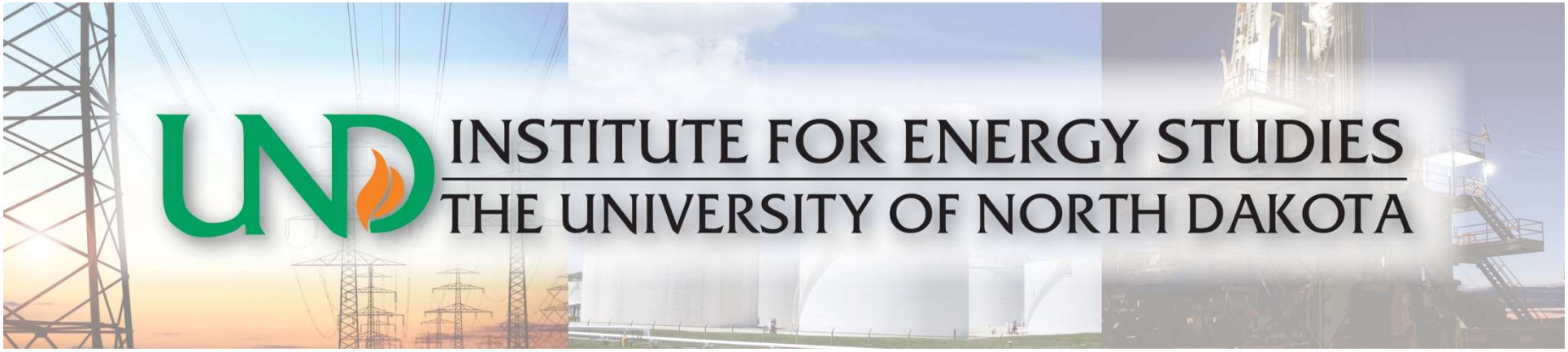

Investigation of Rare Earth Element Extraction from North Dakota Coal-Related Feed Stocks

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Institute for Energy Studies

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2018 NETL Rare Earth Elements Review Meeting
April 10, 2018

DE-FE0027006
Period of Performance:
3/1/2016 to 3/31/2019

Phase 2 Project Team

Technical Team:

- University of North Dakota – Institute for Energy Studies
- Barr Engineering
- Pacific Northwest National Laboratory
- Microbeam Technologies Inc. (MTI)
- MLJ Consulting

Funding Support:

- U.S. Department of Energy – National Energy Technology Laboratory
- Lignite Research Program – North Dakota Industrial Commission
- Great River Energy
- North American Coal Corporation
- Great Northern Properties
- Minnkota Power Cooperative
- UND/ND University System

Advisory Support:

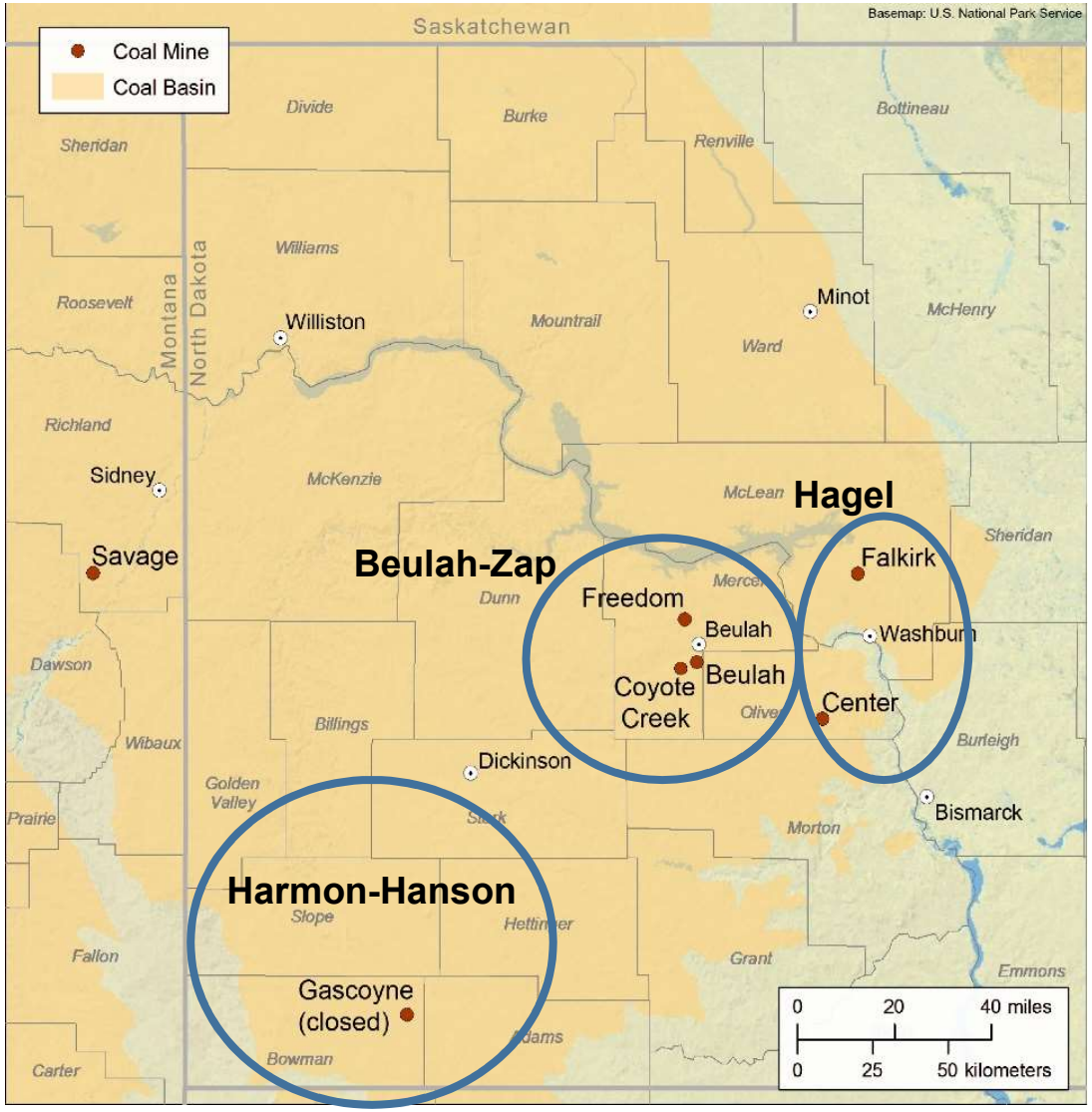
- North Dakota Geological Survey

Presentation Overview

- Background on ND Lignite Industry
- Summary of Phase 1
- Phase 2 Accomplishments to Date
- Phase 2 Next Steps
- Commercialization Concepts
- Acknowledgements
- Questions

ND Lignite Industry

- Host to world's largest lignite deposit at ~350 billion tons
- ~25 billion tons recoverable
- Fort Union group – Paleocene age; 55-65 million years
- State heavily invested in mining/utilization and electric generation – 71% coal electricity in 2016
- Three major coal zones: active mines in Beulah-Zap and Hagel
- ~30 Million tons/yr → 800 year supply
- 7 lignite-fired power plants with > 4,000 MW_e total capacity



Phase 1 Summary

Phase 1 Goal, Objectives and Scope of Work

Overall Goal:

- Develop high performance, economically viable, and environmentally benign concentrating technologies for U.S. coal-related feedstocks

Objectives:

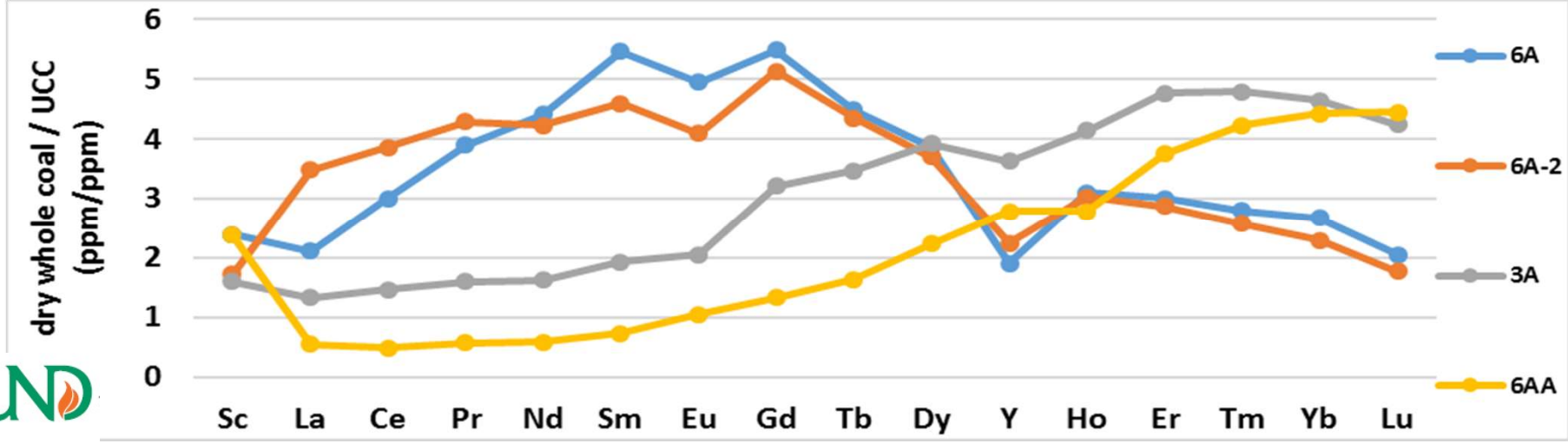
- Identify ND coal-related materials with REE content > 300 ppm
- Develop/test methods to economically concentrate REE to > 2wt%

Scope of Work:

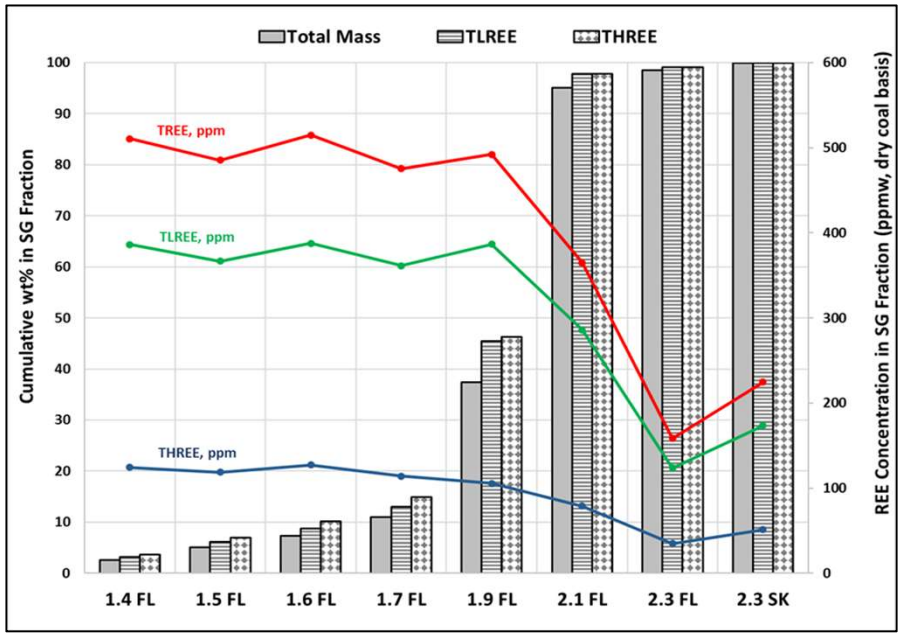
- Sampling
  - Field Samples: Coal, roof, floor, partings
  - Coal Creek Station: DryFining™, fly ash, bottom ash
- Characterization
  - REE abundance
  - Forms and modes of REE occurrence
- Laboratory-scale REE Concentration Testing
- Techno-Economic Analysis
- Bench-scale Design

REE Abundance – Harmon-Hanson Coal Zone

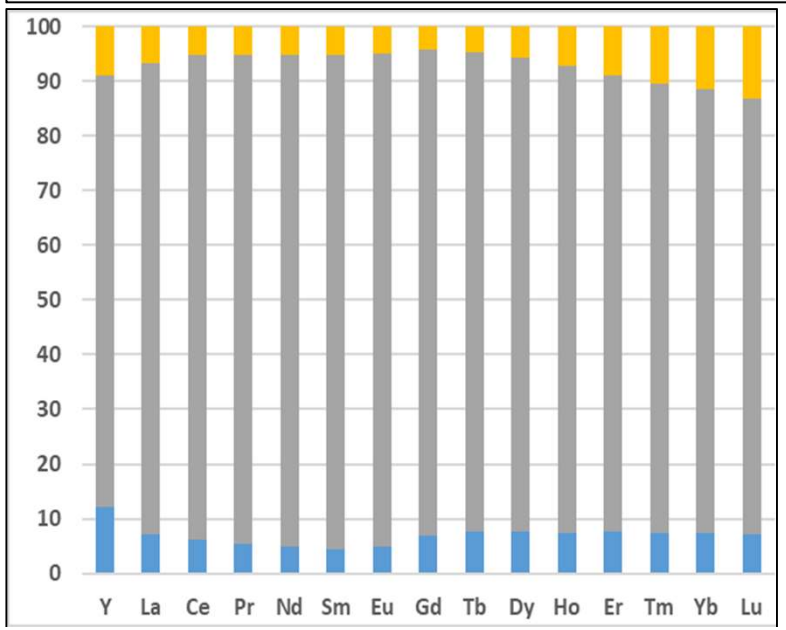
Sample ID	Ash Yield (wt%)	Total REE, ppm (dry mass basis)	Total REE, ppm (ash basis)	HREE/LREE	$ REE/MT Coal
6A-2	36.3	**642**	**1752**	0.28	437
6A	20.1	**564**	**2235**	0.35	599
6A-1	75.5	**449**	587	0.28	307
3A	40.5	**363**	892	0.89	400
3C	60.9	**322**	525	0.43	341
6AA	47.0	212	449	2.06	580
7F	20.9	194	924	0.76	147
15G	32.2	177	541	0.45	242
10	26.2	146	554	0.69	55
5F	15.9	105	659	0.84	137
7E	11.0	76	681	2.19	85
9H	15.7	76	480	1.00	83
5E	10.2	47	462	1.30	86

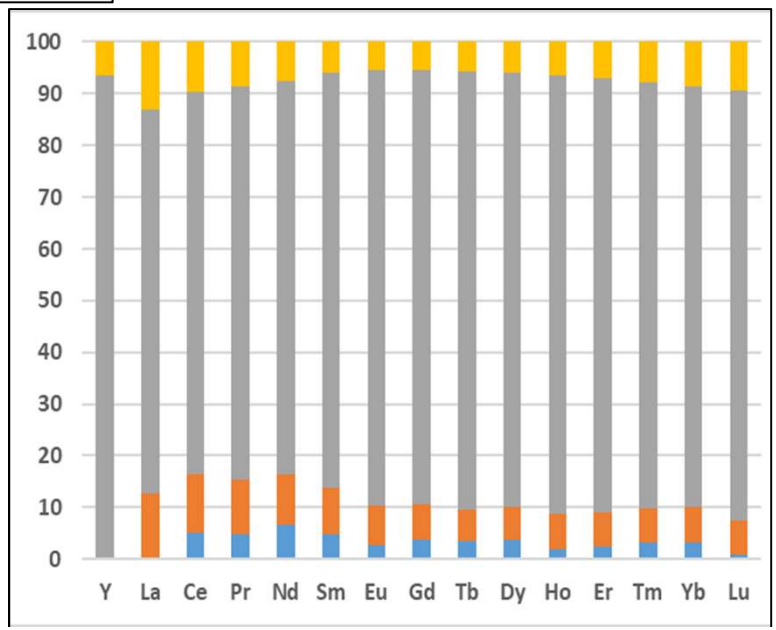


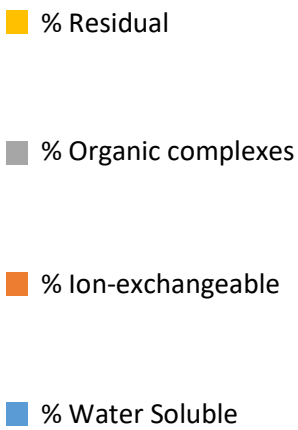
REE Forms and Modes of Occurrence



- Float-sink indicates enrichment in the low SG fractions.
- Sequential solvent extraction testing indicates primarily organic association of the REEs: 85-95%.
- REE in coordination complexes much more prevalent than ion-exchangeable REE.
- Also possibility of carbonates or other HCl-soluble mineral forms





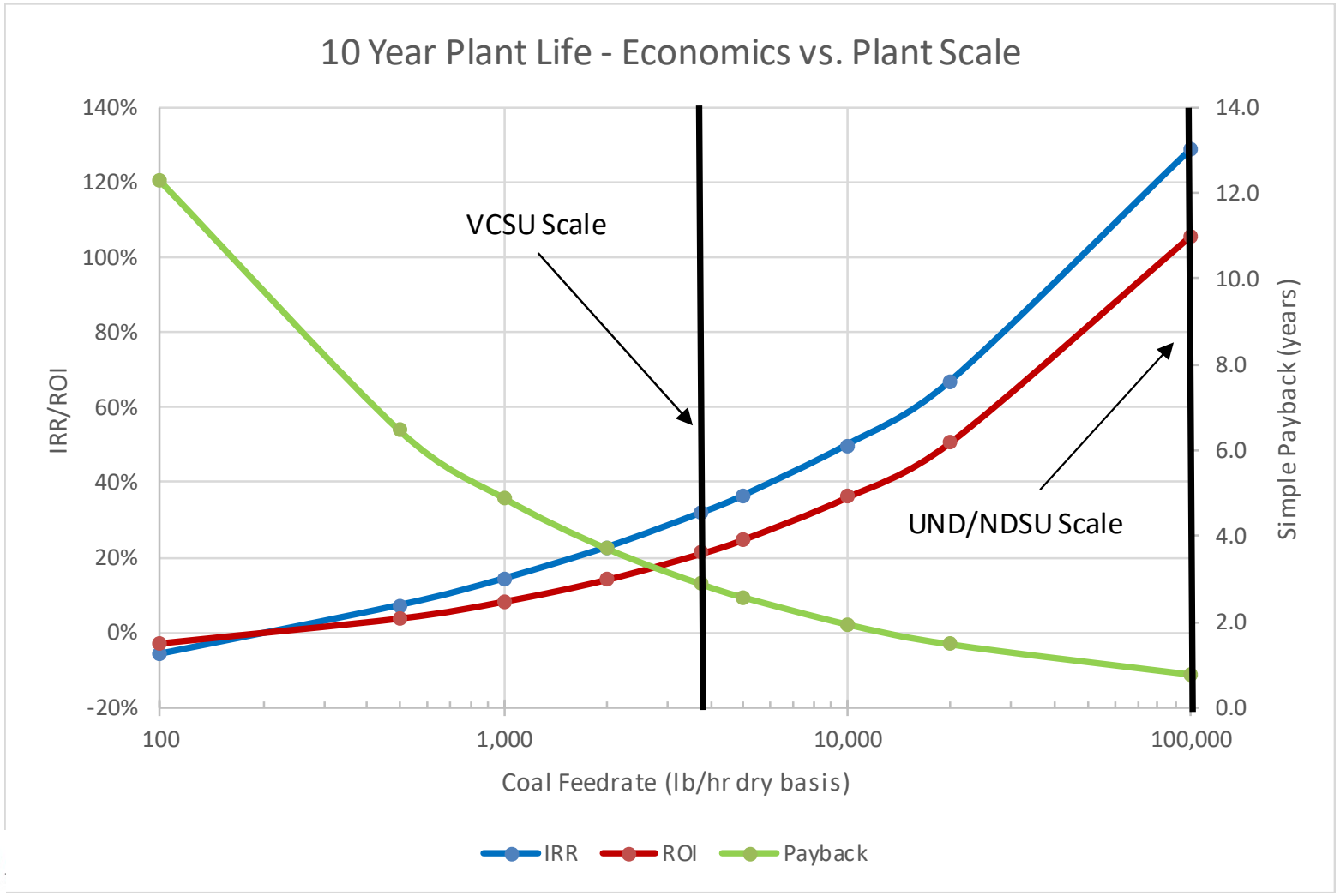


REE Extraction/Concentration Testing - Process Summary and Key Benefits

- REEs easily removed from the **pre-combustion ND lignite** coals due to weak organic bonding
- REE extraction performance summary: **1-step extraction from unprocessed ND lignite**
    1. **>2.0wt%** REE concentration @ 36wt% REE recovery
    2. 1.36wt% REE concentration @ 68wt% REE recovery
    3. 0.8wt% REE concentration @ 86wt% REE recovery
- Also recovers and concentrates several other high value metals: Ge, Ga, Co, V…etc
- Much simpler extraction process than fly ash or mineral-bound REEs (acid/caustic cracking)
- No physical beneficiation required – **process similar to Chinese ion-adsorbed clays**
    1. Solvent-based extraction of REEs from coarsely ground pre-combustion coal
    2. Hydrometallurgy techniques to concentrate REEs in the leachate
- **Mild leaching process** – no high temperatures or pressures; no concentrated acids/bases
- **Selective REE extraction** – only strips the organically associated REEs, leaving the mineral forms and organic matter behind – does not require digestion of entire ore/mineral
- **Coal beneficiation process** – reduces ash yield and preserves organic content/structure; ~100% removal of 'problem' elements such as sodium

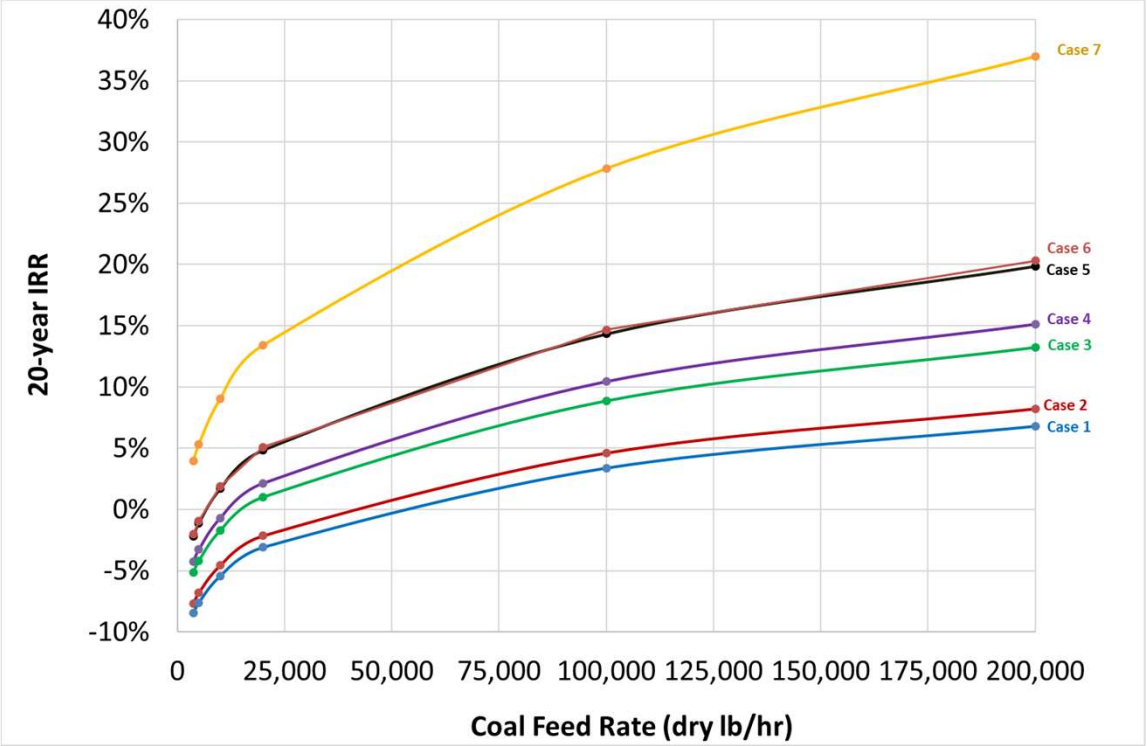
Phase 1 Technical & Economic Analysis (TEA)

Phase 1 TEA – REE with CHP and Activated Carbon Production



Phase 1 TEA – "Stand-Alone" Economics (no activated carbon production)

- Investigated multiple scenarios involving plant scale, REE prices and decreases in CAPEX/OPEX to determine process profitability
- Reduction in economic merit without activated carbon production, but still profitable at > ~25 tph coal feed
- Byproducts likely necessary even at larger scales
- Our approach is unique – clean coal is the byproduct of REE process, thus ability to augment REE revenues



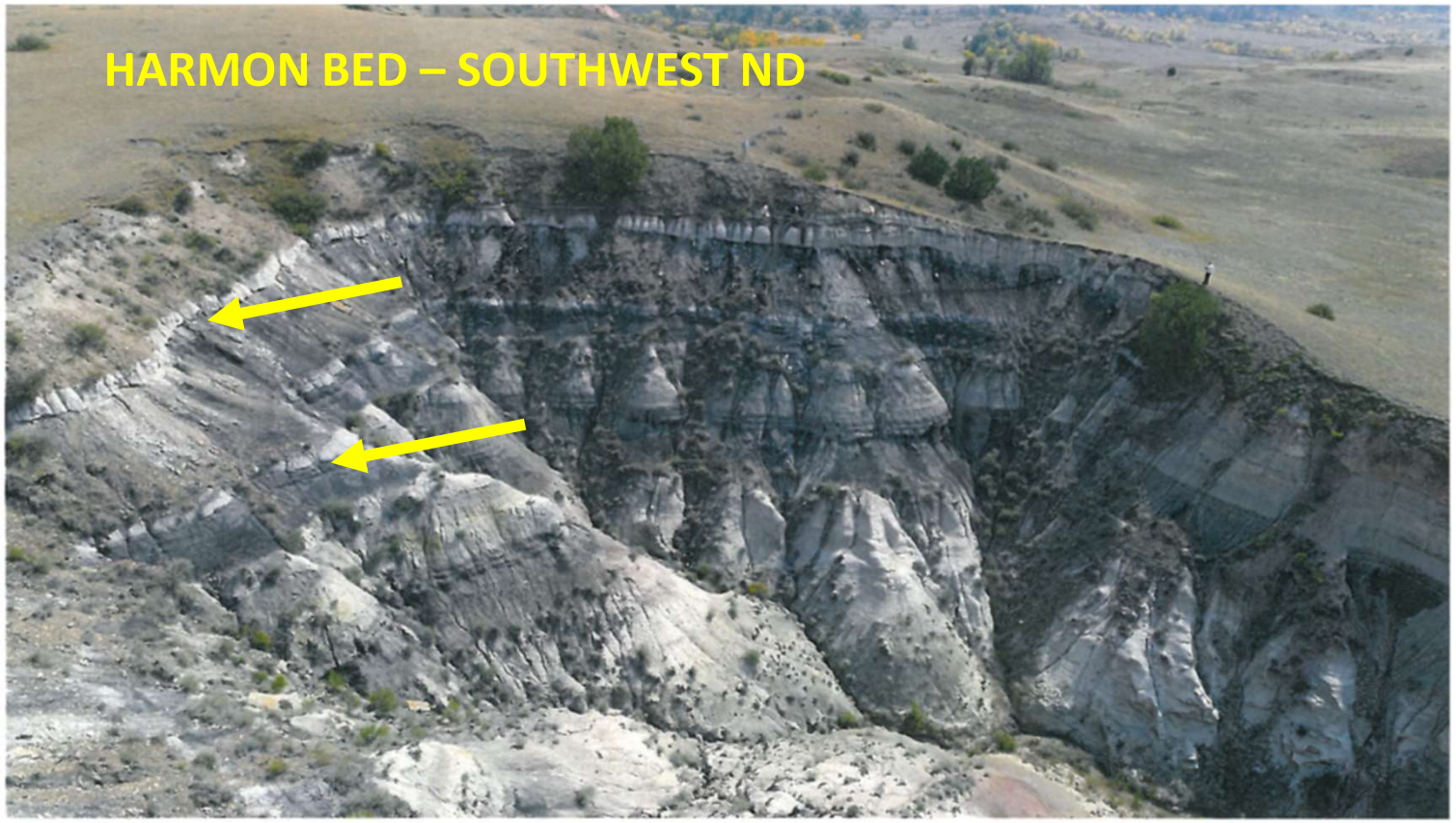
1. Base case analyzed in TEA
2. CAPEX reduced by 10%
3. Target refining only of high-value elements
4. High value elements & CAPEX reduced by 10%
5. Revenue increase by 10%
6. Partial solvent recycle
7. 25% increase in REE prices over 2015

Phase 2 Accomplishments

Phase 2 Objectives and Work Scope

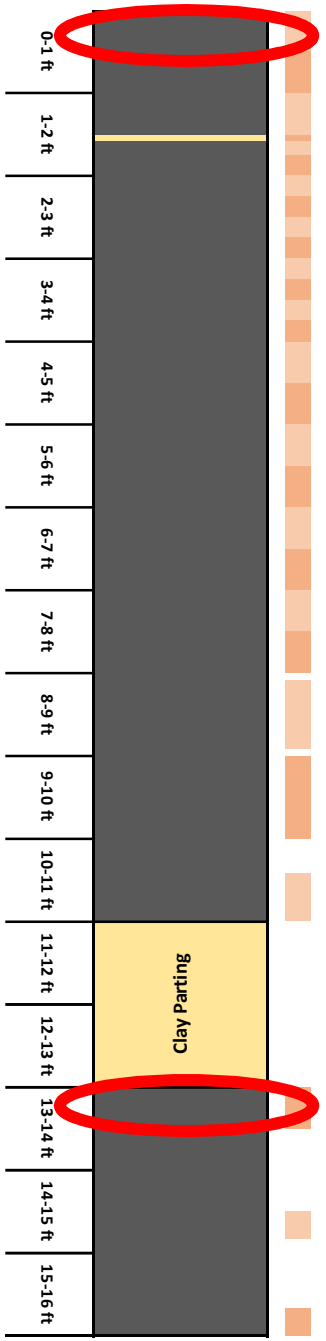
- One of two Phase 2 bench-scale projects awarded under FOA 1202
- Objective: Demonstrate technology at bench-scale (~10 kg coal/hr)
- Phase 1 Testing – batch parametric tests
  - Tune extraction chemistry/conditions to maximize REE selectivity/yield
  - Test additional unit operations to improve overall process and increase REE concentration
  - Optimize conditions and process configuration
- Phase 2 Testing – production testing (~1000 kg total feed)
- Techno-economic assessment
- Preliminary commercialization plan

Feedstock Sourcing for Phase 2



- Focusing on top 6-12” of upper seam and top 6-12” of lower seam
- Upper seam – higher ash material, but > 300 ppm dry coal basis
- Lower seam – very low ash (~5-6wt%), < 300 ppm dry coal basis, but > 1700 ppm ash basis
- Lab testing focusing on upper seam to date

Photo courtesy of North Dakota Geological Survey



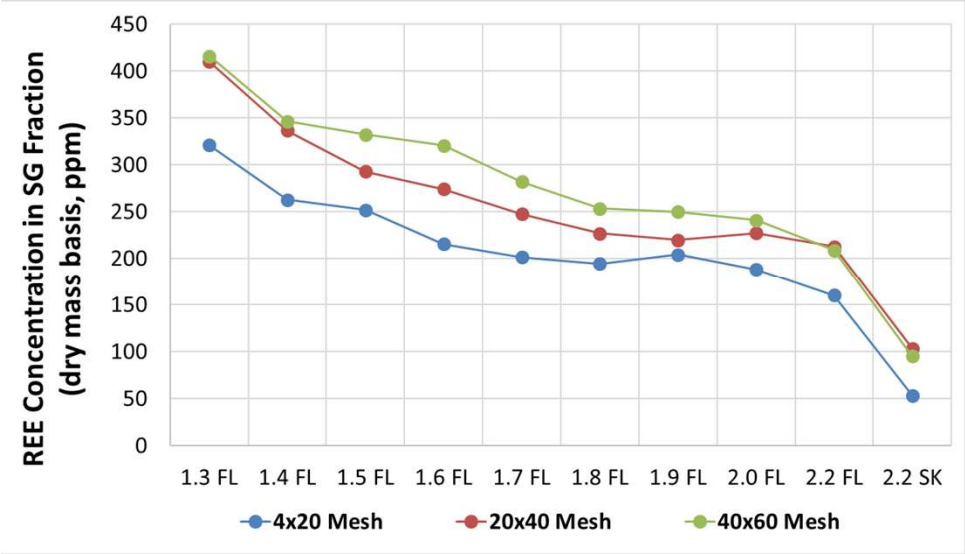
Characterization – Density Separations

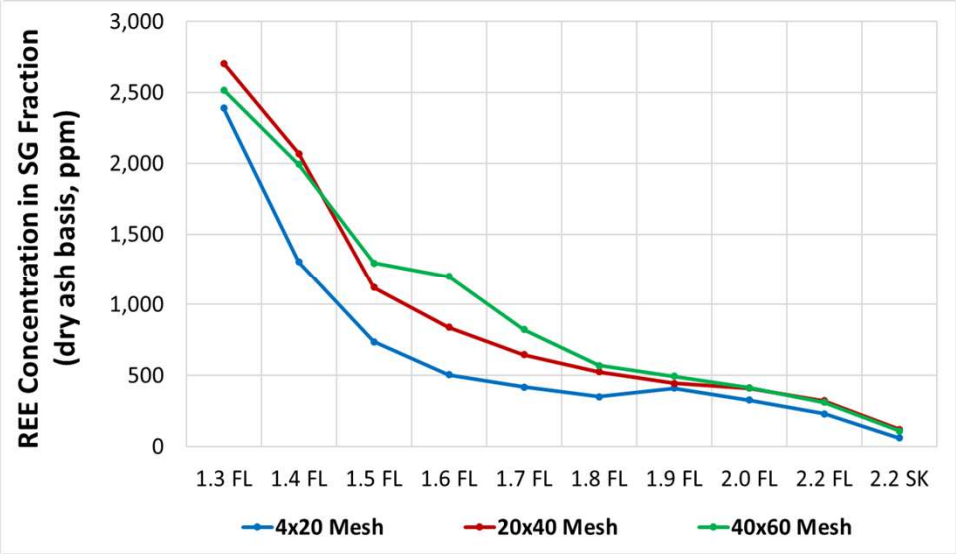
- Goals of Float-Sink Density Separations
  - ✓ SG fractions with highest REE
  - ✓ SG fractions with highest extractable REE
  - ✓ Ash yield as function of SG
  - ✓ Mineralogy as function of SG
  - ✓ Iron partitioning/mineralogy as function of SG
  - ✓ Mineral/REE liberation as function of SG and particle size

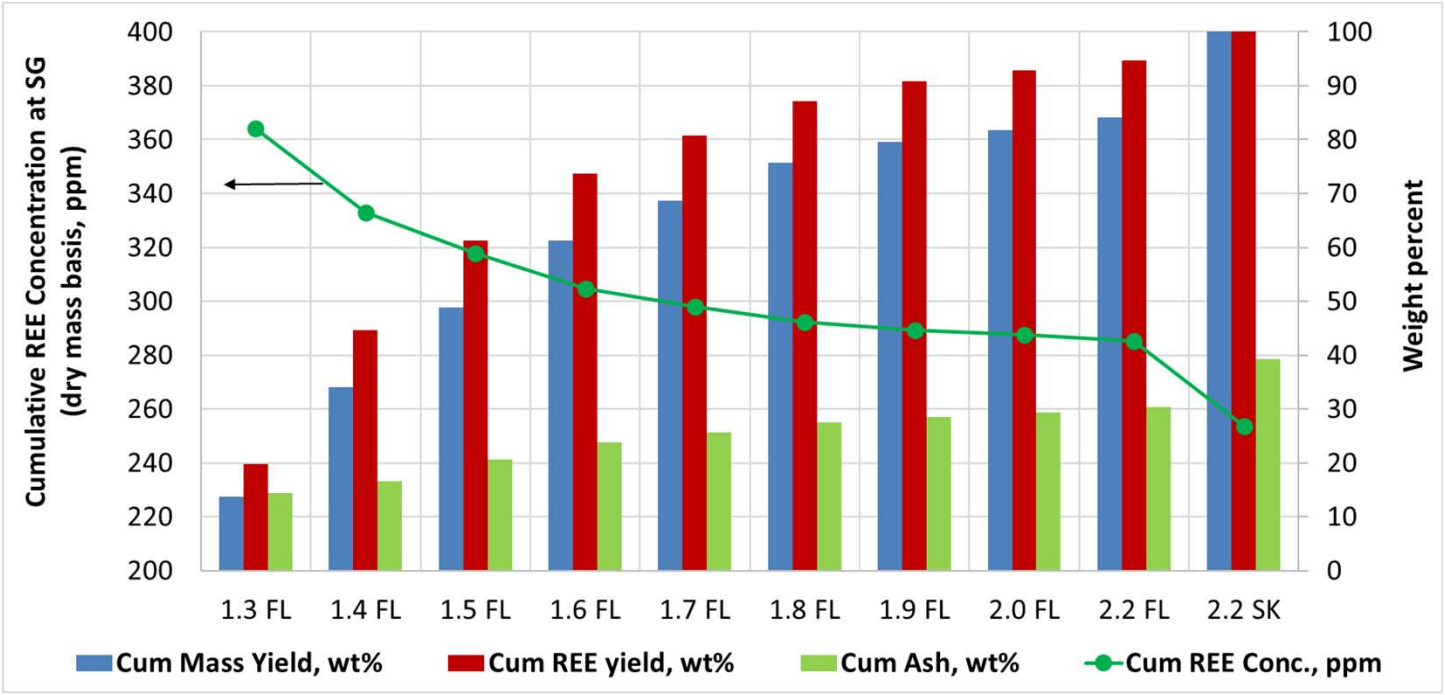
- Ultimate Goal: Select SG range to use for bench-scale testing
  - ✓ Best REE yield (of starting coal)
  - ✓ Best REE extractability
  - ✓ Best total mass yield (of starting coal)
  - ✓ Lowest ash yield of resulting 'clean coal'
  - ✓ Highest REE selectivity (reduce extraction of impurities)

UND

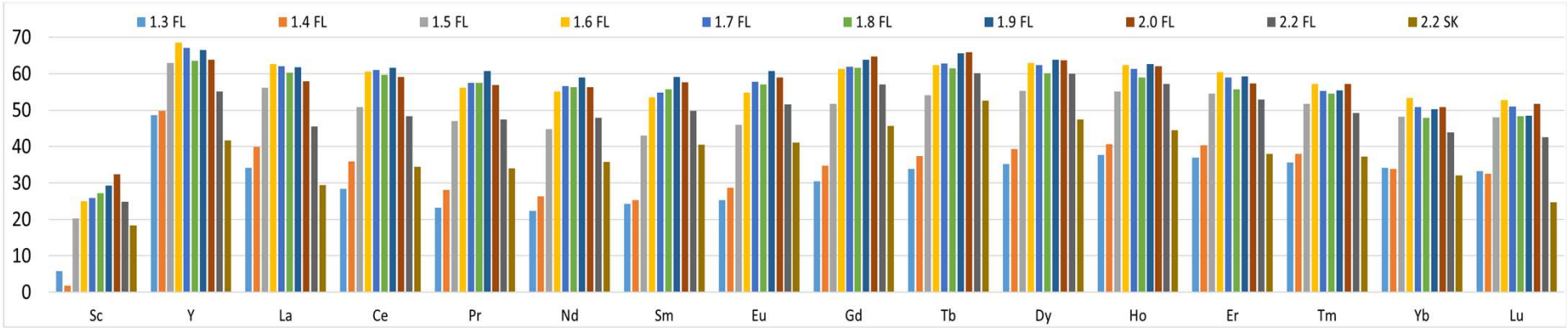
Characterization – Density Separations

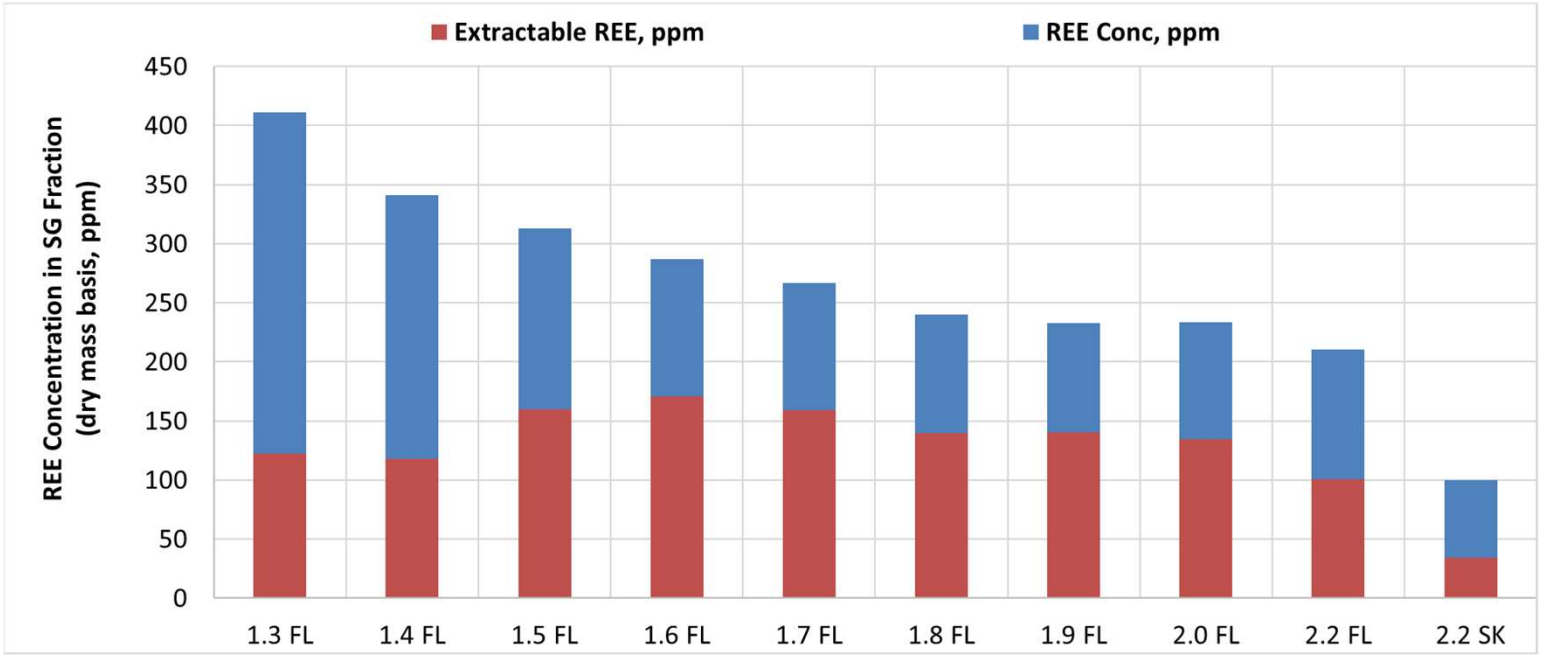






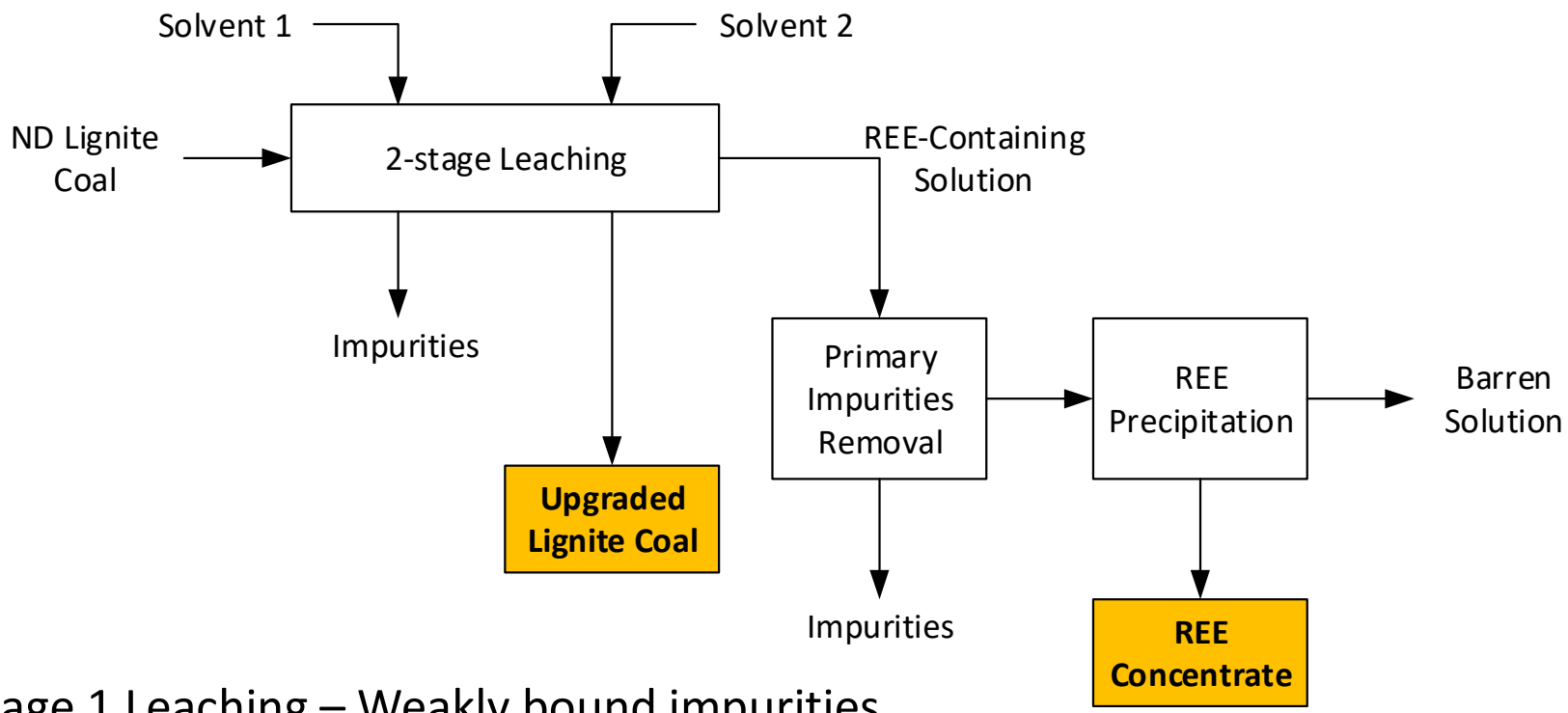
REE Extraction vs. Density Fraction





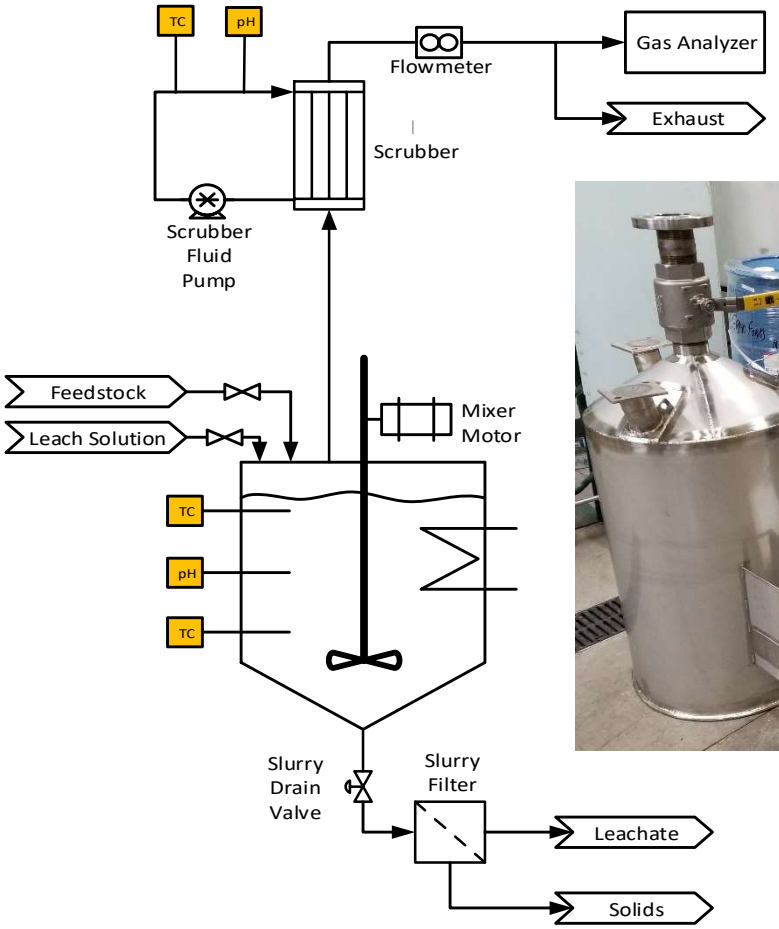
- Short (1hr) contact time – Expect significant improvement with optimization
- Combined results suggest <1.7 SG or <2.0 SG should be the cut point

Bench-Scale Testing: Overall Process Design



- Stage 1 Leaching – Weakly bound impurities
- Stage 2 Leaching – Weakly bound REE
- Impurities removal via pH adjustment and/or oxidation
- REE precipitation via REE-selective precipitating agent
- Preliminary lab-scale tests indicate ability to achieve > 50 wt% REE Product

Bench-Scale Testing: Test System Design



Current Status

- Installing and commissioning bench-scale test system – testing throughout CY18
- Finalizing sample characterization
- 3 Sites selected for large sample collection
  - Mine-sourced lignite – for initial parametric testing
  - Outcrop-sourced lignites – need to wait for spring thaw (May)
- Initiated market analysis
  - Discussion with potential REE refiners – identify concentrate characteristics needed to make salable

Next Steps

- Parametric Testing
  - Process conditions, materials and configuration
- Production Testing
  - ~1000 kg of down-selected feedstock
  - Produce sufficient mass of >2wt% REE concentrate suitable for further processing and detailed characterization
- Commercialization plan & TEA

Commercialization Concepts

Mining Considerations

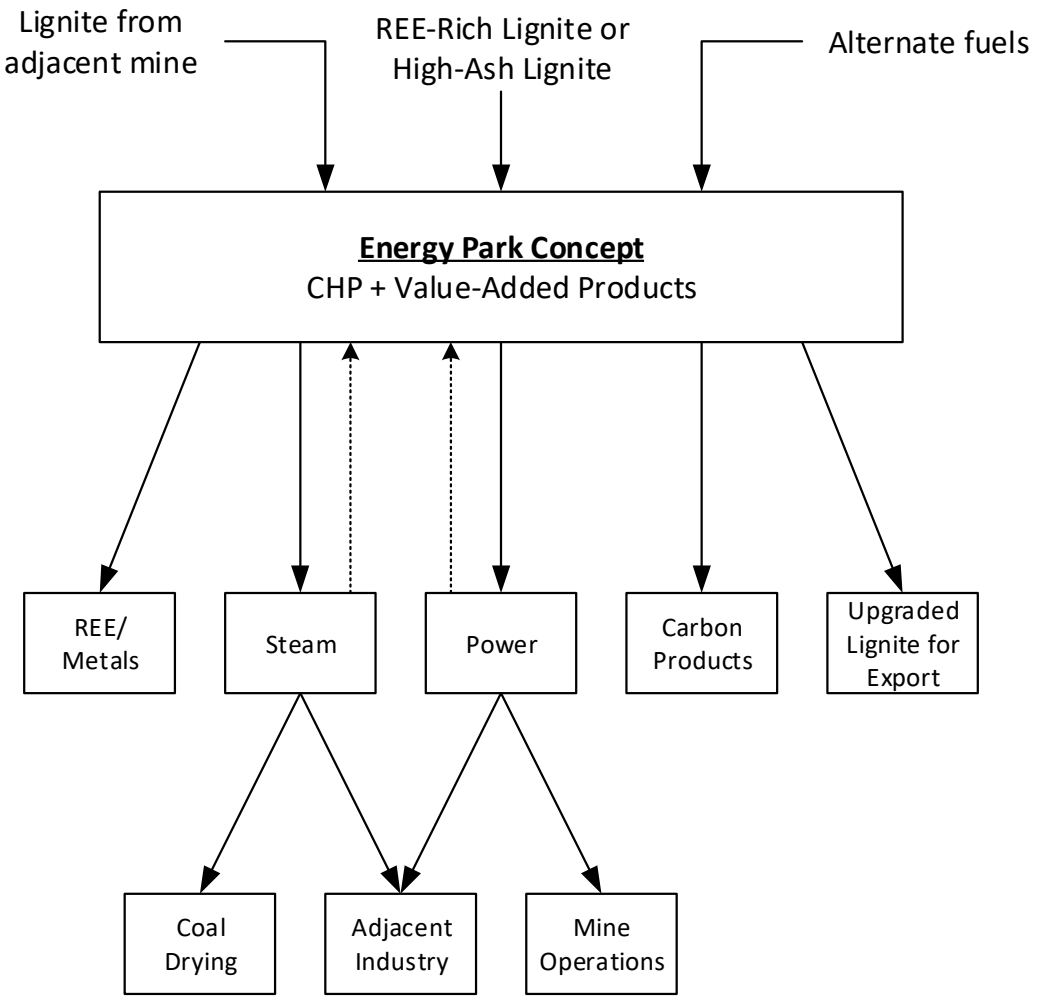
- REE concentrated at margins of roof/floor, below partings and in thin seams
- Low concentration near middle of thick seams
- 'Selective' mining likely needed to separate REE-rich coal from ROM coal – Wirtgen Surface Miner
- Coals near margins and/or thin seams often high ash and may be discarded during mining
- Recovery of REE via UND process can both provide value as well as reclaim and upgrade low-value coal
- Or, starting from lower-ash coal, opportunity to create high-purity carbon-based products with lower cost

ND University System Steam Generation Plants

- Valley City State University currently pursuing installation of activated carbon plant integrated with existing steam generation plant
- Basis of Phase 1 TEA…but really pilot or small demo-scale
- VCSU interested in being platform for pilot testing of fuel conversion technologies and REE
  - ~5 MW_{th} CHP facility: NG and Coal-fired boilers
  - Advanced turbine systems, carbon-based products, coal upgrading, biomass, emissions control systems, CO_2 capture…etc
- https://www.vcsu.edu/president/heat-plant-and-carbon-plant

Integrated Facility Concept

- Potential new mine in Harmon-Hanson coal zone offers next generation opportunities
- Highest value utilization of lignite
- Multiple products
- Potential for export – 800-year supply at current mining rates
- Opportunity to deploy advanced boiler/turbine systems (small coal plant/REMS concepts)
- REE/metals, activated carbon, battery electrode materials, metallurgical coal, carbon fibers…etc
- UND is currently commercializing integrated CHP and activated carbon production system with Valley City State University – **potential host site for pilot-scale demonstrations**



Summary

- Discovered high REE concentration in ND Lignites
- Weakly bound REE – primarily organic association
- Pre-combustion extraction via solvent – process similar to ion-adsorbed clays
- Concentration/recovery of REE via hydrometallurgy
- Potential to exceed >50wt% REE product
- Commercialization concepts:
  - Multi-product integration to augment REE revenues and provide synergies
  - Lignite upgrading and conversion to carbon products
  - VCSU offers pilot-scale testing platform
- Lignite offers promising potential for economical recovery of REE

Acknowledgements

Project Team Members

- Steve Benson, Microbeam Technologies
- Dan Palo, Barr Engineering
- Shane Addleman, PNNL
- Mike Jones, MLJ Consulting
- Ned Kruger, NDGS

Project Sponsor Representatives

- Chuck Miller, NETL Project Manager
- Mike Holmes, Lignite Research Program
- Dennis James, North American Coal
- Charlie Bullinger and Sandra Broekema, Great River Energy
- Craig Bleth, Minnkota Power Cooperative
- Kai Xia, Great Northern Properties
- Rick Tonder, North Dakota University System
- Tisa Mason, Valley City State University

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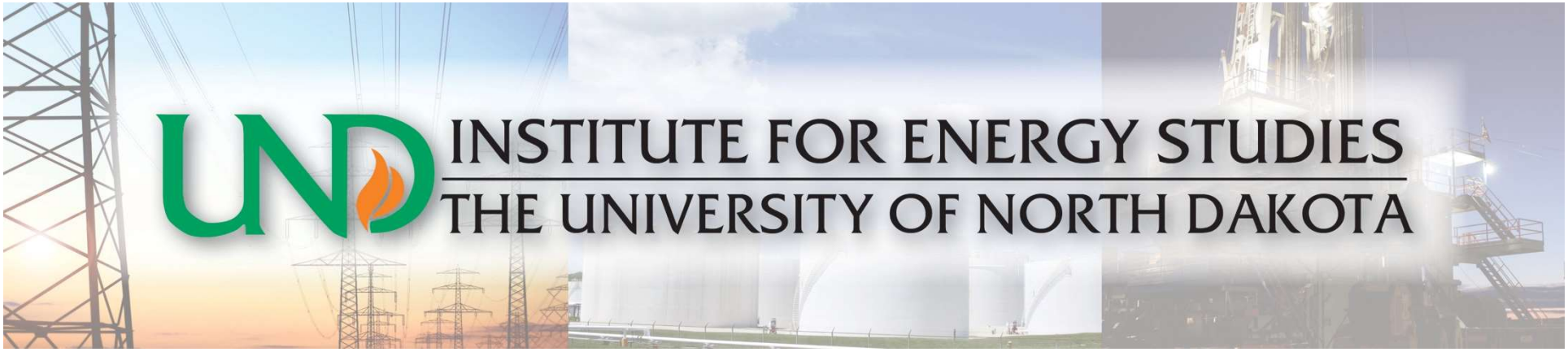
Phase 2 Technical Project Team

- **UND's expertise:**
  - Lignite geology/geochemistry of REEs
  - Advanced analytical techniques involving REEs in coals
  - Chemical/process engineering design and demonstration
- **MTI's expertise (Steve Benson):**
  - Lignite/Low-rank coal inorganic/organic geochemistry
  - Process development/lignite industry experience
  - Business planning/commercialization
- **Barr Engineering's expertise (Dan Palo and team):**
  - Mineral processing, extractive metallurgy
  - Technology and economic feasibility assessment, commercial-scale plant design
  - Market analysis experience
- **PNNL's expertise (Shane Addleman):**
  - REE/F-block chemistry and separations
  - Hydrometallurgy and trace metals recovery technology
- **MLJ Consulting's expertise (Mike Jones):**
  - ND lignite industry
  - Commercialization of lignite-related technologies
- **NDGS' expertise (Ned Kruger, Ed Murphy):**
  - Lignite geology & extensive sample database on REEs



QUESTIONS?

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