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LIFE DRONE
Overview, Objectives and results

LIFE19 ENV/IT/000520
Direct pROduction of New Electrode materials from battery recycling

PROJECT LOCATION: Italy

BUDGET INFO: **Total amount:** 1,720,205 €

% EC Co-funding: 55%

DURATION: Start: 01/09/2020-**End:** 30/06/2024

Amendment approved

PROJECT'S IMPLEMENTORS:

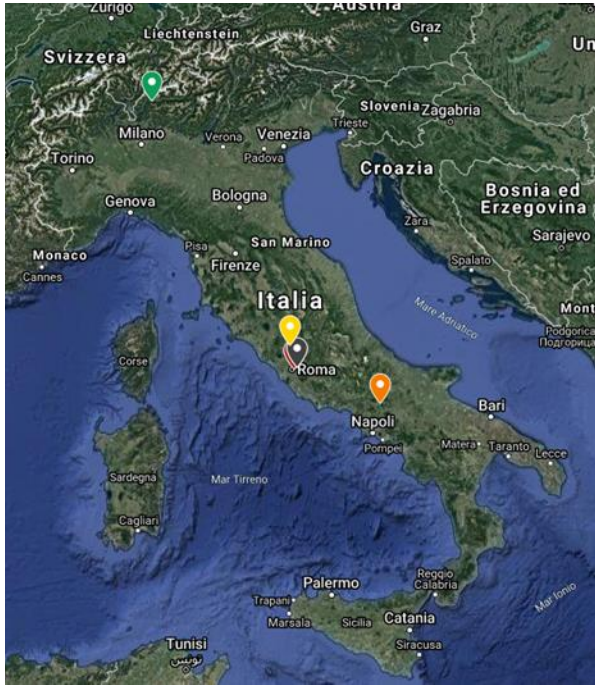
Coordinating Beneficiary:
Technosind srl

Associated Beneficiaries:
ECO RECYCLING srl

FAAM RESEARCH CENTRE SRL (FRC) (Amendment approved)

S.EVal.srl

Sapienza University Department of Chemistry



- Sapienza Università di Roma
- SEVal
- Via di Vannina, 88 - Eco Recycling office
- Technosind Srl
- San Potito Sannitico
- Civita Castellana - Eco Recycling ind. site

OBJECTIVES & SCOPE

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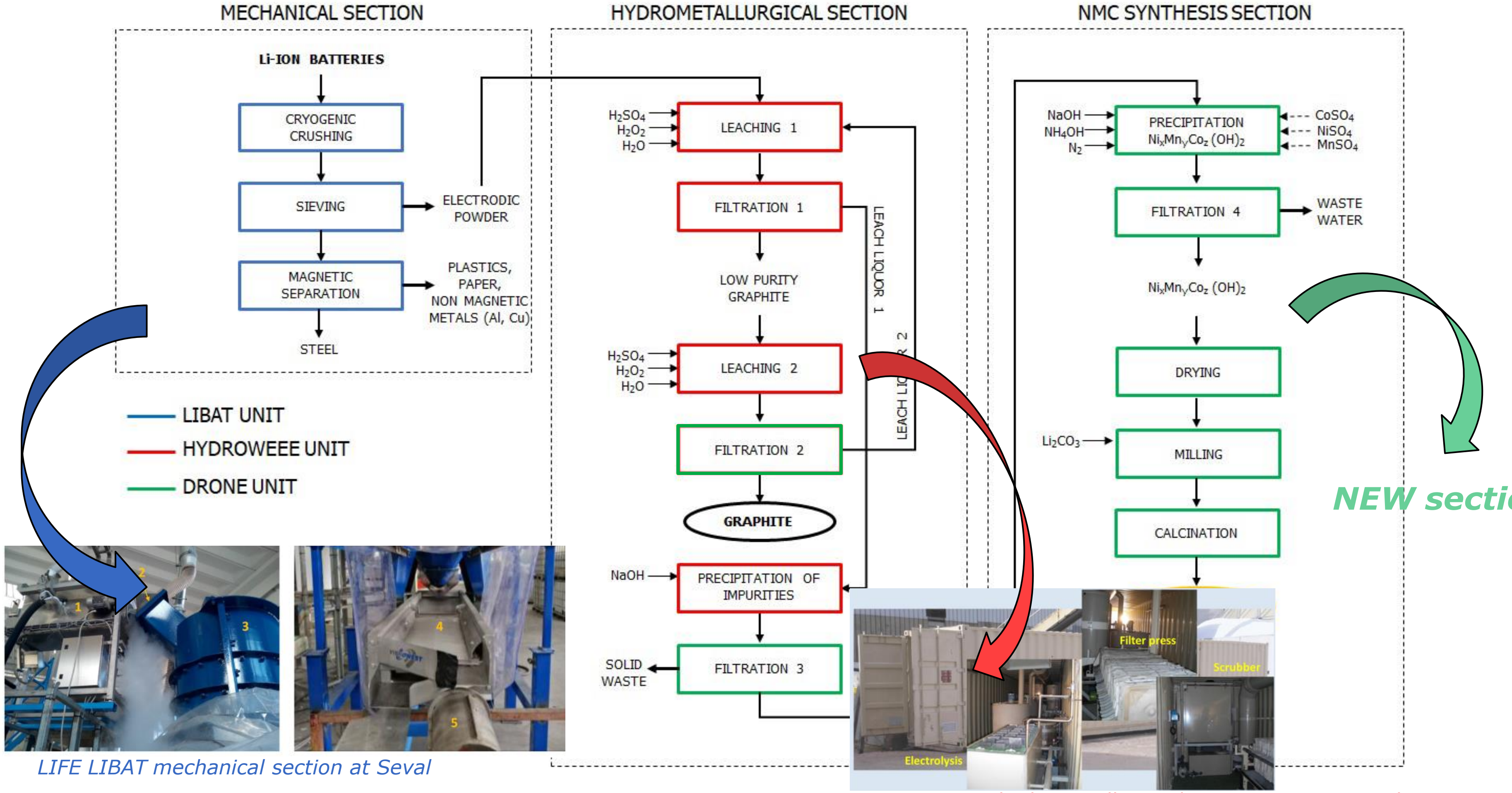
The main project objectives:

- **Design and construction of a mobile plant** to perform the crystallization of the mixed hydroxide and the synthesis of the NMC oxide. Plant design and construction will be performed to ensure a processing capacity of 30 kg/batch and a material recovery yield not lower than 69%.
- **Process demonstration** by treatment of 3 tons of Li-ion batteries (about 1350 kg of electrodic powder) producing 660 kg of NMC oxide
- **Validation** produced of cells for the evaluation of the electrochemical performance of the recovered/synthesized material
- **Life cycle assessment** of the proposed recycling route
- **Elaboration of a replicability plan** evaluating the implementation in a different EU member state
- **Elaboration of a business plan** to drive the large-scale industrial application of the proposed process

Drone process block flow diagram



NEW section

LIFE LIBAT mechanical section at Seval

HYDROWEEE hydrometallurgical section at Eco Recycling

Operability and plant potentiality

- Batch mode running of the prototype.
- The mechanical section is designed to treat maximum 200 kg of batteries per hour.
- The hydrometallurgical section is designed to treat 30 kg of electrodic powder per batch
- The synthesis sections was designed with particular attention to the **Safety** for operators and **environment** due to the presence of hazardous powders.

Batteries sorting

STARTING POINT	1° UPGRADE WITHIN LIFE DRONE	2° UPGRADE WITHIN LIFE DRONE
• The batteries delivered to SEVAL are first loaded into a hopper where the larger battery packs are separated manually. • The batteries of smaller dimensions, most of them quantitatively, are sent to a second hopper from which there is a separation by screening and then a manual sorting.	• SEVAL has trained its staff in order to carry out a further sorting among the LIBs so as to identify and divide the following typologies: LG Li-ion, HG Li-ion and NiMH batteries.	• SEVAL decided to create a local database capable of collecting all the useful data on each selected cell, to be able to recognize the batteries more easily subsequently. An accurate study was carried out for each. • So long as some batteries remained inadequately classified, so to implement and complete the database it has chosen to purchase an X-ray spectrometer (XRF).
Critical aspect All types of LIBs were stored together regardless of both form and chemistry.	Critical aspect Inaccurate distribution according to the concentration of cobalt they presented. Precisely because of this criticality, sorting operations were long and complicated.	Advantage The sorting process has been improved fivefold thanks to the database and to a new RX-spectrometer.

❖ From the sorting activity carried out by SEVAL on the batteries collected, the following fractions of batteries were divided:

TYPE	BATTERIES (kg)	%wt
HG Li-ion	2505	71%
LG Li-ion	670	19%
NiMH	378	11%

Mechanical section

Overview of the mechanical section

A cryogenic pre-treatment is performed to stabilize the batteries and allow them to be ground safely in a mill.

Through dimensional and magnetic separation systems the material is divided into three fractions: steel, non magnetic/paper/plastic and electrodic powder.

The separated fractions are conveyed into three different neutralization reactors

The neutralized electrodic powder is fed to the leaching section

Mechanical section – main units

Pretreatment Unit

Mechanical section and neutralization reactors

Potentiality of mechanical section: 200 kg/day of treated batteries

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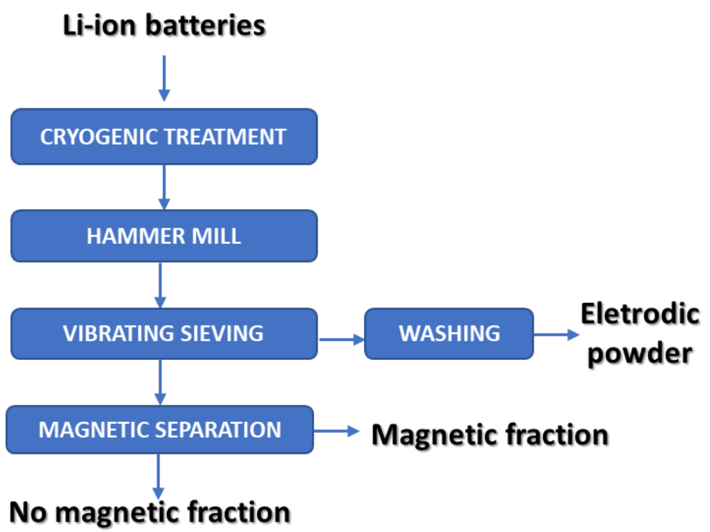
Overview Mechanical section

Optimization of the operating conditions of the pre-treatment section

- The mechanical section is composed of the following equipment: cryogenic unit, grinder, magnetic separator, vibrating sieve and washing system.
- The fractions coming out of the mechanical treatment are the following:
  - fine fraction consisting of electrode powder rich in target metals;
  - coarse magnetic fraction;
  - coarse non-magnetic fraction.



- In order to optimize the operating parameters associated with the equipment that make up the mechanical section, various tests were carried out using the following types of batteries: LG Li-Ion batteries with mixed chemistry, HG batteries with mixed chemistry, HG NMC batteries and NiHM batteries.

TREATMENT	PARAMETERS STUDIED
Cryogenic treatment	Refrigeration temperature of the batteries, storage time in the cryogenic treatment and size of the under-screen of the grinder
Grinding	
Sieving	Screen size
Magnetic separation	Distance of the magnetic tape
Washing treatment	Solid/liquid ratio

Chemical characterization

- All the fractions coming from the pre-treatment have been characterized.

MAGNETIC FRACTION	%
Magnetic fraction (steel)	88,2%
Electrodic powder (black mass)	11,8%

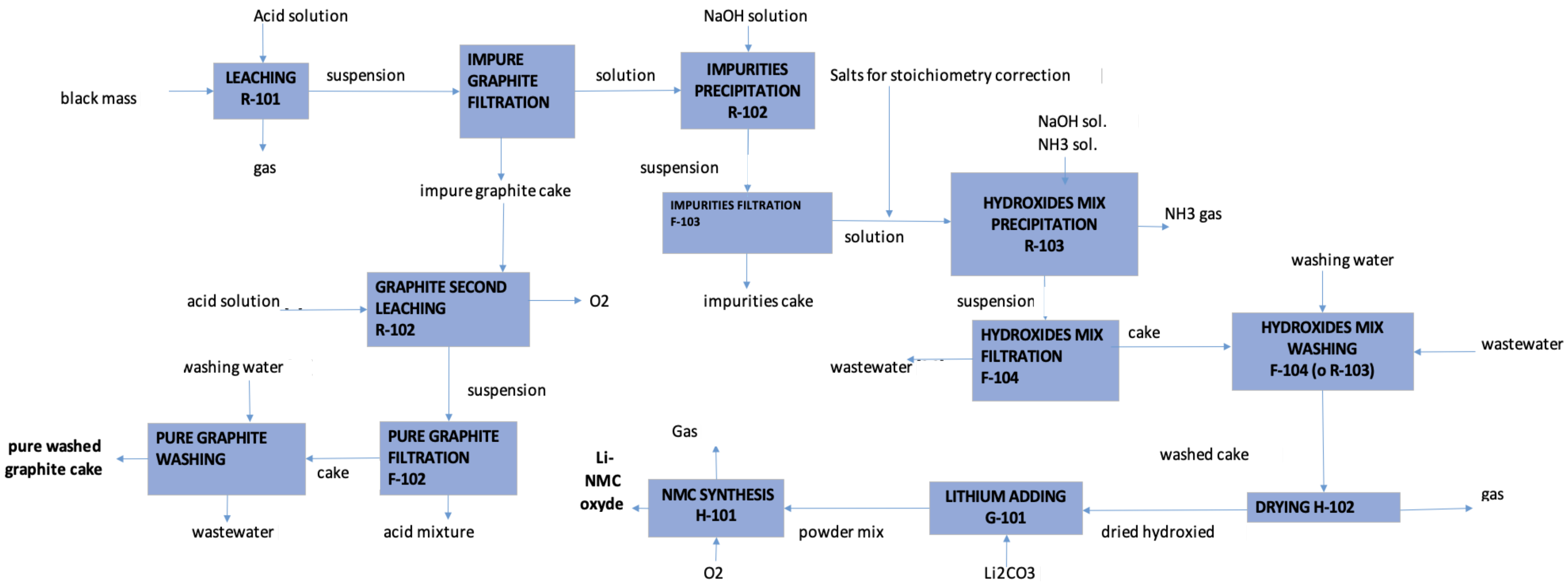
NO MAGNETIC FRACTION	%
Copper and Aluminium	53,9%
Plastic and paper	46,1%

ELECTRODIC POWDER CHEMICAL COMPOSITION			
	HG	LG	NiMH
Metal	(mg/g)	(mg/g)	(mg/g)
Nickel (Ni)	160,3 ± 1,55	90 ± 10	479,3 ± 18,5
Cobalt (Co)	91,6 ± 0,9	36,4 ± 5,6	61,2 ± 2,1
Lithium (Li)	26,5 ± 0,4	31,8 ± 4,7	-
Potassium (K)	-	-	18,2 ± 5,0
Copper (Cu)	4,0 ± 1,7	2,3 ± 1,2	9,3 ± 0,7
Aluminum (Al)	5,8 ± 1,3	2,6 ± 0,7	7,6 ± 0,9
Iron (Fe)	2,1 ± 0,3	2,2 ± 0,4	5,6 ± 0
Calcium (Ca)	-	-	2,1 ± 2,8
Magnesium (Mg)	-	-	10,1 ± 5,0
Zinc (Zn)	-	-	12,4 ± 0,9
Cesium (Ce)	-	-	48,5 ± 1,1
Praseodymium (Pr)	-	-	6,5 ± 3,5
Neodymium (Nd)	-	-	9,1 ± 1,6
Yttrium (Y)	-	-	1,9 ± 0
Lanthanum (La)	-	-	99,8 ± 3,1
Manganese (Mn)	55,3 ± 0,5	122,7 ± 10	25,9 ± 0
Lead (Pb)	-	-	0,6 ± 0
Titanium (Ti)	-	-	0,2 ± 0

Summary of pre-mechanical activity respect to project target

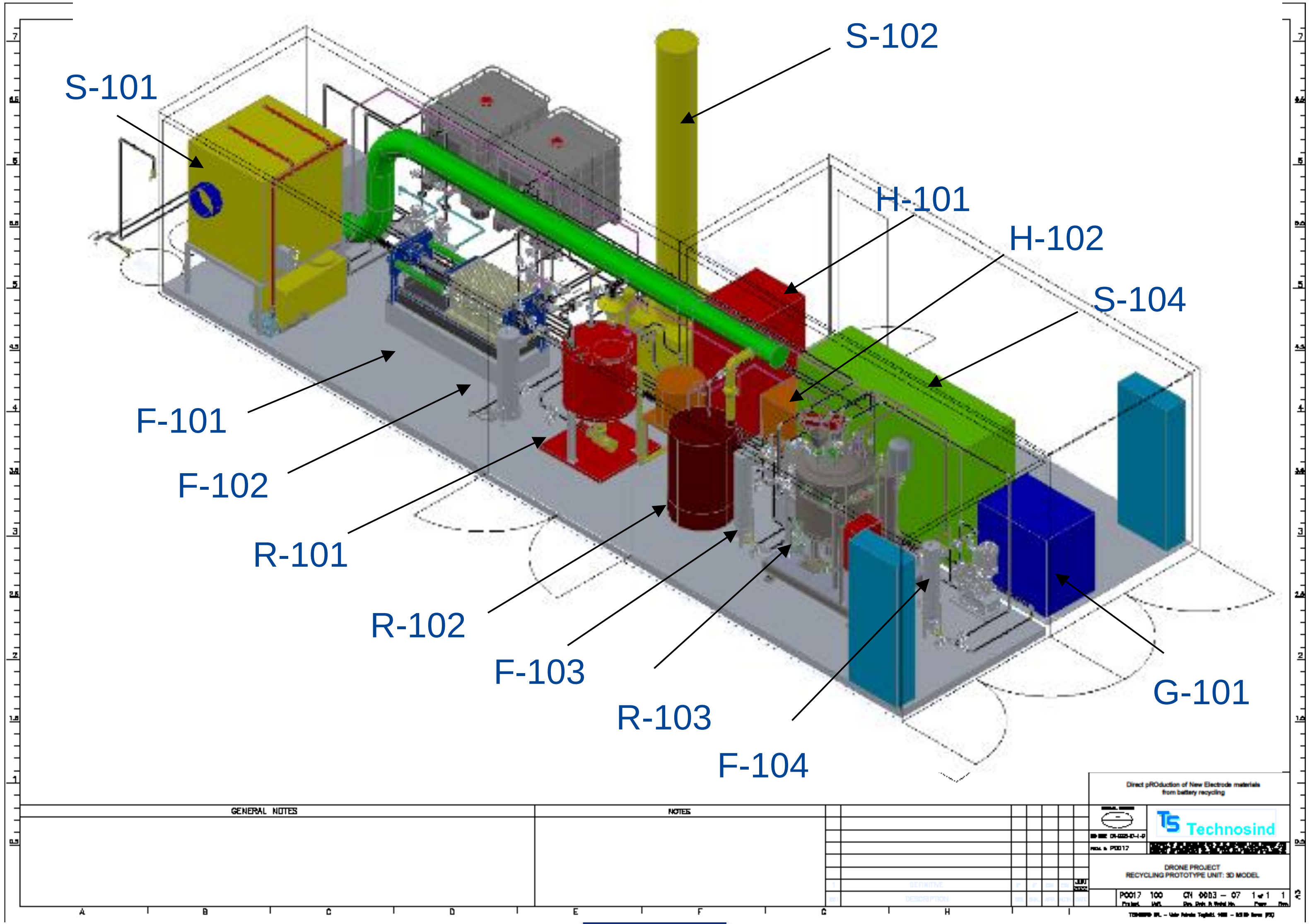
ACTIVITIES	PROJECT GOALS	ACTIVITY STATUS (M45)
Battery collection (Li-ion and Ni-MH batteries)	3000 kg	3553 kg
Aluminium and copper recovery	450 kg	906,7 kg
Plastic and paper recovery	360 kg	
Steel scraps recovery	460 kg	494,5 kg
Electrodic powder recovery	1350 kg	1380

LIFE DRONE block flow diagram: hydrometallurgical and synthesis section process



❖ The hydrometallurgical and synthesis prototype has a potential of 30 kg/batch of black mass.

Layout: 3D model



Outdoor containers overview

Hydrometallurgical section container

Synthesis section container

Hydrometallurgical section overview

Leaching reactor

Purification reactor

Precipitation reactor

Hydrometallurgical section overview

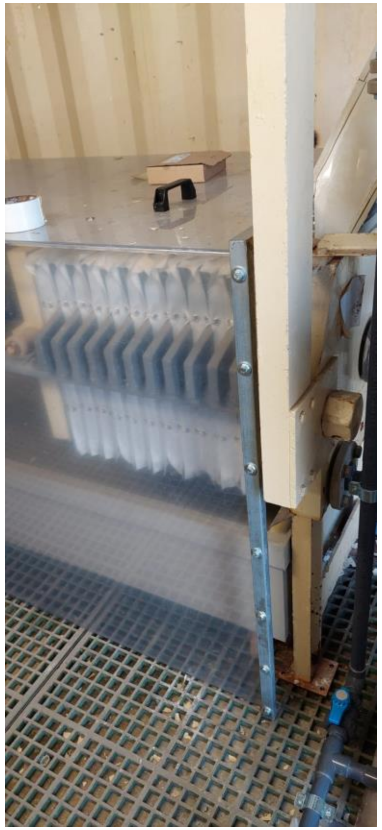
Acid Abatement system

Leaching reactor

Precipitation reactor

Filters

Filterpress
for leachate

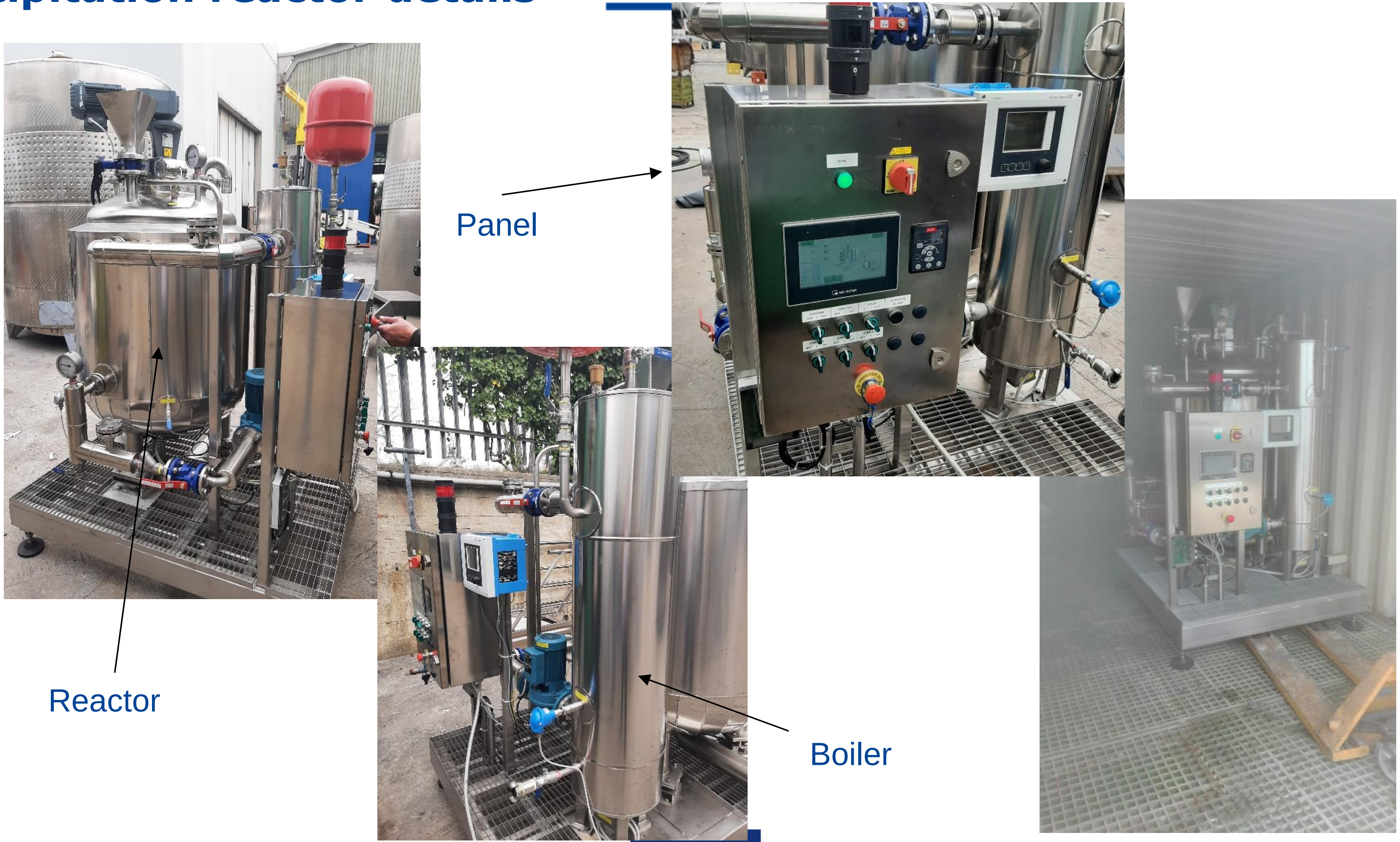


Impurities
filter

NMC hydroxides
filter

High purity
Graphite filter

Precipitation reactor details



Synthesis section

Mill

Isolator

Dryer

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Reagents and ammonia abatement system

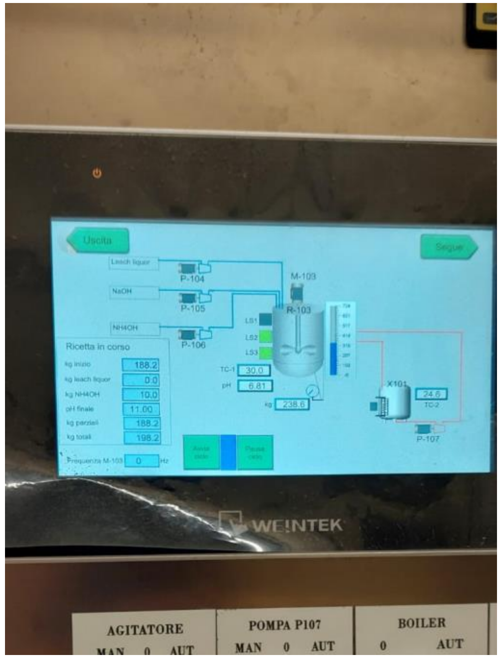
Ball mill and dryer details

Ball mill

Dryer

Control system

The main machines have local control panels but all of them feed into a centralised control system that allows remote management and monitoring



Black mass received from SEVAL

- ECOREC at its technological platform received from SEVAL the black mass coming out of the section of the prototype relating to the pre-treatment of LIBs and NiMHs that have reached the end of their life.

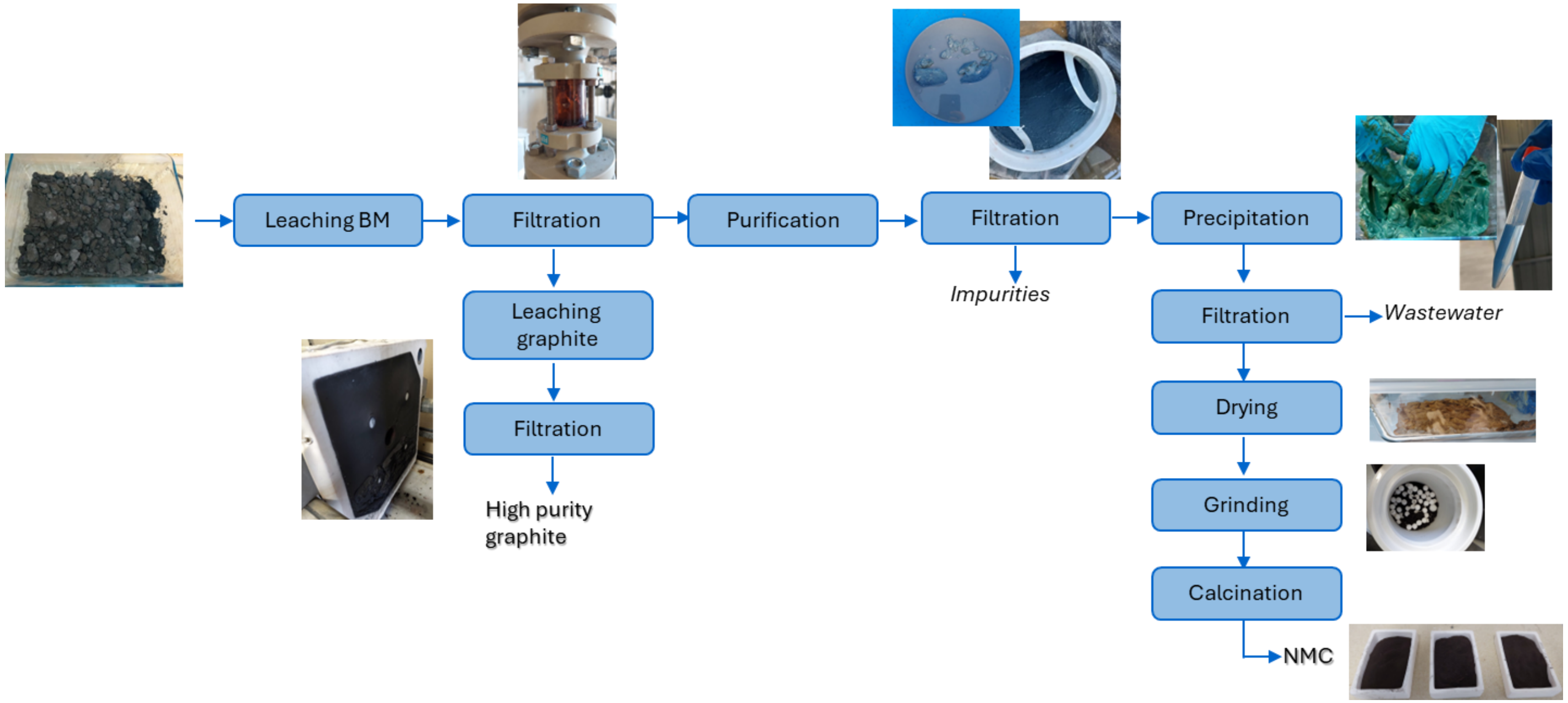
TOTAL BLACK MASS (kg)	PARTIAL AMOUNT (kg)	BATTERY TYPE
1380	1092	HG Li-ion
	201,7	LG Li-ion
	86,25	NiMH

Operating parameters optimized

- During the demonstration campaign, particular attention was given to the optimization of the following operating parameters:

PROCESS STEP	PARAMETERS
Electrodic powder leaching	Temperature and reaction time
Graphite leaching	Temperature, reaction time and L/S ratio
Purification and filtration	pH value, reaction time, mesh size
Hydroxides precipitation, filtration and washing	Time, rotation speed, mesh size, water ratio for washing
Drying	Temperature and time
Grinding	Time, grinding cycle, ratio spheres/solid, rotational speed
Calcination	Ramp speed, temperatures and time

Demonstration campaign: hydrometallurgical and synthesis section



SUMMARY OF THE DEMONSTRATION CAMPAIGN	
Total batches	45
BM treated (kg)	1350
NMC produced (kg)	666
High grade graphite (kg)	461

Procedure to cell assembly

- Electrochemical characterizations were carried out, by UNIROMA, using electrodes prepared by casting a dispersion of the cathodic materials on aluminium foil current collector.

- The dispersion was composed by:
  - 10% of carbon conducting agent (Super P, Timcal)
  - 10% binder (PVDF SigmaAldrich)
  - 80% of cathodic materials
  - N-methyl pyrrolidone (NMP, Sigma-Aldrich) as solvent

CR2032 coin-type cell

- The resulting film was cut into disks of 10 mm diameter and dried.

- The prepared cathodes were assembled in a two electrode R2032 coin-type cells where lithium metal disk was used as negative electrode.

- The employed electrolyte was LiPF6 1 M in an Ethylene Carbonate

- Dimethyl Carbonate, 1:1 v:v solution (LP30).

- Each cell was assembled in an argon-filled glove box.

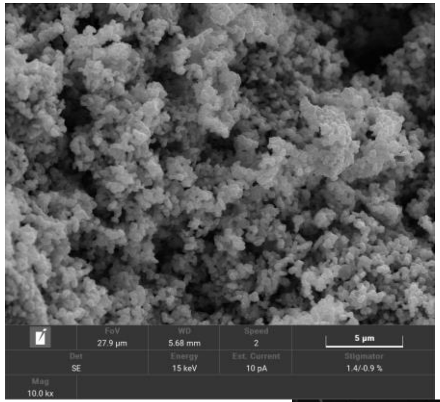
Glove box

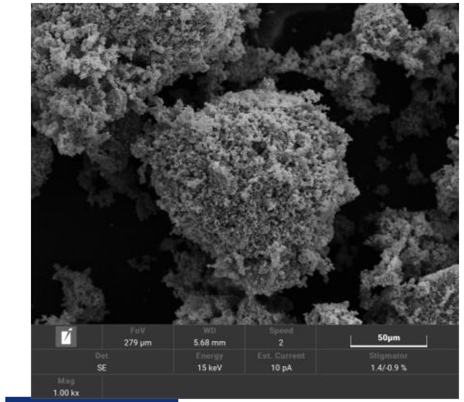
❖ This slide shows the chemical and SEM characterizations and the stoichiometry of the NMC obtained with the optimized synthesis parameters in the prototype and analyzed by UNIROMA.

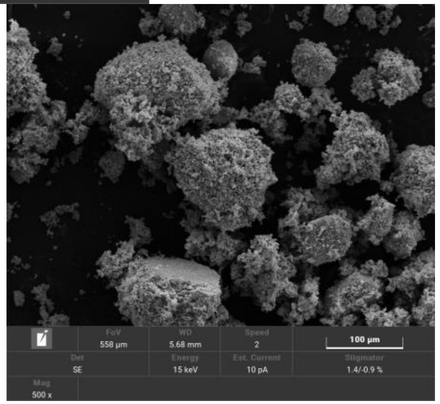
STOICHIOMETRY OF THE CATHODE MATERIALS		
Metal	**Stoichiometry ratio**	**St. dev.**
Li	0,9	0,15
Ni	0,34	0,05
Mn	0,32	0,05
Co	0,33	0,005

AVERAGE METALS CONCENTRATION IN CATHODE MATERIALS		
Metals	**Average concentration (mg/g)**	**St. dev.**
Li	49,1	8,73
Mn	108	20,6
Ni	174	13,3
Co	170	13,4
Fe	0,81	0,22
Al	2,87	0,12
Cu	5,36	0,09

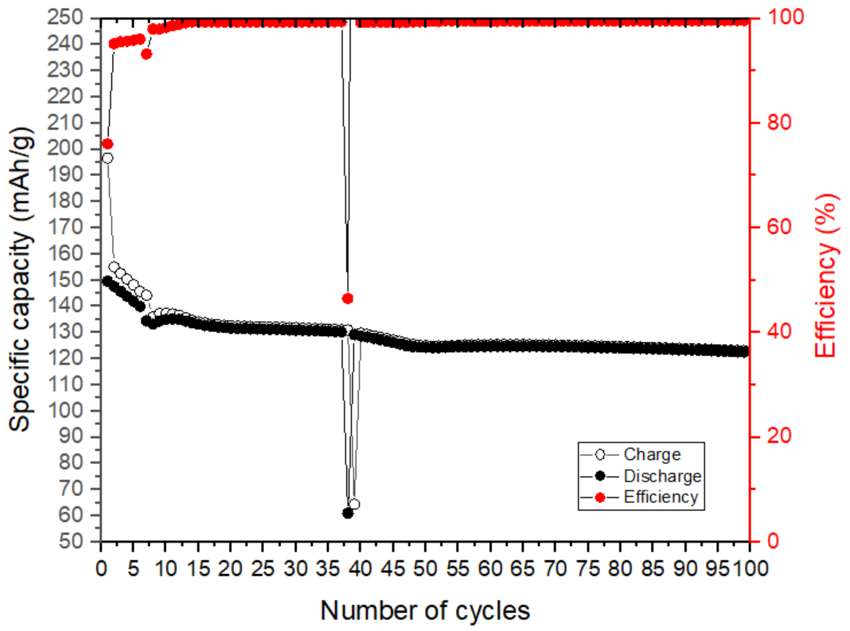
SEM





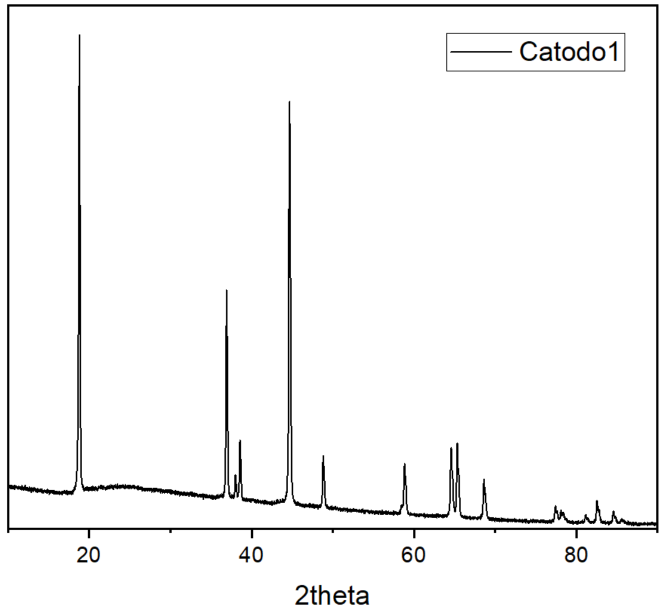


❖ This slide shows the X-ray characterization and the electrochemical characterization of the last NMC obtained with the **optimized synthesis parameters** in the prototype and analyzed by UNIROMA.



Electrochemical characterization

MAX CAPACITY	CAPACITY RETENTION
150 mAh/g	83%



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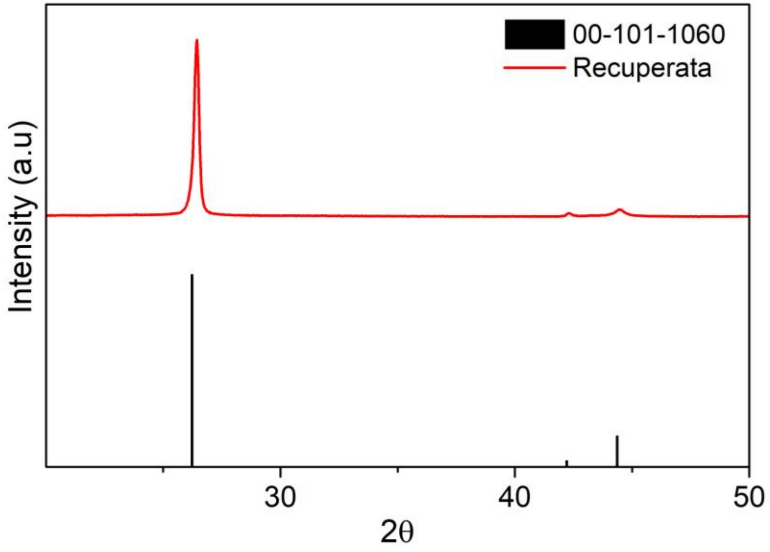
❖ This slide shows chemical characterization and XRD spectroscopy of the graphite obtained with the optimized parameters in the prototype and analyzed by UNIROMA.

Graphite purity

GRAPHITE PURITY	
Element	**Concentration (g/g)**
Co	0,001
Li	0,0006
Ni	< 0,00004
Mn	< 0,00006
Cu	< 0,00009
Fe	0,0003
Zn	< 0,0005
Cd	< 0,0005
Al	0,008



XRD spectroscopy

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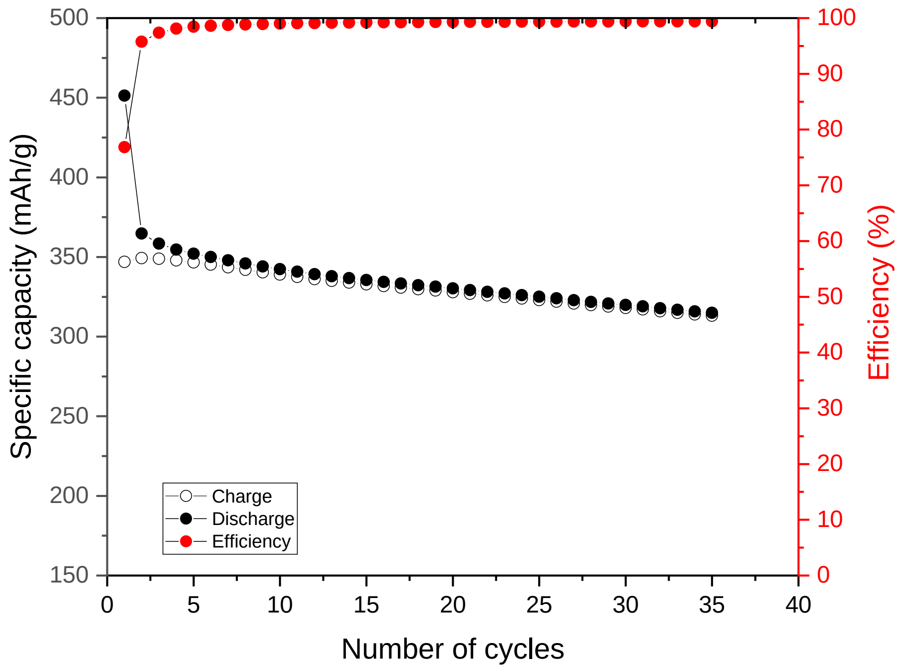
❖ This slide shows electrochemical characterization of the graphite obtained with the optimized synthesis parameters in the prototype and analyzed by UNIROMA.

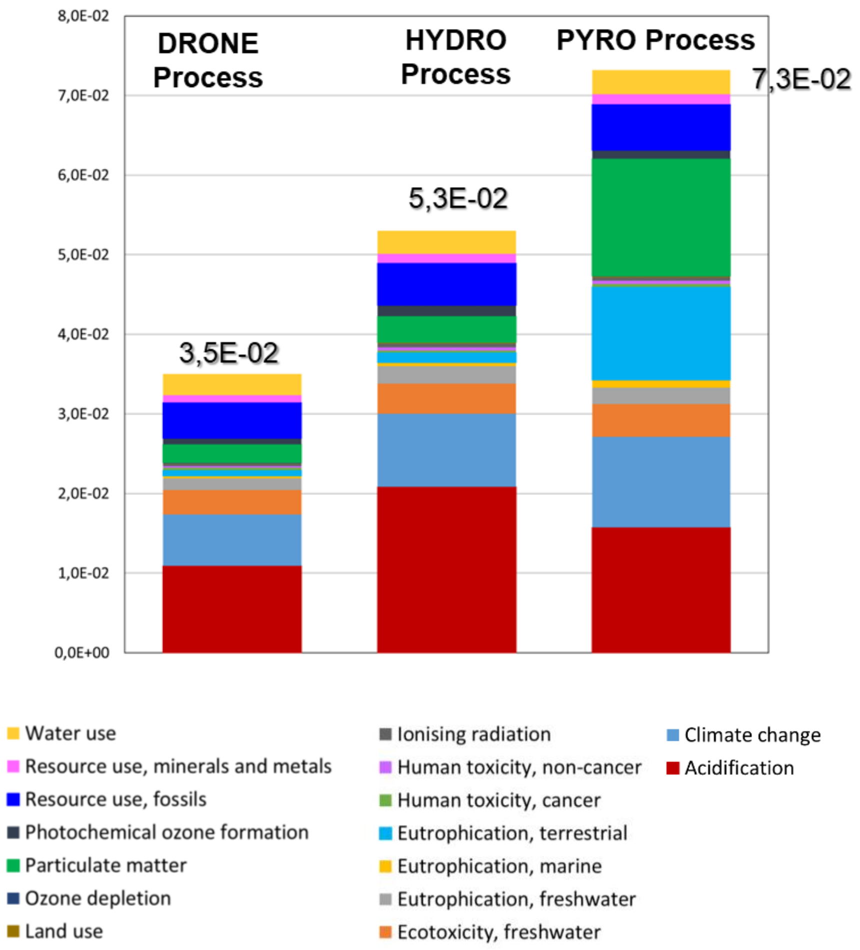


MAX CAPACITY	CAPACITY RETENTION
350 mAh/g	93%

❖ After a thermal treatment, the average size of the graphite particles is reduced from 80 to 35 micron

All project targets relating to the demonstration campaign were achieved

LCA: environmental performance index



The innovative LIFE DRONE process confirms the environmental benefit of the process.

The LIFE DRONE process compared to the hydrometallurgical base case allows a reduction equal to **59%** in terms of kg $CO_{2\ eq}$.
(253 vs 613 kg $CO_{2\ eq}$ /ton batteries)

Full-scale plant process simulation (500 t/y)

- The marketability analysis allow the definition of a **minimal scale** of the industrial plant.
- Assuming the construction of a plant in each Italian region in order to minimise the costs associated with transport, an average capacity of about **500 tons/year** for each plant is estimated.
- **Improvements** with respect to the DRONE prototype:
  - All the L/S separations are carried out in **filter presses**. Such equipment for full-scale plants are more efficient than basket filters, allowing the separation of a solid phase with lower moisture content;
  - An **evaporative crystalliser** and a filter press have also been chosen for the process, in order to process the wastewater resulting after the precipitation step for the NMC synthesis;
  - A **system to produce N2** used in the NMC synthesis section has been added;
  - It is added a **compressor** to produce air for devices;
  - **Automatic solid handling systems** have been added, but they are not foreseen for the prototype scale.
  - **Jet miller grinding** (also for graphite treatment)

Costs and ROI/PBT of the industrial plant (potentiality: 500 t/y)

COSTS	
CAPEX	6.215.000 € (*)
OPEX	3.364.687 €
ROI	31.64 %
PBT	3.16 y

(*)Total investment cost= Equipment free on board + Direct and indirect costs

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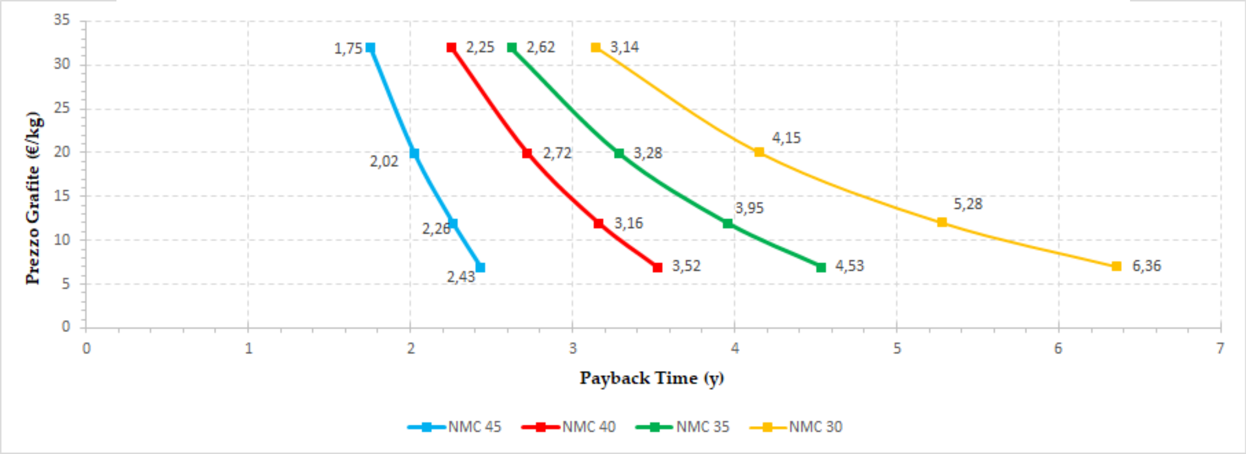
Variation of the graphite and NMC prices

	NMC (€/kg)															
	50				40				35				30			
Grafite (€/kg)	7	12	20	32	7	12	20	32	7	12	20	32	7	12	20	32



- Generally for an investment a suitable Payback period is about **3-5 years**.
- For a plant with a potential of 500 t/year of treated batteries:
  - Considering a NMC selling price between 50-35 €/kg, attractive payback times would be obtained for a possible stakeholder even considering a sales price of graphite equal to the minimum market price (7 €/kg);
  - Considering NMC selling price of 30 €/kg, the plant would give an acceptable payback time up to a selling cost of the graphite equal to about 15-20 €/kg.