REINFORCE

Deliverable 2.3

Materials requirements for battery recycling processes

05/09/2023

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Terms and abbreviations

Al	Aluminum
BM	Black Mass
CAM	Cathode Active Material
Co	Cobalt
Cu	Copper
CRMs	Critical Raw Materials
EVs	Electric Vehicles
EoL	End-of-Life
ETIP	European Technology and Innovation Platform
EU	European Union
LIBs	Lithium-ion Batteries
LFP	Lithium-Iron-Phosphate
Ni	Nickel
NMC	Nickel-Manganese-Cobalt
SOC	State of Charge
SOH	State of Health
VOCs	Volatile Organic Compounds

1. Executive Summary

This document provides an in-depth exploration of the activities carried out within the REINFORCE project, with the primary objective of defining the optimal technical prerequisites for recycling End-of-Life Lithium-Ion Batteries (EoL LIBs). It comprehensively addresses critical facets and considerations related to EoL LIB recycling, including their chemical characteristics, environmental implications, economic viability, and logistical intricacies. Specifically, it delineates the criteria used to assess the suitability of various recycling pathways and offers a comprehensive examination of the pros and cons associated with each decision-making process. Overall, this document lays out the essential requirements that will underpin the recycling of EoL LIBs characterized by diverse chemical compositions.

2. Introduction

WP2 will play a pivotal role in laying the foundations for the successful execution of REINFORCE. This will be achieved by gathering inputs from every stage of the value chain within the EoL batteries sector. These inputs will be instrumental in identifying and precisely defining the essential needs, key performance indicators (KPIs), specifications, and requirements that are indispensable for meeting the exacting demands of the battery industry.

Its objective encompasses (i) defining required data from processing partners to develop decision-making tools for risk identification, automation, costing and cut-off point definition for EoL batteries; (ii) pinpointing needs and requirements of users involved in secondary and tertiary applications; (iii) compiling the technical prerequisites for an optimal recycling method; (iv) establishing the optimal requirements for the development of a helpful battery passport; and (v) establish the design of the disassembly process.

Deliverable (2.3) presents the outcomes derived from task 2.2, which centers around the user requirements for recycling. This task focuses on the pre-processing of EoL batteries that are unsuitable for secondary and tertiary usage. This report establishes a compilation of technical prerequisites for effective recycling. These requisites consider specific cell chemistries (such as NMC and LFP) and the chosen recycling process methodology (such as hydrometallurgical processes or/and direct recycling). A set of KPIs will be derived based on the Battery 2030+ long-term roadmap for recyclability (Figure 1). This will help guide collective research efforts to transform the way batteries are designed, manufactured, and used in real applications.

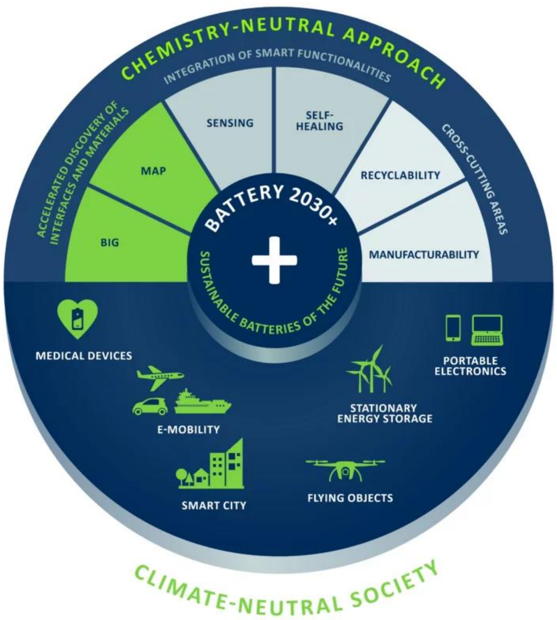

Figure 1. BATTERY 2030+ – a long term-roadmap for forward looking battery research in Europe (1)

A set of target KPIs (Table 6) are proposed base on the three themes established in the Battery 2030+ Roadmap. The latter suggests three overarching themes encompassing six research areas needed to invent the sustainable batteries of the future (table 1).

Table 1. Battery 2030+ Roadmap themes description.

Themes	Description
Accelerated discovery of battery interfaces and materials.	New sustainable materials with high energy and/or power performance that exhibit high stability towards unwanted degradation reactions.
Integration of smart functionalities.	Enhance the lifetime and safety of batteries
Cross-cutting areas.	Manufacturability and recyclability addressed at early stage in the discovery process.

2.1 LIBs recycling overview

The European Union (EU) is committed to countering climate change by transitioning to a sustainable energy framework. Batteries play a pivotal role in electrifying various sectors including transportation, power generation, and industrial applications. The global battery demand, predominantly fueled by the growth of E-mobility and stationary uses, is projected to experience a robust 25% annual increase, potentially reaching a capacity of 2,600 GWh by 2030 (2).

Within this landscape, critical R&I ecosystems and public-private partnerships such as the European Technology and Innovation Platform (ETIP) (3) and the Batteries European Partnership Batt4EU (4) have identified a pivotal goal: to forge sustainable and economically viable recycling procedures to accommodate the impeding influx of EoL batteries. In this line, the EU's battery regulation (5) have set precise recycling targets encompassing recycling yield, material recovery rate, and integration of recycled materials within new batteries (table 2).

Table 2. Recycling targets set in the proposed EU Battery Regulation (5)

Measure	Medium level of ambition	High level of ambition
Recycling efficiencies and recovery of materials	**Lithium-ion batteries and Co, Ni, Li, Cu:** - Recycling efficiency lithium-ion batteries: 65% by 2025 - Material recovery rates for Co, Ni, Li, Cu: resp. 90%, 90%, 35% and 90% in 2025 **Lead-acid batteries and lead:** - Recycling efficiency lead-acid batteries: 75% by 2025 - Material recovery for lead: 90% in 2025	**Lithium-ion batteries and Co, Ni, Li, Cu:** - Recycling efficiency lithium-ion batteries: 70% by 2030 - Material recovery rates for Co, Ni, Li, Cu: resp. 95%, 95%, 70% and 95% in 2030 **Lead-acid batteries and lead:** - Recycling efficiency lead-acid batteries: 80% by 2030 - Material recovery for lead: 95% by 2030
Recycled materials content in new batteries	16% cobalt, 85% lead, 6% lithium, 6% nickel, must come from recovered battery manufacturing waste or post-consumer waste by 2031	26% cobalt, 85% lead, 12% lithium, 15% nickel by 2036
Collection rate for light means of transport batteries	45% by 2028	61% by 2031

Overall, the recycling of LIBs can be executed via three distinct avenues: (i) pyrometallurgical, (ii) hydrometallurgical, and (iii) direct recycling processes (Figure 2). These approaches aim to recover all essential components from the battery, including the cathode material as well as other integral components such as the anode material, current collectors, and electrolyte. For example, the pyrometallurgical and hydrometallurgical approaches involve breaking down the cathode material into its elemental components to recover the so called Critical Raw Materials (CRMs). Conversely, the direct recycling approach sharply centers on restoring the cathode active material without breaking its individual constituents apart. The process itself is also known as cathode healing as the atomic Li deficiency of the spent cathode is rectified while maintaining its crystalline microstructure.

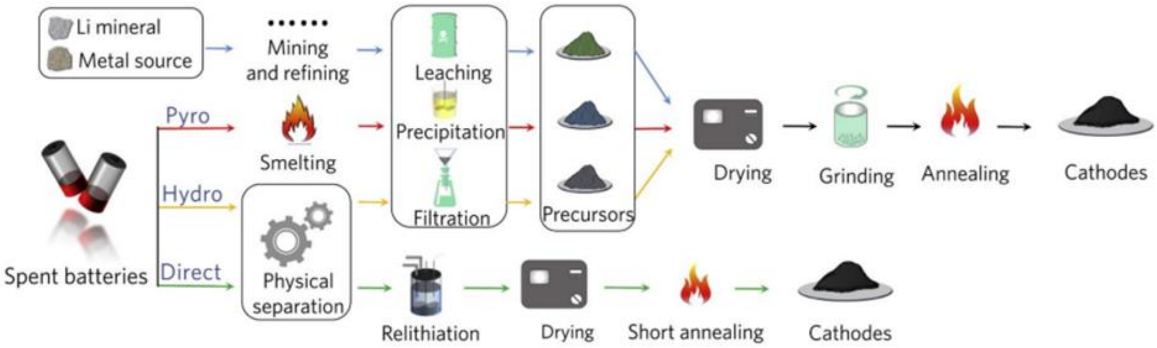

Figure 2. LIBs recycling approaches (6)

The choice of a recycling method is intricately tied to the chemistry of the battery. For nickel (Ni) and cobalt (Co) rich batteries, the pyro/hydrometallurgical methods prove optimal for extracting the high valuable metals from the cathode active material (CAM). In contrast, for batteries comprising component of lesser value, direct recycling emerges as the preferred route, as the recovery of individual CAM precursors lacks economic viability (7).

At present, nickel-manganese-cobalt (NMC) LIBs hold a predominant position within the electric vehicles (EVs) sector (8). This dominance is mainly attributed to the high energy density achievable by the Ni-rich cells. Nevertheless, the elevated cost associated with the precursor metals required for CAM production has prompted battery manufacturers to explore more cost-effective alternatives, resulting in the emergence of lithium-iron-phosphate (LFP) LIBs as prospective substitutes.

As indicated by the analysis published by Wood Mackenzie Power & Renewables, LFP batteries are anticipated to outpace NMCs in terms of market share by 2028 (9). While LFP batteries currently find greater utility in stationary applications owing to their comparatively low energy density and safety profile, their intrinsic qualities such as thermal stability and extended life cycle, coupled with the cost-effectiveness of their components and reduced supply chain vulnerabilities, open up the potential for adoption of LFP in more cost efficient EVs.

In light of these considerations, REINFORCE project will feature both NMC and LFP chemistries battery packs. These chemistries have been selected as representatives of the existing and forthcoming compositions within the LIBs market.

EoL EVs may retain as much as 80% of their initial capacity (10), making them viable for repurposing in other applications, notably in stationary storage applications. Within REINFORCE' overarching scope initiative, a meticulous assessment of these batteries will precede any recycling considerations, with primary focus on their potential for secondary and tertiary usage. For these batteries to be eligible for their secondary and tertiary life applications, they must satisfy specific requirements, including the maintenance of specific state of health (SoH) and capacity (11). All these prerequisites will be meticulously outlined in the forthcoming deliverable 2.1.

In the context of recycling, the potential recyclability of an EoL battery hinges on the fulfillment of specific safety requirements. These requirements are closely linked to potential safety concerns arising from various forms of abuse that the battery undergoes throughout its lifespan. Such abuse encompasses mechanical, electrical, chemical, and thermal factors (12). In order to ensure an effective and secure recycling process, the initial input material, in this case, an EoL battery pack, must meet the material prerequisites established for each phase of the recycling sequence. The specific requisites for each stage are thoroughly delineated in the subsequent section.

NMC batteries

The most effective technoeconomic method for the recycling of NMC-based EoL LIBs is the hydrometallurgical approach. This is primarily due to the value of the metal's components that make the battery (i.e., Co, Ni) and that can be recovered in the process. In essence, the hydrometallurgical technique enables the targeted separation of valuable metals from battery waste, converting them into a liquid phase that subsequently facilitates their recovery in the form of salts. This process specifically targets the CAM with the aim of breaking it into its individual metal components. For this, the cathode material must be first isolated from the rest of the battery components, which is typically done by a combination of mechanical and thermal pre-treatment operations. The hydrometallurgical process itself is less energy-demanding than the pyrometallurgical process, but the use of strong acids and other chemicals commonly results in the generation of high volumes of waste streams (13).

LFP batteries

When dealing with LFP cells, it doesn't make economic sense to break down the LFP CAM structure and individually separate the lithium (Li^+) from the iron phosphate ($FePO_4^-$), since the value of the LFP resides in its crystalline structure rather than in its individual components (Figure 3).

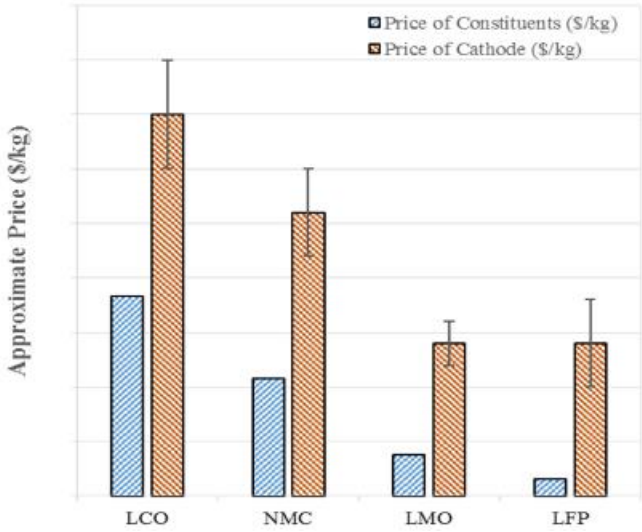

Figure 3. Price of cathodes vs constituents (7)

Instead, a more suitable method for these lower-value materials is the direct recycling approach. This approach involves restoring the LFP cathode's crystal microstructure and compensating for the lithium deficiency in the spent cathode, effectively repairing the LFP CAM itself. Compared to the current pyro/hydro processes, the direct recycling process offers numerous advantages, including reduced energy consumption and lower chemical usage (14). Additionally, it establishes a closed-loop recycling system that eliminates the need for energy-intensive CAM synthesis. However, the success of this process heavily relies on the purity of the spent CAM. Consequently, the battery must be disassembled down to the electrode level to ensure proper separation of the cathode material from other battery components. Following this step, the isolated CAM undergoes further purification to achieve a pristine state. Although the industrial disassembly of batteries into modules, cells, and electrodes is not currently feasible, the REINFORCE project aims to automate these disassembly stages, potentially allowing for the scalability of the direct recycling process. Another avenue for scaling up direct recycling involves directly restoring the black mass (BM). Certain studies propose the option of repairing the CAM using the black mass material generated from a mechanical shredding recycling process (15).

3. Requirements for ideal recycling

3.1 NMC batteries

In the hydrometallurgical process, the battery packs must be first deep discharged to prevent any unwanted side reaction from taking place. There are different possibilities for battery discharging, including physical and chemical methods (16). Insufficient discharge can lead to a thermal runaway during the disassembly or shredding process. The thermal runaway can be triggered by two different mechanisms: Firstly, residual electricity may cause an uncontrolled rise in temperature upon the short-circuiting of the anode and cathode (17). Secondly, a thermal runaway can also result from the presence of Li in the anode, which accumulates in the form of lithiated graphite and can react exothermically with water vapor from the air, triggering a thermal runaway (figure 4) (17).

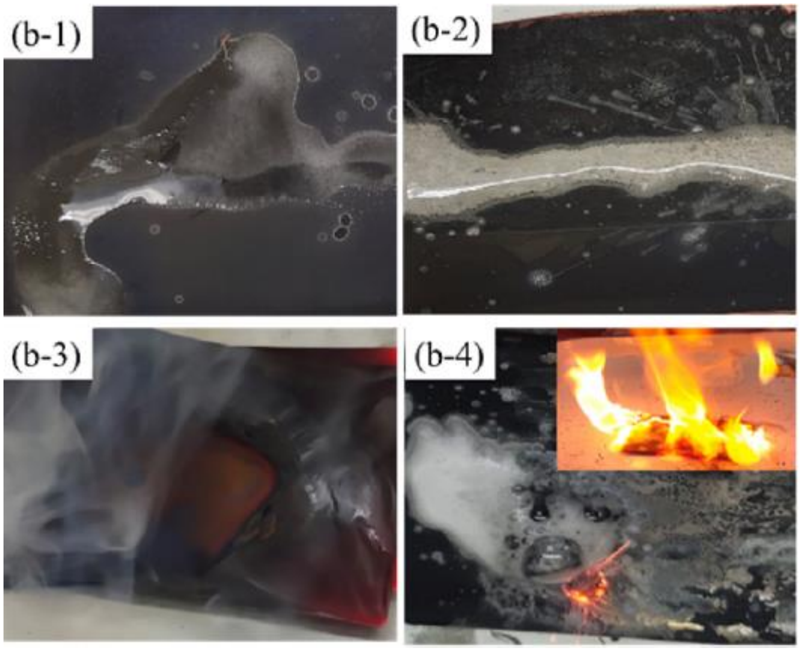

Figure 4. Anode reaction with water at different voltages (b1-0V, b2-2V, b3-3.7V, b4-3.8V) (17)

Ensuring safety during the disassembly and shredding process depends on the battery's state of charge (SOC), which is directly proportional to its voltage. Lower state of charge implies a reduced risk of fire or explosion. However, achieving a complete discharge (i.e., 0 V) is challenging due to the polarization of LIBs. Several research studies have observed a phenomenon known as 'voltage relaxation,' where the voltage increases after the discharge process (18). Wu et al. (17) determined that a safety voltage threshold of 1.5V can help prevent potential runaway scenarios (as depicted in the figure 5), which was verified through various abusing tests.

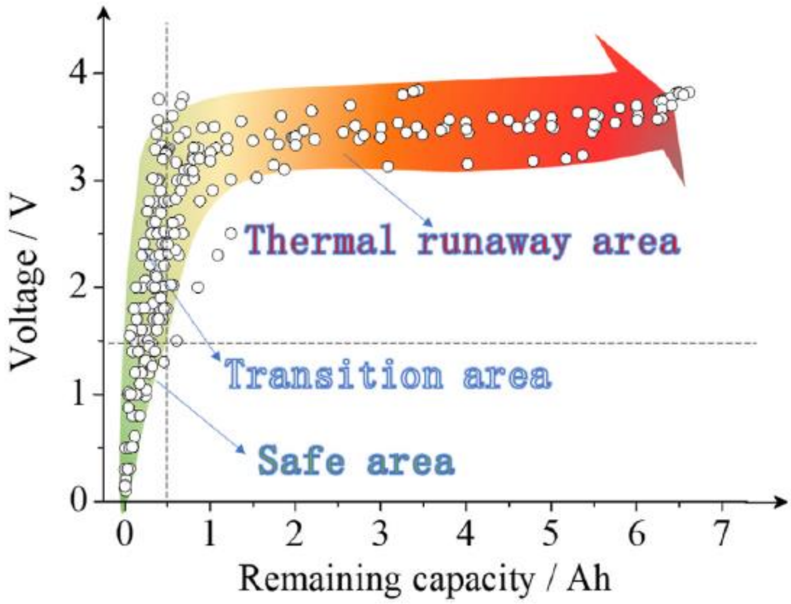

Figure 5. Correlation between potential thermal runaway and voltage (17)

Concerning the disassembly process, it is imperative to dismantle the battery packs to the module level, at a minimum, to ensure compatibility with the shredding size system. Another aspect of concern in battery recycling pertains to potential physical damage. This is crucial due to the possibility of safety hazards arising from electrolyte leakage and short circuits (12).

The modules are then mechanically pulverized and sorted. To shred the battery cells into individual components, the shredding process must take place in a closed system under inert atmosphere. Without it, the electrolyte (i.e., $LiPF_6$) is prone to react with air moisture producing corrosive HF in the system. A closed inert system also minimizes the possibility of a thermal runaway. After shredding, a drying process is applied to remove all volatile organic compounds (VOCs) such as the electrolyte solvents that can then be recovered via condensation. The resulting dry solid mixture goes then through a series of mechanical sorting unit operations to separate plastics cases, aluminum (Al) and copper (Cu) foils from the black mass (BM), which contains the active cathode and anode materials. The anode material, typically graphite, can be separated from the cathode by froth flotation. After this, the hydrometallurgical process can be applied on the isolated CAM to individually recover the battery metals. The overall recycling process scheme is represented in figure 6, and the material requirements to suit the recycling process are listed in table 3.

Different types of leaching agents can be used to dissolve the metals, and different parameters such as pH, residence time, temperature, solid/liquid ratio or rotational speed can be optimized to selectively leach target elements. After the leaching, the metals can be recovered by different techniques, including selective precipitation, solvent extraction and ion exchange.

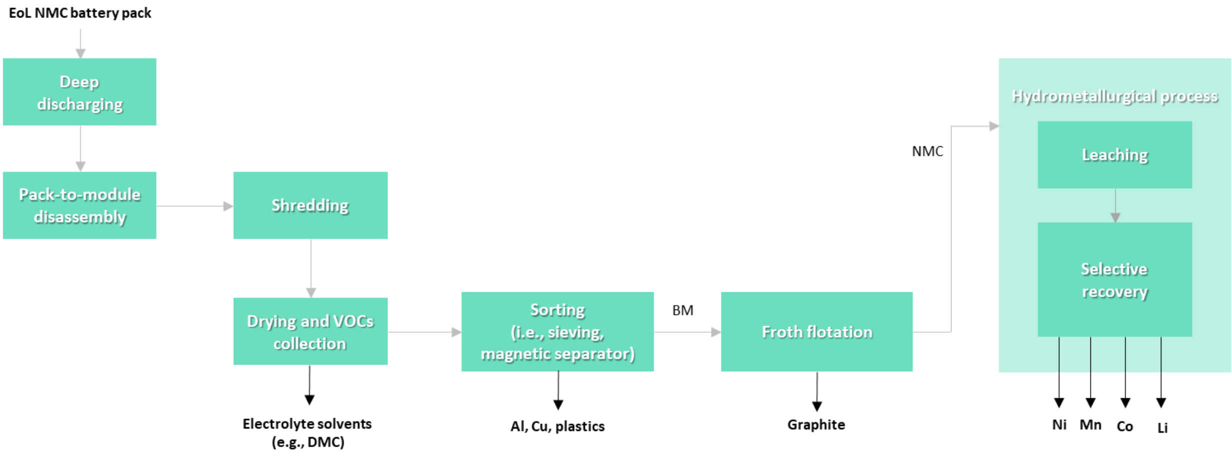

Figure 6 Hydrometallurgical process flow scheme.

Table 3 Requirements for NMC recycling

Process	Process requirement	Entry material requirement
Shredding	Deep discharge (1.5V)	Deep discharging process and state of charge measurements must be applicable on the entry material.
	Pack to module disassembly	Physical damage shouldn't hinder the pack-to-module disassembly.
Drying process	-	-
Mechanical sorting of black mass	Material must be delaminated, and deagglomerated	-
Leaching	-	-

3.2 LFP batteries

Applying direct recycling to the CAM is typically done in two overall steps: (i) a thermal process where a lithiation agent is used to compensate the Li deficiency of the spent $LiFePO_4$, which is then followed by (ii) a high-temperature process to repair its molecular structure.

In order to achieve optimal direct recycling, batteries need to undergo disassembly down to the electrode level. This entails a sequential breakdown, starting from pack-to-module, then module-to-cell, and finally cell-to-electrode disassembly. Following this electrode-level disassembly, the exposed cell comprises various components, including the current collector (such as Al), the CAM, traces of electrolyte solvents (such as DMC, DEC), conductive salt (like $LiPF_6$), binder materials (such as PVDF), and carbon additives.

The purification process of the CAM includes: i) delamination from the current collector, ii) removal of VOCs (electrolyte solvents) and iii) removal of electrolyte salt and binder. The delamination process can be done by dissolving the Al in NaOH. For the removal of the VOCs, the delaminated cathode material can be dried under vacuum, and the VOCs can be recovered via condensation. The electrolyte salt and binder (which are mixed with the CAM in solid form) can be removed by washing the CAM with appropriate solvents, or by applying high temperatures to thermally decompose them. After this, the purified CAM can be repaired. The cathode healing process itself is done in two steps. In the first step, the lithium deficiency of the spent cathode is compensated, for which a lithium source (e.g., LiOH) is necessary. The process can be done via thermal, hydrothermal or electrochemical methods (19). After the relithiation is done, it is necessary to anneal the relithiated CAM to recover the crystalline microstructure. Figure 7 shows the overall recycling process from the discharge step until the CAM healing process.

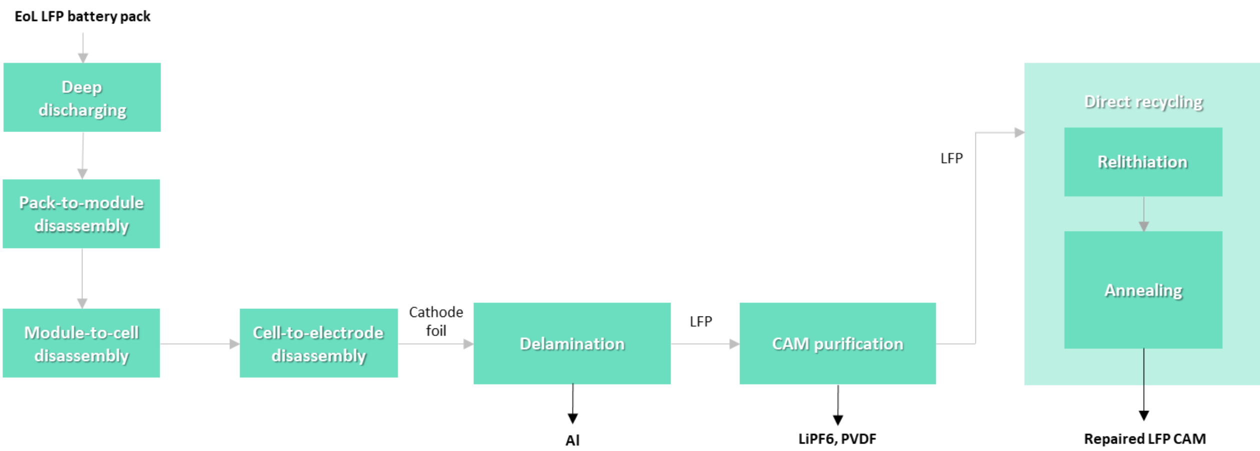

Figure 7. Direct recycling process flow scheme

In the table below (table 4), the requirements for the entry material to suit the proposed recycling process are identified:

Table 4 Requirements for LFP direct recycling

Process	Process requirement	Entry material requirement
CAM isolation and purification	Deep discharging (1.5V)	Deep discharging process and state of charge measurements must be applicable on the entry material.
	Disassembly to electrode level	Physical damage at pack, module or cell level shouldn't hinder the pack-to-module and module-to-cell-to-electrode disassembly.
CAM repairing process	Purified CAM free from: -fluorine -Al - Electrolyte	-

As previously discussed, some studies suggest the possibility of repairing the CAM directly from the black mass material derived from the mechanical shredding recycling process. As it can be observed in figure 8, this approach includes the same steps as the ones previously described in the recycling process for the NMC. First, the battery packs must be deep discharged to avoid thermal runaway during the shredding, and they must be dismantled to the module level. After shredding, VOCs can be recovered during the drying process, and the BM is sorted from the rest of the shredded materials via sieving. After the froth flotation, where graphite is separated from the cathode material, the CAM must be purified to remove the electrolyte salt (e.g., $LiPF_6$) and the binder (e.g., PVDF) before applying the direct recycling process. The input material requirements are listed in table 5.

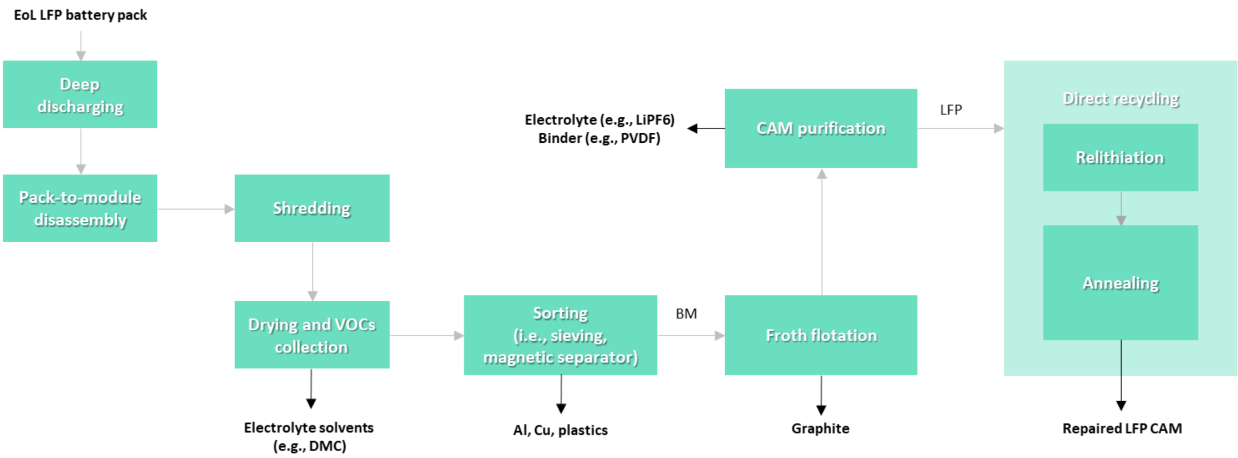

Figure 8. Direct recycling from black mass process flow scheme

Table 5. Requirements for LFP direct recycling from black mass

Process	Process requirement	Entry material requirement
Shredding	Deep discharge (1.5V)	Deep discharging process and state of charge measurements must be applicable on the entry material.
	Pack to module disassembly	physical damage shouldn't hinder the pack-to-module disassembly.
Drying process	-	-
Mechanical sorting of black mass	Material must be delaminated, and deagglomerated	-
Direct recycling	Purified CAM free from: -fluorine -Al - Electrolyte	-

The above recycling approaches show common requirements for the entry material to be suitable for the process. These requirements include deep discharge, SOC measurement, pack-to-module or pack-to-module/module-to-cell-to-electrode disassembly, and no physical damage. In table 6, the general requirements for an ideal recycling are listed along with the respective KPIs.

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Table 6 General requirements and KPIs for ideal recycling.

Requirement - KPIs	Reason
Deep discharge (1.5V) and SOC measurement	To avoid a potential thermal runaway from taking place.
Full battery pack disassembly to module level prior mechanical recycling process.	Battery packs are mostly made of aluminum which will unnecessarily contaminate the battery components after shredding.
>90% electrolyte solvent recovery (i.e., volatile organic compounds (VOCs)) below 90 °C.	To avoid cross contamination within battery components and exposure to environment, most of the VOCs are expected to be collected right after or during shredding process. To avoid electrolyte salt decomposition and thus formation of HF, it is recommended to work at low mild temperatures.
No physical damage	To avoid safety issues related to electrolyte leaking and short circuit, during the discharging and disassembly process as well as during the recycling process itself.
Disassembly at electrode level when applying direct recycling	To successfully achieve direct recycling of the CAM, this needs to be carefully isolated from other components to avoid its contamination which can hinder the recycling effectiveness.
Throrough physicochemical characterization of recycling output material after mechanical processing.	Textural, compositional, elementary, and morphological analyses, among others, are key to understand and enhance the mechanical recycling process itself. It is also a critical step for quality control and for proposing new perspectives for better recycling. Understanding the recycled material will allow to propose suitable battery designs and components in line with the Battery 2030+ Roadmap themes (table 1).

4. Conclusions

The requirements for an ideal recycling have been established, taking into account specific cell chemistries (such as NMC and LFP) and the most efficient recycling process methodologies (such as hydrometallurgical processes or/and direct recycling). The identified requirements include: i) deep discharge, ii) SOC measurement, iii) pack-to-module or pack-to-module/module-to-cell-to-electrode disassembly, and iv) no physical damage. Furthermore, a set of Key Performance Indicators (KPIs) has been established based on the chemistry and battery component considerations. The outcomes presented in this deliverable will serve as a valuable guide for the complex tasks involved in designing and developing batteries that are not only suitable for repurposing but also for efficient recycling.

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