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

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,
2021 Annual Review Meeting (Virtual)
25 May 2021

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Presentation Outline

• Phase 2 Project Description and Objectives
• Project Updates
• Next Steps and Concluding Remarks
Phase II Team

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 Engineer/Process Modeling/TEA
  - Dr. Bryan Sharkey: Process Development, ICP-OES Analysis
  - 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
  - Dr. Robert Jewell: Pozzolanicity Testing
- **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 and additional CMs (Sc, Al)

- **Phase 2 program: 9/29/2017 – 10/31/2021**

- **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 ~$8.75M = $7M DOE funds + $1.75M Cost Share**
Phase 2 Project Objectives

- Overall Objective: Demonstrate Phase 1 REE 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
  - Demonstrate production of high purity REY product and of critical material products (Sc, Al)

<table>
<thead>
<tr>
<th>Performance Parameter</th>
<th>Threshold Value</th>
<th>Objective Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feedstock REY+Sc Content</td>
<td>&gt;300 ppm</td>
<td>&gt;500 ppm</td>
</tr>
<tr>
<td>Return on Investment</td>
<td>&lt; 12 years</td>
<td>&lt; 10 years</td>
</tr>
<tr>
<td>REY-enriched Product</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quantity (REY salts)</td>
<td>100 g</td>
<td>300 g</td>
</tr>
<tr>
<td>REY-enriched Oxide Purity (total REY content - elemental basis)</td>
<td>&gt;85%</td>
<td>&gt;90%</td>
</tr>
<tr>
<td>Sc-enriched Product</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quantity (Salt/Oxide)</td>
<td>1 g</td>
<td>2 g</td>
</tr>
<tr>
<td>Sc-enriched Oxide/Salt Purity (Sc content - elemental basis)</td>
<td>&gt;85%</td>
<td>&gt;90%</td>
</tr>
<tr>
<td>Aluminum Product</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quantity (oxide type material)</td>
<td>100 g</td>
<td>300 g</td>
</tr>
<tr>
<td>Purity (Al content elemental basis)</td>
<td>&gt;50%</td>
<td>&gt;68%</td>
</tr>
</tbody>
</table>
Current Program Status

• **Physical processing – completed:**
  • Collected ~15 tons of coal ash from two different KY plants for physical processing
  • 475 – 550 ppm ash REYSc content

• **Chemical processing operations – initial phase completed/additional operations ongoing:**
  • > 10 tons of coal ash processed to date
  • ~1.2 kg of REE concentrate produced
  • REY product:
    • Initial Phase 2 REYSc product: ~10-68 wt.% (elemental basis)
    • Identified and experimentally tested pathways to increase purity to >85 wt%
  • Sc-product being produced using a PSI-proprietary LLX process
    • 12X enrichment obtained in a single cycle
    • Multiple cycles projected to achieve target purity (>85 wt.%)
  • Al-product of 68-90 wt.% purity obtained in initial runs
# Phase 2 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</td>
</tr>
<tr>
<td>Total REYSc content in final concentrate</td>
<td>&gt;10 wt.% (elemental basis)</td>
<td>REYSc final content of 10 – 60 wt.% has been recorded at pilot scale</td>
</tr>
<tr>
<td>Delivered Concentrate Quantity</td>
<td>0.05 kg</td>
<td>Achieved ~0.5 kg of concentrate production to date. Delivered 51g of concentrate to DOE with &gt;50% REYSc content</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>Yields of 90-93 wt.% recorded in the micropilot and 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 confirmed at pilot scale.</td>
</tr>
<tr>
<td>REY-product</td>
<td>&gt;85 wt.% (elemental basis)</td>
<td>Initial Phase 2 REYSc product: ~10-68 wt.% (elemental basis) Identified pathways to increase purity to &gt; 85 wt.%. Optimization and scale-up ongoing.</td>
</tr>
<tr>
<td>Sc-product</td>
<td>&gt;85 wt. % (elemental basis)</td>
<td>12X enrichment obtained in a single cycle using a PSI-proprietary LLX process Multiple cycles projected to achieve target purity (&gt;85 wt.%).</td>
</tr>
<tr>
<td>Al-product</td>
<td>&gt;56 wt.% (elemental basis)</td>
<td>Al-product of 68-90 wt.% purity obtained on multiple runs</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 Products**: REY, Sc and Al products with high purity

- **Commercially Viable By-products**: Cement substitute, cenospheres, secondary fuel carbon, etc.

*All purity values expressed as elemental relative content*
Feed Ash Material

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

![Ash C](image1)

![Ash D](image2)

![Graph](chart)

**Significant content of Nd (~180 ppm), Y (~50 ppm), and Sc (~25 ppm)**

**Reasonable (~10 ppm) content of Pr, Gd, Dy**
CAER physical pilot plant processed >15 tons of coal ash >50% yield for ash mass fraction for chemical processing
Output of Physical Processing

- Physical processing creates an ash fraction that is a suitable feed to chemical pilot
  - Low carbon content
  - Low magnetics content
  - Small particle size
- Processed ash collected in super sacks, shipped to and processed in the chemical pilot in Sharon, PA
REY-rich material, Sc-rich material and Al-product are produced from coal ash using simple and efficient process steps.
PSI Micropilot Facility

- **PSI micropilot is used to:**
  - Demonstrate target yields and enrichment
  - Determine ash suitability
  - Identify and troubleshoot processing challenges and bottlenecks for the pilot plant
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
WWS chemical pilot plant operational: ~10 tons of coal ash processed to date.
>20% yield for REYSc concentrate, >50% purity (elemental basis).
Produced REYSc concentrate deliverable for the initial Phase II.

Currently focusing on LLX optimization to produce final Phase II deliverable
Product compositions exceed objective key performance parameter (>20 wt.%). Significant quantities of Nd, Y, Sc and HREE in product material.
LLX Optimization: Feedstock Preparation

- Digest from ash dissolution is concentrated via various methods

- Dried material is dissolved to attain target concentration

- Multiple parameters of the resulting solution determine the yield and selectivity of rare earths during subsequent LLX operation

**Feedstock LLX preparation → key steps prior to LLX optimization**
LLX Optimization Approach

- **Liquid-Liquid Extraction (LLX) Optimization Goals**
  - Increase REE yield
  - Increase REE selectivity to increase purity

- **Secondary Optimization goals**
  - Optimize # of stages required in LLX
  - Minimize amount of extractant

- **Steps to Optimization**
  - Feedstock normalization
  - LLX development and evaluation
  - Continuous LLX runs to validate assumptions
  - Iterative parameter optimization (highest recovery and relative content)
  - Samples and data generation, analysis and conclusions
Sc Recovery Summary

- **Methodology:**
  - Validated a company proprietary LLX process for selective recovery of scandium, a high value product
    - Process developed under PSI IRAD project

- **Preliminary Results:**
  - Using this LLX process we were able to increase Sc relative content from 3.3 wt.% in the feed to 41 wt.% in the strip phase
  - Indicates over 12X enrichment in scandium content for a single cycle
  - 51% Sc yield using 2 stripping stages
Sc Recovery Process Relative Content Increase

- Feed contains 3.3 wt.% scandium relative content
- Strip contains 41.6 wt.% scandium relative content (51% yield with two stages)

>12X concentration increase of Sc after LLX
Pozzolanicity Testing – Strength Activity Index

- 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 (%) 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.
Roadmap: Chemical Plant Development Path

- Typical chemical plant project development path:
  1. Conceptual Design
     - WWS Pilot Plant Developed/Operational; Analysis of Alternatives
  2. Pre-feasibility Study
     - Project Option Selected, Process & Technology Risks Retired
     - WWS Pilot Plant Optimized for Anticipated Feedstock(s)
  3. Feasibility Study
     - FEED: Front End Engineering Design
  4. Implementation

The current program brings us through the concept stage, and develops TEA (which is typically done in Pre-Feasibility work)
Capacity, Products and Annual Production

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

- **Ash fractions transported to local markets**
  - Carbon, magnetic ash, > 200 mesh non-magnetic ash

- **Commercial Plant Size:** 1200 tpd ash physical processing plant and 600 tpd chemical processing plant
  - Annual production of major REE salts, Sc salt, and byproducts:

<table>
<thead>
<tr>
<th>Component</th>
<th>Quantity Produced* (tons/year)</th>
<th>Portion of Revenue (%)</th>
<th>Worldwide Market (tons/year)</th>
<th>Market Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>REEs</td>
<td>38.2</td>
<td>1.0</td>
<td>170K</td>
<td>Batteries, Magnets, Alloys, Catalysts</td>
</tr>
<tr>
<td>Scandium</td>
<td>5.8†</td>
<td>26.1</td>
<td>10-15</td>
<td>Alloys, Catalysts</td>
</tr>
<tr>
<td>Carbon</td>
<td>96K</td>
<td>6.2</td>
<td>71.8M</td>
<td>Low-grade Fuel</td>
</tr>
<tr>
<td>Magnetic</td>
<td>20K</td>
<td>5.4</td>
<td>4.0</td>
<td>Magnetite Substitute</td>
</tr>
<tr>
<td>Non-Magnetic &gt;200 Mesh</td>
<td>48K</td>
<td>1.0</td>
<td>0.8</td>
<td>Geopolymer Feed</td>
</tr>
<tr>
<td>Non-Magnetic &lt;200 Mesh</td>
<td>186K</td>
<td>23.9</td>
<td>17.8</td>
<td>Cement Substitute (Pozzolan)</td>
</tr>
<tr>
<td>Cenosphere Product</td>
<td>2K</td>
<td>36.4</td>
<td>~51K</td>
<td>Concrete Additive</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2018 REE Pricing</td>
<td>2011 REE Pricing</td>
<td></td>
</tr>
</tbody>
</table>

*Sc₂O₃ market demand expected to reach 25,000 kg by 2023

Non-REE products significantly offset effects of REYSc commodity price fluctuations

Pricing of non-REESc products varies with general economic conditions
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.
  - Conceptual designs of 1200 tpd commercial plant developed
  - Techno-Economic Analysis (TEA) being developed

- Commercial scale REY and CM production from coal ash possible in 2024-2025 timeframe.

Project will result in a technology that will domestic supply of REY-rich concentrate for downstream separation and refinement into individual REE, as well as critical materials (Sc, Al)
PSI/WWS retained the services of Hatch LTD, Ontario, Canada, to develop a higher fidelity conceptual design of a 1200 tpd ash processing plant:
- CAPEX/OPEX estimates included in conceptual design
- Hatch has extensive experience in designing and building large scale commercial chemical plants worldwide

Plant located at KY ash landfill site: ~ 300 m x 300 m footprint, includes physical and chemical processing plants as well as ash feedstock delivery, preparation, and staging operations.

Conceptual Design by Hatch incorporates robust equipment, and assumes a blank site.
Emerging Applications for REEs Projected to Boost Demand and Increase Prices

- Emerging markets for REEs include wind turbines, electrified vehicles and appliances
- Use of an REE permanent magnet motor can reduce overall vehicle cost by $3,800 USD/vehicle

Source: Lynas Investor Day Presentation, 2019
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) fully operational
  - Optimized processes validated in micropilot plant operations
  - REY concentrate as main product
  - CM recovery (Sc, Al)
  - Beneficiated ash as valuable by-product
Thank You!