High Yield and Economical Production of Rare Earth Elements from Coal Ash

DOE Contract DE-FE0027167 – Phase 2

Physical Sciences Inc., Andover, MA
Center for Applied Energy Research, Lexington, KY
Winner Water Services, LLC, Sharon, PA

Presentation to:
Rare Earth Elements (REE) Program Portfolio,
2020 Annual Review Meeting (Virtual)
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Presentation Outline

• Phase 2 Project Description and Objectives
• Project Updates
• Next Steps and Concluding Remarks
The PSI, CAER, WWS team provides a complete integrated science, technology, engineering, technology transition, and commercialization solution for DOE/NETL

Key Personnel:

- Physical Sciences Inc (PSI):
  - Dr. Dorin Preda: PI/PM, Lead Chemist
  - Dr. David Gamliel: Lead Chemical Engineering/Process Modeling/TEA
  - Dr. Prakash Joshi: Consultant

- University of Kentucky Center for Applied Energy Research (CAER):
  - Dr. James Hower: Coal Geochemistry, Ash Source Selection, Materials Characterization
  - Dr. John Groppo: Mineral/Ash Processing, Feedstock Logistics, Site Qualification

- Winner Water Services (WWS):
  - Mr. Todd Beers: Chemical & Pilot Plant Engineering, and Technology Commercialization
  - Mr. Michael Schrock: Plant Design, Pilot Plant Operations
Phase 2 Project Description

• Area Of Interest (AOI) 2 program: Pilot Scale Technology
  • Phase 1 – Separation technology demonstrated successfully on bench scale
  • Phase 2 – Design, construction and operation of physical and chemical pilot plants to extract rare earth elements (REEs) from coal ash

• 30-month Phase 2 program: 9/29/2017 – 10/31/2020

• Team:  - Physical Sciences Inc. (PSI), Andover, MA
      - Center for Applied Energy Research (CAER), Lexington, KY
      - Winner Water Services, LLC (WWS), Sharon, PA

• Total Contract Value ~$7.5M = $6M DOE funds + $1.5M Cost Share
Phase 2 Project Objectives

Overall Objective: Demonstrate Phase 1 REYSc separation/enrichment technology at pilot scale in a plant(s) with decoupled operating capacities of ~ 0.4 tpd physical processing, and ~ 0.5 tpd chemical processing.

- Both pilot designs are modular and transportable

<table>
<thead>
<tr>
<th>Performance Parameter</th>
<th>Threshold Value</th>
<th>Objective Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feedstock REYSc Content</td>
<td>&gt;300 ppm (Whole Mass Basis)</td>
<td>&gt;500 ppm (Whole Mass Basis)</td>
</tr>
<tr>
<td>Total REYSc Enrichment in Final Concentrate</td>
<td>&gt;10 wt% (Elemental Basis)</td>
<td>&gt;20 wt.% (Elemental Basis)</td>
</tr>
<tr>
<td>Return on Investment</td>
<td>&lt;12 yr</td>
<td>&lt;10 yr</td>
</tr>
<tr>
<td>Delivered Concentrate Quantity</td>
<td>~50 g</td>
<td>~0.5 kg</td>
</tr>
</tbody>
</table>
Current Program Status

- Collected ~15 tons of coal ash from two different KY plants for physical processing
  - 475 – 550 ppm ash REYSc content

- Physical processing of ash completed 8/2019

- Chemical plant construction, shakedown and start-up completed 11/2019. Chemical processing operations ongoing:
  - ~5 tons of coal ash processed to date
  - ~0.5 kg of REE concentrate produced
  - REYSc content of 10-66 wt.% (elemental basis)

- Techno-economic analysis indicates payback period of <10 years for commercial venture
## Phase II Status

<table>
<thead>
<tr>
<th>Performance Attributes</th>
<th>Commercial Target Performance Requirements</th>
<th>Current Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feedstock REYSc Content</td>
<td>&gt;300 ppm (whole mass basis)</td>
<td>Feedstock REYSc content &gt;500 ppm has been achieved by CAER.</td>
</tr>
<tr>
<td>Total REYSc content in final concentrate</td>
<td>&gt;10 wt.% (elemental basis)</td>
<td>REYSc final content of 10 – 20 wt.% has been recorded at Micropilot scale. Enrichment at chemical pilot scale TBD.</td>
</tr>
<tr>
<td>Return on Investment</td>
<td>&lt;12 years</td>
<td>Detailed economic forecasts ongoing. Cost and revenue drivers, potential plant locations, and potential suppliers and purchasers identified and quantified.</td>
</tr>
<tr>
<td>Delivered Concentrate Quantity</td>
<td>0.05 kg</td>
<td>Achieved ~0.5 kg of concentrate production to date.</td>
</tr>
<tr>
<td>Final REE Yield</td>
<td>&gt;10 wt.%</td>
<td>REYSc yields of 10-30 wt.% recorded in Micropilot and Chemical pilot.</td>
</tr>
<tr>
<td>Cement Substitute Yield</td>
<td>&gt;90 wt.%</td>
<td>Consistent cement substitute yields of 90-93 wt.% recorded in the Micropilot. Similar yield at pilot scale. Cement substitute utility confirmed via standardized testing.</td>
</tr>
<tr>
<td>Solvent/ Reagent Recycling</td>
<td>Solvent &gt;98.5 wt.% Reagent &gt;90 wt.%</td>
<td>Solvent recovery of ~97 wt.% &amp; reagent recovery of 93 wt.% recorded in Micropilot. Solvent recycling efficiency expected to increase at pilot scale (ongoing).</td>
</tr>
</tbody>
</table>

**PSI team anticipates that all target performance requirements will be met.**
Project Update
Rare Earths Recovery Process Overview

- Physical separation stage, followed by a chemical separation stage, followed by a post-processing stage

- Proposed Product: REYSc-enriched mixture (dry concentrate)

- Higher Value Products: REY-rich & Scandium-rich concentrates

- Commercially Viable By-products: Cement substitute, cenospheres, secondary fuel carbon, etc.
Feed Ash Material

- Ash from 2 KY coal fire power plants was recovered and used as process feed.

**Ash C**

**Ash D**

**Graph:**
- Significantly concentration of Nd (~180 ppm), Y (~50 ppm), and Sc (~25 ppm).
- Reasonable (~10 ppm) content of Pr, Gd, Dy.

**Bar Graph:**
- Elements: Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu.
- Concentration in ppm.
Physical Processing Pilot

CAER physical pilot plant operational and >15 tons of coal ash processed to date; >50% yield for ash mass fraction for chemical processing
Output of Physical Processing

- Physical processing creates an ash that is a suitable feed to chemical pilot
  - Low carbon content
  - Low magnetics content
  - Small particle size

- Processed ash collected in super sacks and shipped to chemical pilot in Sharon, PA
Chemical Processing Overview

(1) Digestion
REYSc present in coal ash are dissolved.

(2) Pre-Concentration
Reagent is recovered and recycled.

(3) Liquid-Liquid Extraction (LLX)
REYSc are separated from other elements by selective extraction.

(4) Post-Processing
REY is separated from Sc and concentrated further.

Physically Separated Coal Ash

Product 1
REY-rich mixture

Product 2
Sc-rich mixture

REY-rich material and Sc-rich material are produced from coal ash using simple and efficient process steps.
PSI Micropilot Facility

- Feed ash was first processed in PSI micropilot facility to:
  - Demonstrate target yields and enrichment
  - Determine ash suitability
  - Identify processing challenges and bottlenecks
Micropilot Campaign Demonstrated REE Enrichment

- REYSc concentration sequentially increased as material moves through chemical processing stages

Total REYSc relative content in final micropilot concentrate is >10 wt.%, meeting threshold program objective.
Chemical Pilot Operations

• Chemical pilot designed to process 0.5 tons/day of coal ash

• Situated on the floor of a former torpedo factory

• All unit modules are currently operational
Hot and Cold Side Operations

Hot Side Operation

Cold Side Operation

WWS chemical pilot plant operational and ~5 tons of coal ash processed to date; >20% yield for REYSc concentrate, >50% purity (elemental basis). Deliverable REYSc concentrate production ongoing

Plant can process various pulverized feedstocks: coal ash, coal, refuse
Chemical Pilot Plant Status Update

- Multiple ash batches processed from different sources (Ash C, Ash D)
- Materials sampled at various process stages analyzed via ICP-OES/MS
- Final product has REE concentration of up to 66.7% (elemental basis)
  - Further optimization expected to increase final concentrate REE content

<table>
<thead>
<tr>
<th>Pilot Plant Data</th>
<th>Run 1</th>
<th>Run 2</th>
<th>Run 3</th>
<th>Run 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>REYSc Elemental Content</td>
<td>14%</td>
<td>11.9%</td>
<td>66.7%</td>
<td>29.1%</td>
</tr>
<tr>
<td>Ash</td>
<td>Ash D</td>
<td>Ash C</td>
<td>Ash C</td>
<td>Ash C</td>
</tr>
</tbody>
</table>
Dried Product Composition

Product compositions are above objective key performance parameter (>20 wt.%).

Significant quantities of Nd, Y, Sc and HREE in product material.
• Strength Activity Index or SAI: how the coal ash contributes to the strength of concrete.

• Typically measured as the compressive strength of a standard mortar mix with fly ash substituting for 20 wt% Portland cement; a defined period of curing.

• SAI is then compared as a ratio (percent) to a mortar with 100% Ordinary Portland Cement (OPC).

• ASTM C-618 SAI threshold passing criterion is 75% at 7 days or 28 days (Purple line).

• The processed fine ash utilized at 20% replacement of OPC achieved a strength index greater than 75 by 28 days of curing in 5/6 cases.
Techno-economic Modeling Approach – Phase 1

- Chemical processing and economics modeled in Aspen Plus
  - Capital and operating expenses per model
    - Modified per our team’s experience
  - Result: Pro forma spreadsheet model
- Physical processing economics modeled
  - Capital and operating expenses per CAER and WWS experience
  - Result: Pro forma spreadsheet model
- Integrated process economics modeled
- Added capital expenditures of physical and chemical processes
- Modular, transportable physical and chemical processing plants
- Phase I Model: AACE Level 3

Phase 2 model improves fidelity. Currently AACE Class 2-3
Plant Size: 1200 tpd ash physical processing plant and 600 tpd chemical processing plant

Plant Attributes:
- Co-located at ash source to significantly reduce transportation costs; Decoupled operations
- Modular designs for operational flexibility and transportability

Ash fractions shipped to local markets
- Carbon, magnetic ash; > 200 mesh non-magnetic ash

Annual production of major REE salts, Sc salt, and byproducts:

<table>
<thead>
<tr>
<th>Component</th>
<th>Quantity Produced* tons/year</th>
<th>2020 REE Pricing</th>
<th>2011 REE Pricing</th>
<th>Worldwide Market tons/year</th>
<th>Market Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>REEs</td>
<td>38.2</td>
<td>2.0</td>
<td>10.8</td>
<td>170K</td>
<td>Batteries, Magnets, Alloys, Catalysts</td>
</tr>
<tr>
<td>Scandium</td>
<td>5.8†</td>
<td>20.4</td>
<td>35.0</td>
<td>10-15</td>
<td>Alloys, Catalysts</td>
</tr>
<tr>
<td>Carbon</td>
<td>96K</td>
<td>6.7</td>
<td>4.6</td>
<td></td>
<td>Low-grade Fuel</td>
</tr>
<tr>
<td>Magnetic</td>
<td>20K</td>
<td>7.0</td>
<td>4.0</td>
<td></td>
<td>Magnetite Substitute</td>
</tr>
<tr>
<td>Non-Magnetic &gt;200 Mesh</td>
<td>48K</td>
<td>1.1</td>
<td>0.8</td>
<td></td>
<td>Geopolymer Feed</td>
</tr>
<tr>
<td>Non-Magnetic &lt;200 Mesh</td>
<td>186K</td>
<td>23.5</td>
<td>17.8</td>
<td>71.8M</td>
<td>Cement Substitute (Pozzolan)</td>
</tr>
<tr>
<td>Cenosphere Product</td>
<td>2K</td>
<td>39.3</td>
<td>27.1</td>
<td>~51K</td>
<td>Concrete Additive</td>
</tr>
</tbody>
</table>

- Non-REE products significantly offset effects of REYSc commodity price fluctuations
- Pricing of non-REESc products varies with general economic conditions

†Sc₂O₃ market demand expected to reach 25,000 kg by 2023**
Preparing Project for Next Steps

• Our process and equipment are designed to be flexible: modular and transportable.

• Pilot project utilizes standard commercial equipment that lends itself to scaling up via sizing and/or multiple parallel modular units.

• A team experienced in FEED studies, A/E design, and commercial scale plant design and implementation has been assembled.

• Commercial scale REY and CM production from coal ash possible in 2023-2024 timeframe.

Project develops a technology that will provide a domestic supply of REE-rich concentrate for downstream separation and refinement into individual REE, and for recovery of other critical elements.
Concluding Remarks

- U.S. fly ash is an attractive feedstock with rare earths content sufficient for economical recovery of REYSc, particularly, the heavy rare earth elements

- Demonstrated operational pilot plant (0.4 tpd) for physical separation processes
  - Optimized processes to produce selected ash fraction as feedstock for the chemical processing
  - Valuable by-products: cement substitute, cenospheres, secondary fuel carbon

- Pilot plant for chemical processing (0.5 tpd) now operational
  - Optimized processes validated in micropilot plant operations
  - REYSc concentrates as main products (~66% content achieved on elemental basis)
  - Beneficiated ash as valuable by-product

- Commercially viable processes demonstrated by techno-economic analysis
  - Currently AACE Class 2/3

- Next steps include increase in product purity and production of additional CMs
Thank you!
Backups
## AACE Estimate Classes

<table>
<thead>
<tr>
<th>ESTIMATE CLASS</th>
<th>MATURITY LEVEL OF PROJECT DEFINITION DELIVERABLES</th>
<th>END USAGE</th>
<th>METHODOLOGY</th>
<th>EXPECTED ACCURACY RANGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Class 5</td>
<td>0% to 2%</td>
<td>Concept screening</td>
<td>Capacity factored, parametric models, judgment, or analogy</td>
<td>L: -20% to -50%  H: +30% to +100%</td>
</tr>
<tr>
<td>Class 4</td>
<td>1% to 15%</td>
<td>Study or feasibility</td>
<td>Equipment factored or parametric models</td>
<td>L: -15% to -30%  H: +20% to +50%</td>
</tr>
<tr>
<td>Class 3</td>
<td>10% to 40%</td>
<td>Budget authorization or control</td>
<td>Semi-detailed unit costs with assembly level line items</td>
<td>L: -10% to -20%  H: +10% to +30%</td>
</tr>
<tr>
<td>Class 2</td>
<td>30% to 75%</td>
<td>Control or bid/tender</td>
<td>Detailed unit cost with forced detailed take-off</td>
<td>L: -5% to -15%  H: +5% to +20%</td>
</tr>
<tr>
<td>Class 1</td>
<td>65% to 100%</td>
<td>Check estimate or bid/tender</td>
<td>Detailed unit cost with detailed take-off</td>
<td>L: -3% to -10%  H: +3% to +15%</td>
</tr>
</tbody>
</table>

Notes: [a] The state of process technology, availability of applicable reference cost data, and many other risks affect the range markedly. The +/- value represents typical percentage variation of actual costs from the cost estimate after application of contingency (typically at a 50% level of confidence) for given scope.