical detachment of active material (Kar et al., 2025).

In conclusion, compared to landfilling, recycling EoL LIBs represents a safe alternative, both from the points of view of environment and human health protection (Kader et al., 2021; Vieceli et al., 2021; Zhao, 2017), with electrolyte being one of the main sources of pollution (Zhu et al., 2020) and residual voltage the main safety risks (Torabian et al., 2022). Safety issues associated with handling of EoL batteries may be overcome by thorough discharge of residual voltage (Diekmann et al., 2018b). Depending on state of health and state of charge of EoL LIBs, pre-treatment stages of disassembly and cells opening may pose safety issues such as fire hazard (Sommerville et al., 2020) or release of toxic substances (Stehmann et al., 2017). Due to the difficulties in handling organic and halogenic substances released during LIBs recycling pre-treatments (Brückner et al., 2020), dismantling procedures are performed in controlled atmosphere under fume hoods (Grützke et al., 2015b; Marshall et al., 2020; Sommerville et al., 2020).

Eventually, European regulation (European Commission, 2023) outlines the key safety precautions to take during LIBs recycling, such as hazard identification, equipment design, and strict quality control on the products and the processes. Table 2 summarizes the safety precautions identified by European regulations.

Table 2
Safety precautions for LIBs recycling proposed by European regulation (European Commission, 2023).

Safety area	Safety precautions during LIBs recycling
Hazard identification	• Analyse risks based on origin, composition, and reactivity of waste batteries
Equipment safety	• Design equipment to minimize risks (e.g. high voltage)
Product safety	• Address risks from abused or damaged cells (e.g., chemical release, flames).
Handling and storage	• Avoid shocks, heat, water, and temperature variation. • Fire precautions for damaged/defective batteries. • Follow UN regulations for packaging and storage.
Automated procedures	• Use automation to minimize human involvement and reduce exposure to dangerous situation

5. Discussion on economic concerns

EoL LIBs represent valuable secondary sources for metals recovery, particularly those in the cathodes (Zhang et al., 2021). Literature indicates that the concentrations of lithium, cobalt, nickel, and copper in batteries from electric vehicles are higher than in commercial ores (Leon and Miller, 2020). Economic feasibility of LIBs recycling can be enhanced through pre-treatments, as carbothermic reduction via microwave treatment (Fahimi et al., 2023), multi-steps precipitation (Yang et al., 2020), or cobalt purification through roasting (Barik et al., 2016) or reduction in Ar (Pindar and Dhawan, 2020b), and recovering graphite from anodes via froth flotation (Shin et al., 2020). However, a balance must be struck between the lower costs of thermal processing and the higher precision and profits of mechanical treatments (Leon and Miller, 2020).

Profitability in LIBs recycling is often hindered by the high processing costs due to numerous steps and significant energy demands. Labor costs for the initial dismantling stage also pose a substantial expense (Kay et al., 2019). Different configurations and modules number in LIBs are key aspects factoring in the disassembly cost, which can vary from 50.45$ for BAIC battery packs to 186.35$ and 194.11$, respectively for the models used in Peugeot 208 and Nissan Leaf (Lander et al., 2023). In particular, the automation of the disassembly line for LIB recycling requires higher capital investments; however, the potential profits it generates could yield a positive net present value ranging from 2.9 M$ to 14.8 M$, depending on the input flow of waste batteries (Choux et al., 2024). These costs could be reduced by optimizing hybrid disassembly procedures that combine manual labour with automation (Alfaro-Algaba and Ramirez, 2020; Gerbers et al., 2018; Thompson et al., 2021). Another challenge to profitability arises from low-value components and electrolyte solvents, whose market value don't justify their recovery, particularly in batteries with low cobalt content as LFP, expected to dominate the future LIB market (Brückner et al., 2020; Doose et al., 2021).

Furthermore, mechanochemical pre-treatments can improve the efficiency of downstream processes and reducing treatment costs. Mechanochemical activation with dry ice is a cost-effective solution for generating high-value lithium carbonates from spent lithium cobalt oxide (LCO) cathodes (M. Wang et al., 2021), while with Zn powder leaching efficiency of Li could increase from 30 % to 70 % (Xie et al., 2021). Similar effects can be achieved by thermal treatments for metals reduction (D. Wang et al., 2021), which however increase energy demand and subsequently operative costs. Novel solutions based on microwave heating offer a reduction of one order of magnitude in energy demand.(Fahimi et al., 2023). Moreover, cost reduction benefits can be realized by avoiding disposal costs for organic electrolytes, which can be decomposed into gaseous emissions during thermal pre-treatments (Xiao et al., 2017), and in the beneficiation of valuable elements through targeted processes of liberation (through attrition (Pinegar and Smith, 2020) or high voltage fragmentation (Leißner et al., 2018) or concentration processes (e.g. direct vacuum evaporation or distillation for Li recovery (Träger et al., 2015), air current separation (Bi et al., 2021) or froth flotation (Folayan et al., 2021; Zhan et al., 2018).

The economic viability of recycling processes can be significantly enhanced by reducing the consumption of reagents and energy, as these are the primary cost drivers (Woeste et al., 2024). Therefore, processes that operate at low temperatures and in dry conditions, without generating wastewater, are the most promising solutions for ensuring profitability. However, a comprehensive comparison of the economic balances across different processes is challenging, as it is highly dependent on scale and on the local context (e.g. available technology, cost of energy and labor). As a result, the lack of homogeneity in the scale of existing studies complicates such comparisons. In conclusion, the economic analysis of LIB recycling processes reveals that profitability is currently hindered by the high costs of state-of-the-art methods, which heavily rely on manual dismantling and energy-intensive pyrometallurgical and hydrometallurgical treatments. Additionally, the declining market values of recoverable components due to the prevalence of low-cobalt LIBs further challenge profitability. Thus, developing less expensive recycling treatments, particularly for battery chemistries like lithium iron phosphate and lithium manganese aluminum, is essential for future sustainability and economic viability.

6. Conclusions and prospects

Pre-treatments are crucial for achieving high efficiencies in subsequent LIBs recycling treatment stages. This review study classified pretreatment processes into four sequential categories: cell discharge, cell opening, thermal treatments (to remove organic components as electrolytes and binders), liberation of active materials from metallic current collectors and concentration of different material streams for specific recovery treatments. The state of the art and current research about pretreatments for LIBs recycling have made significant progress in addressing critical safety and economic aspects. However, further advancements are required to overcome existing challenges and optimize the overall efficiency, sustainability, and profitability of the recycling processes:

- Discharge is conventionally performed through immersion in 5–10 % NaCl solution. However, literature suggests that this technique releases hazardous gases and only provides a temporary discharge, posing safety risks during the initial dismantling stage. One promising solution to improve discharge processes is the development of optimized conditions for non-corrosive discharge solutions, which offer both environmental and economic benefits. This can be achieved by either replacing the materials used in battery casings with more resistant ones or by using salts in the discharge solution that inhibit corrosion. The latter approach stands out due to its potential for easy scalability, as the use of non-corrosive discharge solutions does not require significant capital investment in new equipment. Instead, conventional NaCl solutions can be replaced with alternative salt solutions.
- Disassembly can be done manually with greater precision but higher costs, or through automated lines. Recent trends focus on combining these two approaches, but they are hindered by diversity and complexity of battery designs. The optimization on disassembly processes could be implemented following different strategies. Modular design based on separable components reduce the time needed by disassembly and therefore labour costs. Besides, modularity in battery packs design is key to automize disassembly processes. Automating the disassembly process is not only about reducing labor costs but also about enhancing safety by minimizing human exposure to hazardous conditions.
- Residual electrolytes and binders are typically removed using thermal treatments or solvents to enhance the liberation of active materials from electrode current collectors. Despite NMP is the standard solvent for dissolving PVDF, its toxicity to human health and the

environment raises significant safety concerns. Although alternatives solvents, like DMSO and Cyrene, have been studied, they do not yet match the performance of PVDF.

- Liberation and concentration of electrode active materials is a critical step, essential for further recycling treatments (mainly hydrometallurgy), depends on the amount and purity of the active powder. Concentration benefits from the different properties of electrode components (such as specific mass, conductivity, or hydrophobicity), often requiring a combination of processes to handle all components and achieve optimal results. Integrating various technologies can enable the recovery of multiple material components, thereby maximizing the potential benefits of material recovery.

The findings of this work highlight how effective pre-treatments positively impact the efficiency and potentially decrease the costs of downstream recycling processes, which play a key role in advancing cleaner production technologies. Mechano-chemical treatments have demonstrated excellent performance due to their high recovery rates and the broad range of recoverable materials, providing a potential path toward more sustainable recycling practices. However, research on this topic is still limited and mostly focused on specific cathode chemistries. Future research should go deeper into mechano-chemical pre-treatments and in the recovery of anodic active materials, optimizing technical performance and minimizing economic and safety concerns. Addressing these gaps could lead to more sustainable and cost-effective recycling processes, driving the wider adoption of cleaner and more circular production technologies in the industry.

CRediT authorship contribution statement

Martina Bruno: Writing – original draft, Visualization, Methodology, Formal analysis, Data curation, Conceptualization. **Silvia Fiore:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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Data availability

Data will be made available on request.

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