Characterization Inform Sustainable Recovery of Critical Minerals from Fossil Energy Waste Feedstocks

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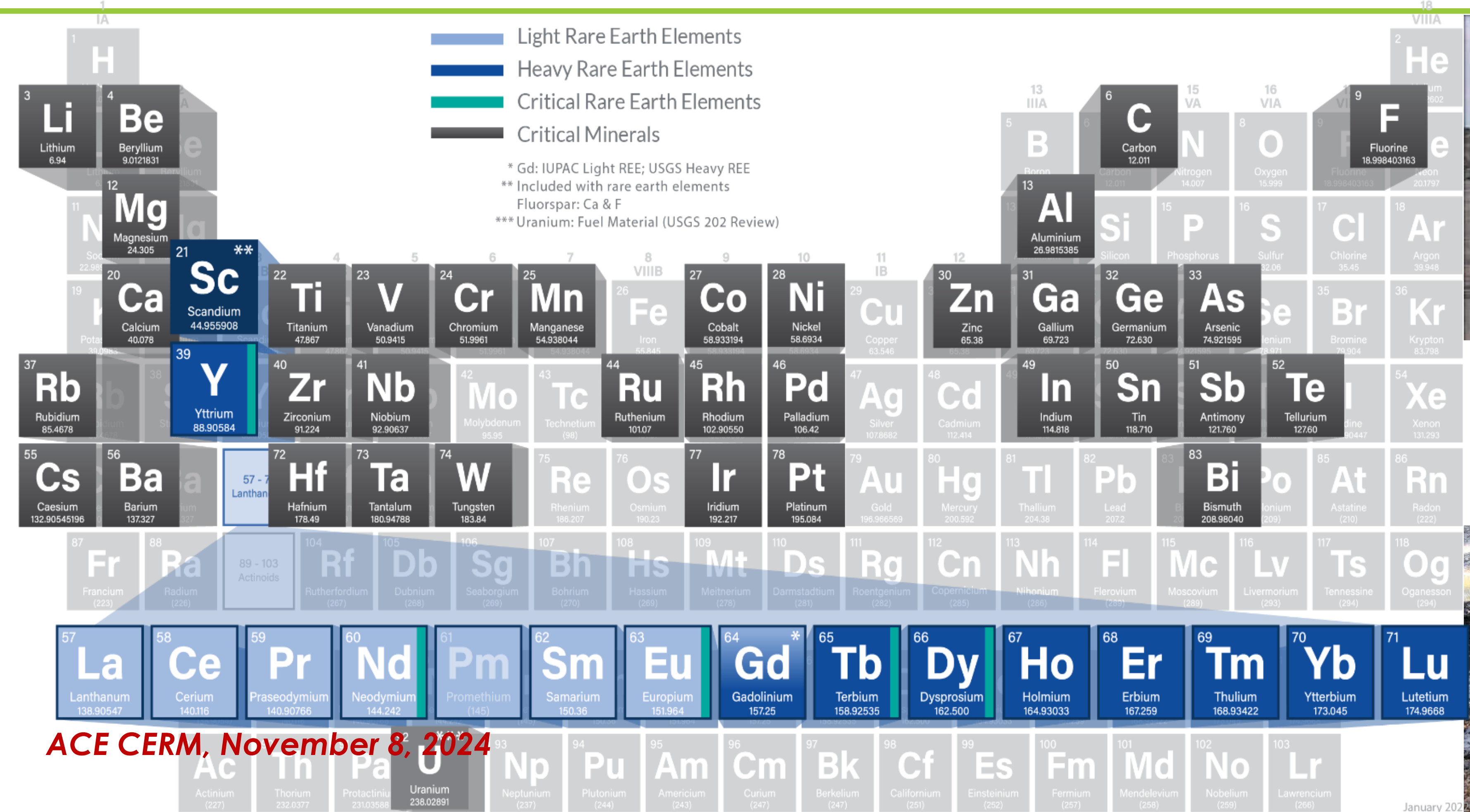
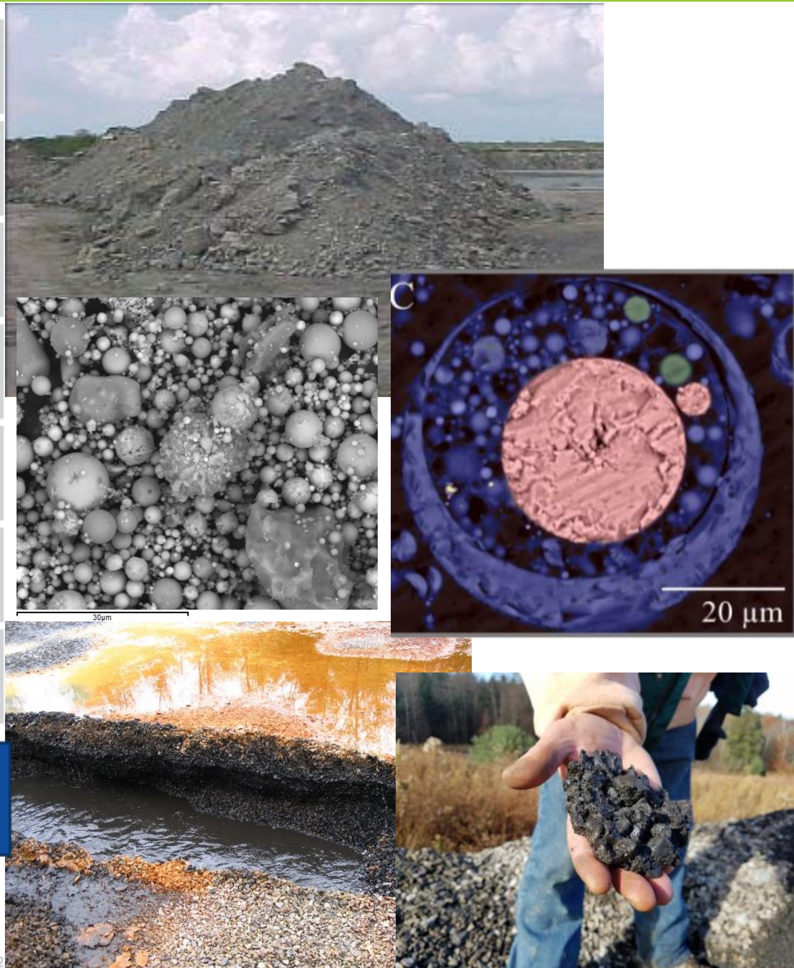


ACE CERM, November 8, 2024



Critical Minerals & Rare Earth Elements (REE)

NATIONAL ENERGY TECHNOLOGY LABORATORY

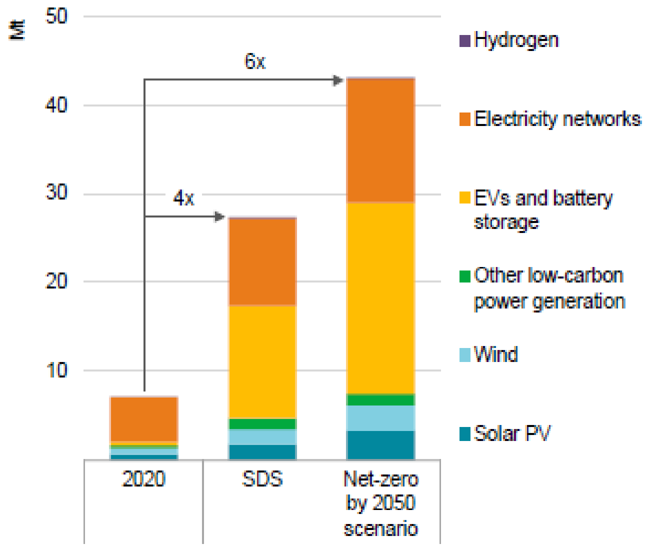
Examples of Critical Minerals Used in Advanced Technologies

Solar panels – Arsenic, Germanium, Indium, Tellurium	**Battery storage –** Cobalt, Graphite, Lithium, Manganese	**Wind turbines –** Aluminum, Rare Earth Elements	**National defense –** Chromium, Gallium, Scandium	**Aviation –** Niobium, Tantalum, Vanadium

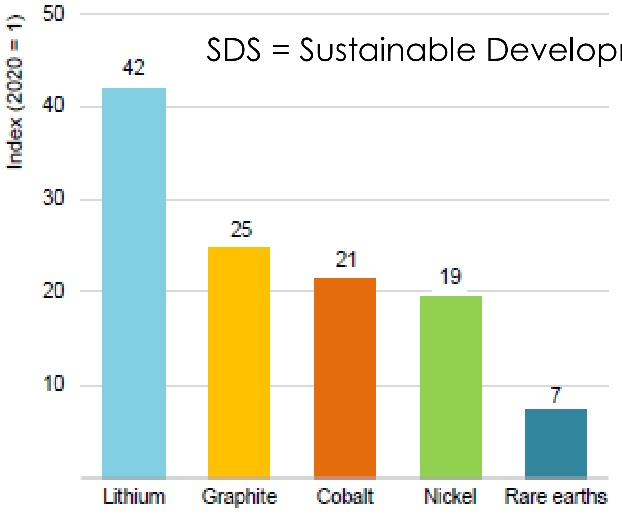
Challenge: Clean Energy Technologies Drive Demand Growth

Mineral demand for clean energy technologies by scenario

Growth to 2040 by sector



Growth of selected minerals in the SDS, 2040 relative to 2020



SDS = Sustainable Development Scenario

Notes: Mt = million tonnes. Includes all minerals in the scope of this report, but does not include steel and aluminium. See Annex for a full list of minerals.

In less than 20 yrs we'll need 5-50x our current CM supplies

Source: https://www.gao.gov/blog/critical-mineral-shortages-could-disrupt-global-supply-chains
https://www.iea.org/reports/the-role-of-critical-minerals-in-clean-energy-transitions/executive-summary

DOE- FECM Critical Mineral Goal

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Where do we get it? and What do we produce?

DOE Fossil Energy Carbon Management Goal:

By 2035, enable unconventional and secondary sourcing for half of the domestic critical mineral needs

- **Demonstrate sufficient resources from domestic unconventional and secondary (U&S) feedstocks, including Fossil Energy wastes (solid and liquid)**
- **Demonstrate economically competitive and environmentally sustainable extraction and processing technologies**

NETL Critical Mineral MYRP

- National prospective Database (Goal 1)
- Resource Characterization (Goal 2)
- Technology Deployment Decision Support Tool (Goal 3)
- Advanced Critical Mineral and Carbon processing (Goal 4&5)
- In-situ recovery using fossil energy infrastructure (Goal 6)

Challenges

U&S feedstocks (solid & liquid)

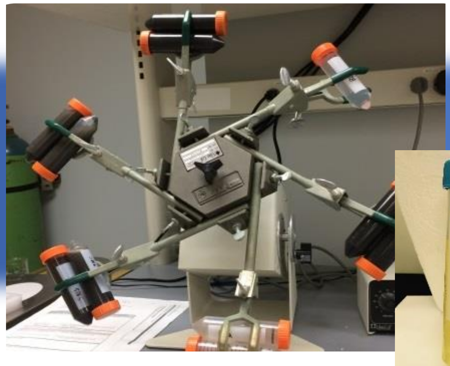
- Low concentrations
- No domestic supply chain (feedstock – end products)
- Which is the best?
- Heterogeneity
- Sustainability

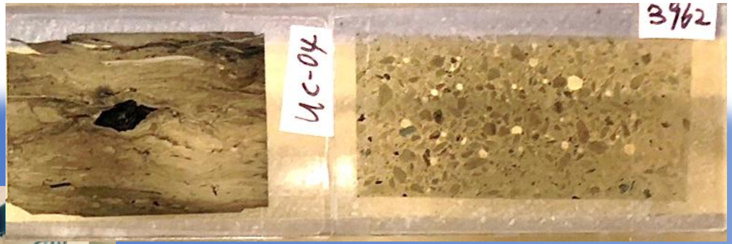
Research Approach

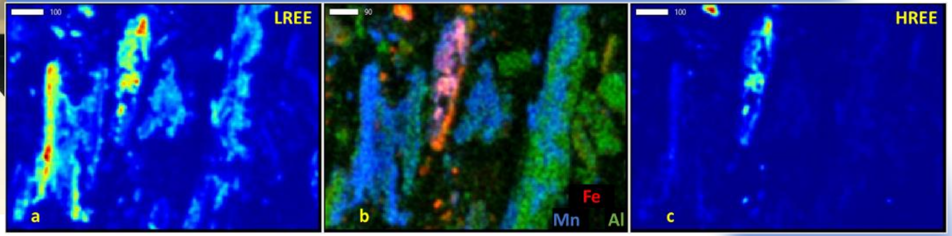
Characterization to Recovery

AMD solids

Fly ash

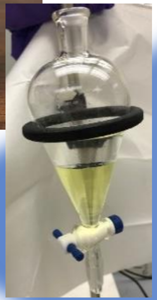


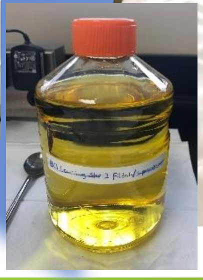


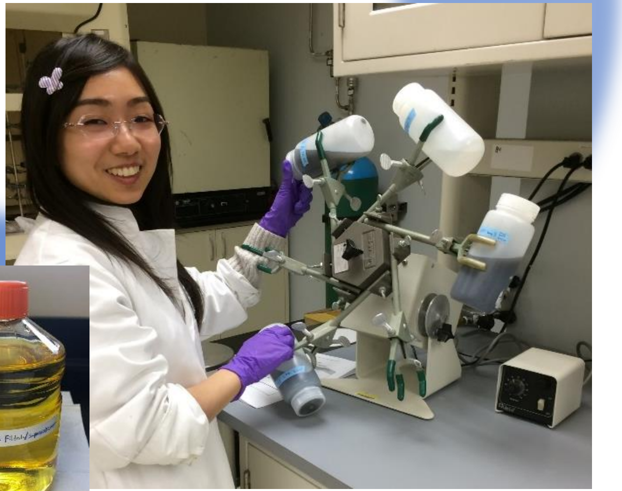


Utilize sequential extraction techniques to **characterize** major CM-hosting solid fractions in different CCBs and to **innovate targeted extractions** for efficient and economical CM recovery.

- A workflow to identify REE & CM host phases & binding environment
- Bulk Chem, Titrations, Sequential Extractions
- Advanced Characterization & Identify targets and Lixiviant
- Selective Extraction Processes Optimize Extraction Efficiency



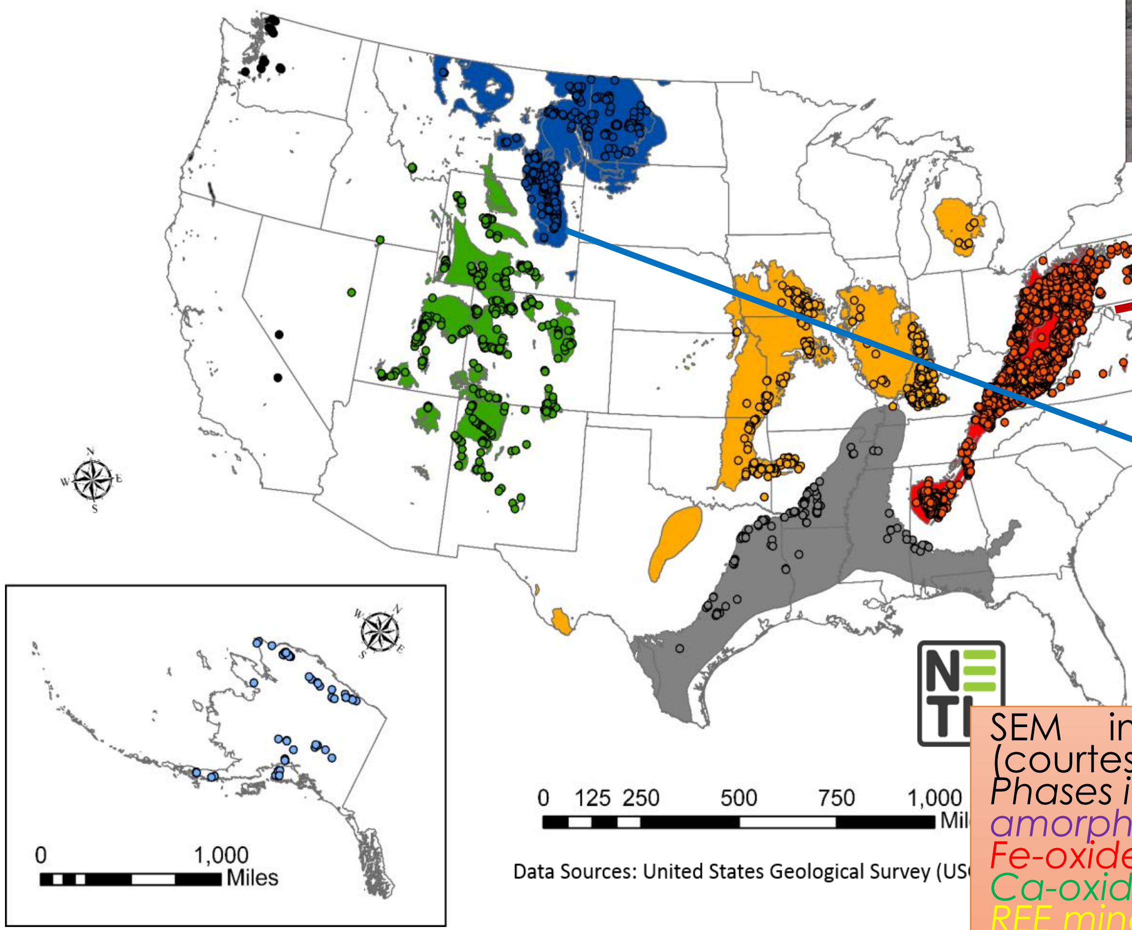




#1: Coal Combustion Ash Wastes as REE Resources

Coal ash: ets. 8,910 REE tons/year, 95% of REE demand in 2018[1]

45 million tons of fly ash in impoundments

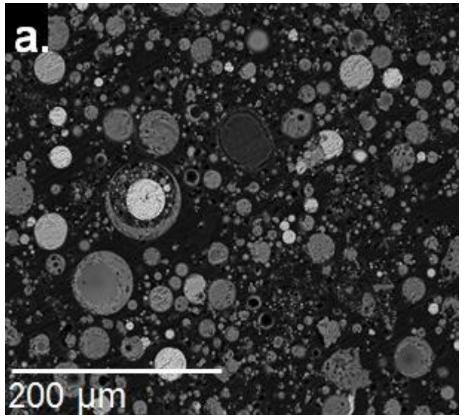


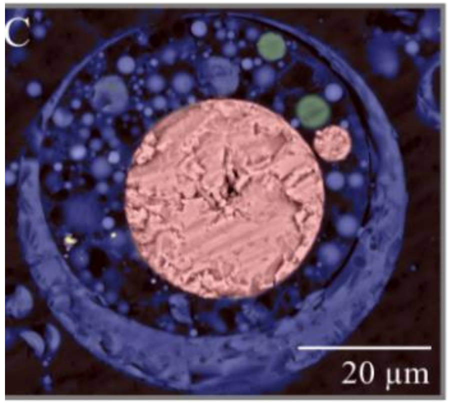
Data Sources: United States Geological Survey (US

SEM image of fly ash (courtesy of Scott Montross)
Phases identified:
amorphous Si-Al -purple
Fe-oxide - red
Ca-oxide - green
REE mineral - yellow

Appalachian basin (APP) coal ash: 5.5 M tons/yr, Al-rich

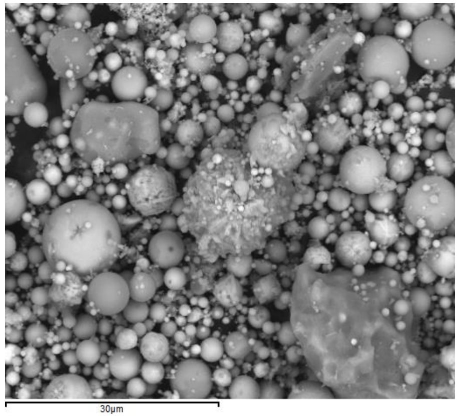
REE encapsulated in AlSi-rich spherical glass

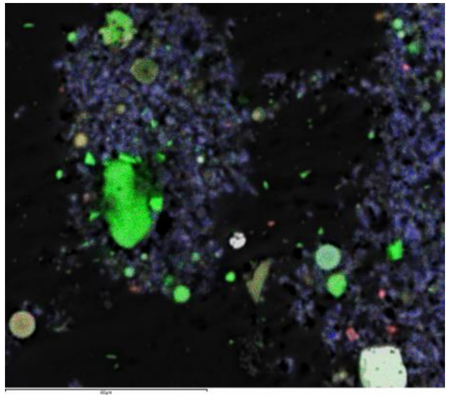




Powder Riven basin (PRB) coal ash: 8.9 M tons/yr, Ca-rich

Amorphous particles with Ca-oxide crust





Synchrotron characterization for Ash Samples

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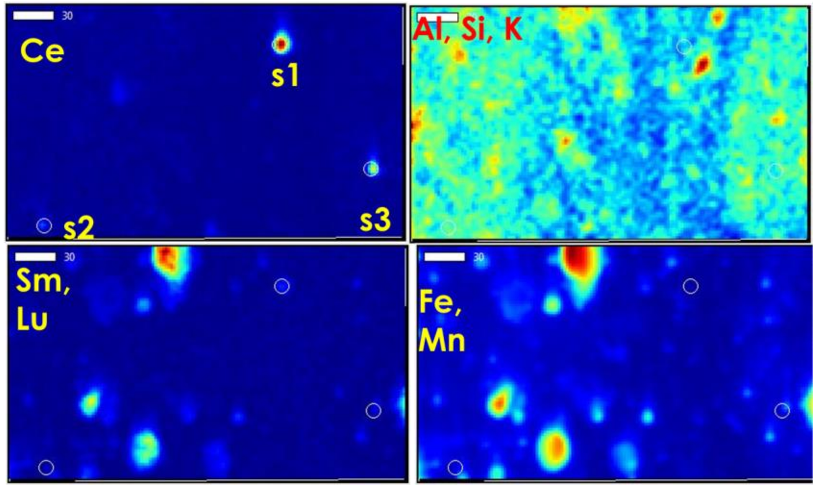
Appalachian Basin Ash (APP) vs Powder River Basin Ash (PRB)

APP coal ash: 5.5 M tons/yr, Al-rich,
306ppm – 797ppm REE, 3-5%wt Ca[1]

- **Bulk Ce XANES: 95%-100% CeIII**



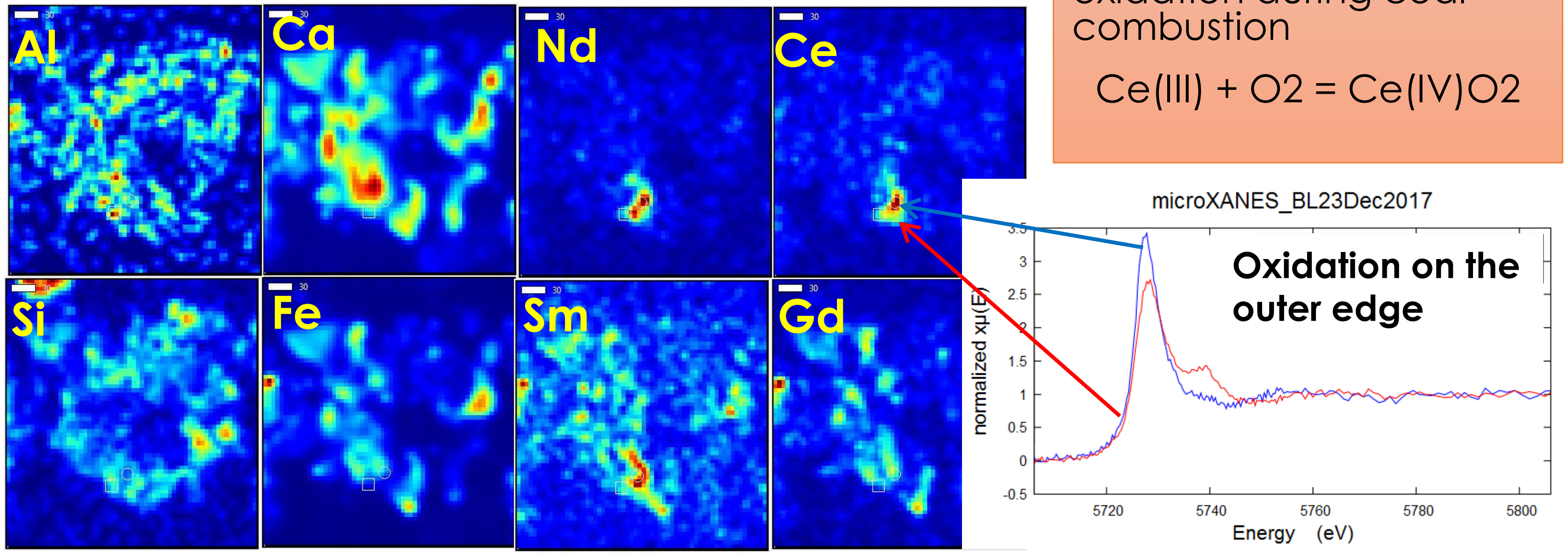
REE encapsulated in AlSi-rich spherical glass, difficult to extract

PRB coal ash: 8.9 M tons/yr, Ca-rich, **24-266 ppm REE, 16.3% - 20.8% Ca[2]**

- **Coal & Fly Ash – 95% Ce(III), < 5% Ce(IV)**
- **Bottom Ash & Ponded Ash ~ 10-30% Ce(IV)**

During coal combustion, Ce diffused into Ca glassy phases: Ce oxidation during coal combustion

$Ce(III) + O2 = Ce(IV)O2$



Amorphous particles with Ca-oxide crust

Light REEs (e.g., Ce, Nd) diffused into Ca-rich AlSi; partial Ce oxidation indicates reactivity; heavy REEs (e.g., Sm, Gd) w/ Fe

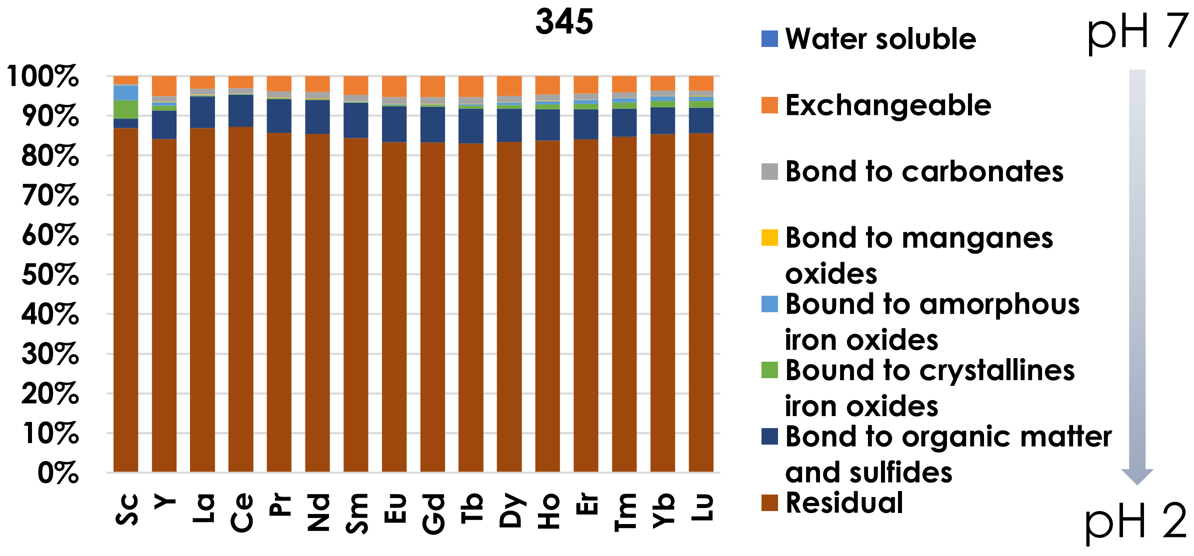
1: M.Y. Stuckman, , et al., IJCG 195 (2018) 125-138.
2: M.Y. Stuckman, et al., (2019) International Pittsburgh Coal Conference

Sequential Extraction for Characterization

Appalachian (APP) Ash versus Powder River Basin (PRB) Ash

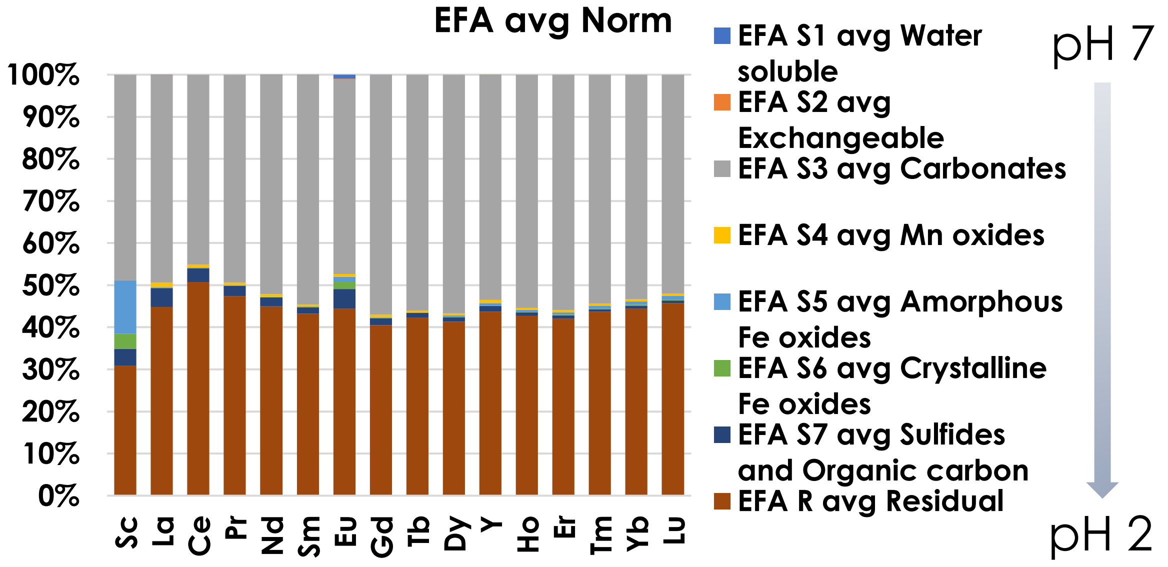
APP Fly Ash 345 (665 ppm REE)[1]

- derived from Appalachian Basin coal (4%wt Ca)
- REE associated mainly with Residual phase(aluminosilicates)



PRB-FA (264ppm REE+Y)[2]

- derived from Powder River Basin coal (20%wt Ca)
- >60% REE released in "carbonate" phase extraction



1: Lin, R. et al (2018) Fuel 232, 124-133;
2: M.Y. Stuckman, et al., (2019) International Pittsburgh Coal Conference

Targeted Rare Earth Extraction (TREE)*

Targeting Calcium-Rich Ashes and AMD solids

TREE Advantages over REE mining:

1. Domestic/ local waste feedstocks
2. No-pretreatment
3. Up to 90% acid reduction
4. No heating/ no pressure
5. No solvent use or reduced solvent use
6. Less waste management cost
7. Additional value streams such as lithium, cobalt, nickel or scandium

2022 R&D 100 FINALIST

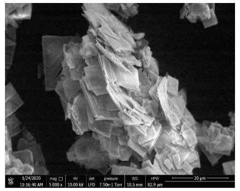
Weak Acid 1 → Step 1 Calcium removal → Calcium-rich leachate

Weak Acid 2 → Step 2 REE extraction → REE leachate → Purify and precipitate → REE products

Weak Acid 3 → Step 3 Lithium, Cobalt or Scandium extraction → Lithium, Cobalt or Scandium leachate → Lithium, Cobalt or Scandium products

AMD solids Fly ash TREE process

Clean REE leachate



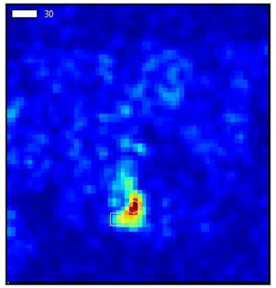
*Stuckman, M.Y., Lopano, C.L. and Tarka, T. (2021) U.S. Patent Pending, Serial No.: 63/053,925

https://netl.doe.gov/node/10318

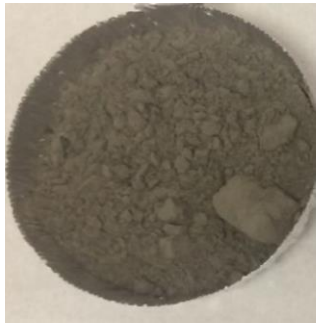
https://www.youtube.com/watch?v=quzSKK4WB2Q&list=LL&index=15

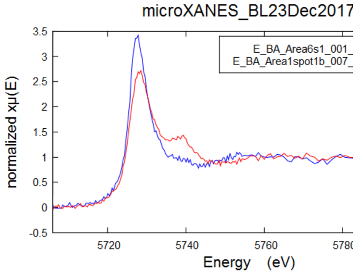
Recovery from Calcium-Rich Ash

Targeting Powder River Basin (PRB) Ashes to Reduce Extraction Steps & Conditions



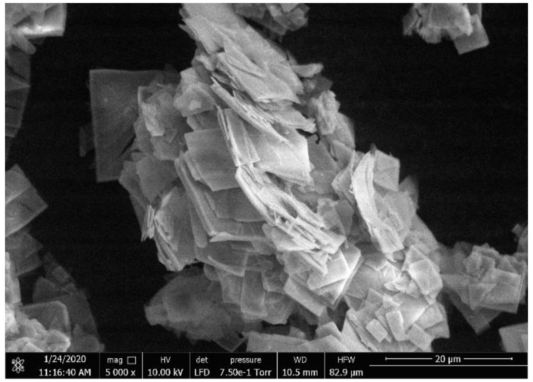
PRB Fly Ash

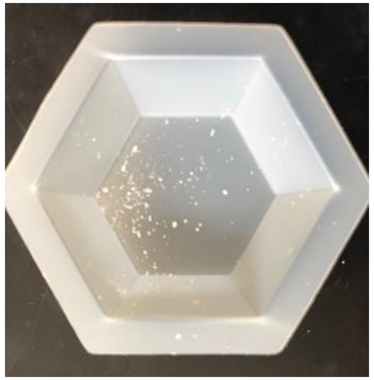




U.S. Patent Pending Serial No.: 63/053,925

96wt% Pure Rare Earth Oxide (REO)





From Bench to Pilot: $1.6 million Technology Commercialization Fund Project

- University of Wyoming School of Energy Resources
- Campbell County
- City of Gillette

State, Campbell County pursue rare earth opportunities

By Greg Johnson, Gillette News Record | Via Wyoming News Exchange Jul 5, 2020 Comments OPEN ACCESS

Rare Earth Elements Project Receives Federal Funding

NEWS DIRECTOR | Article Updated: June 23, 2020 | COMMENTS OFF

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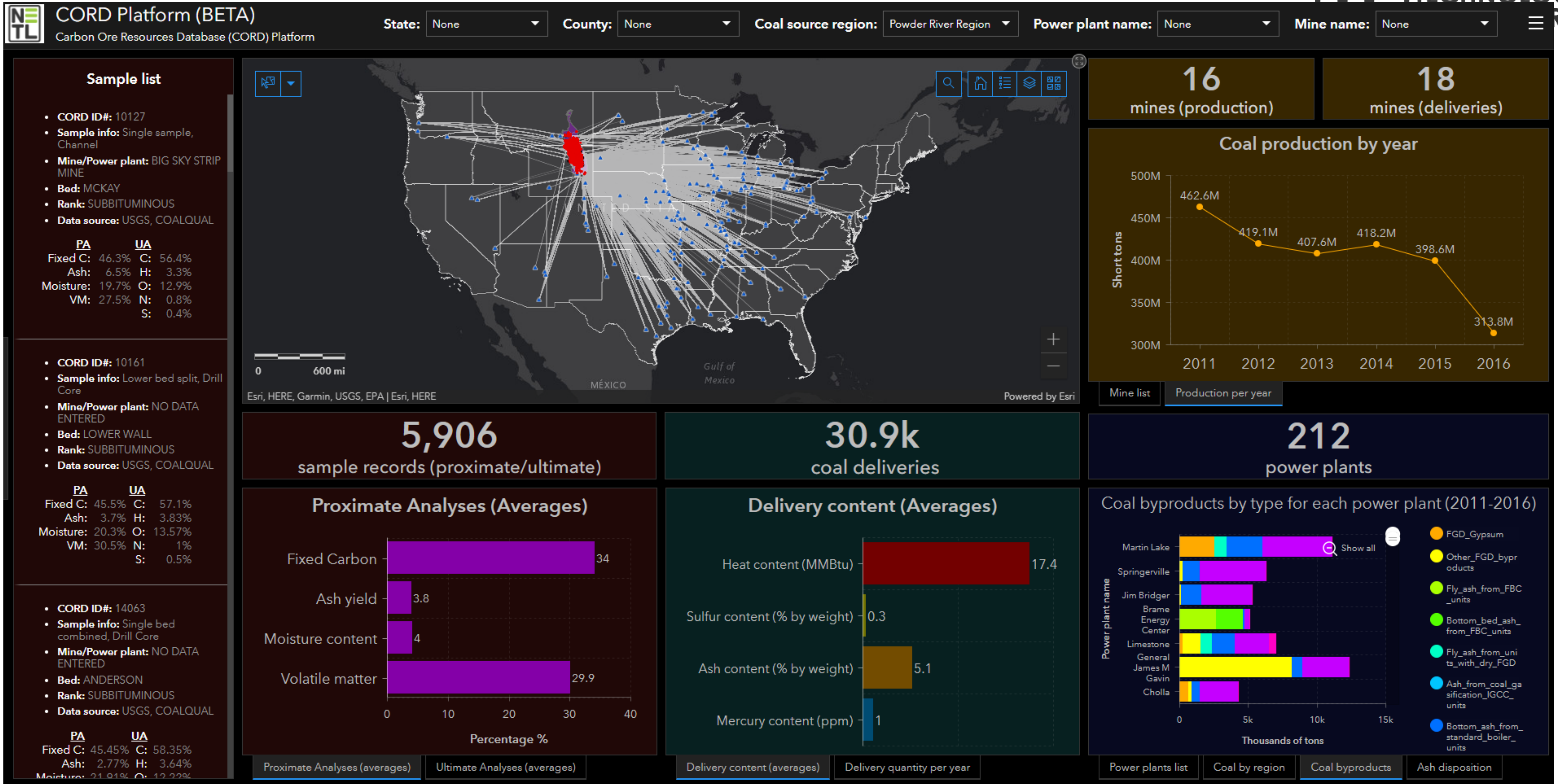
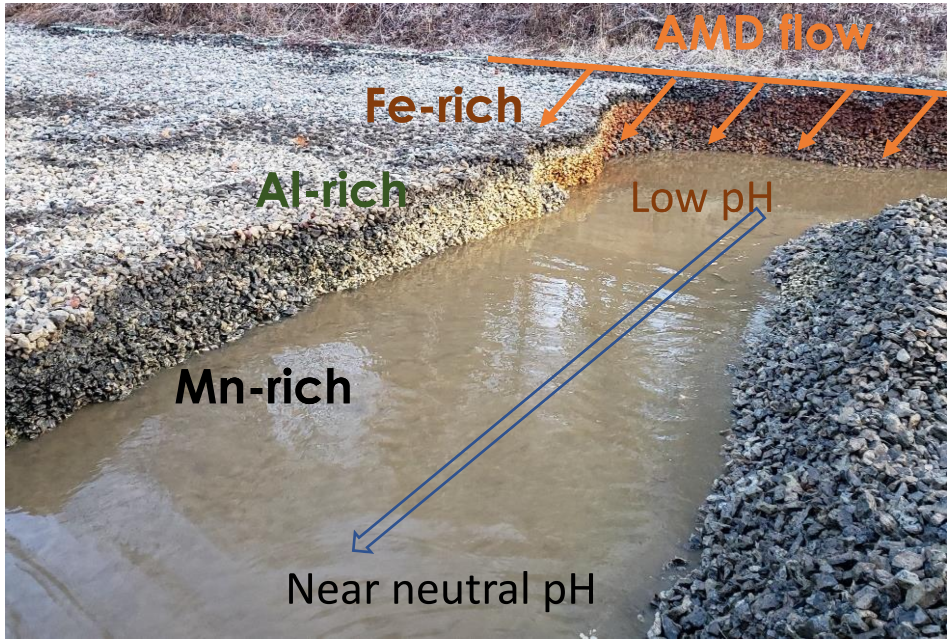
NETL Data Analytics: PRB coal burned across the nation



Justman et al (2022) https://doi.org/10.1016/j.dib.2021.107761

#2: CM in Acid Mine Drainage (AMD) Treatment Systems



Passive Remediation Treatment: No chemical added, >200 systems in PA

~85 billion gallons/year AMD treated

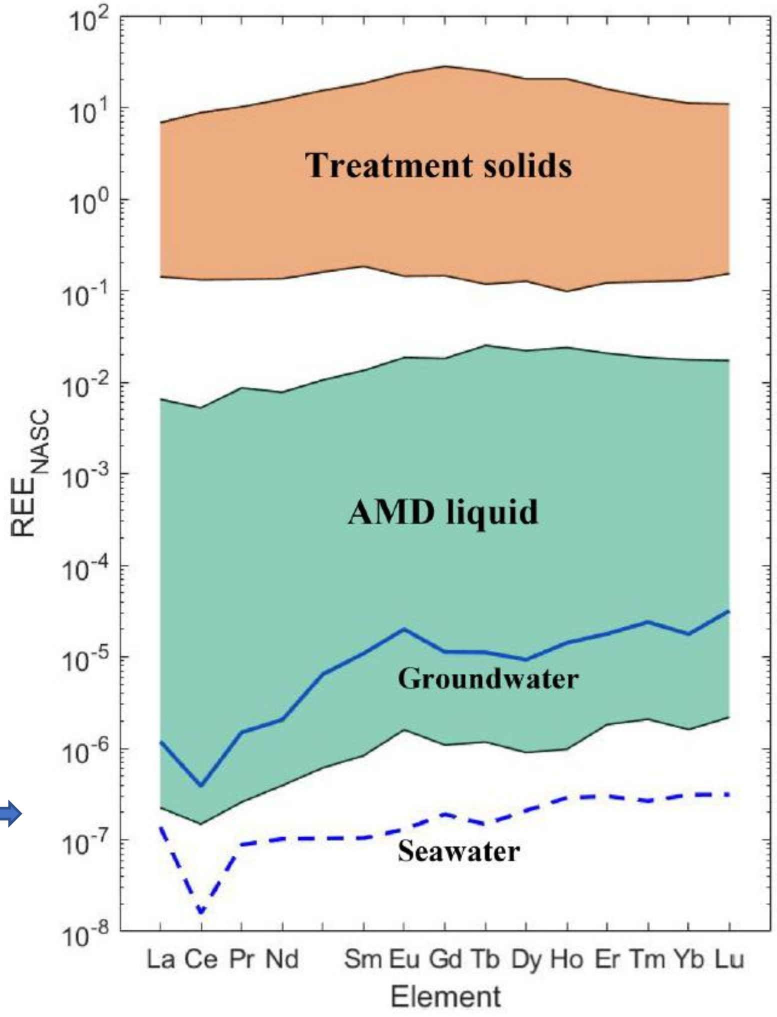
Raises pH of water (Limestone beds)

Precipitate dissolved metals

- 90% REE sequestration rare earth element (REEs) precipitate with Fe, Mn, Al

~18,000 tons/year treatment solids produced: AMD solids

1,102 tons / year REO recovery potential (~40% US annual demand)



Hedin et al. (2019 & 2020), IJCG

Diverse chemical composition of AMD Solids

Unit: wt% for major elements and mg/kg for trace elements

	C	S	Al	Si	Fe	Mn	Mg	Ca	K	Ti	REY	Li	Co	Ni	Cu	Zn	Sr	Ba
Al rich solid	2%	2%	**18.0%**	19.3%	2.1%	0.1%	0.2%	1.2%	0.7%	0.3%	**1113**	38	22	50	106	315	133	216
MnCa rich solid	4%	ND	3.5%	6.1%	0.5%	**18.1%**	0.6%	16.8%	0.4%	0.1%	**1590**	108	6026	8889	89	13585	212	151
AlMnFe rich solid	1%	1%	**15.4%**	9.7%	**5.2%**	**8.5%**	0.2%	2.8%	0.3%	0.1%	**1900**	440	2059	3002	518	5812	53	100

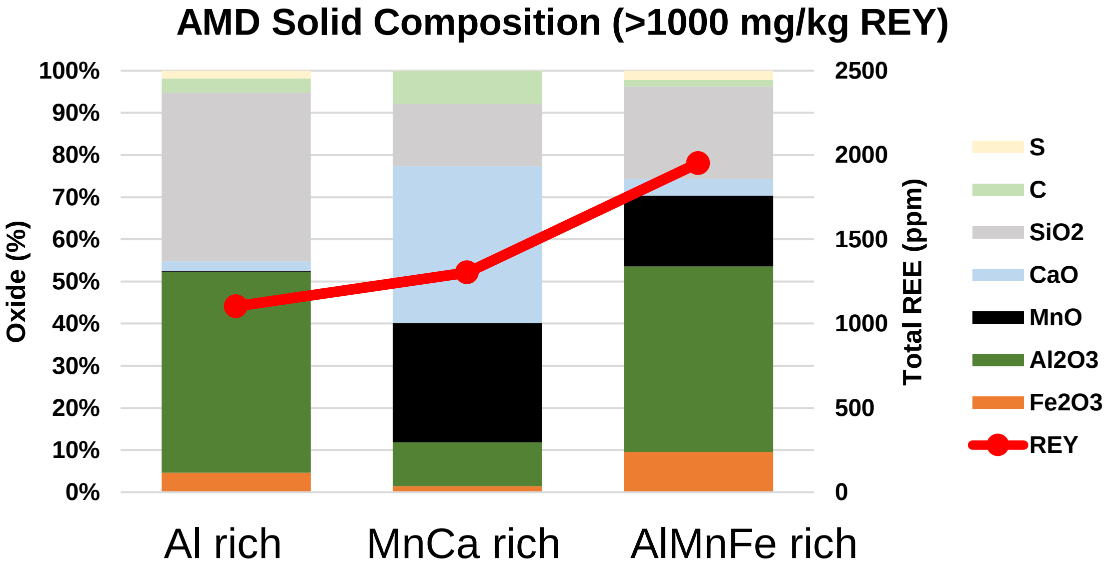
The transition metal contents are sometimes higher than REY; Lithium content is also reasonably high

MnCa-rich solid has higher accumulation of Co, Ni and Zn

Al-rich solids

MnCa-rich solid

Al-, Mn-, Fe-rich solid



Hedin et al., IJCG, 2019; 2020

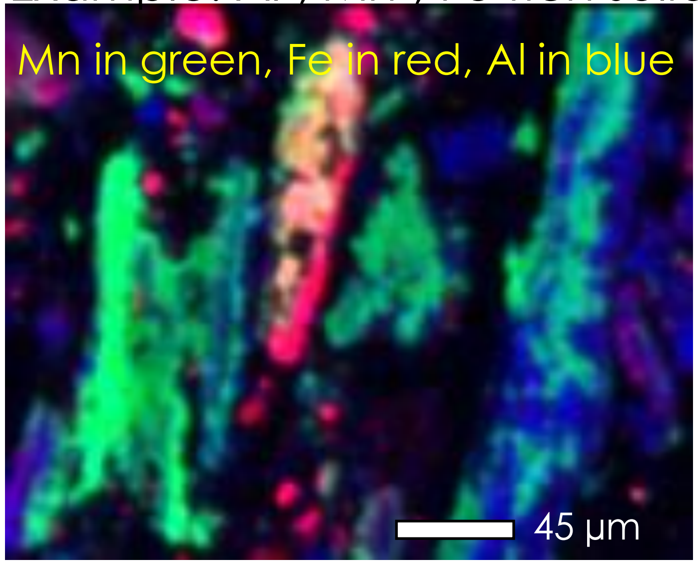
Characterization Summery

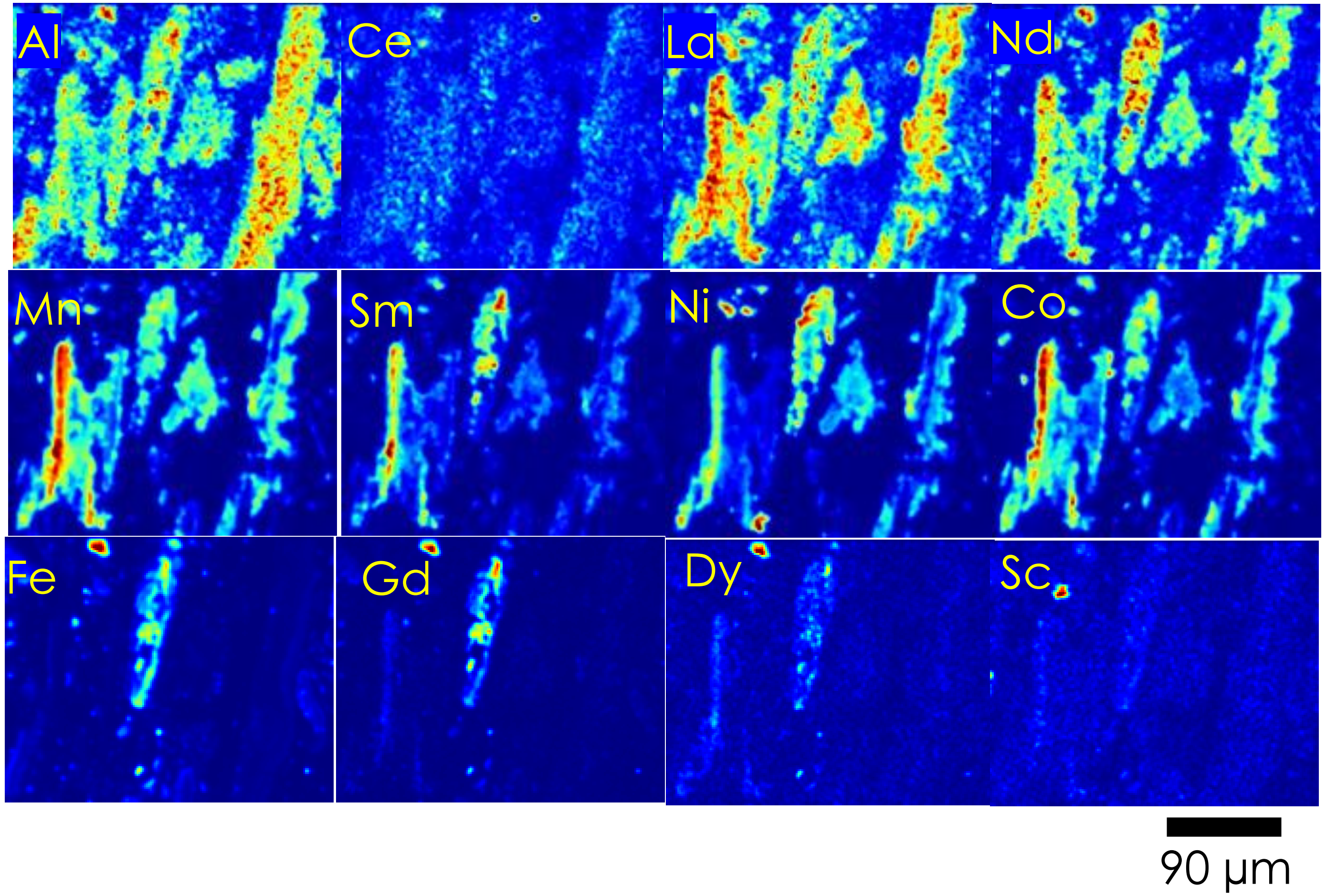
AMD solids have diverse chemical composition, so extractions may need to be tailored.

REEs Co-localized with Al and Mn, selected heavy REEs (Gd,Dy) co-localized with Fe

Co, Ni, Zn co-localized with Mn

Example: Al-, Mn-, Fe-rich solid



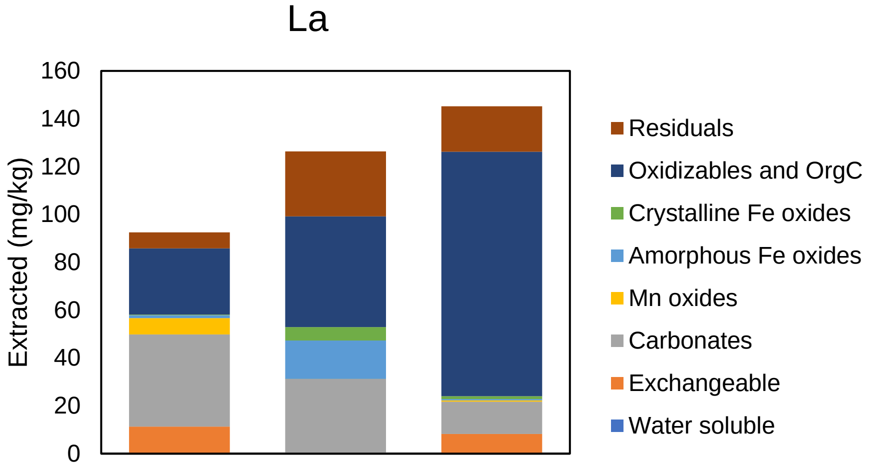


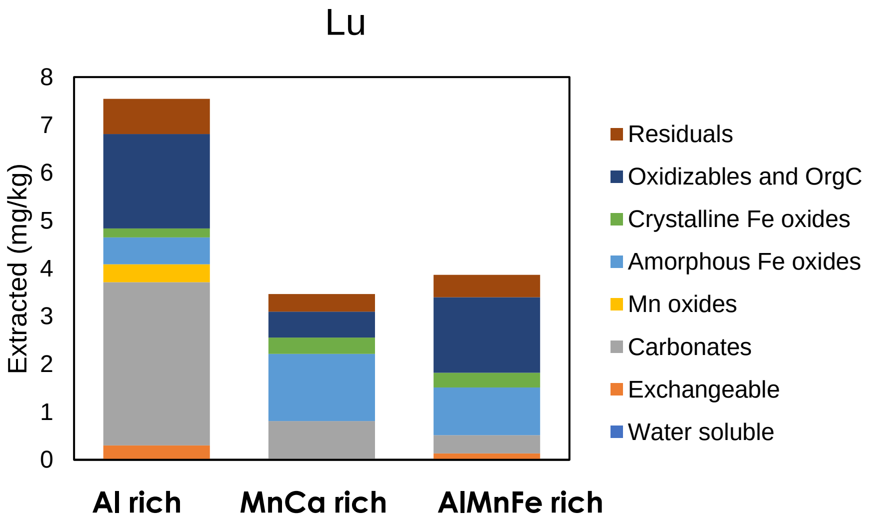
Hedin et al., Chemosphere, 2024

REE in Sq Ex and Geochemical Modeling

More than 80% REY (Al/Mn) extracted

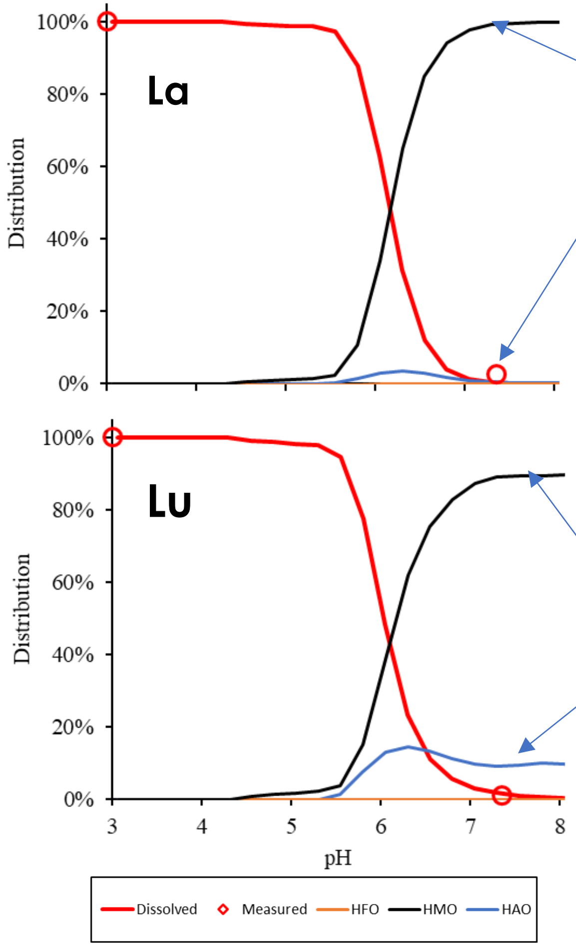
Heavy REE (Lu) extracted in Amorphous Fe oxides phases compared to LREE (La)





PHREEQ-N-AMDTreat developed by C. Cravotta (2022)

Different sorption for La and Lu



100% precipitated at effluent pH Adsorbed mainly on MnO_2 (HMO)

100% precipitated at effluent pH Adsorbed mainly on Mn hydroxides (HMO) and Al hydroxides (HAO)

https://code.usgs.gov/water/phreeq-n-amdtreat

U.S. DEPARTMENT OF ENERGY

Hedin et al., ICARD, 2022; Hedin et al., Chemosphere, 2024

Targeted Rare Earth Extraction (TREE)*

NATIONAL ENERGY TECHNOLOGY LABORATORY

Targeting Calcium-Rich Ashes and AMD solids

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AMD solids Fly ash TREE process

Clean REE leachate

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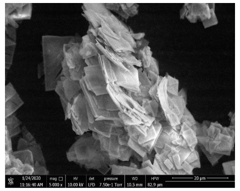
Weak Acid 2 → Step 2 REE extraction → REE leachate → REE products

Weak Acid 3 → Step 3 Lithium, Cobalt or Scandium extraction → Lithium, Cobalt or Scandium leachate → Lithium, Cobalt or Scandium products

Purify and precipitate



https://www.youtube.com/watch?v=quzSKK4WB2Q&list=LL&index=15

Progress: Downstream Optimization-Bypass Solvent Extraction

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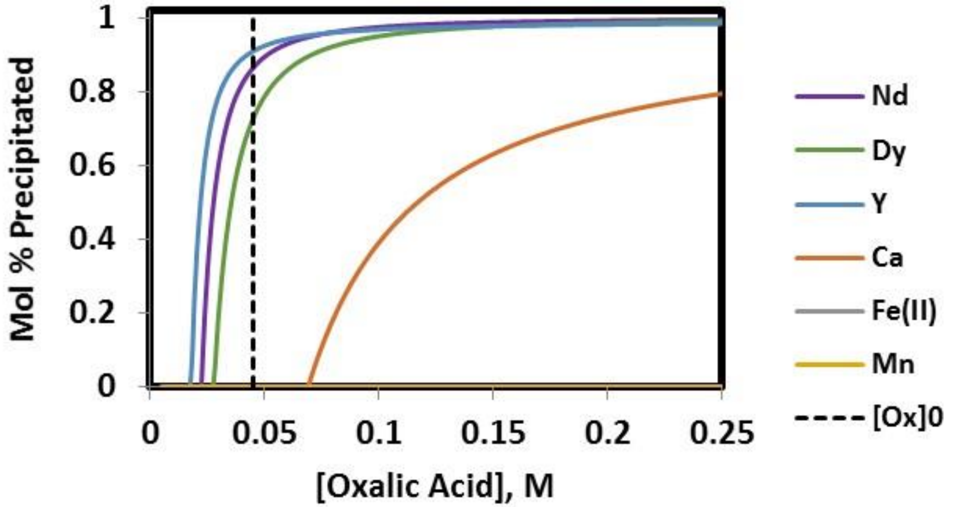
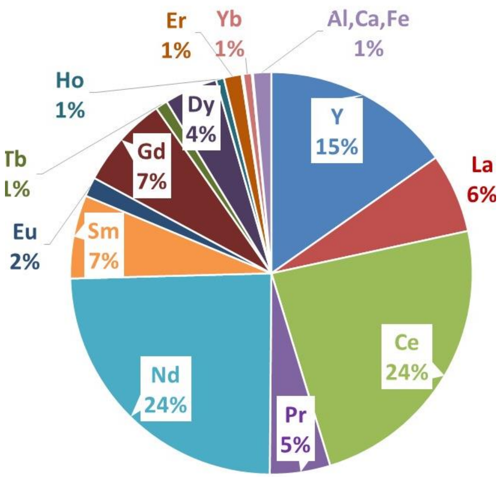
Direct Oxalic Precipitation from Select TREE Leachate

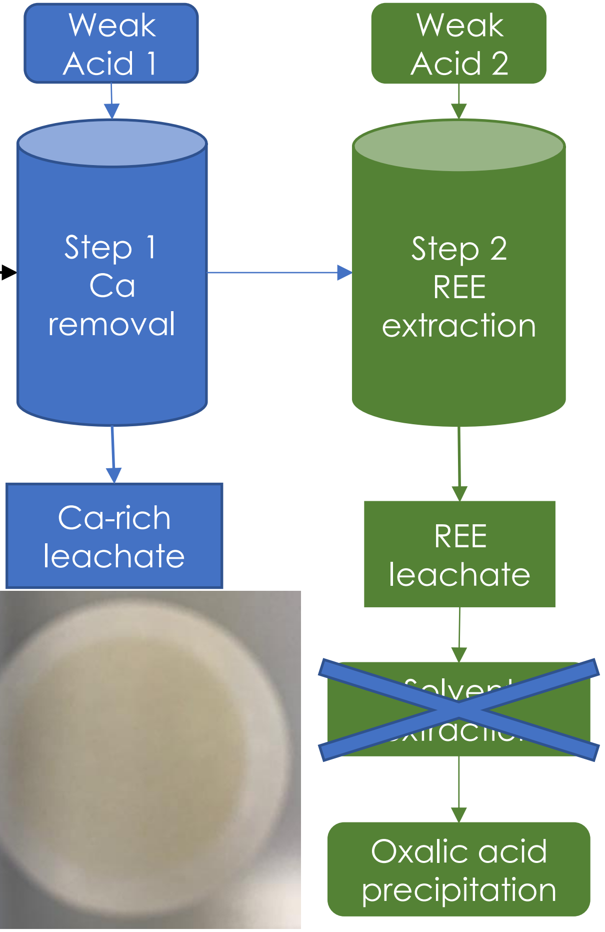
After TREE process, precipitation modeling guided oxalic acid precipitation

- Minimized oxalic acid use
- Successful rejects of Ca
- 99%wt REE oxalate recovered



AMD solid





REE Oxalate, 99%wt

Lithium Ion Sieve from AMD solids (LISA)

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AMD Treatment solids (Al-rich and Mn-rich)

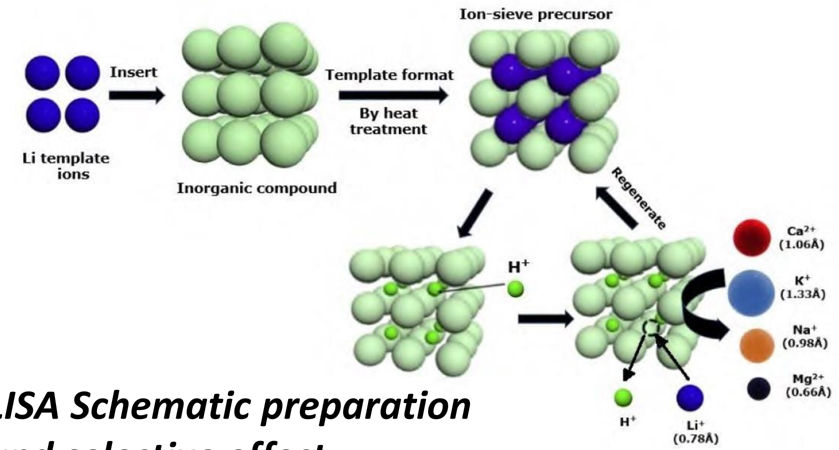
Targeted Rare Earth Extraction (TREE)

Trace metal, Ca/Al removal

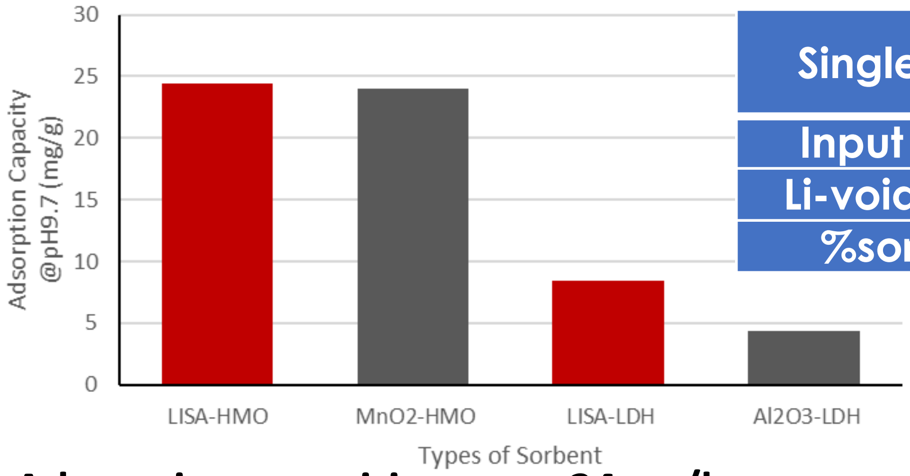
Mn-rich or Al-rich solid residuals

Solid-State Hydrothermal Process

LISA Sorbent



LISA Schematic preparation and selective effect



Single Pass	Li^+ (g/L)	Na^+ (g/L)	K^+ (g/L)
Input brine	1.34	116.1	20.4
Li-void brine	0.55	115.9	19.8
%sorbed	59%	0.17%	2.9%

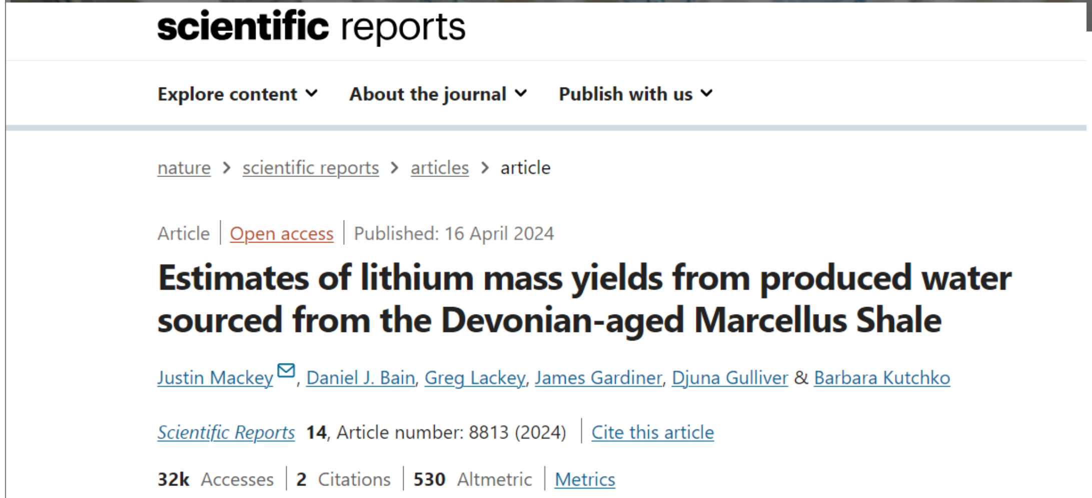
High Li selectivity over other cations in tested brine

Adsorption capacities up to 24mg/kg, comparable to sorbents prepared from chemical reagents.

3rd: CM Resources from O&G Waste & Water

Goal 1 & Goal 5, 6

https://www.pitt.edu/pittwire/features-articles/gas-well-wastewater-lithium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Estimates of lithium mass yields from produced water sourced from the Devonian-aged Marcellus Shale

Justin Mackey, Daniel J. Bain, Greg Lackey, James Gardiner, Djuna Gulliver & Barbara Kutchko

Scientific Reports **14**, Article number: 8813 (2024) | Cite this article

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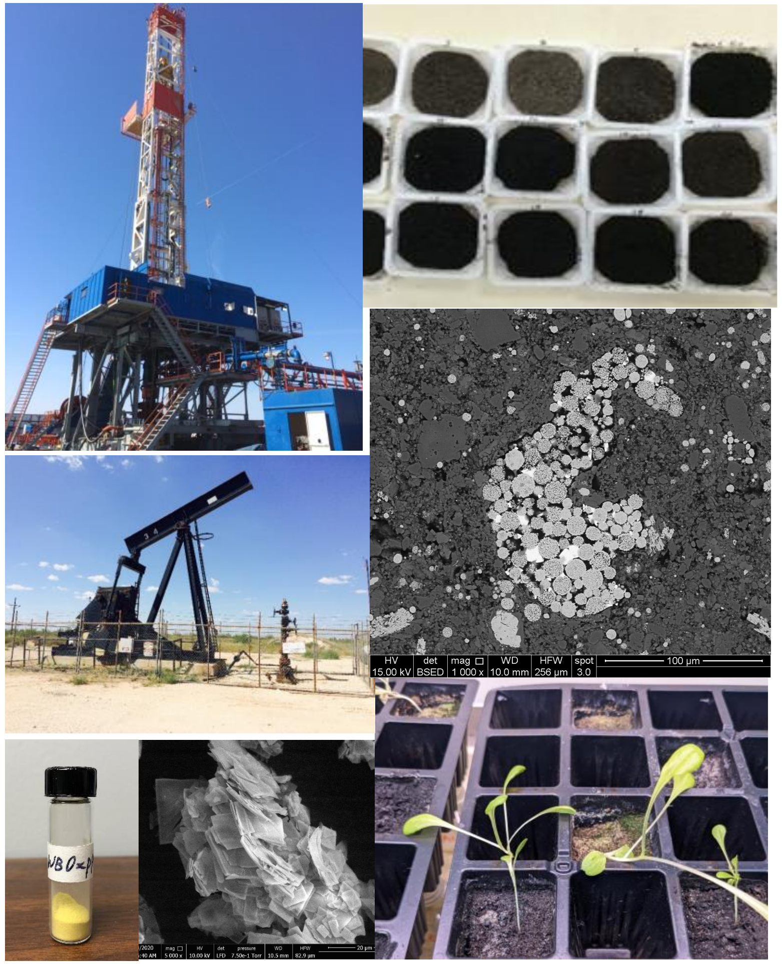
https://www.nature.com/articles/s41598-024-58887-x



- Lithium estimate in O&G produced waters
- CM extraction and beneficial reuse in drill cuttings

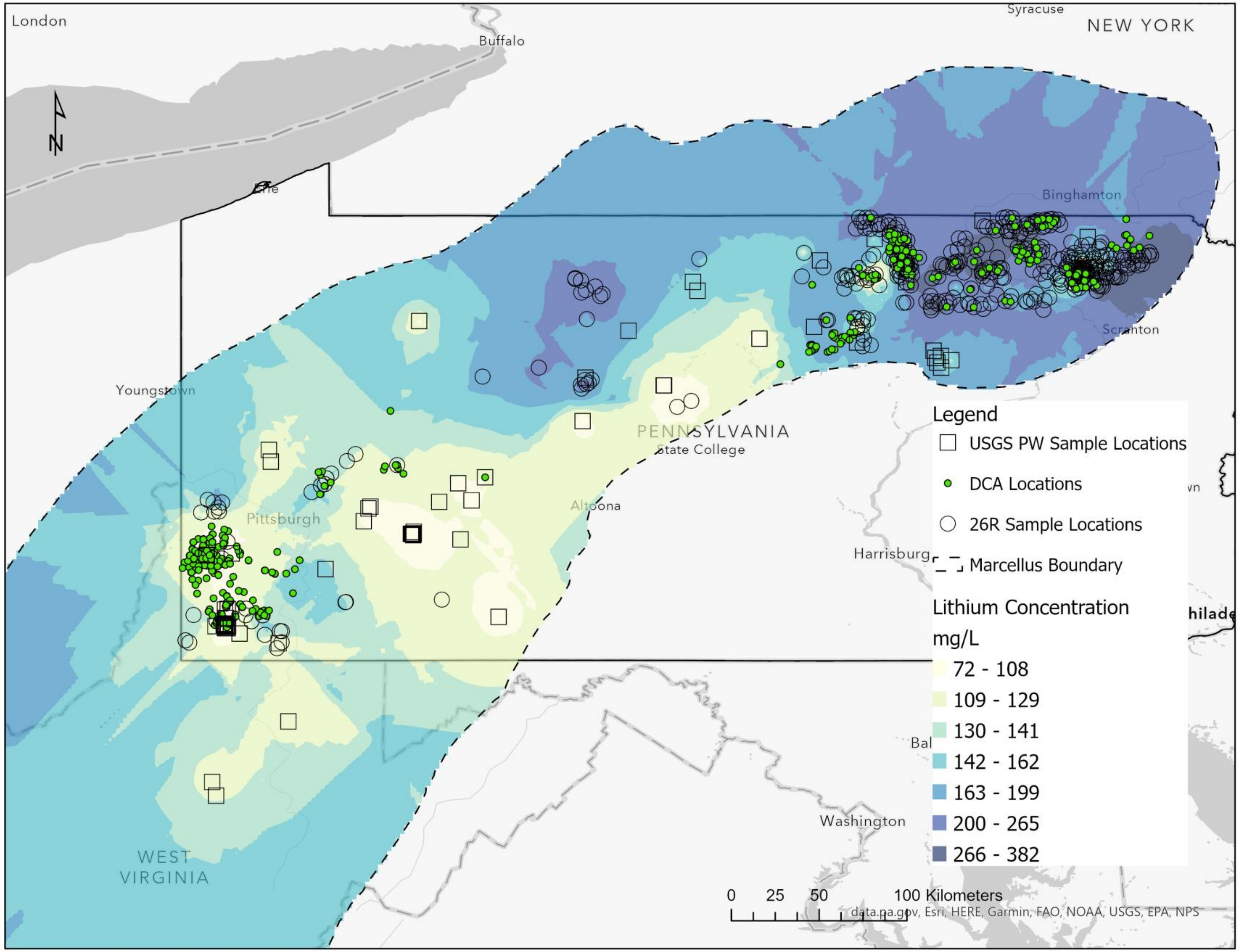
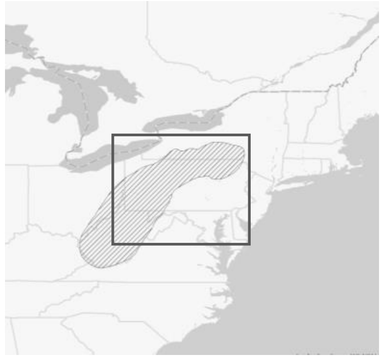


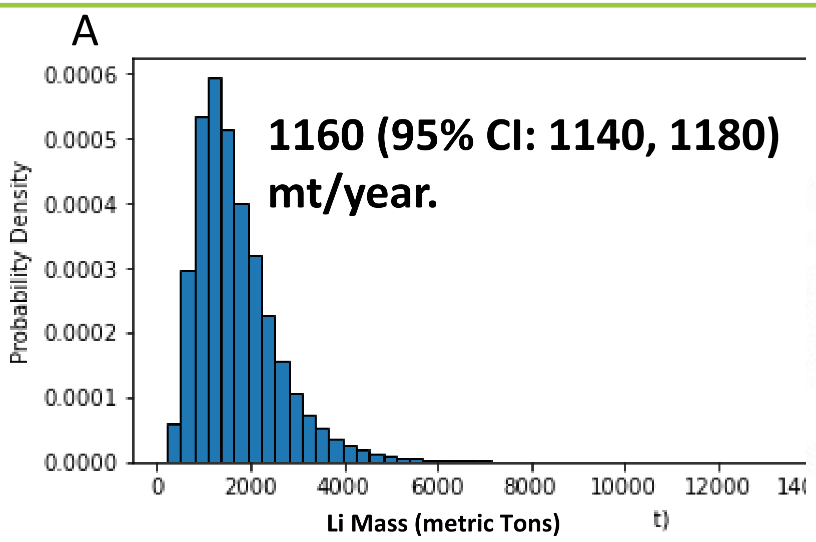
Figure 1. Map of study area showing the Marcellus shale extent, well locations using in decline curve analysis (DCA), PW samples used in this study, and previous USGS sample locations. Kriged lithium concentration layer includes USGS data as well as data reported in this manuscript (USGS, 2018)

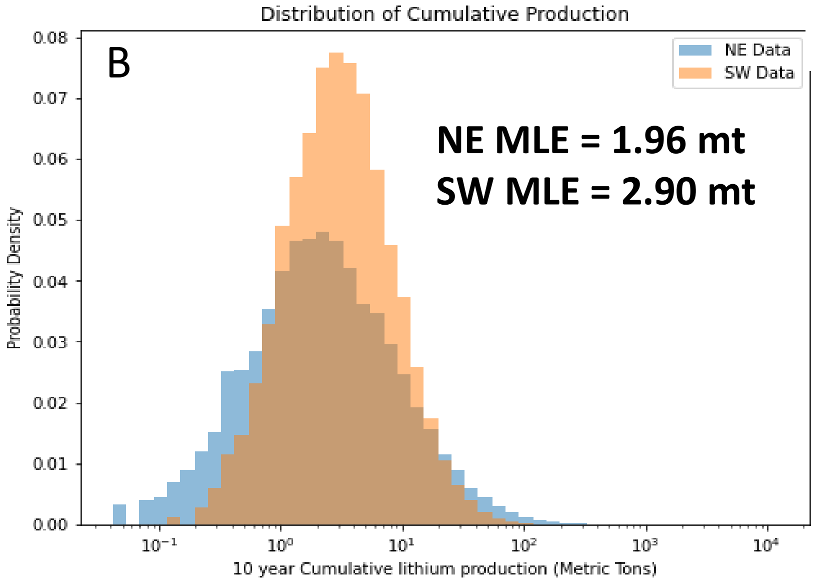


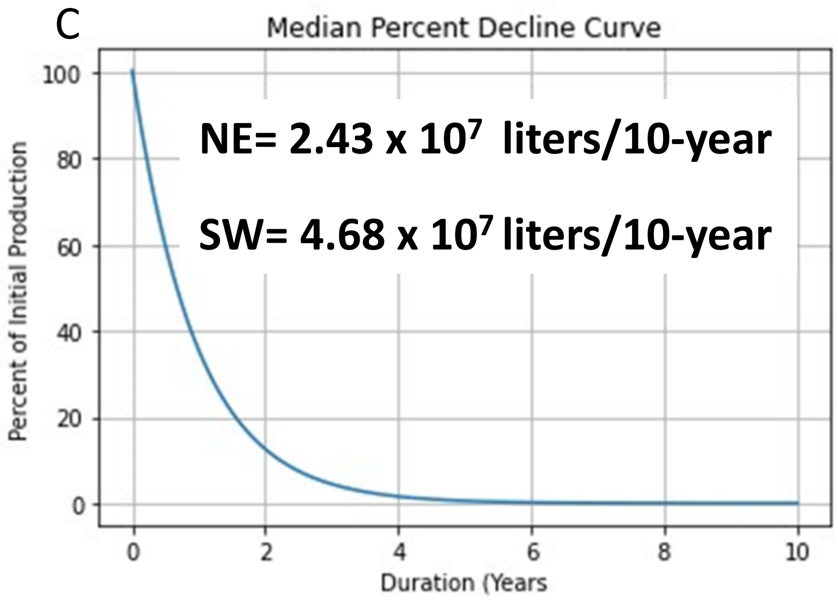
Mackey et al., Scientific Report, 2024

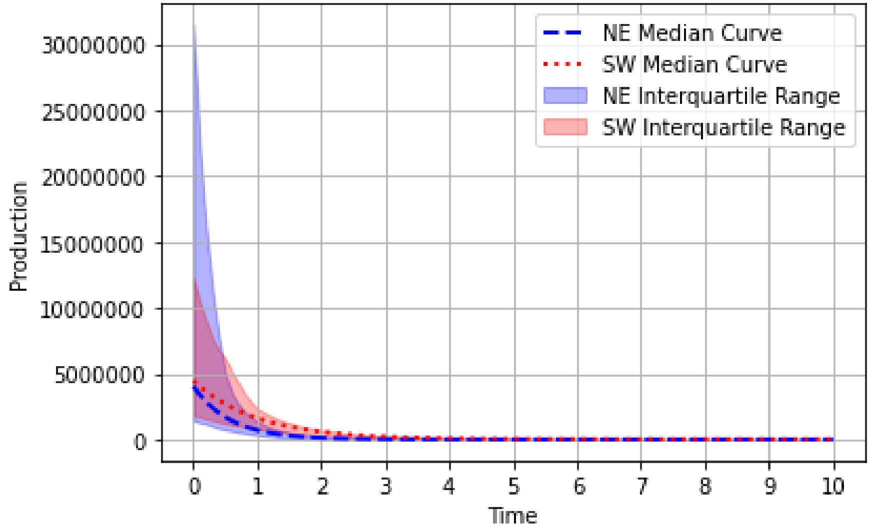
Results:

NATIONAL ENERGY TECHNOLOGY LABORATORY









A) Histogram showing statewide lithium simulation results. MLE of 1,159 mt supplies ~30% of current consumption estimates (USGS, 2023). **B**) Distributions of NE PA and SW PA results from Monte Carlo Simulations. **C**) Plot showing the percent decline in water production of the median Marcellus well over 10 years. **D)** Regional PW decline curve range. Annotation shows regional difference between NE and SW PA 10-year cumulative production water volumes.

Critical mineral source potential from oil & gas produced waters in the United States

NATIONAL ENERGY TECHNOLOGY LABORATORY

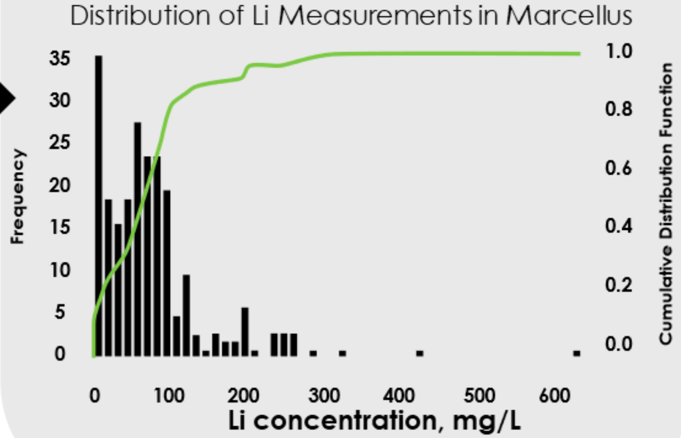
O&G Produced Water:

A Potential Source of Critical Minerals (CM)

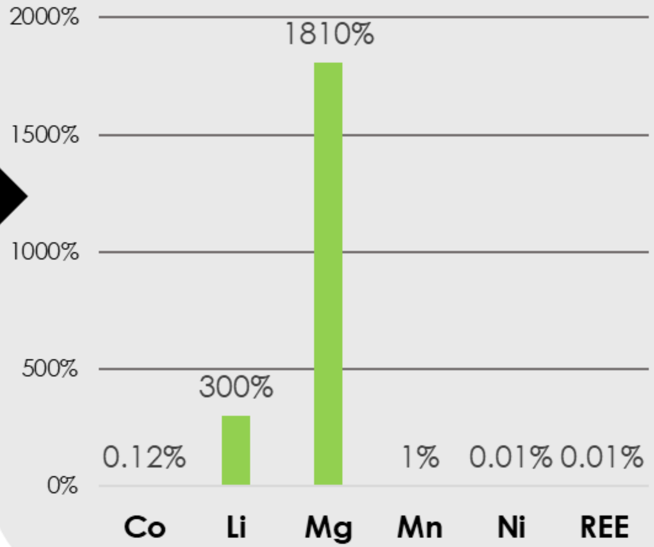
Collect Produced Water CM Data: Ni, Co, Mg, Mn, Li, REEs in Demand

3 Li | 25 Mn | 27 Co | 12 Mg | 28 Ni | 57 La | 58 Ce | 59 Pr | 60 Nd

61 Pm | 62 Sm | 63 Eu | 64 Gd | 65 Tb | 66 Dy | 67 Ho | 68 Er | 69 Tm | 71 Lu



Critical Mineral Yields from Produced Water as % of Demand



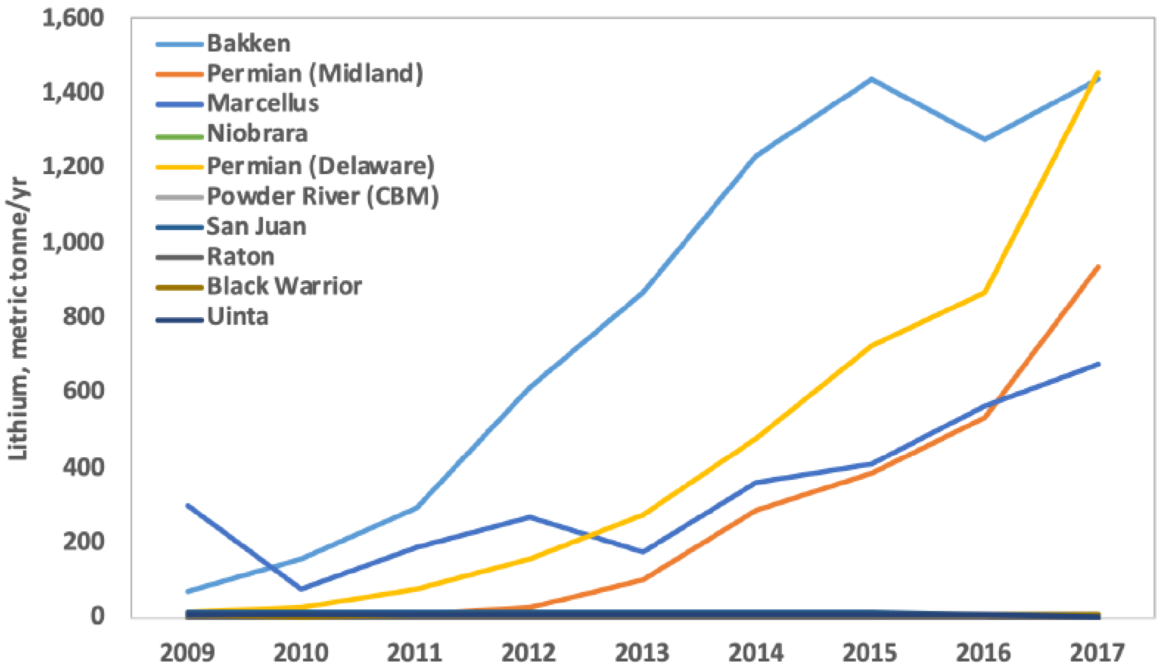
Key finding: Lithium in US PW can meet 300% of current "USGS estimated" U.S. lithium demand

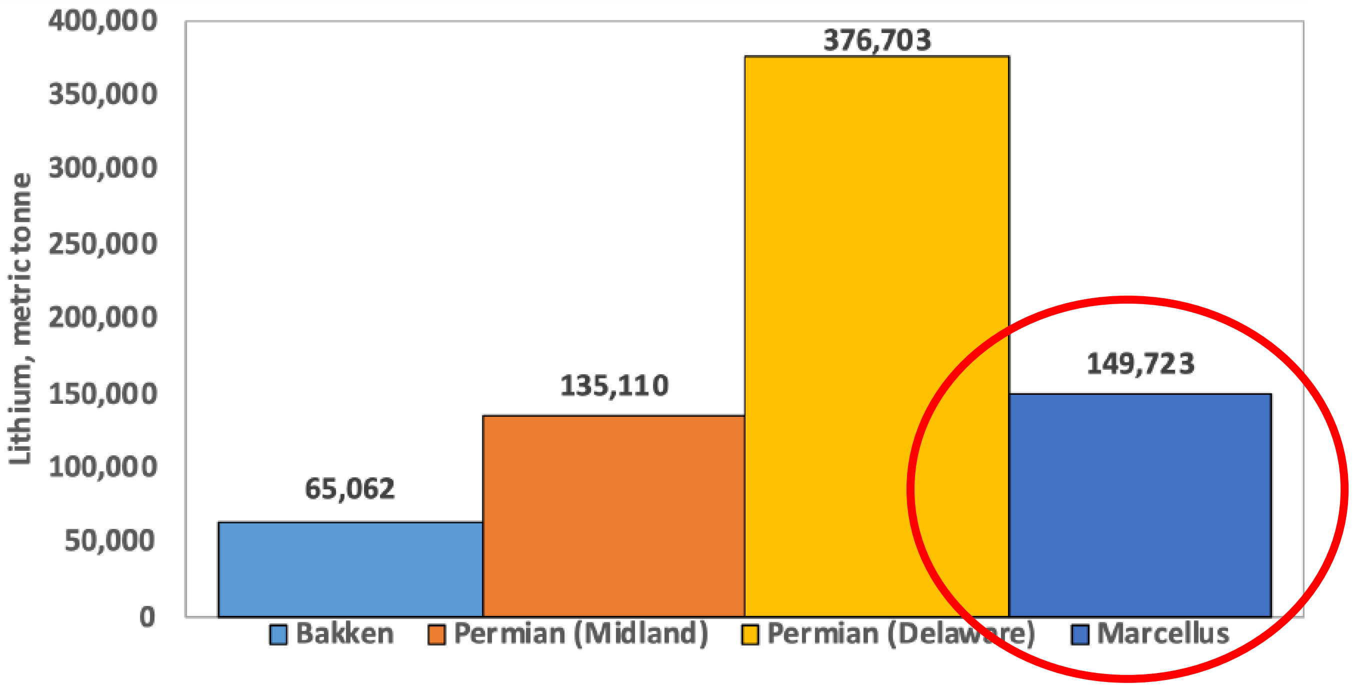
NEWTS DATABASE

Critical mineral source potential from oil & gas produced waters in the United States
Kathryn H. Smith, Justin E. Mackey, Madison Wenzlick, Burt Thomas, Nicholas S. Siefert
Science of The Total Environment, Volume 929, 15 June 2024, 172573

Lithium Resource Assessment

Permian Basin and Marcellus Shale have largest resource potentials





Concentration of Li is ~10 times higher in Marcellus than Permian Basin

Fig.1 Time series of Lithium resource potential calculated from PW volumes presented by Scanlon et al. 2020 and average Li concentration from from USGS PW database (Blondes et al. 2018)

Fig.2. Projected Lithium resource potential calculated from projected remaining PW volumes (Scanlon et al. 2020) and average Li concentration in each formation

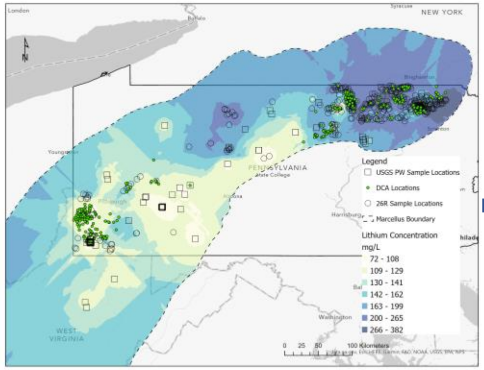
Critical mineral source potential from oil & gas produced waters in the United States, Kathryn H. Smith, Justin E. Mackey, Madison Wenzlick, Burt Thomas, Nicholas S. Siefert, Science of The Total Environment, Volume 929, 15 June 2024, 172573

Accelerated Brine Lithium Extraction (ABLE) Laboratory

IRA Investment: A future facility for DOE researchers and commercial sponsors to test Lithium Separation technologies on clean produced brine at the ~100 gph scale (up to 1000 gpd)

ABLE lab (tentative 2027) as **a test-bed and user facility** for critical mineral recovery will be proposed to

- **Provide pre-treatment**: Hydrocarbon-removal, Scale (NORM)-removal, NF or RO, desalination
- **Provide real-time monitoring** of water chemistry (IC and LIBS) and **process control** (e.g., WaterTap or OLI)
- **Welcome clients** for testing different direct lithium extraction technologies or other critical mineral extraction tests



Marcellus Shale Gas produced water

LISA: Lithium Ion Sieve Sorbent
REE adsorbent
Nanofiltration
Reverse Osmosis
Mineral Softening
Brine crystallizer

Previous case study at WVU

Lithium carbonate

REEs / CMs e.g. Barite

Low Salinity Water

Limestone

Salt Inputs for NaOH & HCl

ABLE lab and proposed modular process at Goal 5

Co-treating flue gas desulfurized effluent and produced water enables novel waste management and recovery of critical minerals - ScienceDirect

NETL Work on Drill Cuttings

NATIONAL ENERGY TECHNOLOGY LABORATORY

900 million barrels (118 million metric tons) of waste send to landfills in 2022
Novel treatment based on characterization:

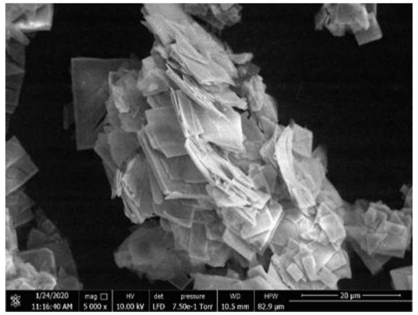
- Environmental Impact reduction (oil and barite removal)
- Targeted recovery of Barite, Transition metals (Co, Ni, Cu), Vanadium, REEs
- Drill cutting conversion to soil supplements

1) Drill Cuttings from Hydraulic Fracturing

2) Novel Treatment and Remediation

3A) Green Roof Application

3B) Critical Mineral Recovery

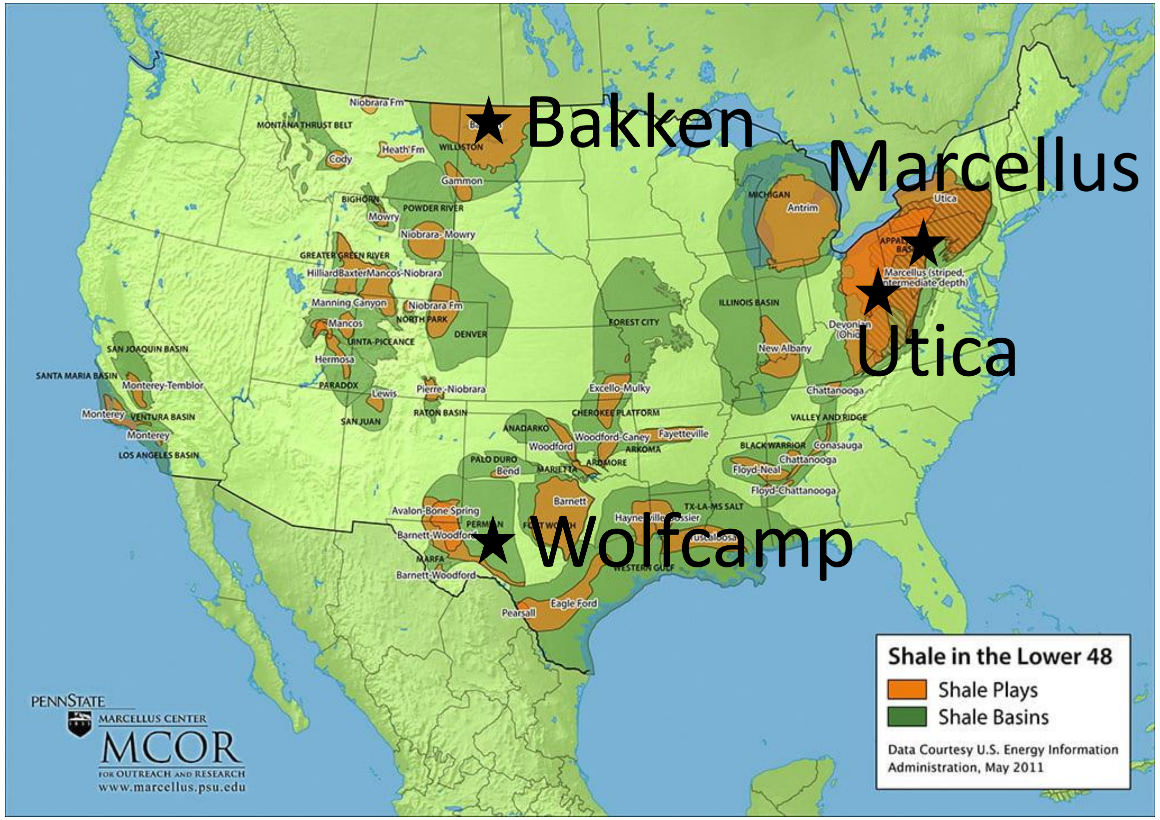


Tentative schematic of drill cutting conversion

Drill Cutting and Core Library at NETL

NATIONAL ENERGY TECHNOLOGY LABORATORY

17 Drill Cuttings and Shale Samples, Marcellus (MS)(8), Bakken (Bak) (3), Utica (UT) (1), Wolfcamp (WC) (5)

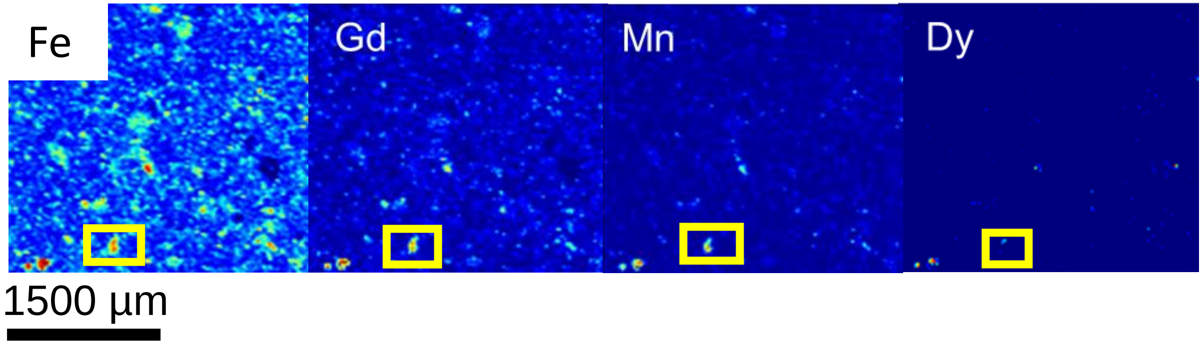


Sample	Ba (ppm)	V (ppm)	Ni (ppm)	Co (ppm)	Li (ppm)	REE (ppm)	TOC (%)
MS-5HA	30167	150	70	14	24	168	2
MS-5HB	51723	917	363	30	4	204	10
MS-5HC	48515	250	97	9	BDL	160	3
MS-5HD	47938	824	228	22	BDL	159	7
MS-476	1302	382	216	30	18	235	4.2
MS-505	1010	465	249	23	BDL	**239**	7.8
MS-548	790	1165	517	33	8	232	13
MS-967	488	1112	518	**28**	6	153	12
BAK-229	188	**1575**	**654**	27	**72**	169	**19**
BAK-254	207	65	BDL	13	23	146	4
BAK-300	110	392	249	33	5	87	8
WC-310	2385	132	103	11	BDL	184	5.6
WC-400	947	148	99	7	BDL	130	4
WC-460a	9561	123	101	9	BDL	183	5.5
WC-460b	2836	113	102	8	BDL	179	5.4
WC-480	1592	138	105	10	BDL	217	4.9
Utica	**76271**	53	33	4	BDL	123	11.1
Deposit Grade		**1000**	**7000**	**400**	**80**	**300**	

- High **Total Organic Carbon** correlates with high CMs such as **REE, V, and Ni**
- Drill cuttings high in barite

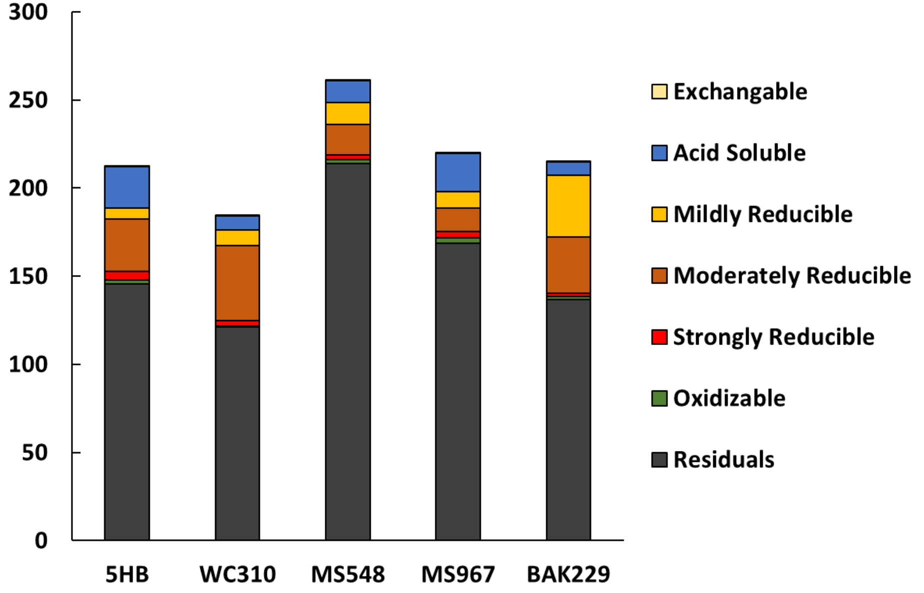
Select REE with FeS (oxidized)

Synchrotron μ-XRF of MS-967



REE extracted via **reducible phases (e.g., oxidized FeS)**

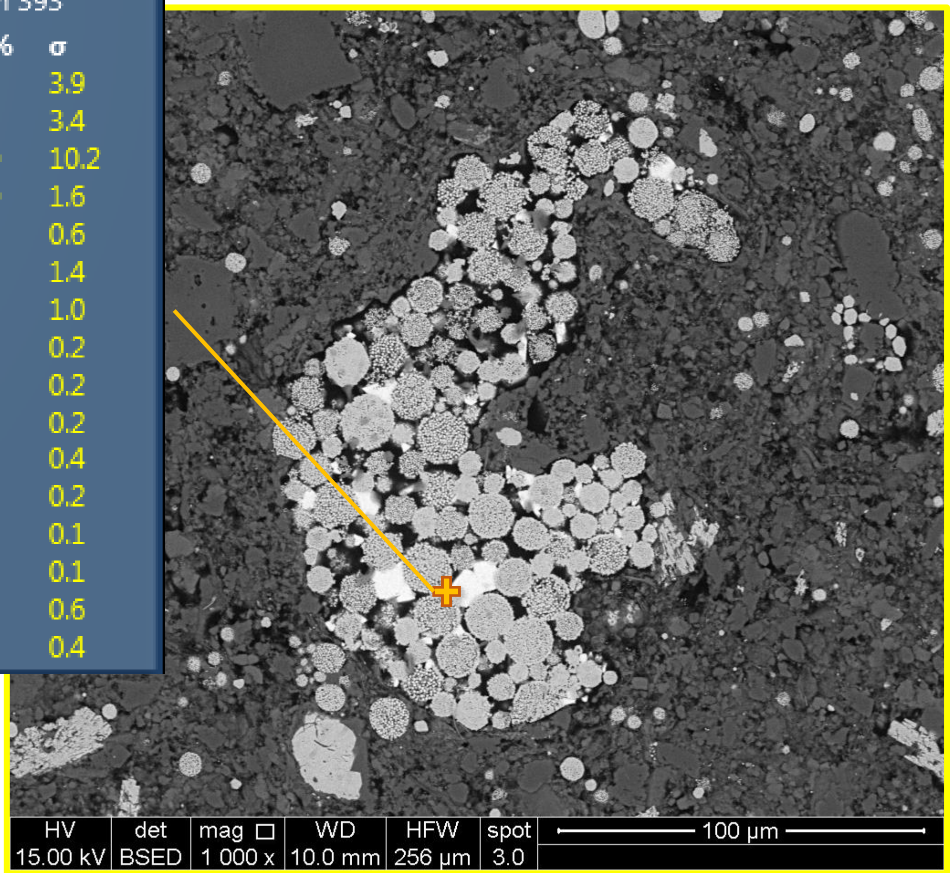
Up to 66% Sm, Eu, Gd recovered



Yellow box in SEM-EDS

Spectrum 393

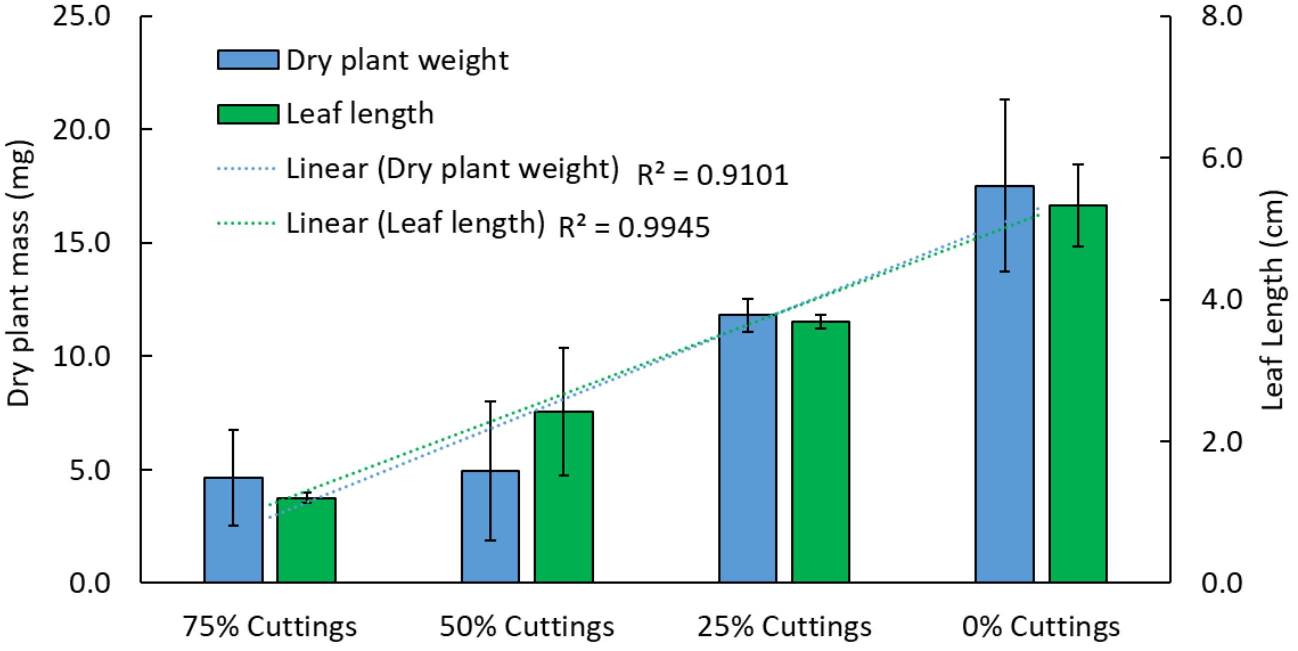
	Wt%	σ
S	29.7	3.9
Fe	25.3	3.4
C	21.0	10.2
O	11.0	1.6
Si	4.2	0.6
Dy	2.6	1.4
Au	1.8	1.0
Na	1.3	0.2
Al	0.9	0.2
Ca	0.7	0.2
Ni	0.5	0.4
P	0.4	0.2
K	0.3	0.1
Mg	0.1	0.1
Gd	0.1	0.6
Sb	0.1	0.4



SEM images of MS-967

Treated Cuttings can be Used as Soil Amendments

- Plants grown for 45 days
  - Fertilized after 2 weeks
- All mixtures of treated cuttings and soil had seedling growth (60 - 100% germination)
- Treated cuttings - died within a week
- Raw cuttings - no growth
- Correlation between plant growth and cutting content in soil



Critical Minerals in Fossil Energy (FE) Wastes

NATIONAL ENERGY TECHNOLOGY LABORATORY

55 Minerals Identified to be Critical to National Security (U.S. Dept. of Interior)

DOE Identifies "Dynamic Dozen" July 28, 2023

Mineral	Top producer	Top supplier	Notable example application	Potential Fossil Energy Feedstocks
Aluminum	China	Canada	Aircraft, power transmission lines, alloys	**AMD solids, fly ash**
Barite	China	China	Oil and gas extractions, lead-acid batteries,	**Drill cuttings**
Cobalt	Congo	Norway	Jet engines, rechargeable batteries	**AMD solids, drill cuttings**
Vanadium	China	China	Alloys, catalyst for green chemistry, coatings	**Drill cuttings**
Lithium	Australia	Chile	Rechargeable batteries, Al-Li alloys for aerospace	**Produced waters**
Manganese	China	South Africa	Aluminum and steel production, lightweight alloys	**AMD solids**
Rare earth elements (REE)	China	China	Catalyst, magnets, aerospace guidance, laser, fiber optics	**AMD solids, fly ash, drill cuttings, waste streams**

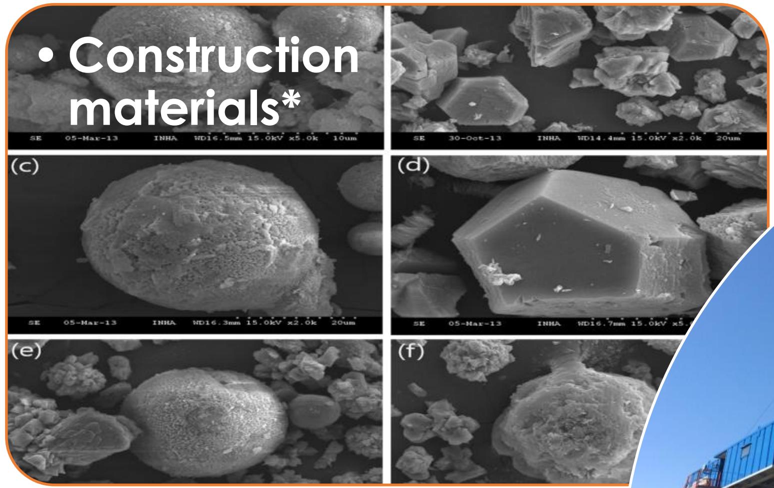
Other critical minerals such as Chromium, Gallium, Germanium, PGM from mining wastes were studied under NETL RIC program.

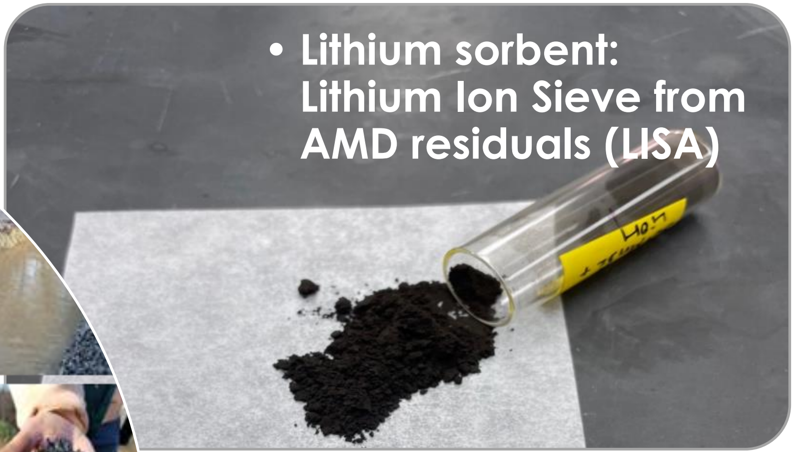
U.S. DEPARTMENT OF ENERGY

https://www.usgs.gov/news/national-news-release/us-geological-survey-releases-2022-list-critical-minerals

Convert Fossil Energy Wastes to Value added Products

"Zero Waste" Strategy





*: Images are from previous or similar studies. Products are under development with current NETL funding.

Acknowledgements

Coffee….Lots of Coffee

- Dr. Barbara Kutchko (NETL)
- Drs. George Guthrie, R. Burt Thomas and Tom Tarka – RIC REE TPL
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- Camille Sicker/Shaffer (Univ. of Pittsburgh)
- Josh Miller (ORISE-NETL)
- Brianna O’Neal-Hankle (MLEF-NETL)
- Pittsburgh Analytical LAB (NETL)
- Dr. Kirk Scheckel (U.S. EPA)
- Dr. Sam Webb, Dr. John Bargar, Dr. Adam Jew, Dr. Nick Edwards, Dr. Sharon Bone (SLAC)

NETL-RIC projects are funded through the U.S. DOE’s Office of Fossil Energy and Carbon Management.

*The synchrotron work was conducted on beamlines **2-3, 4-1, 10-2, 11-1, 7-2** at the Stanford Synchrotron Radiation Lightsource (SSRL), a national user facility operated by Stanford University on behalf of the U.S. DOE’s Office of Basic Energy Sciences, Office of Biological and Environmental Research, and the National Institutes of Health.*

Opportunities

NETL is hiring

- NETL is hiring a cohort to research critical mineral and materials from domestic sources (ORISE fellowship)
- https://netl.doe.gov/sites/default/files/2024-09/Intern%20Postdoc%20Cohort_Template%20R4_Digitial.pdf

Student Internship

Post-graduates

- Mickey Leland Intern Program (MLEF)
- https://orise.orau.gov/mlef/

- Welcome submissions

minerals

an Open Access Journal by MDPI

IMPACT FACTOR 2.5

CITESCORE 3.9

Critical Minerals and Associated Elements in Mine Effluent and Treatment Residuals: Management Strategies and Technologies for Resource Recovery

Guest Editors:

Dr. Mengling Stuckman
National Energy Technology Laboratory, 626 Cochrans Mill Road, P.O. Box 10940, Pittsburgh, PA 15236, USA

Message from the Guest Editors

This Special Issue calls for research on understanding the geochemical transformations and engineering techniques related to the enrichment and behaviors of CMs from one type of unconventional resource, mine waste streams (e.g., coal or metal mines) and treatment precipitates (e.g.,

https://www.mdpi.com/journal/minerals/special_issues/U7Z9S5XK57

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RESOURCES

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