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

Dan Laudal
Institute for Energy Studies
University of North Dakota

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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
- Minn Kota 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

<table>
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<tr>
<th>Sample ID</th>
<th>Ash Yield (wt%)</th>
<th>Total REE, ppm (dry mass basis)</th>
<th>Total REE, ppm (ash basis)</th>
<th>HREE/LREE</th>
<th>$ REE/MT Coal</th>
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</table>

![Graph showing REE abundance in various samples](graph.png)
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

10 Year Plant Life - Economics vs. Plant Scale

- IRR
- ROI
- Payback

Coal Feedrate (lb/hr dry basis)

Simple Payback (years)

VCSU Scale

UND/NDSU Scale
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)
Characterization – Density Separations
• Short (1hr) contact time – Expect significant improvement with optimization
• Combined results suggest <1.7 SG or <2.0 SG should be the cut point
• 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

- Electric Heater
- Mixer
- Motor
- Slurry
- Drain Valve
- Slurry Filter
- Leachate
- Solids
- Feedstock
- Leach Solution
- Gas Analyzer
- Flowmeter
- Exhaust
- Scrubber
- Fluid Pump
- pH
- TC
- Scrubber
- Fluid Pump
- pH
- TC
- Slurry Drain Valve
- Slurry Filter
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
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, CO₂ capture...etc

https://www.vcsu.edu/president/heat-plant-and-carbon-plant
• 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

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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?

Dan Laudal, PI
Manager: Major Projects
Institute for Energy Studies, University of North Dakota
daniel.laudal@engr.und.edu
701-777-3456

Steve Benson, Co-PI
Microbeam Technologies Inc.
sbenson@microbeam.com
701-213-7070