Tunable Electrochemical Pathway for High-purity REM and CM

Project Number : DE-FE0032121

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U.S. Department of Energy

National Energy Technology Laboratory

Resource Sustainability Project Review Meeting

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Project Overview

- Funding Opportunity Announcement
  - DE-FOA 0002404
- Awarded Funding
  - DOE - $200,000 and
  - Cost Share - $50,000
- Overall Project Performance Dates
  - Original – November 30, 2021 – August 30, 2022
  - Extended to – February 28, 2023

Project Overview

Project Participants

- U.S. Department of Energy (DOE)
- North Dakota State Energy Research Center
- Trimeric Corporation
- Current Lighting Solutions, LLC
- Critical Materials Institute, Ames Laboratory
- UND Institute for Energy Studies (IES)
- EERC

Project Overview

Overall Project Goal

Develop a technical research plan for defining and assessing the techno-economic viability of a tunable electrochemical pathway (TEP) for producing individually separated high-purity rare-earth metals (REM) and critical minerals (CM) from lignite coals and combustion by-products originating from the Williston Basin.

Project Overview

Overall Project Objectives

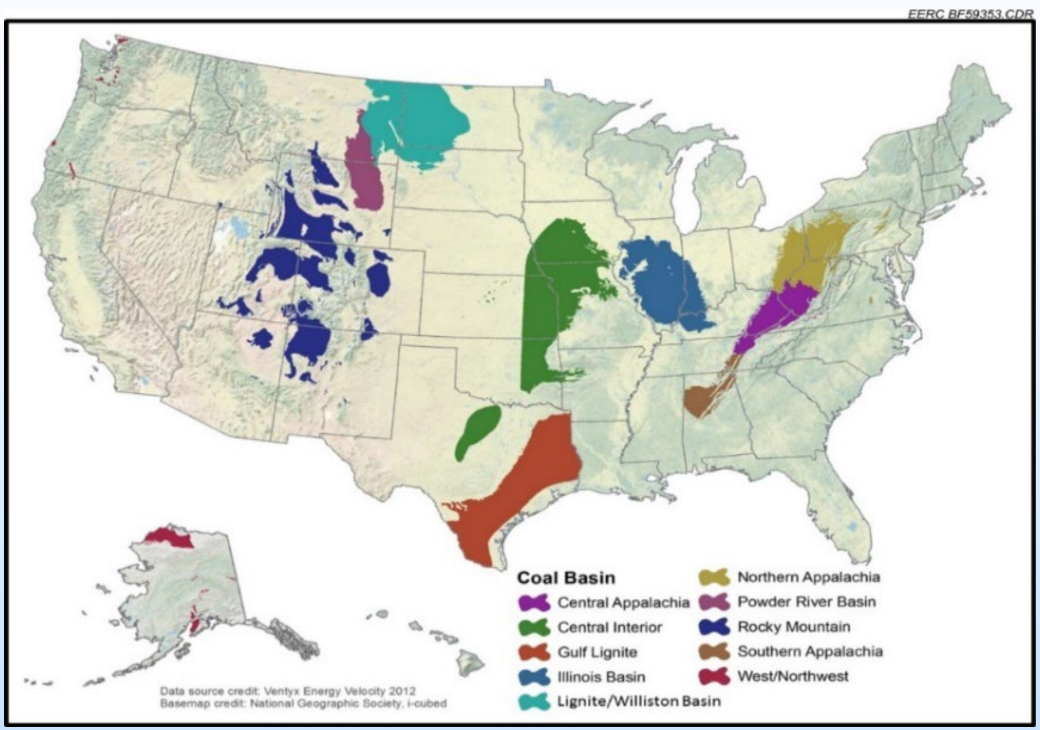
- Identification
  - targeted REE and CM market(s).
  - targeted set of critical materials used in these markets/applications.
- Selection
  - feedstock and currently existing facilities for production of MREO/MRES and CM.
- Description of process for
  - production of individually separated high-purity (ISHP) REO/RES/CM.
  - production of REM.
  - conversion of CM produced in pilot-scale facilities to CM compounds of interest for industrial applications.
- Development of a
  - conceptual process flow diagram illustrating circuit integration for production of REM and CM from coal-based resources.

Technology Background

In prior EERC work,

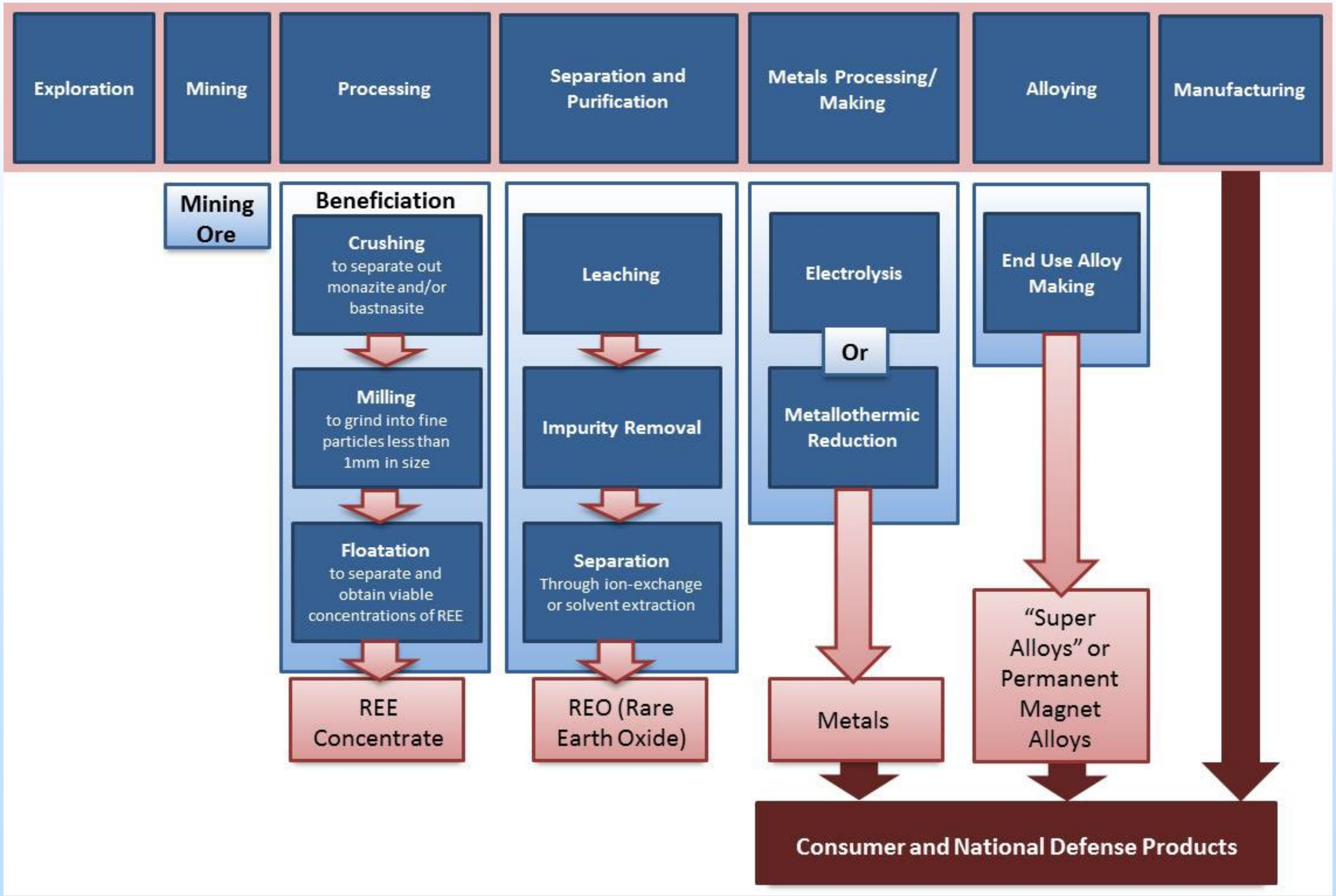
- Williston Basin lignite coal seams were shown to contain REE concentrations in the top 1%–3% of all reported U.S. coals measured to date.

- Major fraction of the REE is bound to the lignite organic matrix by chemical bonds that translate to simpler, cheaper, cleaner extraction compared to higher-rank coals.



Technology Background

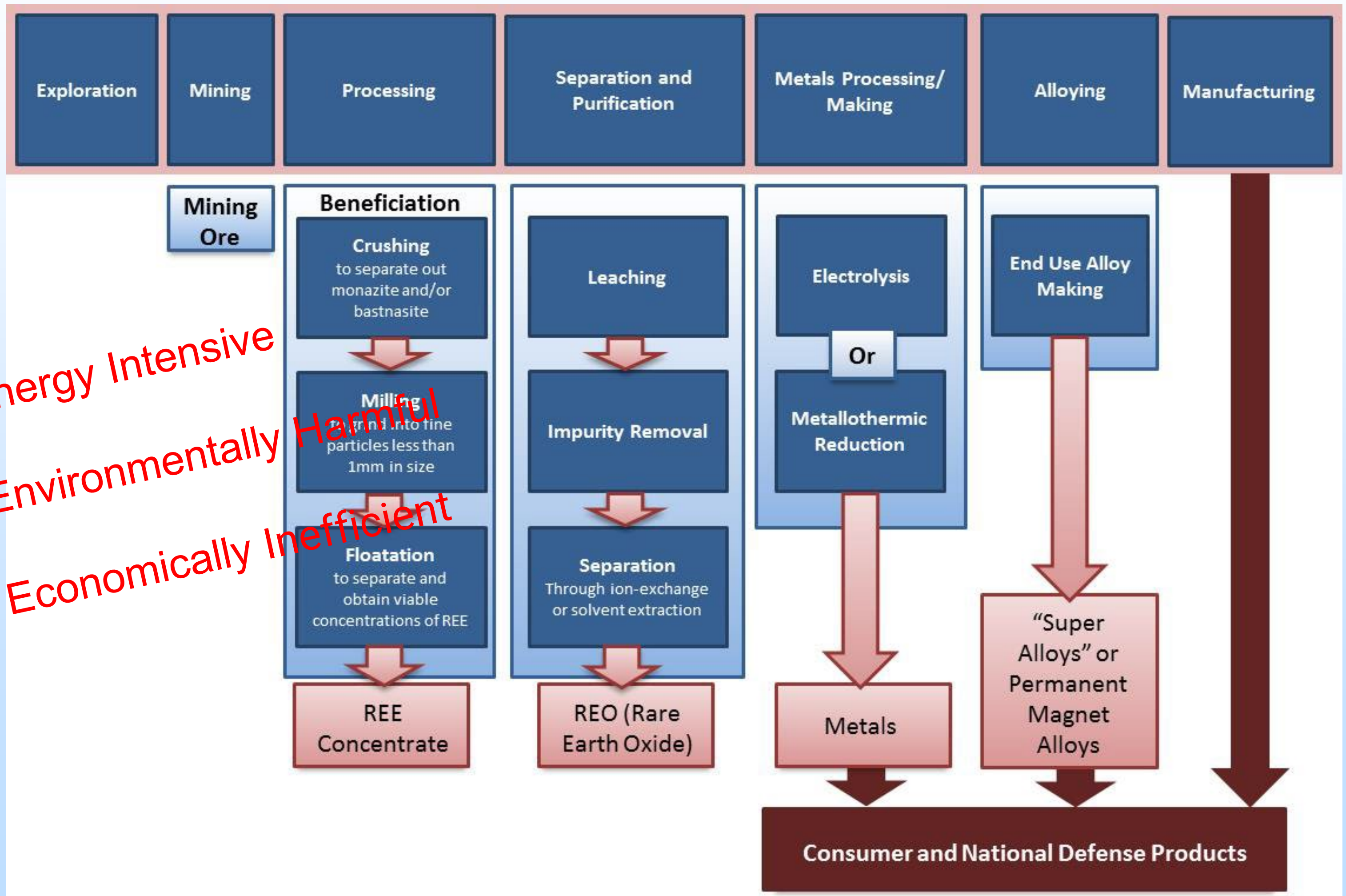
Traditional REEs Processing



Source: Report on Rare Earth Elements From Coal and Coal Byproduct, DOE, 2017

Technology Background

Traditional REEs Processing



- Energy Intensive
- Environmentally Harmful
- Economically Inefficient

Source: Report on Rare Earth Elements From Coal and Coal Byproduct, DOE, 2017

Technology Background

Element	Oxidant	Reductant	E°/V	Electrons	Ref
Th	Th^{4+} +e^-	Th^{3+}	-3.6	1	2
Pr	Pr^{3+} + e^-	Pr^{2+}	-3.1	1	1
Er	Er^{3+} +e^-	Er^{2+}	-3	1	1
Ho	Ho^{3+} +e^-	Ho^{2+}	-2.8	1	1
Yb	Yb^{2+} +2e^-	Yb(s)	-2.76	2	1
Sm	Sm^{2+} +2e^-	Sm(s)	-2.68	2	1
Dy	Dy^{3+} +3e^-	Dy^{2+}	-2.6	1	1
Tm	Tm^{2+} +2e^-	Tm(s)	-2.4	2	1
La	La^{3+} + 3e^-	La(s)	-2.379	3	4
Y	Y^{3+} + 3e^-	Y(s)	-2.372	3	4
Ce	Ce^{3+} + 3e^-	Ce(s)	-2.336	3	1
Er	Er^{3+} +3e^-	Er(s)	-2.331	3	1
Ho	Ho^{3+} +3e^-	Ho(s)	-2.33	3	1
Nd	Nd^{3+} +3e^-	Nd (s)	-2.323	3	1
Tm	Tm^{3+} +3e^-	Tm(s)	-2.319	3	1
Sm	Sm^{3+} +2e^-	Sm(s)	-2.304	3	1
Dy	Dy^{3+} +3e^-	Dy(s)	-2.295	3	1
Tb	Tb^{3+} +3e^-	Tb(s)	-2.28	3	1
Lu	Lu^{3+} +3e^-	Lu(s)	-2.28	3	1
Gd	Gd^{3+} +3e^-	Gd(s)	-2.279	3	1
Dy	Dy^{2+} +2e^-	Dy(s)	-2.2	2	1
Tm	Tm^{3+} +e^-	Tm^{2+}	-2.2	1	1
Yb	Yb^{3+} +3e^-	Yb(s)	-2.19	3	1
Nd	Nd^{2+} +3e^-	Nd (s)	-2.1	2	1
Ho	Ho^{2+} +2e^-	Ho(s)	-2.1	2	1
Sc	Sc^{3+} + 3e^-	Sc (s)	-2.077	3	7
Pr	Pr^{2+} + 2e^-	Pr(s)	-2	2	1
Er	Er^{2+} +2e^-	Er(s)	-2	2	1
Eu	Eu^{3+} +3e^-	Eu(s)	-1.991	3	1
Th	Th^{4+} +4e^-	Th(s)	-1.899	4	1
Th	ThO_2 +4H^+ + 4e^-	Th(s) + 2H_2O	-1.789	4	1
Eu	Eu^{3+} +e^-	Eu^{2+}	-0.35	1	12
Ce	Ce^{4+} + e^-	Ce^{3+}	1.61	1	4
Tb	Tb^{4+} +e^-	Tb^{3+}	3.1	1	3
Pr	Pr^{4+} + e^-	Pr^{3+}	3.2	1	20

Technology Background

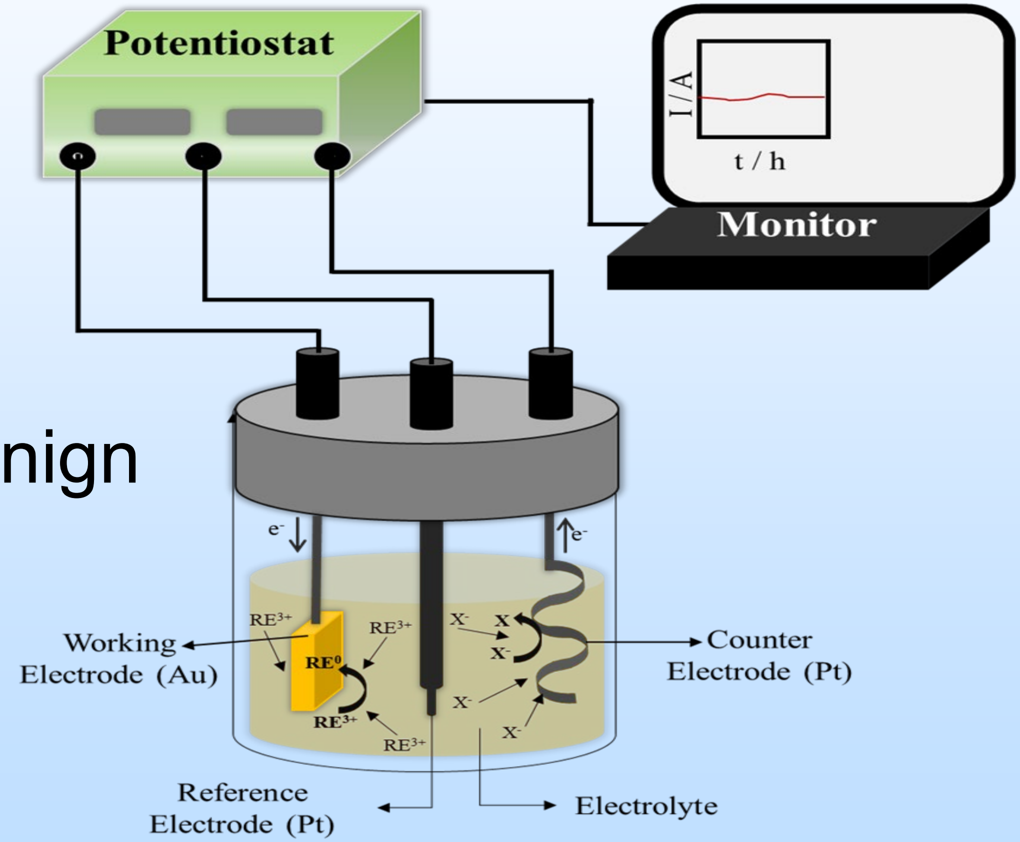
Our Approach to the Processing Challenge

Challenges	Potential Approach
➢ Energy Intensive	➢ Low Temp. process(s)
➢ Environmentally Harmful	➢ Free of Harmful Chemicals
➢ Economically Inefficient	➢ Higher Rate of Recovery

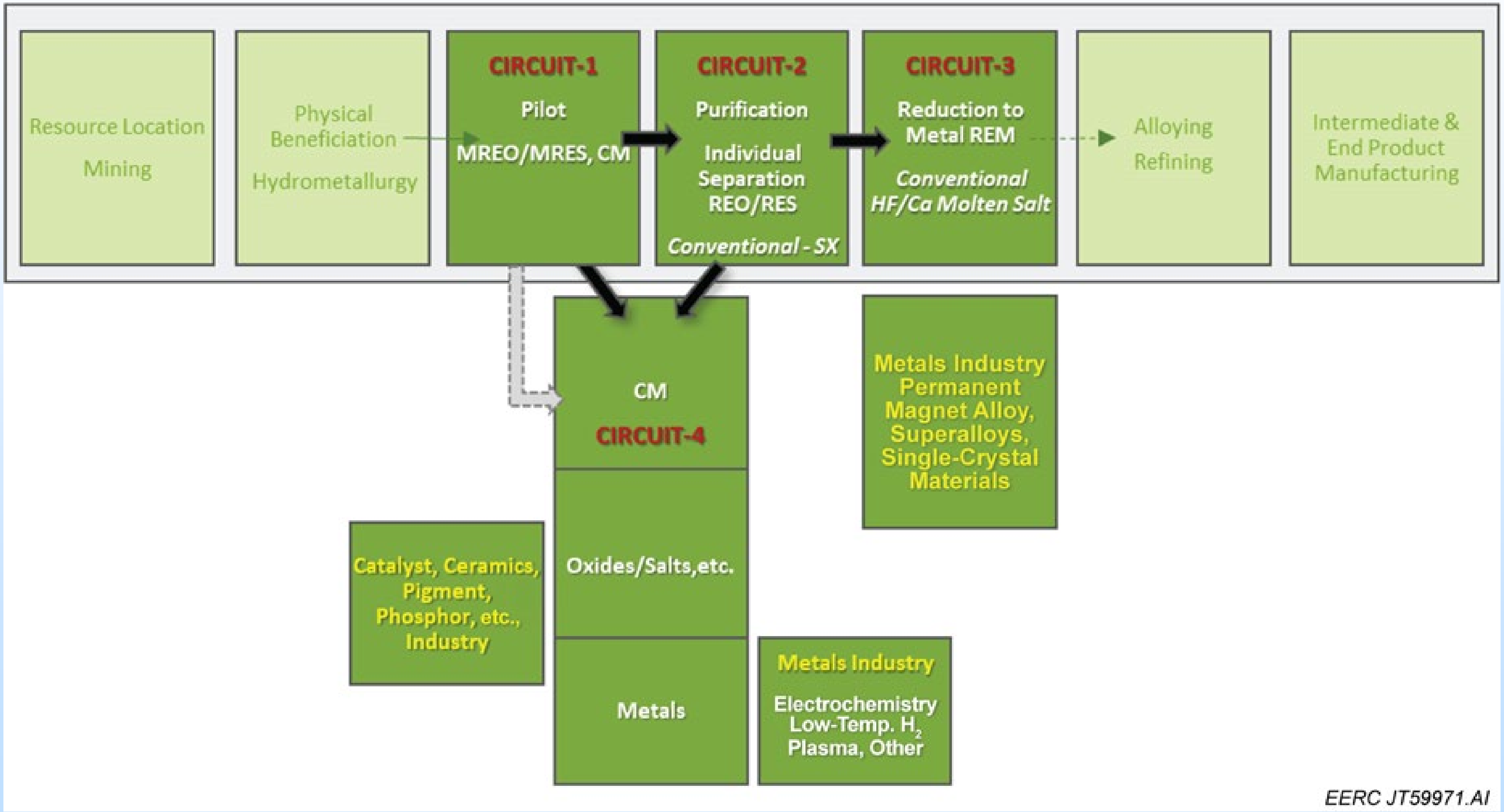
Technology Approach

Tunable Electrochemical Pathway

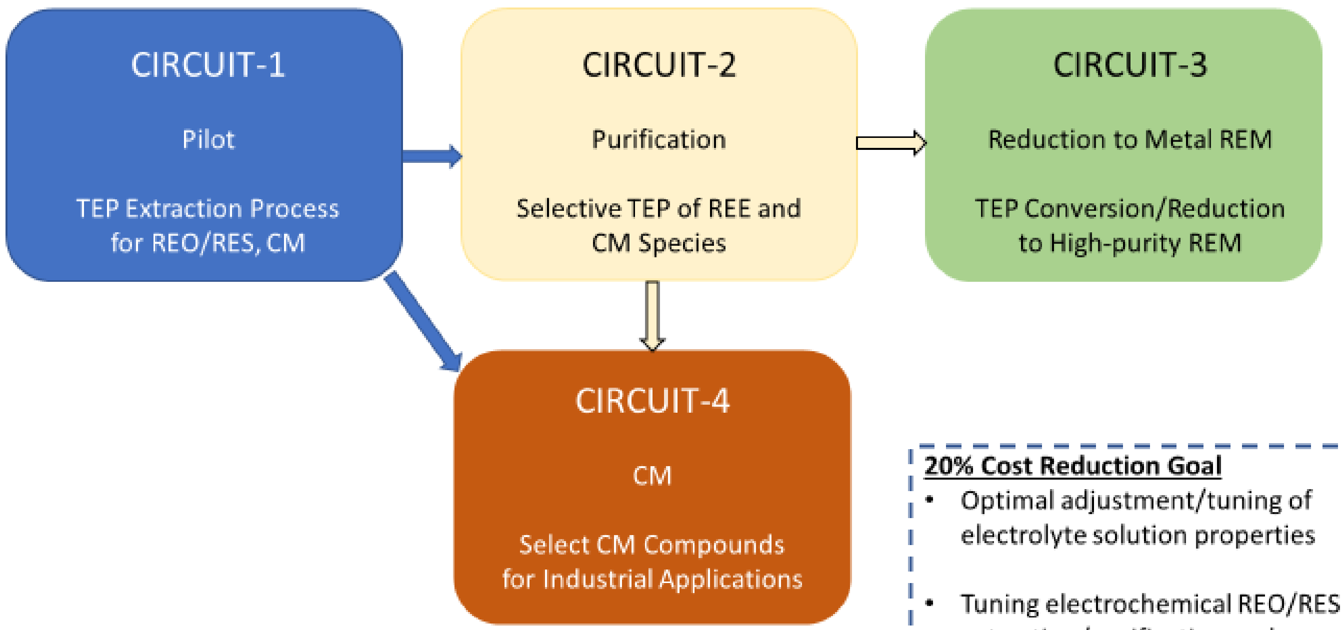
- Low temperature
- Tunable
- Energy Efficient
- Environmentally benign



Technology Approach



Technology Approach



<u>20% Cost Reduction Goal</u>

- Optimal adjustment/tuning of electrolyte solution properties
- Tuning electrochemical REO/RES extraction/purification and reduction to REM operational parameters

Project Scope

Task 1 – Project Management, Planning, and Reporting

Task 2 – Project Teams, Process Development Criteria, Markets, and Industrial Applications

Task 3 – Technical Research Plan

Task 4 – Laboratory Experiments

Project Scope

Project milestones

Task/ Subtask	Milestone (M) Title	PCD	ACD	Verification Method	Comments
3.1	M1 – Circuit 1 Complete	1/31/22	3/31/22	Documented in quarterly report	
3.4	M2 – CM Conversion Assessment Complete	1/31/23		Documented in quarterly report	

Project Scope

Project Deliverables

Task/ Subtask	Deliverable (D) Title	PCD	ACD	Comments
1.0	D1 – Project Management Plan	12/31/21	12/30/21	Complete. Revisions to the project management plan (PMP) shall be submitted as requested by the DOE NETL project manager.
2.0	D2 – Teaming Plan	2/28/23		Due at the end of the period of performance (POP).
2.2	D3 – Process Flow Diagram	2/28/23		By completion of Subtask 2.2.
3.0	D4 – Technical Research Plan Complete	2/28/23		Due at the end of the POP.
3.2	D5 – Purity of Materials Estimated	8/31/22		By completion of Subtask 3.2.
3.2	D6 – Quantity of Resource Identified	8/31/22		By completion of Subtask 3.2.
4.0	D7 – Chemical Characterization Data (EDX)	1/31/23		Due at the end of the POP (to be uploaded to NETL EDX system)
1.0	D8 – Phase 1 Final Technical Report	2/28/23		Due at the end of the POP.

Potential Risks and Risk Mitigation

Perceived Risk	Risk Rating (low, med., high)			Mitigation/Response Strategy
	Probability	Impact	Overall	
Financial Risks				
Failure to Obtain One or More of the Cost-Share Components	Low	High	Med.	The EERC can leverage, and has developed, strategic partnerships with a variety of industry partners to provide cost share.
Cost/Schedule Risks				
Unforeseen Cost and/or Schedule Issues That Prevent Novel Electrochemical Technology from Being Demonstrated	Med.	Med.	Med.	The EERC can develop protocols throughout Task 3 to ensure planning items are addressed efficiently throughout project.
Technical/Scope Risks				
Novel Electrochemical Technology Not Demonstrating 90%–99.99% individually separated high purity Economically.	Med.	High	Med.	EERC experience on previous work of electrochemical demonstrations provides confidence in the fundamental REM and CM recovery; techno-economic data gaps will be gathered and addressed within Task 4.

Potential Risks and Risk Mitigation

Perceived Risk	Risk Rating (low, med., high)			Mitigation/Response Strategy
	Probability	Impact	Overall	
Management, Planning, and Oversight Risks				
Lack of Available Resources	Low	High	Low	The EERC is committed to providing the necessary personnel and facility resources to carry out project activities and will restructure workloads, if necessary, to meet project needs.
Inefficient Communication Leads to Schedule or Cost Overruns	Low	High	Med.	Regular update meetings will be held with the project team to ensure objectives are being pursued and that activities are focused on completing project milestones. Project partners will be included as needed. The planned schedule and budget will be periodically reviewed to ensure no deviations. Communication regarding progress, including any potential deviations from the planned schedule or budget, will occur with the DOE Project Manager via phone calls, e-mail, and quarterly reports.
Loss of PI, Task Lead, or Key Researcher(s) to Health Matters or Attrition	Low	High	Low	Co-PI and co-task lead will lead the project and task, respectively.

Potential Risks and Risk Mitigation

Perceived Risk	Risk Rating (low, med., high)			Mitigation/Response Strategy
	Probability	Impact	Overall	
EHS Risks				
Used Desiccant Brine and/or Precipitated By-Products Not Disposed of Through the Planned, Nonhazardous Means	Med.	Low	Low	A backup plan for using a third-party chemical waste disposal contractor will be identified.
External Factor Risks				
Hazardous Waste Disposal	Low	Med.	Low	Develop contingencies with EHS: use existing UND hazardous waste disposal contractor if required.
Ongoing COVID-19 Challenges	Low	Low	Low	Monitor the situation and its impacts; follow current guidelines.

Progress and Current Status

Task 2 – Project Teams, Process Development Criteria, Markets, and Industrial Applications

Goal to identify project teams, which will consist of subject matter experts and stakeholders from across the entire REE and CM supply chain (Deliverable D2).

- Subtask 2.1 – Feedstock Resource Review
  - Identify a feedstock source through literature review
  - Define potential REE and CM extractability and estimate quantity of resource

- Subtask 2.2 – Markets and Industrial Applications
  - Identify REE and CM markets, international production quantities, U.S. demand, and end-use products
  - Develop a PFD of circuit integration for production of REM and CM (Deliverable D3)

Progress and Current Status

Task 3 – Technical Research Plan

Task 3 will develop a technical research plan encompassing Circuits 1–4 (Deliverable D4).

- Subtask 3.1 – Circuit 1 Pilot Plan
  - Select coal-based materials as feedstock for producing MREO/MRES and CM
  - Investigate REO, RES, or CM compounds produced from MREO/MRES and/or CM
  - Address and mitigate potential impacts of trace contaminant species

- Subtask 3.2 – Circuit 2 Purification Plan
  - Design and development of advanced separation processes to produce ISHP REO/RES and CM
  - Evaluate separation techniques (i.e., SX, io chromatography, etc.) for CM purification (D5)
  - Provide a description of TEP technologies (D6)

Continued…

Progress and Current Status

Task 3 – Technical Research Plan

Task 3 will develop a technical research plan encompassing Circuits 1-4 (Deliverable D4).

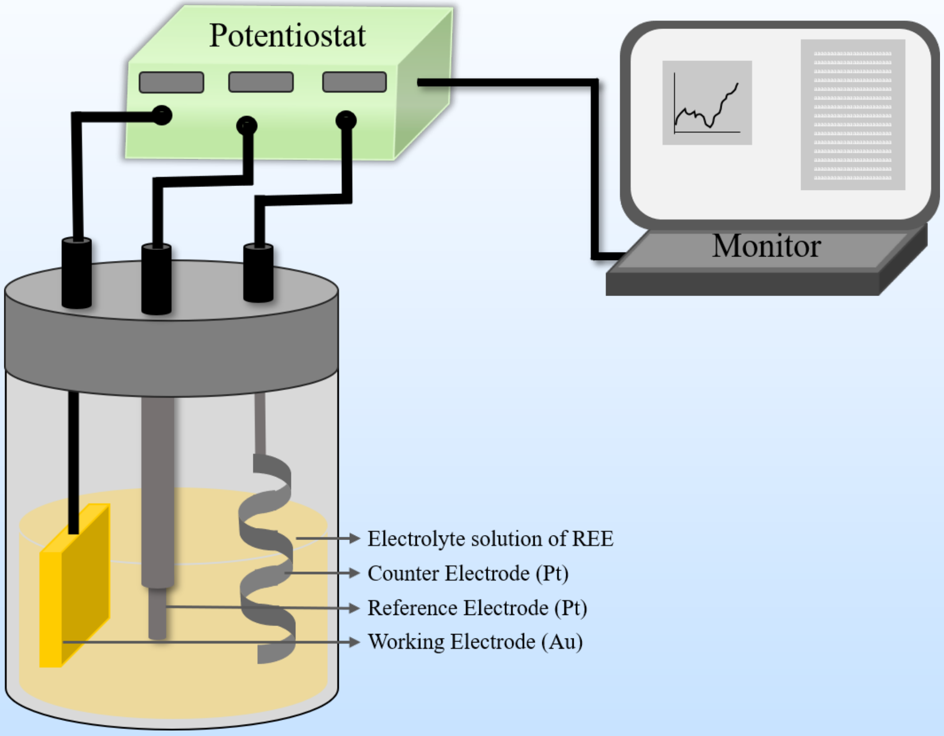
- Subtask 3.3 – Circuit 3 Reduction to Metal Plan
  - Provide summary review of SOTA techniques literature and market utilization of techniques of REM.

- Subtask 3.4 – Circuit 4 Critical Materials Plan
  - Develop plan to utilize conventional or advanced processes to convert CM, other than REE, into CM-oxides for use
  - Provide summary review of SOTA techniques literature and market utilization of these techniques in catalyst, ceramic and pigment, phosphor, battery, etc., industry

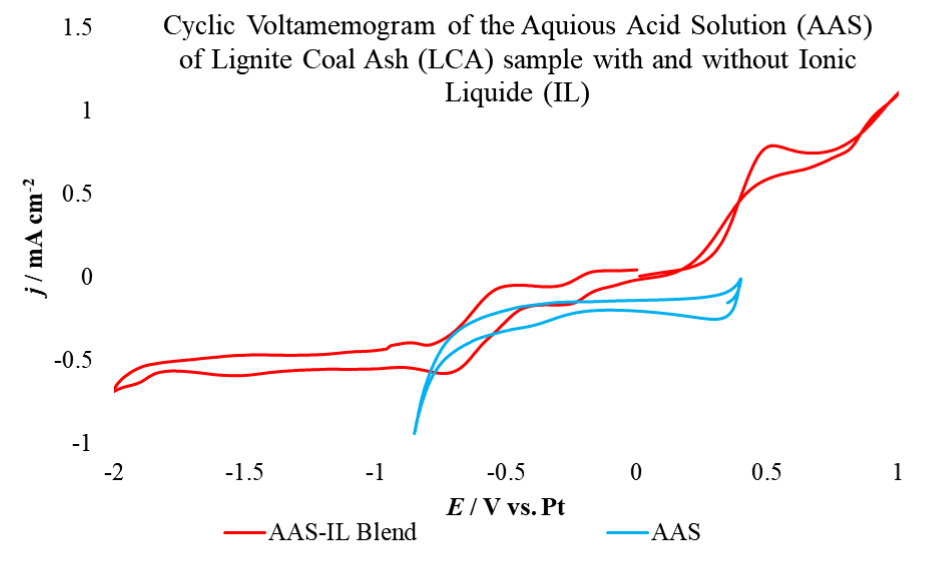
Progress and Current Status

Task 4 – Laboratory Experiments/Data Acquisition

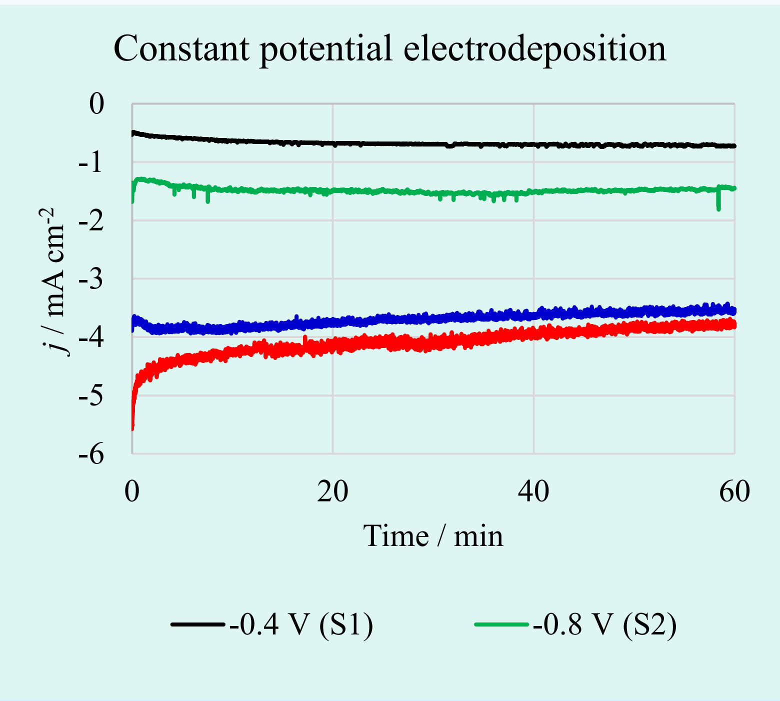
Experimental

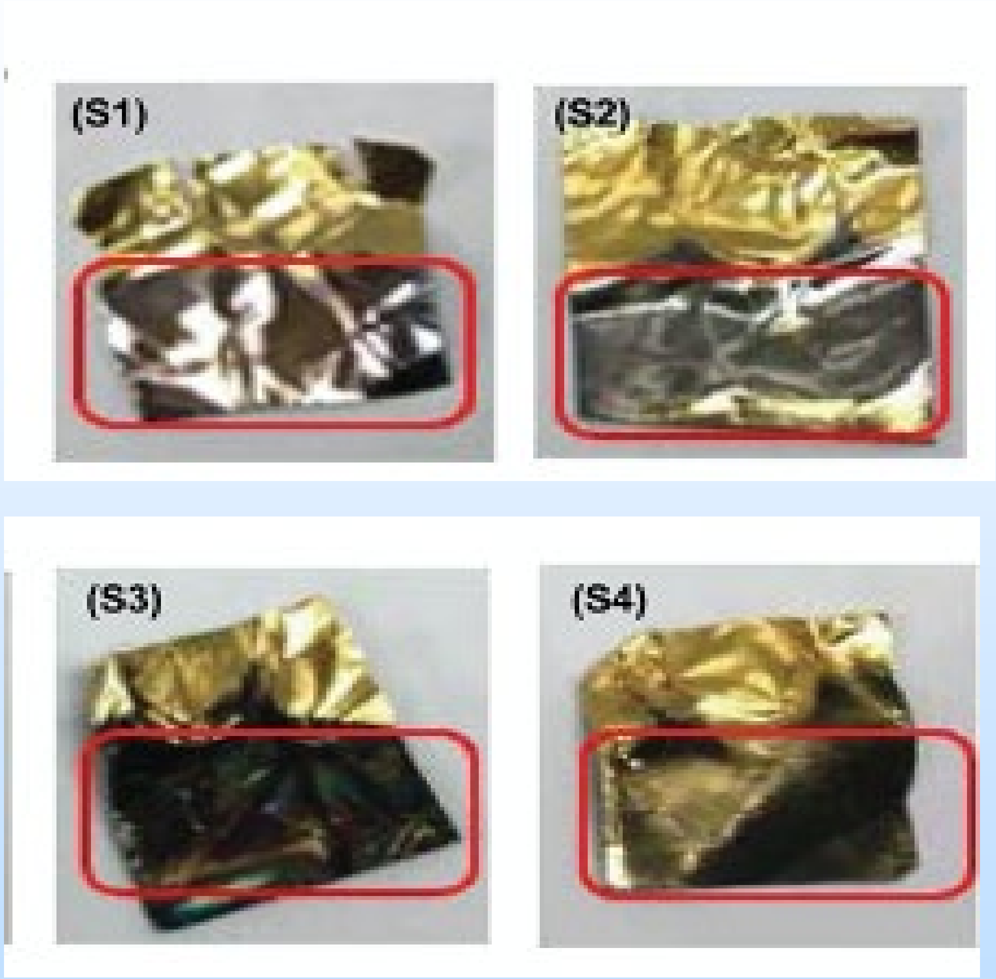


CV in REEs Containing LCA Solution



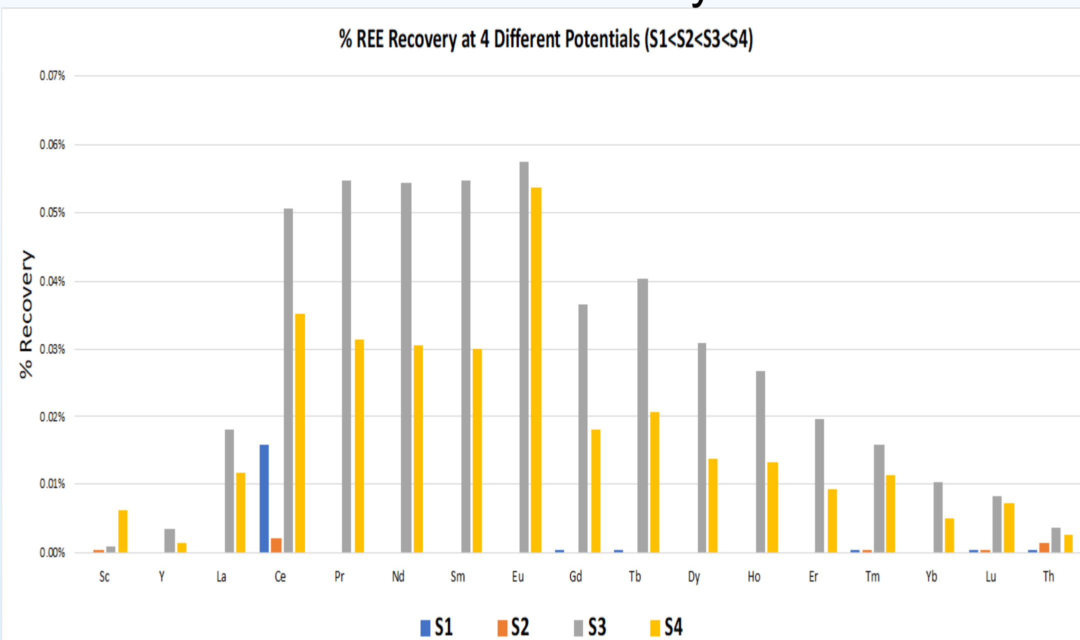
Constant Potential Hold Experiments





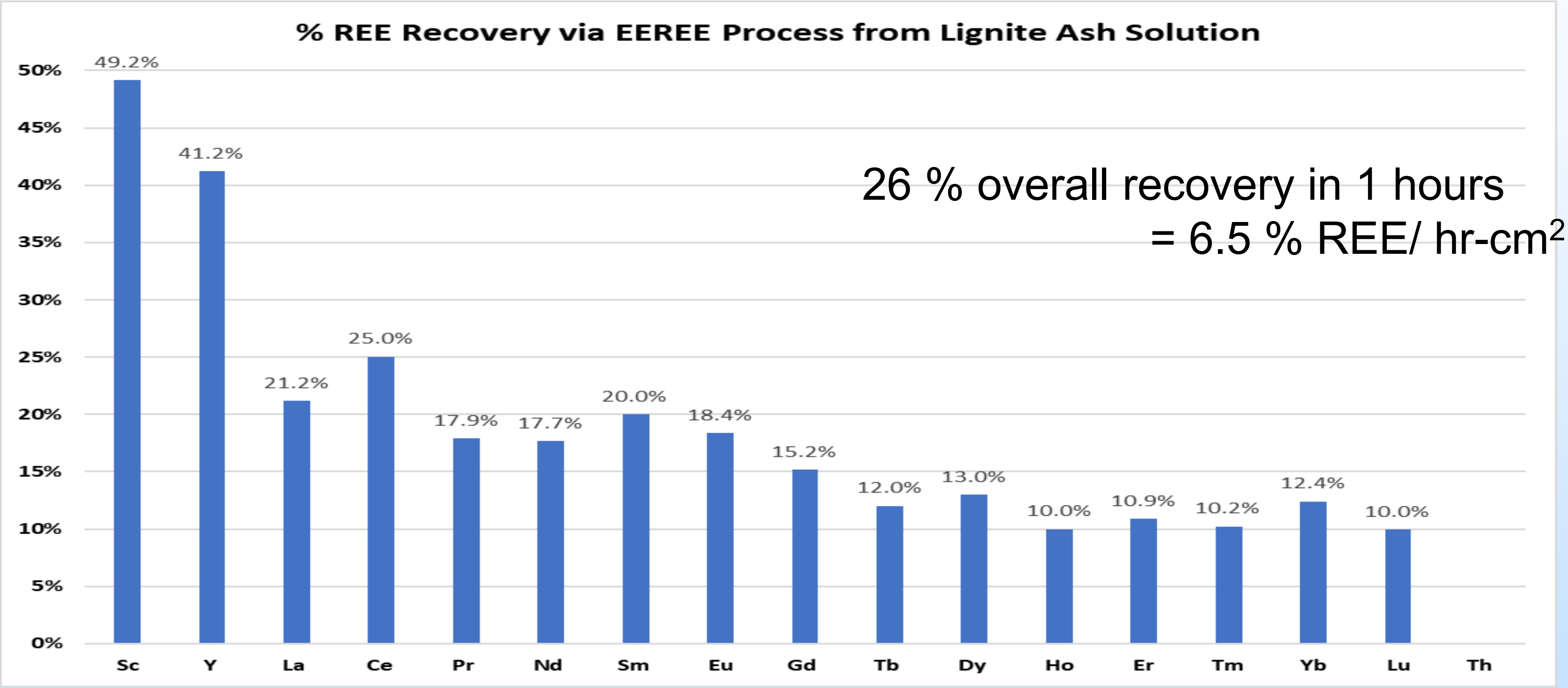
Proof of Concept Testing

ICP-MS Analysis

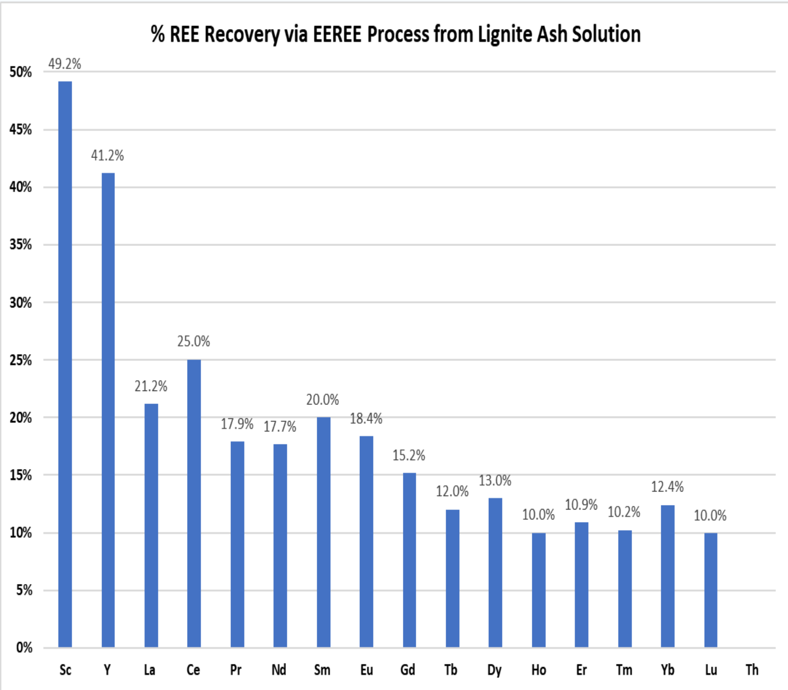


Before and after the digestio

REEs Recovery from LCA Solution



REEs Recovery from LCA Solution



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Sc	$Sc^{3+} + 3e^-$	Sc (s)	-2.08	3	7
Y	$Y^{3+} + 3e^-$	Y(s)	-2.37	3	4
Ce	$Ce^{3+} + 3e^-$	Ce(s)	-2.34	3	1
La	$La^{3+} + 3e^-$	La(s)	-2.38	3	4
Sm	$Sm^{3+} +3e^-$	Sm(s)	-2.30	3	1
Eu	$Eu^{3+} +3e^-$	Eu(s)	-1.99	3	1
Pr	$Pr^{2+} + 2e^-$	Pr(s)	-2.00	2	1
Nd	$Nd^{3+} +3e^-$	Nd (s)	-2.32	3	1
Gd	$Gd^{3+} +3e^-$	Gd(s)	-2.28	3	1
Dy	$Dy^{3+} +3e^-$	Dy^{2+}	-2.60	3	1
Yb	$Yb^{3+} +3e^-$	Yb(s)	-2.19	3	1
Tb	$Tb^{3+} +3e^-$	Tb(s)	-2.28	3	1
Ho	$Ho^{3+} +3e^-$	Ho(s)	-2.33	3	1
Er	$Er^{3+} +3e^-$	Er(s)	-2.33	3	1
Tm	$Tm^{3+} +3e^-$	Tm(s)	-2.32	3	1
Th	$Th^{4+} +4e^-$	Th(s)	-1.90	4	1
Th	$Th^{4+} +e^-$	Th^{3+}	-3.60	1	2

Plans for future development

Develop a technical research plan encompassing Circuits 1–4

Summary Slide

a. TEP could be used to extract REEs and ISHP REEs and CM
b. This project is an essential step toward reestablishing a complete domestic REE/CM value chain based on Williston Basin lignite coal- A resource with an estimated life span—at current utilization rate—of 800 years.

ACKNOWLEDGMENTS AND DOE DISCLAIMER

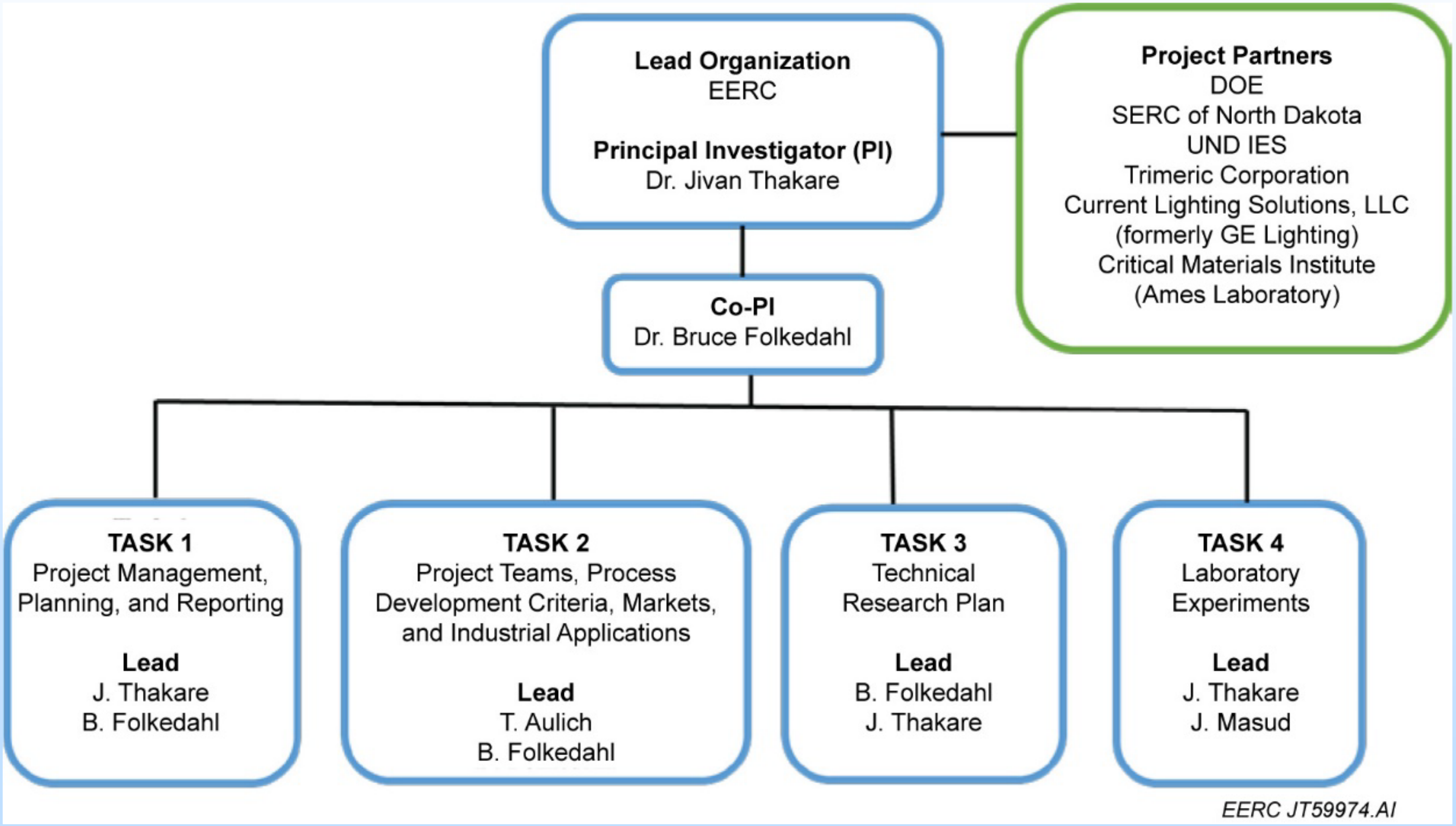
Acknowledgments:

- Special thanks to U.S. Department of Energy (DOE) Project Manager Mark Render and DOE program management team.
- This material is based upon work supported by DOE NETL (National Energy Technology Laboratory) under Award Nos. DE-FE0032121.

Appendix

– These slides will not be discussed during the presentation but are mandatory.

Organization Chart



Organization Chart

Current Team Member	Technical Research Plan	Primary Role
EERC (prime recipient)	Circuits 1–4	Lead Technical Research Plan and project
SERC	Circuits 1–4	Provide cost share
UND IES	Circuit 1	Lignite coal-based source material provider
Trimeric (Subcontractor)	Circuits 1–4	Perform techno-economic and life cycle data gaps and assessments
Current Lighting Solutions, LLC (Subcontractor)	Circuits 3,4	Manufacturing process assessment of REE/CM
Critical Materials Institute (Ames Lab) (Subcontractor)	Circuits 3,4	Manufacturing process assessment of REE/CM