# Scalable Fabrication of a Fibrous Amine-Functionalized Matrix (FAM) Sorbent for Critical Mineral Recovery

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U.S. DEPARTMENT *of* ENERGY

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# Authors and Contact Information

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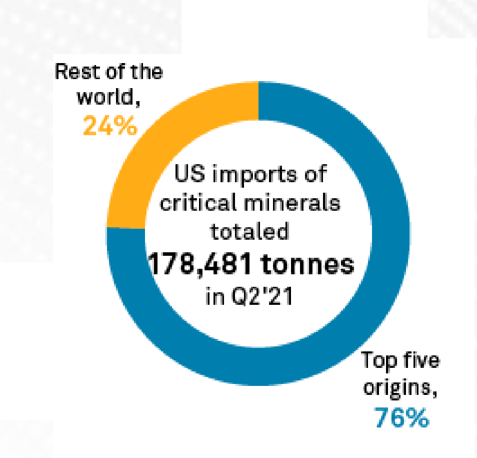
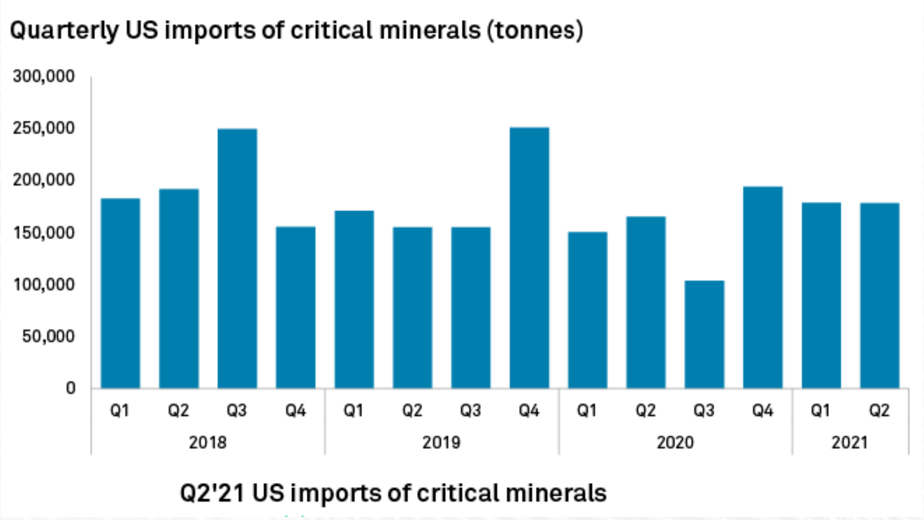
# Background

## Critical Minerals (CM) and Supply Chain Security

**Critical mineral definition**: "…a non-fuel mineral or mineral material essential to the economic or national security of the U.S. and which has a supply chain vulnerable to disruption." – *Energy Act of 2020.*





Data compiled Aug. 6, 2021.
Includes U.S. imports of manganese ores and concentrates; fluorides and complex fluorine salts; tin; niobium, tantalum, vanadium or zirconium ores and concentrates; graphite (natural); manganese oxides; titanium; compounds of rare earth metals; carbonates and sulfates, among others.
Only includes countries that report monthly data to the U.S. Census Bureau.
Source: Panjiva, a business line of S&P Global Market Intelligence, a division of S&P Global Inc.

US critical mineral imports up 7.9% y-o-y in Q2 – report - MINING.COM

# Background

## CMs in Coal Waste/Byproduct

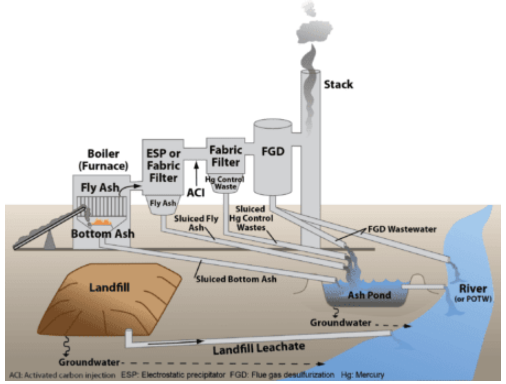
### Combustion Residual Leachate

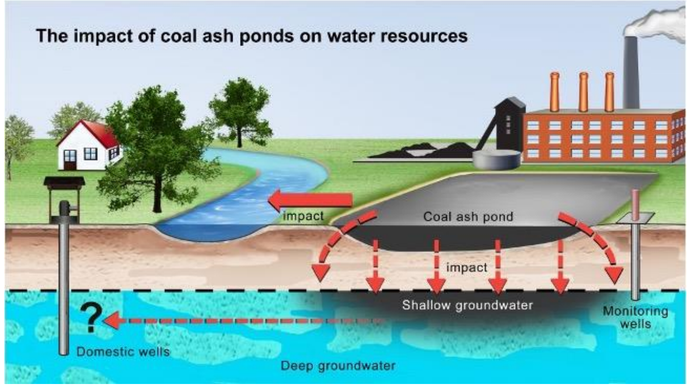
- Combustion residual leachate and impoundment wastewater from coal-fired power plants amount to billions of gallons, containing high **Manganese, Iron, Lithium, Cobalt, Nickel**, etc., among other compounds
- Volumes of leachate and impoundment water were calculated as 7.7-12.1 billion gallons for landfill leachate and up to 240 billion for ash ponds
- Valuable **rare earth elements (REEs)** like Lanthanum, Cerium, Neodymium, and Scandium, plus other CMs such as **Gallium, Germanium, Lithium, Titanium, Vanadium, and Zirconium**, which are crucial for green tech and electronics

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

W.C. Wilfong, T. Ji, Y. Duan, F. Shi, Q. Wang, M.L. Gray, Critical review of functionalized silica sorbent strategies for selective extraction of rare earth elements from acid mine drainage, J. Hazard. Mater., 424 (2022) 127625.

### Acid Mine Drainage (AMD)




[Steam Electric Power Generating Effluent Guidelines - 2024 Final Rule | US EPA]

AMD(coal) has bounty of untapped CM:

- Millions of gallons/day; thousands of miles of streams
- **Magnesium** (259 ppm surveyed max.) – steelmaking, batteries
- **Aluminum** (189 ppm max.) – ubiquitous, found in nearly all sectors
- **Zinc** (30 ppm max.) – metallurgy for producing galvanized steel
- **Manganese** (20 ppm max.) – alloying, and for reducing metals
- **Nickel** (1 ppm max.) – hydrocarbon production

C. Able, D. Rellergert, V. Mazzoni, E. Grol, Assessment of combustion residual leachate volume, composition, and treatment costs, J. Hazard. Mater., 457 (2023)131731.
J. Hower, A. Kolker, HJJ. Hsu-Kim, D. Platta, Rare Earth Elements in coal fly ash and their potential recovery. Book Chapter, 70203337, Eastern Energy Resources Science Center, 2024.
NETL Patents New Process for Extracting Critical Resources from Coal Fly Ash at High Quantities | netl.doe.gov
EPA proposal takes aim at wastewater emissions from coal plants (power-eng.com)

# Background

## Key Challenges for Recovery

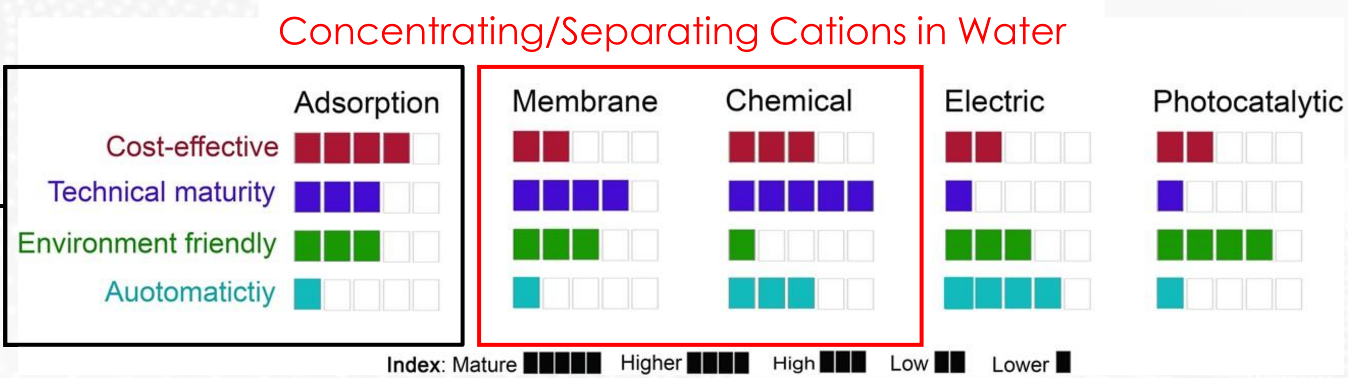
- **Extremely Low Concentrations:** CMs occur at ppm levels or lower
- **Complex Chemical Mixtures:** High salinity (e.g., Na, Ca, K, etc.), competitive heavy metals (e.g., Fe, Mn, Al), organic compounds, suspended solids and fine particles hinder separation
- **Unfavorable Mineral Speciation:** Strong hydrated ions; stable aqueous complexes; colloidal or nanoparticle-bound species limit precipitation, filtration, and sorption
- **Trade-Off Between Selectivity and Recovery:** Selective processes are often costly, short-lived, and regeneration-intensive
- Fouling and scaling
- Regulatory and operational constrains
- pH, etc.

# Background



## Methods of Metal Recovery from Water

### Conventional Technologies




N.A.A. Qasem, R.H. Mohammed, D.U. Lawal, Removal of heavy metal ions from wastewater: a comprehensive and critical review, npj Clean Water, 4 (2021) 366.

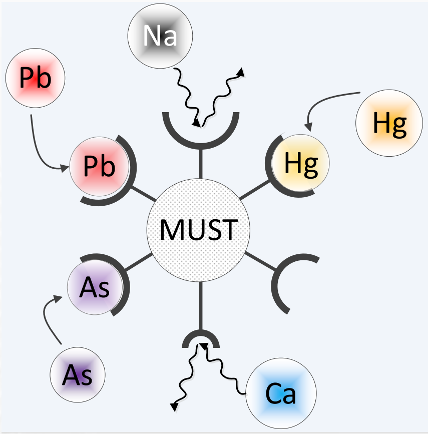
### As-Is or Functionalized…

- **Carbon** (nanotubes, activated carbon)
- **Mineral** (zeolite, silica, clay)
- **Polymer** (chitosan, ion exchange resin, hydrogel)
- **Non-Carbon Bio** (algae, bacteria)

*Polymer network-functionalized silica*

Combine concepts…

### NETL Sorbent Technologies



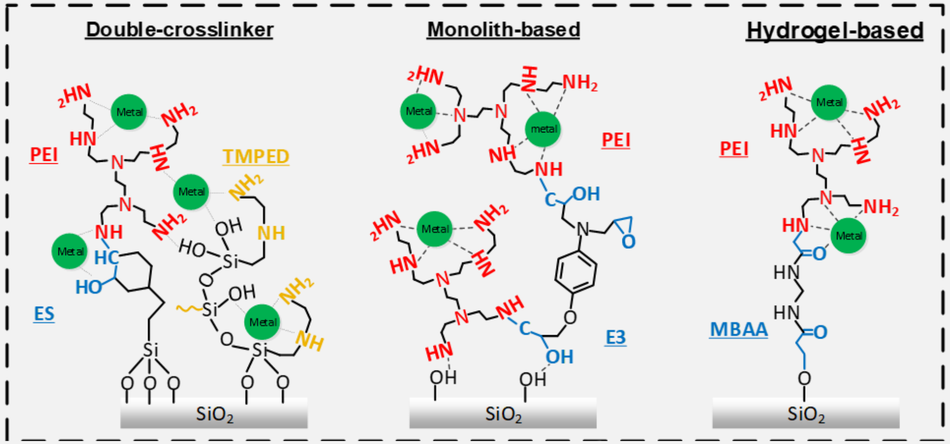

### NETL Sorbent Advantages

- Easily prepared
- Tailored chemistry
- Easily scaled
- Low cost (~$10/kg)
- **Combined adsorption and membrane**

# NETL Sorbent Technology

U.S. DEPARTMENT of ENERGY | NATIONAL ENERGY TECHNOLOGY LABORATORY

## Overview




Clean water is a national and global problem - continued development of Multi-functional Sorbent Technology (MUST) materials and reactor systems is a lucrative solution.

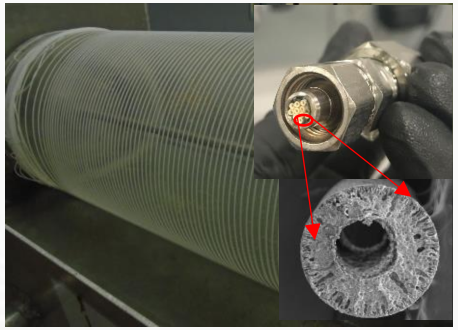
2021 R&D 100 Award

2021 Secretary of Energy's Achievement Award

2022 Edison Award Bronze

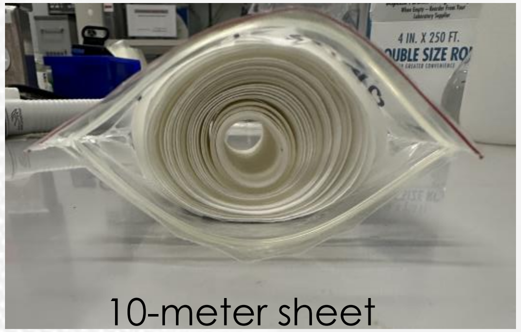
### 1st Gen. - Particles

### 3rd Gen. - Hollow Fiber



### 2nd Gen. - Pellets

### 4th Gen. - Flat Sheet (FAM)





Gray, M.L., Kail. W. K., Wang, Q., Wilfong, W. C., Stable Immobilized Amine Sorbents for REE and Heavy Metal Recovery from Liquid Sources, US 2018/0100065A1. Apr. 12, 2018. Continued licensing by PQ Corporation/Ecovyst.

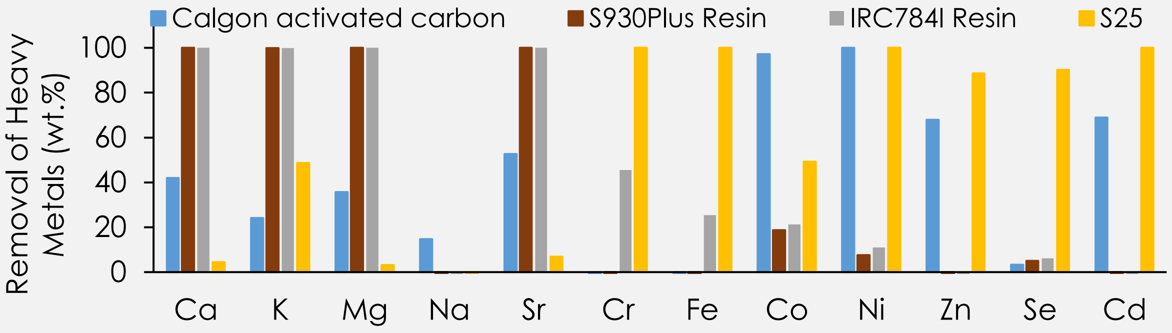
Gray, M.L., Kail. W. K., Wang, Q., Wilfong, W. C., Stable Immobilized Amine Sorbents for Removal of an Organic Contaminate from Wastewater. US 2020/10,836,654. Nov. 17, 2020.

Gray, M.L., Wang, Q., Wilfong, W.C., Shi, F., Fibrous Amine-functionalized Matrix (FAM) for Contaminant Removal from Gaseous and Liquid Sources, Methods of Making and Use, US Patent Application #63434082, Dec. 2022.

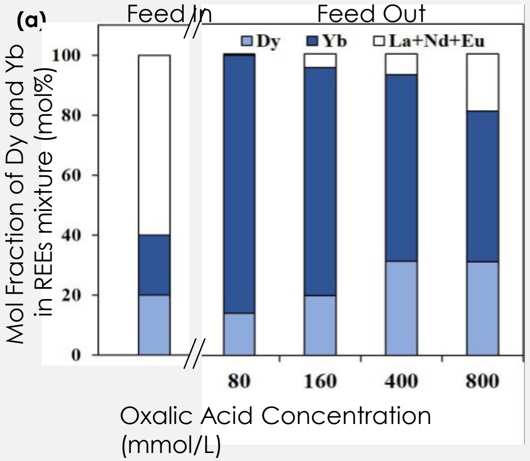
# NETL Particle Sorbent

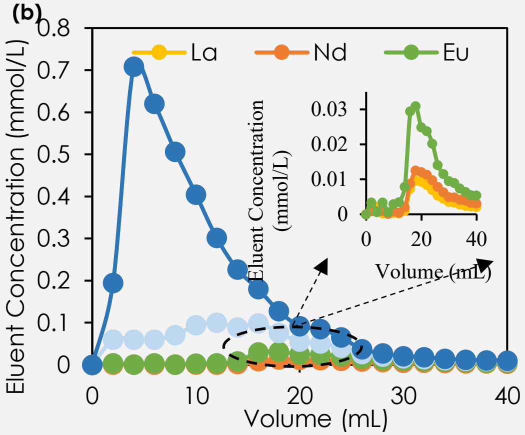
## Accomplishments Overview

### Selective Recovery




**NETL S25** sorbent selectively captured 60% of Co, 90% of Zn, 99% of Ni, and 99% of Fe from Somerset flue gas desulfurization (FGD) wastewater, while simultaneously removed toxic metals such as Cr, Se, and Cd.

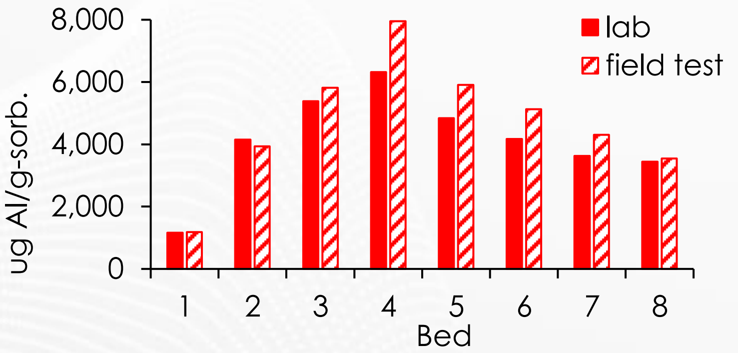


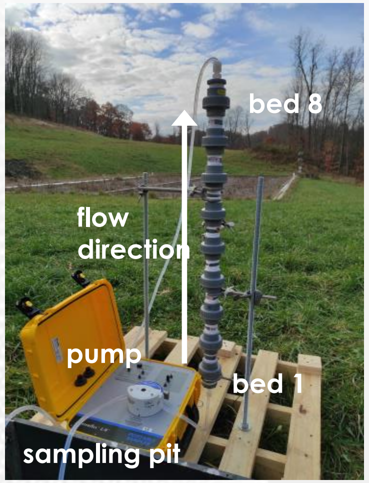

(a) Capture-selective release of Dy and Yb from ideal REE mixture (1.8 mM each $Ln^{3+}$), using oxalic acid. (b) Sequential elution of heavy REE (HREE) from REE-loaded cross-linked MUST with oxalic acid.

Q. Wang, B. Kail, W. Wilfong, F. Shi, T. Tarka, M. Gray, Amine Sorbents for Selective Recovery of Heavy Rare-Earth Elements (Dysprosium, Ytterbium) from Aqueous Solution. ChemPlusChem, 2020. 85(1): p. 130-136.

### Field Test at the Pittsburgh Botanic Gardan




| AMD Metal | ug/L |
|---|---|
| Ca | 117,483 |
| Na | 96,138 |
| Mg | 45,227 |
| K | 1,952 |
| Si | 11,655 |
| **Al** | **11,602** |
| Fe | 1,851 |
| Mn | 1,318 |
| Zn | 172 |
| Ni | 131 |
| Sr | 836 |
| Others | <200 |




W. C. Wilfong, Q. Wang, B. Howard, P. Tinker, K. Johnson, W. Garber, F. Shi, M. Gray, Fractionation of critical metals from authentic acid mine drainage using a multi-bed immobilized amine sorbent setup: A field site study. J of Water Process Engineering, 2024. 58, 104788.

# NETL Particle Sorbent

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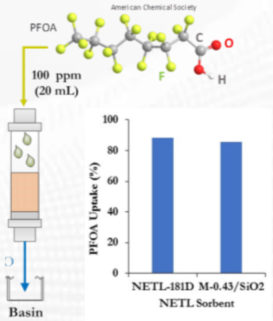
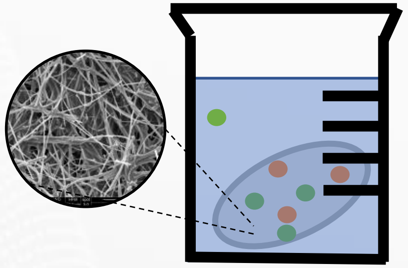
## Achievement Overview

### Removal of Lead from Tap Water

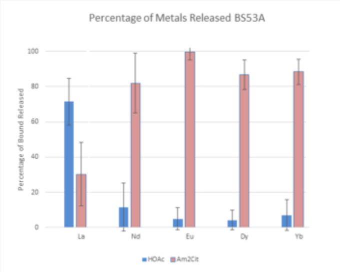

### Removal of Organic Pollutants from Wastewater

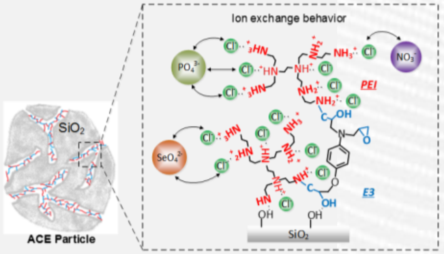

### Extraction of Metals from Organic Solvent



### Separation of REEs from Acid Mine Drainage




### Removal of Heavy Metal from FGD Wastewater




### Removal of Radioactive Metals

Gray, M.L., Kail. W. K., Wang, Q., Wilfong, W. C., Stable Immobilized Amine Sorbents for REE and Heavy Metal Recovery from Liquid Sources, US 2018/0100065A1. Apr. 12, 2018. Continued licensing by PQ Corporation/Ecovyst.

Gray, M.L., Kail. W. K., Wang, Q., Wilfong, W. C., Stable Immobilized Amine Sorbents for Removal of an Organic Contaminate from Wastewater. US 2020/10,836,654. Nov. 17, 2020.

Gray, M.L., Wang, Q., Wilfong, W.C., Shi, F., Fibrous Amine-functionalized Matrix (FAM) for Contaminant Removal from Gaseous and Liquid Sources, Methods of Making and Use, US Patent Application #63434082, Dec. 2022.

# From Particle to Flat Sheet

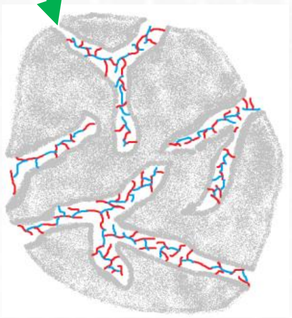
## Same Chemistry Different Format

### NETL Particle Sorbents

$SiO_2$ porous particle supported polyamine network-based sorbents (25-500 µm)

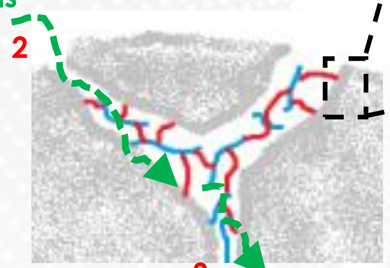
Metals

1

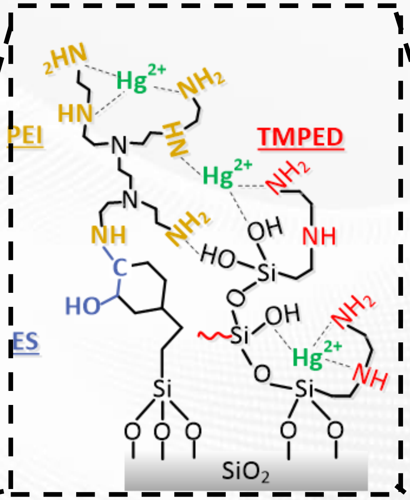


Metals

2



3



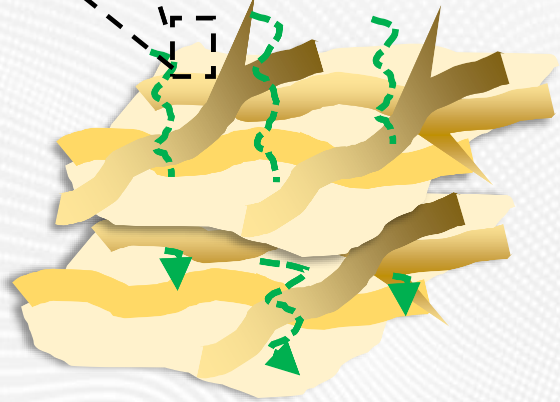


- Particle free
- Simple, scalable
- Fast kinetics
- Regenerable

### NETL Flat Sheet Sorbents (FAM)

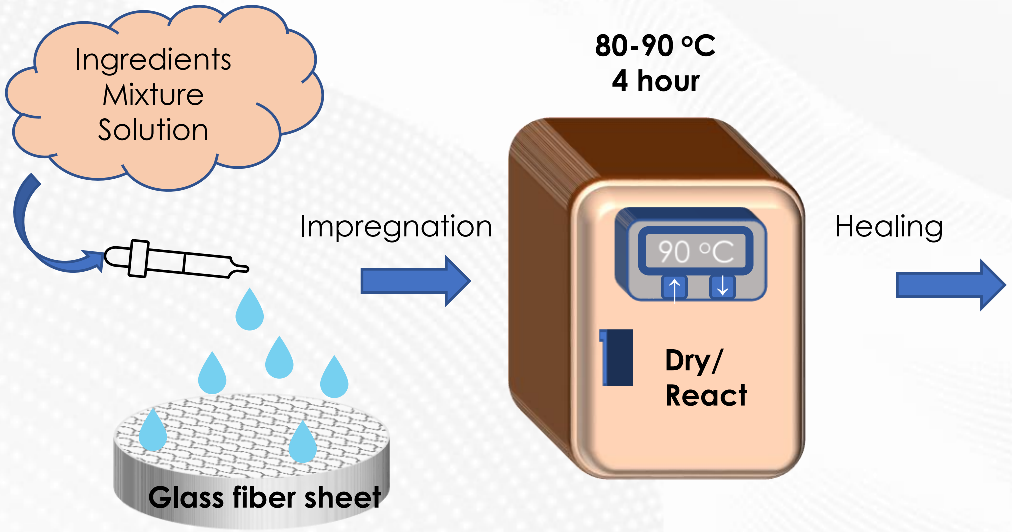
Micro-fiber supported polyamine network-based microfilms

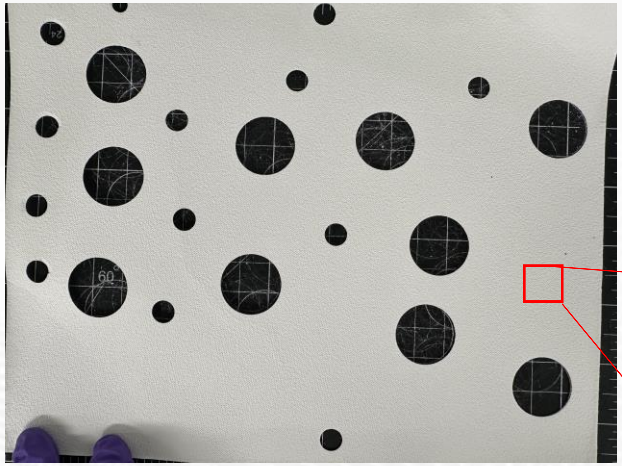
Metals



# FAM Fabrication and Reproducibility

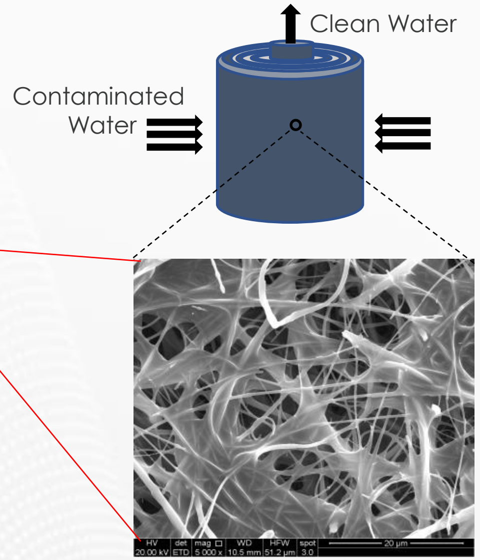
## Overview

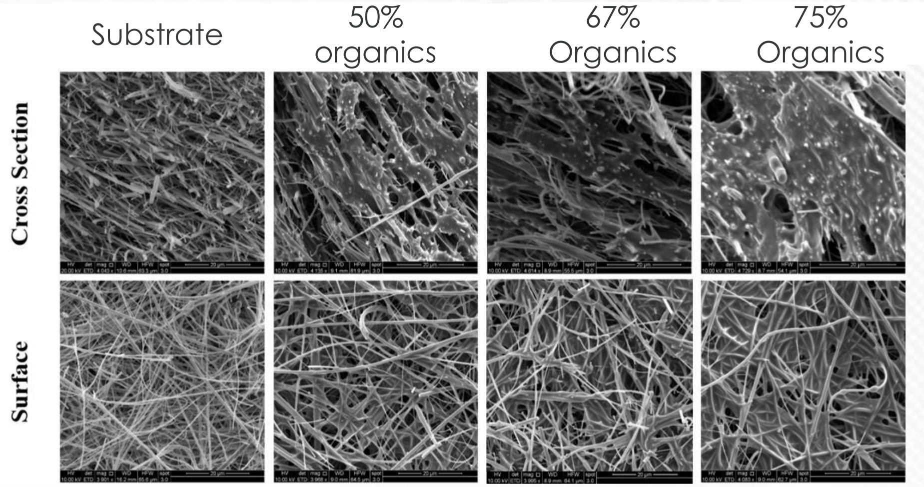


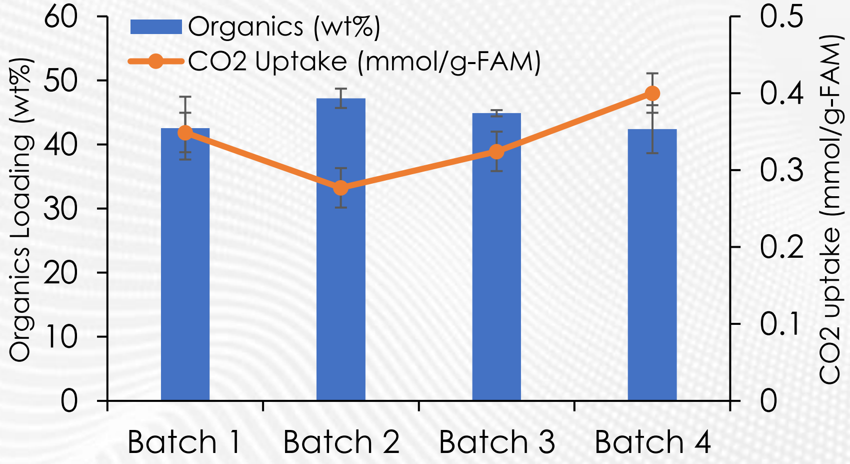



Lab Synthesized 8x10 inches FAM sheet.

Porous glass fiber sheet impregnated with functional PEI species



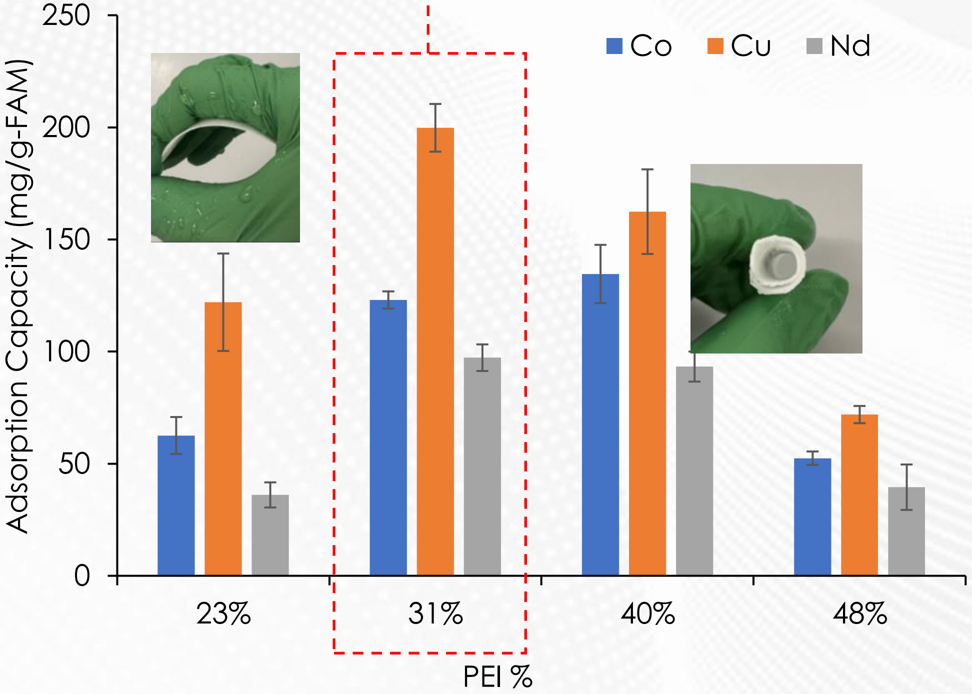



# FAM Performance and Optimization



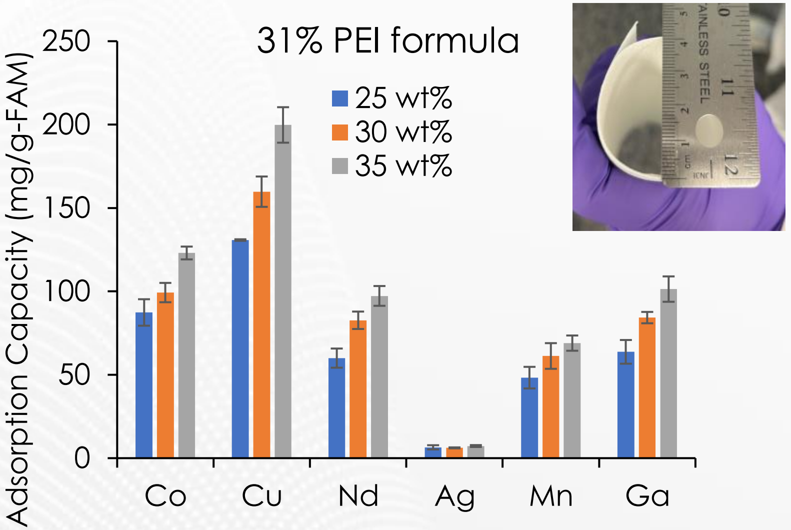
## Trade-Off of the Capacity and Flexibility

### Effect of Formula




- Higher linker ratio results in increased material rigidity and a larger bending radius
- Most metals exhibit high adsorption at a PEI loading of 31-40%

### Effect of Organics Loading



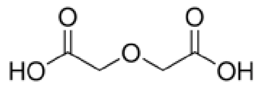
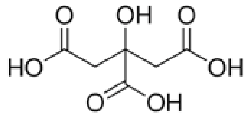
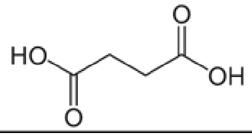
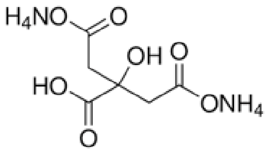
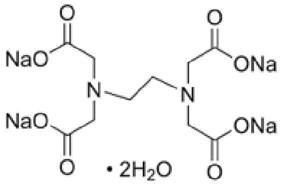

| Ingredients Concentration (wt.%) | Bending Diameter (cm) |
|---|---|
| 25 | 2.5±0.2 |
| 30 | 3.1±0.2 |
| 35 | 3.7±0.2 |

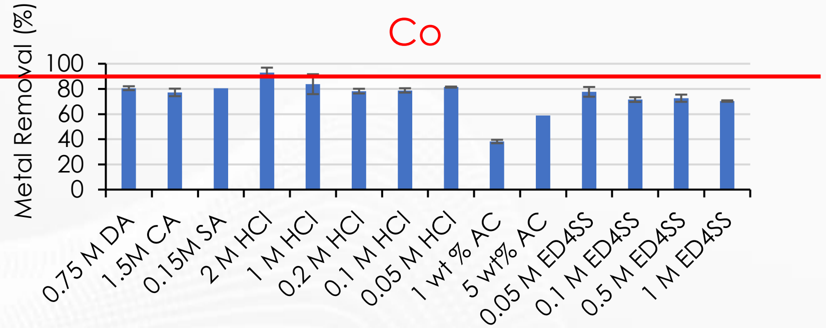
- Higher organic content results in greater metal adsorption
- Higher organic content leads to an increased bending radius

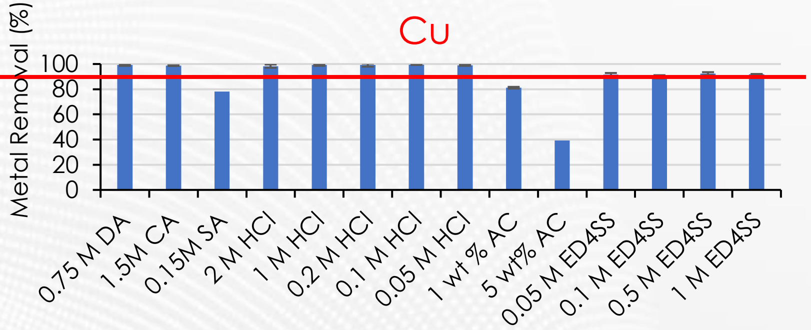
# FAM Performance

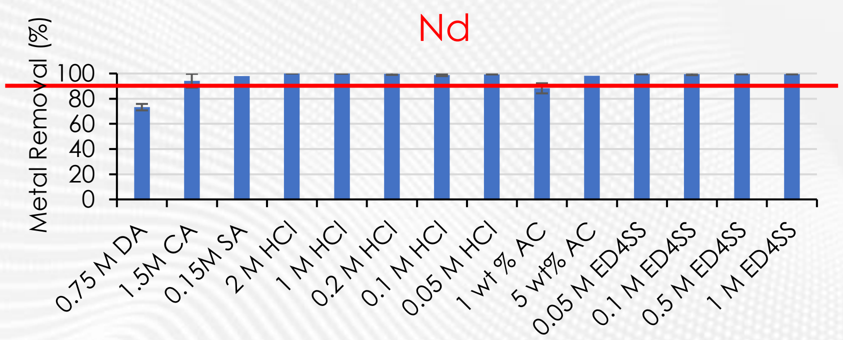


## Regenerability

| | | |
|---|---|---|
| HCl | Hydrochloric Acid | |
| DA | Diglycolic Acid |  |
| CA | Citric Acid |  |
| SA | Succinic Acid |  |
| AC | Ammonium Citrate |  |
| ED4SS | Ethylenediaminetetraacetic acid tetrasodium salt dihydrate |  |



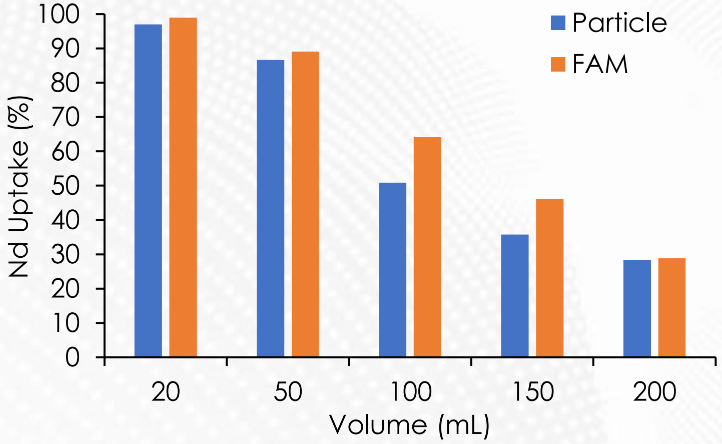



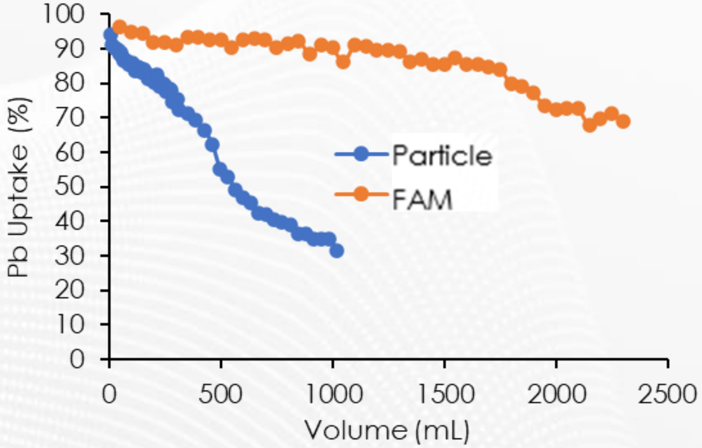
# FAM Performance

## Particle vs. FAM, FAM Cycling Performance

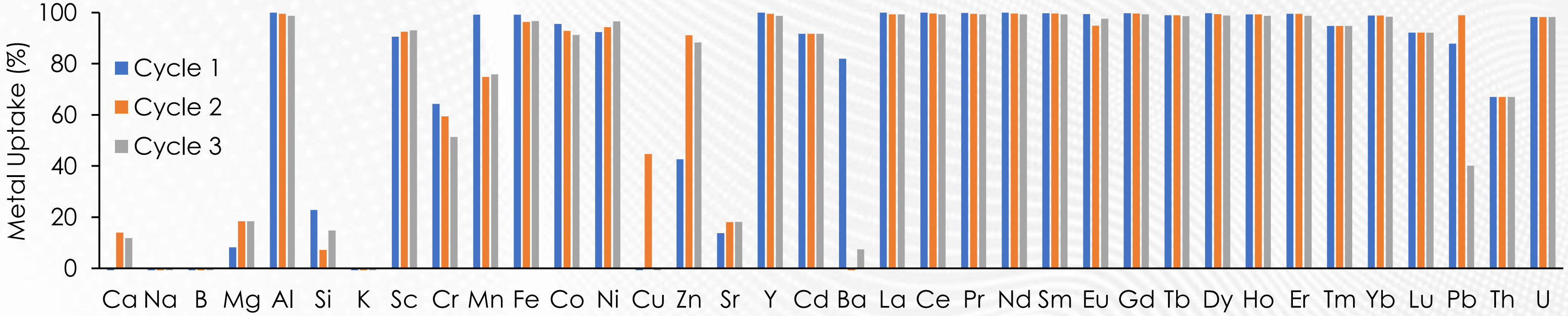
### Particle vs. FAM: Neodymium




### Particle vs. FAM: Lead




### Cycling Test: Critical Mineral Recovery from Pittsburgh Botanic Garden Acid Mine Drainage



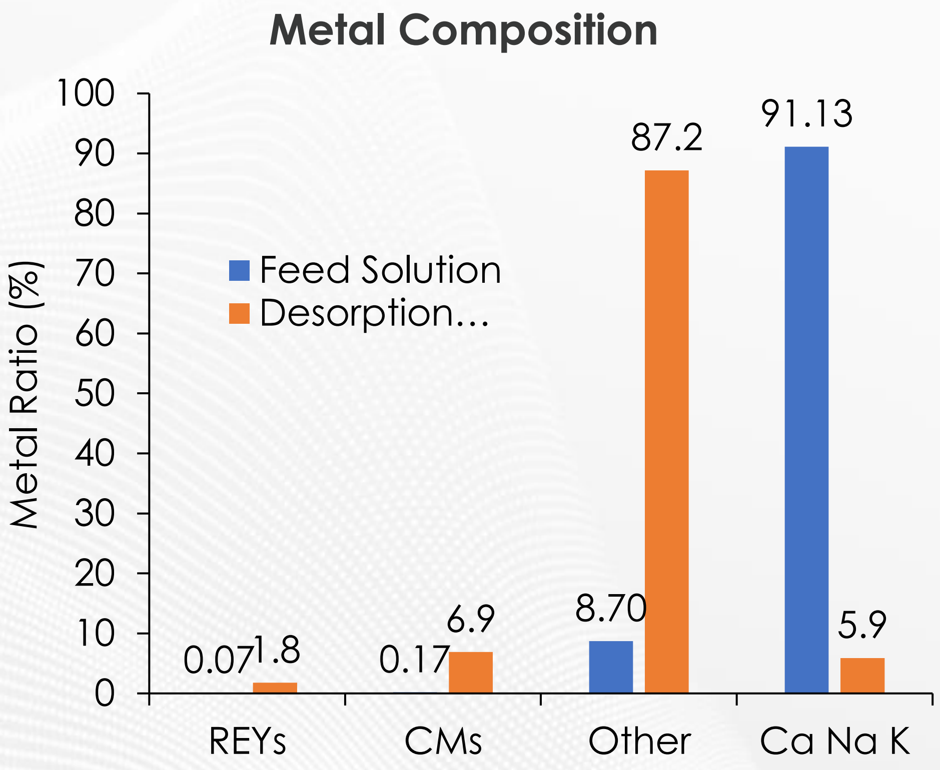
# FAM Performance

## Enrichment of CMs from Coal Ash Leachate

**Concentration Factor**=$[M^+_{eluent}]/[M^+_{fresh}]$

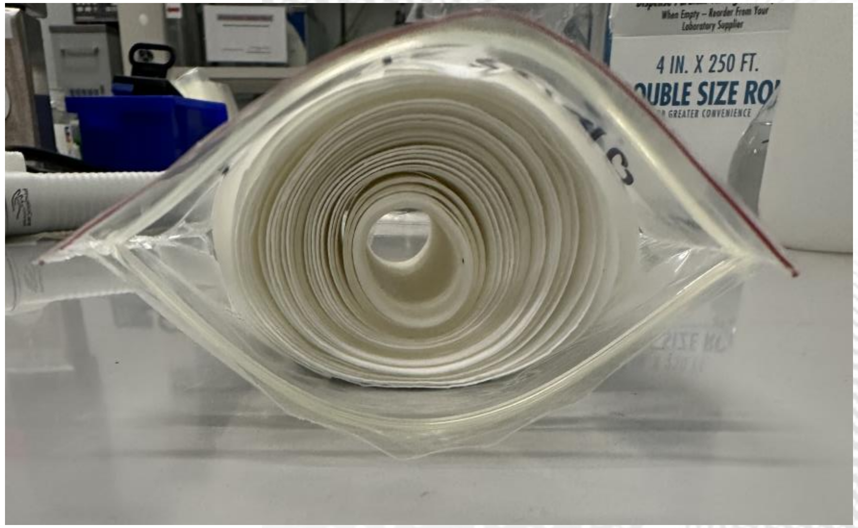
**Enrichment Factor**=$([CM_{eluent}]/[\text{other metals}_{eluent}])/([CM_{fresh}]/[\text{other metals}_{fresh}])$

- Concentrated rare earth elements and Yttrium (REYs) and CMs (**REYs+Ga+Cu+Co+Mn+Ni+V**) by greater than 5.5-10.9X
- Enriched REYs and CMs by up to over 40X



# Conclusions and Continuing Work

- Easy fabrication and scalable production of the FAM sorbent
- High adsorption capacity for CMs
- Strong selectivity toward CMs in diverse coal wastewater streams
- Minimal adsorption of high-salinity background ions (e.g., Ca, K, Na, etc.)
- Tunable mechanical properties for different operational needs
- Good renderability, enabling repeated use and long-term stability
- Continue to study the enrichment of CMs from authentic coal wastewaters
- Further screen substrate and optimize formula for flat sheet sorbent
- Fabricate scalable spiral wound flat sheet material for CM recovery in reactor module
- Design flow channel to promote uniform distribution and reduce pressure drop of the reactor module



# Acknowledgements

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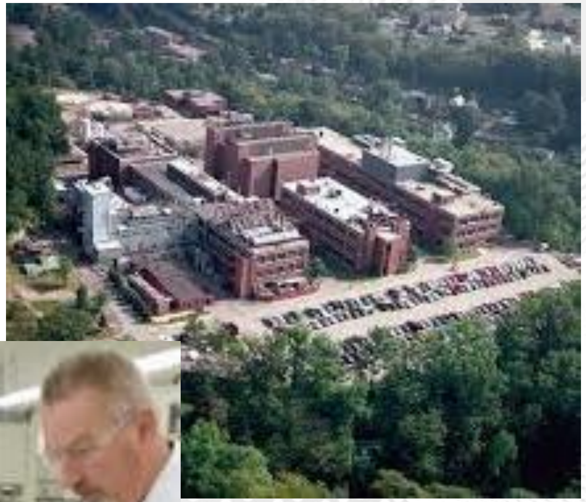
The authors thank Sue Myers and the management at the Pittsburgh Botanic Garden for their time in developing this partnership and for availing the garden grounds to this research.

The authors thank the Pittsburgh Analytical Laboratory, particularly Karen Johnson, William Garber, Robert Thompson, and Phillip Tinker, for performing water and solid sample analyses.



# Call to Action

## How to Partner

### Licenses

NETL looks for licensing partners to mature and deploy Department of Energy (DOE) discoveries through creation of a plan for technology development and deployment, with a high probability for technology commercialization and sharing Intellectual Property (IP) benefits with the public.

### Cooperative Research and Development Agreements (CRADA)

CRADAs allow for joint R&D performed by NETL and participant researchers. These agreements address background IP protection and CRADA-developed IP. CRADAs offer participants the right of first refusal to an exclusive license for jointly developed IP. Cost sharing between NETL and CRADA participant is essential. Small businesses are given preferential consideration.

### Funding Opportunity Announcements (FOA)

NETL leads and partners with organizations to pursue funding opportunities that build upon the lab's unique facilities and capabilities, contingent upon eligibility. In addition, the Department of Energy (DOE) issues Laboratory Calls that are specific to DOE National Laboratory missions, promoting the technology maturation and deployment activities with industry partners.

# NETL Resources

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