Evento finale Tech4Lib: Batterie e Materie Prime Critiche - Sfide e opportunità dell'economia circolare

Novel Approaches for Critical Raw Materials Recovery: Advances in Urban Mining and Lithium Battery Recycling

Daniele Montini*, Elisa Galli, Irene Vassalini, Ivano Alessandri

*SusCheMat Lab and INSTM, Università degli Studi di Brescia

daniele.montini@unibs.it

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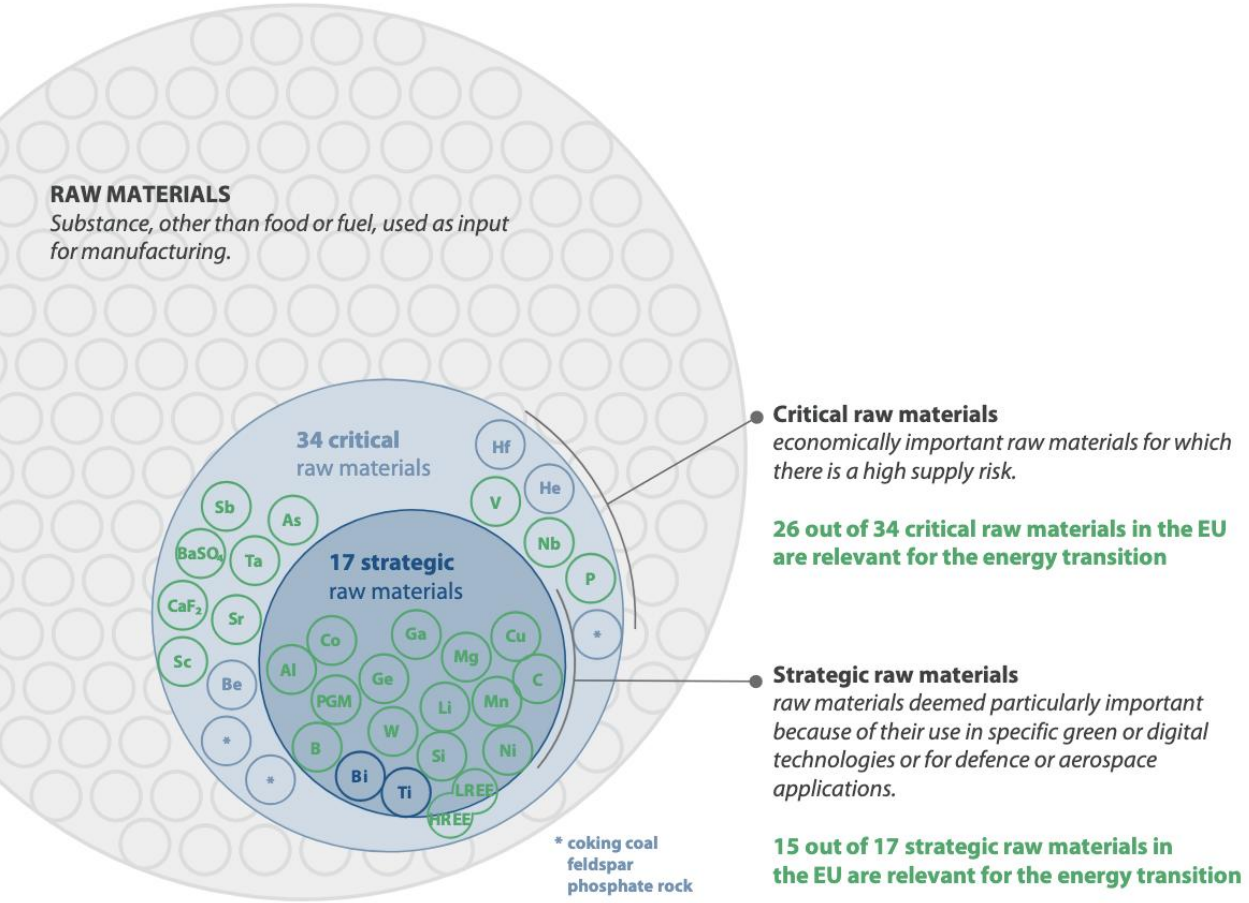
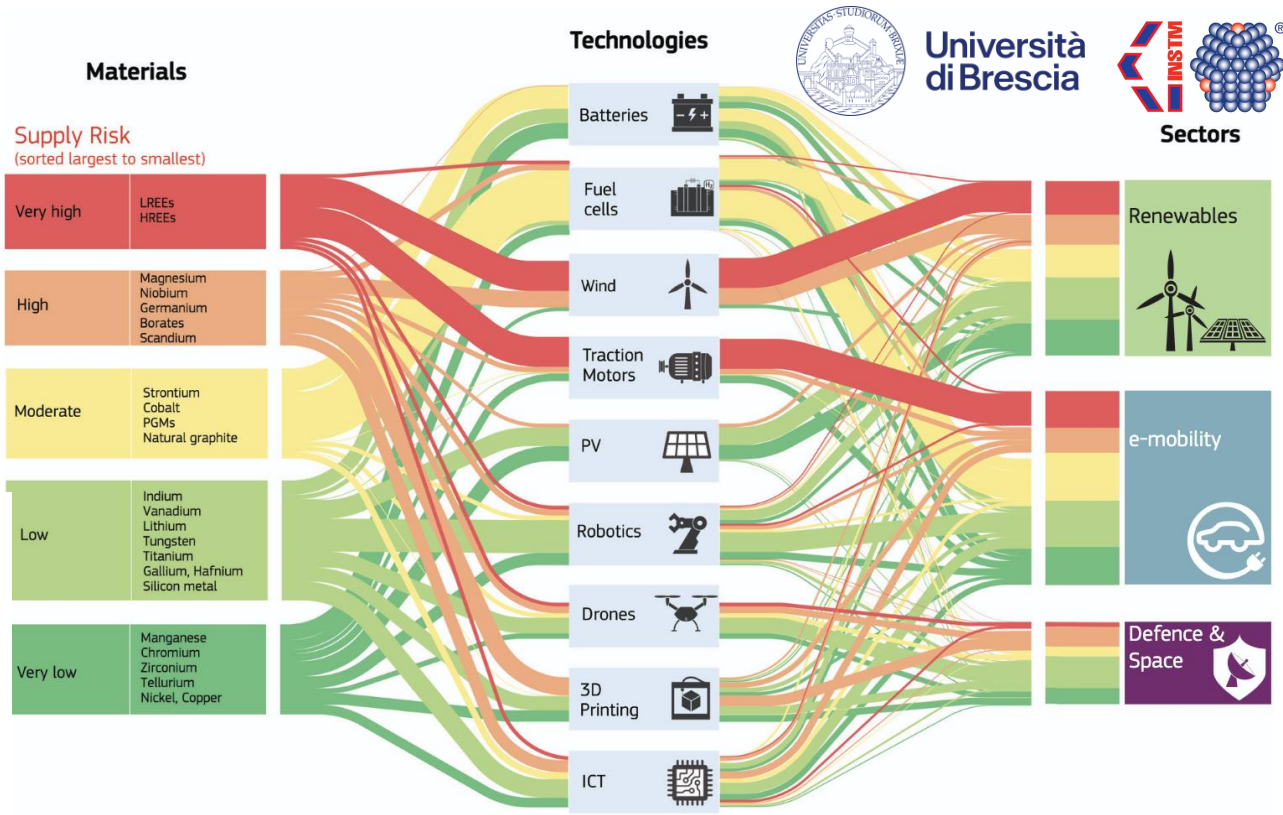
Critical Raw Materials

Economically important resources with a high risk of **supply** disruption that are **essential** for the functioning and integrity of many **industrial ecosystems**.



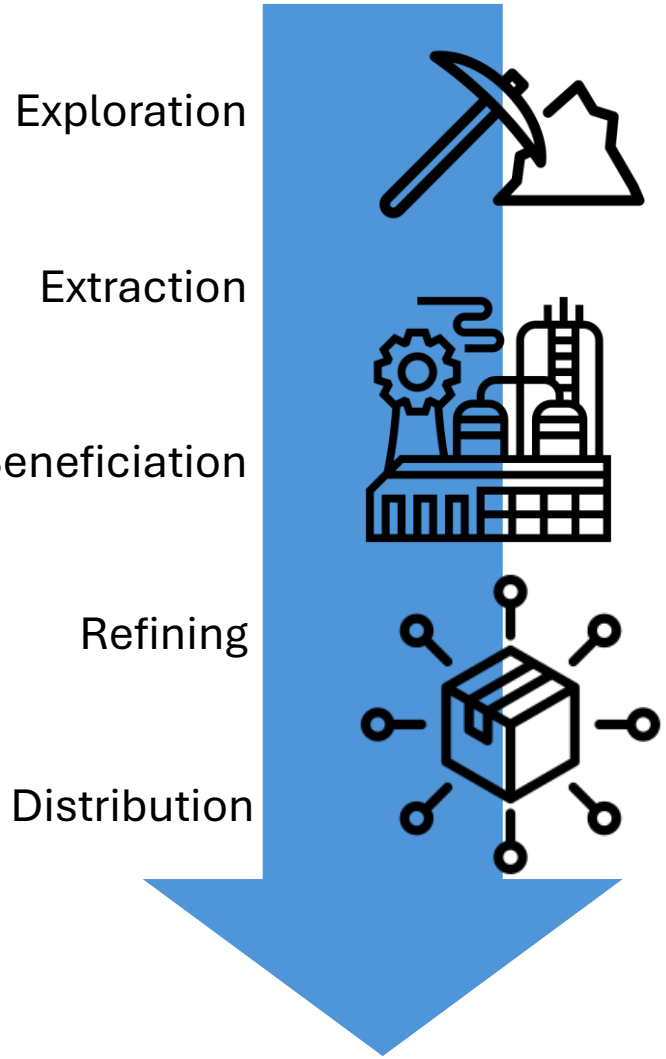


2023 CRMs vs. 2020 CRMs			
aluminium/bauxite	gallium	phosphate rock	vanadium
antimony	germanium	phosphorus	arsenic
baryte	hafnium	PGM	feldspar
beryllium	HREE	scandium	helium
bismuth	lithium	silicon metal	manganese
borate	LREE	strontium	copper
cobalt	magnesium	tantalum	nickel
coking coal	natural graphite	titanium metal	~~indium~~
fluorspar	niobium	tungsten	~~natural rubber~~

Legend:
Black: CRMs in 2023 and 2020
Red: CRMs in 2023, non-CRMs in 2020
~~Strike:~~ Non-CRMs in 2023 that were critical in 2020

European Commission

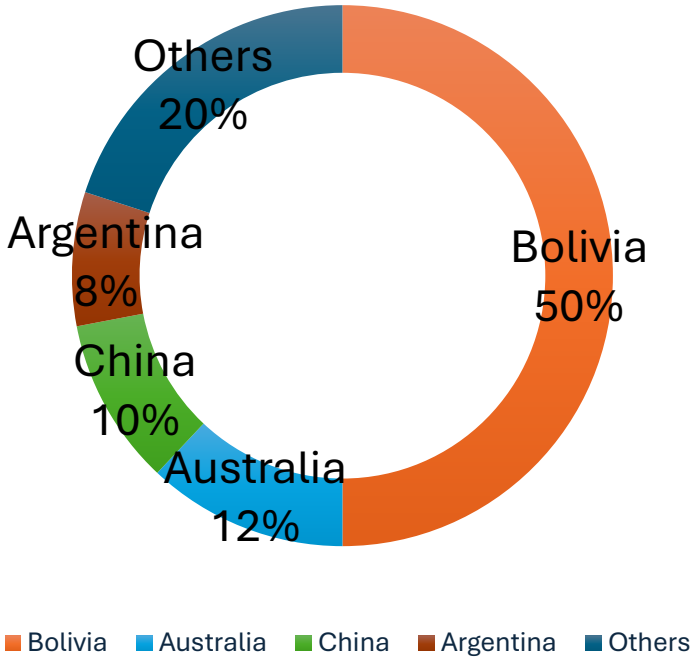
CRMs Supply chain



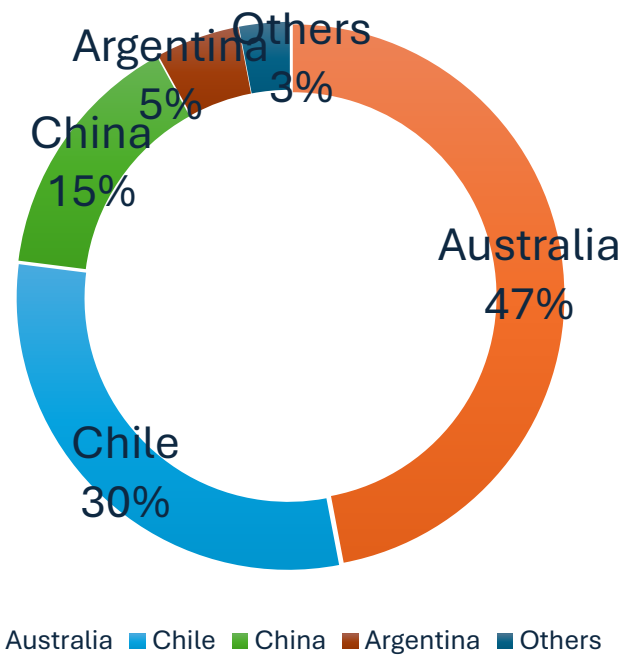
Production -> Distribution -> Use ->
Waste treatment -> Land filling

3 Li Lithium 6.941	28 Ni Nickel 58.693	27 Co Cobalt 58.933	25 Mn Manganese 54.938
EU Reserves	**1.2%**	**4.2%**	**4.4%**	**0%**
EU Primary prod.	0.1%	1.5%	0.7%	0%

Lithium Primary Producers



Lithium Refinery Producers



European Critical Raw Materials Act

Regulation that entered into force in **2024** to secure a reliable, sustainable, and diversified supply of critical minerals

4 main **2030** benchmarks for strategic raw materials:

EU EXTRACTION

At least 10% of the EU's annual consumption from EU extraction

EU PROCESSING

At least 40% of the EU's annual consumption from EU processing

EU RECYCLING

At least 25% of the EU's annual consumption from EU recycling

EXTERNAL SOURCES

Not more than 65% EU's annual consumption of each strategic raw material at any relevant stage of processing from single third country

Urban Mining: a new resource

Recovery of valuable materials from end-of-life products and industrial waste streams.

Hidden Stock of Critical Raw Materials:

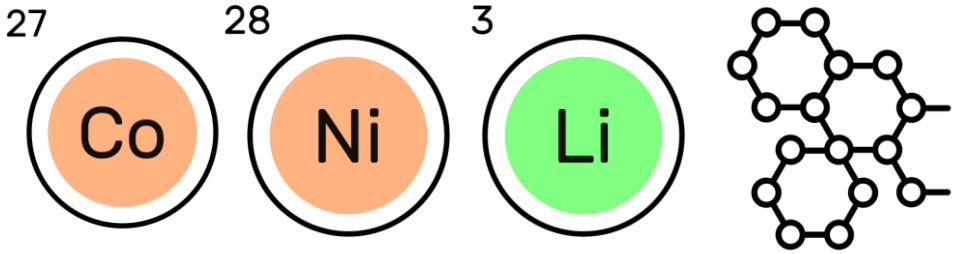
European Commission
@EU_Commission

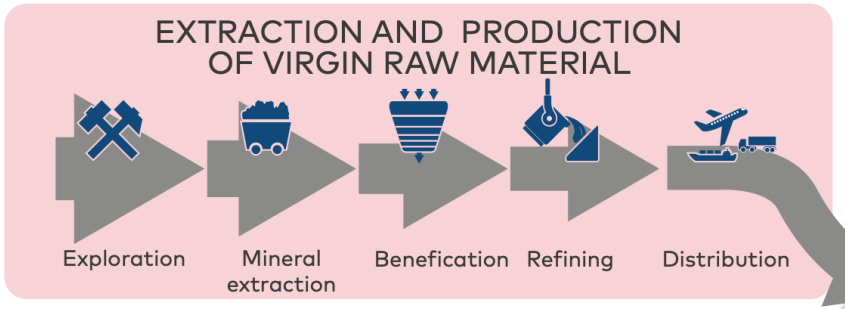
There are some 700 million unused phones in the EU.

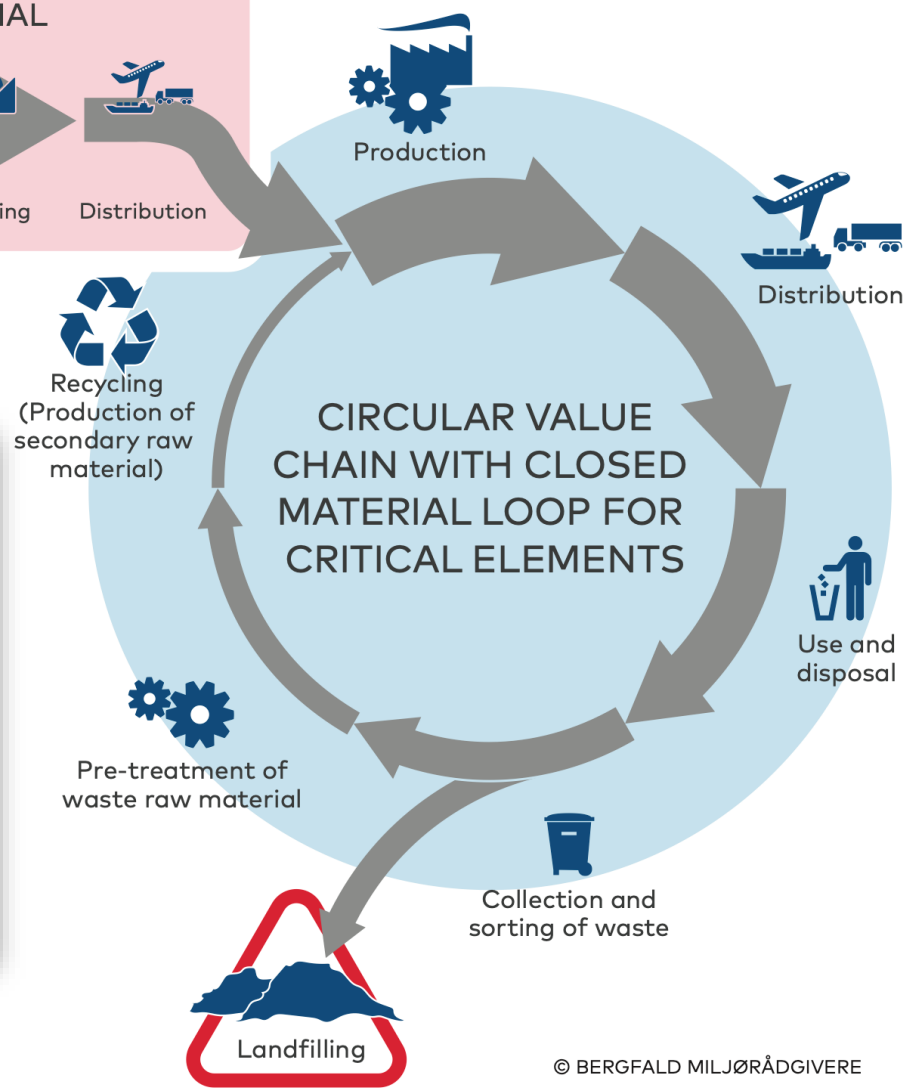
If we recycle them, we can:

- Reuse lithium, cobalt, and rare earth elements
- Reduce our reliance on imports
- Increase our resilience against global market disruptions

link.europa.eu/3MJKR4







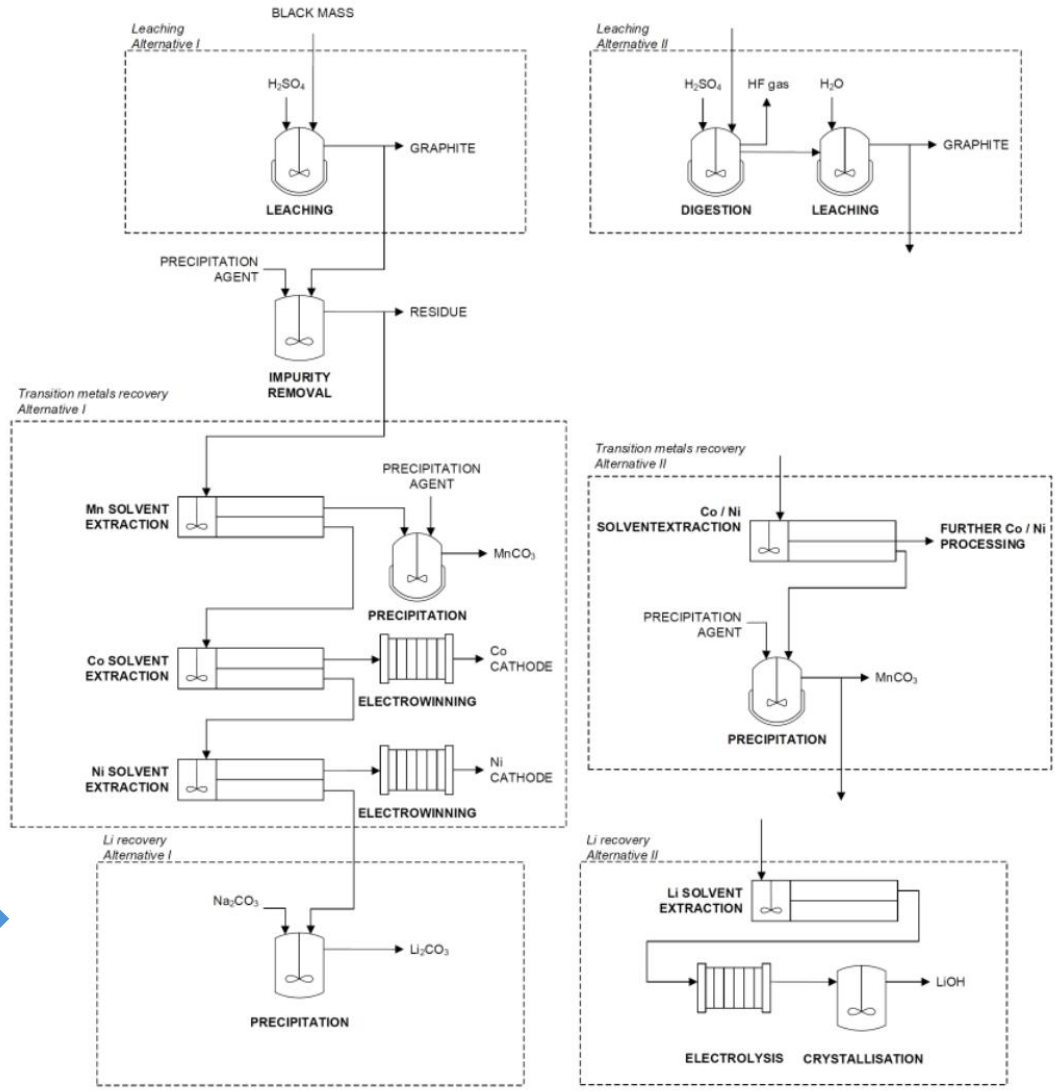
Metal recovery from waste stream

Pyrometallurgy

- Robust
- Hazardous gas emission
- Slag production
- Lithium losses

Hydrometallurgy

- Good yields
- Rely on strong hazardous acids

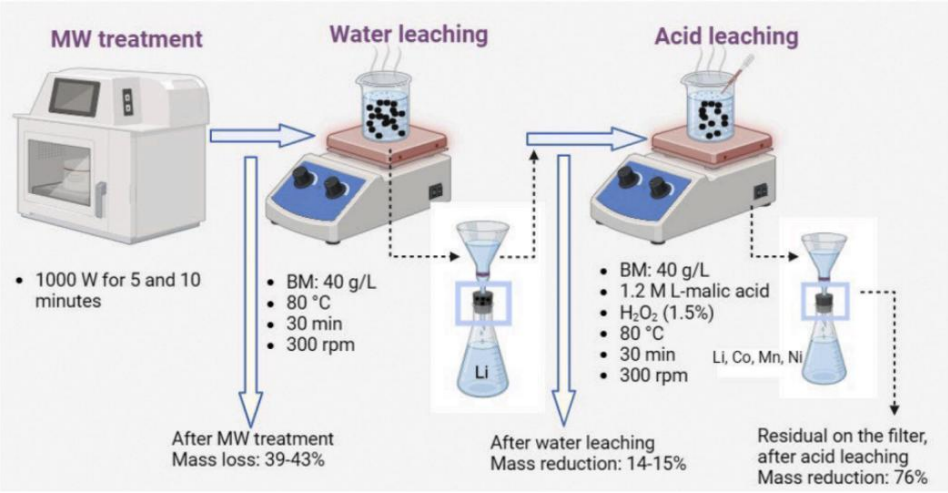


Brückner et al. 2020; Haga et al. 2019; Hanisch et al. 2019; Lipp, 2017

Tech4Lib Project

Tech4Lib
Spent lithium-ion battery recovery!

Foundend by: Fondazione CARIPLO



The Approach:

Establish circular economy approach for the raw materials necessary for LIBs production

The Solution:

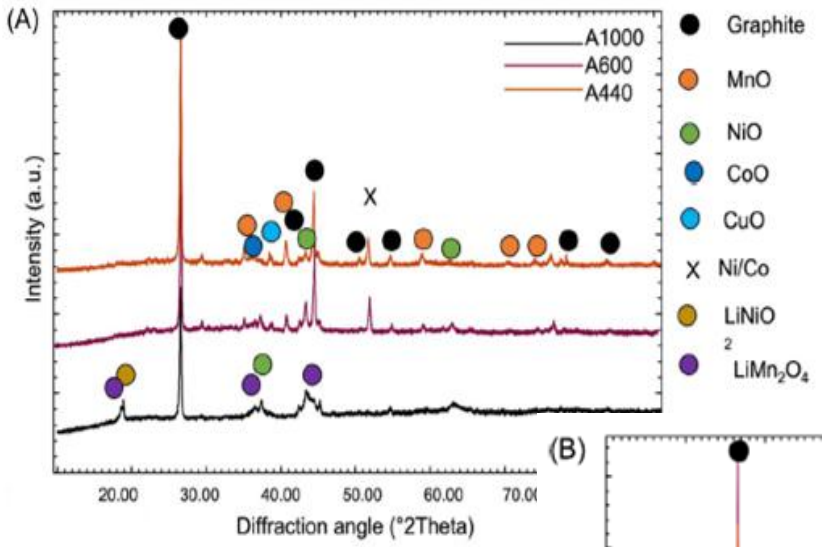
Obtain carbothermic reactions at lower energies by a new microwave technology

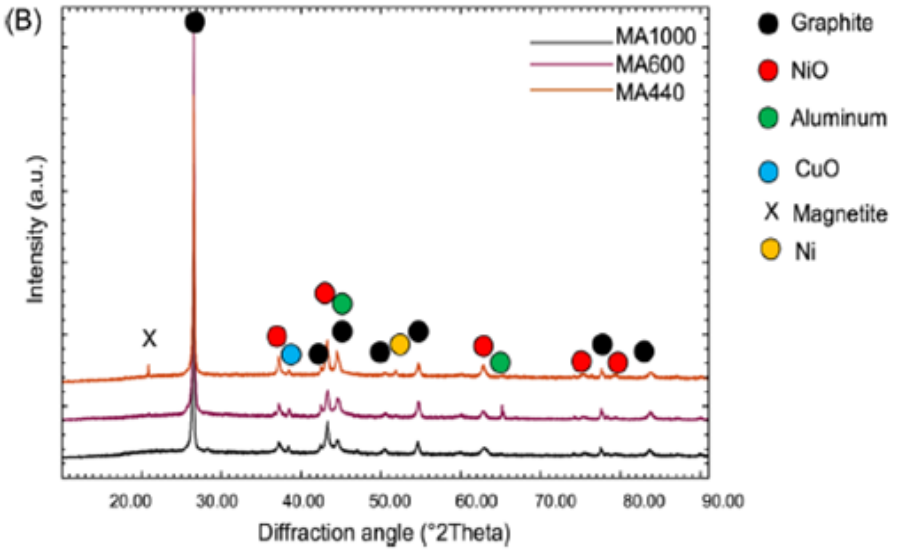
Extract Li by using only a water solution

Extract other metals (Co, Mn, Ni, Cu) by combining leachate streams obtained by **food waste**

wt (%)	Al	Co	Cu	Fe	Mn	Ni	Li
BM	5.4±0.1	5.2±0.2	5.3±0.1	0.7±0.1	5.1±0.1	8.5±0.2	2.2±0.1
BM < 0.5 mm	2.9±0.1	5.7±0.2	2.7±0.1	0.8±0.1	6.1±0.1	9.9±0.2	2.6±0.1

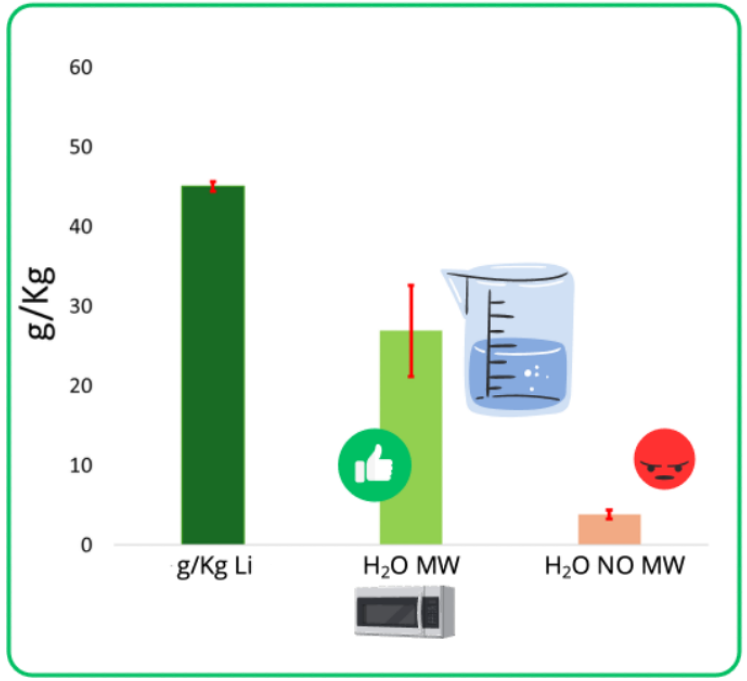
- **Ni**, **Co**, and **Mn** are the main constituents of the LIB cathodes;
- Li is around 2.6%;
- Fe origins from battery casing contamination.



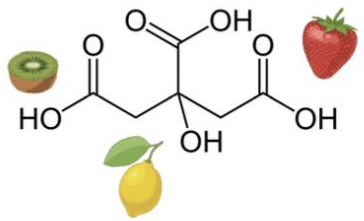


At 440 W and 600 W no peaks attributed to LMO, LNO, and LCO, indicating that carbothermic reactions have caused the formation of corresponding metal oxides.

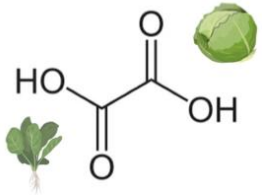
Extraction with water



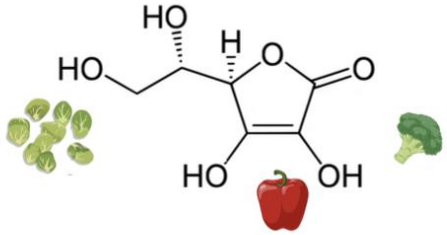
Selective Leaching with Organic Acids



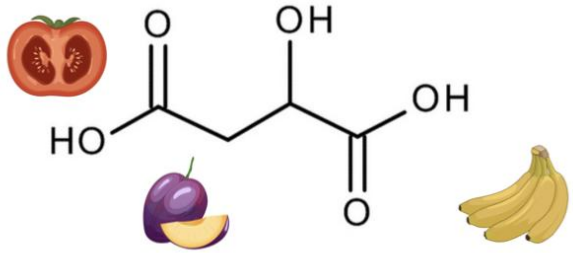
Acido citrico



Acido ossalico



Acido ascorbico



Acido malico

40 g/L, 80°C, 30 min,
300 rpm

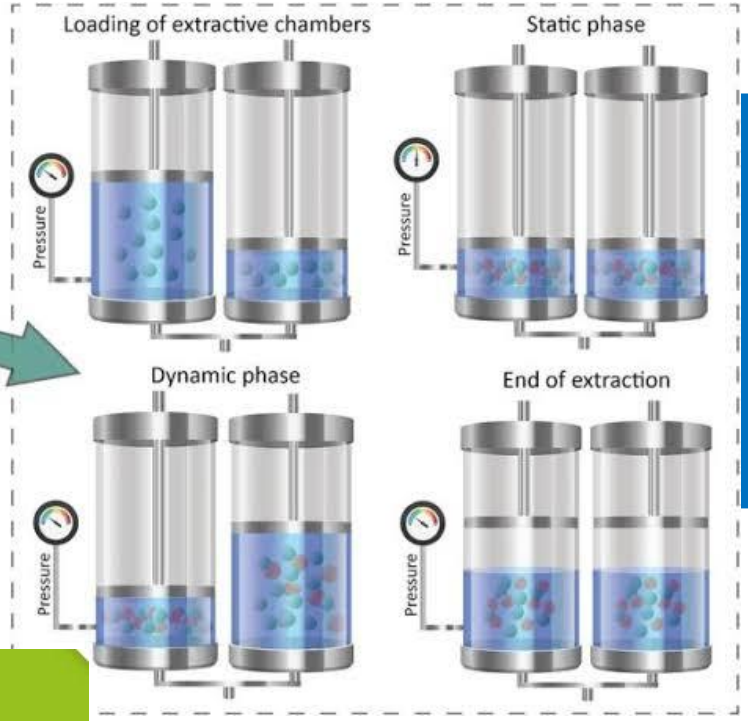
H_2O_2 (1.5%)

Selective Leaching with Organic Acids

Extraction from Food Waste

Food Waste

Naviglio Extractor



40 g/L, 80°C, 30 min,
300 rpm
1.2 M Acido Malico
H_2O_2 (1.5%)

- High efficiency enabled by two-phase extraction process
- Operates at room temperature
- Low energy demand

Selective Leaching with Organic Acids

Separation emerges from the interplay of coupled equilibria and reaction mechanisms

- Time
- Temperature
- Volume
- Stirring

•*Ligand acid–base equilibrium*

Oxalic acid: $pK_{a1} \approx 1.23$, $pK_{a2} \approx 4.19$
Malic acid: $pK_{a1} = 3.40$, $pK_{a2} = 5.11$
Citric acid: $pK_{a1} = 3.13$, $pK_{a2} = 4.76$, $pK_{a3} = 6.40$

•*Metal–ligand complexation equilibrium*

$M^{n+} + x\ L^{y-} \rightleftharpoons [ML_x]^{(n-xy)}$

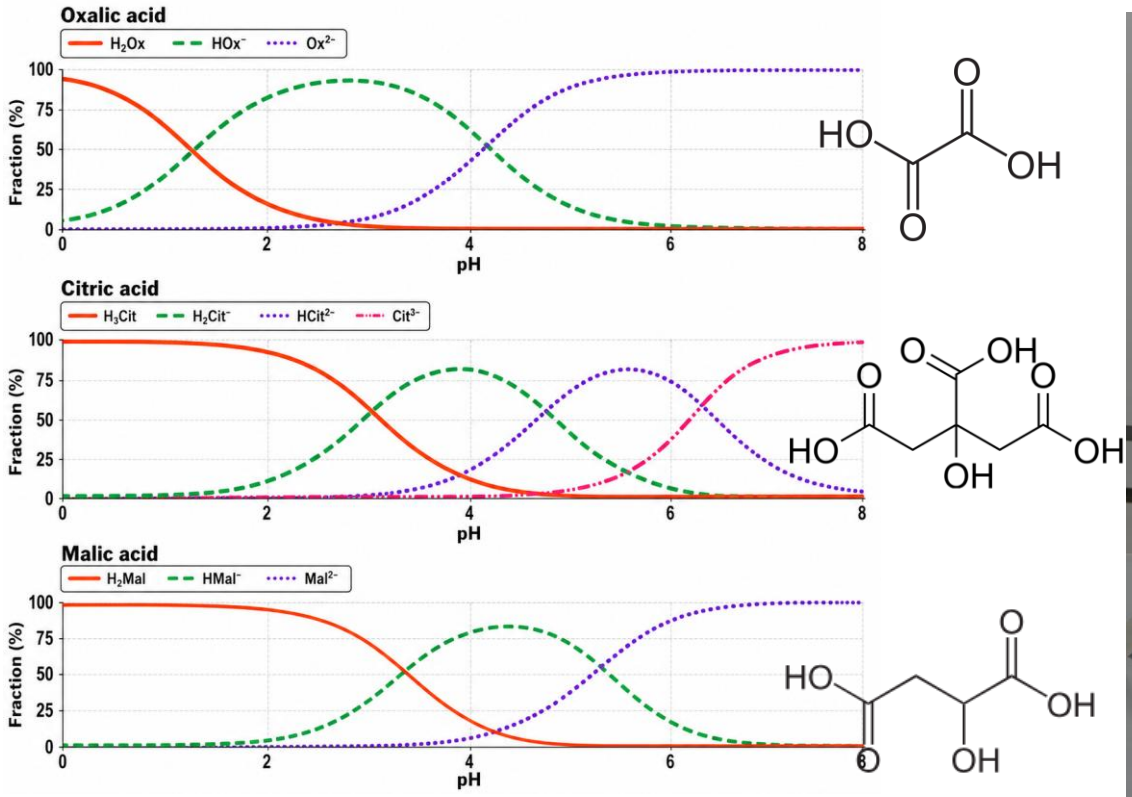
Stability follows the Irving–Williams series:

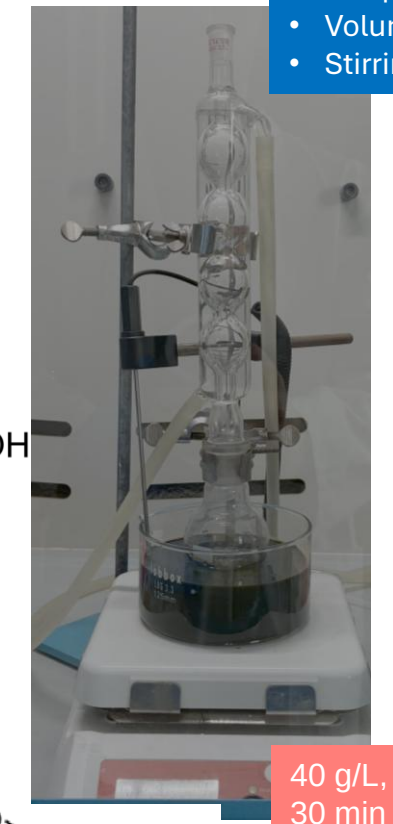
$Mn^{2+} < Fe^{2+} < Co^{2+} < Ni^{2+}$

•*Solubility equilibrium of the resulting salt*

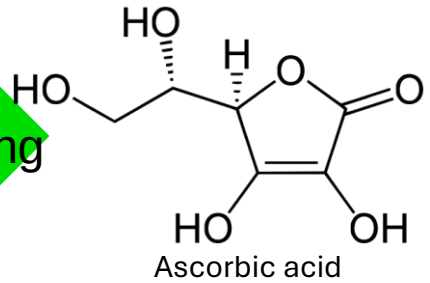
$M_2(C_2O_4)_n\ (s) \rightleftharpoons 2\ M^{n+} + n\ C_2O_4^{2-}$; $Ksp = [M^{n+}]^2[C_2O_4^{2-}]^n$

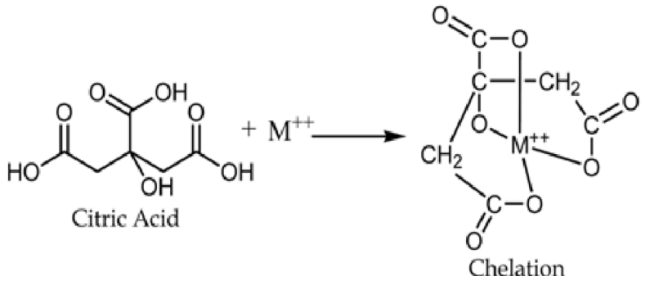
•Reduction of transition metals enhances the metal dissolution altering leaching selectivity



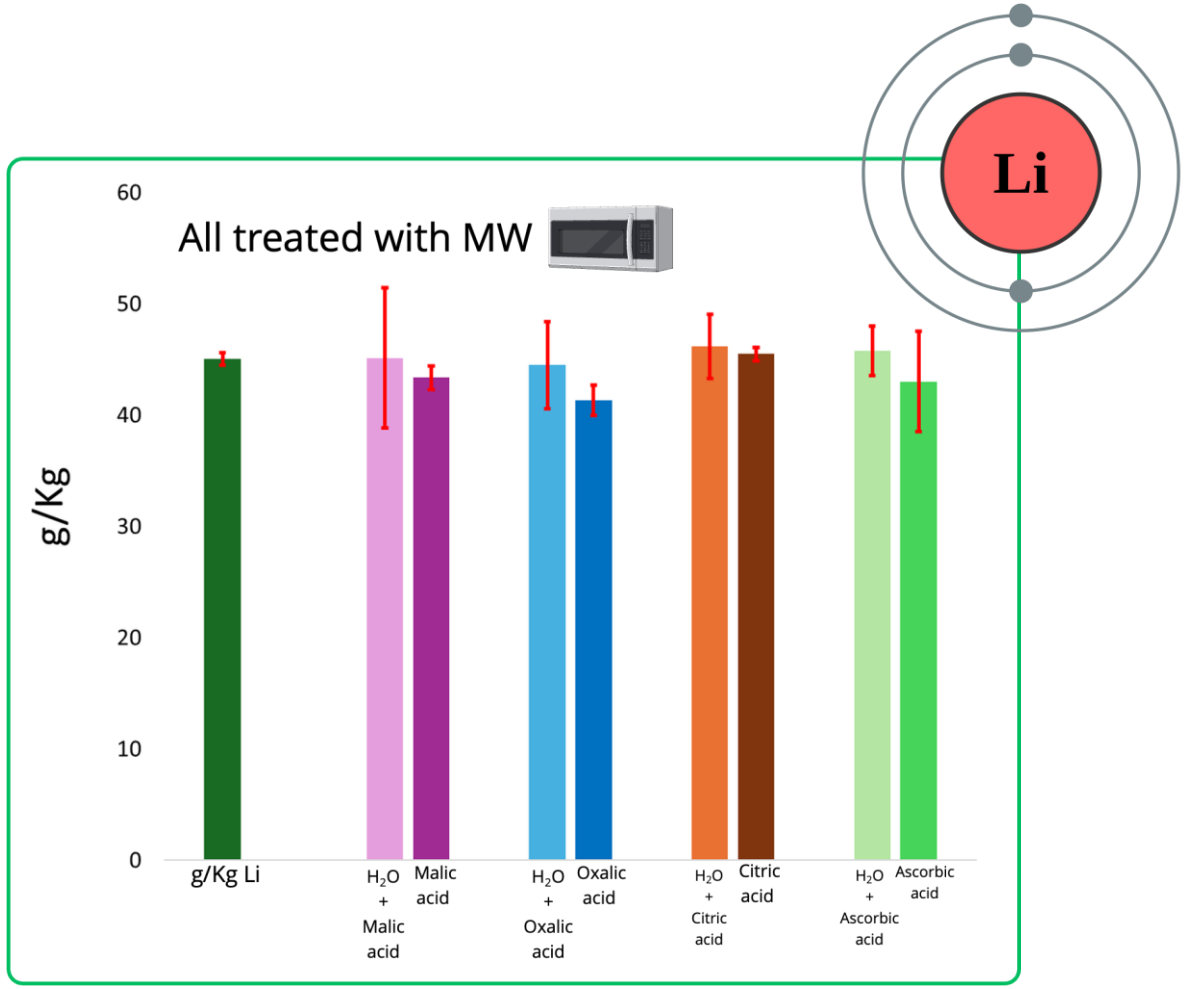


40 g/L, 80°C
30 min
300 rpm
H_2O_2 1,5 % v/v


Ascorbic acid

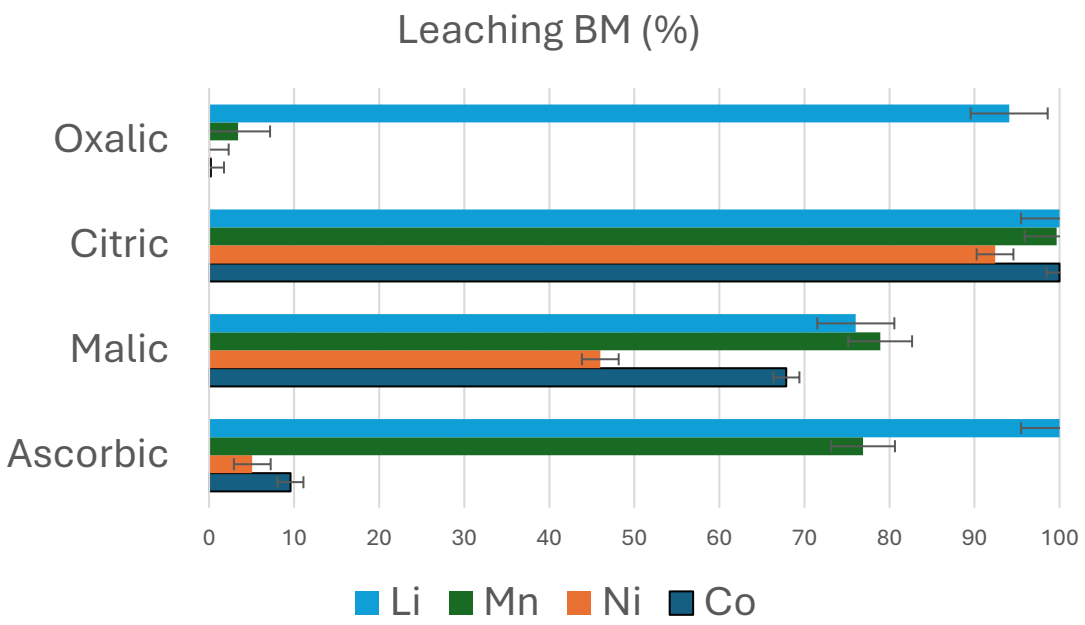


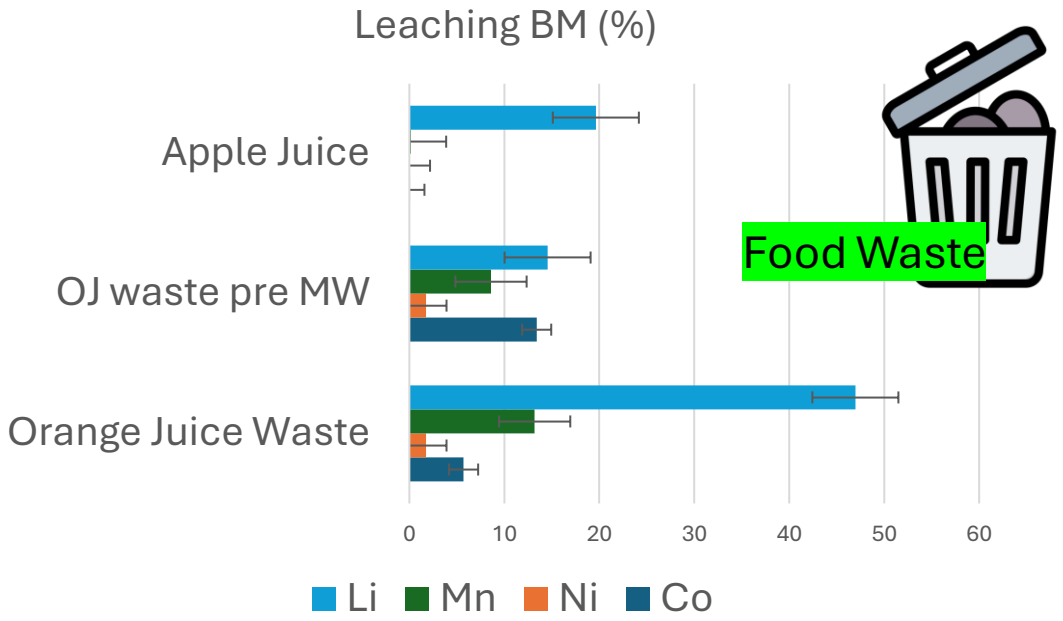
Selective Leaching with Organic Acids



Microwave-assisted extraction resulted in comparable Li recovery for all tested organic acids

Selective Leaching with Organic Acids





- **Citric acid**: Universal complexing agent. Good yields, but low selectivity.
- **Malic acid**: Lower affinity for nickel
- **Oxalic acid**: Forms poorly soluble oxalates with Mn, Ni, Co
- **Ascorbic acid**: Reducing agent with high efficiency for lithium and Mn.

- Li extraction efficiency is **improved** by MW treatment
- The effect of ascorbic acid appears to be the **dominant factor** in the extraction process.

Key Conclusions:

•Microwave processing achieves lithium recovery comparable to pyrometallurgical routes, with lower energy demand and shorter treatment times.

•Organic acid leaching represents a promising and sustainable strategy for lithium extraction from spent batteries.

•Further investigation of reaction mechanisms and process parameter interactions is needed to improve recovery efficiency and elemental speciation control.

Thank you for your attention!

SusCheMat Lab

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Spent lithium-ion battery recovery!

https://tech4lib.unibs.it/

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