Deliverable D2.4

Report on pre- and post-recycling materials characterization

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Feasible Recovery of critical raw materials through a new circular Ecosystem for a Li-Ion Battery cross-value chain in Europe

WP2 - End-of-life LIBs Collection and Characterisation, Digital Tools and Battery Passports deployment

D2.4 – Report on pre- and post- recycling materials characterization

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Versions

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List of Abbreviations

ACRONYMS	DESCRIPTION
EOL	End-of-Life
SOC	State of Charge
SOH	State of Health
LIB	Li-ion Battery
EHF	Electrohydraulic Fragmentation
XRD	X-Ray Diffraction analysis
AA	Atomic Absorption
SEM	Scanning Electron Microscopy
EDX	Energy Dispersive X-ray
XRF	X-ray Fluorescence
BMA	Black Mass
PRE	Precursors
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
LE	Light Elements
NMC	Nickel-Manganese-Cobalt
LNMO	Lithium Nickel Manganese Oxide
EIS	Electrochemical Impedance Spectroscopy

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1. Introduction

1.1 Context of the deliverable

The deliverable D2.4 aggregates and expands the findings from a series of material characterizations conducted before and after recycling processes for lithium-ion battery (LIB) materials. As part of the FREE4LIB project, the primary objective is to evaluate the chemical, structural, and morphological attributes of critical components, focusing on cathode and black mass materials. These evaluations aim to optimize recycling strategies, enhance material recovery efficiency, and support the development of a circular economy for LIBs.

This deliverable collects the results of the work performed in ST2.2.2 on coordination of data management, together with reports of experimental measurements performed in WP3 and WP4. These results include the work performed for collection and organization of many material characterizations performed on EOL and recycled materials, as well as the ones gathered along the development of the recycling processes studied within the FREE4LIB project. Materials techniques used for material analysis includes X-ray Photoelectron Spectroscopy (XPS), X-Ray Fluorescence (XRF), Atomic Absorption (AA), ICP-OES, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), particle size distribution, powder flowability and many others.

Some of the tests discussed in D2.4 have already been presented in previous deliverables of WP3. The aim of this deliverable is to expand and relate these reports by publishing extensive data of the experimental characterizations and making an explicit connection between materials resulting from different processes applied in sequence. These additional data allow for comparisons between material sources and process outcomes, as well as analysis of materials at intermediate steps. This possibility provides critical information to evaluate the efficacy of the recycling process with different input compositions, identify possible shortcomings and improvements in the development of these processes.

1.2 Connections to other tasks

The content of this deliverable is strongly connected to several part of the project, since it gathers contributions from data management in WP2 and experimental activities in WP3 and WP4. Specifically, the data gathering and management on materials and experimental characterizations has been

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performed in task T2.2 (ST2.2.2), with a strong connection with task T2.3 (ST2.3.1) for the organization and upload of data in the cloud battery recycling platform. Characterization reports in the context of the development and validation of recycling processes have been elaborated in tasks T3.2, T3.3, T3.4, T3.5, T3.6, T3.7, T3.8, T3.9, while those supporting the scale-up of these technologies have been completed in T4.2, T4.3, T4.4, T4.6, T4.7, T4.9.

The additional information included in this report will be used to interpret the results of the electrochemical tests performed in ST2.2.1 as well as the results coming from development tasks in WP3. The experimental reports will also provide important information for the further development and upscaling of several recycling processes performed in different tasks of WP4.

The content of this deliverable is also supposed to provide input to ST2.3.1 for what concerns the organization of data and upload of the characterization reports in the could platform, as well as T5.4 for the selection of the variables to be included in the Battery Passport Concept. Selected data from D2.4 can also be used as input for the activity on modelling of recycling processes within ST2.3.2.

1.3 Structure of the report

The Introduction section provides a context for this deliverable, explaining its focus and the main objectives, and outlines the interdependencies between D2.4 and other project tasks, emphasizing data collection, management, and experimental contributions.

The Data Collection and Management section shows some details on the organization and reporting of experimental data within the project consortium.

The Materials Characterizations along the Recycling Processes section presents detailed analyses of end-of-life (EOL) materials and recycled components. Characterization techniques such as XRF, SEM, and XRD were applied to assess the properties of plastics, cathode foils, and black mass materials.

Black mass production subsection explores processes for generating black mass from LIBs, focusing on pyrometallurgical and electrohydraulic fragmentation methods. Techniques for dismantling and thermal deactivation are discussed alongside the resulting material's composition. Extraction of metals (CSIC) subsection delves into the hydrometallurgical extraction of critical metals such as lithium, cobalt, and nickel from black mass.

From precursors to electrodes subsection examines the transformation of recycled precursors into cathode powders and ceramic pigments. It discusses

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synthesis techniques, including Flame Spray Pyrolysis, and highlights the challenges of composition variability. The next sub section (Direct cathode recycling) focuses on the direct recycling of cathode materials, emphasizing re-lithiation processes and structural integrity analyses. The Post recycling subsection provides a detailed evaluation of the electrochemical properties of recycled materials. Techniques such as cyclic voltammetry and impedance spectroscopy are used to assess cycling stability and charge transfer.

Finally, the section Conclusions synthesizes the key findings from D2.4, highlighting the effectiveness of recycling strategies and the quality of recovered materials.

2. Data Collection and Management

2.1 Data Reporting

The FREE4LIB project used a large number of cells for the production of different materials, coming from several vehicles and spanning different formats. To organize the large amount of material produced and keep track of information along the production and transformation of materials, FBK created a Cells and Materials Tracking Database. This database anticipated the structure of the online Recycling Platform and served as a preliminary storage of data until the platform itself was operative.

To organize the data, each cell involved in the project was named with a specific code with the format XXX X Pnn Mnn Cnn, where X are predefined codes and nn are progressive numbers. Different parts of the code identify the name and type of the source vehicle and the position within the pack and the module. Below an example of a cell code:

Vehicle code	Vehicle type	Pack n.	Module n.	Cell n.
HKO	E	P01	M04	C23

VEHICLE TYPE LEGEND	
E	Full Electric Vehicle
P	Plug-In Hybrid Vehicle
H	Hybrid Vehicle
L	Light Means of Transport

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A naming system was also employed for the materials, in the format of a three letters code identifying the material type, followed by a progressive number (XXXnn).

Materials	Code
Cathode foil	CFO
Anode foil	AFO
Cathode powder	CPO
Anode powder	APO
Cathode paste	CPA
Black mass	BMA
Leachate	LEC
Leaching residual	LRE
Metal oxide	MOX
Precursor	PRE
Metal	MET
Graphite	GRA
Polymer	POL
Plastic	PLA
Electrolyte	ELE

To obtain an effective and uniform data reporting across the whole project, a standard structure for experimental reports have been created and shared with all the partners. Each report gathers information on the tested material, such as name, type, source (cells, modules, other materials, etc.) and upstream processes applied on it, details on the characterizations performed, such as test objective, applied techniques and test procedures, and experimental results and outcomes. Given the variability of applied techniques, several data visualization formats have been adopted depending on the report.

In the following chapters, each characterization report is provided as a hyperlink. To ensure clarity for the readers, we maintain a consistent template for these hyperlinks throughout the report. Each report is labelled as "XXXnn-CHT," representing the material name followed by the characterization techniques applied to the materials.

2.2 Data Management

Each partner has been responsible for the continuous update of the shared Cells & Materials Tracking Database and the online Recycling Platform for what concerned the disassembled or produced cells and materials. FBK coordinated the activity by regularly checking the databases and promoting and supporting the resolution of possible inconsistencies or misalignments. The same

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approach has been applied for the completion of experimental reports and their review.

3. Materials Characterizations along the Recycling Processes

3.1 End-of-Life Materials

3.1.1 Plastics (AIMPLAS)

Our partner AIMPLAS conducted an in-depth analysis of recycled thermoplastic materials obtained from dismantled EV battery components. The materials were characterized to evaluate their physical and mechanical properties, ensuring suitability for recycling as secondary raw materials in battery plastic parts or other high value applications. Recycled materials from EV battery packs of the Hyundai Kona model were analysed. Three specific components—expanded panels, frames, and fittings—were processed to recover polypropylene (EPP), polyphenylene ether (PPE), and polyamide (PA66), respectively. The upstream process involved dismantling, sorting, grinding, and extrusion or injection moulding to create test specimens. Standardized tests were conducted, including tensile, flexural, and impact resistance tests, along with heat deflection and hardness evaluations. Results were obtained according to international standards such as UNE-EN ISO 27, UNE-EN ISO 178, and UNE-EN ISO 75. The table below summarizes the key physical and mechanical properties of the tested materials:

Material Code	Material Type	Source	Property	Unit	Value
PLA07 PLA07-Phys	Recycled EPP panels	EV battery pack	Density	kg/m³	66
			Melt flow rate (230°C/2.16 kg)	g/10 min	16.1
PLA06 PLA06-Phys	Recycled PPE from frames	EV battery module	Tensile stress at break	MPa	78.1

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			Tensile strain at break	%	3.1
			Flexural modulus	MPa	4050
			Flexural strength	MPa	118
			Charpy impact resistance Notch @ 23°C	kJ/m²	9.64
			Charpy impact resistance Unnotch @ 23°C	kJ/m²	34.58
			Heat Deflection Temperature 1.8 MPa	°C	122
			Heat Deflection Temperature 0.45 MPa	°C	128
			Rockwell M hardness	--	167
PLA05 PLA05-Phys	Recycled PA66 from fittings	EV battery pack	Tensile stress at break	MPa	77.2
			Tensile strain at break	%	2.2
			Flexural modulus	MPa	5050

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			Flexural strength	MPa	141
			Charpy impact resistance Notch @ 23°C	kJ/m²	6.3
			Charpy impact resistance Unnotch @ 23°C	kJ/m²	32.44
			Heat Deflection Temperature 1.8 MPa	°C	233
			Heat Deflection Temperature 0.45 MPa	°C	243
			Rockwell M hardness	--	177

3.1.2 Foils (CARTIF)

The report from CARTIF provides an in-depth analysis of cathode and anode materials extracted from lithium-ion battery modules. These materials, including cathode foils and anode foils, were manually processed following a deep discharge procedure and subsequently dried in a vacuum oven to eliminate any residual electrolytes. To examine their chemical composition, structural attributes, and surface morphology, a range of advanced analytical techniques were utilized, such as X-ray Fluorescence (XRF), X-ray Powder Diffraction (XRD), Scanning Electron Microscopy (SEM), and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES).

The cathode foils (Material Code: CFO03) featured distinct layers, with black and whitish sides analysed separately. XRF revealed consistent elemental compositions CFO03-XRD-XRF across all sides, dominated by nickel (37.1%-37.4%), manganese (9.7%-9.8%), cobalt (7.4%-7.5%), and oxygen (35%),

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alongside trace elements such as sulphur, phosphorus, and fluorine. Aluminium was present in significant amounts, forming the substrate for the active material layers. XRD confirmed the presence of lithium cobalt manganese nickel oxide (NMC) and aluminium, while corundum was observed in the whitish side.

The cathode and anode foils (Material Code: CFO02) underwent SEM analysis to study CFO02-SEM their surface morphology. The cathode foils displayed a layered structure on both black and whitish sides, while the anode foils featured uniform black layers. Cross-sectional imaging revealed the structural integrity and uniformity of the layers, ensuring their reliability in lithium-ion battery systems.

The next set of Material (Material Code: CPO20) from Hyundai Kona, type E, pack P06, module 01 and cell 19. The modules were discharged by deep discharge and the cells have been manually dismantled. The cathode foils had been previously dried in a vacuum oven to remove the electrolyte. Subsequently, the sides of the cathode were scrapped of the foil to get the cathode powder to analyse. In this module, the cells had a pink paper wrapper and their cathode foils with distinct sides (Side A: whitish, Side B: less whitish) were analysed. XRD confirmed CPO20-XRD-XRF lithium cobalt manganese nickel oxide (NMC622) as the main phase. XRF quantified metal and metal oxide percentages, showing near-identical compositions for both sides.

The foils' (Material Code: CFO07) morphology and structure analysis included CFO07-XRD. SEM analysis showed uniform morphology in the anode foils (Material Code: CFO06). SEM revealed CFO06-SEMclear surface differences, important for recycling. These findings highlight consistent quality, aiding recycling efficiency. The Scraped cathode powder (Material Code: CPO19) analysis by ICP-OES CPO19-ICP-OES showed Ni (23%-24%), Mn (6%), Co (%), and Li (6.8%). Protective Al2O3 coatings were more prominent on the whitish side, providing stability.

The another set of material characterisation (Material Code: CPO17) from Hyundai Kona, type E, pack P05, module 03 and cell 25. In this module, the cells had a yellow paper wrapper. This type of cells, have the cathode foils with different sides: one black side (side A) and other whitish side (side B), one black side (side C) and a blacker one (side D) or both sides black (side E). In this case, all sides were analysed by ICP-OES CPO17-ICP-OES. It revealed the cathode material to be of type NMC622, with average values of Nickel (27.3%), Manganese (9.3%), Cobalt (9.0%), Lithium (6.4%), and traces of Aluminium (1.2%).

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3.2 Black mass production

3.2.1 Pyrometallurgical process (ACCUREC)

The recovery of black mass (BM01) from end-of-life (EOL) lithium-ion batteries (LIBs) through pyrometallurgical methods are handled by our partner ACCUREC. The process begins with the critical pre-treatment of the batteries. First, the batteries are dismantled manually or mechanically to separate casings, current collectors, and separators. Following this, the batteries are subjected to thermal deactivation at low temperatures to eliminate residual electrolytes, which are both flammable and toxic. This step also enhances the safety of subsequent high-temperature processes. Finally, the pre-treated batteries are shredded into fine particles, yielding a homogenous feedstock comprising cathode and anode materials, binders, and any residual impurities.

The pyrometallurgical processing involves smelting the shredded feedstock in a high-temperature furnace. During this phase, organic binders and plastics are combusted, releasing energy that contributes to the heating process. Carbon additives and graphite act as reducing agents, facilitating the separation of metal oxides into individual elements or alloy phases. Heavier metals like nickel (Ni) and cobalt (Co) melt into a separate phase that can be collected and reused in battery manufacturing. Simultaneously, lithium (Li), graphite, and other lighter components remain in the slag, forming the substance known as black mass. This black mass, enriched with valuable materials such as lithium compounds, cobalt, and nickel oxides, serves as a crucial feedstock for further refinement through hydrometallurgical methods. The produced BM01 was characterised by other partner CSIC by using different techniques which will be explained in the coming chapters.

3.2.2 Electrohydraulic fragmentation (Fraunhofer)

Electrohydraulic fragmentation (EHF) is an innovative method for recovering black mass from end-of-life lithium-ion batteries (LIBs). The process is handled by Fraunhofer and it begins with pre-treatment, where LIB cells are discharged, dismantled, and mechanically shredded. This step weakens the structural bonds and exposes internal components, making subsequent fragmentation more efficient. In the fragmentation stage, pre-shredded materials are immersed in a dielectric fluid and subjected to high-voltage electrical pulses. These pulses create shockwaves that travel through the liquid and material, exploiting mechanical differences between layers. This results in selective separation of active materials, such as cathode coatings, from metallic foils. The fragmented material is then sieved, with finer particles forming the black

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mass (BM03 and BM02), primarily comprising cathode materials (e.g., lithium cobalt oxide, graphite). The coarser fractions, including aluminium and copper foils, are separately recovered for recycling. The process is energy-efficient, scalable, and enables high-purity material recovery.

Fraunhofer conducted the EHF treatment and derived the black mass materials (Material code: BMA03-BMA04 & BMA05-BMA14). The below reports (BMA005-BMA014-ICP-OES & BMA005-BMA014-ICP-OES-1) provide the details of material characterisation to assess the chemical composition, quality determination and contamination levels. The key data was gathered using ICP-OES to quantify elemental concentrations.

Sample ID & Observation	Al (wt%)	Co (wt%)	Cu (wt%)	Fe (wt%)	Li (wt%)	Mn (wt%)	Ni (wt%)
2303_F4L_dho_01Aa Lower impurities: active material concentration balanced	0.954	4.30	1.22	0.0191	2.43	4.06	13.4
2303_F4L_dho_01Ab Comparable impurity and active material levels to 01Aa.	0.902	4.29	1.24	0.0148	2.47	4.09	13.3
2302_F4L_dho_01Ba Higher impurities observed compared to shorter processed samples (A).	2.66	6.28	2.75	0.0408	3.37	5.99	17.9
2302_F4L_dho_01Bb Increased active material concentration at expense of higher impurities.	2.23	6.40	2.74	0.0391	3.36	6.12	18.3
2305_F4L_dho_7a Excellent active material concentration: impurity control improved.	1.78	8.30	3.24	0.0394	4.02	7.73	25.5
2305_F4L_dho_7b Highest Ni and Co values; suitable for advanced applications	1.82	9.05	3.37	0.0390	4.28	8.31	27.4

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Based on the above data, samples 2305_F4L_dho_7a and 7b demonstrated the highest concentrations of Ni, Co, and Mn, making them favourable for high-performance cathode applications. The BM produced by Fraunhofer was characterised by CSIC to determine the chemical composition and to characterize the mineralogy and morphology.

3.3 Extraction of metals (CSIC)

The process of extracting valuable metals from spent LIBs involves several key stages. The Pre-Treatment and production process such as Pyrometallurgy and Electrohydraulic fragmentation (EHF) was done by our partners (ACC, FRA). Then the next key crucial step of extraction of metals weas handled by CSIC (BM01 and BM03) and Sakarya university (BM01 and BM03). Black mass undergoes leaching in acidic solutions, extracting metals into solution. Selective precipitation and solvent extraction techniques then isolate individual metals like lithium, cobalt etc.

3.3.1 Leaching and Liquid-liquid extraction

The process revolves around processing black mass (BMA01, BMA02 and BMA03) to extract valuable materials, with the production of several precursors (PRE). These precursors are obtained from different leaching solution and deep eutectic solvents. The detailed process explanation about this work was submitted as Deliverable (D3.3 - Development, testing and validation of recycling solutions for cathode and black mass at lab-scale). Then the precursor obtained from both BM's is characterised by Atomic Absorption (AA) and X-Ray diffraction analysis (XRD). For the black mass (BMA02), the impurities were found high. So single characterisation was done and explained below. In the chemical characterization report, BMA02 (Material code: BMA02) is identified (BMA02-AA) as containing high concentrations of metals crucial for the cathode-active material regeneration. Analytical processes ensure accurate measurement of elemental compositions, providing critical data for downstream recovery. The structural analysis through (BMA02-XRD.pdf) highlights the crystalline phases of metal oxides and other compounds, establishing BMA02 as an efficient feedstock for recycling. The precursor obtained from BMA01 was explained below then followed by BM03. These key results obtained are based on the hydrometallurgical process and combined pyro-hydrometallurgical process.

Material Code	Source	Key Results

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BMA02 BMA02-AA & BMA02-XRD	FRAUNHOFER (HKO E P07 M01-03)	Ni: 16.703%, Mn: 5.056%, Li: 3.222%, Co: 5.452%, Cu: 1.224%, C: 53.4%; Graphite, Li, Ni, Mn oxides.
BMA03 BMA03-AA & BMA03-XRD	FRAUNHOFER (HKO E P07 M01-03)	Ni: 16.579%, Mn: 4.205%, Li: 2.754%, Co: 5.147%, Cu: 3.228%, C: 40.3%; Graphite, Li, Ni, Mn oxides.
PRE01 PRE01-AA & PRE01-XRD	BMA01	Li: 17.29%, F: 0.85%, P: 0.09%; Li_2CO_3 and LiF identified.
PRE06 PRE06-AA & PRE06-XRD	BMA03	Li: 17.44%, F: 0.61%, P: 0.06%; Li_2CO_3 and LiF identified.
PRE02 PRE02-AA & PRE02-XRD	BMA01	Ni: 12.58%, Co: 4.31%, Mn: 3.98%, Al: 3.50%, Li: 1.21%; Graphite, Ni/Co phases.
PRE07 PRE07-AA & PRE07-XRD	BMA03	Ni: 15.94%, Co: 4.99%, Mn: 4.68%, Al: 2.15%, Li: 0.59%; Graphite, Ni/Co phases.
PRE03 PRE03-AA & PRE03-XRD	PRE02_BM01	Nickel, cobalt, manganese oxalate hydrates identified.
PRE08 PRE08-AA & PRE08-XRD	PRE02_BM03	Ni: 14.47%, Co: 5.57%, Mn: 4.43%, Cu: 4.78%, Li: 0.11%.
PRE11 PRE11-AA & PRE11-XRD	PRE02_BM01	Ni: 17.91%, Co: 5.71%, Mn: 4.59%, Cu: 0.03%, Li: 0.11%.
PRE14 PRE14-AA & PRE14-XRD	PRE02_BM03	Ni: 17.44%, Co: 5.48%, Mn: 6.11%, Cu: 0.01%, Li: 0.08%.
PRE04 PRE04-AA & PRE04-XRD	PRE03_BM01	Ni: 0.75%, Co: 13.41%, Mn: 5.06%, Li: 0.01%; Cobalt sulfate hydrate identified.

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PRE09 PRE09-AA & PRE09-XRD	PRE03_BM03	Ni: 0.79%, Co: 17.68%, Mn: 5.99%, Li: 0.01%; Cobalt sulfate hydrate identified.
PRE12 PRE12-AA & PRE12-XRD	PRE03-2_BM01	Ni: 1.46%, Co: 20.38%, Mn: 9.58%, Cu: 0.99%, Li: <0.01%; Cobalt sulfate hydrate identified.
PRE15 PRE15-AA & PRE15-XRD	PRE03-2_BM03	Ni: 0.79%, Co: 17.68%, Mn: 5.98%, Cu: 7.81%, Li: 0.01%; Cobalt sulfate hydrate identified.
PRE05 PRE05-AA & PRE05-XRD	PRE03_BM01	Ni: 20.28%, Co: 0.69%, Mn: 6.54%, Li: 0.36%; Hydrate phases identified.
PRE10 PRE10-AA & PRE10-XRD	PRE03_BM03	Ni: 22.15%, Co: 0.58%, Mn: 4.49%, Li: 0.33%; Nickel and manganese oxalate hydrates identified.
PRE13 RE13-AA & PRE13-XRD	PRE03-2_BM01	Ni: 25.63%, Co: 0.65%, Mn: 4.55%, Cu: 0.01%, Li: 0.14%; Nickel chloride hydrate identified.
PRE16 PRE16-AA & PRE16-XRD	PRE03-2_BM03	Ni: 22.22%, Co: 0.56%, Mn: 5.37%, Cu: 0.006%, Li: 0.10%; Nickel chloride hydrate identified.

3.4 From precursors to electrodes

3.4.1 Cathode powder (Torrecid and Sakarya)

This report aims to evaluate the structural, chemical, and functional properties of various materials. The below table provides consolidated material characterization results for various samples, focusing on cathode powders, ceramic pigments, and their corresponding preparation methods. The techniques applied primarily include XRD, SEM-EDX, and electrochemical performance evaluations. Category recycled refers to cathode powder or ceramic pigments produced with recycled precursors or BM.

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Material Code	Type	Key Observations	Category (EOL, commercial, recycled)
CP001	Cathode Powder LNMO	XRD and SEM-EDAX show standard LNMO characteristics CP001-XRD-SEM-E.Chem	Commercial - LNMO-NEI
CP003	Cathode Powder LNMO	Good crystallinity after two-phase sintering CP003-XRD	Commercial
CP004	Cathode Powder LNMO	Identification vis XRD - LiNi0.5Mn1.5O4 CP004-XRD	Commercial
CP005	Cathode Powder NMC622	Electrochemical performance meets supplier specifications CP005-XRD-E.Chem	Commercial – MSE supplies
CP007	Cathode Powder NMC811	Identified morphology via SEM-EDAX CP007-XRD-SEM-EDX	Commercial - BASF
CP009	Cathode Powder NMC811	Particle size and chemical composition align with expectations CP009-XRD-SEM-EDX	Commercial - NMC811 TOB
CP012	Cathode LNMO using recycled material	High crystallinity: composition analysis shows reduced cobalt contamination CP012-XRD-SEM-EDX	Recycled
CP013	Cathode Powder NMC622	SEM-EDAX reveals deviation in composition CP013-XRD-SEM-EDX	Recycled
CP014	Cathode Powder LNMO	XRD analysis confirms structural integrity CP014-XRD-SEM-EDX	Recycled

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CPO24	Cathode Powder NMC622	Defective cobalt content revealed by SEM-EDAX CPO24-XRD-SEM-EDX	Recycled
CPO25	Cathode Powder NMC622	XRD shows low lithiation levels CPO25-XRD	Recycled
CPO26/27	Ceramic Pigment NiMnFeCo	Feasible for pigment applications with good colorimetric properties CPO26-CPO27-XRD-Calori.Values	
CPO30	NMC622	Good crystallinity; XRD confirms structure CPO30-XRD-SEM-EDX	Commercial
CPO31	LNMO	Al contamination identified during SEM-EDAX analysis CPO31-XRD-SEM-EDX	Commercial
CPO32	NMC622	XRD pattern confirms successful synthesis CPO32-XRD	Recycled
CPO37	LNMO with low cobalt content / CSIC source partner	XRD shows LNMO and secondary NiMnO peaks CPO37-XRD	Recycled
CPO38	LNMO	Similar patterns to CPO37; cobalt content managed CPO38-XRD	Recycled
CPO40	LNMO /CSIC source partner	High crystallinity confirmed by XRD CPO40-XRD	Recycled
CPO42	NMC622 /CSIC source partner	XRD confirms successful synthesis CPO42-XRD	Recycled
CPO43	NMC622 /CSIC source partner	Good lithium intercalation; validated by XRD CPO43-XRD	Recycled

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CP044	NMC622 /CSIC source partner	XRD shows defects in lithium intercalation CP044-XRD	Recycled
CP047	Ceramic Pigment NiMnCoFe	Viable as a pigment with competitive colorimetric values CP047-XRD-CaIr.Val	Recycled
CP028	NMC622 synthesis with CP021 (85% recycled)	Precursors adjusted with NiO, MnO_2, CoO. · Successfully synthesized NMC622 phase CP028-XRD-SEM-EDS	Recycled
CP032	NMC622 synthesis with CP021 (68% recycled)	Precursors adjusted with NiO, MnO_2, CoO, Li_2O. · Formation of the NMC622 phase confirmed CP032-XRD-SEM-EDS	Recycled

The next set of table below summarizes the critical details of the characterization results for cathode materials (NMC622) sourced from CARTIF. The materials were analysed for structural and morphological properties using X-ray diffraction (XRD) by IREC.

Material Code	Type	Key Observation
M1 M1-XRD	Cathode material, NMC622	XRD pattern coincides with NMC622 standard pattern (Ref: 01-084-9845, ICSD Code: 291469).
M2 M2-XRD	Cathode material, NMC622	XRD pattern coincides with NMC622 standard pattern (Ref: 01-084-9845, ICSD Code: 291469).
M3 M3-XRD	Cathode material, NMC622	XRD pattern coincides with NMC622 standard pattern (Ref: 01-084-9845, ICSD Code: 291469).

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The XRD analysis confirms the structural integrity of all three samples (M1, M2, M3) by matching their diffraction patterns with the NMC622 reference standard. Although morphological details are not explicitly included in the outcomes, the consistency in XRD results across samples suggests uniform phase composition.

3.4.2 Cathode powder (IREC and Lurederra)

This report consolidates the material characterization results for Li-NMC811 and NMC622 active nano powders synthesized using Flame Spray Pyrolysis Technology. Physicochemical properties, including surface area, particle size, and structural analysis, were evaluated using BET (Lurederra) and XRD (IREC) techniques.

Material Code	Type	Surface Area (m^2/g)	Particle Size	Key Observations
CPO33	Li-NMC811 cathode nano powder	53.8	25.13 nm	Dark brown powder with 1% mass loss; no impurities detected CPO33-BET
CPO44A	Li-NMC811 synthesized under low temperature	1.19	1.13 µm	XRD confirms alignment with NMC811 standards; black powder, no impurities detected CPO44A-BET-XRD
CPO48	Li-NMC811 (optimized version of CPO33)	0.81	1.68 µm	XRD confirms structural similarity to NMC811; dark brown powder CPO48-BET-XRD
CPO49	Li-NMC811 (further optimized CPO33B)	1.24	1.09 µm	XRD pattern matches NMC811; optimized structural and morphological properties CPO49-BET-XRD
CPO11	NMC622 cathode nano powder	36.5	31.7 nm	Black powder with 4.5% mass loss; no impurities detected CPO11-BET

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3.5 Direct cathode recycling

3.5.1 Characterization of cathode foils and paste (CARTIF)

The report from CARTIF focuses on the detailed characterization of cathode and anode materials sourced from lithium-ion battery modules. The materials tested included cathode paste which were processed manually after a deep discharge procedure and dried in a vacuum oven to remove residual electrolyte. Various analytical techniques, such as X-ray Fluorescence (XRF), X-ray Powder Diffraction (XRD), Scanning Electron Microscopy (SEM), and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), were employed to determine the chemical composition, structural properties, and surface morphology of the materials.

The cathode paste (Material Codes: CPO16 and CPO15), obtained by scraping the layers CPO16-XRD-XRF of cathode foils, was analysed to further understand the composition of the active material. In this module, the cells had a green paper wrapper. This type of cells, have the cathode foils with different sides: one side black (side A) and other side whitish (side B), and one side whitish (side C) and other side less whitish (side D). XRF revealed that the black layers contained a high concentration of nickel oxide (up to 50.6%), manganese oxide (13%-14.1%), and cobalt oxide (9.4%-10.4%). Aluminium oxide was more prevalent in the whitish layers due to its role in protective coatings. ICP-OES analysis showed that CPO15-ICP-OES the black layers consisted of an NMC622 composition, with nickel (35.3%), manganese (9.9%), cobalt (6.2%), and lithium (6.5%) being the major components.

SEM imaging CFO04-SEM revealed distinct surface textures between black, whitish, and mixed sides of the cathode (Material Code: CFO04), while maintaining overall structural integrity. Elemental analysis (Material Code: CPO18) using XRF CPO18-XRD-XRF confirmed elevated levels of Nickel (~47.3%), Manganese (~12.2%), and Cobalt (~15.0%), along with protective aluminium oxide (Al2O3) layers on the whitish side (~6.14%). XRD analysis revealed that all sides of the cathode foils shared a uniform phase: lithium cobalt manganese nickel oxide. Structural analysis (Material Code: CFO05) through XRD showed consistent CFO05-XRD crystalline phases of lithium cobalt manganese nickel oxide across all sides, with no significant differences observed.

The next set of samples included cathode powders from HKO modules (Material Code: CPO39) and VMU modules (Material Code: CPO48). This material

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(Material Code: CP039) was manually dismantled and underwent electrochemical relithiation. ICP-OES CP039-ICP-SEM analysis indicated a composition of Nickel (36.69%), Manganese (10.57%), Cobalt (8.01%), and Lithium (6.31%). The VMU sample (Material Code: CP048) was also manually dismantled and vacuum dried. ICP-OES CP048-ICP-OES analysis showed Nickel (18.658%), Manganese (17.097%), Cobalt (18.673%), and Lithium (7.160%).

The final set of samples involves the characterization of cathode and precursor powders derived from end-of-life lithium-ion batteries through hydrothermal re-lithiation and ultrasonication delamination processes. The materials analysed include cathode powder (CP049) and re-lithiated precursors (PRE17), both sourced from disassembled cells of HKO E, pack P06, module 02, and cell 11. The cathode powder (CP049-ICP-OES) was obtained via manual dismantling and ultrasonication delamination after deep discharge of the battery modules. Vacuum drying removed residual electrolytes, and inductively coupled plasma optical emission spectroscopy (ICP-OES) was employed to determine the chemical composition. The analysis revealed the composition as NMC622, with 36.93% nickel, 10.60% manganese, 8.38% cobalt, and 6.21% lithium, establishing its suitability as a cathode active material. The re-lithiated precursor (PRE17-ICP-OES) was synthesized through a hydrothermal re-lithiation process using cathode powder as feedstock. ICP-OES analysis under three different operating conditions showed varying compositions of nickel (35.75–39.16%), manganese (7.89–11.23%), cobalt (6.27–8.81%), and lithium (4.83–7.82%).

3.5.2 Characterization of cathode retails (EURECAT)

The cathode materials of lithium-ion batteries (LIBs) were characterized to assess their suitability for recycling and reuse. The two analysed materials originated from vehicle code VQ3, vehicle type H, Pack P01, Module M01, and cells 1 to 12 and vehicle code HKO, vehicle type E, Pack P01, Module M04, and cells 1 to 15 with modules and cells manually dismantled and prepared for testing. The goal was to determine the chemical composition of the cathodic material, particularly focusing on nickel-manganese-cobalt (NMC) chemistries, through advanced characterization techniques including Aqua Regia digestion with ICP-MS, XRF analysis and SEM analysis.

On conducting ICP-MS characterisation, the large cathodes CF008-ICP-MS primarily exhibited an NMC 6:3:2 composition with high nickel content (257 mg/g) alongside manganese (124 mg/g), cobalt (91 mg/g), aluminium (102 mg/g), and lithium (60 mg/g). The smaller cathode displayed an NMC 1:1:1 composition, with lower nickel (134 mg/g) and lithium (52 mg/g)

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concentrations but higher cobalt (126 mg/g) and aluminium (176 mg/g). The reddish coloration in some cathodes was attributed to copper contamination.

Similarly, for XFR analysis reinforced the findings from ICP-MS. It showed CFO08-XRF normalized percentages of key elements for reddish, large black, and small black cathodes. Nickel remained the dominant element, with percentages of 28.8% in reddish cathodes, 29.3% in large cathodes, and 15.7% in small cathodes. Manganese, cobalt, and light elements (LE) were also prominent across all samples, while reddish cathodes showed elevated copper levels (2.8%), affirming the ICP-MS results. The compositional differences between small and large cathodes further validated the distinct NMC chemistries.

The another set of characterization revealed that the cathode material (vehicle code HKO, vehicle type E, Pack P01, Module M04, and cells 1 to 15) adheres to a 622 NMC (nickel-manganese-cobalt) chemistry, making it suitable for direct recycling. SEM-EDX analysis CFO01-SEM-EDX showed an intact, complex surface morphology typical of high-performance cathodes, with significant levels of nickel (Ni), cobalt (Co), manganese (Mn), and lithium (Li), along with trace zirconium (Zr) particles that may enhance structural stability.

XRF analysis CFO01-XRF & CFO01-XRF-1 confirmed the primary elemental composition, with nickel (29.17–34.91%), manganese (8.76–10.25%), and cobalt (7.91–9.51%), alongside secondary fractions of aluminium (5.92–12.49%) and minor amounts of sulphur, phosphorus, and zirconium.

ICP-MS CFO01-ICP-MS provided precise quantitative data, confirming lithium (5.9%), nickel (35%), manganese (11.1%), cobalt (11.5%), and aluminium (6.4%) in the sample, reinforcing the NMC 622 composition. Collectively, these results highlight that the material is chemically stable, retains its functional properties, and requires minimal processing for reuse, supporting its suitability for direct cathode recycling.

3.6 Post recycling

The following report provided by Sakarya University consolidates the electrochemical post recycling characterization data for different materials synthesized or treated under specific conditions by partners using recycled precursors. The key elements analysed include charge/discharge cycling, cyclic voltammetry, and electrochemical impedance spectroscopy (EIS).

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Material Code	Material Type	Source Partner	Process
CPO30	NMC622 (solid-state synthesis)	Torrrecid	Mixture of oxides/sulfates CPO30-E.Chem
CPO32	NMC622 (surface coprecipitation)	Torrrecid	Precursors as sulfates CPO32-E.Chem
CPO38	NMC622 (PR03 with oxide adjustments)	Torrrecid	75% recycled PR03 CPO38-E.Chem
CPO25	NMC622 (CP021 adjusted)	Torrrecid	95% recycling CP021 CPO25-E.Chem
CPO28	NMC622 (CP021 adjusted)	Torrrecid	85% recycling CP021 CPO28-E.Chem
BMA03-1	Graphite (Fraunhofer BM) – Anode Powder	CSIC	Hydrometallurgical treatment BMA03-E.Chem
BMA01-1	Graphite (Accurec BM) – Anode Powder	CSIC	Hydrometallurgical treatment BMA01-E.Chem
BMA01-2	Graphite (Accurec BM) – Anode Powder	CSIC	Combined pyro-hydro treatment BMA01-E.Chem-2
BMA03-2	Graphite (Fraunhofer BM) – Anode Powder	CSIC	Combined pyro-hydro treatment BMA03-E.Chem-2
CPO09	NMC811	Torrrecid	CPO09-E.Chem
CPO11	NMC811	Lurederra	Binder variations CPO11-E.Chem
CPO24	NMC622 (recycled black mass)	Torrrecid	Relithiation with Li2O CPO24-E.Chem
GRA01	Graphite - BM from ACCUREC	CSIC	Hydrometallurgical treatment (H_2SO_4 + O_3) GRA01-XRD-SEM-EDS
GRA02	Graphite - BM from ACCUREC	CSIC	Combined pyro-hydrometallurgical

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			treatment GRA02-XRD-SEM-EDS
GRA03	Graphite - BM from FRAUNHOFER	CSIC	Hydrometallurgical treatment (H_2SO_4 + O_3) GRA03-XRD-SEM-EDS
GRA04	Graphite - BM from FRAUNHOFER	CSIC	Combined pyro-hydrometallurgical treatment GRA04-XRD-SEM-EDS

The electrode preparation for the tested materials followed a standardized methodology to ensure consistency and reliability in the results. For NMC-based materials, polyvinylidene fluoride (PVDF) was used as the binder at a concentration of 10 wt%, while graphite-based materials utilized carboxymethyl cellulose (CMC) at 5 wt%. Super P carbon black was added as a conductive agent at 10 wt% for NMC electrodes and 5 wt% for graphite electrodes, with the active material accounting for 80 wt% and 90 wt%, respectively.

Electrochemical characterization involved three primary tests. Charge/discharge cycling was conducted over a voltage range of 4.2-2.7 V for NMC materials and 1.5-0.1 V for graphite, with current rates of C/20 and C/10. Cyclic voltammetry (CV) was performed under similar voltage ranges with a scan rate of 0.1 mV/s to analyse the redox reactions and reversibility. Electrochemical impedance spectroscopy (EIS) was carried out across a frequency range of 0.01 Hz to 100 kHz, providing insights into charge transfer resistance and interfacial characteristics.

The results revealed that materials such as CPO30 and CPO38 demonstrated excellent cycling stability and specific capacity retention, making them strong candidates for high-energy cathode applications. The graphite-based materials BM01-2 and BM03-2 exhibited significant improvements in charge transfer and reversibility due to optimized processing, particularly through combined pyro-hydrometallurgical treatments. Nyquist plots showed reduced impedance in materials like CPO24, suggesting effective relithiation and superior electrochemical performance.

Material-specific observations highlighted the potential of CPO30 and CPO38 for further optimization and commercial scaling. Graphite-based anodes, particularly BM01-2 and BM03-2, emerged as promising candidates for lithium-ion battery applications due to their enhanced processing and stable

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cycling. The relithiated NMC material CP024 demonstrated an innovative approach to integrating lithium, paving the way for sustainable and high-performance battery designs.

Another partner IREC also conducted some characterisation tests on recycled materials. The consolidated report presents a comprehensive evaluation of the structural, morphological, and electrochemical properties of various Lithium Manganese Nickel Oxide (LMNO) materials and related compositions.

Material Code & Partners	**Source Partner**	**SEM/XRD Observations**	**Electrochemical Performance**
CP031 TORRECID CP031-XRD-SEM-E.Chem	Commercial	XRD matched LMNO standard peaks; uniform morphology	Capacity: C/10 = 120.3 mAh/g, C/2 = 38.9 mAh/g; low retention.
CP035 IREC CP035-SEM-XRD-E.Chem	Commercial TOPSE	XRD consistent with LMNO standard; good particle uniformity	Capacity: C/10 = 140.9 mAh/g, C/2 = 110.4 mAh/g; stable cycling.
CP040, CP041 TORRECID CP040-XRD-SEM-E.Chem & CP041-XRD-SEM-E.Chem	Recycled	XRD confirmed phase match, but high impedance observed	Impedance > 10 kΩ; poor capacity and cyclability.
CP037 IREC CP037-SEM-XRD-E.Chem	TORRECID	XRD aligned with LMNO; reduced particle aggregation	Avg. capacity: 35.14 mAh/g after 100 cycles (initial: 66.71 mAh/g).
CP006 IREC CP006-E.Chem	TORRECID	XRD indicated reduced crystallinity vs. commercial NMC622	Half the capacity of NMC622; stable but low C-rate performance.
CP012, CP014 TORRECID CP012-XRD-SEM-E.Chem &	Recycled	XRD matched LMNO but SEM showed poor morphology (CP012)	CP012: No electrochemical activity; CP014: Capacity = 108.2 mAh/g (C/10).

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CPO14-XRD-SEM-E.Chem			

4. Conclusions

The systematic characterization of LIB materials validates the effectiveness of the proposed recycling processes, underscoring their role in sustainable battery production. The results of D2.4 contribute significantly to the advancement of recycling technologies, promoting economic and environmental sustainability within the LIB value chain. These reports establish the necessity for LIB recycling to mitigate raw material shortages and environmental impact. Analytical techniques were selected to maximize insights into material properties. Cathode foils and black mass materials derived from end-of-life LIBs were characterized to understand their chemical composition and structural properties. Analysis revealed significant retention of high-value elements like Ni, Mn, Co, and Li, which are essential for battery functionality. This highlights the feasibility of recovering these materials through controlled dismantling and processing.

4.1 Black mass production

This report highlights the effectiveness of processing black mass materials for high-value applications, emphasizing the importance of balancing impurity control with active material retention. By refining processing methods and adopting robust quality control measures, black mass can be a valuable feedstock for sustainable material production in the energy and electronics sectors.

4.2 Extraction of metals

The process described for extracting valuable metals from spent lithium-ion batteries (LIBs) showcases an efficient multi-stage recycling method. The approach ensures the recovery of critical materials such as lithium, cobalt, nickel, manganese, and graphite through chemical processing and advanced techniques. For both BM01 and BM03 feedstocks, the outlined steps—including leaching, selective precipitation, and crystallization—highlight the systematic recovery of valuable precursors like lithium carbonate, graphite, cobalt sulphate, and mixed metal oxalates. These precursors are key inputs for manufacturing new battery materials.

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The recovery rates and purity of the products emphasize the effectiveness of the method. High lithium and cobalt recovery are notable, alongside the efficient separation of nickel and manganese. By employing tailored processes for each precursor, the methodology reduces material wastage, contributes to sustainability, and minimizes reliance on virgin resources.

4.3 From precursors to electrodes

The findings in this report underscore the potential of recycled materials in producing high-quality cathode powders and ceramic pigments. While the use of recycled precursors presents certain challenges, such as composition variability and contamination risks, the overall feasibility and sustainability benefits are significant. By refining synthesis techniques and implementing robust quality controls, recycled materials can play a pivotal role in advancing sustainable material development.

The findings underline the potential of Flame Spray Pyrolysis Technology in producing high-quality cathode nano powders with tuneable properties. By tailoring synthesis conditions, it is possible to balance surface area and structural integrity, enabling these materials to meet specific application needs in energy storage and advanced material systems.

4.4 Direct cathode recycling

Across all samples, the primary cathode materials displayed high Nickel content with notable variations in Manganese and Cobalt levels. These variations reflect differences in material preparation and sources. Structural analyses consistently identified lithium cobalt manganese nickel oxide phases, confirming material homogeneity across cathode foils. The relithiated sample (CP039) exhibited favourable chemical composition and morphology for potential reuse.

The comprehensive characterization of the cathode retails confirms their compatibility with direct cathode recycling. The integrity of the NMC 622 composition, alongside the observed doping with zirconium, positions the material as a candidate for direct reuse in battery manufacturing. By minimizing waste and reducing chemical processing, direct recycling offers a cost-effective and environmentally friendly pathway for reintroducing spent cathode materials into the battery supply chain. These findings contribute to advancing circular economy principles in energy storage technologies.

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4.5 Post recycling

The characterization reveals significant insights into the behaviour of LMNO materials under varying preparation and testing conditions. While materials like CPO35 show promise for commercial application, others highlight critical challenges in using recycled precursors.

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