

Li-ion Battery Recycling: A Summary Review

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Abstract – A short review of battery recycling technologies is presented. Main components and materials of battery cells, modules and packs are identified, highlighting their compositions and contents. Main component of packs is aluminium, followed by the cathode materials, anode materials, copper and polymers. Safe discharge of the spent batteries is mandatory, and the subsequent dismantling allows separation and valorisation of the aluminium casings, electronic components, polymers and steel. The resulting cells or modules are pretreated by shearing and drying, and the solvent and evolving gases shall be safely treated and disposed. Physical separation operations, including secondary shredding, sieving, gravity separation, among others, are applied to separate the Al, Cu and polymer fragments from the electrode powders (black mass). Metallurgical processing, by hydro or pyrometallurgy, allows further recovery of the metals in pure forms, which are then sent back to the market. Nowadays, several hydrometallurgical processes are being introduced, allowing high recovery yields of the most important metals such as lithium, cobalt, nickel and manganese. By this way, a more sustainable management of the resources can be achieved.

Keywords: Li-ion batteries; Recycling; Pretreatments; Pyrometallurgy; Hydrometallurgy

1. Introduction

The decarbonization of the economy by the so-called energy transition implies the implementation of “green technologies” to the energy conversion, energy storage and mobility. Batteries have a crucial role in this transformation, as they are a versatile energy storage technology, applied whether in vehicles, microgrids, energy communities, and the electrical grid. For this transition, Li-ion battery technology needs huge amounts of materials like lithium, cobalt, nickel, manganese, copper, graphite, therefore their sustainable management involves the responsible extraction of raw materials, the substitution of less available materials, and, finally, the recycling of end-of-life batteries.

Li-ion battery recycling is very demanding due to the high complexity of assembly, and to the large variety of constituting components and materials. Safe, versatile and efficient processes are needed to allow maximum recovery of the important battery components. This paper presents a short review of the state-of-the-art of battery recycling, addressing topics such as battery composition, as well as recycling processes and technologies.

2. Materials in Li-ion Batteries

Li-ion batteries are composed of cells that can be assembled in modules and packs. The assembling can be arranged in series or parallel, allowing to define the voltage and the capacity. The size depends on the application, ranging from small mobile phone batteries to the large-scale e-vehicles battery and even larger battery blocks used in electric grid energy storage.

Three main commercial cell shapes have been identified: pouch, prismatic and cylindrical. In pouch cells, the electrodes are stacked in rectangular flat sheets, while in cylindrical cells, the electrode arrangement is in jelly-roll form. In prismatic cells, a different arrangement is typically observed; the electrode sheets are wrapped in a pseudo-prismatic roll. The cell interior consists of sheets of cathode, separator and anode.

The cell is composed of three core elements, namely:

- The cathode, where the reduction semi-reaction occurs (in discharge conditions and the opposite when charging). Common cathodes are oxides of Li and a transition metal M (Co, Ni, Mn, Al), with formula LiMO_2 , where Li is in monovalent state while the transition metals are in trivalent (average) state, as well as lithium iron phosphates [1].
- The anode, where the oxidation semi-reaction occurs (in discharge conditions and the opposite when charging), is generally composed of graphite, which can intercalate Li ions in its structure when charged.
- The electrolyte, which provides charge transport, in ionic form, between the two electrodes, closing the circuit, is also significant. In LIBs, the electrolyte used is the salt LiPF_6 , dissolved in an organic solvent: dimethyl carbonate (DMC), diethyl carbonate (DEC) or ethyl-methyl carbonate (EMC), among others [2], and their mixtures.

The most common commercial cathodes are:

- LCO – Lithium cobalt oxide, LiCoO_2 , has a layered structure.
- NCM – Lithium nickel-cobalt-manganese oxide, $\text{Li}(\text{Ni}_x\text{Co}_y\text{Mn}_z)\text{O}_2$, with $x,y,z=0.33$ (NCM333 or NCM111), $x=0.6, y,z=0.2$ (NCM622), or $x=0.8, y,z=0.1$ (NCM811), among other combinations. The NCM has a layered structure.
- NCA – Lithium nickel-cobalt-aluminium oxide, $\text{Li}(\text{Ni}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05})\text{O}_2$, has a layered structure.
- LMO – Lithium manganese oxide, LiMn_2O_4 , has a spinel structure.
- LFP – Lithium iron phosphate, LiFePO_4 , has an olivine structure.

NCM cathodes are the more widely used and the current trend is to decrease Co content by increasing the Ni content, due to the disadvantage of using cobalt (scarcer, with supply problems and more expensive, thus classified as critical). Recently, alternative chemistries have been proposed, including the Li and Mn-rich layered cathodes (LMR-NCM), with general formula $x\text{Li}_2\text{MnO}_3 \cdot (1-x)\text{LiMO}_2$ ($\text{M}=\text{Ni}, \text{Co}, \text{Mn}$). These cathodes exhibit higher specific capacity and energy density; however, further developments are necessary to improve certain characteristics and stability [3,4], such as initial capacity loss, voltage and capacity losses during cycling, and electronic conductivity issues. Other chemistries in development include the evolution of the LFP system by introducing doping metals like manganese (LMFP), to overcome the disadvantages of the LFP system of low cell energy density.

Regarding the anodes, several candidates for graphite substitution have been proposed for the coming years [5], including nanostructured carbons, metal alloys, Si-C composites, silicon metal, Si-Li alloys and, eventually, lithium-metal, with an anticipated substantial increase in cell specific capacity relative to current graphite usage.

Both the active electrode materials (graphite and Li-metal oxide) are used in powder form. The film powders are deposited as a thin layer on current collector foils, which are made of aluminium (in the cathode) and copper (in the anode) with the addition of a binder (PVDF, but also CMC in the case of the anode) that promotes adhesion [6]. In the cathode, the powder also contains a certain amount of carbon to allow conductivity between the oxide electrode particles.

The electrodes are separated by a layer of fibrous polymeric material – the separator – which allows the transfer of electrolyte ions but avoids electric contact between the electrodes (preventing short-circuit). Separators are made of microporous polyolefin, namely polypropylene, polyethylene, or mixtures of both, in intercalated films, normally trilayers [7,8]).

The average composition of a LIBs pack is shown in Fig. 1, calculated from data taken from literature. The cells represent 56% of the total pack weight, the module housing stands for 10wt%, the pack housing for 29% and the electronics/cables for 5.0%. By aggregating similar materials existing in different parts, the materials/substances balance, depicted in Fig 1b, shows that aluminium is clearly the most relevant metal, representing 33%; beyond pack and module housings, aluminium is also present in the cells, namely in the foils and from cell housings. Cathode materials are near 15%, while carbon (essentially graphite from the anode) represents 10%. Copper from cables and wiring, electronics and from cell components, stands for 10%, while stainless steel is 9.5%. Polymers accounts for approximately 11%, existing in several parts of packs, modules and cells.

It can be stated that, in general, the cells are approximately 50% of a pack with the cathode and anode materials accounting for around 50% of the cell, or 25% of the pack. These values can be utilised to convert a metallic content of a cell to a pack, or vice-versa. As an example, it can be said that the Li content in the electrode materials (anode and cathode mixture) is about 4%, indicating that a cell contains near 2% Li, or alternatively, a pack contains 1% Li.

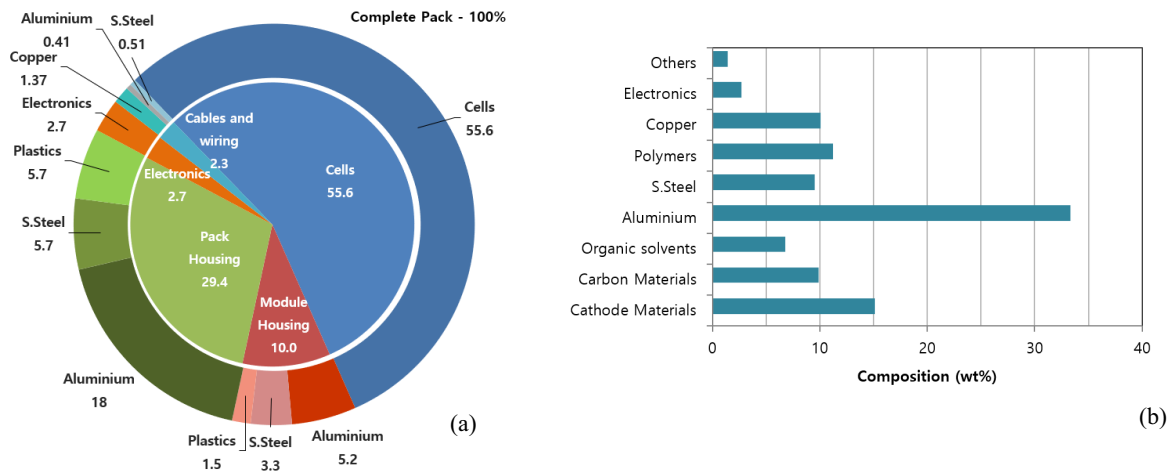


Fig. 1: Materials balance of a Li-ion battery pack of an electric vehicle: (a) distribution by main components; (b) distribution by main materials/substances.

Table 1: Overall average materials balance of Li-ion cells.

Component	Average (wt%)	
Cathode	42.2	
Al foil	10.8	
Powder	31.4	
Electrode material	27.2	
Binder	2.4	
Carbon	1.8	
Anode	32.9	
Cu foil	15.6	
Powder	17.3	
Graphite	15.9	
Binder	1.4	
Electrolyte and Separator	15.7	
Electrolyte	12.2	
Solvent carrier	12.2	
Salt (LiPF ₄)	0.056	
Fibre separator	3.5	
Casings and others	9.2	
Cell case	6.6	
Others	2.6	

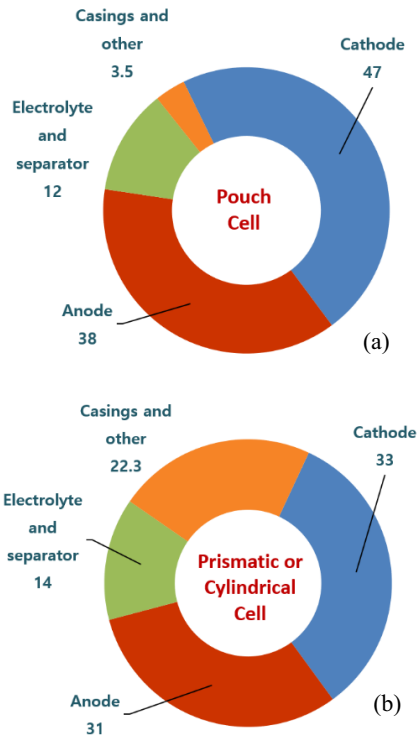


Fig. 2: Specific cases of materials balance of Li-ion cells: (a) pouch type cell; (b) prismatic or cylindrical cell.

Regarding the cells, the material weight distribution is presented in Table 1. It is evident that the cathode plates contribute 42% of the total weight, the anode 33%, the electrolyte and separator 16%, and the casings and other components 9%. Fig. 2 emphasizes some differences in composition between pouch-type cells and cylindrical or prismatic-type cells. The later exhibit a higher concentration of casing materials and, proportionally, a lower concentration of electrode powders. Concerning the metal contents of cells, typical values are 1.6-2.5% Li and 15-20% transition metals (Ni, Co, Mn) for NCM-type chemistry, and 1.1-1.3% Li and 9-11% Fe for LFP-type chemistry. Spent batteries are therefore excellent metal concentrates, constituting a very interesting secondary raw material.

3. Recycling of Li-ion Batteries

The recycling of batteries is a very complex process involving several steps, from the initial spent packs to the final products, with relevance to metal commodities that can be used to manufacture new electrodes. Two main steps can be identified (Fig. 3): (a) pretreatment, and (b) metallurgical processing. Large-size batteries, namely the industrial ones such as those used in electric vehicles, are always deactivated (discharged) and dismantled into modules or cells. The cell's internals are then accessed through a first stage of treatment by coarse crushing, where the volatiles (organics) are removed, and the resulting solids follow the metallurgical treatment. LIBs of portable devices do not require discharge or dismantling. Two metallurgical routes are used: pyrometallurgy or hydrometallurgy. The latter requires a preliminary physical treatment to concentrate the electrode powders and to remove the coarse scrap.

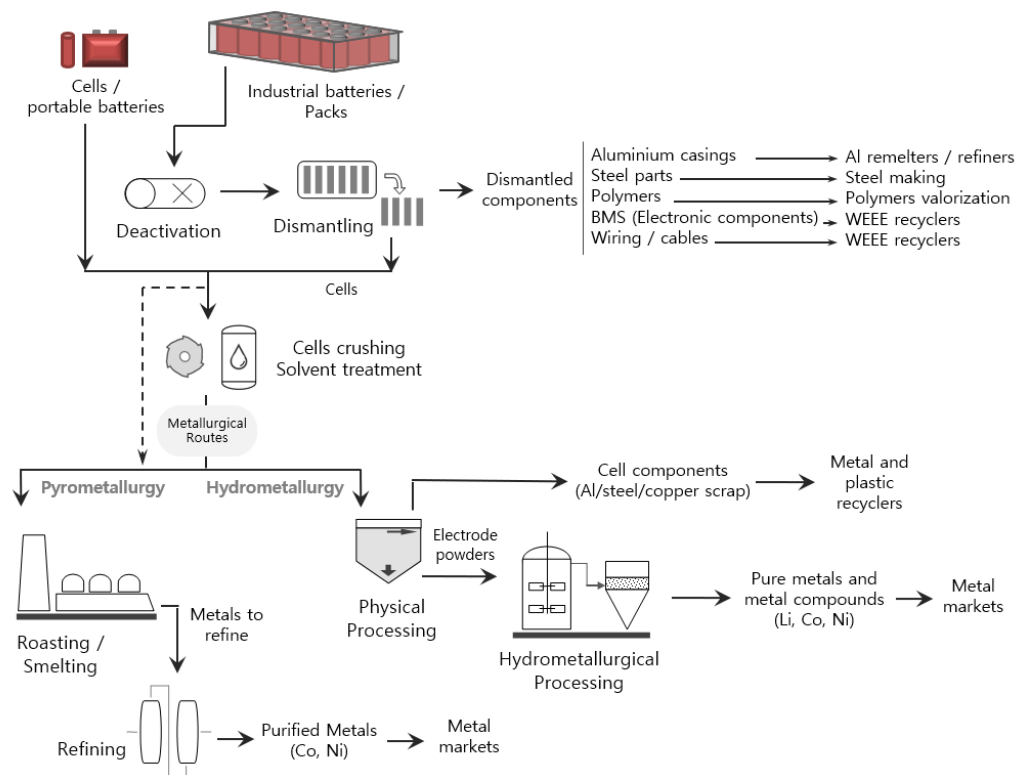


Fig. 3: Diagram of the main steps of the LIBs recycling.

3.1. Deactivation and Dismantling

Battery pack voltages are high, averaging at approximate 400V; however modern batteries can exhibit significantly higher voltages. Therefore, the handling of spent batteries must be conducted with the utmost caution due to the potential risk of fire or explosion, attributable to the ignition of the electrolyte in the event of elevated temperatures, causing the

thermal runaway. This phenomenon can arise as a consequence of short-circuiting, namely if batteries came from inadequate usage, accidents or internal malfunctions. Handling by skilled professionals is mandatory. As first step of pretreatments, deactivation is strongly recommended by means of electrical discharge, freezing, immersion in an aqueous electrolyte or other procedures [9,10,11]. Ideally, discharge should be facilitated by mechanisms that allow recovering the stored energy. Afterwards, the dismantling of large (industrial) LIBs can be carried out with greater safety [12], allowing to recover several components of the packs and modules, such as the peripheral casings (predominantly aluminium), steel, some plastics and electronic components. All these fractions have valorising solutions in already established recycling circuits. Electronic components such as the battery management system (BMS) and wiring/cables can also be valorised by recyclers of waste of electric and electronic equipment.

3.2. Pre-metallurgical Treatments

After the dismantling, a series of operations prior to metallurgy are applied. The mechanical processing of battery cells or modules is carried out by means of coarse crushing or shredding [9,13], usually involving the use of rotary shears. This step aims to access the interior of cells, exposing their components for subsequent treatment. Special care is required to avoid overheating, due to mechanical action, with the potential for ignition and fire. It is also important to note that small batteries are not discharged during this process, which can lead to an increase in temperature during crushing. This operation is typically carried out in an environment with low oxygen concentration, either by purging nitrogen or carbon dioxide, or by conducting the process under vacuum. Cryogenic treatment can be applied to reduce reactivity [13]. Released gases containing part of the solvent (from the electrolyte carrier) as well as fluoride compounds (from the decomposition of the electrolyte salt) need to be properly treated. Drying of solids to remove all the solvent is a subsequent step. The solvent is then condensed and collected, or, alternatively, extracted by organics such as NMP (N-methyl-2-pyrrolidone) [9,13]. Reusing the solvent may be a possibility, although it is not normally practiced. The operations described here can be avoided if the destination is the pyrometallurgical processing, as it will be discussed later.

After shredding and solvent removal, a series of physical separation operations are applied (Fig. 4) to separate the electrode powders – a concentrate called black mass – from the scrap. A secondary grinding can benefit the liberation of the powders by using more intensive impact milling such as hammer mill, cutting mill or other adequate shredding options [14]. Some processes claim the use of a thermal treatment to remove or to weaken the binders, thus facilitating particle liberation. Several physical operations can be used [11,15], and will be described below:

Sieving: this is a method of separating the electrode powders from coarse materials containing fragments of the separator, the Cu and Al foils and the casings.

Magnetic separation: this is a process that allows for the removal of iron magnetic materials.

Density separation / elutriation: Several techniques can be applied to separate particles of different densities and shapes. The use of the zig-zag column classifier, for instance, utilises an ascending air flow to drag light particles such as plastics.

Eddy current separation: allows separate non-ferrous conducting metals such as Al and Cu, to be separated from other materials by repulsion from a magnetic field.

Vibrating tables: with an appropriate slope, speed and vibrating intensity, enhance the separation of metal particles of different densities such as Al and Cu.

The final powder concentrate, the black mass, is then sent to the hydrometallurgical processing stage.

3.3. Metallurgical Processing

3.3.1. Pyrometallurgy

The pyrometallurgy involves high temperature processing, where the cells containing the valuable metals are melted and further refined and recovered [16]. It is considered a straightforward method for managing scrap batteries, as it does not necessitate any physical pretreatment. The application of high temperatures causes the cells, modules or small packs to lose integrity and break down. Additionally, the carbon and organic materials (e.g. solvents and polymers) are burnt allowing energy recovery within the furnaces. However, if the process cannot easily handle some types of contaminants, the previous steps of crushing and organics removal can be implemented prior to pyrometallurgy. These facilities are usually operated at

high capacity to optimize energy balance, given the energy-intensive nature of these processes. Prior to melting, preheating or roasting may be applied to allow batteries to break and to remove the organics. The charge is then heated to temperatures of up to 1500°C, where the valuable metals such as cobalt and nickel are melted and recovered, while other metals such as lithium and aluminium are slagged. Consequently, the pyrometallurgical approach is focused on cobalt and nickel, with lithium being lost. The possibility of Li recovery from the slag is always a technical option, but slags are very refractory to chemical attack and would require an intensive grinding to liberate Li-bearing particles and dissolve them. The thermal route is also usually associated with gaseous emissions, which require special attention to control. If organic combustion is incomplete, hazardous gas emissions can occur. Examples of industrial applications include the Umicore, Inmetco, Accurec and Nippon Recycling processes.

3.3.2. Hydrometallurgy

The hydrometallurgical route has been shown to be a more versatile process, with the capacity to maximize metal recovery (Li included), and to be adapted to a wide range of treatment capacities and has lower energy consumption. It is also easily adaptable to variations in metal contents in the feed (an important consideration due to the permanent change in the cathode chemistry). However, it should be noted that these processes have more unit operations and are normally associated with high reagent consumption and the need for wastewater treatment. Due to versatility, maximum metal recovery and less energy needs, more recent industrial processes have been developed by hydrometallurgy.

As previously referred, the hydrometallurgy option is always associated with a previous physical process (Fig. 4). After shredding and solvent removal, a series of physical separation operations are applied to separate the black mass for chemical treatment. Hydrometallurgy is a complex series of operations [15,16,17], the most relevant of which are described below:

Leaching: this is the core operation, where the metals are dissolved in aqueous media in the presence of appropriate reagents, including an acid (e.g. sulfuric acid or hydrochloric acid) and a reducer (e.g. hydrogen peroxide or a sulphurous agent). The role of the reducing agent is to enhance the leaching of transition metals (Co, Ni, Mn), by lowering their oxidation state to (+2), thus increasing their solubility. The final solution contains the metals (the previously referred and lithium) in soluble sulphate or chloride forms, depending on the acid used. Other leaching media have been proposed (e.g. organic acids, such as oxalic, citric, among others) but not industrially applied. The insoluble solids, desirably a carbon-rich concentrate (essentially graphite), can be valorised, though this necessitates further processing. The recyclers normally do not claim valorisation of this stream. The aqueous leachates undergo hydrometallurgical separation and recovery.

Separation operations: some leachant contaminants like Fe, Cu and Al can be initially removed by a controlled neutralization at relatively low pH values or by cementation (case of copper). Further precipitation of transition metals prior to lithium is a possibility, namely as hydroxides by neutralization with an alkaline agent until a higher pH is attained, but this option will lead to a mixed precipitate (Ni,Co,Mn)OH₂. Nevertheless, this product can be valorised and refined in other metallurgical plants or eventually used as a raw material to produce new cathodes. Some years ago, recycling plants dealing only with LCO batteries could produce a pure cobalt hydroxide product, but this is no longer the case with the emergence of new LIB systems. When the maximum valorisation of the metals is required, other operations can be applied, the most efficient of which is solvent extraction (SX). SX involves the use of specific organic solvents, that are immiscible with water, selectively extracting certain metals which are then stripped, yielding a new aqueous solution containing a single metal. Cobalt-nickel separation by SX is well documented in the metallurgical processing of Co-Ni mineral resources, and it has been successfully employed in the recycling of batteries. Other separation operations can be applied such as ion exchange or specific/selective precipitation methods, can also be used as final purification/refining steps.

Recovery operations: After separation, the pure metal streams dissolved in aqueous solutions can be recovered as commodities. Cobalt and nickel can be recovered as metals by electrolysis or as compounds by precipitation (hydroxides, oxides, carbonates) or crystallization (e.g. as sulphates). Concerning lithium, the most usual recovery method is through the process of precipitation as carbonate, but the alternative of producing lithium hydroxide has nowadays been considered, as it serves as an optimal precursor for the synthesis of cathode materials. Production of lithium hydroxide can be accomplished through the implementation of a membrane process, specifically electrodialysis, a method that has recently been considered as a potential upgrade to some lithium metallurgical processes and further expanded to encompass recycling processes.

Examples of industrial applications are the JX Nippon Mining and Metals, Recupyl/Valibat, Retrieval and Duesenfeld processes.

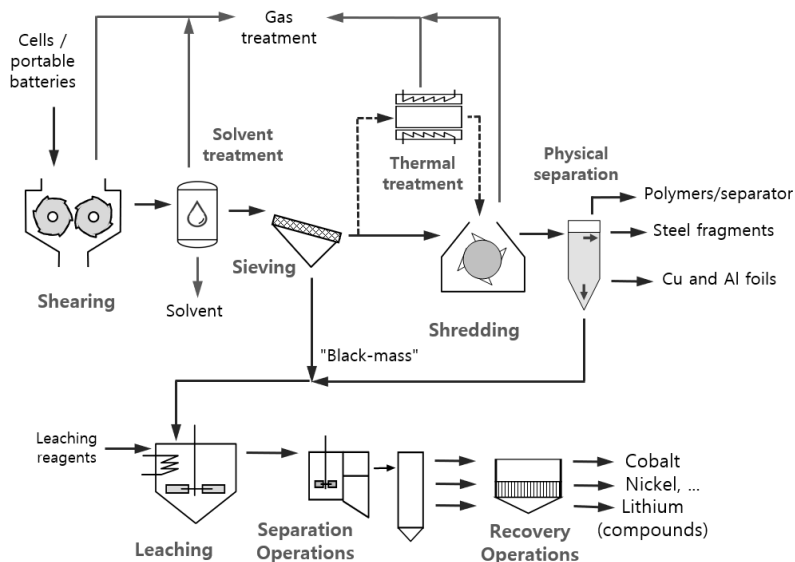


Fig. 4: Main operations of pretreatments (after dismantling), physical processing and hydrometallurgical recovery.

3.4. Direct Recycling

Direct recycling is the third method of battery recycling [18,19], in which powder materials of the electrodes are recovered in their pure forms to allow for the adjustment of composition and the remanufacturing of new electrodes. Normally, it involves the recovery of the cathode, and eventually the anode, along with other components, through dismantling and physical separation, without or with minimum chemical treatment. The difficulty is to obtain materials with enough purity to be directly recycled to produce new battery components, and for that aim the separation steps are quite challenging.

4. Conclusions

The recycling of LIBs is a process that involves a series of complex steps and operations. In the pre-treatments, safe discharge is mandatory, and dismantling enables the recovery of components (aluminium parts, plastics and electronic materials) that are then sent to established recycling circuits. In the following pre-treatments (also called pre-metallurgical), dismantled cells or modules need to be opened through a primary coarse grinding that also allows the removal of the organic solvents from the electrolyte and other volatile substances. The pre-metallurgical operations include secondary shredding and physical separation techniques, removing metal scrap (essentially Al and Cu) and other polymers, and producing an electrode powder concentrate (black mass). Metallurgical processing, by pyrometallurgy or hydrometallurgy, allows the recovery of metals in their purest forms. Pyrometallurgy is more energy-intensive and is focused on recovery of cobalt and nickel, while hydrometallurgy is more complete regarding metals recovery (lithium included), but is more reagent-consuming. Current processes are mostly carried out via the hydrometallurgical route, involving operation such as leaching, precipitation, and solvent extraction. The recycling of metals promotes circularity, reduces the risk of supply chain disruptions, and ensures the availability of raw materials for battery production, thereby contributing to the ongoing progress of the energy transition.

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