eNiCoN

Sustainable processing of Europe’s low grade sulphidic and lateritic Ni/Co ores and tailings into battery grade metals

Brecht Dewulf, Philippe Muchez, Hannah Hughes, Anders Sand, Mari Lundström, Arne Peys, Kostas Komnitsas, Anna Kritikaki, Stelios Tabouris, Lefteris Kaklamanos, Giorgian Dinu, Lieven Machiels, Peter Tom Jones, Koen Binnemans

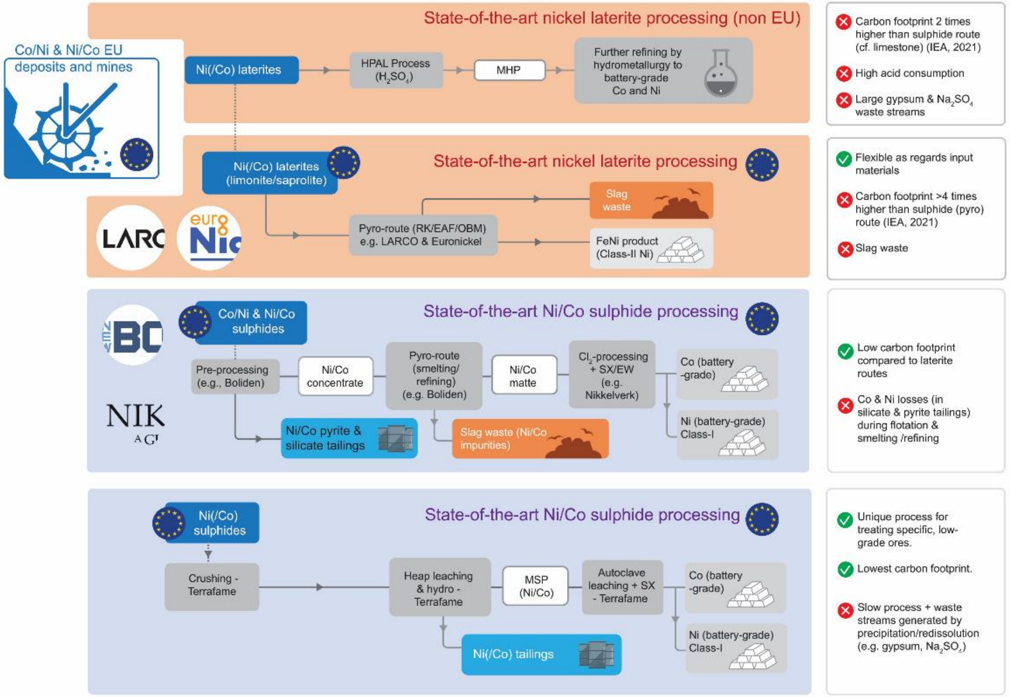
This project has received funding from the European Union's EU Framework Programme for Research and Innovation Horizon Europe under GA No 101058124

https://enicon-horizon.eu/

“Unlock the potential of Europe’s (low-grade) Ni/Co resources”

Domestic European Ni/Co processing:

- Improve existing flowsheets
- Focus on near-zero waste processing – Circular Hydrometallurgy
- Development of new HCl-based route for laterites, sulphides and secondary sources



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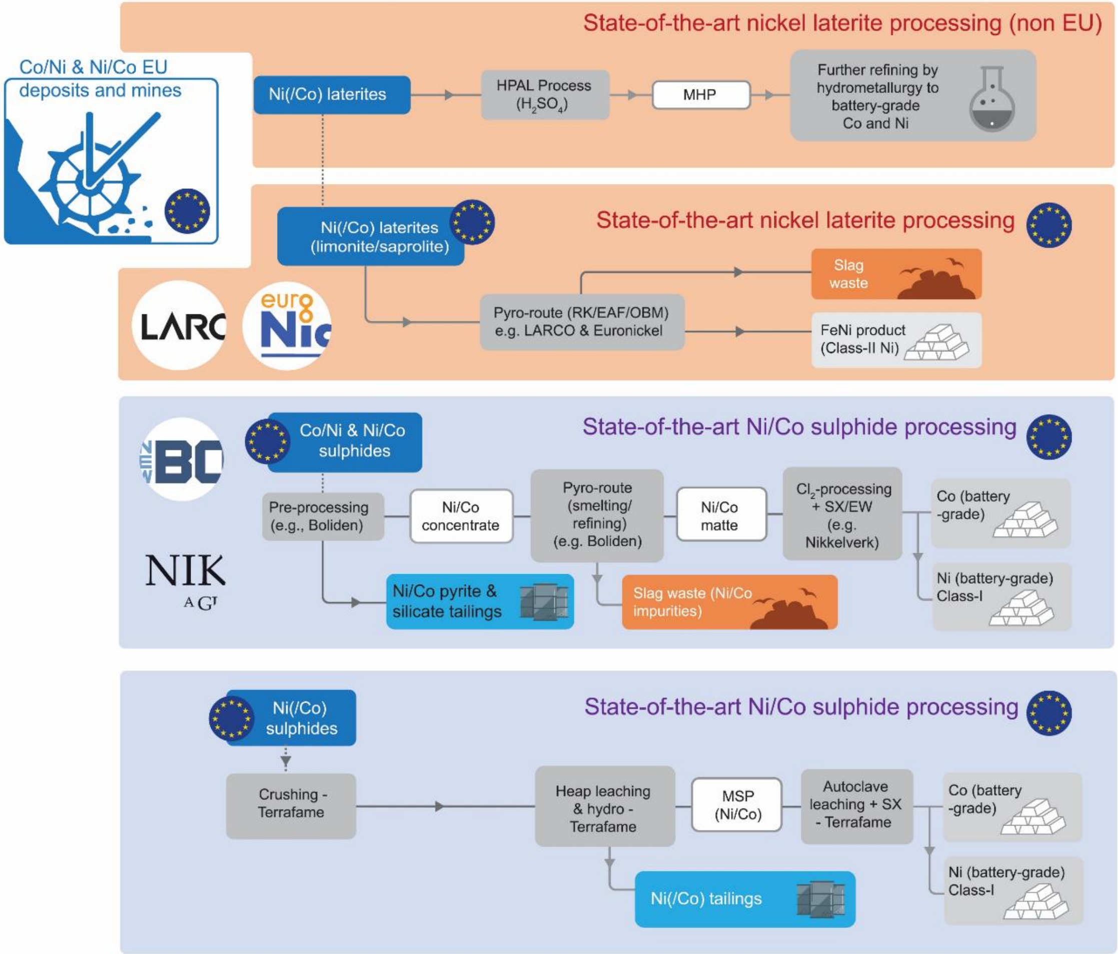

- ✗ Carbon footprint 2 times higher than sulphide route (cf. limestone) (IEA, 2021)
- ✗ High acid consumption
- ✗ Large gypsum & Na_2SO_4 waste streams

- ✓ Flexible as regards input materials
- ✗ Carbon footprint >4 times higher than sulphide (pyro) route (IEA, 2021)
- ✗ Slag waste

- ✓ Low carbon footprint compared to laterite routes
- ✗ Co & Ni losses (in silicate & pyrite tailings) during flotation & smelting /refining

- ✓ Unique process for treating specific, low-grade ores.
- ✓ Lowest carbon footprint.
- ✗ Slow process + waste streams generated by precipitation/redissolution (e.g. gypsum, Na_2SO_4)

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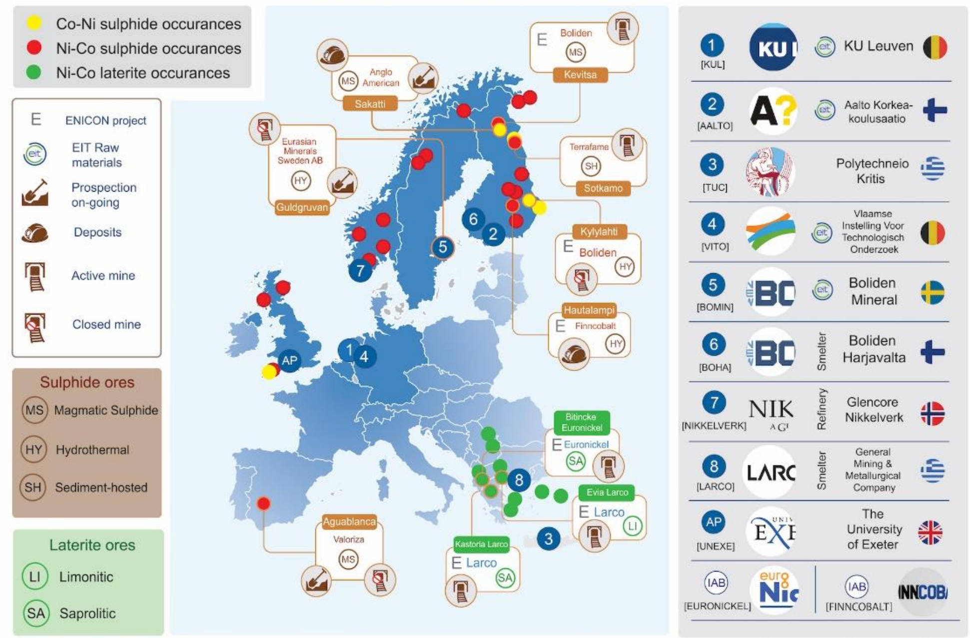
Materials

eNiCoN



- Laterites

- Ni/Co sulphide concentrate

- Class II FeNi
- MHP/MSP

- Silicate and pyrite tailings

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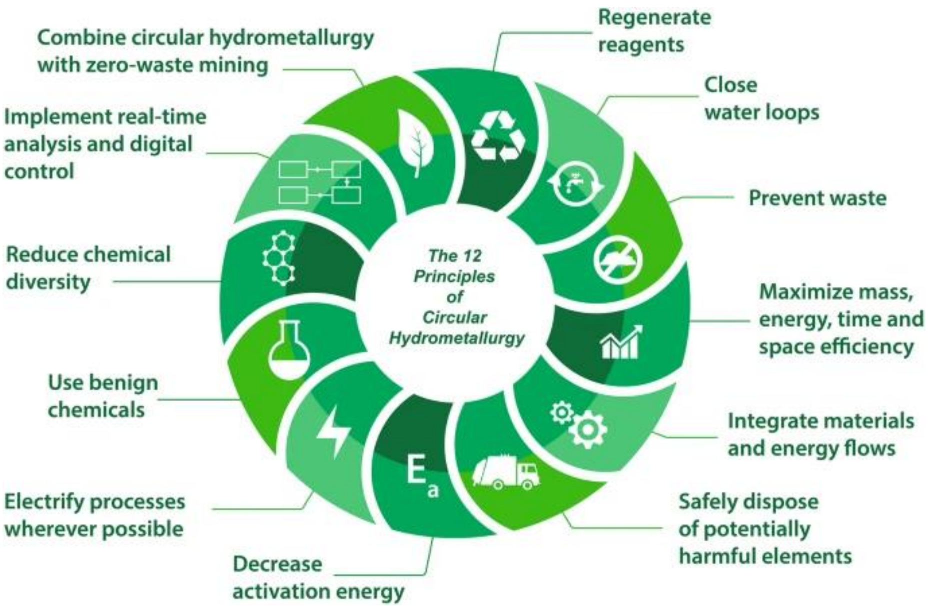
Circular Hydrometallurgy



Binnemans, K.; Jones, P. T. The Twelve Principles of Circular Hydrometallurgy. *J. Sustain. Metall.* **2022**, *9* (1), 1–25. https://doi.org/10.1007/s40831-022-00636-3.

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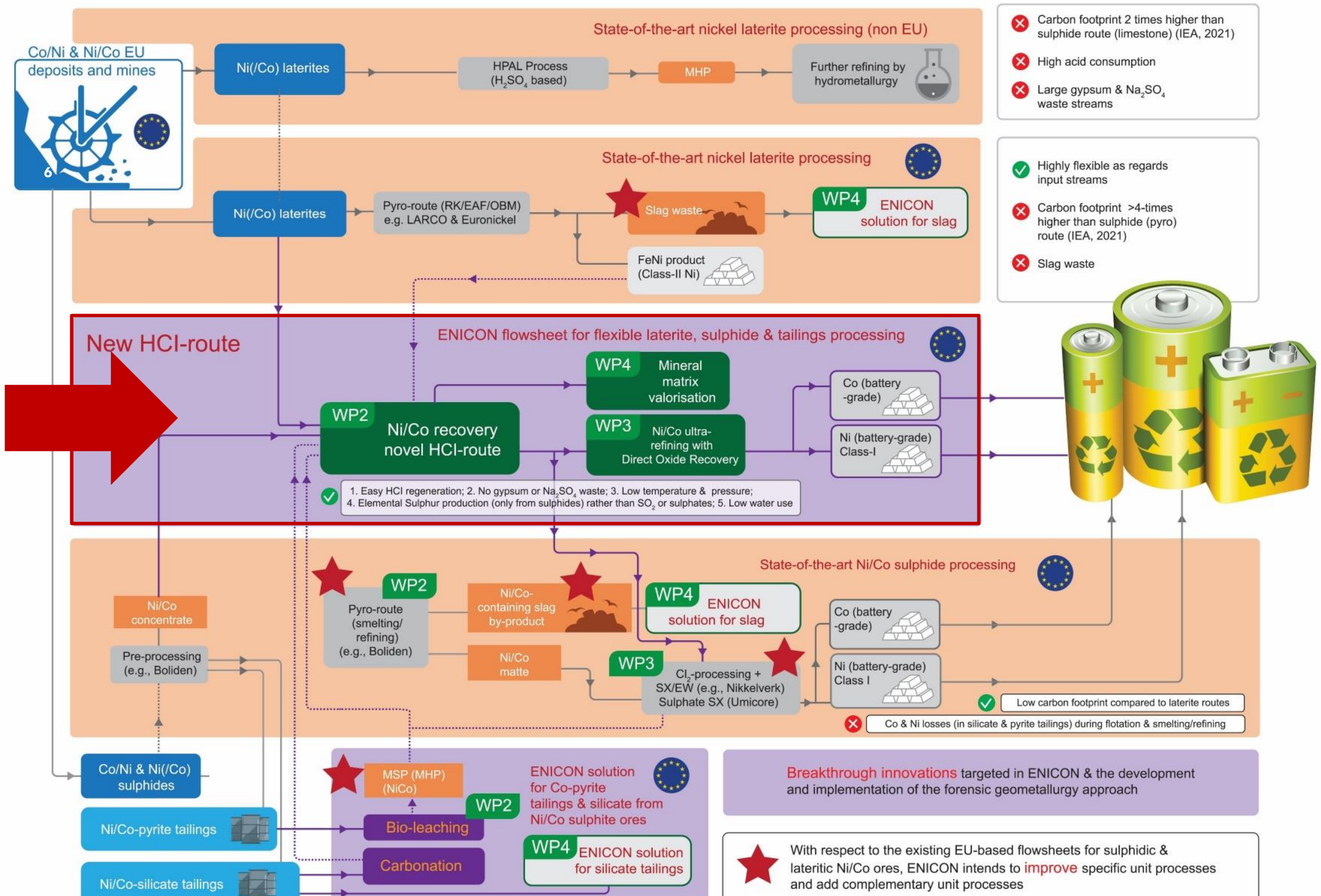

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Project summary

- HCl-route for processing ore materials/concentrates, intermediates and waste streams
- Ultra-refining: Direct oxide recovery
- Matrix/slag valorization
- Support through:
  - Forensic geometallurgy
  - Thermodynamic modeling of leaching, solvent extraction and hydrolytic distillation
  - LCA-TEA

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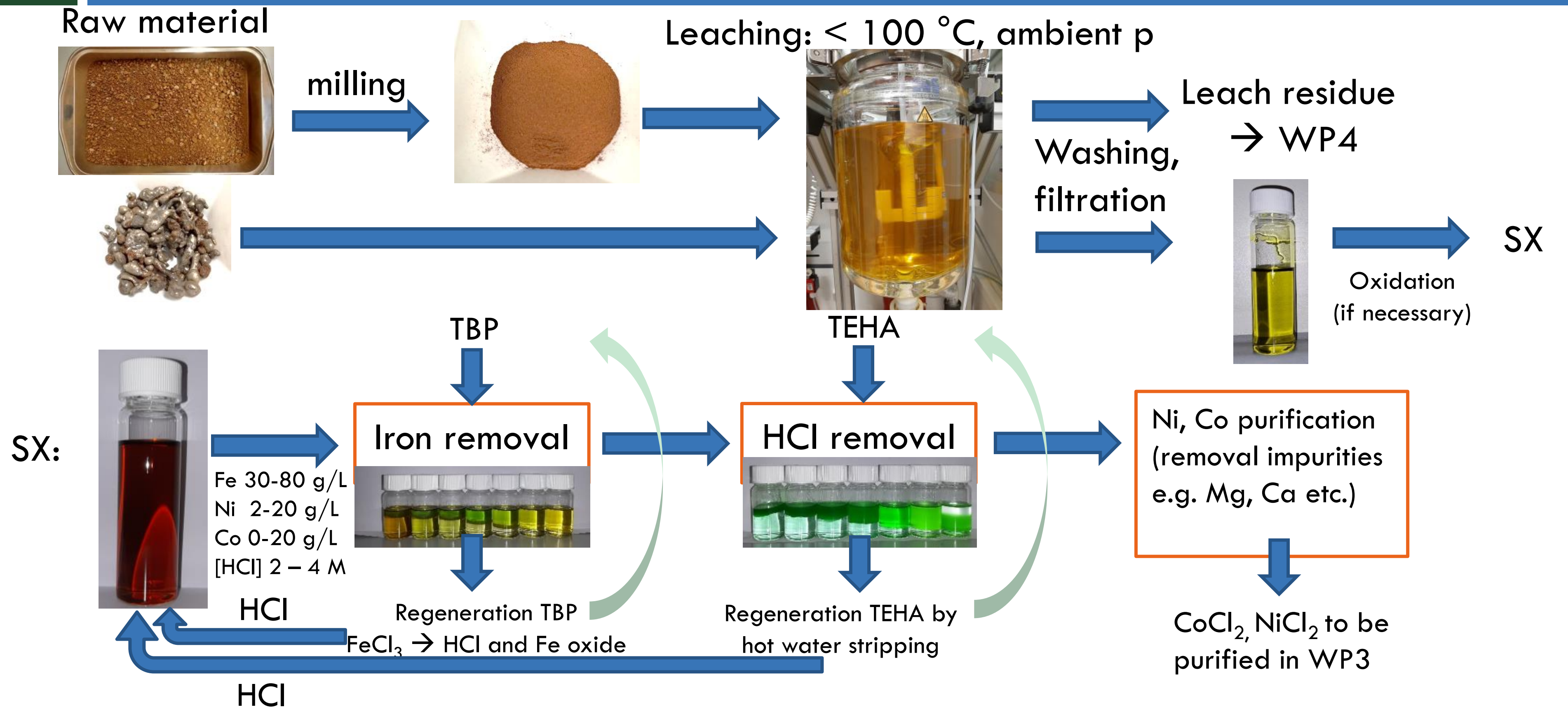
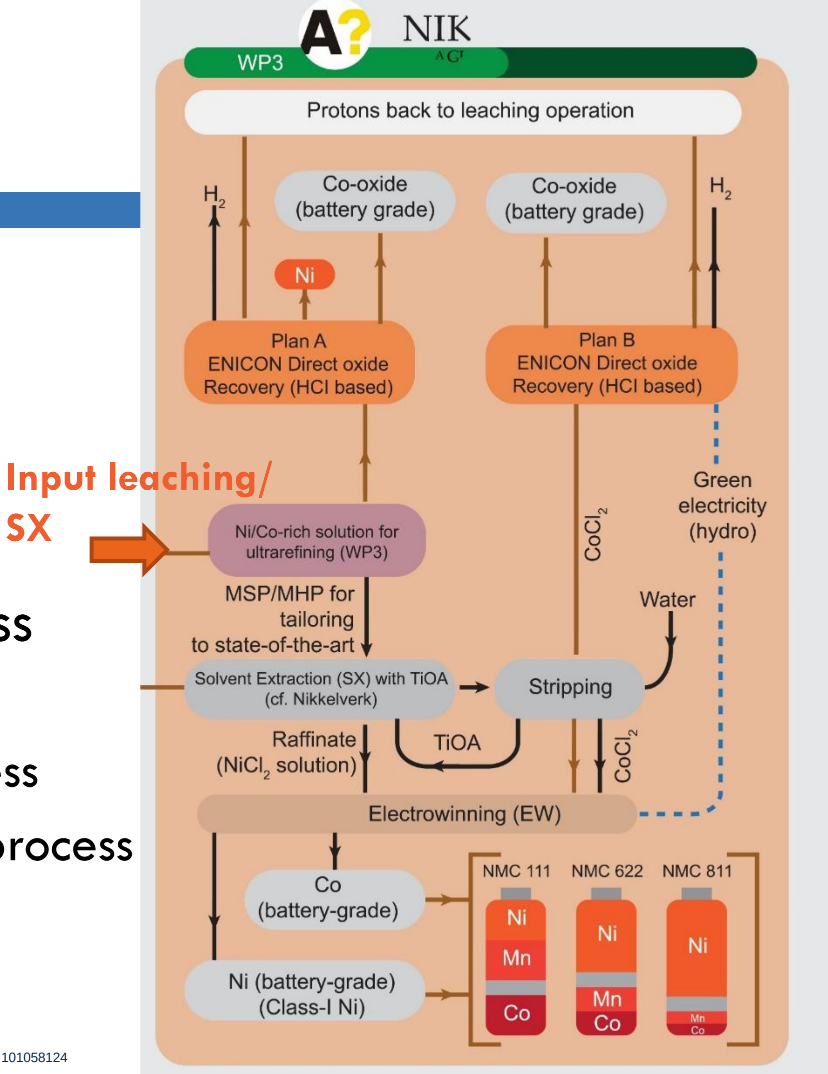
HCl-route for Ni/Co recovery: leaching and solvent extraction



HCl-route for Ni/Co recovery: Direct oxide recovery

- Selective electrochemical Co-oxide recovery and Ni or H_2 recovery at cathode → chemical-free Ni/Co separation
- Ultra-refining by Cl_2-SX/EW process Nikkelverk of MHP/MSP from
  - Strip solution after Ni/Co HCl-SX process
  - PLS bioleaching or oxidative leaching process (pyrite tailings)



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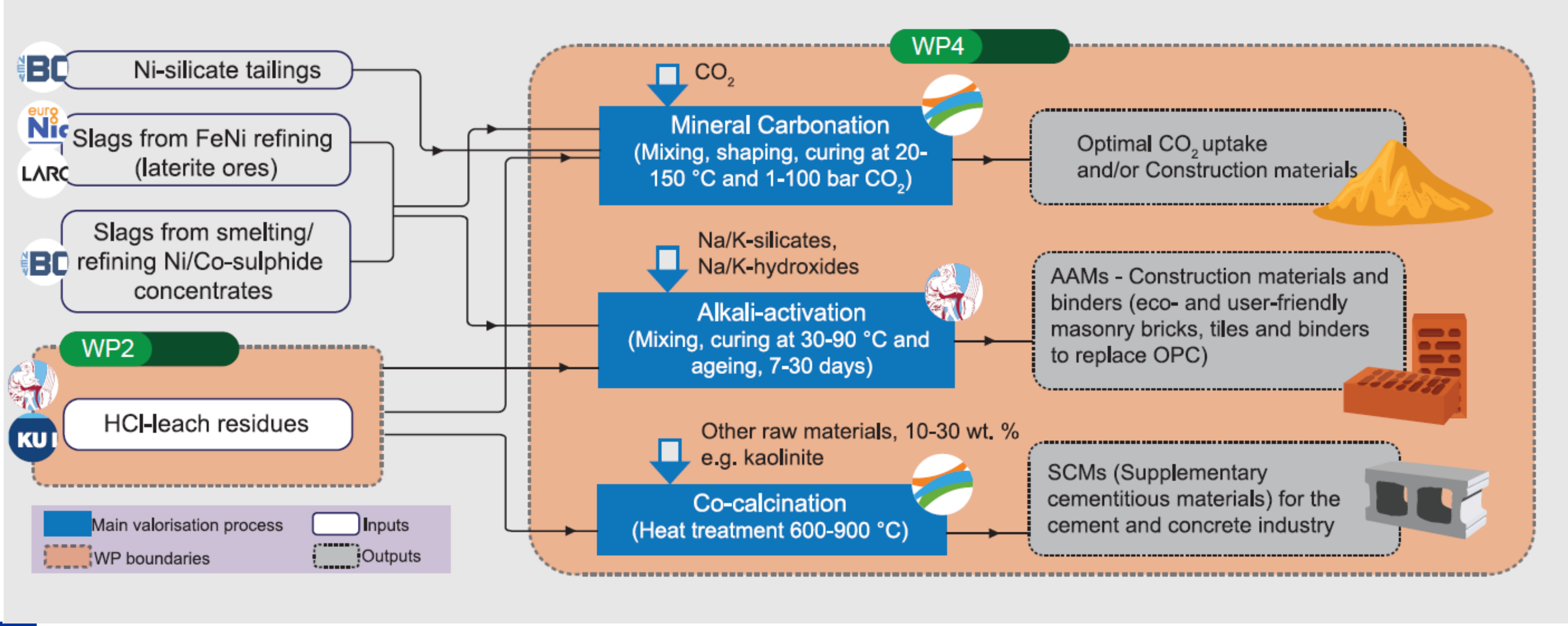
Reuse of mineral matrix and residues

- Stabilization/storage of residues containing Ni, Zn, Pb etc.
- Avoid landfilling and producing potentially profitable products
- CO_2 lean production of cement, concrete and other construction materials



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Conclusions

ENICON contributes to sustainable/responsible production of Co and Ni from European resources

- Besides laterites/sulphidic concentrates, also focus on processing intermediates and waste streams
- HCl-route as a best-in-class alternative for HPAL
- Focus on circular hydrometallurgy – reducing chemicals consumption and carbon footprint
- Valorization of mineral matrix/slags for construction materials

Impact
- Scientific: breakthrough research leading to major scientific advances in eco-friendly battery metal production
- Economic/technological: adapting matrix valorization and HCl-flowsheets in metal processing industries
- Societal: ENICON solutions lead to reduction of direct CO_2-emissions and near-zero waste metallurgy – potential increased global competitiveness EU battery industry, new resources, new jobs

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Gas-diffusion electrocrystallization (GDEx)

Xochitl Dominguez-Benetton

Funded by the European Union under Grant Agreements No 101069685, 101069644 and 101058163. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the European Climate, Infrastructure and Environment Executive Agency (CINEA). Neither the European Union nor the granting authority can be held responsible for them.

As we shift from a fossil fuel-based economy to an electrified one, the game-changer lies in our commitment to embrace reusing, remanufacturing, and recycling as the foundation of our practices.

This entails minimizing the extraction of primary resources, which, if we must insist on obtaining, should also be done sustainably.

Source: https://circulareconomy.europa.eu

Battery passport

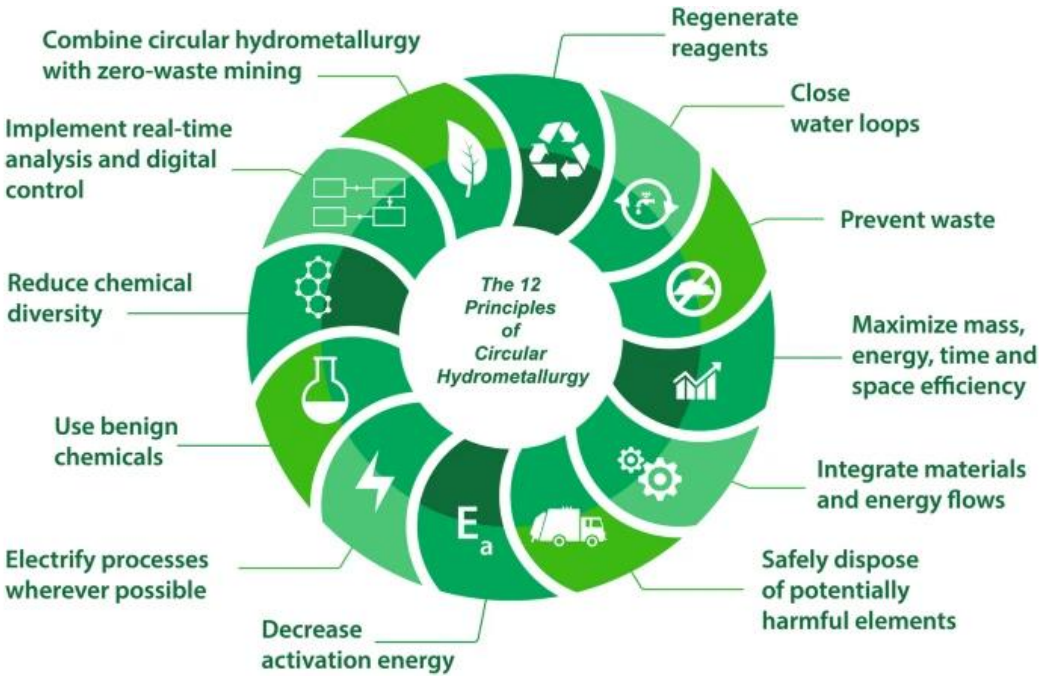
- Value chain transparency and impact
- Minimum acceptable standards for **sustainable, circular and responsible batteries**
- Validating and tracking progress towards achieving **sustainable, circular and responsible batteries**

What value does this effort hold if we don’t also commit to ensuring the sustainability of the underlying processes?

mining – metallurgy – manufacturing - recycling

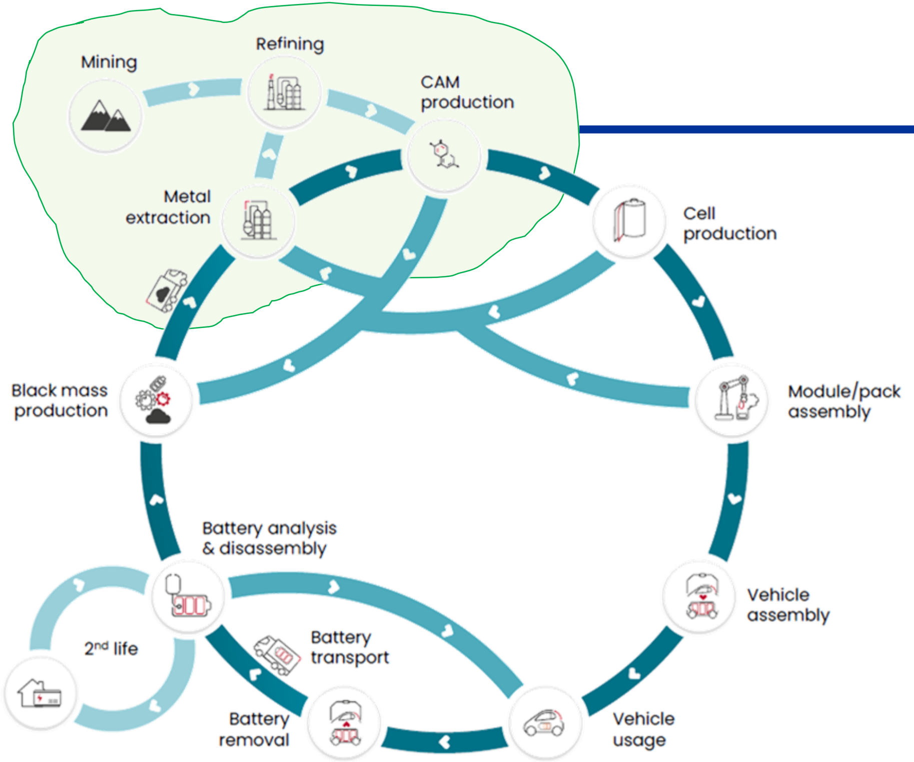
Future mining – metallurgy – manufacturing – recycling
Batteries included

- Less pyrometallurgy
- More hydrometallurgy
- No adverse environmental impact



Source: Binnemans & Jones. Journal of Sustainable Metallurgy (2023) 9:1–25

Battery value chain (circular economy)

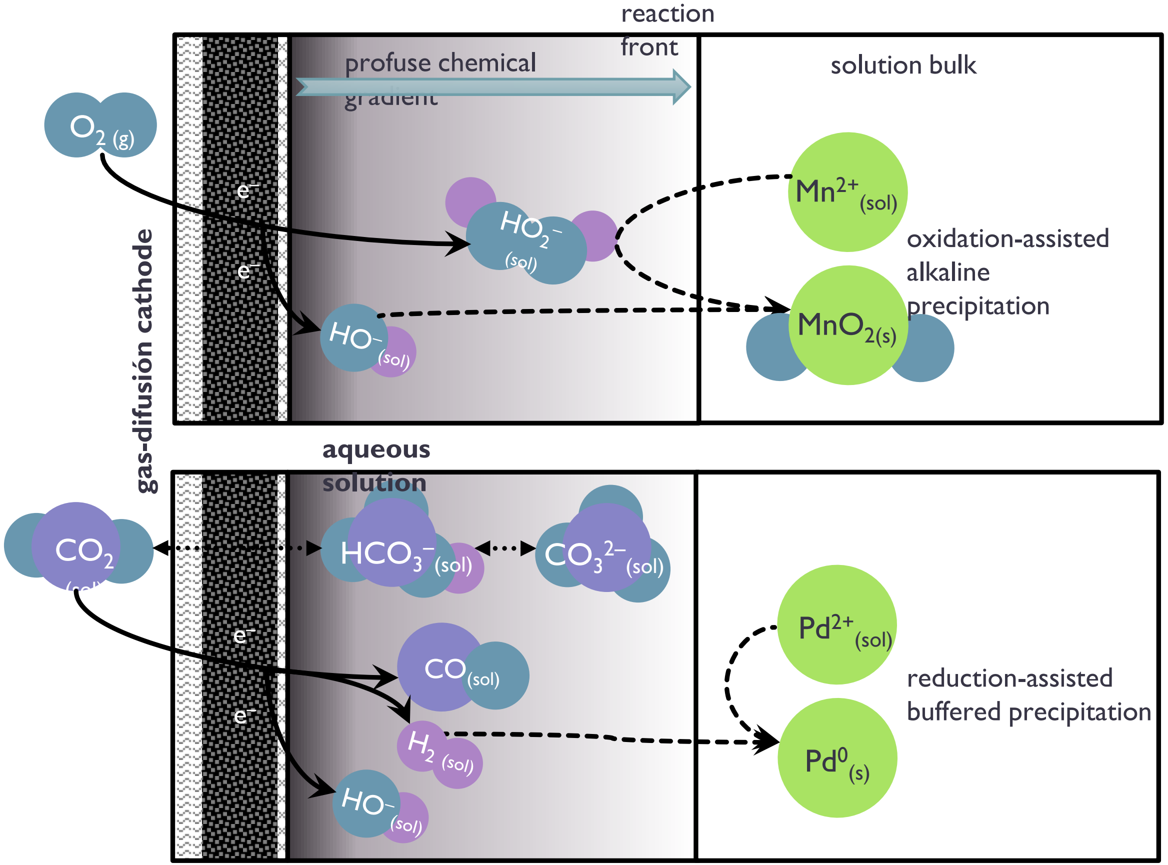


Source: FEV Consulting

Our focus is here

- Battery recycling
- Lithium mining (brines)

Technology focus: Gas-Diffusion Electrocrystallization (GDEx)

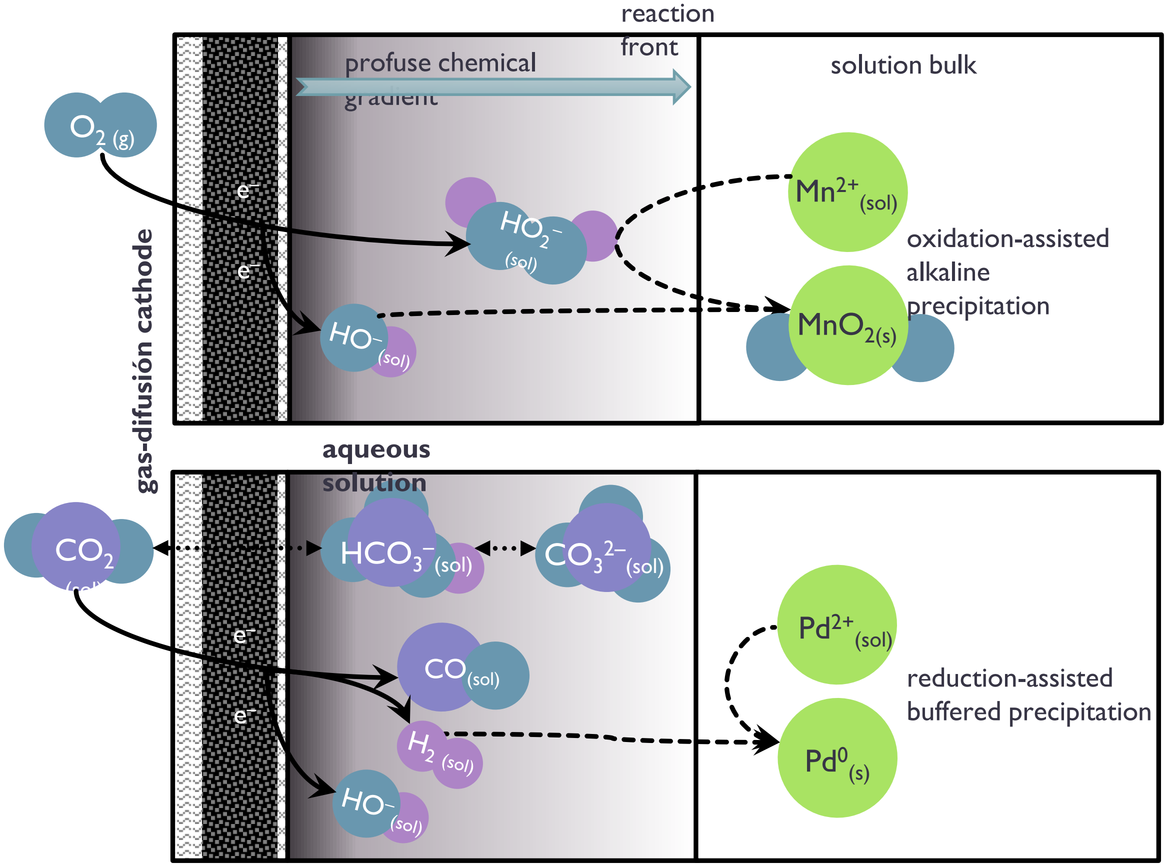


Prato et al. (2020) *J. Mat. Chem. A.* 8:11674
Eggermont et al. (2021) *React. Chem. Eng.*,6:1031.

- **Process of reactive precipitation of metals in solution with oxidizing or reducing agents produced in-situ by the electrochemical reduction of a gas, in a gas-diffusion electrode.**
- **The precipitates obtained are nanoparticle agglomerates.**

Martinez-Mora et al. (2023) *RSC Sust.* 1:454.

Technology focus: Gas-Diffusion Electrocrystallization (GDEx)



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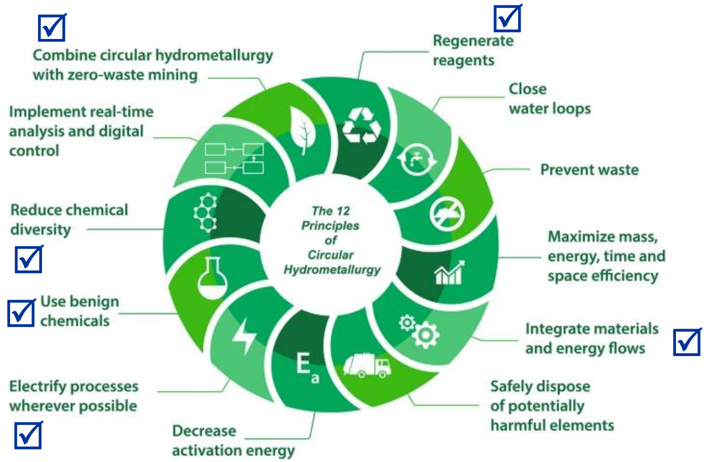


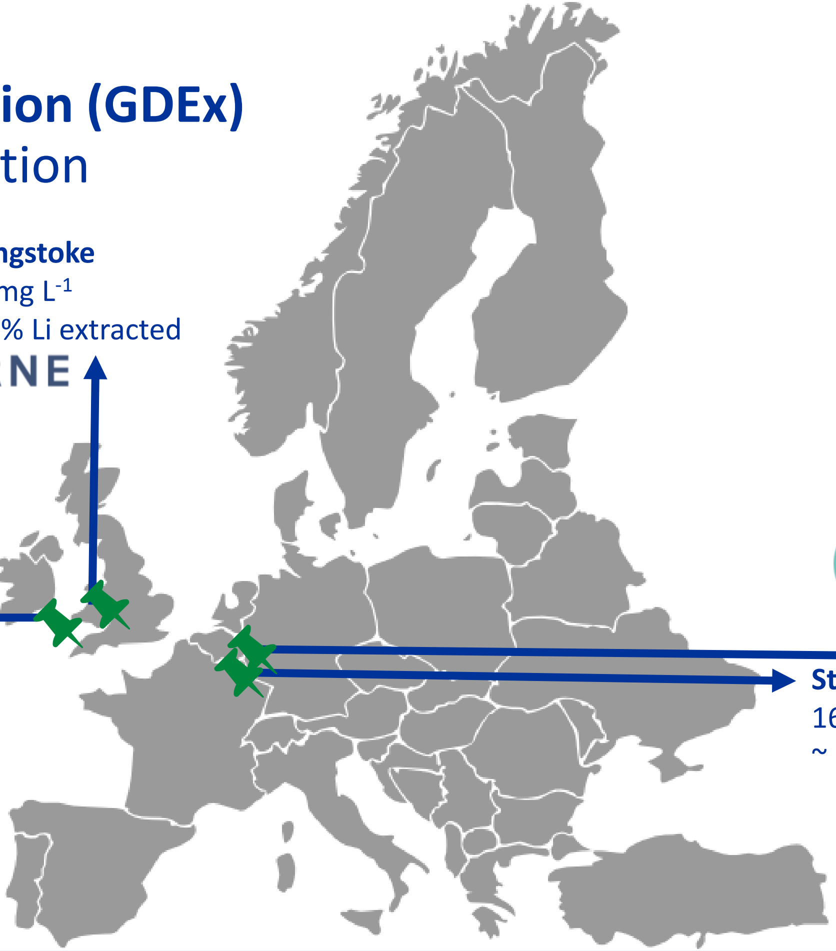
Martinez-Mora et al. (2023) *RSC Sust.* 1:454.

Gas-Diffusion Electrocrystallization (GDEx)

Selective Li-extraction

Geothermal brines

Others in progress



LiCORNE

Basingstoke
170 mg L^{-1}
~ 99 % Li extracted

CRM geothermal

Cornwall
40 mg L^{-1}
~75% Li extracted

LiCORNE

Germany
140 mg L^{-1}
~ 99% Li extracted

Strasbourg
160 mg L^{-1}
~ 70% Li extracted

3–10 kWh kg^{-1}
$LiOH \cdot H_2O$ equivalent

Patent pending.

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Gas-Diffusion Electrocrystallization (GDEx)

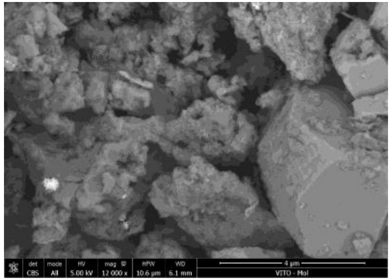
Li-extraction

Geothermal brines

Case: Li^+ 40 mg L^{-1}

Single-step treatment

1:0.5



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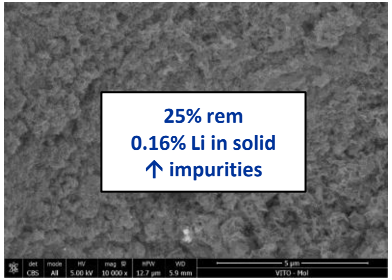

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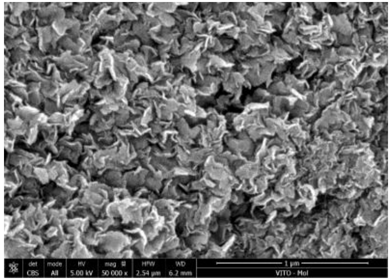

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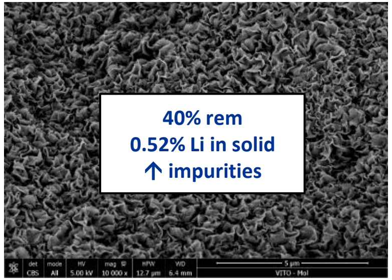
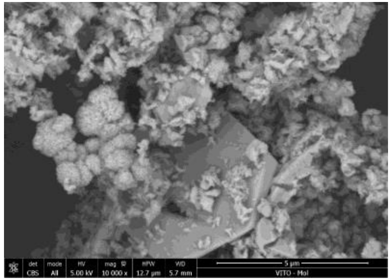


Morphology characteristic of LDH

Two-step squential treatment

No supplements



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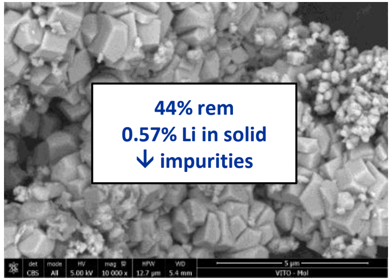


Muégano structure

Cleaner LDH

Compromise: higher energy consumption

Extraction and processing decoupled!

Gas-Diffusion Electrocrystallization (GDEx)

Li-extraction

Geothermal brines

• Economic assessment (15 y)

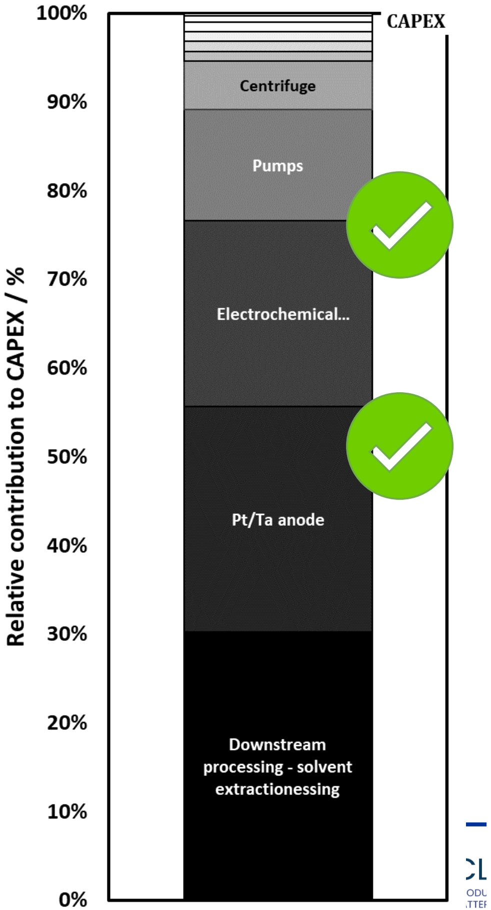
Total CAPEX (considering lang factor)
117 M€ (4 €/kg product)

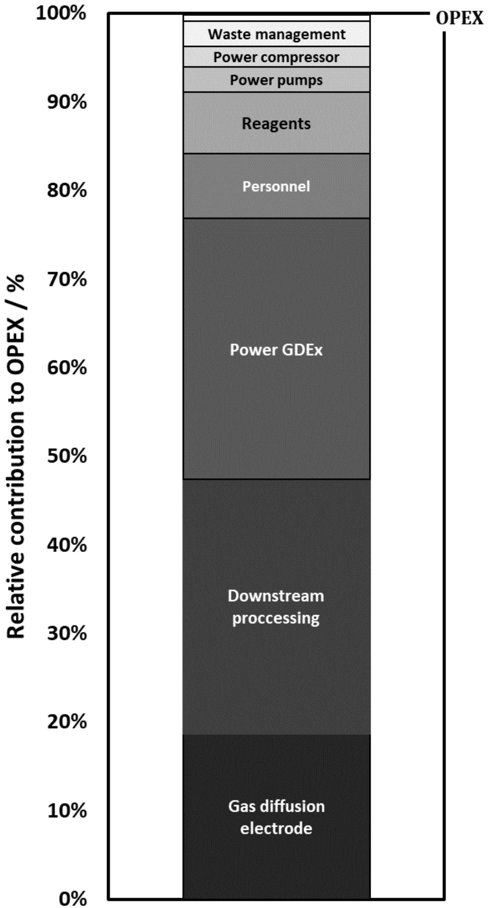
Total OPEX
27 M€ / year (13 €/kg product)

NPV 212 M€ **ROI** 181%
Operating profit margin 48%

Challenges:

- TRL-7 meeting KPIs for market readiness
- Still to optimize:
  - Cost of GDE
  - Energy efficiency GDEx
  - Downstream processing (battery grade materials)





Gas-Diffusion Electrocrystallization (GDEx)

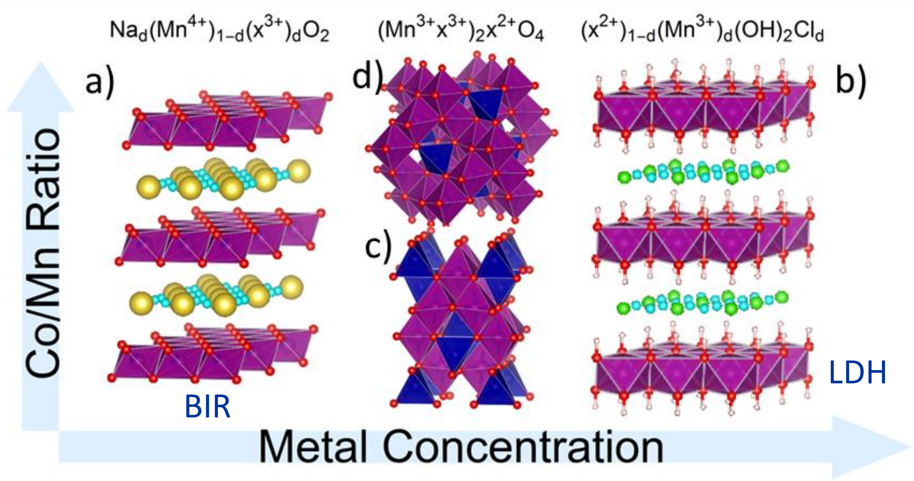
NMC recycling

Batteries

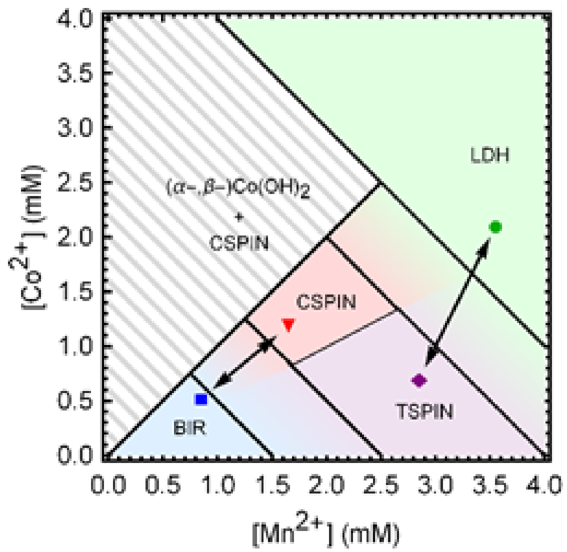
O_2 as the gas feed – No additives

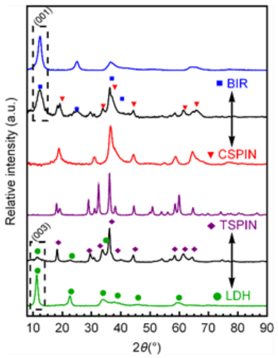
Differentiation of structural groups

SPINc



SPINt





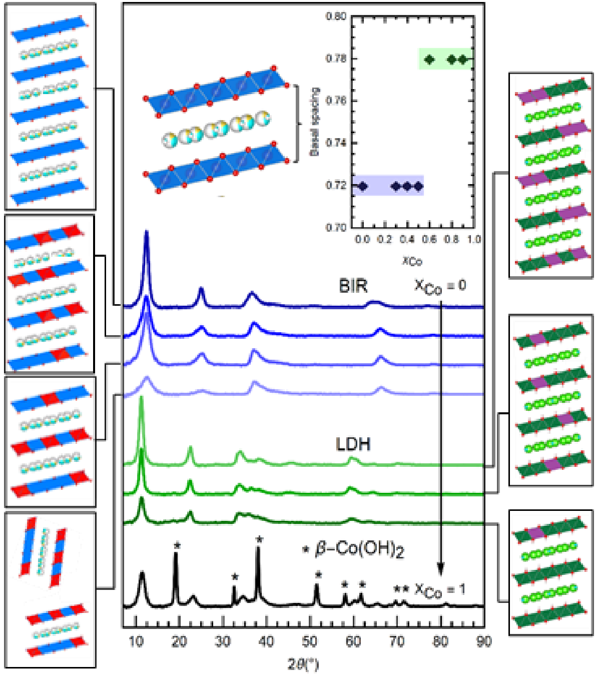
Prato et al. (2020) J Mat Chem A doi: 10.1039/D0TA00633E.

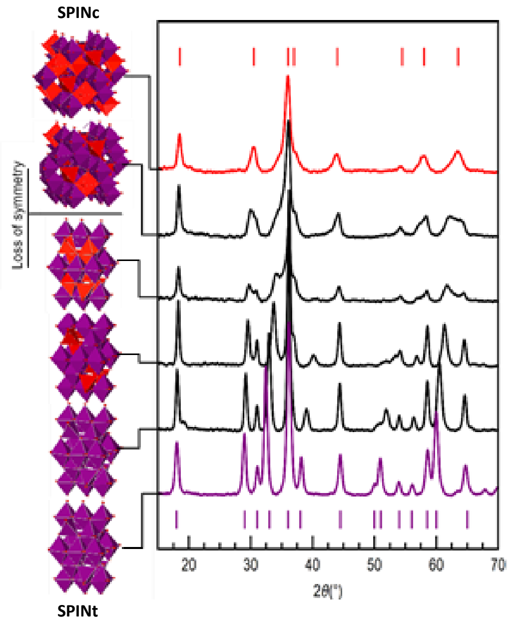
Gas-Diffusion Electrocrystallization (GDEx)
NMC recycling

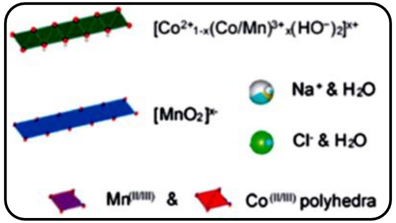
Batteries

Libraries of nanostructures:
precise control of phases, stoichiometries, particle size/distribution, crystallite size

O_2 as the gas feed – No additives







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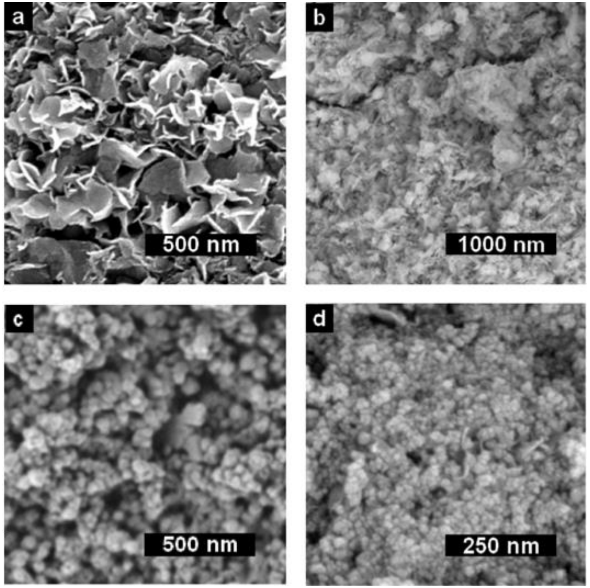
Gas-Diffusion Electrocrystallization (GDEx)

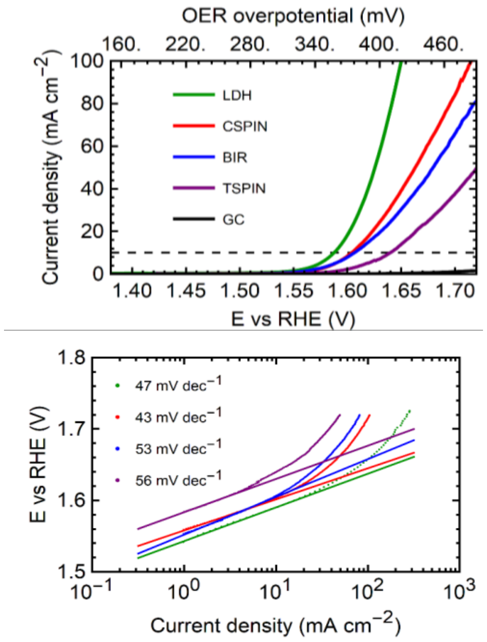
NMC recycling

O_2 as the gas feed – No additives



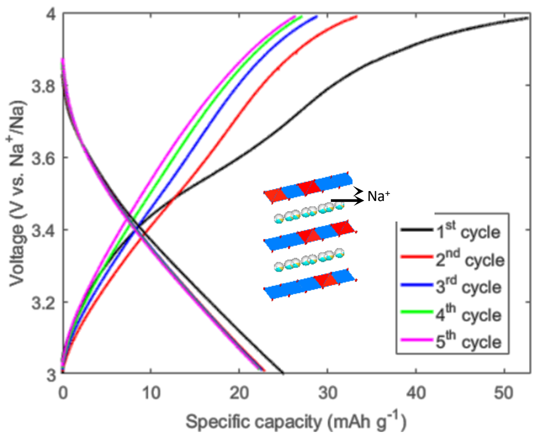
Functionality

Electrocatalysis (O_2 evolution)



Batteries

Na-ion batteries



- First 5 cycles, BIR $Co_{0.25}Na_{0.75}MnO_2$
- I_{appl} = 0.004 mA cm^{-2} (0.1C)
- Cut-off potentials between 3 V and 4 V
- First charge specific capacity: 53 mA h g^{-1}
- Reversible capacity 25 mA h g^{-1}

Prato et al. (2020) J Mat Chem A doi: 10.1039/D0TA00633E.

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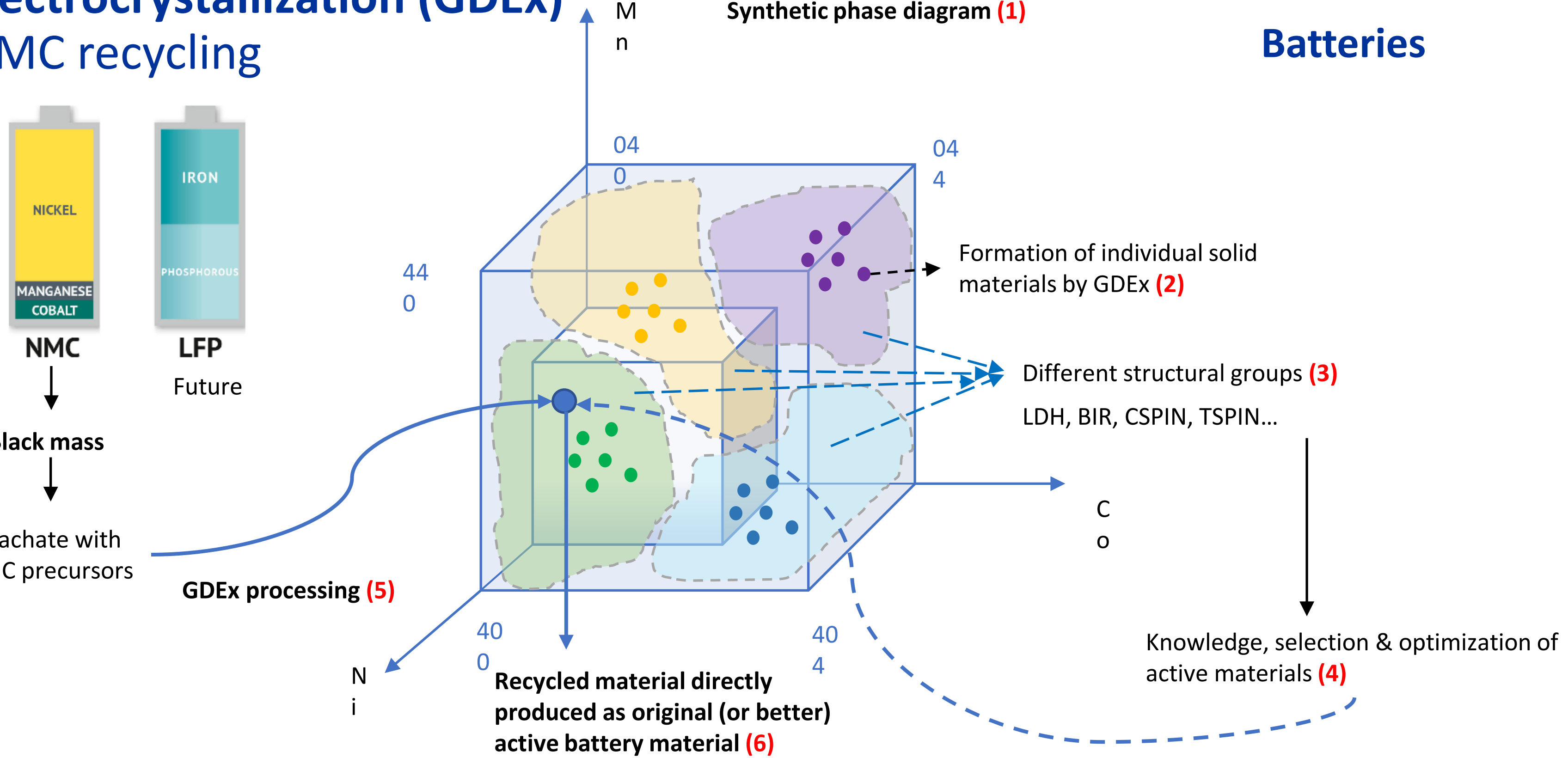
Gas-Diffusion Electrocrystallization (GDEx)

NMC recycling

Batteries



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Gas-Diffusion Electrocrystallization (GDEx)
NMC recycling

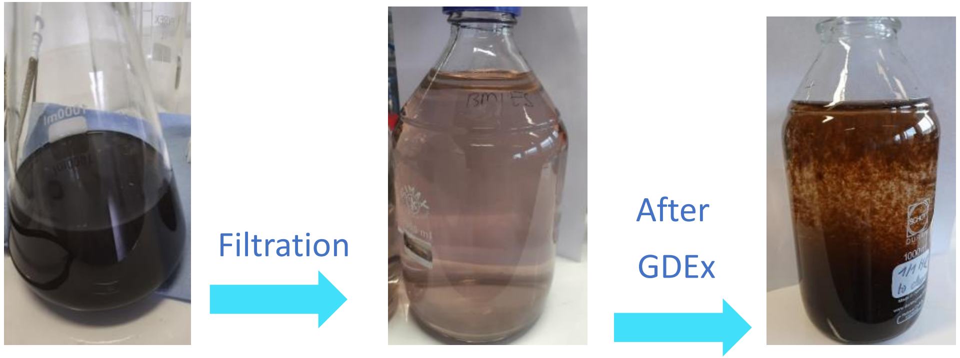
Batteries

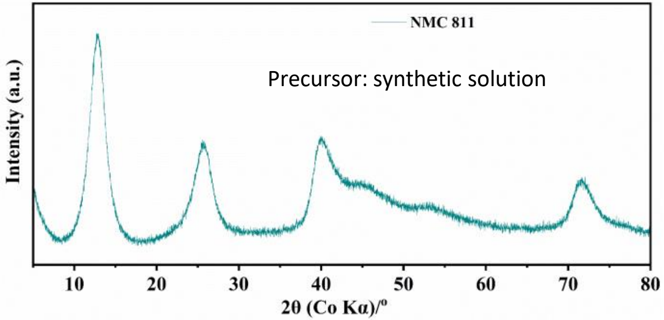
Synthetic phase diagram (1)

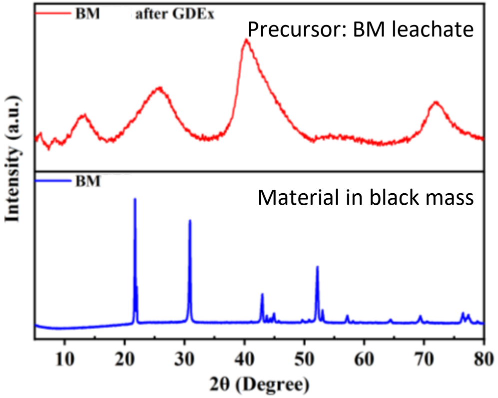
NMC 811 Precursor – Solid product Stoichiometry

Metal	Co	Mn	Ni
Concentration (mg/kg)	55184	60590	392474
Molecular weight	58.933	54.938	58.693
Moles	0.9	1.1	6.7
Molar ratio	0.1	0.1	0.8

BM leachate







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Conclusion
Gas-Diffusion Electrocrystallization (GDEx)

We cannot have sustainable batteries with unsustainable practices.

Electrochemistry is not only the future.

It is the solution.

- GDEx is a robust and versatile electrochemical technology
- It enables the effective, sustainable, and economically viable metal recovery from low-grade primary, waste, spent and off-specification materials
- Transforming them directly into functional materials for electrochemical energy applications and more.

Gas-diffusion electrocrystallization (GDEx)

Xochitl Dominguez-Benetton

Funded by the European Union under Grant Agreements No 101069685, 101069644 and 101058163. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the European Climate, Infrastructure and Environment Executive Agency (CINEA). Neither the European Union nor the granting authority can be held responsible for them.

METALLICO-Demonstration of battery metals recovery from primary and secondary resources through a sustainable processing methodology

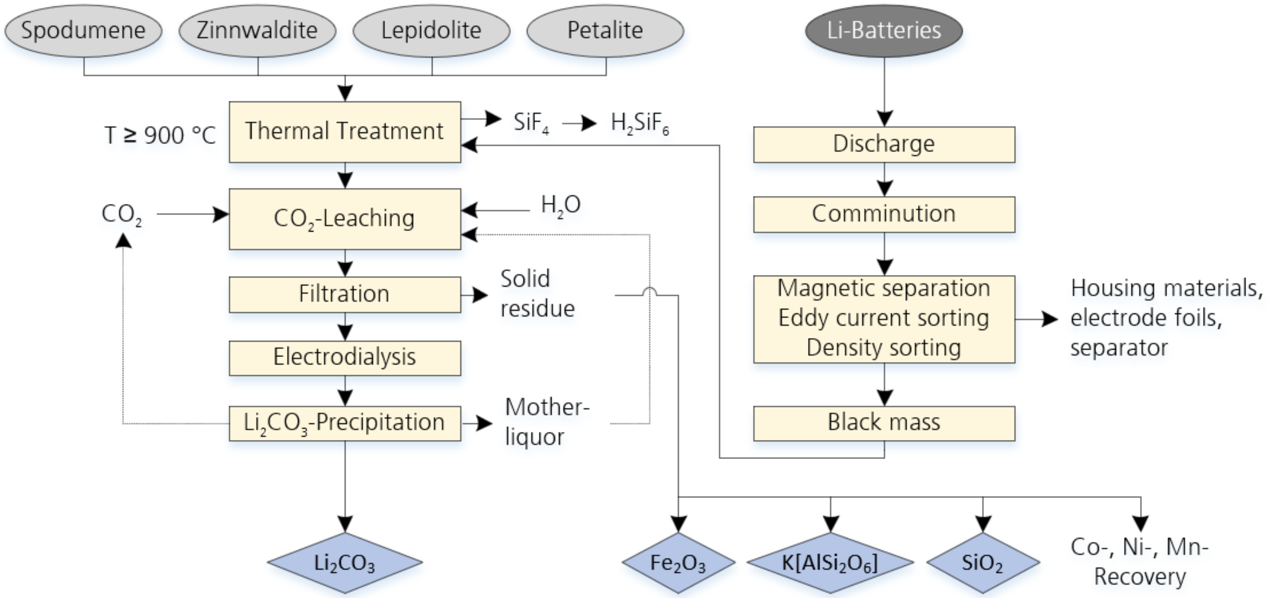
Cluster Hub Annual Event
Brussels, Belgium. 16 NOV 2023

Dr. Sandra Pavón, Alexander Nickol, Dr. Sebastian Hippmann, Prof. Dr. Alexander Michaelis

Fraunhofer Institute for Ceramic Technologies and Systems IKTS

Lithium Recycling

COOL-Process



Recycling in primary product quality

Recycling of the residual materials

Holistic recycling of primary and secondary raw materials

M. Bertau, G. Martin, C. Pätzold, DE 1020152217590; M. Bertau, G. Martin, DE102016204360.9; H.-G. Jäckel,, U.A. Peuker, L. Wuschke, *Chem. Ing. Tech*. **2014**, *86*, 806-813; G. Martin, C. Pätzold, M. Bertau, *Int. J. Min. Process*. **2017**, *160*, 8-15; G. Martin, A. Schneider, W. Voigt, M. Bertau, *Min. Eng.* **2017**, *110*, 75-81; D. Kaiser, S. Pavón, M. Bertau, *Chem. Ing. Tech*. **2021**, *93*, 1833-1839; S. Pavón, D. Kaiser, M. Bertau, *Chem. Ing. Tech*. **2021**, *93*, 1840-1850; 30. S. Pavón, D. Kaiser, R. Mende, M. Bertau. *Metals* **2021**, *11*, 259.

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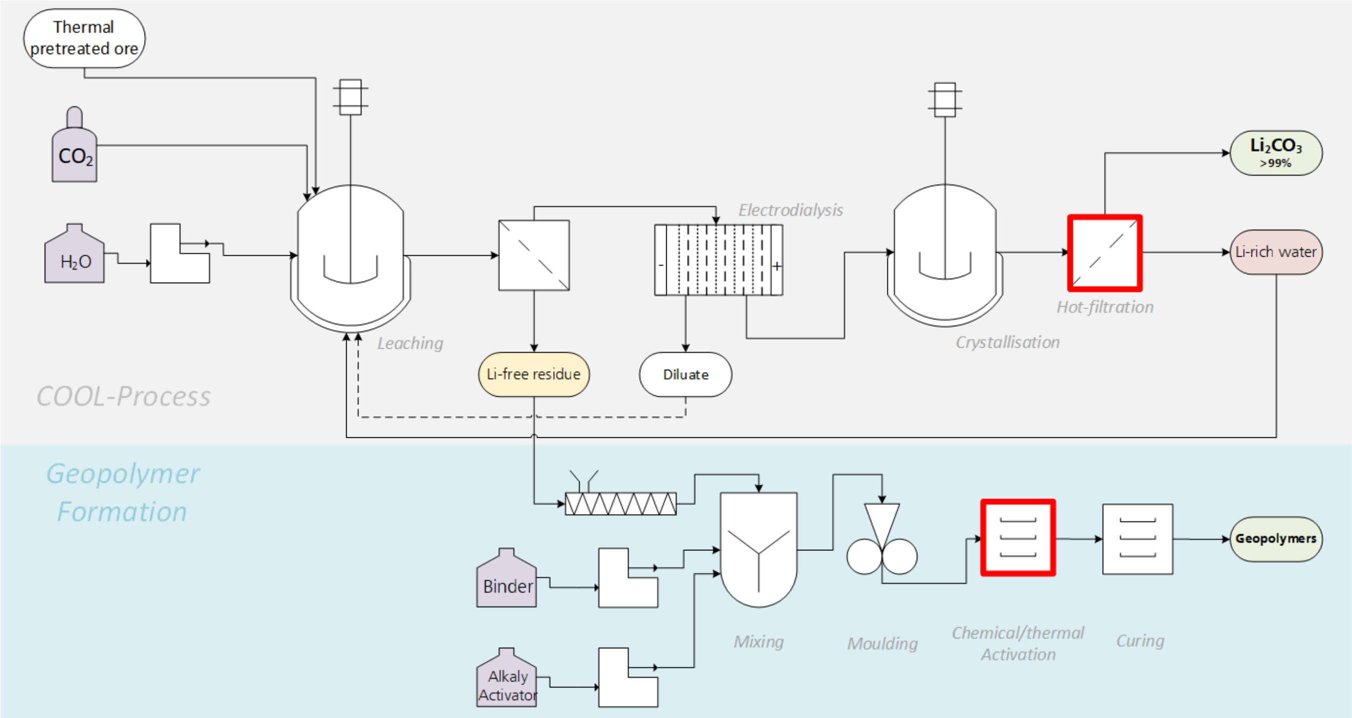
Recycling process validation

COOL+ Process



M. Bertau, G. Martin, C. Pätzold, DE 1020152217590; M. Bertau, G. Martin, DE102016204360.9; H.-G. Jäckel,, U.A. Peuker, L. Wuschke, *Chem. Ing. Tech*. **2014**, *86*, 806-813; G. Martin, C. Pätzold, M. Bertau, *Int. J. Min. Process*. **2017**, *160*, 8-15; G. Martin, A. Schneider, W. Voigt, M. Bertau, *Min. Eng.* **2017**, *110*, 75-81; D. Kaiser, S. Pavón, M. Bertau, *Chem. Ing. Tech*. **2021**, *93*, 1833-1839; S. Pavón, D. Kaiser, M. Bertau, *Chem. Ing. Tech*. **2021**, *93*, 1840-1850; 30. S. Pavón, D. Kaiser, R. Mende, M. Bertau. *Metals* **2021**, *11*, 259.

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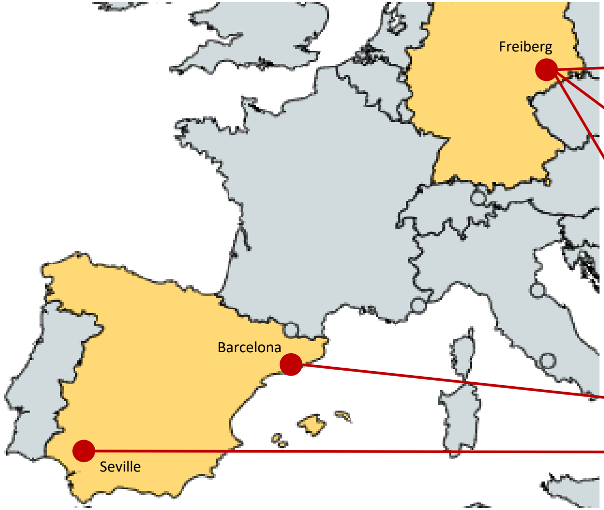
Lithium recovery from primary raw materials

Value chain

Partners involved



- TUBAF (TU Bergakademie Freiberg – Die Ressourcenuniversität. Seit 1765.): Lab-scale optimisation
- Fraunhofer IKTS: **Validation in pilot plant**
- G.E.O.S., idener (Scientific Computing): Simulation and modelling
- CETAQUA (Water Technology Centre), idener (Scientific Computing): Implementation; full scale-up

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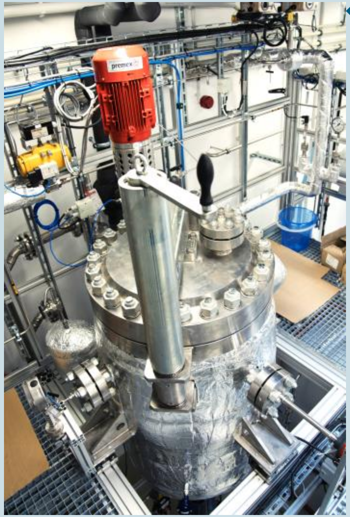
Challenges for cost-effective operation

COOL Process in TRL 5 possible?



200L- Autoclave reactor

Electrodialysis

Crystallisation/
Precipitation

- Decreasing **S:L ratio** for higher throughput and less wastewater production
- Connecting several reactors for quasi-continuous operation
  - Effective use of unconsumed CO_2
  - Reuse of mother liquor

G. Martin, PhD thesis, TU Bergakademie Freiberg, Freiberg, **2017;** M. Bertau, G. Martin, C. Pätzold, DE102015221759A1, **2017**.

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Challenges for cost-effective operation

COOL Process in TRL 5 possible?

COOL-Process parameters

- Thermal pre-treatment at T = 1100°C for 2 h
- Milling and sieving → Particle size ≤ 250 µm
- Digestion with *sc*-CO_2 at 230 °C; 100 bar; 2h; **S:L = 1:90 – 1:45**

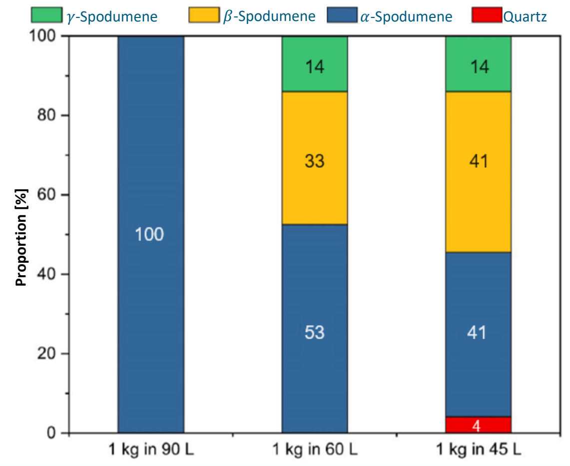
Challenge

- How to concentrate the Li-solution after leaching step?
    - Li-concentration in solution ≈ 10 mg/l (conductivity 0.3 mS cm^{-1})
    - About 500 L of the solution

Possible solutions

- Reverse osmosis (saturation?)
- Ion exchange resin (recharging?)

Composition of the leaching residue



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Thank you for your attention!

Dr. Eng. Sandra Pavón
Recycling & Green Battery

METALLICO

Funded by the European Union

The project has received funding from the European Union's Horizon Europe- the Framework Programme for Research and Innovation (2021-2027) under grant agreement no 101091682

Raw materials from geothermal fluids: Occurrence, enrichment, extraction

Katrin Kieling

GFZ Potsdam

"Materials for batteries" Cluster Annual Meeting I 19 October 2023

Funded by the European Union

CRM-geothermal: Project facts and challenge

- **The challenge**: Large amounts of Critical Raw Materials (CRM) are needed for the energy and digital transition

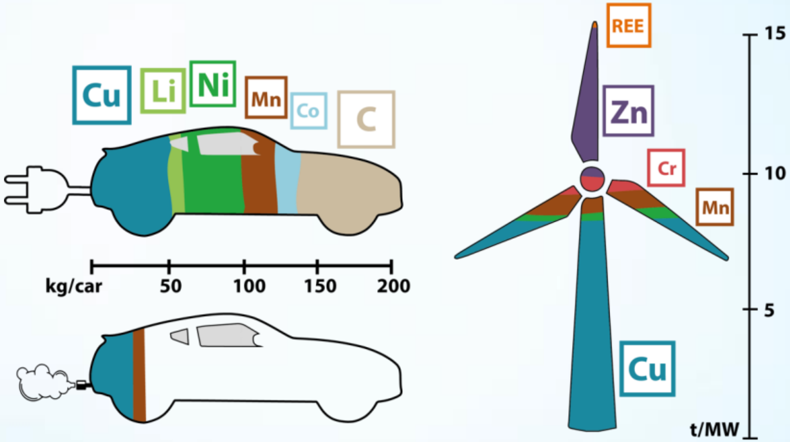

Illustration of the increasing need for critical materials that comes with the transition to renewable energies and the transition to electric cars. Data from https://www.iea.org/reports/the-role-of-critical-minerals-in-clean-energy-transitions. (Source: Ch. Kusebauch, GFZ)

CRM-geothermal: Project facts and challenge

- **The challenge**: Large amounts of Critical Raw Materials (CRM) are needed for the energy and digital transition

Lithium extraction at the Insheim geothermal power plant

Lithium Made in Germany from Thermal Water

Source: Energy & Management Powernews, July 28, 2020

The important battery raw material lithium could possibly be extracted in Germany. At the Insheim geothermal power plant, the extrac thermal water is being tested.

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By Kira Taylor | EURACTIV Est. 5min 30. Aug. 2021 (updated: 1. Sept. 2021)

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CRM-geothermal: Project facts and challenge

- **The challenge**: Large amounts of Critical Raw Materials (CRM) are needed for the energy and digital transition
- **CRM-geothermal solution**: Extraction of Critical Raw Materials from geothermal fluids


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CRM-geothermal: Project facts and challenge

- **The challenge**: Large amounts of Critical Raw Materials (CRM) are needed for the energy and digital transition
- **CRM-geothermal solution**: Extraction of Critical Raw Materials from geothermal fluids
- **Horizon Europe** call „HORIZON-CL4-2021-RESILIENCE-01-06 - Innovation for responsible EU sourcing of primary raw materials"
- **~7,9 Mio € funding** (EU, UK and Switzerland)
- Project lifetime: **May 2022 – April 2026**


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Concept

CRM-geothermal proposes to combine the **extraction of heat/electricity** and the **extraction of valuable elements from geothermal fluids**.

Advantages are:

- ✓ Maximising returns on investment
- ✓ Avoiding additional land use
- ✓ Minimising environmental impact of mining.

Objectives

- Establish an **overview of the potential for raw materials** in geothermal fluids,
- **Determine the source of selected CRM**, their mobility and potential for sustained extraction from geothermal brines;
- Develop and optimise **innovative extraction technologies** for selected CRM;
- Assess the **environmental-social-economic viability** and foster ethical sourcing of CRM;
- **Demonstrate at a pilot site** the extraction technology at the scale of a mini-plant and evaluate the system's sustainability

Expected Impacts

- Combined extraction has the potential to **cover a significant percentage of current and future needs of certain CRM** to the EU
- A more **resilient and domestic CRM supply chain** for the EU by reducing imports exposed to market and political risks
- More **trustworthy and ethical supply chains** for certain CRM
- Helping to **bridge the gap between societal resistance to raw materials extraction and the increasing demand for raw materials**
- Greater number of **viable geothermal projects**

Source: Cornish Lithium Ltd

Thank you for your attention!

Katrin.Kieling@gfz-potsdam.de

https://crm-geothermal.eu

crm-geothermal @CrmGeothermal

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