Rare Earth Element Extraction and Concentration at Pilot-Scale from North Dakota Coal-Related Feedstocks

2020 NETL Annual Crosscutting Technologies Meeting
Nolan Theaker, Institute for Energy Studies
Team Members

Project Team Members
- UND Institute for Energy Studies
- Microbeam Technologies Inc.
- Barr Engineering Co.
- Rare Earth Salts LLC
- MLJ Consulting LLC
- North Dakota Geological Survey

Project Sponsor Representatives/Executive Advisory Team
- U.S. Department of Energy NETL
- Lignite Research Program
- North American Coal
- Great River Energy
- Minnkota Power Cooperative
- BNI Energy
- Great Northern Properties
- Critical Materials Institute
- North Dakota University System
- Valley City State University
Presentation Overview

- Project Objectives and Scope
- Accomplishments to Date
- Project Next Steps
- Next Steps for Commercialization
- Applicability to Strategic Goals
- Questions
Technology Background

• Extracts REE from low rank coals (LRCs) utilizing weak acids
  – Weak organic associations, rather than mineralized forms (carboxylic acid)

• Utilizes the pre-combustion coal for the feedstock
  – Generates a reduced-ash unique byproduct
    • Usable for AC, humic acid
    • Low fouling ash for boilers
Technology Background (cont)

- Lignite often identified with high HREE/LREE ratios
  - Economically favorable distributions for usage

- Process developed produces other CM
  - High-value Ge and Ga concentrates
Project Objectives and Scope

• As outlined by DOE – NETL in FOA 2003:
  – Development of pilot scale near 5-25% of commercial capacity of >2% REO concentrates utilizing coal-based resources of >300 ppm concentration TREE
  – Economic and environmentally-friendly extraction of REEs from coal

• Goal is to validate the REE extraction from LRC technology at a relevant scale for commercial deployment
Project Objective - Scale

• Previous economic analysis → 5 ton/hr coal output with activated carbon usage
  – Defined as commercial demonstration – able to operate profitably without external financing

• Pilot scoped for 10% capacity of this – 0.5 ton/hr coal output
  – REO Concentrate production of ~100 grams/hr
  – Coal output utilization not included within pilot scope
Project Objective - Coal

• Blended, cleaned coal basis utilized for process feed
  – Lower-REE coal from active mine blended with high-REE coal collected from outcrop seam
  – Coal spiraling utilized as coal cleaning process to remove mineral-rich tailings

• Up to 300 tons of >300 ppm material gathered to date on cleaned-coal basis
Project Objective – REO Purity

• Bench-scale testing resulted in >50% concentrate production as primary product
  – Poor controls on oxalate feed rate resulted in <85% produced in laboratory testing

• Pilot-scale testing and additional sensors/controls expected to keep concentration of primary concentrate >65%
  – Additional research in solid-state REE purification has identified path to >95% concentration
Project Objective - Commercialization

• Identify requirements and knowledge needed for effective commercialization of the technology
  – Including technology and non-technology needs

• Technology Needs:
  – Technology robustness to variable feedstocks (mining variability)
  – Impact of water quality on process streams’
  – Long-term steady-state data
  – Cost improvements as a plus
Project Updates

• Coal acquired from two locations
  – <300 ppm, high HREE/LREE ratio from active mine
    • Substantial improvements by coal cleaning expected due to high clay content within mined sample – UND choice
  – >300 ppm coal from outcrop sample – H-Bed
    • Collected with assistance from NDGS and with state permits

• Pilot design completed with process sizing and control loop development

• Feasibility Study complete with recommendations (to DOE)
Coal Acquisition – Freedom Mine

• 450 tons of 160-200 ppm coal extracted from the Freedom Mine (Buelah, ND) courtesy of NACC
  – Gathered from two distinct fields of coal, with one separated into two variable size fraction piles

• Ash content near 45%, expected strong benefit of spiraling
  – High due to the inclusion of clay layers near the margin of the seam – UND choice
Coal Acquisition – H-Bed

- High-REE content H-Bed sampled in southwestern ND
  - 40 super sacks collected, approximately 44 tons
  - Average of ~480 ppm, with average ash near 35%

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Dry Ash (wt%)</th>
<th>Sc</th>
<th>Y</th>
<th>La</th>
<th>Ce</th>
<th>Pr</th>
<th>Nd</th>
<th>Sm</th>
<th>Eu</th>
<th>Gd</th>
<th>Tb</th>
<th>Dy</th>
<th>Ho</th>
<th>Er</th>
<th>Tm</th>
<th>Yb</th>
<th>Lu</th>
<th>Total REE (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>H Bed - Bags 1-3</td>
<td>36.49</td>
<td>23.0</td>
<td>47.0</td>
<td>70.5</td>
<td>171</td>
<td>21.4</td>
<td>87.2</td>
<td>19.0</td>
<td>4.24</td>
<td>16.6</td>
<td>2.38</td>
<td>12.8</td>
<td>2.30</td>
<td>6.12</td>
<td>0.82</td>
<td>5.14</td>
<td>0.71</td>
<td>490.2</td>
</tr>
<tr>
<td>H Bed - Bags 4-6</td>
<td>36.80</td>
<td>23.9</td>
<td>40.4</td>
<td>51.8</td>
<td>127</td>
<td>16.5</td>
<td>69.7</td>
<td>15.9</td>
<td>3.59</td>
<td>14.1</td>
<td>2.01</td>
<td>11.2</td>
<td>2.09</td>
<td>5.85</td>
<td>0.81</td>
<td>5.41</td>
<td>0.76</td>
<td>391.0</td>
</tr>
<tr>
<td>H Bed - Bags 7-9</td>
<td>21.58</td>
<td>21.9</td>
<td>60.3</td>
<td>78.1</td>
<td>172</td>
<td>21.3</td>
<td>86.0</td>
<td>18.3</td>
<td>4.25</td>
<td>17.8</td>
<td>2.61</td>
<td>14.8</td>
<td>2.81</td>
<td>7.66</td>
<td>1.03</td>
<td>6.47</td>
<td>0.88</td>
<td>516.2</td>
</tr>
<tr>
<td>H Bed - Bags 10-12</td>
<td>30.00</td>
<td>21.3</td>
<td>56.0</td>
<td>101</td>
<td>222</td>
<td>26.2</td>
<td>104</td>
<td>21.8</td>
<td>4.94</td>
<td>19.7</td>
<td>2.82</td>
<td>14.8</td>
<td>2.63</td>
<td>6.88</td>
<td>0.90</td>
<td>5.47</td>
<td>0.75</td>
<td>611.2</td>
</tr>
<tr>
<td>H Bed - Bags 13-15</td>
<td>28.24</td>
<td>16.0</td>
<td>47.5</td>
<td>63.6</td>
<td>144</td>
<td>16.9</td>
<td>66.9</td>
<td>13.7</td>
<td>3.13</td>
<td>13.1</td>
<td>1.93</td>
<td>10.9</td>
<td>2.12</td>
<td>5.92</td>
<td>0.80</td>
<td>5.06</td>
<td>0.70</td>
<td>412.3</td>
</tr>
<tr>
<td>H Bed - Bags 16-18</td>
<td>24.03</td>
<td>13.2</td>
<td>50.2</td>
<td>60.5</td>
<td>124</td>
<td>13.7</td>
<td>52.1</td>
<td>10.2</td>
<td>2.36</td>
<td>10.6</td>
<td>1.62</td>
<td>9.5</td>
<td>1.92</td>
<td>5.43</td>
<td>0.72</td>
<td>4.46</td>
<td>0.63</td>
<td>361.1</td>
</tr>
<tr>
<td>H Bed - Bags 19-21</td>
<td>26.65</td>
<td>19.2</td>
<td>43.7</td>
<td>47.2</td>
<td>111</td>
<td>13.3</td>
<td>55.1</td>
<td>11.8</td>
<td>2.69</td>
<td>11.3</td>
<td>1.71</td>
<td>10.0</td>
<td>1.98</td>
<td>5.67</td>
<td>0.79</td>
<td>5.06</td>
<td>0.70</td>
<td>341.2</td>
</tr>
<tr>
<td>H Bed - Bags 22-24</td>
<td>31.13</td>
<td>22.5</td>
<td>54.0</td>
<td>65.9</td>
<td>157</td>
<td>19.4</td>
<td>81.3</td>
<td>17.8</td>
<td>4.09</td>
<td>17.0</td>
<td>2.49</td>
<td>13.9</td>
<td>2.59</td>
<td>7.13</td>
<td>0.98</td>
<td>6.22</td>
<td>0.85</td>
<td>473.2</td>
</tr>
<tr>
<td>H Bed - Bags 25-27</td>
<td>51.54</td>
<td>19.1</td>
<td>46.5</td>
<td>59.3</td>
<td>141</td>
<td>18.1</td>
<td>76.2</td>
<td>16.7</td>
<td>3.79</td>
<td>15.3</td>
<td>2.20</td>
<td>12.1</td>
<td>2.26</td>
<td>6.19</td>
<td>0.83</td>
<td>5.42</td>
<td>0.73</td>
<td>425.7</td>
</tr>
<tr>
<td>H Bed - Bags 28-30</td>
<td>41.55</td>
<td>23.7</td>
<td>56.6</td>
<td>65.2</td>
<td>155</td>
<td>19.5</td>
<td>81.6</td>
<td>18.0</td>
<td>4.15</td>
<td>16.9</td>
<td>2.45</td>
<td>13.7</td>
<td>2.57</td>
<td>7.15</td>
<td>0.98</td>
<td>6.17</td>
<td>0.85</td>
<td>474.5</td>
</tr>
<tr>
<td>H Bed - Bags 31-33</td>
<td>33.58</td>
<td>23.6</td>
<td>58.4</td>
<td>68.7</td>
<td>167</td>
<td>20.9</td>
<td>87.8</td>
<td>19.3</td>
<td>4.42</td>
<td>18.1</td>
<td>2.62</td>
<td>14.5</td>
<td>2.71</td>
<td>7.55</td>
<td>1.02</td>
<td>6.50</td>
<td>0.89</td>
<td>504.0</td>
</tr>
<tr>
<td>H Bed - Bags 34-36</td>
<td>31.03</td>
<td>26.6</td>
<td>53.9</td>
<td>83.6</td>
<td>211</td>
<td>27.3</td>
<td>115</td>
<td>25.8</td>
<td>5.80</td>
<td>22.1</td>
<td>3.04</td>
<td>15.6</td>
<td>2.72</td>
<td>7.11</td>
<td>0.94</td>
<td>5.98</td>
<td>0.80</td>
<td>607.3</td>
</tr>
<tr>
<td>H Bed - Bags 37-39</td>
<td>32.63</td>
<td>24.8</td>
<td>45.5</td>
<td>77.8</td>
<td>199</td>
<td>25.5</td>
<td>106</td>
<td>23.3</td>
<td>5.09</td>
<td>18.9</td>
<td>2.62</td>
<td>13.3</td>
<td>2.29</td>
<td>6.14</td>
<td>0.82</td>
<td>5.25</td>
<td>0.71</td>
<td>557.0</td>
</tr>
<tr>
<td>H Bed - Bag 40</td>
<td>17.68</td>
<td>23.8</td>
<td>52.5</td>
<td>81.0</td>
<td>202</td>
<td>26.2</td>
<td>110</td>
<td>24.3</td>
<td>5.41</td>
<td>20.8</td>
<td>2.97</td>
<td>15.9</td>
<td>2.87</td>
<td>7.95</td>
<td>1.09</td>
<td>7.21</td>
<td>0.98</td>
<td>585.0</td>
</tr>
</tbody>
</table>
Continuous Pilot Design Summary

• Included within the continuous scope of the pilot:
  – Coal crushing and preparation (to topsize of -4 mesh)
  – Coal spiraling and mineral-rich tailings disposal
  – Leaching of REE and filtration/washing of coal
  – Solution purification and REE precipitation
  – Wastewater treatment of spend waste

• REE concentrates will be fired to oxides batch-wise as produced (kg/day)

• All processing permits (industrial safety, air, water) have been issued
Feasibility Study - Economics

- Plant Scope: 5.5 ton/hr AC feed for REE extraction
  - Includes CAPEX and OPEX of AC plant
    - Salables: AC, REE Concentrates, Ge, Ga
    - Consumables: Acid, Base, Oxalic Acid, Coal Feed, Labor, Electrical/Heat, Maintenance, and REE/CM refining costs
  - Discounted payback at 6.2 years

- Utilizes data from bench-scale data (DE-FE0027006)
  - REE Recovery
  - Consumable Usage
Project Next Steps

- Following Go/No-Go Decision (September 30\textsuperscript{th})
  - Bench-scale testing of blended coal feed to evaluate parametrics critical for pilot-scale operability
  - Procure, construct, and shakedown pilot facility at host site identified
  - Operate pilot facility to process at least 100 tons of >300 ppm material
  - Utilize bench-scale REO separations technology at Rare Earth Salts to evaluate refining costs/viability
  - Update Feasibility Study to reflect pilot recoveries and extrapolated costs
Bench-Scale Testing

• Testing to include:
  – Effect of water quality on REE processing (purity, recovery, etc.)
    • Testing tap, RO, and DI planned
  – REE concentrate purity enhancement
    • Sensors added to bench-scale system to mimic pilot planning
    • Solid-state post-calcining concentrate improvement
  – Parametrics for pilot scale coal blend and steady-state information
    • Acid concentration/pH, base usage, precipitant concentration, seed crystal usage
Pilot Procurement & Construction

• All major and ancillary equipment specified during pilot design to vendor-level specifications
  – Vendors have been contacted and quotes available

• 6-Month period of procurement/construction of plant starting October 1
  – With commissioning, anticipate pilot start near June/July 2021

• Equipment sized for nameplate of 500 kg/hr coal output
Pilot Layout Plans
Pilot Testing Plans

1. Test variable equipment and control setups for operability/reduced chemical or residence time requirements

2. Confirm parameters specified by bench-scale data for optimal economic point

3. Establish long-term steady state (>100 hours in total) and evaluate feedstock variance effects
Bench-Scale REO Separation

• Preliminary separation at lab scale based upon bench-scale concentrates produced
  – Evaluate potential of reducing processing time/costs for lignite-based REE concentrates

• Bench scale processing of pilot products generated (kgs)
  – Using the chosen optimal processing from lab-scale
Technology to Market Path

• Upon successful pilot testing:
  – Commercial demonstration scale viewed as next step for technology
  – Validation of REO concentrate separation valuable for commercialization

• All flows necessary for commercial environmental evaluation will be analyzed during pilot operation
  – Solid, liquid, and gas outputs and required control mechanisms to suit
  – Economic analysis at higher accuracy based on steady-state pilot data
Challenges Remaining for Comm.

• Detailed resource characterization of REE within lignite reserves
  – At bankable level of detail over geographic and stratigraphic layers of seams
  – Development of mine plants to suit these reserves

• Commercial demonstration viability and long-term economic picture
  – Required for significant investment in larger plants to match coal-fired utilities
Potential Commercial Partners

• Lignite owners/miners/users
  – On the project, have two owners, two miners, two users
  – Also have letters of support/interest from two additional owners/users

• Project partners include:
  – Most (>50%) of lignite holders and users within ND
  – All major (>1,000,000 tons/yr) mines with ND
  – Major support from the ND state government
Summary

- Developed technology has been demonstrated to be economic, even at small scales
  - Combined with high-value carbon utilization

- Pilot designs are complete for 500 kg/hr coal output system
  - 10% scale of planned commercial demonstration
  - Construction/operation to continue after Go/No-Go decision September 30th

- Strong industrial and governmental support from ND for project success

- Additional resource characterization/development, particularly of high-REE potential sites in southwestern ND needed
Summary

• Discovered high REE concentrations in ND lignite
• REE weakly bound – primarily as organic complexes
• Pre-combustion extraction permits sale of upgraded coal products
  – REE concentration through selective precipitations
• Preliminary parametric testing complete
  – Finalizing operating parameters and equipment for semi-continuous testing
• Commercialization pathways
  – Multiple products for synergistic economic approach
  – VCSU/GRE Spiritwood for pilot/commercial demo opportunities
Disclaimer - DOE

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.
This presentation was prepared by the University of North Dakota pursuant to an agreement partially funded by the Industrial Commission of North Dakota and neither UND nor any of its subcontractors nor the Industrial Commission of North Dakota nor any person acting on behalf of either:

(A) Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

(B) Assumes any liabilities with respect to the use of, or for damages resulting from the use of, any information, apparatus, method or process disclosed in this report.

Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the Industrial Commission of North Dakota. The views and opinions of authors expressed herein do not necessarily state or reflect those of the Industrial Commission of North Dakota.
Questions?

CONTACT INFORMATION

Nolan Theaker, Co-Principal Investigator
Research Engineer, Institute for Energy Studies
nolan.theaker@und.edu
701-777-6298

Dr. Michael Mann, Project Manager
Director, Institute for Energy Studies
michael.mann@und.edu
701-777-3852