Enhanced Validation of Advanced Battery Supply Chains (EVALS) Overview

Presenter: Katharine Harrison

National Renewable Energy Laboratory

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Center For Extreme Batteries Spring Meeting

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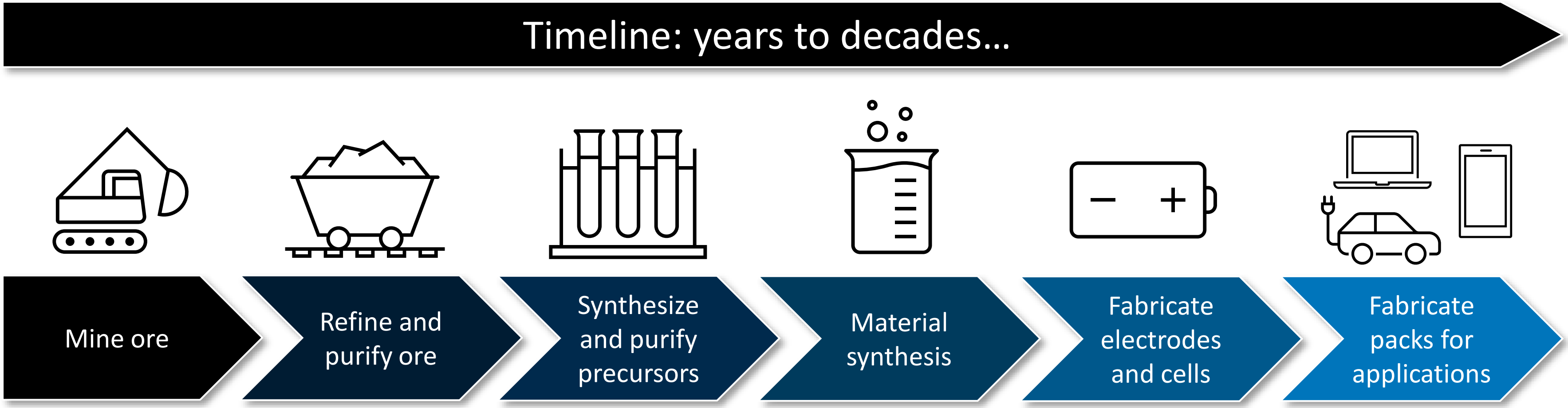
Photo by Josh Bauer, NREL 61725

Significant time and cost barriers to setting up domestic battery supply chain



Mining and refining can take years to develop because mines have different impurities (unless supplier can leverage existing mine).

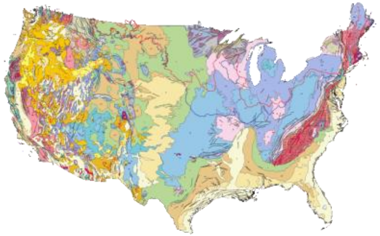
Materials producers require years to develop battery grade precursors that optimize processing for purity and performance.

Once material suppliers meet qualifications, many more years are required for validation in specific applications.

EVALS: Accelerate the path from mine to battery and improve supply chain resiliency

NREL | Idaho National Laboratory | Argonne National Laboratory

Materials Processing → Active Material Production → Cell Manufacturing → Deployment



OEM = original equipment manufacturer
EV = electric vehicle

EVALS primary goal is to reduce the time and cost associated with adopting domestic sources by:

Better understanding source impurity impacts on material properties and performance to inform qualification decisions and cost-performance tradeoffs.

Developing suite of tools and processes to decrease validation time for domestic battery materials to support material manufacturers and automative EV OEMs.

$LiFePO_4$ (LFP) was selected as an initial example material for EVALS, but EVALS is *not* an LFP project.

US gigafactories online FY26 show interest in US manufacturing (all chemistries).[1]

AA Portable Power
AKASOL
Alion Science and Technology
AllCell Technology
Altair Nanotechnologies
American Battery Factory
American Battery Solutions
American Lithium Energy
Auto Motive Power
Battery Specialties
Blue Line Battery
BMW Spartanburg Plant
BorgWarner-Romeo Power
Braille Battery
Cell-Con
Clarios
Conamix
Coreshell Technologies
Cuberg
Custom Power
Dantona Industries
EaglePicher Technologies
Electric Power Systems
Electrochem Solutions
EnerSys
EnerSys
Enovate Medical
eNow
EnPower
Envia Systems
Envision AESC Bowling Green Plant
Envision AESC Smyrna Plant
Exponential Power
Exponential Power
Exponential Power
Factorial
Flexodes
Flux Power
Ford Rawsonville Plant
Ford-SK BlueOval
Ford-SK BlueOval City
General Motors Brownstown Battery
GM-LG Ultium Cells
GM-LG Ultium Cells
GM-LG Ultium Cells
GS Yuasa Lithium Power
Highpower International
Honda Brownstone Battery
Hyundai-SK
Inventus Power Woodridge Facility
Kore Power
LG Energy
LG Energy
Lithion Battery
Lithion Battery
Lithion Battery
LytEn
Magnis
Mercedes Battery Plant
Microvast
Navitas Systems
Octillion Power Systems
Our Next Energy
Polyplus Battery
ProTechnologies
Proterra
Proterra
Proterra
Rivian
Saft America
Saft America
Samsung SDI
Simpliphi Power
Soelect, Inc.
Sparkz Inc.
StateVolt
Statevolt
Stellantis-Samsung Kokomo Plant
Tenergy Corp
Tesla Gigafactory Texas
Tesla Giga New York
Tesla Gigafactory 1
TNR Technical
Toyota
Trojan Battery
Wabtec
Xalt Energy
Yotta Energy
Zakuro
Zeus Battery Products
Zinc8 Energy

1. https://electronsx.com/battery-gigafactories.php

LFP demand is growing rapidly and US demand projected >4x higher in 2030 (180 GWh/y) than 2025.[2]

LFP contains fewer critical materials than conventional $LiNi_xMnyCo_{1-x-y}O_2$ (NMC) – more available in US.

2. Source: BloombergNEF https://cen.acs.org/energy/energy-storage-/Lithium-iron-phosphate-comes-to-America/101/i4

EVALS Thrust Structure

Senior Leadership Team: Eric Dufek (PI), Tony Burrell, Jack Vaughey

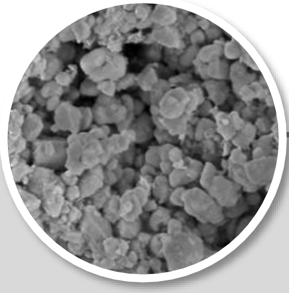
Industrial Advisory Board

Caleb Stetson

Process Knowledge and Sourcing

- US sources and their impurities – case studies
- Inform impurity evaluation and cost-performance tradeoffs

Katie Harrison



Materials

- Materials synthesis with and without target impurities
- Characterization to understand impurity impacts and screen

Andy Jansen

Electrode and Cell Development

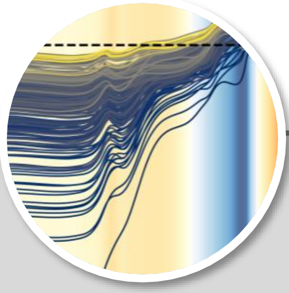
- Electrode slurry and coating development
- Cell design and fabrication

Tanvir Tanim

Advanced Test Development/Analysis

- Tiered test protocol development
- Electrochemical and physical characterization

Peter Weddle



Models and Predictions

- Design of experiments and life prediction
- Inform cost-performance tradeoffs

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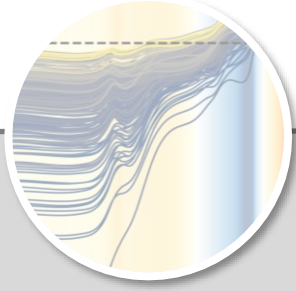
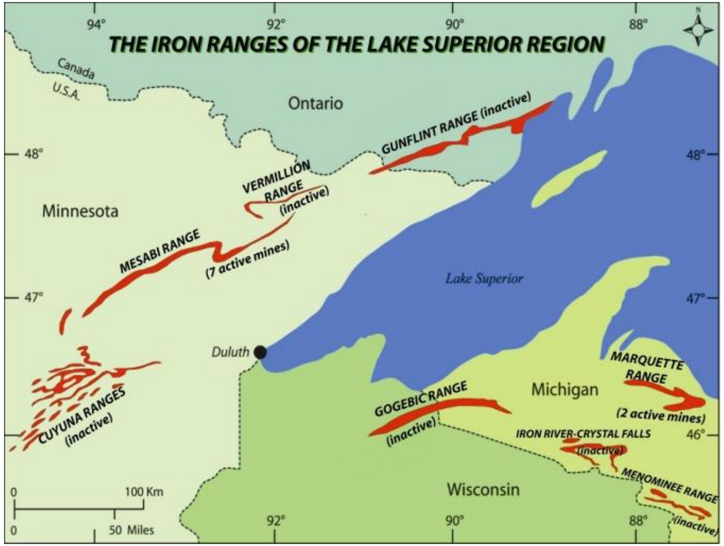
				
Process Knowledge and Sourcing	**Materials**	**Electrode and Cell Development**	**Advanced Test Development/Analysis**	**Models and Predictions**
US sources and their impurities – case studies	Materials synthesis with and without target impurities	Electrode slurry and coating development	Tiered test protocol development	Design of experiments and life prediction
Inform impurity evaluation and cost-performance tradeoffs	Characterization to understand impurity impacts and screen	Cell design and fabrication	Electrochemical and physical characterization	Inform cost-performance tradeoffs

Iron resource case study: Minnesota Mesabi Iron Range mined since late 19th century

Process Knowledge and Sourcing



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By W.F. Cannon (USGS) Public Domain, https://commons.wikimedia.org/w/index.php?curid=39161950

Taconite is the major orebody mineral mined in the region.

Infrastructure, workforce, and transportation networks.

Fe required for LFP is <<1% of that required for steel.

All Fe currently mined to make low-silica magnetite pellet product for steel manufacture.

Mining, Comminution, and Beneficiation Flowsheet to Form Iron Ore Product for Steelmaking

Drill → Blast → Shovel → Haul → 1° and 2° Gyratory Crushing → Rail Transport to Concentrator → 3° and 4° Cone Crushing

Rod Milling → Dry Magnetic Separation → Ball Milling → Rougher Magnetic Separation → Gravity Separation → Screening

→ H_2O Recovery → Finisher Magnetic Separation → Thickening → Filtration → Balling → Binding → Pelletization

Counterproductive in sourcing Fe for LFP

Iron resource case study: orebody and processing impurities

Process Knowledge and Sourcing

Apply knowledge of **geology** and **processing** to domestic primary resources to understand feasibility for **battery precursor** products.

Iron Ore Pellet Composition Targets *for Steelmaking*

Composition (weight %)	Min	Target	Max
Fe	65		
Silica	4.60	4.80	5.00
CaO	0.77	0.85	1.20
P	0.016	0.021	0.021
$Na_2O + K_2O$		0.062	0.073
Moisture (H_2O)		2.75	4.30

Sadeghi, B., Najafizadeh, M., Cavaliere, P., Shabani, A. and Aminaei, M., 2024. Effect of composition and processing conditions on the direct reduction of iron oxide pellets. Powder Technology, 444, p.120061. (open access)

LFP has different impurity concerns than steel.

Magnetite concentrate products are analyzed for suitability for steel production, not for alternative minor products.

Example: P induces steel embrittlement, so it must be minimized in steel, but small P impurity may not degrade LFP.

Also have to consider impurity impacts from processing agents (clay binders, frothing agents, flocculants, etc.).

When converting Fe ore to LFP precursors, what impurities are likely?

Process Knowledge and Sourcing

Taconite iron ore pellets for compositional and leaching studies.

Most common Fe ore impurities are refractory (high melting temperatures and chemically resistant).

- Silica, alumina, magnesia not mobilized in leaching.

Aqueous matrix matters for impurity removal (e.g., Ca).

- Sparingly soluble in sulfate environment as $CaSO_4$.
- Moderately soluble in basic environment as $Ca(OH)_2$.
- Highly soluble in chloride environment as $CaCl_2$.

Solvent extraction oxidizes Fe to Fe^{3+} and other trivalent ions tend to accompany (e.g., Cr^{3+}, Al^{3+}).

Near neighbor elements are often difficult to separate.

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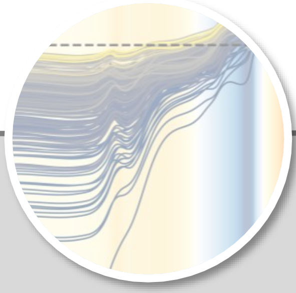


Models and Predictions

Design of experiments and life prediction

Inform cost-performance tradeoffs

Accelerating material sources from mine to batteries requires an example LFP synthesis process to introduce impurities

Materials

Impurity identification from source analysis

Synthesis with and without target impurities

Materials characterization

Downselect and send materials for electrode and cell fabrication

98.5% of LFP is made in China today by a two-step process.

$FeSO_4 + H_3PO_4 \rightarrow FePO_4$

$FePO_4 + Li_2CO_3 +$ dextrose $\rightarrow LiFePO_4$-C

$FeSO_4$ is waste biproduct in TiO_2 manufacturing in China[1-3] that minimizes cost and uses local resources.

Synthesis approaches and considerations vary in USA.

Precursor cost/availability and environmental regulations in US differ from China.

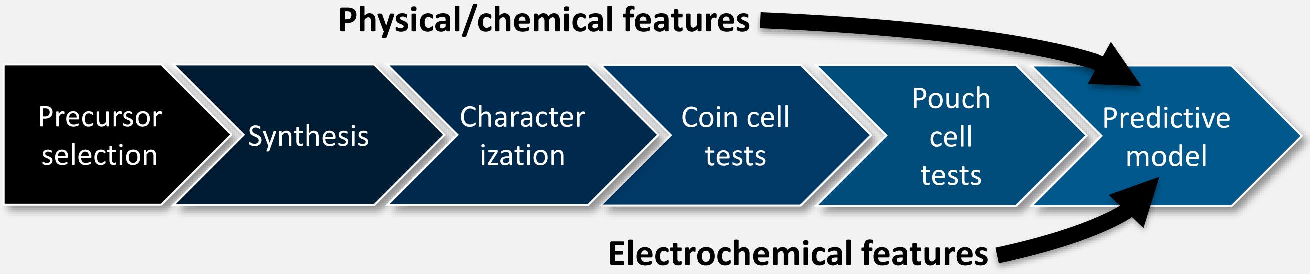
Some targeting Fe/Fe oxides to avoid sulfate waste[1-2] or purchasing Chinese-manufactured $FePO_4$.

Impurity impacts depend on synthesis process.

EVALS using two-step process to demonstrate tools for validating sources and accelerating mine to battery path.

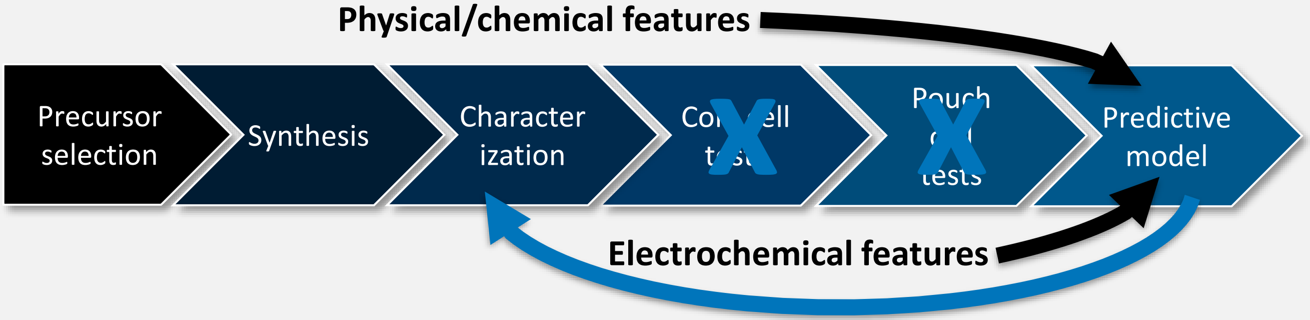
1. https://cen.acs.org/energy/energy-storage-/Lithium-iron-phosphate-comes-to-America/101/i4
2. Y. Caprara, "Identifying the pinch points in the LFP supply chain," Battery Materials Review (2022), 5 (2).
3. J. Quan, S. Zhao, D. Song, T. Wang, W. He, G. Li, Sci. of Total Environ. 819 (2022) 153105.

Correlating materials characterization and electrochemical performance may inform rapid screening protocols



Train models with materials and electrochemical characterization.

Correlating materials characterization and electrochemical performance may inform rapid screening protocols



Train models with materials and electrochemical characterization.

Reduce electrochemical matrix with materials characterization screening tools and protocols.

Correlating materials characterization and electrochemical performance may inform rapid screening protocols

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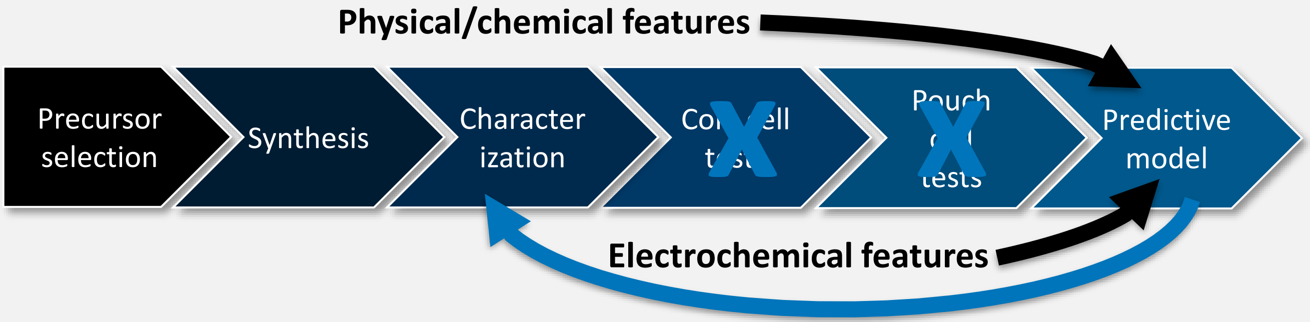


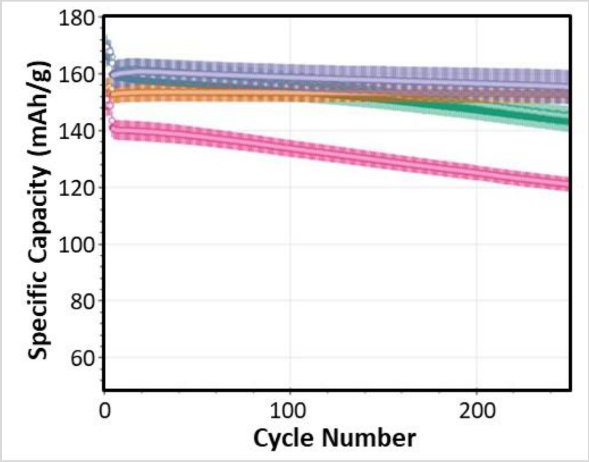
Train models with materials and electrochemical characterization.

Reduce electrochemical matrix with materials characterization screening tools and protocols.

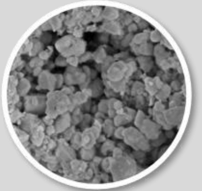
Need to link materials signatures to performance.

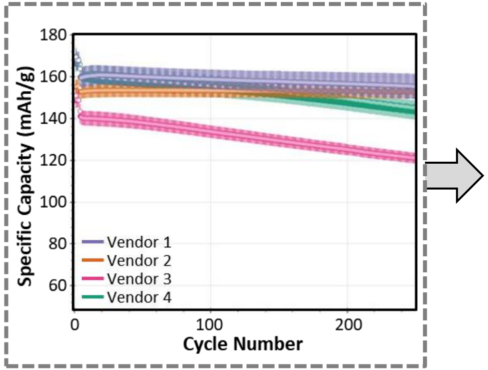
Four **commercial LFP** materials show vary different performance in low loading half cells.

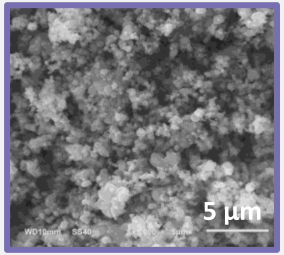
Characterizing to correlate material properties with performance and most effective screening tools.

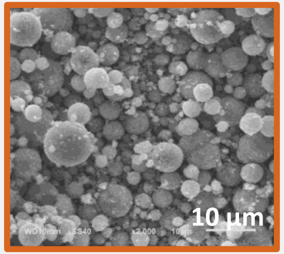


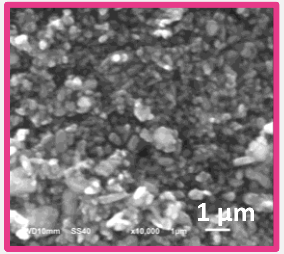
Commercial LFPs show distinct bulk physical and chemical signatures



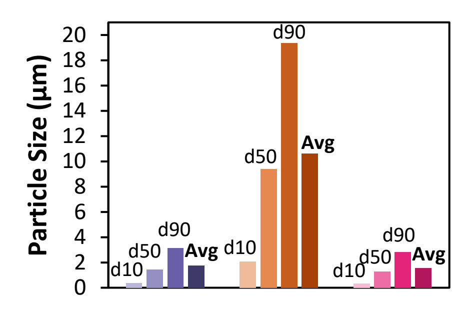


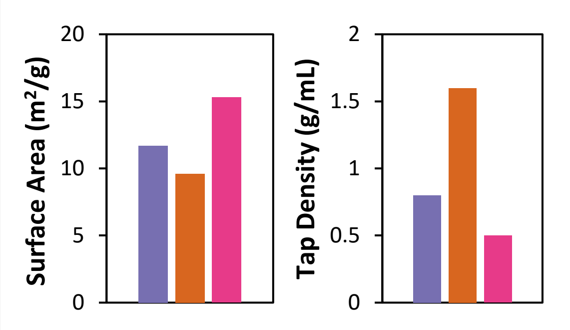




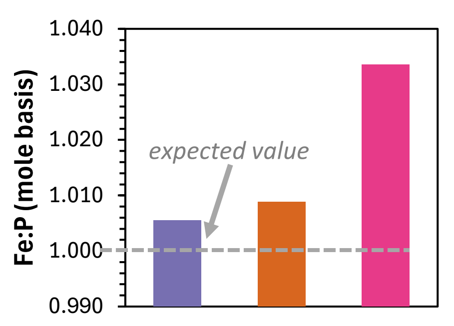


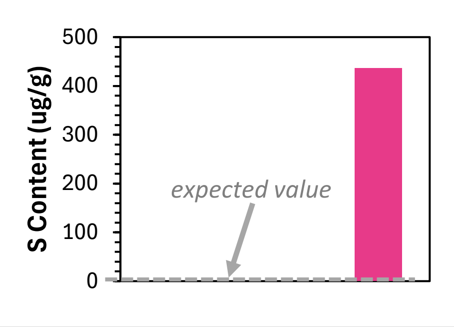
Vendor 1 Vendor 2 Vendor 3





Physical Characterization





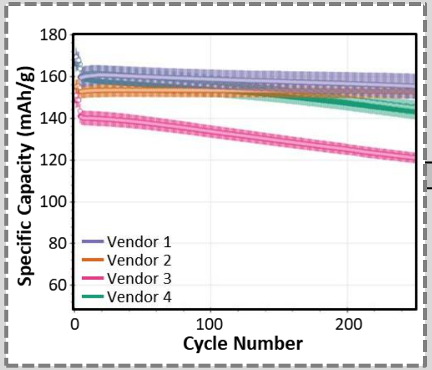
Chemical Characterization

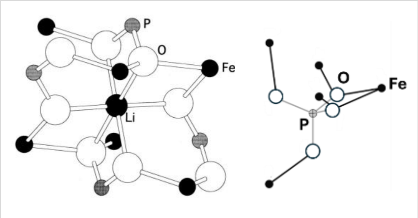
Performance not simply correlated to particle size, surface area, or tap density (despite large variation).

Poor performance correlated with higher than 1:1 Fe:P and presence of S impurities.

Solid state NMR sensitive to atomic and electronic ^{6}Li and ^{31}P environments and EPR sensitive to magnetic and electronic structure of Fe

Materials

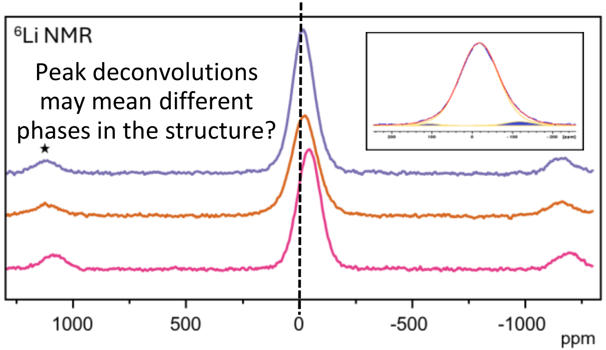




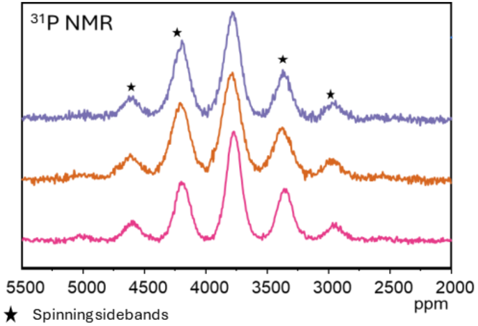
EPR and NMR sensitive to changes between commercial LFPs but still determining relation to structural features.

NMR = nuclear magnetic resonance
EPR = electron paramagnetic resonance

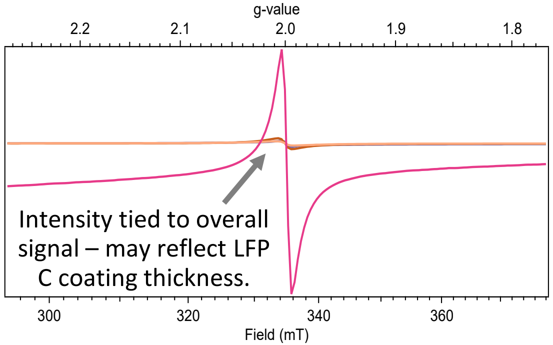
Shift/width changes with Li stoichiometries, local disorder, and defects sites.



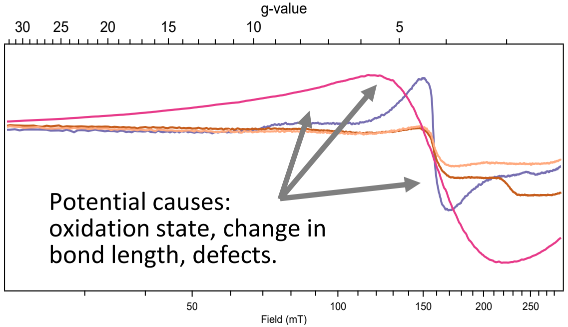
Sideband manifold span informs local symmetry and interactions between paramagnetic metals.



EPR signal indicates magnetic disruption (no signal for Fe^{2+} in LFP).



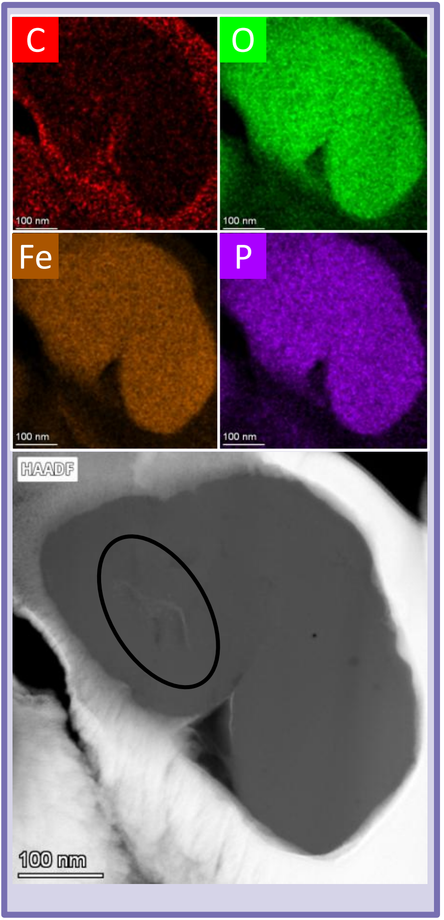
EPR fine structure informs disrupted magnetic state cause.



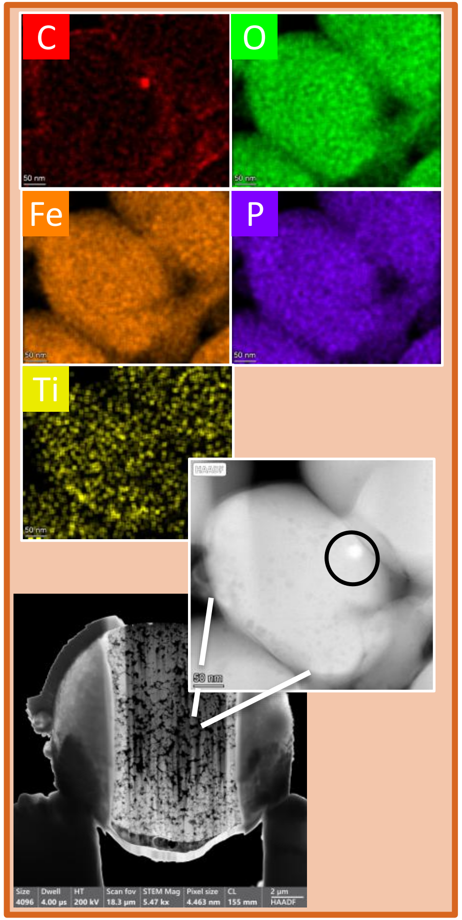
Probing Performance Differences with STEM/EDS and 4D STEM


Materials

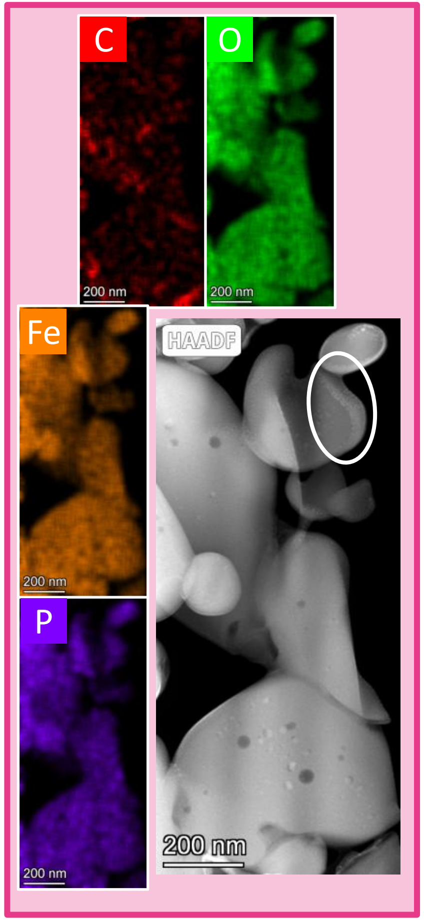
Vendor 1

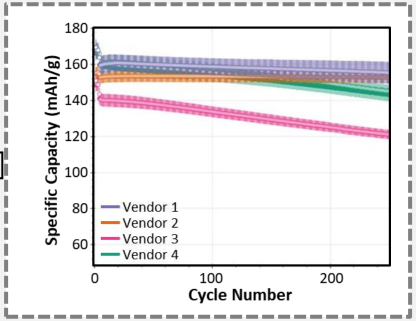


Vendor 2



Vendor 3





Particle-scale compositional heterogeneity evident.

C/Ti coating uniformity varies.

Atomic-level defects (vacancies, anti-site disorder, substitutions).

STEM = scanning transmission electron microscopy
EDS = energy dispersive spectroscopy

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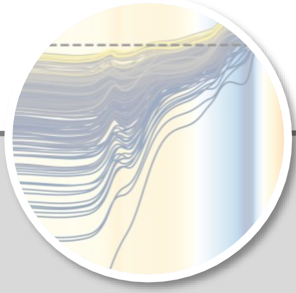


Models and Predictions

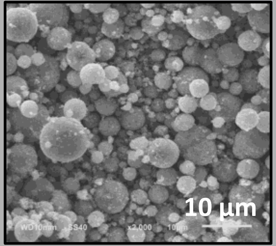
- Design of experiments and life prediction
- Inform cost-performance tradeoffs

High quality electrode coatings and cells from CAMP will enable robust electrochemical testing and statistically relevant results

Cell and Electrode Development

LFP slurry optimization → Coating development → Cell design and fabrication

Commercial and EVALS-synthesized LFP materials.

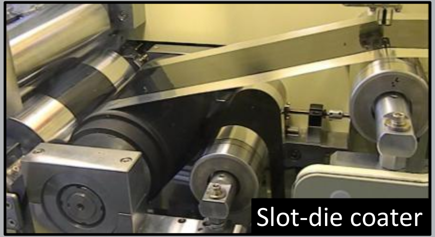


CAMP = Argonne's Cell Analysis, Modeling, and Prototyping Facility

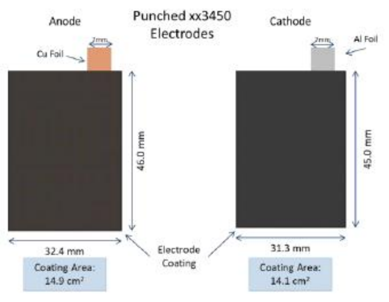
Roll-to-roll doctor blade with slurry hopper not ideal for high surface area materials but only requires 20-50 g of material.


Reverse-comma coater

Actively pumps slurry through slot so ideal for high surface area electrodes but need hundreds of g of material.


Slot-die coater

CAMP capable of semi-automated pilot-scale manufacturing in dry room.



92% active LFP, 2.3 mAh/cm², 35% porosity, N/P = 1.05-1.1

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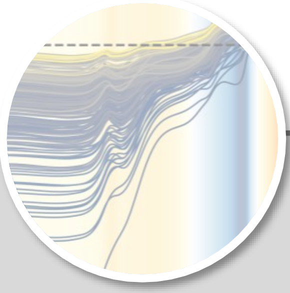
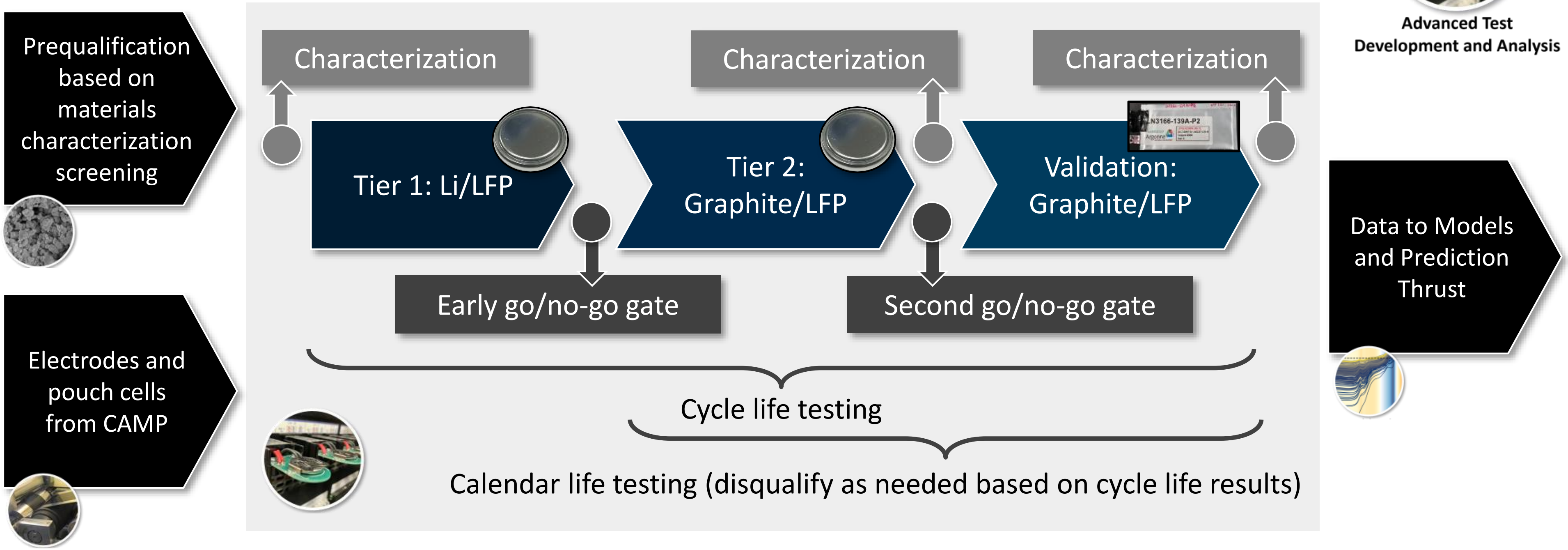


Models and Predictions

- Design of experiments and life prediction
- Inform cost-performance tradeoffs

Tiered testing protocols help reduce long-term cycling test matrix

Advanced Test Development and Analysis



Support rapid decision-making on impurities using approaches that combine both electrochemical and non-electrochemical data.

Innovate testing diagnostics to accelerate testing while preserving key signatures that enable machine learning/artificial intelligence approaches.

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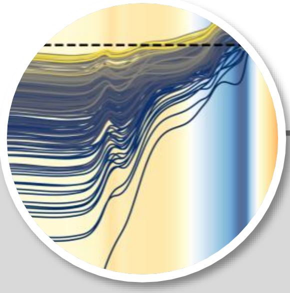

Models and Predictions

- Design of experiments and life prediction
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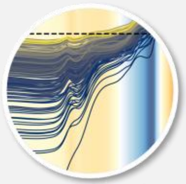
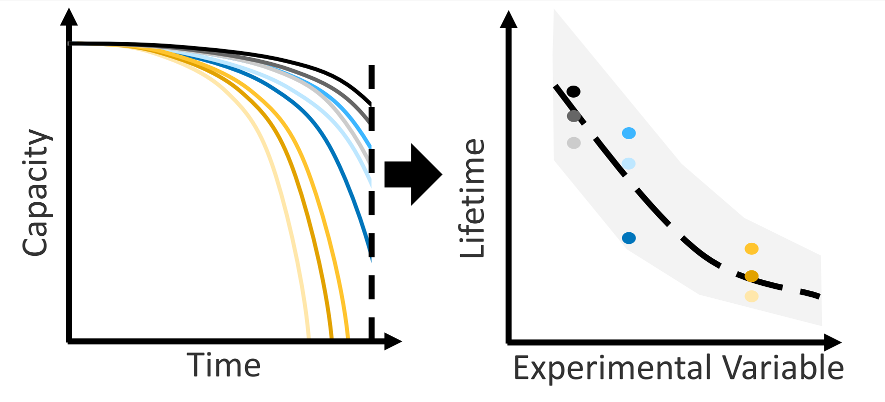
Develop models to predict lifetime, reduce test matrix/time, and inform cost-performance tradeoffs



Models and Predictions

Traditional life testing

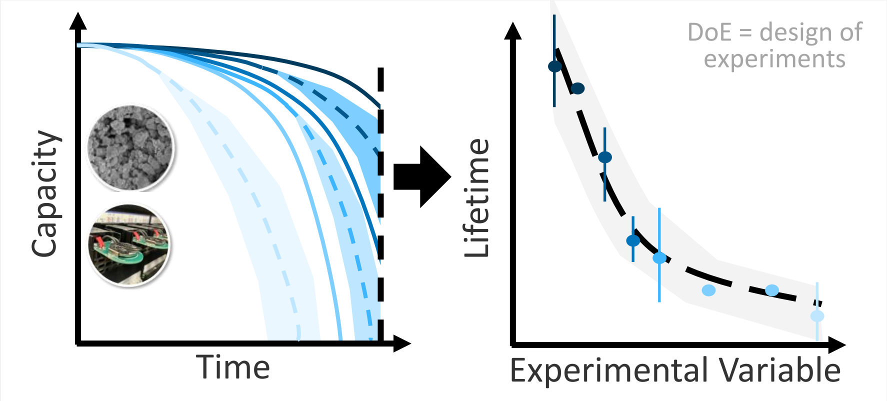
Batch testing cells in test matrix by cycling replicate cells to failure in parallel is inefficient use of time and channels.



Channel	Testing Weeks									
1										
2										
3										

DoE and early life prediction

Develop early prediction models and employ them to reduce test matrix and inform next experiment.

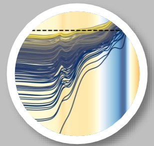


Channel	Testing Weeks												
1													
2													
3													

Cost-performance tradeoffs

Inform TEA to understand cost impacts of bad-actor impurity removal.

Couple material purity and processing information with performance.



TEA = technoeconomic analysis

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Questions? Comments? Thank you.

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Photo from Getty-181828180

Why focus on impurities and tool development?

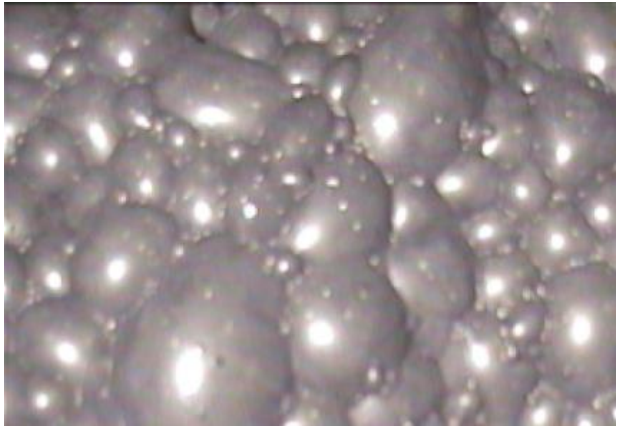
Identified Challenges

- Each geologic source has different impurity levels.
- New source validation is time consuming.
- Low uniformity in purification methods and tolerance levels across materials production and cell manufacturing.
- High-throughput, combinatorial analysis is insufficient and costly.

Opportunities for Tools to Save Time and Cost

- Location-specific forecasting of impurities and impacts.
- Test matrix reduction methods decrease time and resources for combinatorial, high-throughput approaches.
- Coordinated characterization, testing, prediction, and modeling tools.
- Expanded parameter extraction from early data.
- Integrated tools strengthen links between performance and cost as a function of processing and impurities.
- Transferable insights across materials and cell designs.
- Potential for qualification standardization.

Iron resource case study: product impurities (arise from resource AND processing methods)



Aldrich, C. and Liu, X., 2021. Monitoring of flotation systems by use of multivariate froth image analysis. Minerals, 11(7), p.683. (open access)

Iron Ore Pellet Production: Entrained Reagents and Consumables

Consumable	lb/Mt pellet product
Grinding Media (Fe)	4.48
Diamine ($CH_2(NH_2)_2$)	0.144
Flocculant [$(C_6H_{11}XO_4)_n$]	0.027
Soda Ash (Na_2CO_3)	**1.557**
Frother ($CH_3CH_2CH_2CH_2CH(CH_2CH_3)CH_2OH$)	0.027
Bentonite [$(Na,Ca)_{0.33}(Al,Mg)_2(Si_4O_{10})(OH)_2 \cdot nH2O$]	**9.0**
Organic ($C_xH_yO_z$)	0.50
Fluxstone (Ca-rich melting-point depressant)	**26.93**

Compositional targets ignore the reagents used in separations.

Entrained reagents/impurities problematic in downstream *steel production* are tracked; these will differ for battery precursors.