Electrochemical Safety Research Institute (ESRI)

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NAATBatt 2025 | Extending Range
Member Update Presentations (Upstream): Session 3A-10
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Discoveries in Safety™

Introduction:

UL Research Institutes (ULRI) is committed to **Building Resilience for a Sustainable Future**. In line with this mission, the Electrochemical Safety Research Institute (ESRI) is focused on enhancing safety science by assessing recycling methods through fundamental research.

- To ensure safe lithium-ion batteries (LIB) end-of-life management
- To address transition metal shortages for sustainability
- To reduce environmental impact
- To conserve natural resources

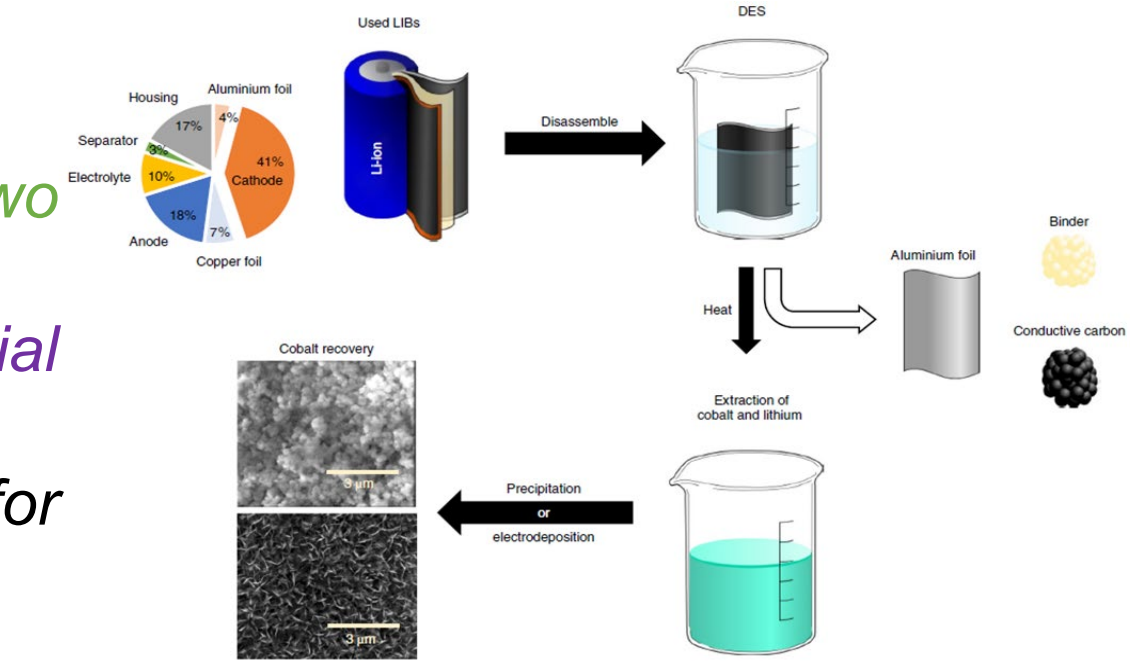
Research Objectives

ESRI collaborates with Professor Pulickel M. Ajayan at Rice University to enhance recycling processes, focusing on efficient and environmentally friendly methods for recovering cathode materials.

RICE — Team — UL Research Institutes | Electrochemical Safety

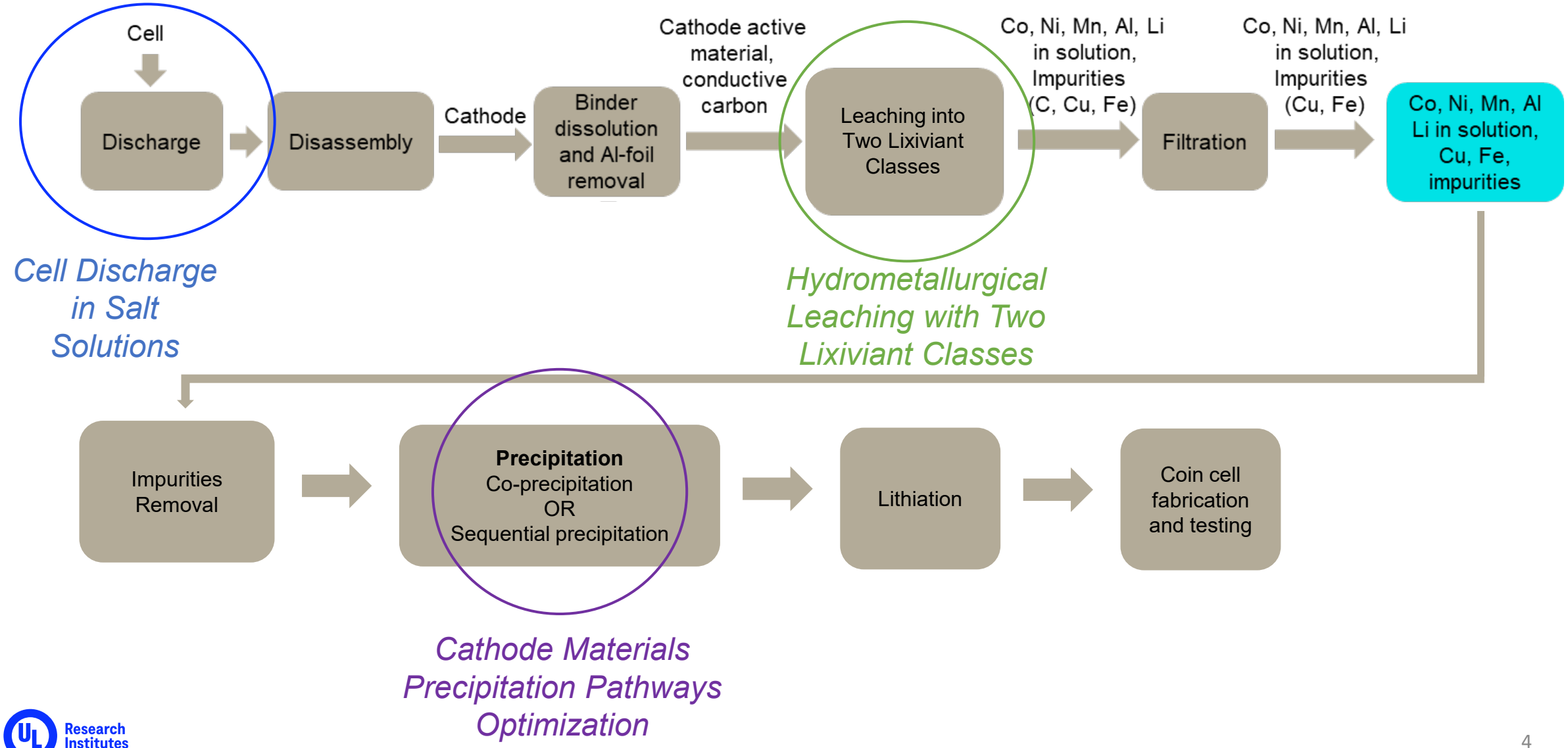
Approaches:

1. *Cell Discharge in Salt Solutions*
2. *Hydrometallurgical Leaching with Two Lixiviant Classes*
3. *Optimization of Cathode Material Precipitation Pathways*
4. *Microwave-Assisted Direct Methods for Recycling LIB Cathodes*



P.M. Ajayan et al., *Nat Energy* **2019**, 4, 339

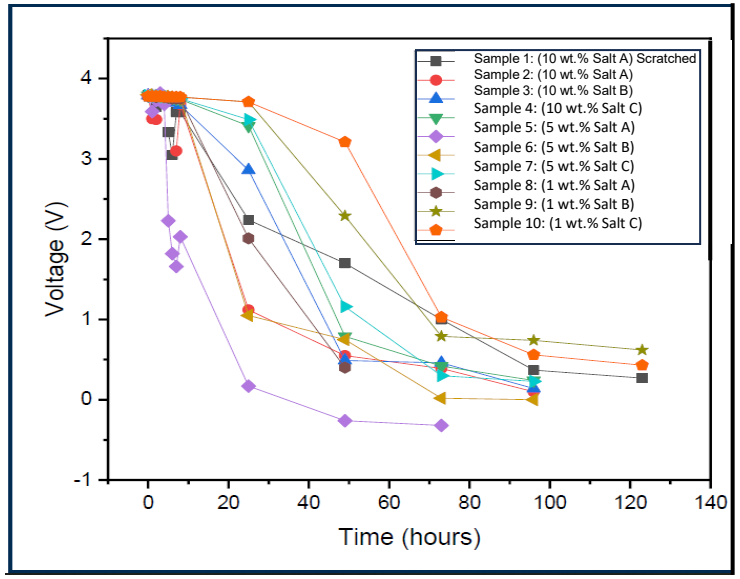
Research Flowchart:
Year 1 & Year 2: Hydrometallurgical Recycling

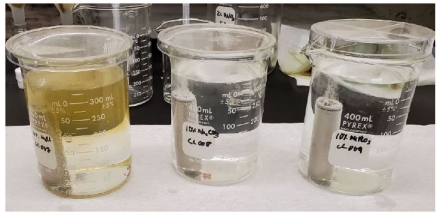


The Study of Cell Discharge in Salt Solutions

Pouch Cells

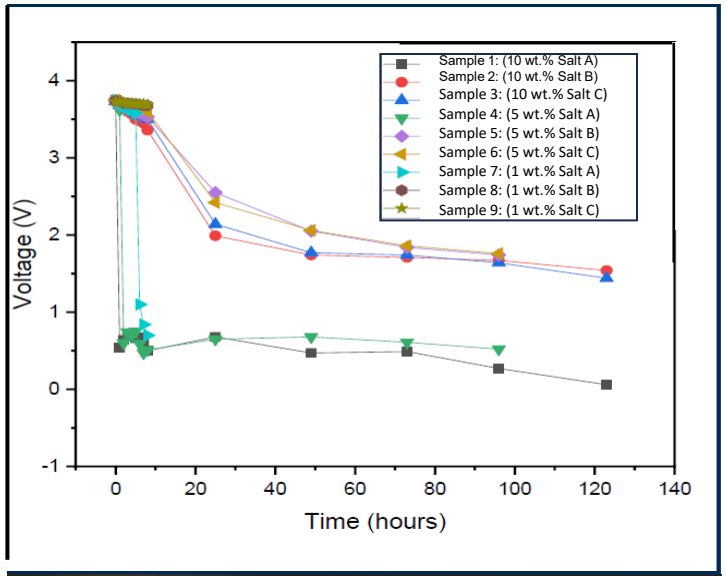
Pouch cell samples were discharged by immersing them in various salt solutions. The reaction was followed by measuring the drop in voltage over time. Only one sample with surface scratching. Results show that after 48h, all cells were discharged below 2.5V, except *Sample 10 (1 wt.% Salt C)* and the surface scratching does not speed up the discharge process.





Cylindrical Cells

Cylindrical cell samples were discharged by immersing them in various salt solutions. The reaction was followed by measuring the drop in voltage over time. Results show that in 8h, all cells immersed in *Salt A (1%, 5%, 10% wt.)* were discharged below 1V, and within 48h, all cells with *Salt B and C (1%, 5%, 10% wt.)* were below 2.5V.



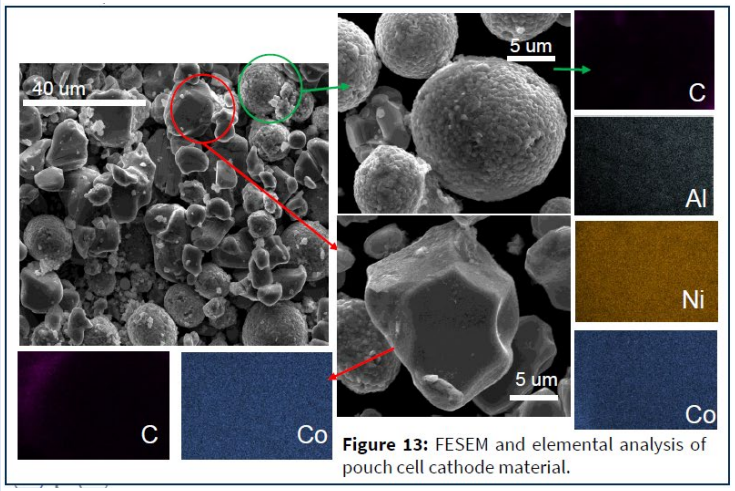


Figure 13: FESEM and elemental analysis of pouch cell cathode material.

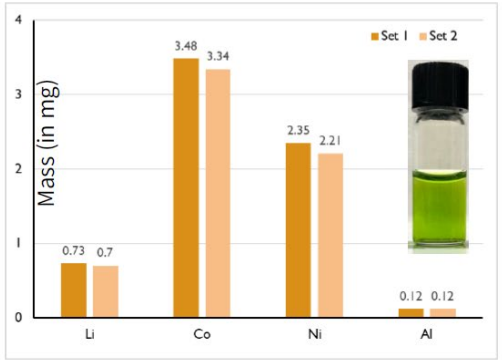
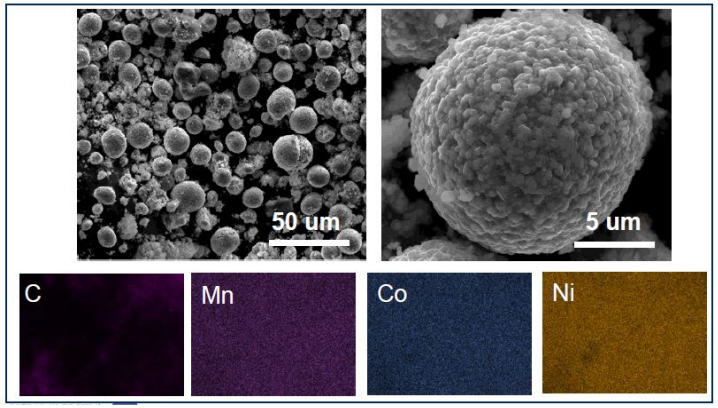


Figure 14: Mass of different elements determined from ICP-MS.

The pouch cell composition was determined to be a two types of cathode chemistries by Rietveld refinement of PXRD pattern, FESEM and ICP-OES → LCO and NCA



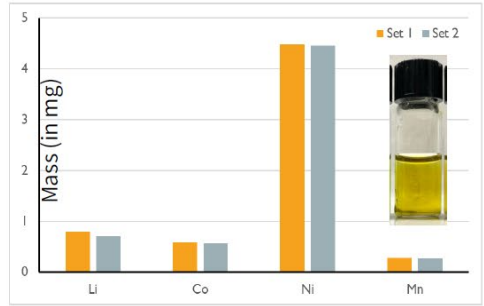
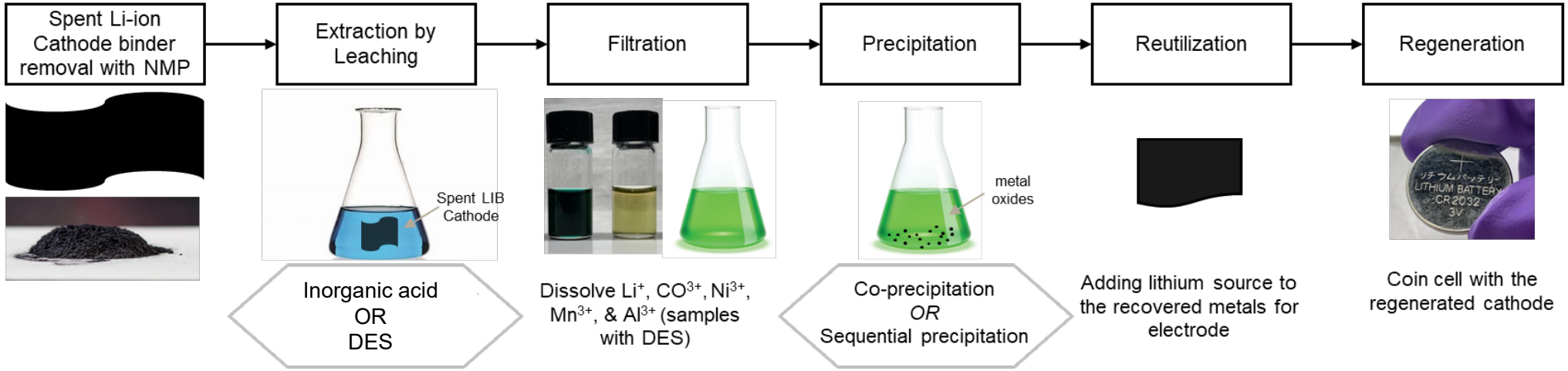


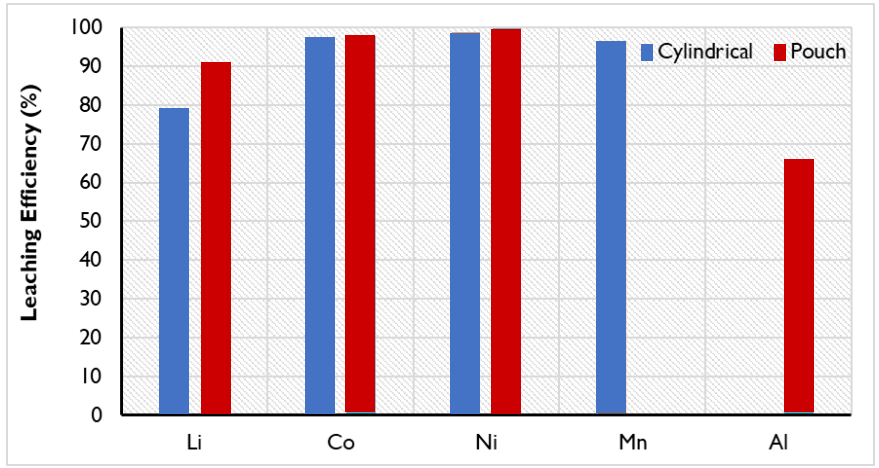
Figure 12: Mass of different elements determined from ICP-MS.

The pouch cell composition was determined to be a single types of cathode chemistry by FESEM and ICP-OES → NMC

The Study of Hydrometallurgical Leaching with Two Lixiviant Classes



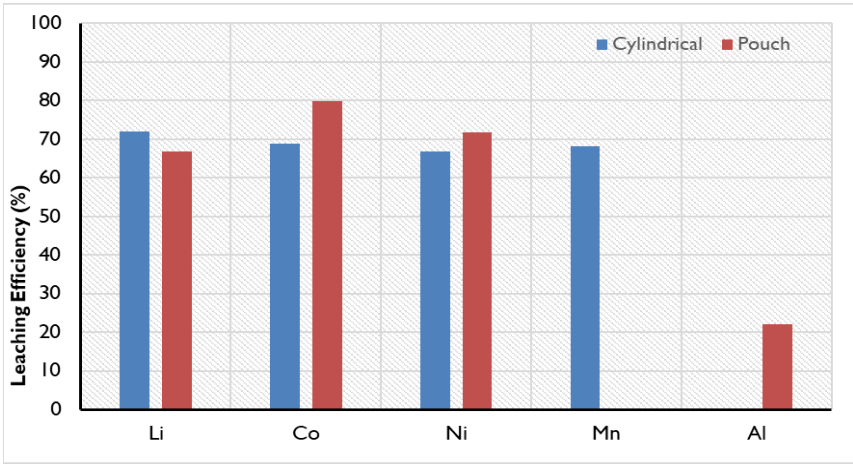
Leaching in <u>inorganic acids</u>



> 90% efficiency by ICP-OES

Leaching in a <u>deep eutectic solvent</u> (DES)

Patent no.: US11,591,670 B2

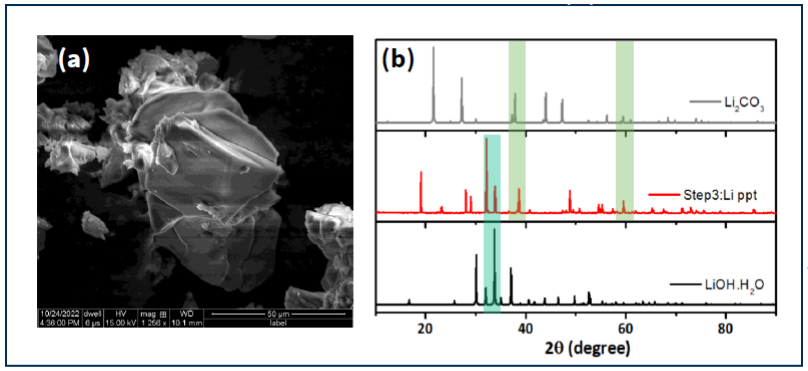


< 90 % efficiency by ICP-OES;
Increase efficiency after optimized formulation

Optimization of Cathode Materials Precipitation Pathways

Co-Precipitation

Current focus:
Lithiation and heat treatment reactions (data not shown)



(a) SEM of the Li precipitate. **(b)** PXRD patterns of the precipitate.

Leachate solution → Precipitate with Al, Fe

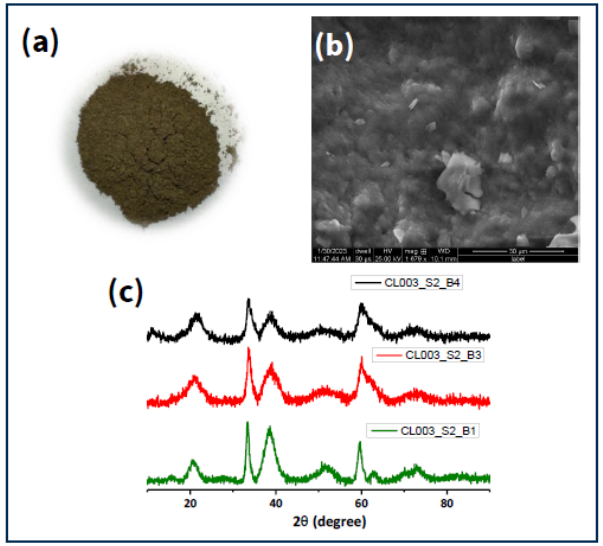
Liquor 1 → Transition Metal Co-Precipitate

Liquor 2 → Li_2CO_3 precipitate

This set of co-precipitation data were analyzed from inorganic acid leachate.

Table 1: Precipitation percentages of different metals from the leachate

Element	Precipitation%
Co	98.9
Ni	97.6
Mn	94.3



(a) Photograph of the precipitate. **(b)** SEM image of the precipitate. **(c)** PXRD patterns of the precipitates.

→ The co-precipitation reaction is more suitable for a single cathode chemistry, in attempt to maintain the stoichiometry of the recovered cathode materials prior to lithiation, followed by heat treatment reactions. Current work focus on the optimization of lithiation using different lithium sources.

Sequential Precipitation

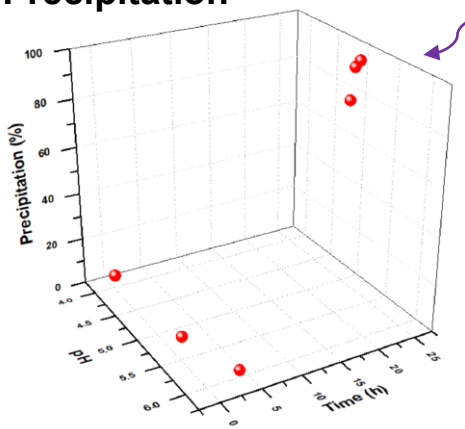


Figure: Summary of Al precipitate (%) conditioned at different pH over time (h).

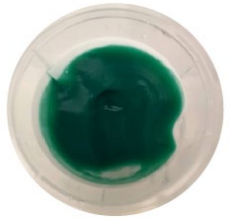
STEP 1: Al Removal: Leachate Solution → $Al(OH)_3$ precipitate (ppt) + Filtrate 1

STEP 2: Ni Removal: Filtrate 2 + Ni ppt

STEP 3: Co Removal: Filtrate 3 + $Co(OH)_2$ ppt

STEP 4: Li Removal: Filtrate 4 + Li_2CO_3 ppt

Current focus:
Better separation of oxide or hydroxide Co and Ni metals.



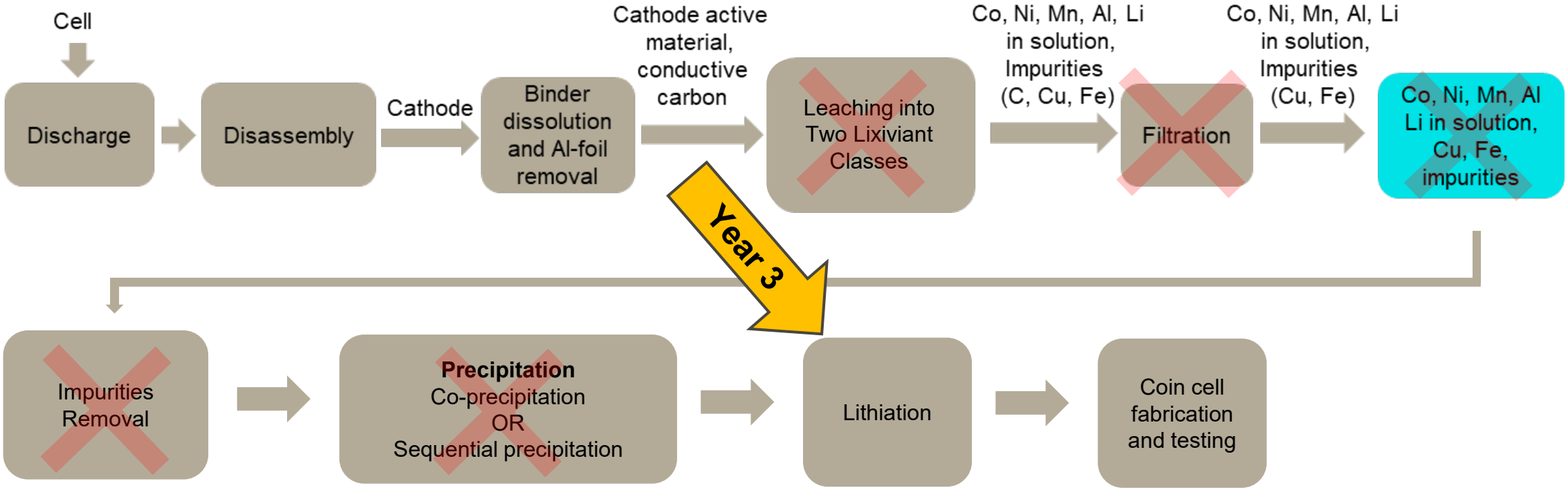
A mixture of Co and Ni precipitates was detected by ICP while adjusting the pH.

→ The sequential precipitation reaction is more suitable for a blended cathode chemistries, in attempt to crash out individual metals for recycling.

Research Flowchart:

Year 3: Microwave-Assisted Direct Methods for Recycling

- Develop methods to recover battery-grade cathodes while preserving their chemical integrity.
- Investigate the effectiveness of microwaves in eutectic and hydrothermal regeneration.
- Minimize processes like chemical treatments and mechanical separation to enhance energy efficiency and sustainability.

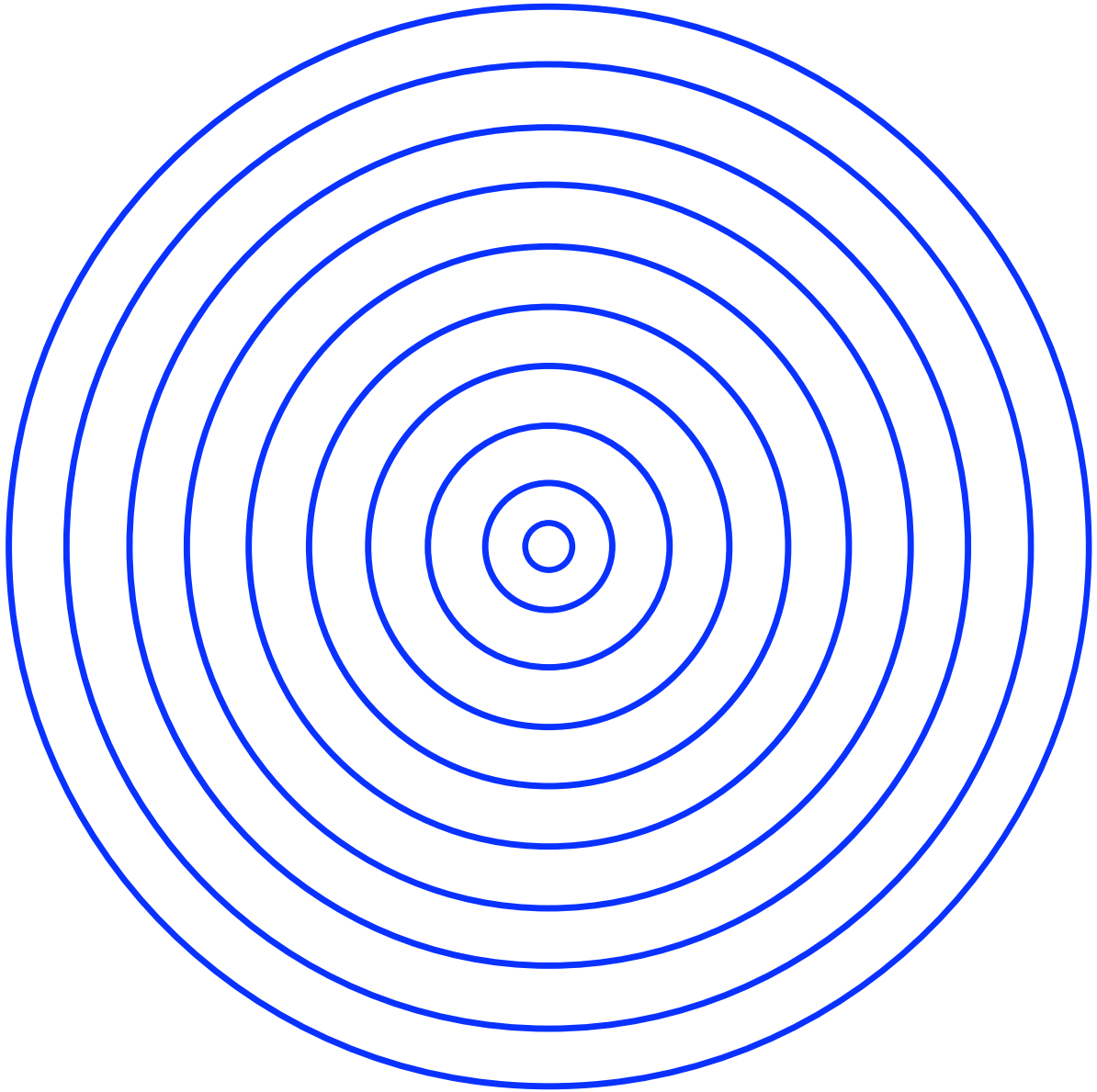


Regeneration Methods:

1. Eutectic and Organic
2. Hydrothermal

In addition …

Besides our collaborative research effort, **ULRI | ESRI** is also involved in public safety campaigns and initiatives to proactively raise public awareness and advocate the importance of battery recycling through knowledge sharing and education.



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