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

Physical Sciences Inc., Andover, MA, Equinox Chemicals LLC, Albany, GA, and Center for Applied Energy Research, Lexington, KY

2016 Crosscutting Research and Rare Earth Elements Portfolios Review

Pittsburgh, PA 19 April 2016

Project Objective

- Overall (Phases I and II): Develop and Demonstrate a Pilot Scale Plant to Economically Produce from Coal Ash High-yield REE Concentrates and Commercially Viable Co-products using Environmentally Safe Physical / **Chemical Enrichment Processes**
- Phase 1 performance metrics

Performance Parameter	Threshold Value*	Objective Value
Feedstock REE (REE + Yttrium) Content	300 ppm	> 500 ppm
Total REE (REE + Yttrium) Enrichment in Final Concentrate	~ 5 wt% REE (Elemental)	> 10 wt% REE (Elemental)

Project Schedule .0 Program Management & Planning 3.5 Plant Engineering, Modeling & Techno-Econ Analysis PSI = Physical Sciences Inc. Andover, MA UK/CAER = University of Kentucky, Lexington, KY / Center for Applied Energy Research ECL = Equinox Chemicals LLC, Albany, GA

Sampling and Characterization of Feedstocks - I Physical Sciences Inc. Objective: To characterize the selected ash feedstock materials via photomicrography and via optical and spectrometric techniques in order to guide the fractionation and selection of the materials for physical and chemical separation processes

 Specific feedstock source requirements for this program are: 1. Availability of high REE ash (>300 PPM) sources for pilot plant demonstration

Task 2.0

- 2. Availability of high REE ash (>300 PPM) sources for commercial scale plant
- Strategies for extension to ash sources nationwide will be developed
- Ash morphologies characterized via optical techniques:
- Optical Microscopy, XRD, SEM-EDS, SHIMP, HR-TEM, and SEM-FIB
- Data used to identify REE distribution within ash particles
 - · Identify chemical process operations needed for REE extraction

Task 4.0 System Design - I

- Objective: Develop plant design concepts
- Pilot plant design for further development in Phase II
- Physical and Chemical Separation Plants
- Implement techno-economic model from Task 3.0
- Physical Separation Plant Design: Select appropriate processing strategy for selected substrates
- Identify size and capacity of each unit process
- Select most appropriate classification approach
- Identify size and capacity of each unit process
- Implement flowsheet from Task 3.0 into techno-economic model
- Design physical processing plant to meet Phase 2 objectives

Proposed Technology Combustion Feed Physical • Products and Co-products: REE-enriched dry nitrates mixture (concentrate) - Beneficiated ash, Carbon, Magnetic ash

Phase I Milestones

Based on start date of 1 March 2016

Milestone	Program	Planned Completion
willestone	Month	Date
Kickoff Meeting @ DOE/NETL	1	10 March 2016
Sampling and Characterization Plan	1	29 March 2016
Technical Interchange Mtg. (TIM) #1 @UK/CAER	3	31 May 2016
Interim Review # 1 @PSI	6	31 August 2016
TIM #2 @ ECL	9	30 November 2016
Phase 1 Feasibility Study Results	11	31 January 2016
GO/NOGO	12	28 February 2017
Interim Review #2 @ DOE/NETL	12	28 February 2017
Phase 1 Design Package	14	31 May 2017
Phase 1 Summary Report	14	31 May 2017
Phase 2 Proposal	14	31 May 2017
TIM#3 Teleconference	15	31 May 2017
Phase 1 Final Briefing @DOE/NETL	18	31 August 2017

Task 2.0

TOT Sampling and Characterization of Feedstocks - II

- All studies, with the exception of SHRIMP, are underway or will be started soon, on UKY/CAER/KGS campuses
- SHRIMP will be conducted in May 2016 at USGS facility on Stanford campus (subcontract from CAER to USGS)

Technique	Scale	Advantages	Limitations
Optical microscopy	Lower limit of a few Microns	Micron-scale and large descriptions	Not chemically based
X-ray diffraction	Single to few percent	Mineral determination	Very low-% minerals can be lost in background
ICP techniques	Whole sample	Chemical analysis of whole samples	Bulk analysis
SEM-EDS	Sub-micron	Chemical analysis of specific areas	Limited use for low concentration trace elements
SHRIMP	Few micron	High precision chemical analysis; milling allows measurements in 3-D	Area measured may be larger than minerals
HR-TEM	Few nanometers	Chemical analysis nano-scale areas	Surface or thin sample technique; limitations with larger particles
SEM/FIB	Nanometer	High precision chemical analysis; milling allows measurements in 3-D	Very small area may limit precision

Task 4.0 System Design - II

- Chemical Separation Plant Design:
- Integrate physical separation plant design with chemical separation plant design below
- Develop PFD and preliminary PID for plot plant
- Determine sizing and processing capacity of equipment for specific unit operations
- Specify equipment for unit operations
- Determine power and utility requirements
- Prepare preliminary layout drawings
- Prepare cost estimates for Phase II pilot plant

Project Team & Budget

- Physical Sciences Inc. Develop/Optimize Chemical Separation Processes, Recycling and Waste Handling Processes, and Demonstrate REE Extraction/Re-extraction **Processes under Continuous Flow**
- Center for Applied Energy Research Ash Selection, Collection, & Characterization, Physical Separation Process and Plant Design
- Equinox Chemicals LLC. Techno-economic modeling, pilot plant design of the complete process including physical and chemical separation processes
- Budget: \$1250 K, inclusive of \$250K Cost Share

Task 1.0 Project Management and Planning

- Project (both phases) will be managed by PSI
- Responsible for the overall technical, financial, & schedule performance
- Work performed by the three organizations PSI, ECL, and CAER
- PSI will create a Project Management Plan
- Updated quarterly based on technical results, specific problems encountered, financial status, and per redirection of technical goals and
- PSI will be responsible for all formal and informal communications on this project
- With DOE/NETL as well as PSI's collaborating team members
- Formal reports as called out in the contract
- Reports/Presentations on technical and financial progress/status per contract - Informal communications via e-mail, phone, and fax
- Keep DOE/NETL apprised of new developments, technical problems, or contractual issues in a timely manner

Task 3.0 Feasibility Study

- Objective: Investigate
- Regional and national feedstocks for their REE contents quantities for eventual pilot/commercial recovery of REEs,
- 2. Waste management characterization and proposed processes to minimize or reduce environmental impacts
- 3. Physical Processing of Feedstock
- 4. Chemical Processing of Feedstock
- 5. Reagent Recycling and Waste Disposal
- 6. Continuous Extraction Process Demonstration on lab scale
- 7. Process Modeling and Techno-Economic Analysis of the above
- a. Mass/water/energy balances
- b. Capital, operating, and maintenance costs per unit of input and output

Task 5.0 Technology Development Support

- Objective: Conduct experiments and analyses to support Tasks 2.0, 3.0, and 4.0 to reduce technical and program risks.
- This is a team cost share task
- Support to include:
- Ash sampling and characterization
- Physical separation
- Chemical separation
- Bench scale REE recovery experiments
- Continuous flow laboratory testing
- Specialized equipment and analytical testing
- Techno-economic Modeling
- Pilot plant design