

***High Yield, Economical and Environmentally Benign Production of  
Rare Earth Elements from Coal Ash***

**Phase II Final Summary Report**

DoE Federal Grant No.: DE-FE0027167

Sponsoring Program Office: DoE – NETL

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Revised June 24, 2022

**Acknowledgement:** This material is based upon work supported by the U.S. Department of Energy under Award DE-FE0027167.

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## Abstract

Fly ash stored in landfills and ponds across the United States is an attractive, abundant domestic resource for the cost-effective recovery of rare earth elements (REE) and other critical minerals (CM). Physical Sciences Inc. (PSI) and its team members, Winner Water Services (WWS) and University of Kentucky/Center for Applied Energy Research (UK/CAER) successfully executed a multiphase program that developed technologies and their implementation in a pilot plant. We demonstrated plant operations for cost-effective and environmentally-friendly production of rare earth element oxide (REO) concentrates, and the critical minerals scandium and aluminum (in the forms of salts or oxide products), from coal ash. We also constructed and demonstrated a research-scale (0.5 kg/day) micropilot facility to validate the key physical and chemical processing operations, predict yields, and troubleshoot process bottlenecks. The project team then designed, constructed and operated two decoupled pilot plants: (1) an operational pilot plant for physical separation processes with capacity of 0.4 metric tons per day (tpd), where we optimized processes to produce selected ash fractions as the feedstock for chemical processing and as valuable byproducts such as cenospheres, magnetic ash, and secondary fuel carbon, and (2) an operational pilot plant for chemical ash processing with a capacity of 0.5 tpd that developed optimized processes for the production of: (a) REO concentrates, (b) critical minerals (Sc, Al), and (c) beneficiated ash as a valuable byproduct suitable for cement applications.

In Phase I, the project team (with Equinox Chemicals in place of WWS) developed and demonstrated the feasibility of the physical and chemical separation processes, developed the design of a pilot plant, and began the development of a preliminary techno-economic model.

In the baseline (initial) Phase II program, the project team developed and demonstrated the above pilot scale plant, producing salable REE concentrates, including Y and Sc (REYSc), plus commercially viable byproducts, using environmentally safe and high-yield physical and chemical enrichment processes. The team successfully demonstrated chemical pilot design, construction, shakedown, and operations of the plant. We produced the Phase II deliverable REYSc concentrate ((50 g of >60 wt.% purity REYSc salts on elemental basis), generated the feed for the Phase II follow-on program, identified processing challenges for future optimizations, and refined the techno-economic model.

In the Phase II follow-on program, the project team: (1) developed and demonstrated processes to increase the REE amount by 3X (content basis) and convert the Phase II REE salt mixture to an oxide mixture, (2) produced/delivered >38 g of REO mixture with >85 wt.% purity (elemental basis); (3) developed processes to recover critical minerals scandium and aluminum from intermediate streams; (4) produced/delivered > 1 g of scandium salt mixture with >85 wt. % purity (elemental basis); (5) produced/delivered > 100 g of aluminum oxide type material with >70% wt. purity (elemental basis); and (6) updated the techno-economic model from the baseline Phase II program to assess CAPEX and OPEX of a commercial operation.

This program has developed extensive databases on process chemistry, unit operations, plant engineering, and techno-economics that will enable further scale-up toward commercial plant design. Specific future developments will be focused on achieving dramatic savings in energy, reagent usage, and operating costs. The combined results will contribute significantly for maturing the technologies of REE recovery from coal byproducts and promote the establishment of domestic REE and CM supply chains.

## 1. Executive Summary

This report summarizes the results of work performed by the Physical Sciences Inc. (PSI) team (collaborators outlined for each Phase below) during the multiphase program DE FE0027167. The work performed on the program and the corresponding results/achievements include:

- Phase I (Team: Physical Sciences Inc. (PSI), University of Kentucky/Center for Applied Energy Research (UK/CAER), and Equinox Chemicals, LLC (ECL))
  - Developed and demonstrated the feasibility for physical and chemical separation processes.
  - Developed a preliminary design of pilot plant built and operated in Phase II.
- Phase II (Team: Physical Sciences Inc. (PSI), University of Kentucky/Center for Applied Energy Research (UK/CAER), and Winner Water Services, LLC (WWS))
  - Completed detailed design, developed and demonstrated a pilot scale plant to produce salable REE concentrates, including Y and Sc (REYSc), and commercially viable co-products, using environmentally safe, and high-yield physical and chemical enrichment processes.
  - Demonstrated chemical pilot design, construction, and shakedown
  - Produced the Phase II deliverable REYSc concentrate ((50 g of >60 wt.% purity REYSc salts).
  - Generated the LLX feed for the Phase II follow-on program.
  - Identified processing challenges for future optimization and refined the techno-economic model.
- Phase II follow-on (Team: Physical Sciences Inc. (PSI) and Winner Water Services, LLC (WWS))
  - Developed and demonstrated processes to increase the REE production by 3X and convert the Phase II REE salt mixture to REO mixture.
  - Produced/delivered >38 g of REOs with >85% purity (elemental basis).
  - Developed processes to recover critical minerals scandium and aluminum from intermediate streams.
  - Produced/delivered > 1 g of scandium salt mixture with >85 wt.% purity (elemental basis).
  - Produced/delivered > 100 g of aluminum oxide type material with >70 wt.% purity (elemental basis).
  - Updated the techno-economic model from baseline Phase II program to assess CAPEX and OPEX of a commercial operation.

This program also developed extensive knowledge on process chemistry, unit operations, plant engineering, and techno-economics that will enable further scale-up and commercial plant design. Specific developments will contribute to significant savings in energy, reagent usage, and operating costs. The combined results will contribute significantly for maturing the technologies of REE recovery from coal ash.

**Table 1** summarizes the key performance/process parameters we achieved. Details are provided below in the sections of individual phases of the program and in the supporting information section.

**Table 1.** Key Findings and Process Parameters

Key Parameter(s)	Selected Value(s)
<ul style="list-style-type: none"> <li>• Weight% (wt%) of REY in deliverable concentrate</li> </ul>	40-60 wt. % Elemental basis (Phase II) 85-90% wt. Elemental basis (Phase II add-on)
<ul style="list-style-type: none"> <li>• Products from PSI's process: coarse non-magnetic ash used as geopolymer, carbon, magnetite substitute, aqueous REYSC solution, Product 1 (REY-rich Dry Mixture), Product 2 (Sc-rich Dry Mixture)</li> </ul>	<p>Pilot scale process produced all the anticipated products and byproducts at high yields and relative content. Selected products were analyzed as outlined in various sections of this report.</p> <p>Anticipated uses of byproducts: coarse non-magnetic ash usable as geopolymer, carbon-rich fraction usable as secondary fuel and as inexpensive feed for higher value carbon forms, magnetic magnetite substitute usable in heavy media separation, product 1 usable for REY separation and refining, Product 2 usable for Sc separation and refining, beneficiated ash after digestion usable as cement substitute.</p>
<ul style="list-style-type: none"> <li>• Plant capacity (1-5 tons (0.5-2.5 cubic meters) of ash processed per day)</li> </ul>	0.4 tons-per-day – physical processing 0.5 tons-per-day – chemical processing
<ul style="list-style-type: none"> <li>• Mesh size (&lt;200 mesh), type of laboratory analyses (EDS, HRTEM), and analytical results (counts as a function of voltage), and images</li> </ul>	The coal ash fraction with a particle size of <200 mesh, non-magnetic, with carbon removed was selected for the chemical processing. Various analyses (ICP, HRTEM, microscopic imaging) indicated high REE concentrations (>500 ppm).
<ul style="list-style-type: none"> <li>• Site-specific sources of ash feedstock, type of laboratory analysis (ICP-MS), REYSc concentration, tons of ash reserve, tons of REYSc reserve, life of reserve expressed as % of U.S. annual demand for “x” years</li> </ul>	Ash from two sites from Eastern Kentucky (Phase II Dale ash and an additional feedstock) were used for the pilot runs. Table with ash locations was provided at the end of Phase I
<ul style="list-style-type: none"> <li>• Concentration of individual REEs, ppm on a whole sample basis, in selected ash fraction</li> </ul>	A typical analysis indicated good content of individual REYSc, with significant content of Nd (~170 ppm), Y (~70 ppm), and Sc (~40 ppm), and reasonable (>~10 ppm) content of Pr, Gd, Dy, Er, Tb.
<ul style="list-style-type: none"> <li>• REY yield (%), REY wt% concentration, Sc yield (%), Sc wt% concentration</li> </ul>	REY and Sc yields of 10-30 wt.% were recorded in micropilot and chemical pilot operations. The concentrations of the samples at the conclusion of the program were >85% wt. for both the REY and Sc products.

Key Parameter(s)	Selected Value(s)
<ul style="list-style-type: none"> <li>REY and Sc relative content (enrichment wt%) for REY, LREE, and HREE+Y through progressive processing steps, including Post Process Step 1 and Post Process Step 2</li> </ul>	Phase I indicated significant enrichment to generate products with relative REYSc content as high as 30 wt.%. Further developments in Phase II and Phase II add-on showed the capability for further enrichment to generate products with the following concentration on elemental basis: REY (>85%); Sc (>85%); Al (>68%)
<ul style="list-style-type: none"> <li>Ton per day capacity, products, and annual production for individual REEs, carbon, magnetic, non-mag &gt; 200 mesh, non-mag &lt; 200 mesh) in a commercial scale REE processing plant, and number of years to provide Return on Investment; degree of recycling/reuse of reagents required</li> </ul>	A capacity of 1200 tons-per-day physical processing with a corresponding of ~600 tons-per-day chemical processing is the commercial baseline. Number of years for the return of investment will depend on selected plant capacity, recyclability/reuse of reagents (>90% threshold/>95% objective), make-vs-buy/import decisions on highest-use reagents, byproduct market volumes and market penetration including effects of prevailing economic conditions, economics of critical minerals production (e.g., aluminum), REYSc pricing environment including international aspects, public infrastructure contributions from the government for REYSc as critical/strategic materials, structure and terms of public/private partnership, and government subsidies and price supports.

## 2. Summary of Phase I Work

In Phase I, the project team developed and demonstrated feasibility of the physical and chemical separation processes, developed a preliminary design of a pilot plant to be built and operated in Phase II and began the development of a preliminary techno-economic model. Key findings of this program are summarized below. Details of the performed work are provided in Appendix A (Phase I Summary Report).

### 2.1 Program Goals and Achievements

The broad objective of the Phase I program was to demonstrate the feasibility of developing a pilot scale plant to economically produce high-yield rare earth element concentrates and commercially viable co-products from coal ash using environmentally safe physical and chemical enrichment processes. The project specific metrics and the achieved the values presented in **Table 2**. All major requirements of the proposed Phase I threshold/objective goals were met or exceeded.

**Table 2.** Phase 1 Performance Metrics and Results Achieved

Performance Parameter	Threshold Value	Objective Value	Phase I Results
Feedstock REE (REE+Yttrium) Content	300 ppm	> 500 ppm	> 500 ppm
Total REE (REE+Yttrium) Relative Content in Final Concentrate	~ 5 wt% REE (Elemental)	> 10 wt% REE (Elemental)	30 wt% (Elemental)

## 2.2 Key Results

### 2.2.1 Ash Feedstock Characterization and Selection

The project team considered and screened/landfilled ash at several power plants, **Table 3**. The table shows our measured values of REE on ash basis via the ASTM D6357-11-1 standard. Based on the REE content, size of reserves, and logistics considerations, the Dale ash (#6) was selected as the feedstock for the physical/chemical separation processes for the initial Phase II:

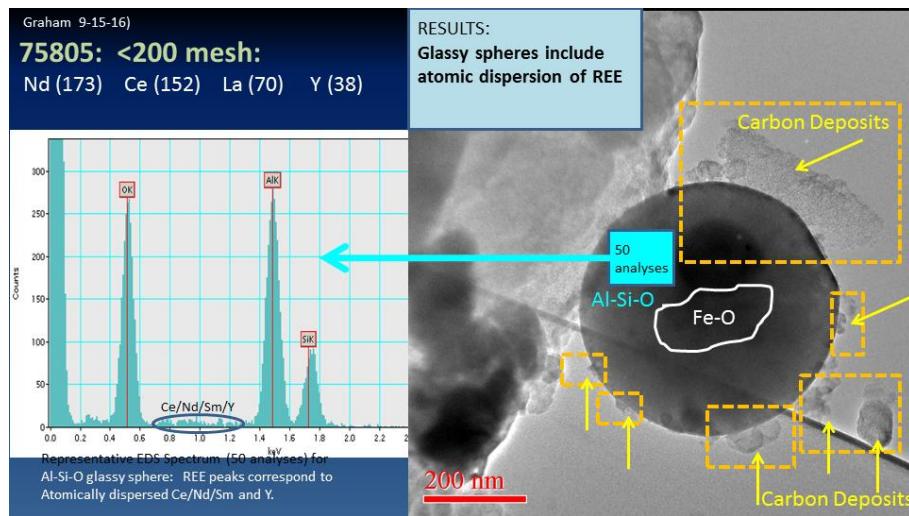
**Table 3.** Phase I Coal Ash Samples

#	Sample Ash Description	Location	Total REE +Y (ppm)
1	Coal Ash from Cooper Power Station	Burnside, KY (Pulaski County)	794
2	Coal Ash from Williams Plant	Goose Creek, SC (Berkeley County)	669
3	Coal Ash from KSU Stoker Plant	Kentucky State U. (Franklin County)	1113
4	Coal Ash from EW Brown Plant	Burgin, KY (Mercer County)	622
5	Coal Ash from Northampton Power Plant	Northampton, PA (Northampton County)	492
6	Coal Ash from Dale Power Station	Ford, KY (Clark County)	528

Approximately 20 samples were collected from several locations (ash pond shown in **Figure 1**) and a composite sample was created for analysis and experimental work.

**Figure 1.** Ash pond overview.

A preliminary assessment of the REE content in a Kentucky power plant ash fraction was performed by High Resolution Transmission Electron Microscopy (HRTEM) analysis (**Figure 2**). The results indicate high Nd content and provided confirmation that REEs are tied up with glassy aluminosilicate matrix.



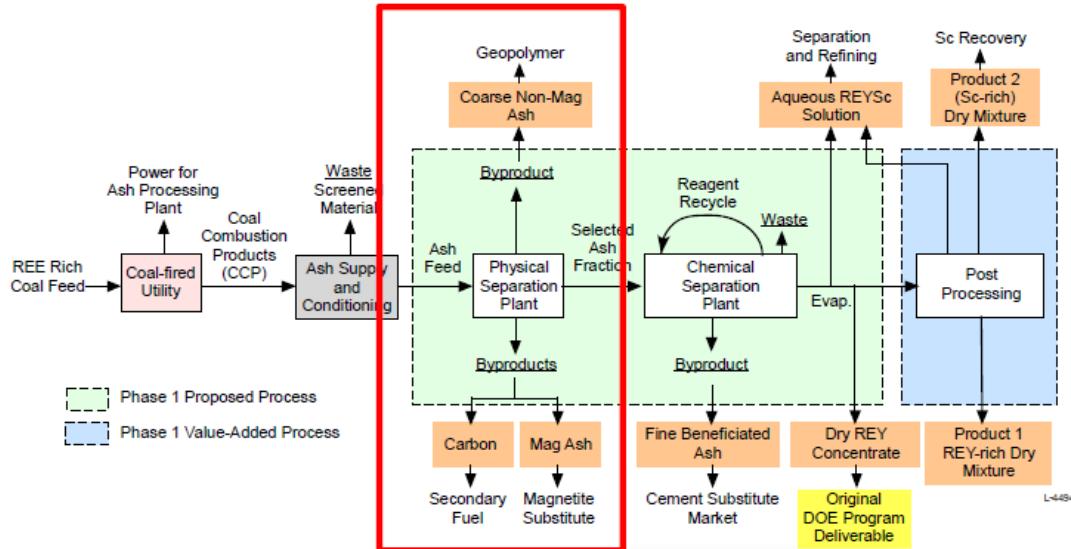
**Figure 2.** HRTEM analysis.

The ash selection and characterization findings in Phase I are summarized below:

- Ash source analyses and selection completed
  - Analyzed four PCC power plant ash sources that use primarily Eastern KY coal & one FBC power plant ash source that uses anthracite/culm from Central PA
    - Cooper, Williams, Brown, and Dale (PCC), plus Northampton (FBC)
  - All ash sources analyzed via ICP-MS; REYSc content > 500 ppm
- Ash from Dale power station, Ford, KY was selected as ash source for plant demonstration on Phase 2 of this program
  - REYSc = 556 ppm, exceeding 500 ppm objective requirement
  - ~ 2Mt of ash reserve; ~1200 tons REYSc reserve (~10% of US annual demand)
  - Adequate for 1200 tpd commercial scale plant (~1.5% annual demand for ~ 7 y)
  - A vast reserve for an envisioned 1-5 tpd pilot plant
- Feed ash, physically separated ash, and chemically pre-treated ash characterized via various optical/other techniques
  - REE concentrates present in glassy ash particles in feed ash
  - REE also present in carbon deposits around glassy particles

## 2.2.2 Physical Separation Process

The physical separation (**Figure 3**) entailed standard mineral processing methods that were applied to coal ash to generate a selected feedstock for the subsequent chemical separations.

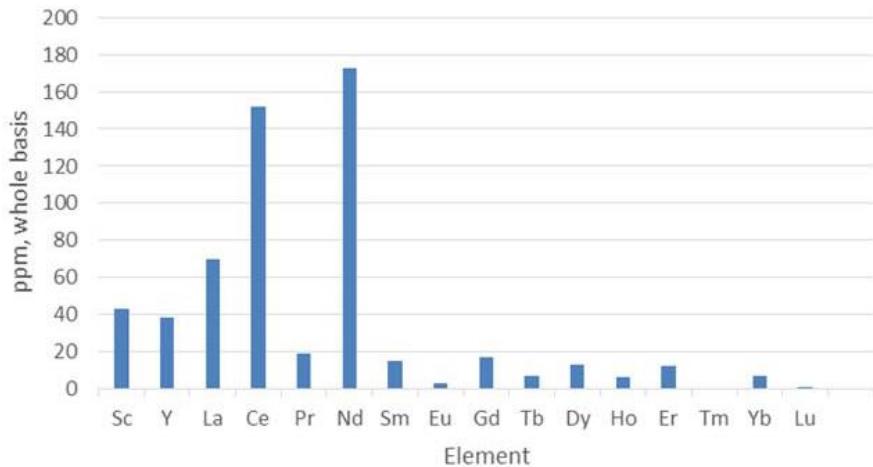


**Figure 3.** Physical separation step outlined in the context of the overall process.

Key Phase I findings of the physical separation are summarized below:

- Each of the five ash sources were separated into five fractions
  - +60 mesh ( $>250 \mu\text{m}$ ): Oversize, discarded
  - Froth product (principally C): Use as supplementary fuel, higher C products
  - Magnetic: Use as cheap magnetic substitute
  - Non-magnetic:
    - +200 mesh ( $>75 \mu\text{m}$ ): Use as conventional cement substitute
    - -200 mesh ( $<75 \mu\text{m}$ ): High REY content fraction for chemical processing
- Developed flowsheets for physical separation process
- Developed a spreadsheet economic model of the process
- Integrated physical processing with the chemical process model, and used for techno - economic assessment

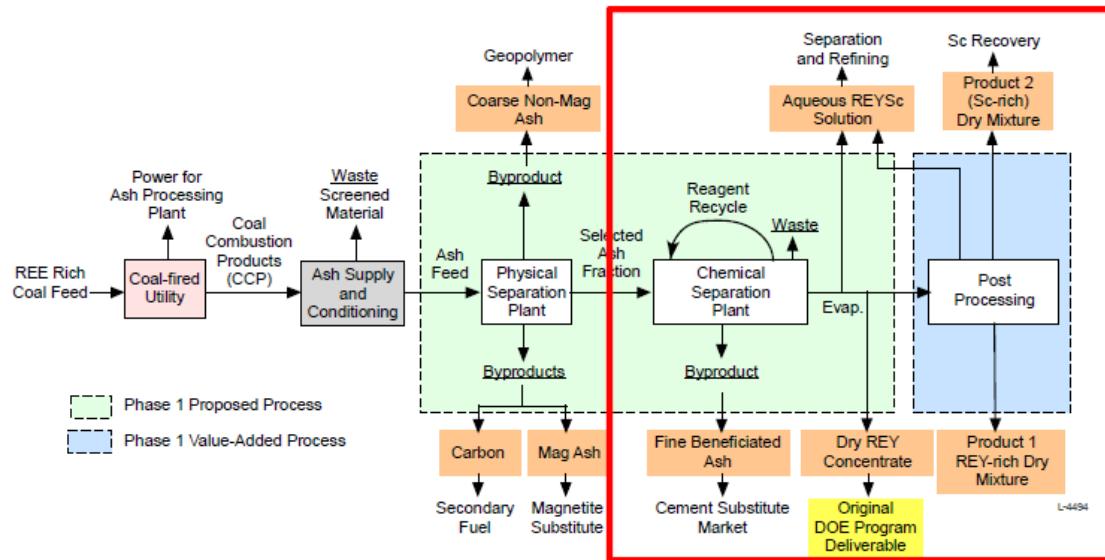
Further analysis of the selected fraction indicated good content of individual REYSc, with significant content of Nd (~170 ppm), Y (~70 ppm), and Sc (~40 ppm), and reasonable ( $>10 \text{ ppm}$ ) content of Pr, Gd, Dy, Er, Tb (Figure 4).



**Figure 4.** Individual elemental REYSc content for the selected ash fraction.

### 2.2.3 Chemical Separation Process

The chemical process (**Figure 5**) was optimized and applied to the selected ash fraction generated by the physical processing steps discussed above:



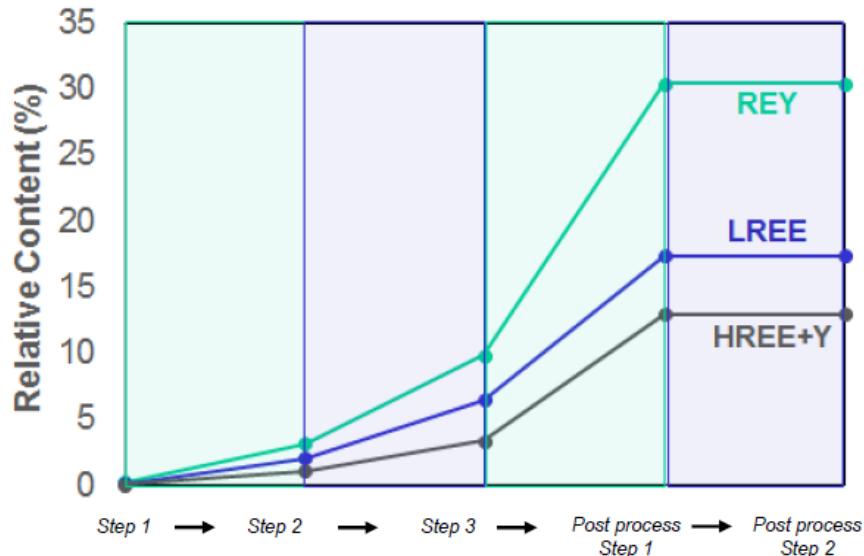
**Figure 5.** Chemical separation step outlined in the context of the overall process.

Key Phase I findings of the chemical separation are summarized below:

- Determined optimal parameters for each process step to recover REE from selected ash at high yield consistent with high enrichment
- Developed post-processing steps that yield two higher value product streams:
  - REY-rich: REY Yield > 18%, Conc > 30 wt%; Sc Yield ~ 18%, Conc ~ 0.5 wt%
    - REE conc >> objective target (10 wt%)
  - Sc-rich: Sc Yield ~ 18%, Conc ~ 1.5 wt%; REE Yield > 4%, Conc ~ 6 wt%
    - REE conc >> threshold target (5 wt%)
- Reduced process time for individual steps and avoided elevated temperatures

- Experimental development of process scale up
- Process implementable on commercial scale; requires different optimization parameters for project economics
  - Tradeoff some of the high REYSc yield/enrichment to achieve better economics

The postprocessing steps were demonstrated to provide additional enrichment (**Figure 6**). For instance, post-processing step 1 led to 3X increase in REY enrichment and efficient separation from other contaminants to generate a product well-suited for additional REY separation.



**Figure 6.** Analysis of Phase I postprocessing steps.

### 3. Summary of Phase II Work

In the baseline Phase II, the project team developed and demonstrated a pilot scale plant to produce salable REE concentrates, including Y and Sc (REYSc), and commercially viable co-products, using environmentally safe, and high-yield physical and chemical enrichment processes. The project team successfully demonstrated a chemical pilot design, construction, and shakedown in two stages, produced the Phase II deliverable REYSc concentrate ((50 g of >60 wt.% purity REYSc salts), generated the feed for the Phase II follow-on program, identified processing challenges for future optimization, and refined the techno-economic model. Key findings of this program period of performance are summarized below. Details of the performed work are provided in the supporting info section.

#### 3.1 Program Goals and Achievements

The broad objective of the initial Phase II was to demonstrate REYSc separation/enrichment technology in pilot 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). The project specific metrics and the achieved values are presented in **Table 4**. All major requirements of the proposed Phase II threshold/objective goals were met or exceeded.

**Table 4.** Phase II Project Metrics and Achieved Values

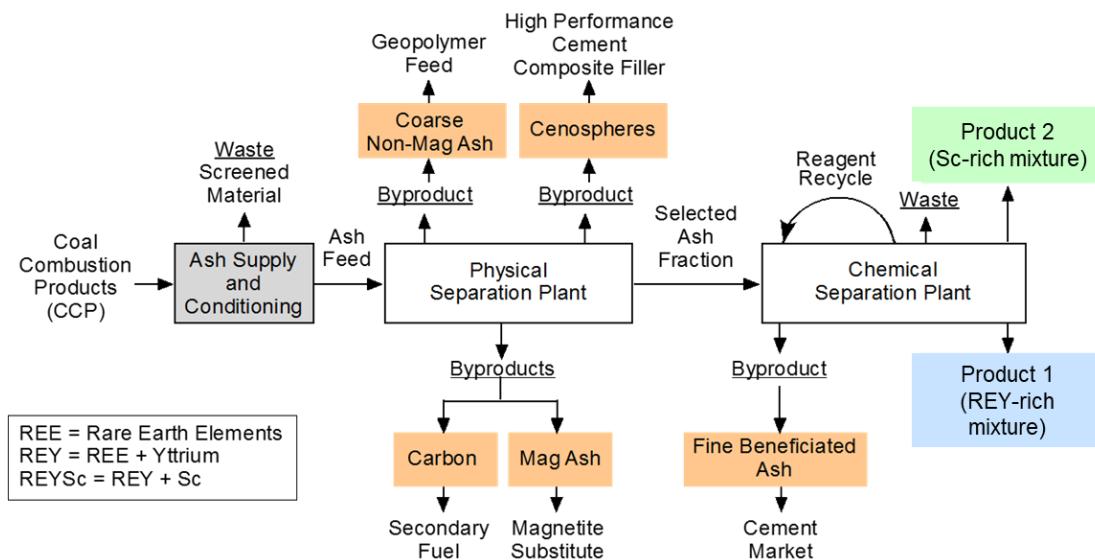
Performance Parameter	Threshold Value	Objective Value	Achieved Value
Feedstock REYSc Content	>300 ppm (Whole Mass)	>500 ppm (Whole Mass Basis)	475 – 515 ppm
Total REYSc Enrichment in Final Concentrate	>10 wt% (Elemental Basis)	>20 wt.% (Elemental Basis)	56 wt.% (Elemental Basis)
Return on Investment	<12 yr	<10 yr	Carryover to Phase II Follow-on program
Delivered Concentrate Quantity	~50 g	~0.5 kg	51 g

### 3.2 Key Results

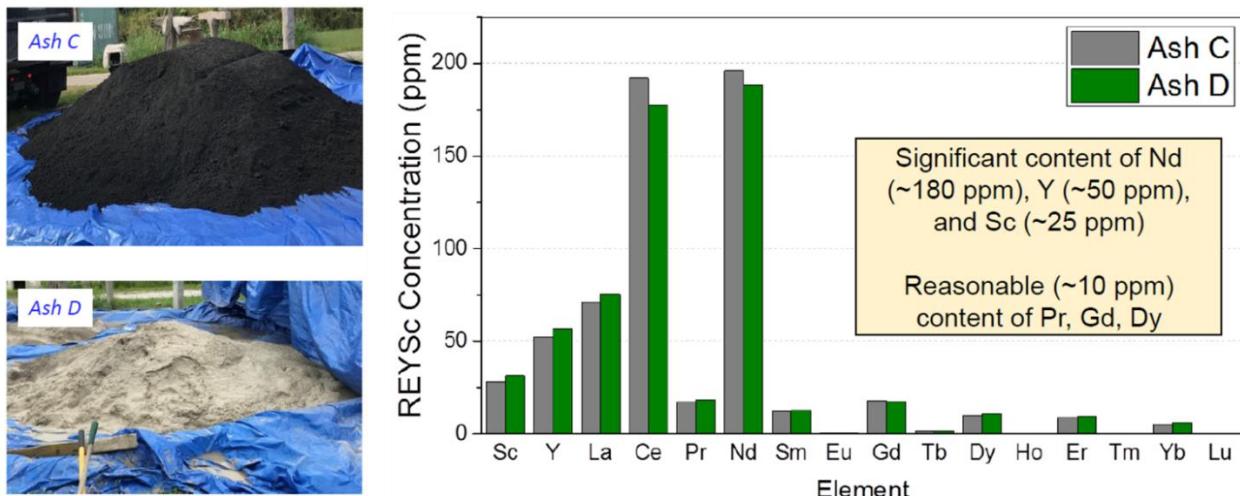
#### 3.2.1 Pilot Plants and Unit Operations

construct and operate pilot plants for the physical and chemical separation steps. The integrated process comprised a physical separation stage, followed by a chemical separation stage, followed by a post-processing stage.

The primary products were REY-rich and Sc-rich concentrates. The key byproducts included beneficiated ash cement substitute, a magnetic ash product, cenospheres and a carbon product used as a secondary fuel. The Phase II process is shown in **Figure 7**.

**Figure 7.** Phase II Process for the REY and Sc recovery from coal ash

Ash from two eastern KY coal fire power plants was recovered and used as process feedstocks. Analysis of both ash sources (Ash C and D in **Figure 8**) indicated significant content of Nd (~180 ppm), Y (~50 ppm), and Sc (~25 ppm) as well as reasonable (~10 ppm) content of Pr, Gd, Dy elements.



**Figure 8.** Ash feedstocks used for the Phase II program.

The physical separation plant at the UK/CAER location was built and operated at a capacity of 0.4 tpd. Approximately 20 tons of coal ash were processed with > 50% yield to produce ~ 10 tons of the fraction suitable for chemical processing (carbon-removed, non-magnetic ash with particle size < 75 microns). An overview of the physical separation plant showing the equipment for various unit operations is presented in **Figure 9**.



**Figure 9.** Physical Separation Pilot Plant (0.4 tpd capacity).

Physical processing created an ash fraction that is a suitable feed to chemical separation. Key properties are: (1) low carbon content; (2) low magnetics content and (3) small particle size. Processed ash collected in super sacks, shipped to and processed in the chemical pilot. **Figure 10** shows a comparative photographic analysis of the physical and chemical processing feedstocks. Note that the color changes from dark black to brown, indicating removal of carbon.

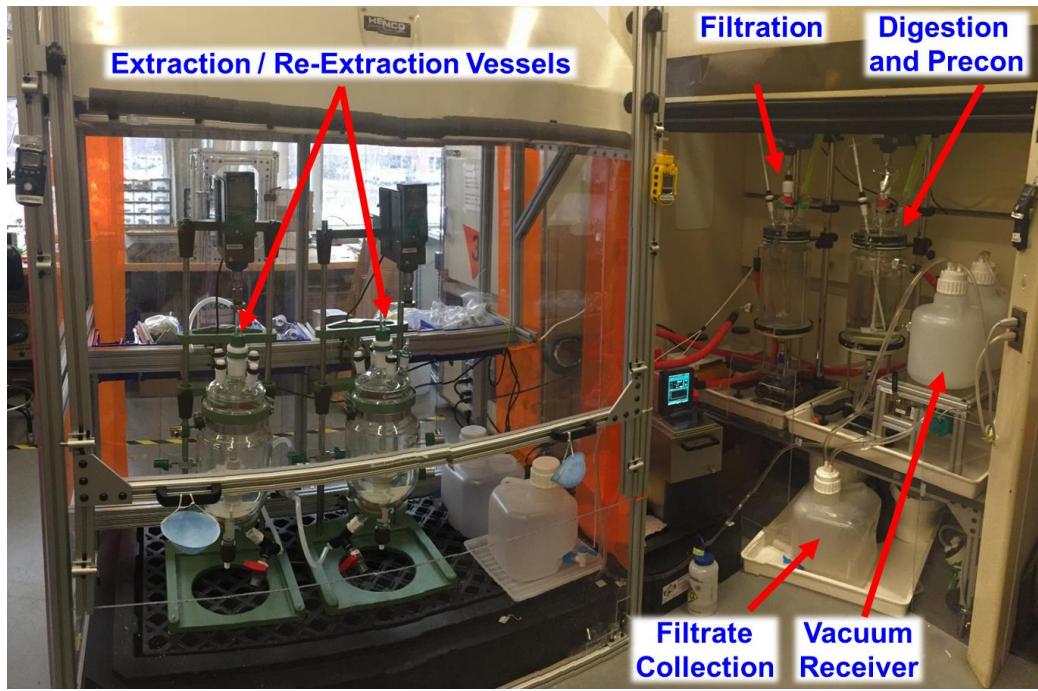
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in super sacks, shipped to and processed in the chemical pilot. **Figure 10** shows a comparative photographic analysis of the physical and chemical processing feedstocks:



**Figure 10.** Photographic analysis of the physical and chemical processing feedstocks.

In order to streamline the chemical pilot plant operations and reduce risk, the project team developed a micropilot plant (0.5 kg/day capacity). The micropilot facility demonstrated target yield and enrichment performance requirements and was successfully used for validating and troubleshooting chemical pilot processes. Key micropilot unit operations include digestion, filtration, pre-concentration, LLX feed preparation, and liquid-liquid extraction and re-extraction. **Figure 11** outlines equipment used for critical unit operations. Approximately 15 kg of coal ash was processed through the PSI micropilot facility.



**Figure 11.** Micropilot Plant (0.5 kg/day capacity).

The project team designed, constructed and operated a chemical processing pilot plant (0.5 tpd capacity) based on the process development from the Phase I and micropilot efforts. The chemical processing pilot is located at the WWS facility in Sharon, PA. This pilot plant (**Figure 12**) processed ~ 8 tons of coal ash and was demonstrated to produce REE concentrates with >15% yield and >50% purity (elemental basis).

The chemical pilot plant contains all of the equipment necessary to extract REE metals from coal ash, and purify the REEs to meet the target concentrations. The pilot plant contained two LLX systems, a bench top system (capacity of ~1 gal/hr), and a commercial system (capacity ~50 gal/hr), denoted LLX1 and LLX2 in the figure, respectively.



**Figure 12.** Pilot Plant for Chemical Processing: plant overview (left); hot side operation- digestion and preconcentration steps (center) and Cold Side Operation – LLX units (right).

### 3.2.2 Qualification of Selected Products and Byproducts

As discussed above, the operation of the Phase II pilot plants resulted in the production of significant amounts of valuable products and byproducts. Selected materials were qualified for their commercial potential using concentration measurements and functional evaluation.

The main product, REY concentrate was analyzed by ICP-OES and ICP-MS and demonstrated for adequate purity for subsequent refining and separation. Multiple ash batches were processed and the materials were sampled at various process stages analyzed via three independent methods:

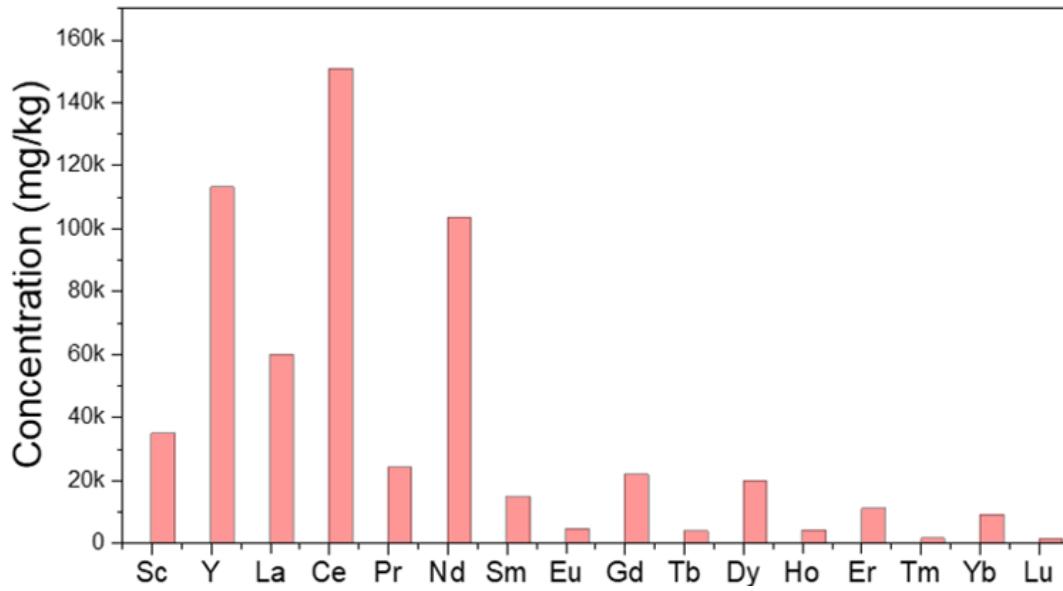
- ICP-MS Analysis at Environmental Service Lab, Pittsburgh, PA (WWS)
- ICP-OES Analysis at University of Massachusetts Lab, Lowell, MA (PSI)
- ICP-MS Analysis at National Analytical Lab, Londonderry, NH (PSI)

The comparative analyses of the final liquid samples produced upon chemical processing of the ash feedstocks are shown in **Table 5** (sample 3 was used to generate the Phase II deliverable sample):

**Table 5.** Analysis of the Final Samples

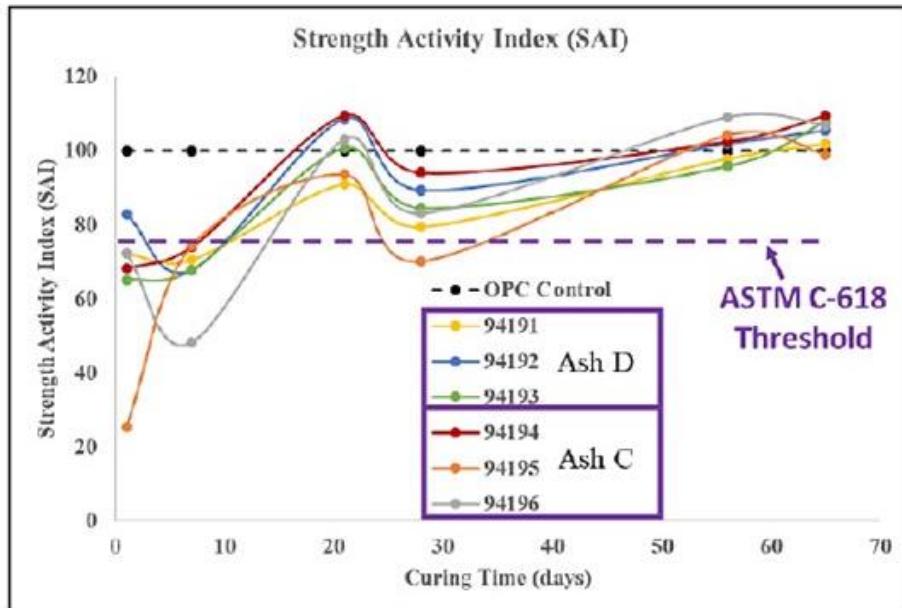
REYSc Rel Content	Sample 1	Sample 2	Sample 3	Sample 4
WWS/ESL ICP-MS	n.d.	10.2%	<b>88.4%</b>	n.d.
PSI/UML ICP-OES	14%	11.9%	<b>66.7%</b>	29.1%
Quantity of REYSc Salt Mixture Produced	597 g	~330 g	<b>66 g</b>	~214 g

The REE product contained significant quantities of the valuable Nd, Y, Sc and HREE content as exemplified in **Figure 13**:



**Figure 13.** REYSc analysis of a product sample.

The ash cake materials resulting from the digestion step were functionally qualified for the use as cement substitutes. ASTM C-618 protocol was used to qualify the samples using as criterion the >75% of strength activity index (SAI) after 28 days metric. As shown in **Figure 14**, five out of six samples (originating from both ash feedstocks) passed the ASTM C-618 requirements. These results indicate that the ash cake is suitable as a cement substitute and the digestion process does not greatly impact cement suitability.



**Figure 14.** ASTM C-618 evaluation of ash cake samples resulted upon digestion runs.

### 3.2.3 Techno-economic Considerations

In Phase II the project team further developed key elements of the techno-economic model. Economics of the physical and chemical processing steps were modeled using Aspen Plus and spreadsheet analyses. The plant size used for the analysis was 1200 tpd for the ash physical processing plant and 600 tpd for chemical processing plant. Key plant attributes are: co-location at ash source to significantly reduce transportation costs, decoupled operations and modular designs for operational flexibility and transportability. Ash fractions shipped to local markets used for analysis were: carbon, magnetic ash, and the >200 mesh non-magnetic ash. **Table 6** outlines the annual production of major REE salts, Sc salt, and byproducts. The analysis indicated that non-REE products significantly offset effects of REYSc commodity price fluctuations (pricing of non REYSc products varies with general economic conditions):

**Table 6.** Analysis - Annual Production of Major REE Salts, Sc Salt, and Byproducts

Component	Quantity Produced <sup>1</sup> tons/year	Portion of Revenue (%)		Worldwide Market tons/year	Market Application
		2018 REE Pricing	2011 REE Pricing		
REEs	38.2	1.0	10.8	170K	Batteries, Magnets, Alloys, Catalysts
Scandium	5.8	26.1	35.0	10-15	Alloys, Catalysts
Carbon	96K	6.2	4.6		Low-grade Fuel
Magnetic	20K	5.4	4.0		Magnetite Substitute
Non-Magnetic >200 Mesh	48K	1.0	0.8		Geopolymer Feed
Non-Magnetic <200 Mesh	186K	23.9	17.8	71.8M	Cement Substitute (Pozzolan)
Cenosphere Product	2K	36.4	27.1	~51K	Concrete Additive

**Table 6** shows the quantity of each product produced per year at a nominal feed rate of 1200 tpd of coal ash. The high quantity products are those directly derived from the ash feed material (carbon, magnetic ash, cenospheres and non-magnetic ash fractions). These materials are important for the economic picture because they represent consistent revenue sources that are independent from REE markets. It's also important to note that these quantities are far from the Worldwide market quantity, in order to avoid market saturation. The table also shows the portion of revenue derived from each of the product streams under 2018 and 2011 REE pricing. At 2018 REE prices, only 27% of the revenue is derived from REYSc products. This is because the price of REEs was relatively low in that year. However, under 2011 REE pricing, nearly 45% of the revenue comes from REYSc products, due to the very high prices of selected elements in that year.

## 4. Summary of Phase II Follow-on Work

In the Phase II follow-on program, the project team: (1) developed and demonstrated processes to increase the REE production by 3X and convert the Phase II REE salt mixture to REO mixture, (2) produced/delivered >38 g of REOs with >85% purity (elemental basis); (3) developed processes to recover critical minerals such as scandium and aluminum from intermediate streams; (4) produced/delivered > 1 g of scandium salt mixture with >85 wt.% purity (elemental basis); (5) produced/delivered > 100 g of aluminum oxide type material with >70 wt.% purity (elemental basis); (6) updated the techno-economic model from initial Phase II program to assess CAPEX and OPEX of a commercial operation.

### 4.1 Program Goals and Achievements

The overall goal of The Phase II follow-on program was to demonstrate the production of REE products with enhanced purity, recovery of critical minerals (CMs) and development of techno-economic analysis for further technology maturation and commercial transition. Specific objectives included:

(1) demonstrate a process to convert the salts mixture with >10-20 wt% REYSc (relative content) from Phase II program to >85 wt.% REY-enriched (relative content) oxides mixture; (2) Produce Sc-enriched

oxide mixture with >85% wt.% REYSc relative content; (3) Increase total quantity of REY in deliverable REY-enriched product by >3X; (4) Develop a process to recover the critical material aluminum from intermediates of the REYSc extraction process developed under Phase II program; (5) demonstrate production of a >50 wt.% (relative content) aluminum oxide product; (6) update the techno-economic model developed under the Phase II program for a commercial scale plant that will provide < 10-12 years return on investment. The key performance parameters are shown in **Table 7** and the target deliverable samples are shown in **Table 8**.

**Table 7.** Key Performance Parameters

Performance Parameter		Threshold Value	Objective Value
<b>Feedstock REY+Sc Content</b>		>300 ppm	>500 ppm
<b>Return on Investment</b>		< 12 years	< 10 years
<b>REY-enriched Product</b>	<b>Quantity (REY salts)</b>	100 g	300 g
	<i>REY-enriched Oxide Purity (total REY content - elemental basis)</i>	>85%	>90%
<b>Sc-enriched Product</b>	<b>Quantity (Salt/Oxide)</b>	1 g	2 g
	<i>Sc-enriched Oxide/Salt Purity (Sc content - elemental basis)</i>	>85%	>90%
<b>Aluminum Product</b>	<b>Quantity (oxide type material)</b>	100 g	300 g
	<i>Purity (Al content elemental basis)</i>	>50%	>68%

**Table 8.** Deliverable Samples

Product	Type	Deliverable Quantity of Product	Relative Content
REY-enriched Product	Salt	100 g	>85%
Sc-enriched Product	Salt	1 g	>85%
Aluminum oxide product	Oxide-type	100g	>50%

**Table 9** and **Figure 15** outline the metrics and the appearance of samples deliverables that were produced/delivered (KPP requirements met or exceeded). The REY delivered samples samples are the oxide equivalent of the originally proposed salt forms. These samples have the same or higher purity as the original deliverables and provided the DOE customer with samples positioned further in the supply chain as the REY refining processes are commercially designed to use oxides as feedstock.

**Table 9.** Phase II Follow-on Sample Deliverables

Item	Threshold	Objective	Delivered
REY Product	>85% Purity 100 g REY Salts	>90% Purity 300 g REY Salts	22 g of >85% REY 16 g of >90% REY Oxides Concentrate
Sc Product	>85% purity, 1 g	>90% purity, 2 g	~1 g at >85% Sc Salt Concentrate
Al Product	>50% Purity, 100 g	>68% Purity, 300g	101 g at >70% Purity Oxide-type Product

**Figure 15.** Sample product deliverables – photographic analysis.

## 4.2 Key Results

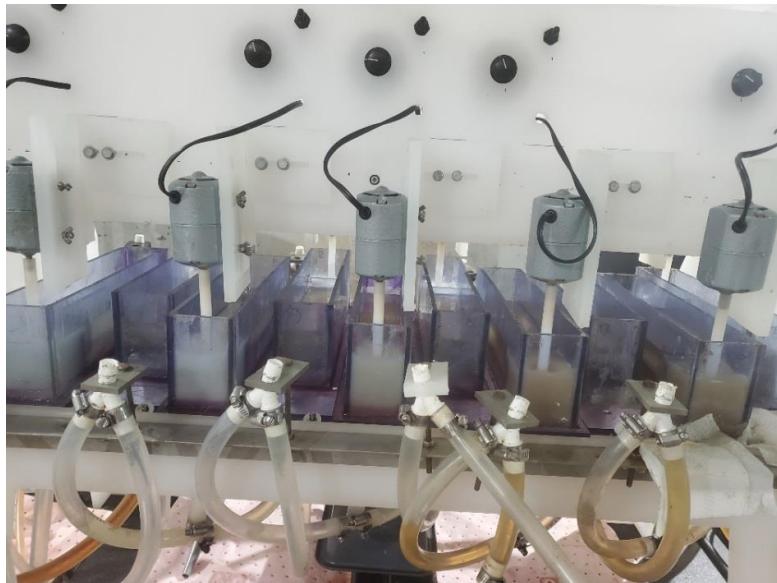
### 4.2.1 Production of REOs: REY-enriched Product

The REY enriched product was generated using a combination of LLX and post-processing steps.

The LLX process was further optimized based on the initial Phase II findings. Multiple runs were performed and key findings are summarized below.

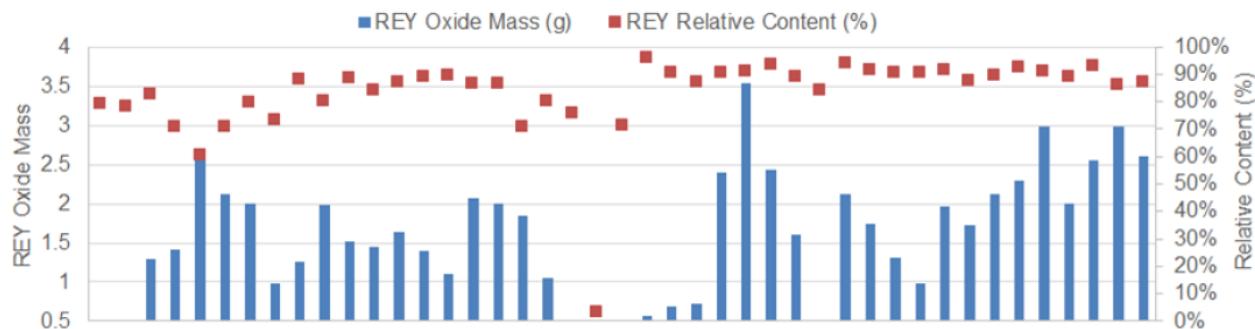
- Use of specific extractants allows selective extraction of rare earth salts
- Two important measures of performance are:
  1. High yield of REE salt from the feed solution
  2. Purity of REE salts evaluated by relative content
    - Selectivity of REE salts over other components
    - Reduces impurities levels
    - Achieved purities (relative content) of 40-60% using LLX feed optimization
    - Post processing steps were developed to increase REY purity from the LLX to >85%

To produce program deliverables the counter current liquid-liquid extraction (CCLLX) circuit (**Figure 16**) was operated in continuous campaigns. These campaigns produced REE solution that were further refined to meet program relative content goals.



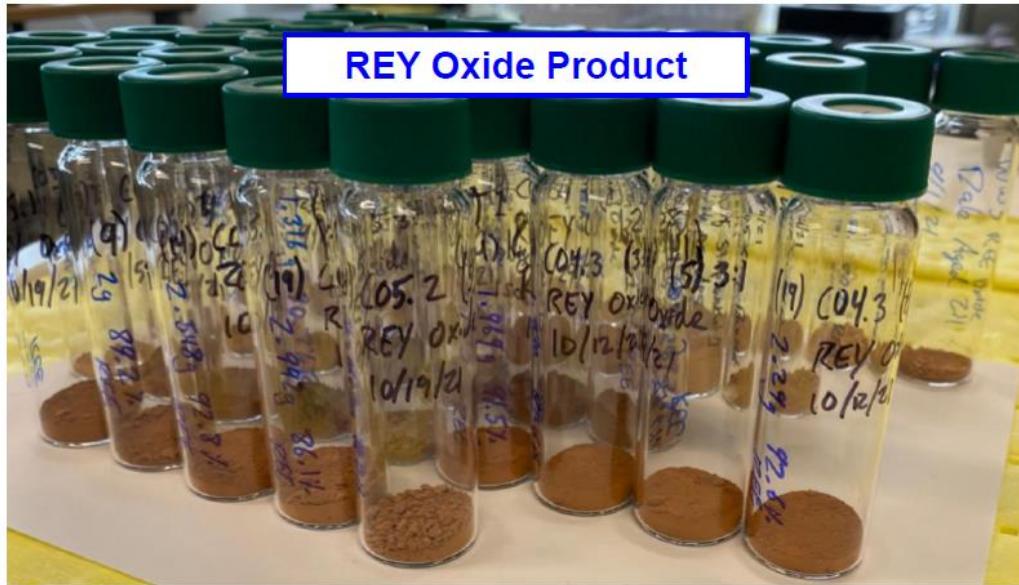
**Figure 16.** CCLLX circuit.

The post-processing steps were developed and PSI and scaled-up in the pilot plant operations by WWS. The REY oxide product was produced in multiple production runs. **Figure 17** outlines the results with the amount of REY oxide produced shown in blue and the corresponding REY relative content shown in red for each REY oxide production run. As shown in **Figure 17**, consistent high product purity was obtained after post processing optimization:



**Figure 17.** Analysis of REY oxide production runs.

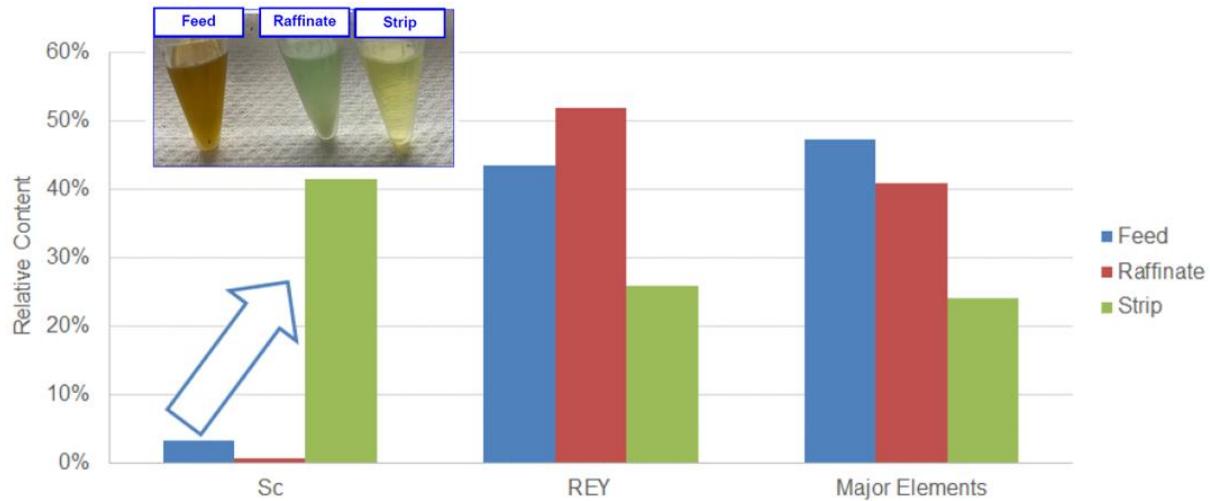
The LLX optimization resulted in an LLX product with an REY purity consistently in the range of 40-60% relative content. The post-processing processing steps were demonstrated to consistently generate REY oxide material (~90% relative content) from the LLX products. The scaled-up post processing steps generated the REY oxide concentrates deliverable materials (22 g of >85% REY content) and 16 g of >90% REY content – **Figure 18**. The samples were delivered to DoE.



**Figure 18.** REY oxide product.

#### 4.2.2 Production of CMs: Scandium Product

Production of the CM scandium product was performed from the dry precipitate resulting from post-processing steps. The project team validated an LLX process (developed under IRAD funding) for the selective recovery scandium, a high value critical mineral. In the initial runs, using this LLX process the project team was able to increase Sc relative content from 3.3 wt.% in the feed to 41 wt.% in the strip phase which indicated >12X enrichment in scandium content for a single cycle A 51% Sc yield was achieved using two stripping stages. The analysis of this initial runs is shown in **Figure 19**:



**Figure 19.** Scandium recovery process – initial runs.

Two liquid-liquid extraction (LLX) cycles were used to achieve the required Sc relative content. The project team generated a total product (three samples) of 1 g with >85% Sc relative content. The samples (**Figure 20**) were delivered to DoE:

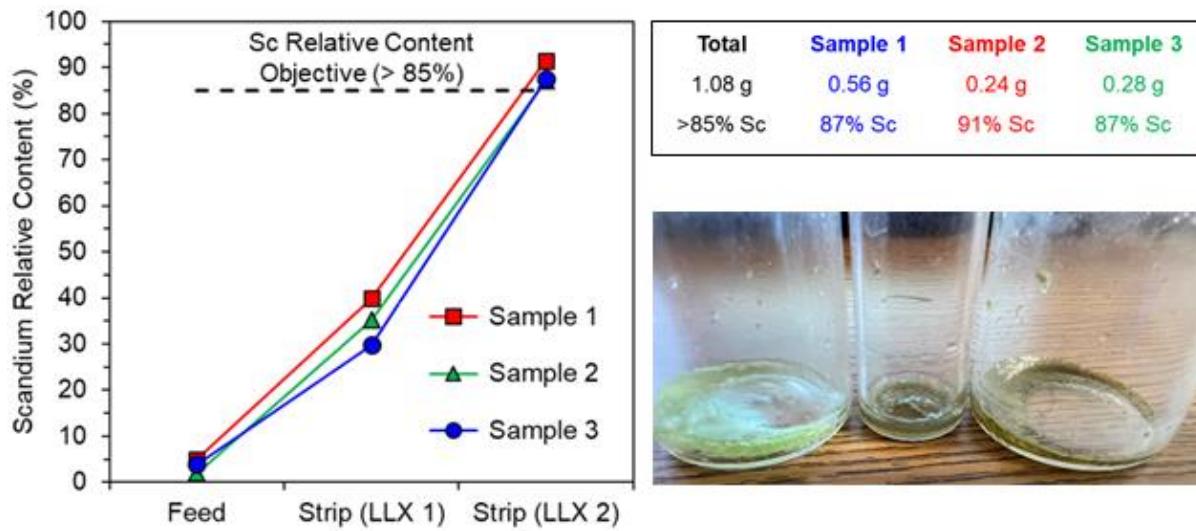


Figure 20. Sc product samples.

#### 4.2.3 Production of CMs: Aluminum Product

The project team developed a process to recover the critical mineral aluminum for the feedstock provided by the LLX waste stream (see **Figure 5**). This feedstock was enriched in aluminum as analysis of various samples indicated ~ 45% Al relative elemental content. Recovery of aluminum represents an additional potential value stream from the LLX waste product. The aluminum recovery process was developed by PSI and scaled up by WWS. Key results are:

- Developed and demonstrated a bench scale process to selectively recover aluminum from LLX waste stream
- Optimized, scaled up process and produced 100 g of material with >70% purity (**Figure 21**)
- SEM analysis indicated similar morphologies of the bench scale and pilot materials (**Figure 21**)

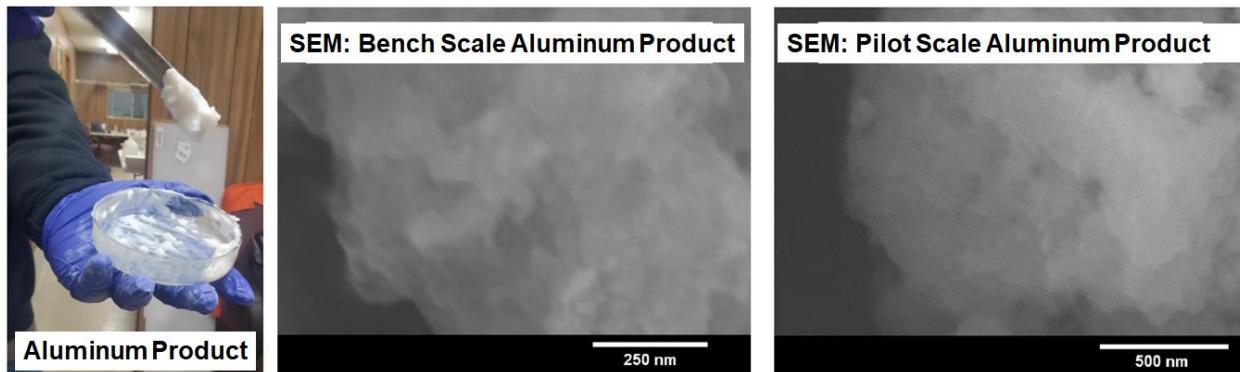


Figure 21. Photographic and SEM analyses of the aluminum product.

WWS produced five batches of aluminum product based on the scaled up aluminum recovery process. Samples were combined, ground to a powder by mortar and pestle, and elemental analysis was performed on the powder using ICP-OES. The project team produced 101 g of aluminum oxide type product with >70% aluminum relative content that was delivered to DoE (**Figure 22**):

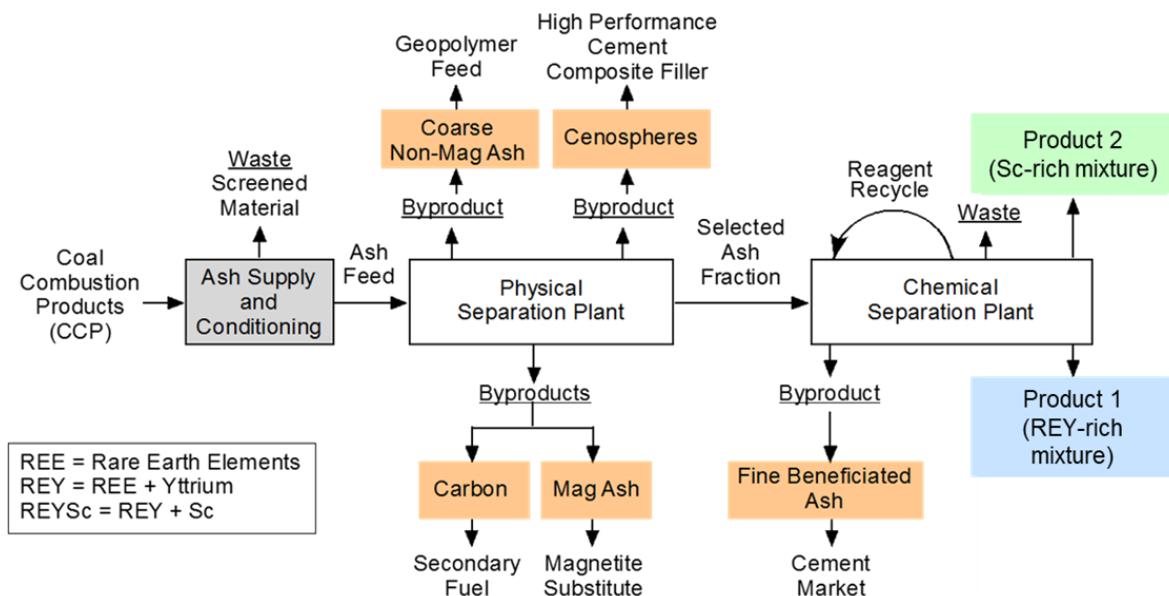


**Figure 22.** Final aluminum product.

#### 4.2.4 Techno-Economic Analysis (TEA)

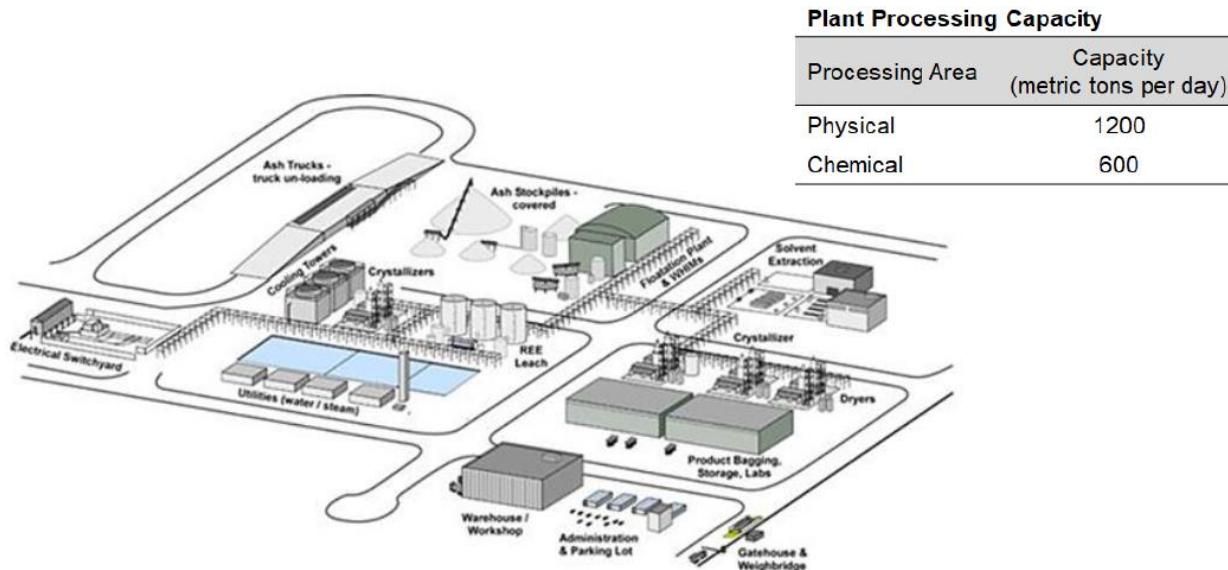
TEA was performed using the findings of initial Phase II and Phase II add-on programs. An updated techno-economic model from initial Phase II program to assess CAPEX, OPEX and revenues of a commercial operation was developed as discussed below.

**Figure 23** shows the process diagram that was used for the techno-economic model development. The process comprised a physical separation stage, followed by a chemical separation stage, followed by a post-processing stage. The primary products are REY-rich and Sc-rich concentrates. The byproducts are cement substitute, cenospheres, secondary fuel and magnetics. CAPEX and OPEX estimates are based on REYSc concentrate.



**Figure 23.** Process diagram for the techno-economic model.

A commercial plant model (**Figure 24**) was developed (Hatch Ltd contracted for CAPEX and OPEX estimates) using the following assumptions: (1) plant located at ash landfill site; (2) onsite physical and chemical processing; (3) model includes ash feedstock delivery, preparation, and staging operations:



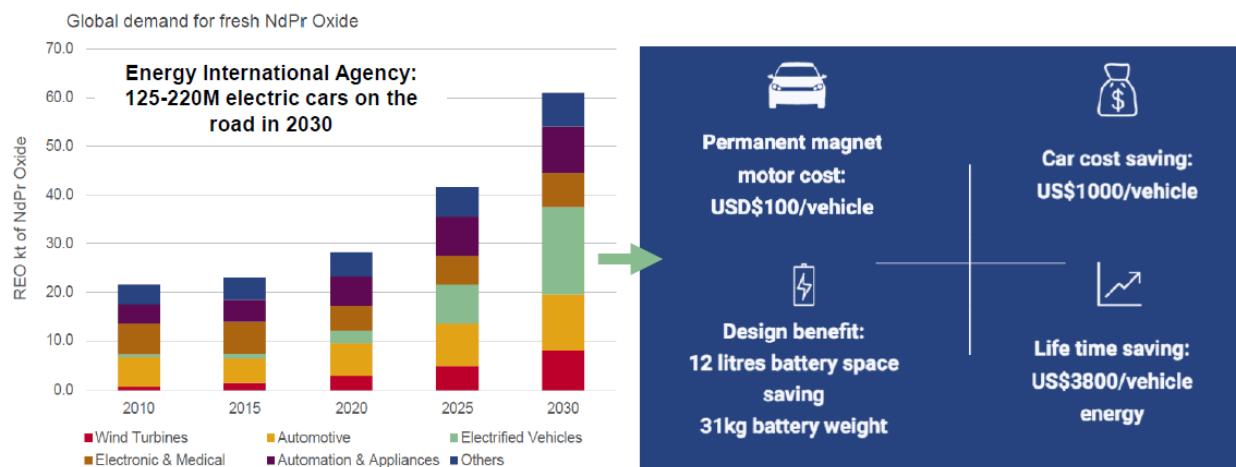
**Figure 24.** Commercial plant model.

**Table 10** outlines the updated annual production (relative to Phase II base program – see **Table 6**) of major REE salts, Sc salt, and byproducts. The analysis indicated that non-REE products significantly offset effects of REYSc commodity price fluctuations (pricing of non REYSc products varies with general economic conditions):

**Table 10.** Updated Analysis - Annual Production of Major REE Salts, Sc Salt, and Byproducts

Component	Quantity Produced tons/year	Portion of Revenue (%)		Worldwide Market tons/year	Market Application
		2020 REE Pricing	2011 REE Pricing		
REEs	38.2	2.0	10.8	170K	Batteries, Magnets, Alloys, Catalysts
Scandium	5.8	20.4	35.0	10-15	Alloys, Catalysts
Carbon	96K	6.7	4.6		Low-grade Fuel
Magnetic	20K	7.0	4.0		Magnetite Substitute
Non-Magnetic >200 Mesh	48K	1.1	0.8		Geopolymer Feed
Non-Magnetic <200 Mesh	186K	23.5	17.8	71.8M	Cement Substitute (Pozzolan)
Cenosphere Product	2K	39.3	27.1	~51K	Cpncrete Additive

The project team updated the techno-economic model from initial Phase II program to assess CAPEX and OPEX of a commercial operation. The analysis took into account emerging applications for REEs projected to boost demand and increase prices. Emerging markets for REEs included wind turbines, electrified vehicles and appliances. For instance, the use of an REE permanent magnet motor was estimated to reduce overall vehicle cost by \$3,800 USD/vehicle (Lynas Investor Day Presentation, 2019, **Figure 25**):



**Figure 25.** Emerging markets analysis.

Using these estimates the TEA resulted in the following observations:

- CAPEX was constructed using pricing and sizing of actual equipment necessary
- OPEX is heavily controlled by energy consumption, reagents and recycling efficiency, and labor.
- Byproducts are major sources of revenue adding to the REYSc revenues.
- Projected increases in REYSc prices will result in a faster/earlier positive cash flow.
- Cash flow depends on plant design, public/private partnership agreements, product and byproduct pricing, financing terms, and conditions including government support.
- Earnings are highly sensitive to inflation on the revenue side and emergence of new product markets

## 5. Conclusions and Recommendations for Further Developments

This program demonstrated that selected U.S. fly ash deposits are attractive feedstocks for the cost-effective recovery of rare earth elements (REE) and other critical minerals (CM). The project team successfully executed a multiphase program that developed and demonstrated pilot plant operations for cost-effective and environmentally-friendly production of rare earth elements oxide concentrates (REO) and critical minerals scandium and aluminum (in the forms of salts or oxide products). The team designed, constructed and operated two decoupled pilot plants: (1) an operational pilot plant with a capacity of 0.4 tpd for physical separation that used optimized processes to produce selected ash fractions as (a) feedstock for subsequent chemical processing and (b) as valuable by-products such as cement substitute, cenospheres, magnetic ash and secondary fuel carbon, and (2) an operational pilot plant with a capacity of 0.5 tpd for chemical processing that used optimized processes validated in PSI's micropilot plant for the production of: (a) REO concentrates, (b) critical minerals recovery (Sc, Al), and (c) beneficiated ash as valuable byproduct suitable for cement applications.

This program developed extensive databases on process chemistry, unit operations, plant engineering, and techno-economics that will enable further scale-up and commercial plant design. Specific developments will contribute to significant savings in energy, reagent usage, and operating costs. The combined results will contribute significantly for maturing the technologies of REE recovery from coal ash. The project team recommends further work to identify technology gaps and approaches to their closures based on the

program results, and eventual commercial implementation. **Table 11** lists the technology gaps that need to be closed on path to commercialization (identified during performance of Phase I and Phase II programs).

**Table 11.** Preliminary List of Technology Gaps Identified in Phase I and Phase II Programs

<u>Process Operation</u>	<u>Demonstrated Pilot Scale Technology</u>	<u>Alternate Technologies / Improvements</u>	<u>Evaluation Objective</u>	<u>Evaluation Methods</u>	<u>Comments</u>
Carbon Removal from Ash	Froth Floatation	Electrostatic Precipitation, Fluidized Bed Roasting	Minimize feed ash loss on ignition (LOI), maximize decarbonized ash yield	Bench scale technology evaluation, pilot scale testing	Use data from literature & other PSI/WWS programs
Ash Digestion	Single Stage Digestion	Sequential Digestion	Minimize acid consumption and energy expenditure	Micropilot and pilot scale testing	Use data from literature & other PSI/WWS programs
Rare Earth Purification	Counter-Current Liquid-Liquid Extraction (LLX)	Solid Phase Extraction, Co-Current LLX, Post-processing	Maximize REE purity and yield, and extractant consumption, increase equipment lifetime	Pilot scale testing	Reproducibly demonstrate high REE purity at pilot scale
Scandium Recovery	Batch Process	Continuous Liquid-Liquid Extraction	Maximize scandium product purity and yield	Bench scale and pilot scale testing	Scandium is important for process economics
Aluminum Recovery	Batch Process	Commercial filtration, washing and drying equipment, alternate reagents	Maximize aluminum product purity and yield	Bench scale and pilot scale testing	Aluminum represents an abundant critical mineral in coal ash
Acid Recovery / Recycle from Raffinate	To be demonstrated	Ion Exchange, Conversion to a value-added Product	Weight recovery of salt	Thermo-gravimetric analysis, oven calcination	Essential for high recovery of acid

Our team recognizes that key formal design and feasibility studies must be completed in order to retire technical risks and to seek funding from government as well as private investors for a commercial venture. Toward this goal, we suggest the roadmap below following the conclusion of the above-described Phase I and Phase II programs and conduct of additional studies to close the technology gaps in **Table 11**. A typical chemical plant project development path is outlined in **Figure 26** (key stages of the roadmap are presented below):

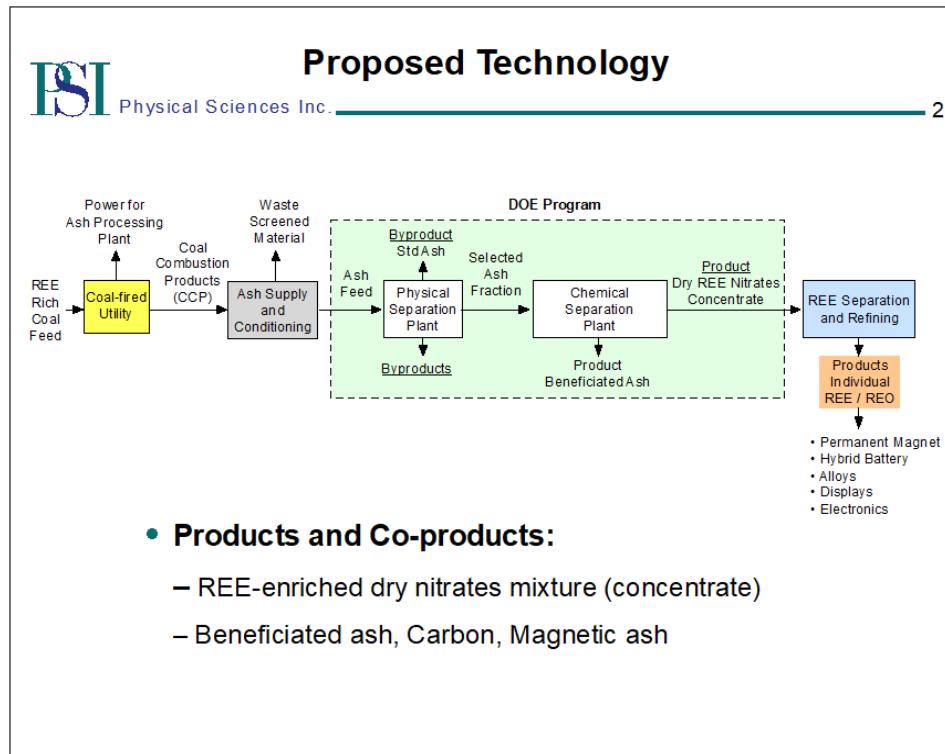
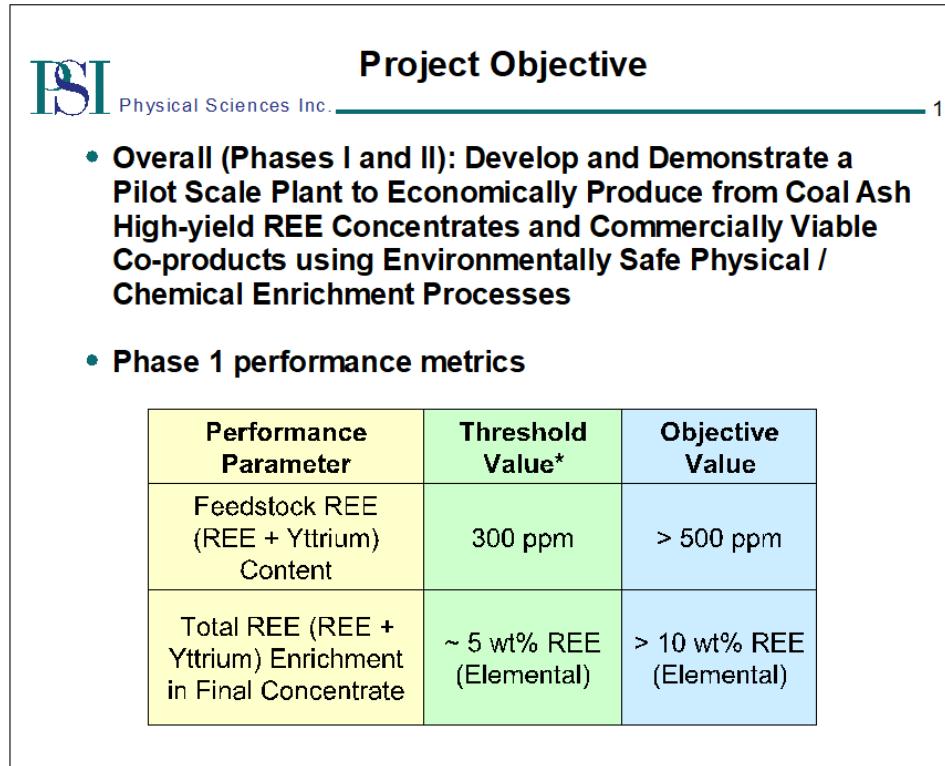


**Figure 26:** Roadmap - Chemical Plant Development Path

1. *Conceptual Design Study (Indicated Accuracy – 50% to + 50%)*
  - a. Engineering Concept Screening
  - b. Analysis of Alternatives (AoA)
  - c. Identification of Requirements and Cost of Pre-feasibility/Feasibility Studies
  - d. Outcome: Business Planning, Technology Assessment, Project Options
2. *Pre-feasibility Study (Indicated Accuracy – 30% to +30%)*
  - a. Techno-Economic Analysis (TEA)
  - b. Validity of Business Case
  - c. Outcome: Select Best Project Option, Set Project Objectives, Target Cost and Schedule, and GO/NOGO Decision Points
3. *Feasibility Study (Indicated Accuracy – 10% to +20%)*
  - a. Project Control Baseline, Monitor Budget Variations
  - b. Outcome: Detailed Project Basis consistent with Project Objectives and Business Plan, ROI Projects, and Project Implementation Planning

## 6. Supporting Information

1. ***"High Yield and Economical Production of Rare Earth Elements from Coal Ash" Poster presentation at: 2016 Crosscutting Research and Rare Earth Elements Portfolio Reviews, Pittsburgh, PA, 19 April 2016***



**Project Team & Budget**

**Physical Sciences Inc.** 3

- **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**

**Project Schedule**

**Physical Sciences Inc.** 4

Task	GFY 2016						GFY 2017											
	CY 2016			CY 2017			CY 2016			CY 2017								
	M	A	M	J	A	S	O	N	D	J	F	M	A	J	J	A		
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1.0 Program Management & Planning																		
2.0 Feedstock Sampling & Characterization																		
3.0 Feasibility Study																		
3.1 Physical Processing of Feedstock																		
3.2 Chemical Processing of Feedstock																		
3.3 Reagents Recycling & Waste Disposal																		
3.4 Continuous Process Evaluation																		
3.5 Plant Engineering, Modeling & Techno-Econ Analysis																		
– Go / No Go Evaluation																		
4.0 System Design																		
4.1 Physical Separation Plant Design																		
4.2 Chemical Separation Plant Design																		
5.0 Technology Development Support																		
Milestones, Technical Interchange Meetings & Reviews																		
Report																		

PSI = Physical Sciences Inc. Andover, MA  
 UK/CAER = University of Kentucky, Lexington, KY / Center for Applied Energy Research  
 ECL = Equinox Chemicals LLC, Albany, GA

Updated PMP ~ Sampling & Char. Plan ~ Only #1 Only #2 Only #3 Only #4 Only #5 ~ Ph 1 Final Brief @ NREL

Kickoff @ PSI @ UK/CAER Int. Rev #1 @ PSI TM #1 @ ECL Go / No Go Int. Rev #2 @ NREL Ph 1 Design Package, Ph 1 Summary Report

Int. Rev #1 @ PSI TM #2 @ ECL Go / No Go Int. Rev #2 @ NREL Ph 1 Final Brief @ NREL

TM #3 @ ECL Telcon Ph 1 Design Package, Ph 1 Summary Report

Ph 1 Final Brief @ NREL



Physical Sciences Inc.

## Phase I Milestones

5

- Based on start date of 1 March 2016

Milestone	Program Month	Planned Completion 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



Physical Sciences Inc.

## Task 1.0

### Project Management and Planning

6

- **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 priorities
- **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 2.0

### ESI Sampling and Characterization of Feedstocks - I

Physical Sciences Inc.

7

- **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
  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 2.0

### ESI Sampling and Characterization of Feedstocks - II

Physical Sciences Inc.

8

- 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 3.0 Feasibility Study

9

- **Objective:** Investigate
  - 1. 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 4.0 System Design - I

10

- **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



## Task 4.0 System Design - II

11

- **Chemical Separation Plant Design:**

- Integrate physical separation plant design with chemical separation plant design below
- Develop PFD and preliminary PID for pilot 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



## Task 5.0

### Technology Development Support

12

- **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

2. *"High Yield and Economical Production of Rare Earth Elements from Coal Ash", Presentation to: 2017 Rare Earth Elements Portfolio Review (DOE/NETL), Pittsburgh, PA, 22 March 2017*

**PSI** Physical Sciences Inc.

U.S. DEPARTMENT OF ENERGY | NETL NATIONAL ENERGY TECHNOLOGY LABORATORY

VG-2017-018

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

Physical Sciences Inc., Andover, MA (PI – Dr. Prakash Joshi)  
 Equinox Chemicals LLC, Albany, GA, and  
 Center for Applied Energy Research, Lexington, KY  
 DOE Award #: DE-FE0027167  
 Period of Performance: 3/1/2016 – 8/31/2017

*Presentation to:*  
 2017 Rare Earth Elements Portfolio Review (DOE/NETL)  
 Pittsburgh, PA  
 22 March 2017

Acknowledgement: This material is based upon work supported by the U.S. Department of Energy under Award DE-FE0027167.  
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Physical Sciences Inc. 20 New England Business Center Andover, MA 01810

**PSI** Physical Sciences Inc.

U.S. DEPARTMENT OF ENERGY | NETL NATIONAL ENERGY TECHNOLOGY LABORATORY

VG-2017-018 -1

## Project Goals and Objectives

- Overall Project Goal (Phases 1 and 2)**
  - Develop a pilot scale plant to demonstrate feasibility of economically producing from coal ash
    - High-yield REYSc concentrates, and
    - Commercially viable byproducts and co-products, using
    - Environmentally safe physical and chemical enrichment processes
- Phase 1 Project Objectives:**

*Phase 1 performance metrics:*

- Threshold Values > 2 wt% requirements of the solicitation
- Threshold values will be used for the GO/NOGO evaluation criteria

Parameter	Proposed Performance Range	Phase 1 Results and Values Used
Wt% of REY in dry nitrate concentrate final product	~ 5 wt%, threshold > 10 wt%, objective	> 10 wt%; Prop. Prod. > 30 wt%; Product 1
Ash feedstock REY Content	~ 300 ppm threshold > 500 ppm objective	556 ppm

Milestone	Program Month	Planned Completion 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	08 December 2016
Phase 1 Feasibility Study Results	11	31 January 2016
GO/NOGO	12	21 February 2017
Interim Review #2 @ DOE/NETL	12	21 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



Physical Sciences Inc.

## Outline



VG-2017-018 -2

- Process Overview
- Project Specific Objectives
- Ash Source: Selection and Characterization
- Physical Processing
- Chemical Processing
- Techno-Economic Model
- Summary



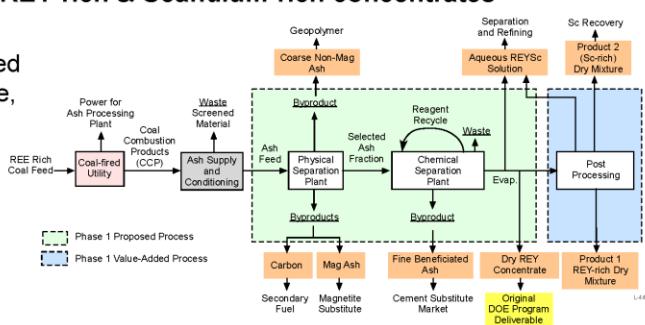
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## Process Overview



VG-2017-018 -3

- 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
- By-products:
  - Conventional/beneficiated ash as cement substitute, carbon as supplementary fuel, magnetic ash (magnetite substitute), geopolymers





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**Project Specific Objectives**

VG-2017-018 -4

- **Characterize ash feedstocks for total REYSc (REE + Y + Sc) content as well as distribution of the REE within ash particle morphologies**
  - Measurement protocol: ASTM D6357-11-1
- **Optimize the bench scale processes for REYSc enrichment already developed by PSI**
  - REE Enrichment Metric: ~ 5%
- **Perform key process experiments to anchor model below**
- **Develop a techno-economic process model that includes:**
  - Ash feedstock and reagent inputs, recycling of reagents, REYSc and by-product outputs of commercial value, accounting for market forecasts, mass balances and energy expenditures, and capital and operating expenses
- **Use above model to design a pilot scale REE enrichment plant**
  - Nominal capacity 1-5 tons (0.5-2.5 m<sup>3</sup>) of ash processed per day
  - Identify parameters for economical operation of plant



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VG-2017-018 -5

**Ash Source:  
Selection and Characterization**



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**Selected Ash Source:  
Dale Location**

VG-2017-018 -6



- Plant located near Ford, KY



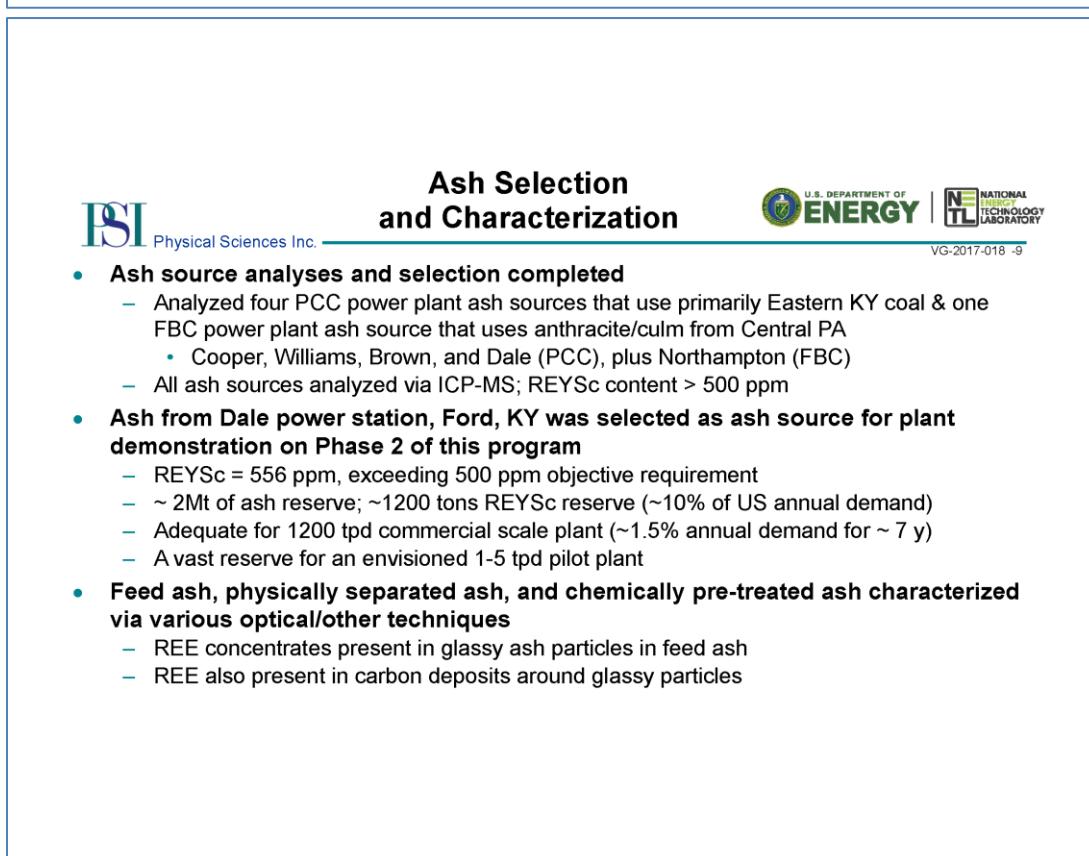
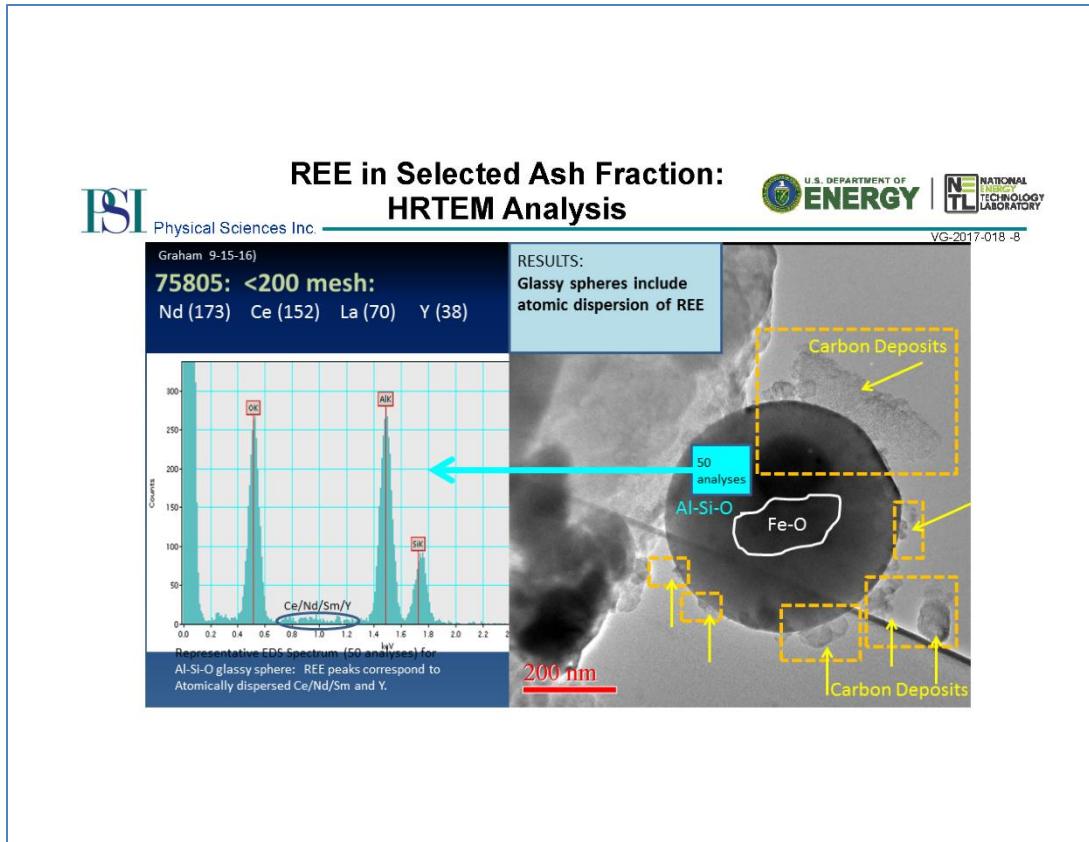
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**Selected Ash Source:  
Dale Ponds and Power Plant**

VG-2017-018 -7

*July 2014 sampling trip*

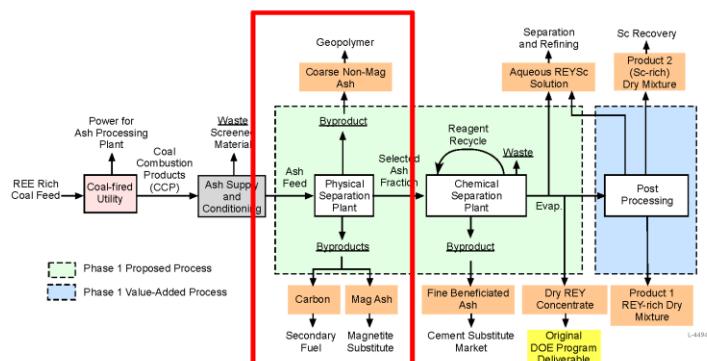
- 20 samples collected from several locations; a composite sample created for analysis and experimental work





VG-2017-018-10

## Physical Processing



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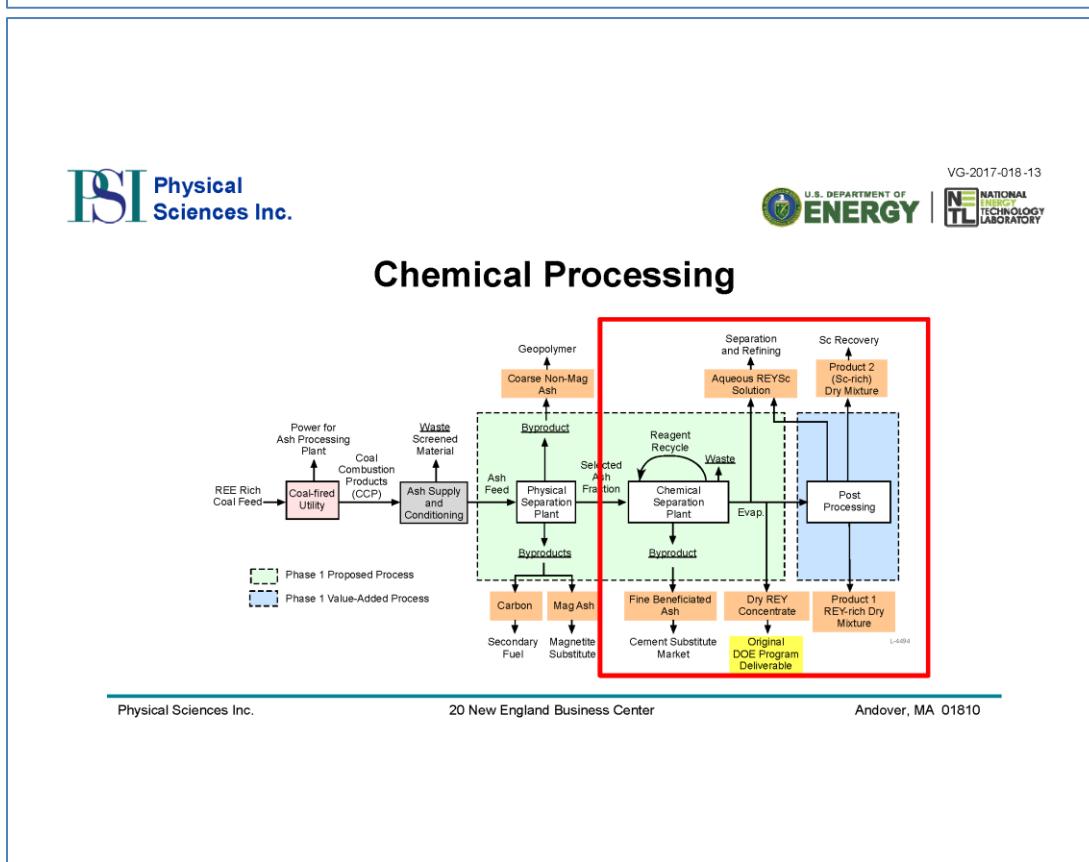
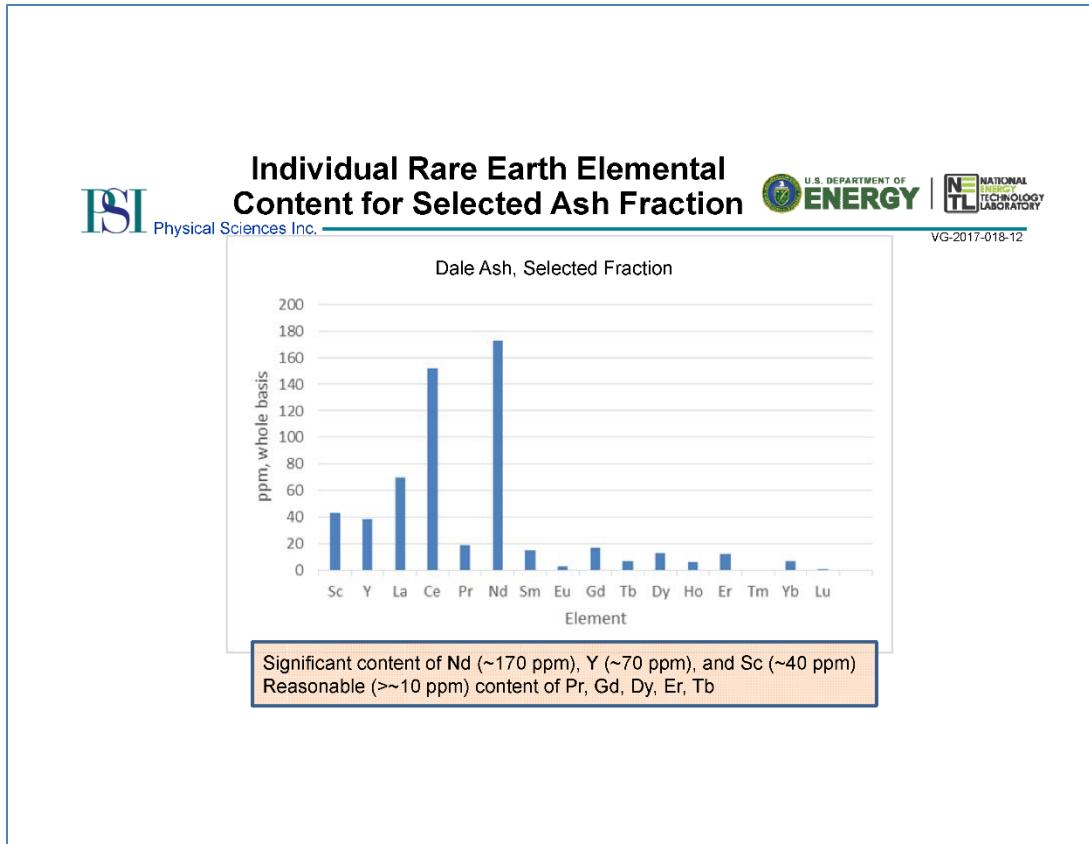


## Summary of Results



VG-2017-018-11

- **Each of the five ash sources were separated into five fractions**
    - +60 mesh ( $>250 \mu\text{m}$ ): Oversize, discarded
    - Froth product (principally C): Use as supplementary fuel, higher C products
    - Magnetic: Use as cheap magnetic substitute
    - Non-magnetic:
      - +200 mesh ( $>75 \mu\text{m}$ ): Use as conventional cement substitute
      - -200 mesh ( $<75 \mu\text{m}$ ): High REY content fraction for chemical processing
  - **Developed flowsheets for physical separation process**
  - **Developed a spreadsheet economic model of the process**
  - **Integrated physical processing with the chemical process model, and used for techno - economic assessment**





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## Summary of Results



VG-2017-018-14

- Determined optimal parameters for each process step to recover REE from Dale ash at high yield consistent with high enrichment
- Developed post-processing steps that yield two higher value product streams
  - REY-rich: REY Yield > 18%, Conc > 30 wt%; Sc Yield ~ 18%, Conc ~ 0.5 wt%
    - REE conc >> objective target (10 wt%)
  - Sc-rich: Sc Yield ~ 18%, Conc ~ 1.5 wt%; REE Yield > 4%, Conc ~ 6 wt%
    - REE conc >> threshold target (5 wt%)
- Reduced process time for individual steps and avoided elevated temperatures
- Experimental development of process scale up
- Process implementable on commercial scale; requires different optimization parameters for project economics
  - Tradeoff some of the high REYSc yield/enrichment to achieve better economics



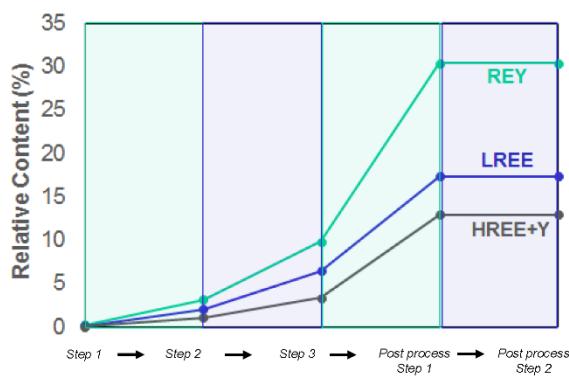
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## REY and Sc Relative Content (Enrichment wt%)



VG-2017-018-15

- Post-processing step 1 leads to:
  - 3X increase in REY enrichment
  - Efficient separation from other contaminants
  - Product well-suited for REY separation





VG-2017-018-16

## Techno-Economic Modeling

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### Techno-Economic Modeling Approach



VG-2017-018-17

- **Chemical processing and economics modeled in Aspen**
  - Capital and operating expenses per model
    - Modified per Equinox experience
  - Result: Pro forma spreadsheet model
- **Physical processing economics modeled**
  - Capital and operating expenses per CAER experience
  - Result: Pro forma spreadsheet model
- **Integrated process economics modeled**
  - Added capital expenditures of physical and chemical processes
  - Physical process decoupled from chemical
  - Physical *and* chemical processing performed on-site at Dale pond
    - Save transportation costs

**Techno-Economic Modeling Results**  
**Capacity, Products and Annual Production**



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VG-2017-018-18

- **A nominal 1200 tpd physical processing plant and 600 tpd chemical processing plant needed for**
  - Return on Investment (ROI) of < 5 years
- **Both physical and chemical processing plants**
  - Collocated at ash source to eliminate transportation costs; Decoupled ops
  - 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, Sc, and Y nitrate concentrate (kg)**

	LREE					H	R	E	E	Other HREE		
Sc	La	Ce	Pr	Nd	Eu	Gd	Tb	Dy	Y	Ho, Er, Tm, Yb, Lu		
11K	11K	9K	300	10K	350	2K	300	2K	14K	300-800		

➤  $\text{Sc}_2\text{O}_3$  production worldwide (2013) ~ 10K kg, Our oxide equivalent ~ 3000 kg

- **Annual production of byproducts (kg)**

Carbon	Magnetic	Non-mag > 200 mesh	Non-mag < 200 mesh
100K	21K	50K	124K

**Summary**



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VG-2017-018-19

- **The PSI team has effectively addressed the technical and economic aspects of recovery of trace REYSc elements from coal ash for our proposed physical and chemical separation processes**
- **Ponded ash from Dale power plant in Ford, KY was selected as the ash source for demonstration of key process elements on pilot scale as well as a potential source for a commercial scale plant**
  - > 500 ppm REYSc
- **Both physical and chemical processing plants are collocated at ash source for process economy; their modular designs will permit transportability from one ash source to another**
  - 1200 tpd (~ 500 m<sup>3</sup>/d) physical processing, 600 tpd (~ 250 m<sup>3</sup>/d) chemical processing
- **For our processes with the 600 tpd throughput, reagent costs have the most significant influence on Return On Investment (ROI)**
  - High efficiency recycling/re-use of reagents is required

3. *"High Yield and Economical Production of Rare Earth Elements from Coal Ash", Presentation to: Rare Earth Elements (REE) Program Portfolio. 2018 Annual Review Meeting, Pittsburgh, PA, 10 April 2018*



VG-2018-024

## 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  
2018 Annual Review Meeting  
Pittsburgh, PA  
10 April 2018

Acknowledgement:  
This material is based upon work supported by the U.S. Department of Energy under Award DE FE0027167.

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Andover, MA 01810

VG-2018-024-1



## Presentation Outline

- Phase 2 Programmatic
- Rare Earths Recovery Process Overview
- Phase 1 Results Summary
- Phase 2 Overview
- Phase 2 Objectives
- Phase 2 Tasks, Schedule, Milestones, and Deliverables
- Current Status
- Summary



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## Phase 2 Programmatic

VG-2018-024-2

- **Area Of Interest (AOI) 2 program: Pilot Scale Technology**
  - Existing separation technology previously demonstrated successfully on bench scale
  - Ready or near-ready for design at pilot scale
    - Pilot plant design to be delivered at end of Phase I
  - Ready for scale up to commercial scale at completion of Phase II
- **30-month Phase 2 program: 9/29/2017 – 3/31/2020**
  - Phase 1 ended 8/31/2017
- **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**

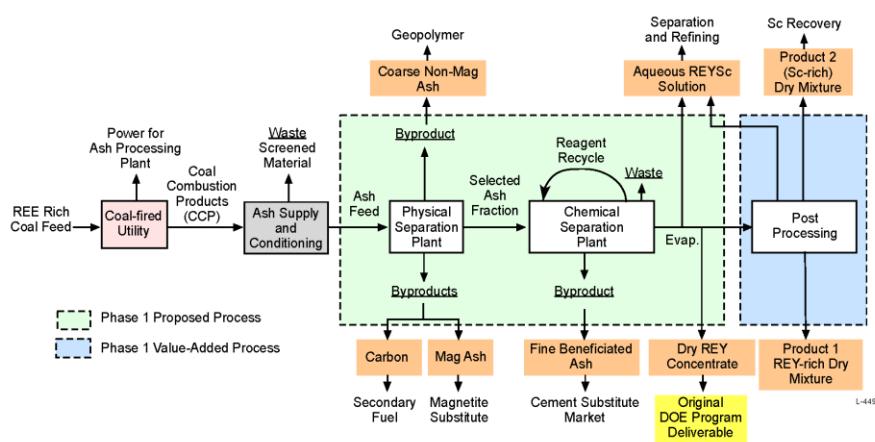


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## Rare Earths Recovery Process Overview

VG-2018-024-3

- **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**
- **By-products: Cement substitute, secondary fuel carbon, .....**



## Phase I Results Summary



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## Ash Source Selection

VG-2018-024-5

- Ash from eastern KY coal selected for Phase 2 developments
- The average REYSc content of 556 ppm measured from a composite of 20 ash samples > 300 ppm (DOE requirement)



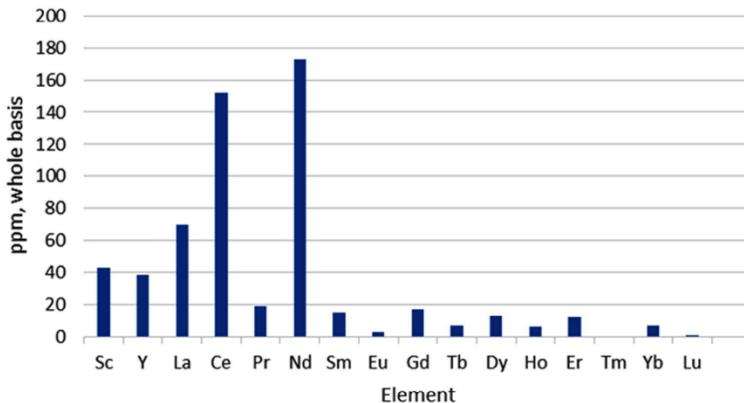
Sample	Sc	Y	$\Sigma$ REE	$\Sigma$ REY	LREE/HREE
Composite	33	59	457	516	7.46



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## Individual Rare Earth Elemental Content for Selected Ash Fraction

VG-2018-024-6



Significant content of Nd (~170 ppm), Y (~70 ppm), and Sc (~40 ppm)  
Reasonable (>~10 ppm) content of Pr, Gd, Dy, Er, Tb

6



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## Chemical Processing of Selected Ash Fraction

VG-2018-024-7

- Developed chemical processes for the selected ash fraction to recover REYSc with high yield and high enrichment in two final concentrate products of commercial value.
  - Concentration expressed on elemental basis i.e. the content of REY or Sc relative to that of all elements)
- **Product 1: REY-rich concentrate:**
  - REY Yield > 18%, Conc > 30 wt%
  - Sc Yield ~ 18%, Conc ~ 0.5 wt%
  - REY concentration >> objective target (10 wt%)
- **Product 2: Sc-rich concentrate:**
  - Sc Yield ~ 18%, Conc ~ 1.5 wt%
  - REY Yield > 4%, Conc ~ 6 wt%
  - REE conc >> threshold target (5 wt%)



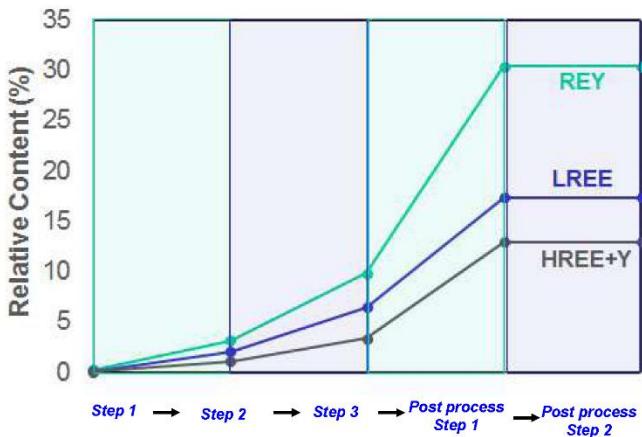
## Post-Processing for REY and Sc Relative Content

Physical Sciences Inc.

VG-2018-024-8

- **Post-processing step 1 leads to:**

- 3X increase in REY enrichment
- Efficient separation from other contaminants
- Product well-suited for REY separation



## Techno-economic Modeling Approach

Physical Sciences Inc.

VG-2018-024-9

- **Chemical processing and economics modeled in Aspen**
  - 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 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**



## Capacity, Products and Annual Production

Physical Sciences Inc.

VG-2018-024-10

- **Designed a nominal 1200 tpd physical processing plant and 600 tpd chemical processing plant**
  - Return on Investment (ROI) of < 5 years
- **Both physical and chemical processing plants**
  - Decoupled operations
  - Modular designs for operational flexibility and transportability
- **Byproduct ash fractions shipped to local markets**
- **Annual production of major REE, Sc, and Y concentrate (kg)**

	LREE					H	R	E	E		Other HREE
Sc	La	Ce	Pr	Nd	Eu	Gd	Tb	Dy	Y	Ho, Er, Tm, Yb, Lu	
11K	11K	9K	300	10K	350	2K	300	2K	14K	300-800	

## Phase II Overview



## Overall Project Goal

VG-2018-024-12

- Develop and demonstrate a pilot scale plant to economically produce salable rare earth element-rich concentrates, including yttrium, scandium, and commercially viable co-products from coal ash feedstock; using environmentally safe, and high-yield physical and chemical enrichment processes.

- Phase 2 Project Metrics

Performance Parameter	Threshold Value	Objective Value
Feedstock REYSc <sup>†</sup> Content	>300 ppm (Whole Mass Basis)	>500 ppm (Whole Mass Basis)
Total REYSc Enrichment in Final Concentrate	>10 wt% (Elemental Basis)	>20 wt% (Elemental Basis)
Return on Investment <sup>*</sup>	<7 y	<5 y
Delivered Concentrate Quantity <sup>§</sup>	~50 g <sup>§</sup>	~0.5 kg <sup>§§</sup>

REYSc = Rare Earth Elements Plus Yttrium and Scandium, \*Scale-dependent ~ 600 tpd, & Ten 5g split samples, 5g split sample required per solicitation.



## Phase II Team

VG-2018-024-13

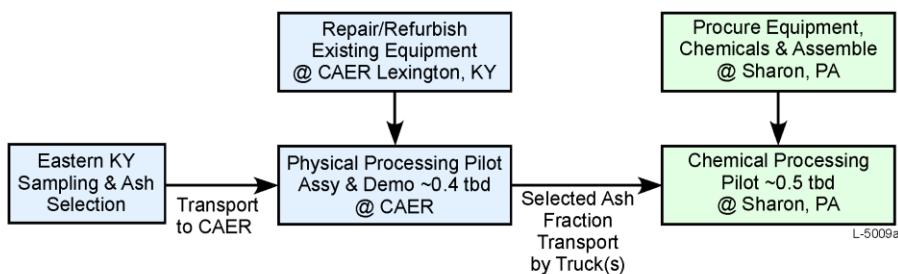
- The CAER, PSI, WWS team provides a complete integrated science, technology, engineering, technology transition, and commercialization solution for DOE/NETL
- Key Personnel:
  - **PSI:**
    - Dr. Prakash Joshi, PI/PM
    - Dr. Dorin Preda, Lead Chemist
    - Dr. Matthew Boucher, Lead Chemical Engineer/Process Modeling
  - **CAER:**
    - Dr. James Hower, Coal geochemistry, materials characterization
    - Dr. John Groppo, Mineral processing, feedstock logistics, site qualification
  - **WWS:**
    - Mr. Todd Beers: Chemical Engineering and technology commercialization
    - Mr. Michael Schrock, Chemical Engineering; plant design

## Phase 2 Scope – I Overall Program



VG-2018-024-14

- Demonstrate the Phase 1 REYSc separation/enrichment technology in pilot plant(s) with *decoupled* operating capacities of ~ 0.4 tpd physical processing, and ~ 0.5 tpd chemical processing.
  - Both pilot designs will be *modular* and *transportable*.



L-5009a

## Phase 2 Scope – II Physical Processing Pilot



VG-2018-024-15

- The physical pilot plant comprises existing equipment at the CAER facility in Lexington, KY, which is being repaired/refurbished for use in Phase 2
- The physical pilot will be decoupled from the chemical pilot located at the WWS facility in Sharon, PA.
  - ~ 40 tons of selected ash will be transported to CAER facility
- Physical processing pilot, will be operated over < ~ 4 weeks for a significant demonstration while conserving project funds, producing 10 to 20 tons of the chemically processable ash fraction
- The selected ash fraction will then be transported to the WWS plant in Sharon, PA for chemical processing



Physical Sciences Inc.

## Phase 2 Scope – III Chemical Processing Pilot

VG-2018-024-16

- Chemical processing pilot will utilize WWS's existing, proven solvent extraction equipment to the maximum extent possible, and also procure additional equipment
- The chemical pilot will be operated over ~ 2 to 4 weeks for a significant demonstration while also conserving project funds, producing ~ 50 to 500 grams of deliverable REYSc-enriched concentrate
- Pilot will demonstrate the high recovery/ recycling of reagents at > 95% efficiency.
- Establish processes for environmentally safe disposal of waste products



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## Phase 2 Scope – IV

### Chemical Micropilot & Techno-Economic Modeling

VG-2018-024-17

- A ~1-5 kg/d Micropilot Plant will be developed at PSI, Andover, MA
  - Quick turnaround validation of pilot plant processing parameters and to provide data for chemical pilot plant design
- A critical part of Phase 2 will be to refine, enhance, and validate the Phase 1 Aspen-based Techno-Economic Model of REYSc recovery from coal ash using the results from physical and chemical pilot plant operations
  - Fidelity of the resulting Phase 2 model is expected to be AACE Class 2
  - Model will be used to develop design of a commercial scale plant for profitable (ROI < 5 y) REYSc at the conclusion of Phase 2

## Phase II Objectives



Physical Sciences Inc.

## Phase 2 Project Objectives - I

VG-2018-024-19

### Phase 2 Objective:

- Demonstrate the REYSc separation/enrichment technology developed in Phase 1 in a pilot scale plant with operating capacity of 0.1-1 metric ton per day (tpd)

### Specific Objectives:

1. Refine and complete detailed design of the chemical pilot plant(s) from Phase 1
  - Modular, transportable designs
2. Assemble and operate a *micropilot (chemical) plant* for quick turnaround validation of pilot plant processing parameters, and provide data for chemical pilot plant design



Physical Sciences Inc.

## Phase 2 Project Objectives - II

VG-2018-024-20

3. Assemble pilot scale plant at CAER for physical processing of ash
4. Construct a *modular* pilot scale chemical plant at WWS facilities
5. Demonstrate operation of the physical pilot plant using the power plant (ash feedstock selected in Phase 1)
  - Modular, mobile CAER plant that uses the selected ash feedstock
  - Operation at ash source, decoupled from chemical pilot plant



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## Phase 2 Project Objectives - III

VG-2018-024-21

6. Demonstrate operation of the chemical pilot plant using the selected ash fraction produced by the physical pilot plant
  - Operation at WWS facility in Sharon, PA
    - Selected ash fraction transported to this facility from Lexington, KY
7. Refine and enhance the Phase 1 techno-economic model using results of above physical and chemical pilot plant operations
  - Produce AACE Class 2 costing fidelity model in Phase 2
    - Current Phase 1 model is AACE Class 3
8. Develop and provide design of a commercial scale plant for profitable REYSc recovery from coal ash at Phase 2 conclusion
  - Use the above refined Phase 2 techno-economic model
  - ROI metrics as previously stated

## Phase II Tasks, Schedule, Program Milestones, and Deliverables



### Phase 2 Tasks

VG-2018-024-23

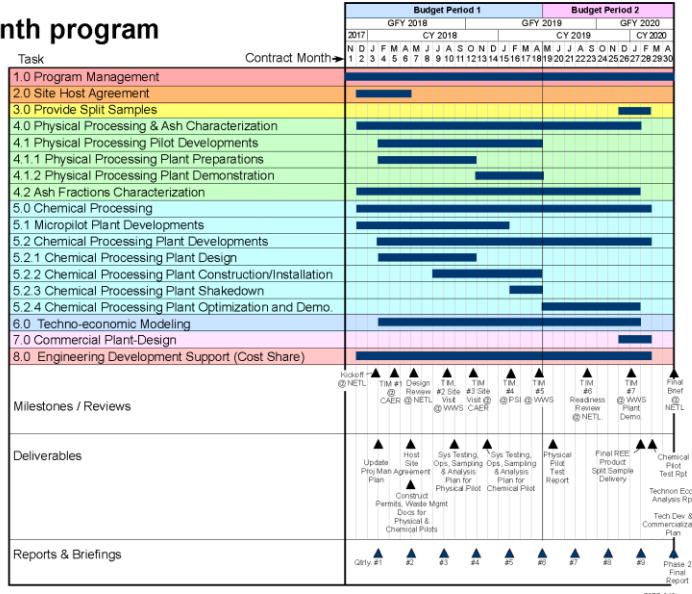
Task #	Title	Lead/Performing Organizations	Performance Period (Months)	Budget Period
1.0	Project Management and Planning	PSI	1-18	1
1.0	Project Management and Planning	PSI	19-30	2
2.0	Site Host Agreement	PSI	2-6	1
3.0	Provide Split Samples	PSI	26-28	2
4.0	Physical Processing Demo & Ash Characterization	UK/CAER	2-27	1&2
4.1	Physical Processing Pilot Implementation	UK/CAER	4-18	1
4.1.1	Physical Processing Plant Preparations	UK/CAER	4-12	1
4.1.2	Physical Processing Plant Operational Demo	UK/CAER	12-18	1
4.2	Ash Fractions Characterization	UK/CAER	2-18	1
4.2	Ash Fractions Characterization	UK/CAER	19-27	2
5.0	Chemical Processing	WWS/PSI	2-28	1&2
5.1	Micropilot Plant and Experimental Developments	PSI	2-16	1
5.2	Chemical Processing Pilot Development	WWS/PSI	4-28	1&2
5.2.1	Chemical Pilot Plant Design	WWS/PSI	4-12	1
5.2.2	Chemical Pilot Plant Construction	WWS/PSI	9-18	1
5.2.3	Chemical Pilot Plant Shakedown	WWS/PSI	16-18	1
5.2.4	Chemical Pilot Plant Optimization and Demo	WWS/PSI	19-27	2
6.0	Techno-Economic Modeling	PSI	4-18	1
6.0	Techno-Economic Modeling	PSI	19-27	2
7.0	Commercial Plant Design	WWS/PSI	26-28	2
8.0	Engineering Development Support	PSI	2-18	1
8.0	Engineering Development Support	PSI	19-28	2



## Phase 2 Schedule

VG-2018-024-24

- 30-month program



TIM = Technical Interchange Meeting

7377-043



## Phase 2 Milestones and Deliverables

VG-2018-024-25

Milestone	Program Month	Planned Completion Date
Kickoff Meeting	1	24 January 2018
Updated Project Management Plan	1	31 January 2018
Technical Interchange Meeting (TIM) #1 @CAER	3	15 March 2018
Quarterly Report #1	3	31 January 2018
Design Review @ NETL	6	04 May 2018
Host Site Agreement	6	04 May 2018
Construction, Waste Mgmt Docs for Phys & Chem Pilots	6	04 May 2018
Quarterly Report #2	6	04 May 2018
TIM #2 @ WWS (Chem Plant Site Visit)	9	10 August 2018
Quarterly Report #3	9	27 July 2018
Sys Test, Ops, Sample & Analysis Plan for Phys Pilot	10	24 August 2018
TIM #3 @ CAER (Physical Pilot Site Visit/Demo)	12	26 October 2018
Quarterly Report #4	12	29 October 2018
Sys Test, Ops, Sample & Analysis Plan for Chem Pilot	13	30 November 2018
Quarterly Report #5	15	28 January 2019
TIM #4 @ PSI (Micropilot Site Visit)	15	08 February 2019
TIM #5 @ WWS (Chemical Pilot Shakedown Tests)	18	26 April 2019
Quarterly Report #6	18	29 April 2019
Physical Pilot Test Report	19	04 June 2019
Quarterly Report #7	21	29 July 2019
TIM #6 @ NETL (Readiness Review)	22	04 September 2019
Quarterly Report #8	24	28 October 2019
TIM #7 @ WWS (Chemical Pilot Demo)	26	06 January 2020
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Final REYS Product Split Sample Delivery	27	07 February 2020
Chemical Pilot Test Report	28	28 February 2020
Techno-Economic Analysis Report	28	28 February 2020
Technology Development and Commercial Plant Design	28	28 February 2020
Phase 2 Final Report	30	30 April 2020
Phase 2 Final Briefing @ NETL	30	30 April 2020

## Current Status

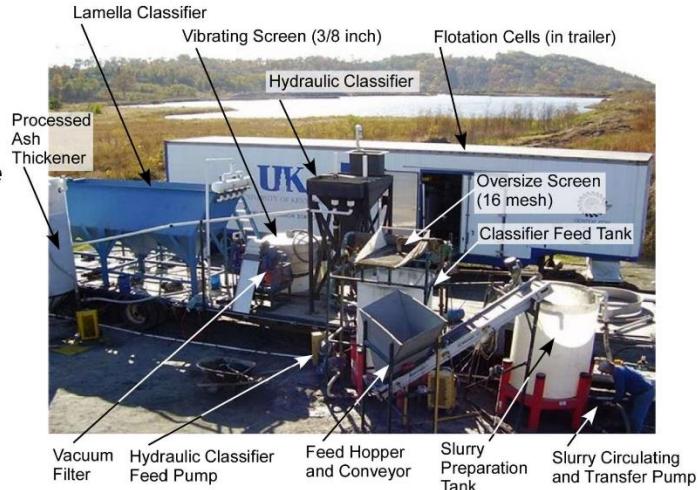


### Modular, Transportable Physical Processing Plant

Physical Sciences Inc.

VG-2018-024-27

- **Reconditioned 40' Trailer for Transport & Use**
- **Reconditioned 2" Hydrocyclone**
- **Reconditioned 4'x4' Hydraulic Classifier**
- **Assembling 2x30 liter Flotation Cells**
- **Assembling 1'x1.5' Drum Magnet**



L-5145

Ash Feedstock site agreement with EKPC  
for access, logistics, sampling, and transportation  
has been negotiated in currently in final round of signatures



## Modular, Transportable Chemical Processing Plant

Physical Sciences Inc.

VG-2018-024-28

- Winner's facility (formerly a Westinghouse torpedo production plant) in Sharon, PA is currently being refurbished with structural, spatial and electrical/water/ utilities infrastructure for the plant
- Detailed plant design currently in progress:
  - Building upon Phase 1 Final Design Package, Feasibility Report, and Aspen Techno-Economic Model(s)
  - Initial discussions to review requirements and design approach on 03/27/2018
- Design review ~ 4 May 2018



Physical Sciences Inc.

## Micropilot Plant

VG-2018-024-29

- Plant, hoods, exhausts assemblies done
- Safety Reviews, and Safety Walkthrough completed
- Chemicals monitoring and waster disposal procedures in place
- Plant Standard Operating Procedure (SOP) approved by Safety Committee
- Plant testing has commenced: Four 5 L reactors plus peripherals





## Summary

VG-2018-024-30

- The Physical Sciences Inc., Winner Water Services, and Center for Applied Energy Research Phase 2 program has been initiated and is progressing per our proposed plan
  - Kickoff meeting held at NETL 24 January 2018
- Host site agreement with EKPC for access, logistics, sampling, facilitates, transportation of selected ash feedstock....has been negotiated in currently in final round of signatures
- Refurbishment, assembly, and construction of Physical Processing Pilot is well underway at CAER, Lexington Site preparations for the Chemical Processing Pilot in progress at Winner Water facilities in Sharon, PA
- Detail design of Chemical Processing Pilot has been initiated
- Micropilot Plant at PSI in Andover, MA is ready for operation with initial data expected this week

4. *"High Yield and Economical Production of Rare Earth Elements from Coal Ash", Presentation to: Presentation to: Rare Earth Elements (REE) Program Portfolio, 2019 Annual Review Meeting, Pittsburgh, PA, 9-11 April 2019*

 VG-2019-41

## 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,  
2019 Annual Review Meeting, Pittsburgh, PA,  
9-11 April 2019

Acknowledgement:  
This material is based upon work supported by the U.S. Department of Energy under Award DE FE0027167.

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Physical Sciences Inc. 20 New England Business Center Andover, MA 01810

 VG-2019-41-1

## Presentation Outline

- Phase 2 Programmatic
- Rare Earths Recovery Process Overview
- Phase 2 Tasks, Schedule, Milestones, and Deliverables
- Summary of Phase 2 Results to Date



## Phase 2 Programmatic

VG-2019-41-2

- **Area Of Interest (AOI) 2 program: Pilot Scale Technology**
  - Existing separation technology previously demonstrated successfully on bench scale
  - Ready or near-ready for design at pilot scale
    - Pilot plant design to be delivered at end of Phase I
  - Ready for scale up to commercial scale (design) at completion of Phase II
- **30-month Phase 2 program: 9/29/2017 – 9/31/2020**
  - Phase 1 ended 8/31/2017
- **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**



VG-2019-41

## Rare Earths Recovery Process Overview

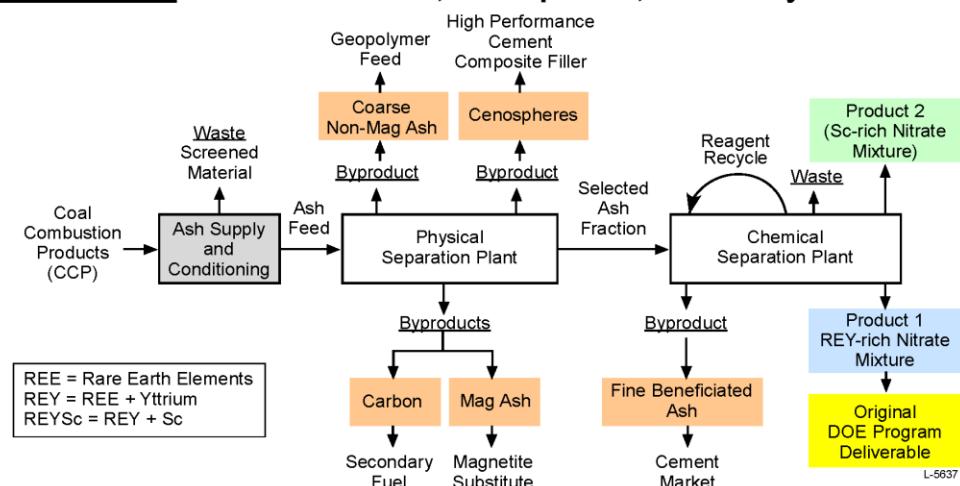


Physical Sciences Inc.

VG-2019-41-4

## 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
- By-products: Cement substitute, cenospheres, secondary fuel carbon...

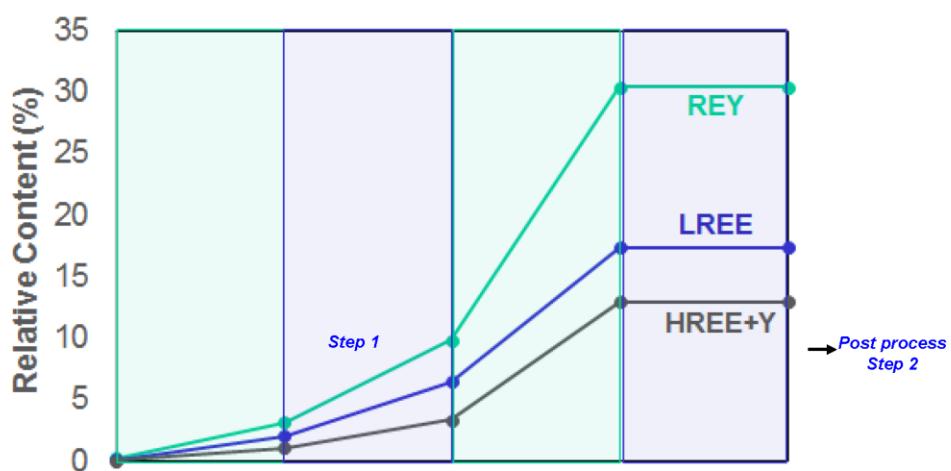


## Post-Processing for REY and Sc Relative Content

Physical Sciences Inc.

VG-2019-41-5

- Post-processing leads to:
  - 3X increase in REY enrichment
  - Efficient separation from other contaminants
  - Product well-suited for REY separation





VG-2019-41

## Phase 2 Overview

Physical Sciences Inc.

20 New England Business Center

Andover, MA 01810



## Overall Project Goal

VG-2019-41-7

- **Develop and demonstrate a pilot scale plant to economically produce salable rare earth element-rich concentrates, including yttrium, scandium, and commercially viable co-products from coal ash feedstock; using environmentally safe, and high-yield physical and chemical enrichment processes.**
- **Phase 2 Project Metrics**

Performance Parameter	Threshold Value	Objective Value
Feedstock REYSc <sup>†</sup> Content	>300 ppm (Whole Mass Basis)	>500 ppm (Whole Mass Basis)
Total REYSc Enrichment in Final Concentrate	>10 wt% (Elemental Basis)	>20 wt% (Elemental Basis)
Return on Investment <sup>*</sup>	<7 y	<5 y
Delivered Concentrate Quantity <sup>§</sup>	~50 g <sup>§</sup>	~0.5 kg <sup>§§</sup>

REYSc = Rare Earth Elements Plus Yttrium and Scandium, \*Scale-dependent ~ 600 tpd, &Ten 5g split samples, 5g split sample required per solicitation.



## Phase II Team

VG-2019-41-8

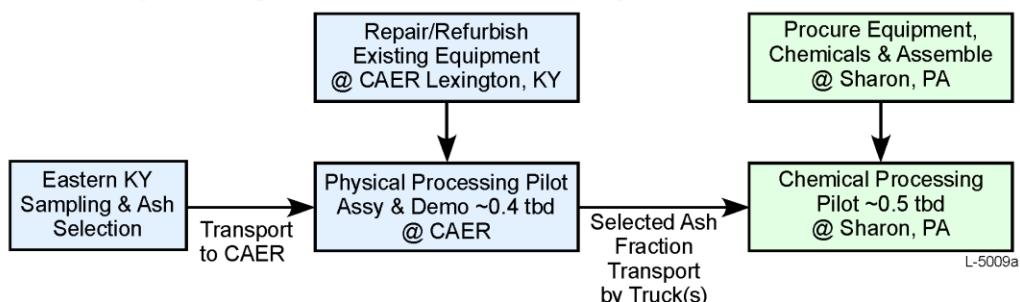
- The CAER, PSI, WWS team provides a complete integrated science, technology, engineering, technology transition, and commercialization solution for DOE/NETL
- Key Personnel:
  - PSI:
    - Dr. Prakash Joshi, PI/PM
    - Dr. Dorin Preda, Lead Chemist
    - Dr. David Gamliel, Lead Chemical Engineer/Process Modeling/TEA
  - CAER:
    - Dr. James Hower, Coal geochemistry, materials characterization
    - Dr. John Groppo, Mineral processing, feedstock logistics, site qualification
  - WWS:
    - Mr. Todd Beers: Chemical Engineering and technology commercialization
    - Mr. Michael Schrock, Chemical Engineering; Plant Design



## Phase 2 Scope – I Overall Program

VG-2019-41-9

- Demonstrate the Phase 1 REYSc separation/enrichment technology in pilot plant(s) with *decoupled* operating capacities of ~ 0.4 tpd physical processing, and ~ 0.5 tpd chemical processing.
  - Both pilot designs will be *modular* and *transportable*



L-5009a

Phase	Start TRL	End TRL
1	3	4
2 (Current)	4	5
2 (End)	5	6+



Physical Sciences Inc.

## Phase 2 Scope – II Physical Processing Pilot

VG-2019-41-10

- The Physical pilot plant comprises existing equipment at CAER facility in Lexington, KY, which was repaired/refurbished for Phase 2
- The physical pilot is decoupled from the chemical pilot located at the WWS facility in Sharon, PA.
  - ~ 20 tons of selected ash transported to CAER facility
- Physical processing pilot, will be operated over < ~ 4 weeks for a significant demonstration while conserving project funds, producing 10 to 20 tons of the chemically processable ash fraction
- Selected chemical processing operations may be collocated with physical processing to minimize amount of material to be processed in the chemical (pilot) plant, and to save transportation costs
- Selected ash fraction to be transported to the WWS plant in Sharon, PA for chemical processing



Physical Sciences Inc.

## Phase 2 Scope – III Chemical Processing Pilot

VG-2019-41-11

- Chemical processing pilot utilizes WWS's existing, proven solvent extraction equipment to minimize costs, with additional equipment procured for pre-extraction operations and for reagent compatibility
- The chemical pilot will be operated over ~ 2 to 4 weeks for a significant demonstration while also conserving project funds, producing ~ 50 to 500 grams of REYSc-enriched product deliverable
- Environmentally safe disposal of waste products
- Pilot will demonstrate the high recovery/ recycling of reagents; > 95%

## Phase 2 Scope – IV

### Chemical Micropilot & Techno-Economic Modeling



Physical Sciences Inc.

VG-2019-41-12

- A ~1-5 kg/d Micropilot Plant has been developed at PSI, Andover, MA
  - Quick turnaround validation of pilot plant processing parameters and to provide data for chemical pilot plant design
- A high fidelity Aspen-based Techno-Economic Model of REYSc recovery from coal ash using the results from physical and chemical pilot plant operations
  - Refine, enhance, and validate the Phase 1 model
  - Fidelity of the resulting Phase 2 model to be AACE Class 2
  - Model will be used to develop design of a commercial scale plant for profitable (ROI < 5-7 y) REYSc at the conclusion of Phase 2

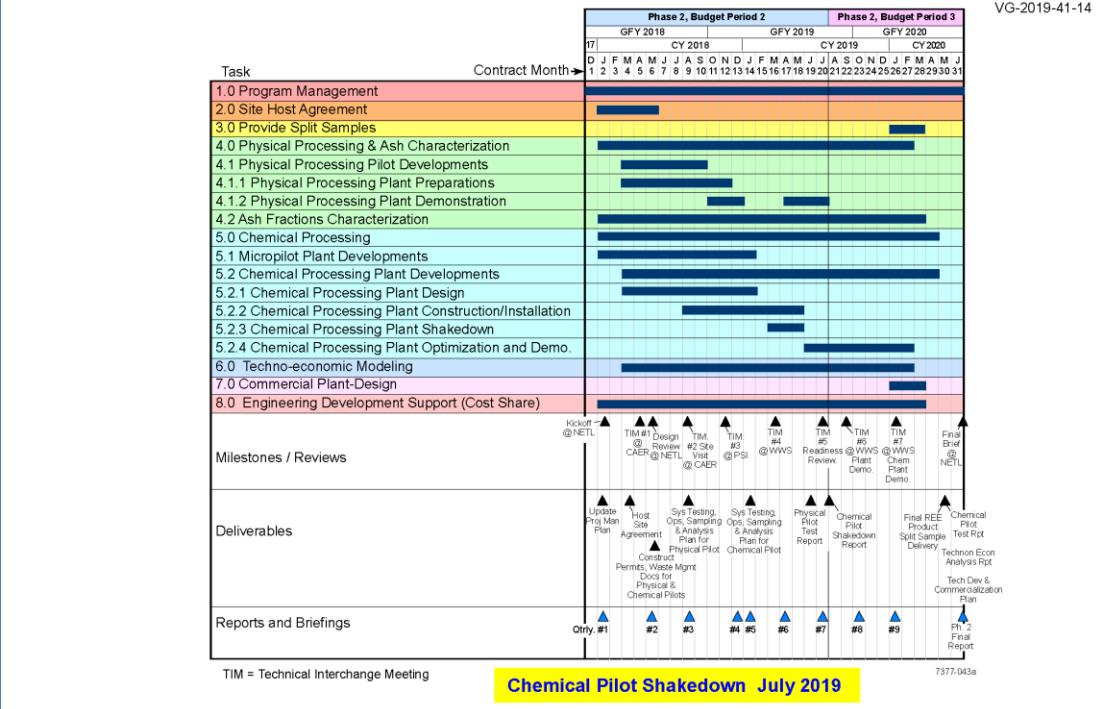


VG-2019-41

## Phase 2 Tasks, Schedule, Milestones, and Deliverables



## Phase 2 Schedule



## Phase 2 Milestones and Deliverables

VG-2019-41-15

Milestone	Program Month	Planned Completion Date
Kickoff Meeting	1	24 January 2018
Updated Project Management Plan	1	31 January 2018
Technical Interchange Meeting (TIM) #1 @CAER	3	15 March 2018
Quarterly Report #1	3	31 January 2018
Design Review @ NETL	6	04 May 2018
Host Site Agreement	6	04 May 2018
Construction, Waste Mgmt Docs for Phys & Chem Pilots	6	04 May 2018
Quarterly Report #2	6	04 May 2018
TIM #2 @ WWS (Chem Plant Site Visit)	9	10 August 2018
Quarterly Report #3	9	27 July 2018
Sys Test, Ops, Sample & Analysis Plan for Phys Pilot	10	24 August 2018
TIM #3 @ CAER (Physical Pilot Site Visit/Demo)	12	26 October 2018
Quarterly Report #4	12	29 October 2018
Sys Test, Ops, Sample & Analysis Plan for Chem Pilot	13	30 November 2018
Quarterly Report #5	15	28 January 2019
TIM #4 @ PSI (Micropilot Site Visit)	15	08 February 2019
TIM #5 @ WWS (Chemical Pilot Shakedown Tests)	18	26 April 2019
Quarterly Report #6	18	29 April 2019
Physical Pilot Test Report	19	04 June 2019
Quarterly Report #7	21	29 July 2019
TIM #6 @ NETL (Readiness Review)	22	04 September 2019
Quarterly Report #8	24	28 October 2019
TIM #7 @ WWS (Chemical Pilot Demo)	26	06 January 2020
Quarterly Report #9	27	31 January 2020
Final REYSc Product Split Sample Delivery	27	07 February 2020
Chemical Pilot Test Report	28	28 February 2020
Techno-Economic Analysis Report	28	28 February 2020
Technology Development and Commercial Plant Design	28	28 February 2020
Phase 2 Final Report	30	30 April 2020
Phase 2 Final Briefing @ NETL	30	30 April 2020



VG-2019-41

## Summary of Phase 2 Results To Date

Physical Sciences Inc.

20 New England Business Center

Andover, MA 01810



## Phase II Status

VG-2019-41-17

Performance Attributes	Commercial Target Performance Requirements	Current Status
Feedstock REYSc Content	>300 ppm (whole mass basis)	Feedstock REYSc content >500 ppm has been achieved by CAER.
Total REYSc content in final concentrate	>10 wt.% (elemental basis)	REYSc final content of 10 – 20 wt.% has been recorded at Micropilot scale. Enrichment at chemical pilot scale TBD.
Return on Investment	<7 years	Detailed economic forecasts ongoing. Cost and revenue drivers, potential plant locations, and potential suppliers and purchasers identified and quantified.
Delivered Concentrate Quantity	0.05 kg	Should be readily achievable based on chemical pilot mass balance calculations
Final REE Yield	>10 wt.%	REYSc yields of 10-15 wt.% recorded in Micropilot. Chemical pilot yields TBD.
Cement Substitute Yield	>90 wt.%	Consistent cement substitute yields of 90-93 wt.% recorded in the Micropilot. Yield at chemical pilot scale TBD.
Solvent/ Reagent Recycling	Solvent> 98.5 wt.%, Reagent>90 wt.%	Solvent recovery of ~97 wt.% & reagent recovery of 93 wt.% recorded in Micropilot. Solvent recycling efficiency expected to increase at pilot scale.

**At present, PSI expects that all target performance requirements are achievable.**



Physical Sciences Inc.

## Ash Source Selection

VG-2019-41-18

- Ash from eastern KY coal selected for Phase 2 developments
- The average REYSc content of 556 ppm measured from a composite of 20 ash samples > 300 ppm (DOE requirement)



- Example:

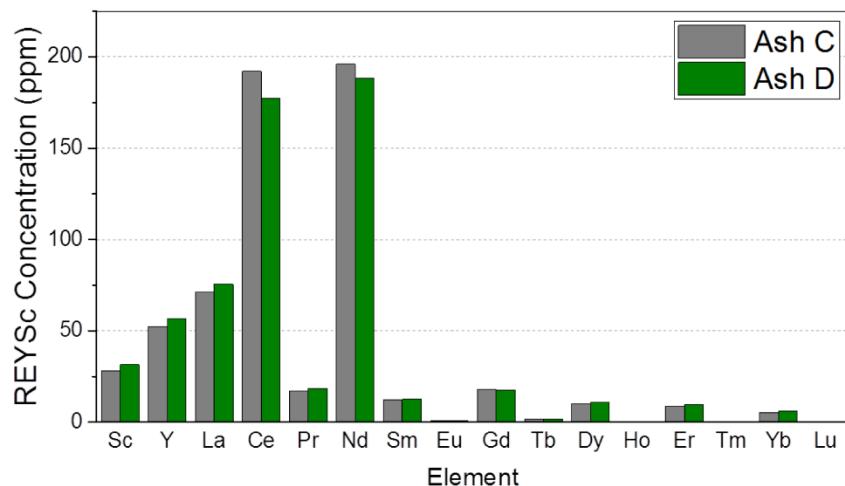
Sample	Sc	Y	$\Sigma$ REE	$\Sigma$ REY	LREE/HREE
Composite	33	59	457	516	7.46

## Individual Rare Earth Elemental Content for Selected Ash Fraction



Physical Sciences Inc.

VG-2019-41-19

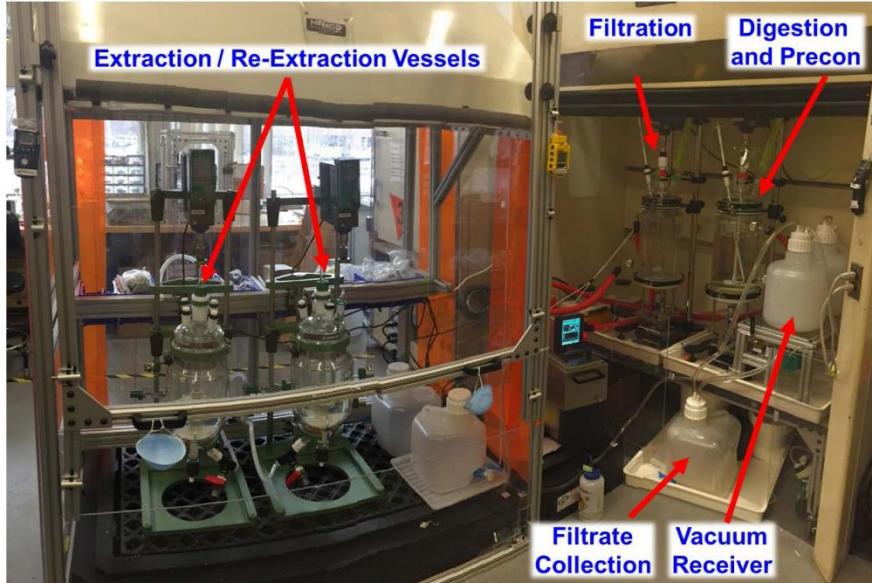


Significant content of Nd (~180 ppm), Y (~50 ppm), and Sc (~25 ppm)  
 Reasonable (~10 ppm) content of Pr, Gd, Dy



## PSI Micropilot Facility

VG-2019-41-20



**PSI Micropilot facility is operational and has demonstrated target yield and enrichment performance requirements**



## Micropilot Processing of Selected Ash Fraction

VG-2019-41-21

- Developed chemical processes for the selected ash fraction to recover REYSc with high yield and high enrichment in two final concentrate products of commercial value.
  - Concentration expressed on elemental basis i.e. the content of REY or Sc relative to that of all elements)
- **Product 1: REY-rich concentrate:**
  - REY Concentration > 15 wt%
  - REY concentration >> threshold target (10 wt%)
  - REY:Sc 100:1
  - LREE:HREE ratio = 2.3:1
- **Product 2: Sc-rich concentrate:**
  - REY:Sc 6:1
- **Process improves LREE:HREE Ratio by 2X in Product 1**
  - Starting Coal Ash LREE:HREE Ratio = 5:1



Physical Sciences Inc.

## CAER Physical Processing Pilot Plant

VG-2019-41-22



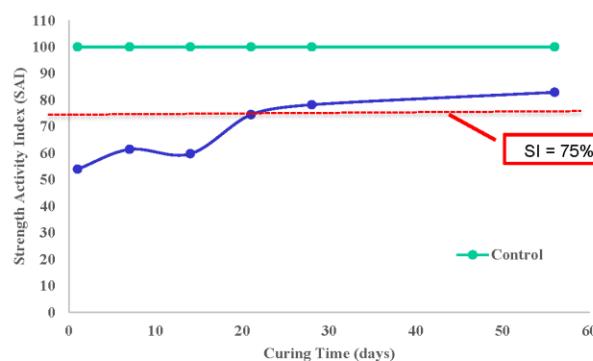
CAER physical pilot plant is operational and ~8 tons of coal ash have been processed; ~ 50% Yield for ash mass fraction for chem processing



Physical Sciences Inc.

## Pozzolanicity Testing – Strength Activity Index

VG-2019-41-23



- **Strength Activity Index or SI:** 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 at a defined level for Portland cement after a defined period of curing (Blue Line).
- SI is then compared as a ratio percent to a mortar with all Portland cement (Green Line).
- ASTM SI criterion is 75% at 7 days or 28 days (Red line). The processed fine ash utilized at 20% replacement of OPC achieved a strength index greater than 75 by 28 days of curing.



Physical Sciences Inc.

## WWS Chemical Processing Pilot Plant

VG-2019-41-24

- Detailed design completed
- Long lead time equipment has been ordered
- Infrastructure (utilities) installation in progress
- Plant assembly beginning May 2019
- Shakedown to begin July 2019



Physical Sciences Inc.

## Techno-economic Modeling Approach – Phase 1

VG-2019-41-25

- Chemical processing and economics modeled in Aspen
    - 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 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



## AACE Estimate Classes

VG-2019-41-26

ESTIMATE CLASS	MATURITY LEVEL OF PROJECT DEFINITION DELIVERABLES Express as % of complete definition	Secondary Characteristic		
		END USAGE Typical purpose of estimate	METHODOLOGY Typical estimating method	EXPECTED ACCURACY RANGE Typical variation in low and high ranges <sup>(a)</sup>
Class 5	0% to 2%	Concept screening	Capacity factored, parametric models, judgment, or analogy	L: -20% to -50% H: +30% to +100%
Class 4	1% to 15%	Study or feasibility	Equipment factored or parametric models	L: -15% to -30% H: +20% to +50%
Class 3	10% to 40%	Budget authorization or control	Semi-detailed unit costs with assembly level line items	L: -10% to -20% H: +10% to +30%
Class 2	30% to 75%	Control or bid/tender	Detailed unit cost with forced detailed take-off	L: -5% to -15% H: +5% to +20%
Class 1	65% to 100%	Check estimate or bid/tender	Detailed unit cost with detailed take-off	L: -3% to -10% H: +3% to +15%

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.

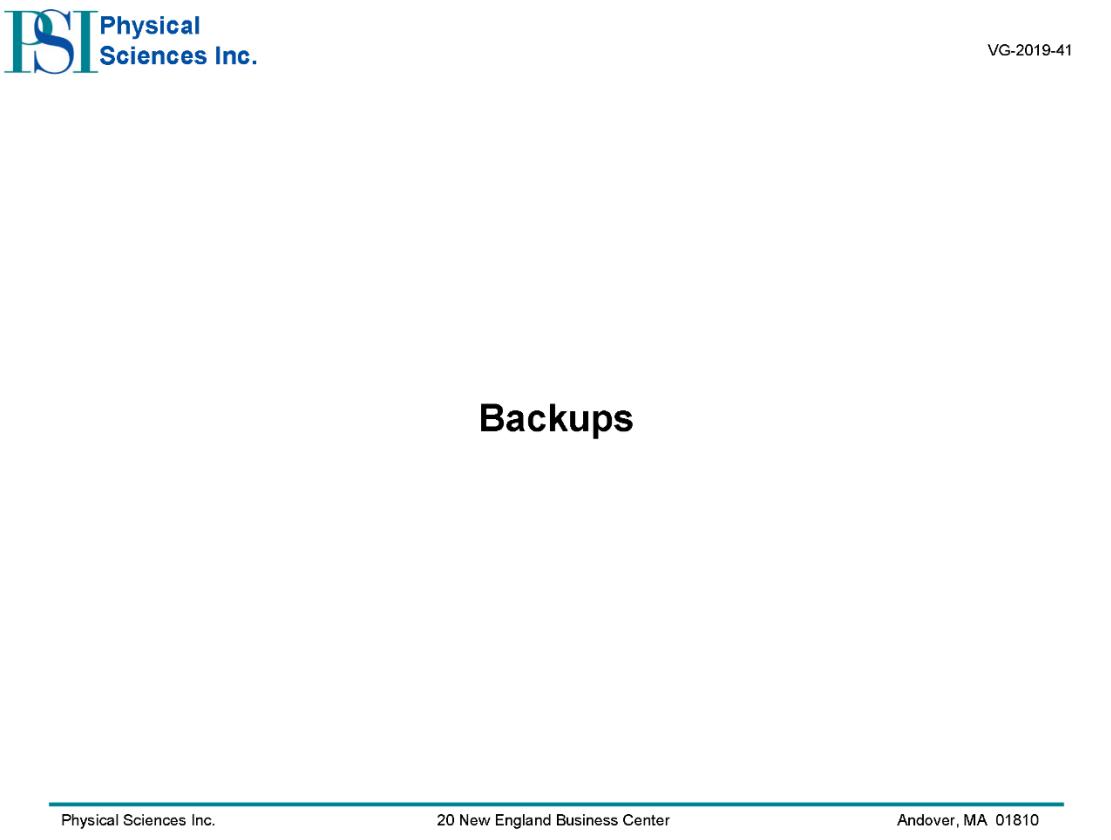
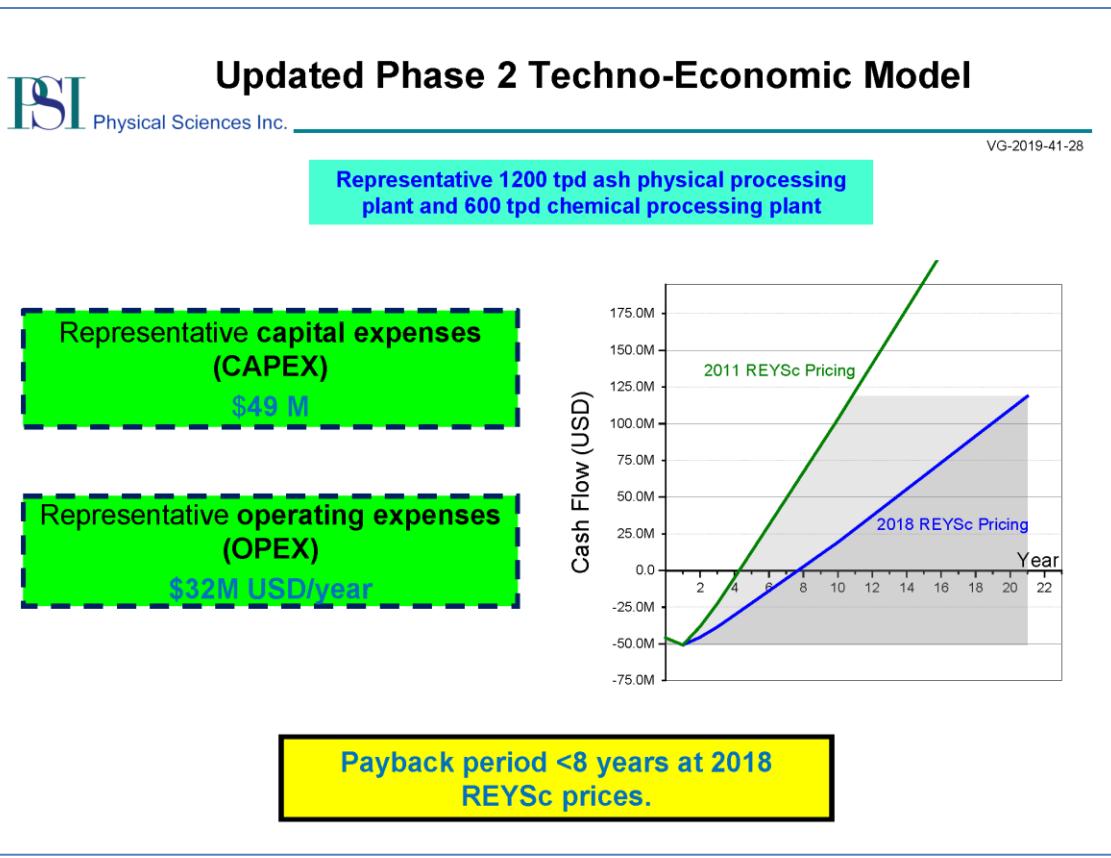


## Summary of Phase II Economic Model Updates

VG-2019-41-27

- Defined waste streams, calculated daily waste yield and determined handling/disposal costs based on vendor quotes
- Incorporated costs associated with coal ash excavation and transportation of dry nitrate to a chemical plant
- Refined material acquisition costs at scale based on vendor quotes
- Evaluated product transportation methods and associated distance-costs (truck, rail, barge)
- Defined standards for use of ash cake as a Portland cement substitute
- Defined a protocol for screening ash from a given site for PSI process suitability
- Investigated potential markets and customers for REE-rich product. Identified REE-oxides as a potential market entry point
- Phase II Model: AACE Level 2

PSI TEA is currently between AACE Class 3 and AACE Class 2 model





## Phase 2 Project Objectives - I

VG-2019-41-30

### Phase 2 Objective:

- Demonstrate the REYSc separation/enrichment technology developed in Phase 1 in a pilot scale plant with operating capacity of 0.1-1 metric ton per day (tpd)

### Specific Objectives:

1. Refine and complete detailed design of the chemical pilot plant(s) from Phase 1
  - Modular, transportable designs
2. Assemble and operate a *Micropilot (chemical) plant* for quick turnaround validation of pilot plant processing parameters, and provide data for chemical pilot plant design



## Phase 2 Project Objectives - II

VG-2019-41-31

3. Assemble pilot scale plant at CAER for physical processing of ash
4. Construct a modular pilot scale chemical plant at WWS facilities
5. Demonstrate operation of the physical pilot plant using the power plant (ash feedstock selected in Phase 1)
  - Modular, mobile CAER plant that uses the selected ash feedstock
  - Operation at ash source, decoupled from chemical pilot plant



## Phase 2 Project Objectives - III

VG-2019-41-32

- 6. Demonstrate operation of the chemical pilot plant using the selected ash fraction produced by the physical pilot plant**
  - Operation at WWS facility in Sharon, PA
    - Selected ash fraction transported to this facility from Lexington, KY
- 7. Refine and enhance the Phase 1 techno-economic model using results of above physical and chemical pilot plant operations**
  - Produce AACE Class 2 costing fidelity model in Phase 2
    - Current Phase 1 model is AACE Class 3
- 8. Develop and provide design of a commercial scale plant for profitable REYSc recovery from coal ash at Phase 2 conclusion**
  - Use the above refined Phase 2 techno-economic model
  - ROI metrics as previously stated

5. ***"High Yield and Economical Production of Rare Earth Elements from Coal Ash", Presentation to: Presentation to: Rare Earth Elements (REE) Program Portfolio, 2020 Annual Review Meeting (Virtual), 15 September 2020***

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)  
15 September 2020

**NATIONAL ENERGY TECHNOLOGY LABORATORY**  
VG-2020-105

Acknowledgement:  
This material is based upon work supported by the U.S. Department of Energy under Award DE-FE0027167.

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U.S. DEPARTMENT OF ENERGY 0

Presentation Outline

**NATIONAL ENERGY TECHNOLOGY LABORATORY**  
VG-2020-105

- Phase 2 Project Description and Objectives
- Project Updates
- Next Steps and Concluding Remarks

U.S. DEPARTMENT OF ENERGY 1

## 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 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



2

## 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



3

## 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

Performance Parameter	Threshold Value	Objective Value
Feedstock REYSc Content	>300 ppm (Whole Mass Basis)	>500 ppm (Whole Mass Basis)
Total REYSc Enrichment in Final Concentrate	>10 wt% (Elemental Basis)	>20 wt.% (Elemental Basis)
Return on Investment	<12 yr	<10 yr
Delivered Concentrate Quantity	~50 g	~0.5 kg



4

## 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



5

## Phase II Status



VG-2020-105

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

PSI team anticipates that all target performance requirements will be met.



6



VG-2020-105

## Project Update



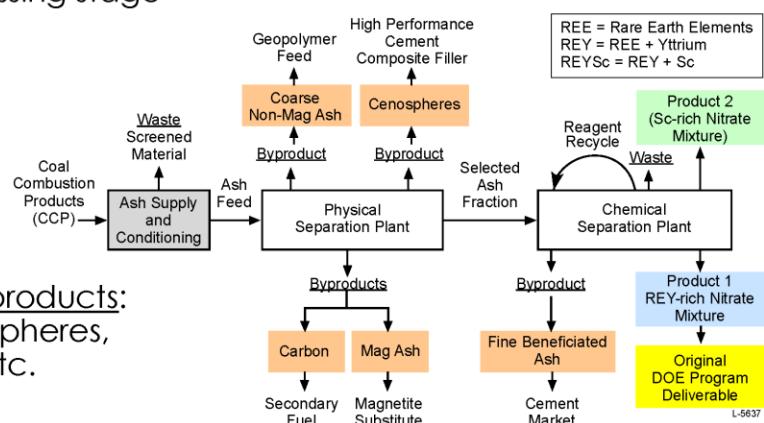
7

## Rare Earths Recovery Process Overview



VG-2020-105

- 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.



8

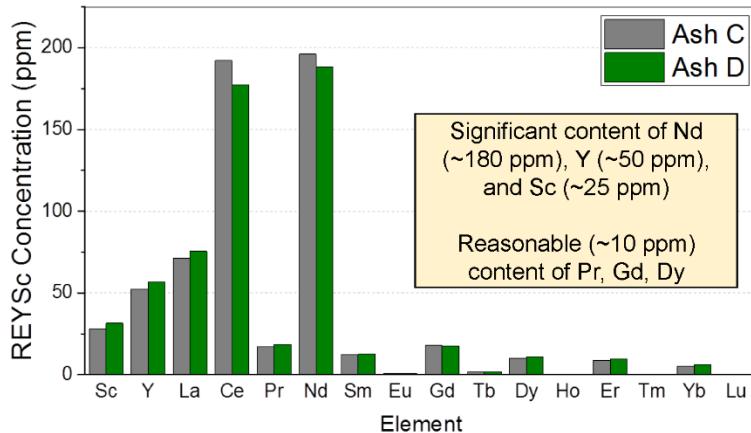
## Feed Ash Material



VG-2020-105



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



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## 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



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## Output of Physical Processing

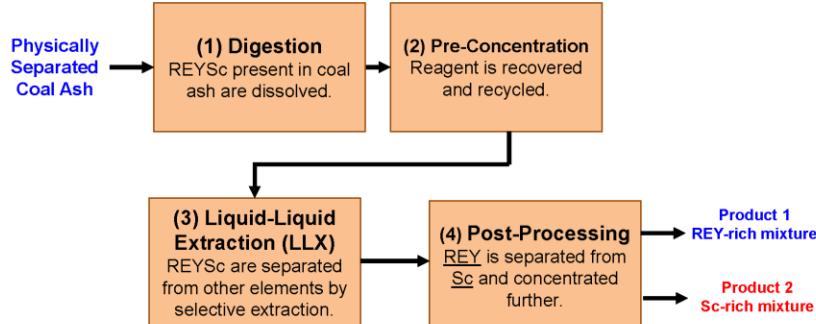


- Physical processing creates an ash that is a suitable feed to chemical pilot
  - Low carbon content
  - Low magnetics content
  - Small particle size
- Processed ash collected in super sacks and shipped to chemical pilot in Sharon, PA



11

## Chemical Processing Overview



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

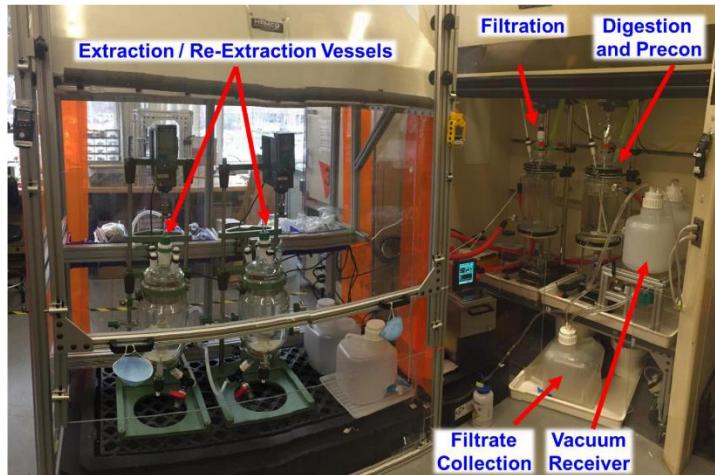


12

## PSI Micropilot Facility

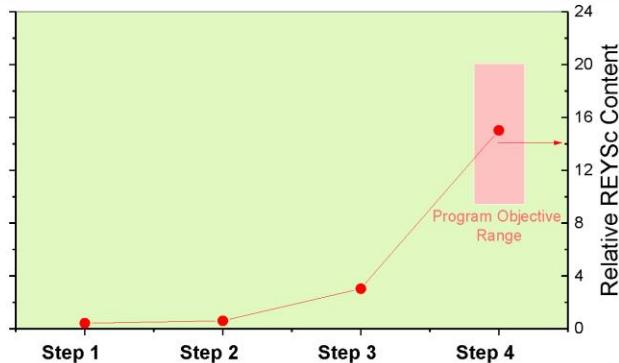


- Feed ash was first processed in PSI micropilot facility to:
  - Demonstrate target yields and enrichment
  - Determine ash suitability
  - Identify processing challenges and bottlenecks



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## 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.



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## 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

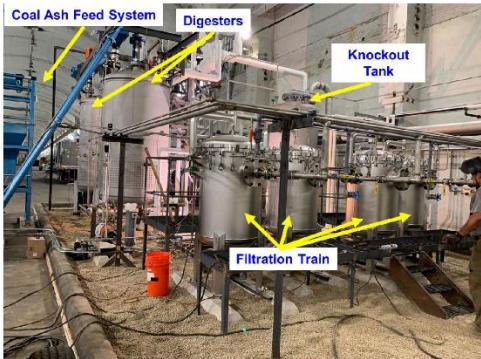


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## Hot and Cold Side Operations



VG-2020-105



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



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## Chemical Pilot Plant Status Update



VG-2020-105

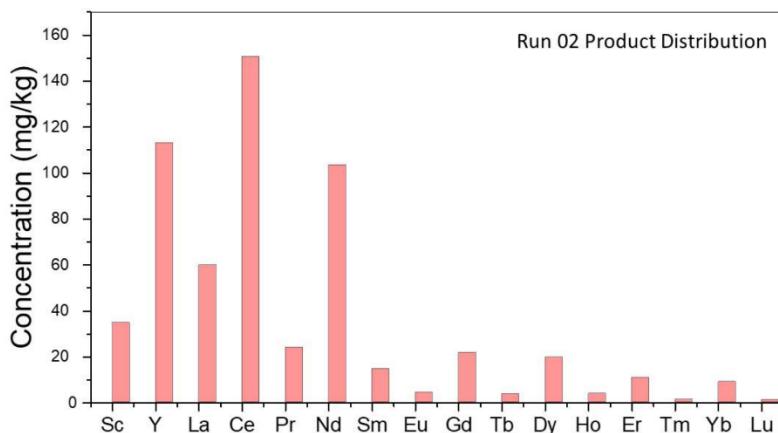
- Multiple ash batches processed from different sources (Ash C, Ash D)
- Materials sampled at various process stages analyzed via ICP-OES/MS
- Final product has REE concentration of up to 66.7% (elemental basis)
  - Further optimization expected to increase final concentrate REE content

Pilot Plant Data	Run 1	Run 2	Run 3	Run 4
REYSc Elemental Content	14%	11.9%	66.7%	29.1%
Ash	Ash D	Ash C	Ash C	Ash C



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## Dried Product Composition



Product compositions are above objective key performance parameter (>20 wt.%).  
Significant quantities of Nd, Y, Sc and HREE in product material.

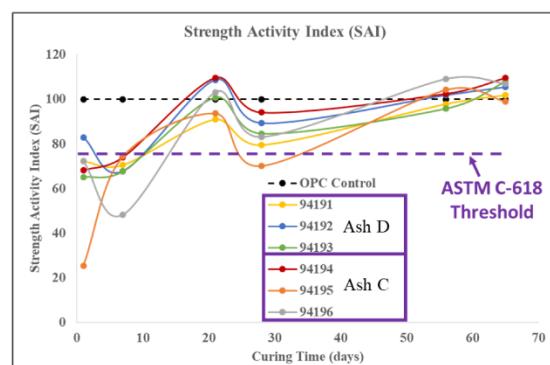


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## Pozzolanicity Testing – Strength Activity Index



- 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 (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.



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## 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



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## Capacity, Products and Annual Production



**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:

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

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

<sup>†</sup>Sc<sub>2</sub>O<sub>3</sub> market demand expected to reach 25,000 kg by 2023\*\*



21

## 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.



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## 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**



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# Thank you!



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## Backups



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## AACE Estimate Classes



ESTIMATE CLASS	MATURITY LEVEL OF PROJECT DEFINITION DELIVERABLES Express as % of complete definition	Secondary Characteristic		
		END USAGE Typical purpose of estimate	METHODOLOGY Typical estimating method	EXPECTED ACCURACY RANGE Typical variation in low and high ranges <sup>(a)</sup>
Class 5	0% to 2%	Concept screening	Capacity factored, parametric models, judgment, or analogy	L: -20% to -50% H: +30% to +100%
Class 4	1% to 15%	Study or feasibility	Equipment factored or parametric models	L: -15% to -30% H: +20% to +50%
Class 3	10% to 40%	Budget authorization or control	Semi-detailed unit costs with assembly level line items	L: -10% to -20% H: +10% to +30%
Class 2	30% to 75%	Control or bid/tender	Detailed unit cost with forced detailed take-off	L: -5% to -15% H: +5% to +20%
Class 1	65% to 100%	Check estimate or bid/tender	Detailed unit cost with detailed take-off	L: -3% to -10% H: +3% to +15%

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.



6. *"High Yield and Economical Production of Rare Earth Elements from Coal Ash", Presentation to: Presentation to: Rare Earth Elements (REE) Program Portfolio, 2021 Annual Review Meeting (Virtual), 25 May 2021*



VG-2021-081

## 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

#### Acknowledgement:

This material is based upon work supported by the U.S. Department of Energy under Award DE FE0027167.

#### Disclaimer:

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Physical Sciences Inc.

20 New England Business Center

Andover, MA 01810



## Presentation Outline

VG-2021-081-1

- **Phase 2 Project Description and Objectives**
- **Project Updates**
- **Next Steps and Concluding Remarks**



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## Phase II Team

VG-2021-081-2

- **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 Gamlie: 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 Hover: Coal Geochemistry, Ash Source Selection, Materials Characterization
    - Dr. John Groppe: 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



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VG-2021-081-3

## 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

VG-2021-081-4

- **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)

Performance Parameter		Threshold Value	Objective Value
Feedstock REY+Sc Content		>300 ppm	>500 ppm
Return on Investment		< 12 years	< 10 years
REY-enriched Product	Quantity (REY salts)	100 g	300 g
	REY-enriched Oxide Purity (total REY content - elemental basis)	>85%	>90%
Sc-enriched Product	Quantity (Salt/Oxide)	1 g	2 g
	Sc-enriched Oxide/Salt Purity (Sc content - elemental basis)	>85%	>90%
Aluminum Product	Quantity (oxide type material)	100 g	300 g
	Purity (Al content elemental basis)	>50%	>68%



## Current Program Status

VG-2021-081-5

- **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



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## Phase 2 Status

VG-2021-081-6

Performance Attributes	Commercial Target Performance Requirements	Current Status
Feedstock REYSc Content	>300 ppm (whole mass basis)	Feedstock REYSc content >500 ppm has been achieved
Total REYSc content in final concentrate	>10 wt.% (elemental basis)	REYSc final content of 10 – 60 wt.% has been recorded at pilot scale
Delivered Concentrate Quantity	0.05 kg	Achieved ~0.5 kg of concentrate production to date. Delivered 51g of concentrate to DOE with >50% REYSc content
Final REE Yield	>10 wt.%	REYSc yields of 10-30 wt.% recorded in micropilot and chemical pilot.
Cement Substitute Yield	>90 wt.%	Yields of 90-93 wt.% recorded in the micropilot and pilot scale. Cement substitute utility confirmed via standardized testing.
Solvent/ Reagent Recycling	Solvent >98.5 wt.% Reagent >90 wt.%	Solvent recovery of ~97 wt.% & reagent recovery of 93 wt.% recorded in micropilot. Solvent recycling efficiency confirmed at pilot scale.
REY-product	>85 wt.% (elemental basis)	Initial Phase 2 REYSc product: ~10-68 wt.% (elemental basis) Identified pathways to increase purity to > 85 wt.%. Optimization and scale-up ongoing.
Sc-product	>85 wt. % (elemental basis)	12X enrichment obtained in a single cycle using a PSI-proprietary LLX process Multiple cycles projected to achieve target purity (>85 wt.%)
Al-product	>56 wt.% (elemental basis)	Al-product of 68-90 wt.% purity obtained on multiple runs

PSI team anticipates that all target performance requirements will be met



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VG-2021-081-7

## Project Update

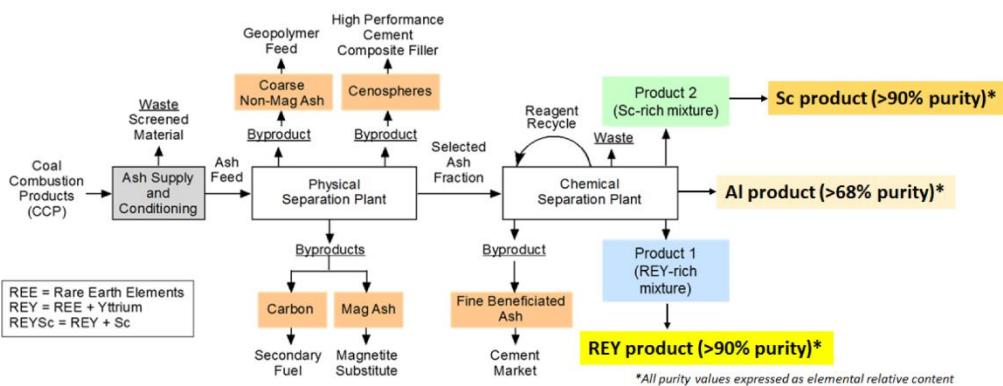


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VG-2021-081-8

## 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.



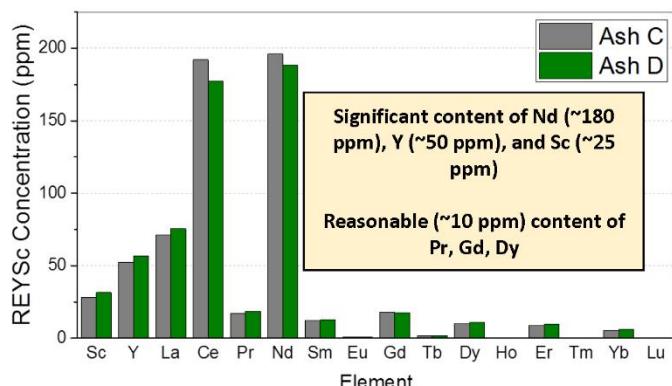
Physical Sciences Inc.

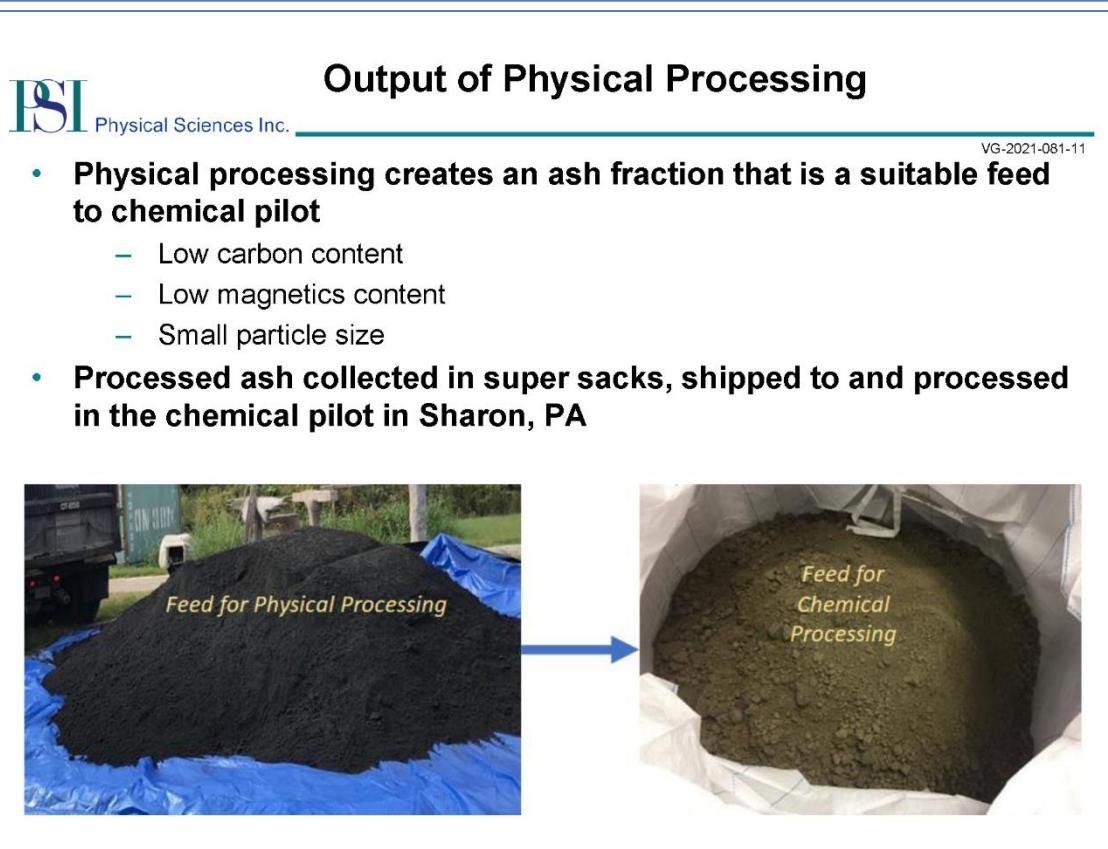
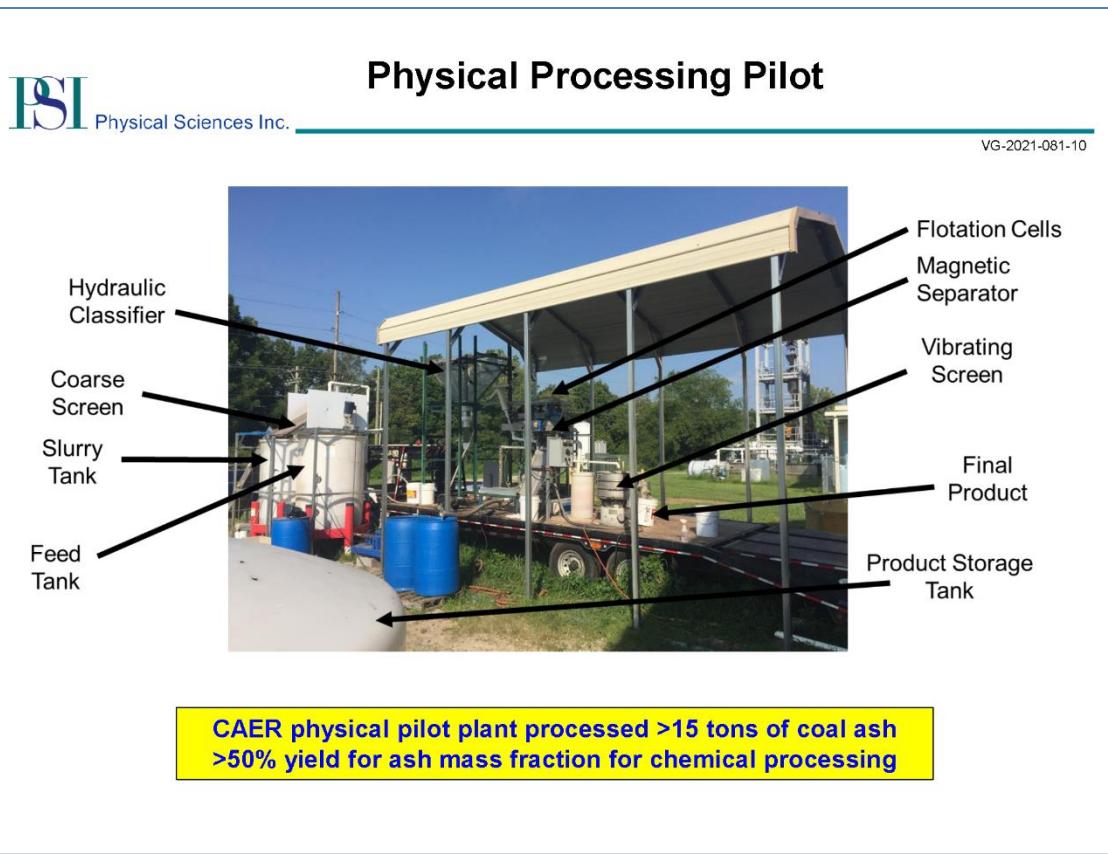
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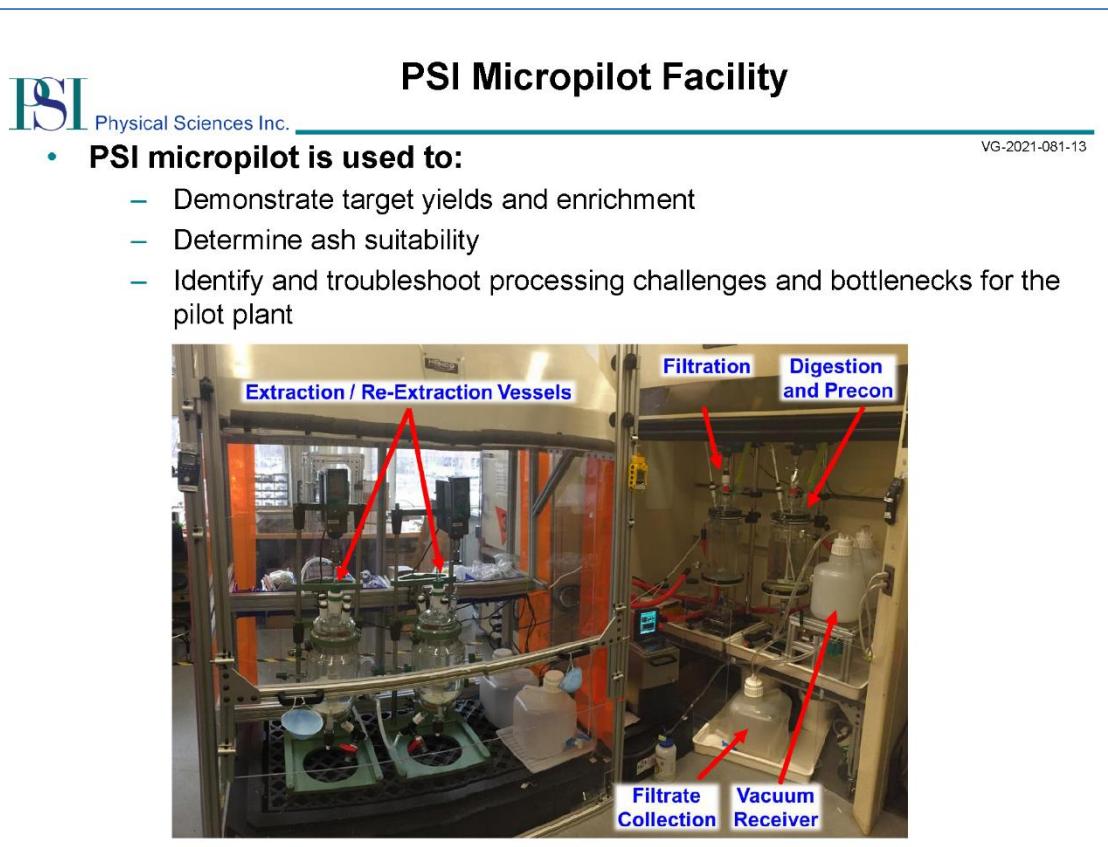
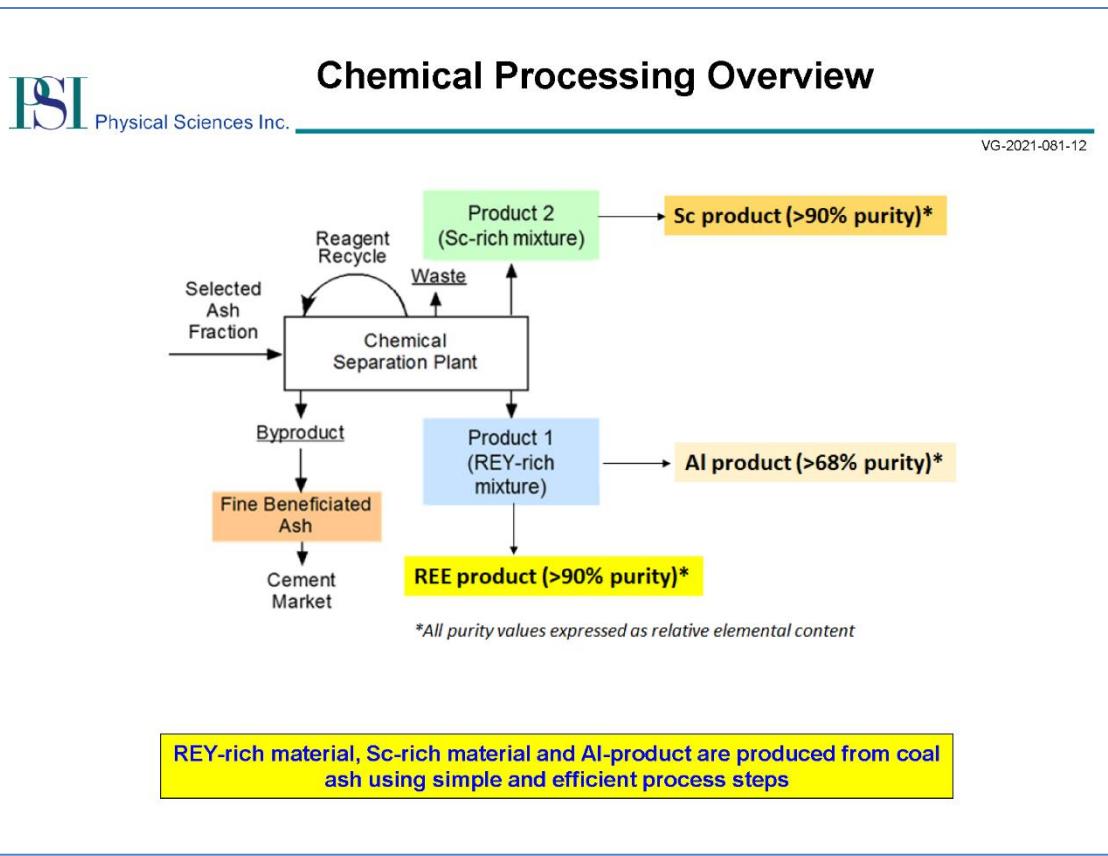
## Feed Ash Material



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







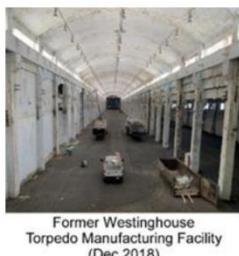


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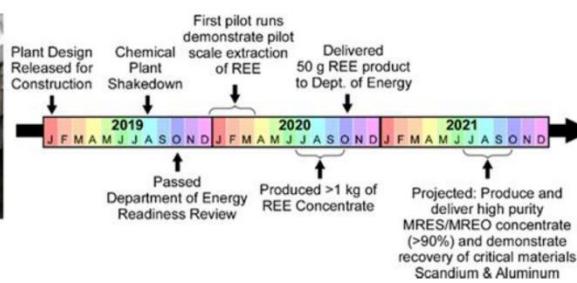
## Chemical Pilot Operations

VG-2021-081-14

- 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



Former Westinghouse  
Torpedo Manufacturing Facility  
(Dec 2018)



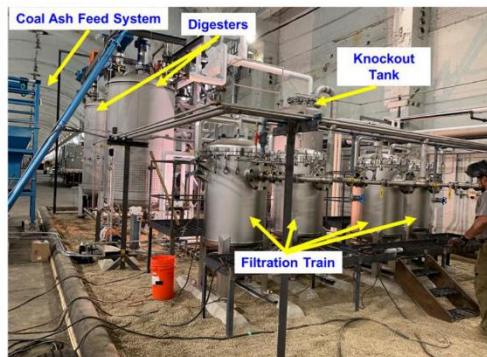
REE Recovery Pilot Plant  
(Jan 2021)



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## Hot and Cold Side Operations

VG-2021-081-15



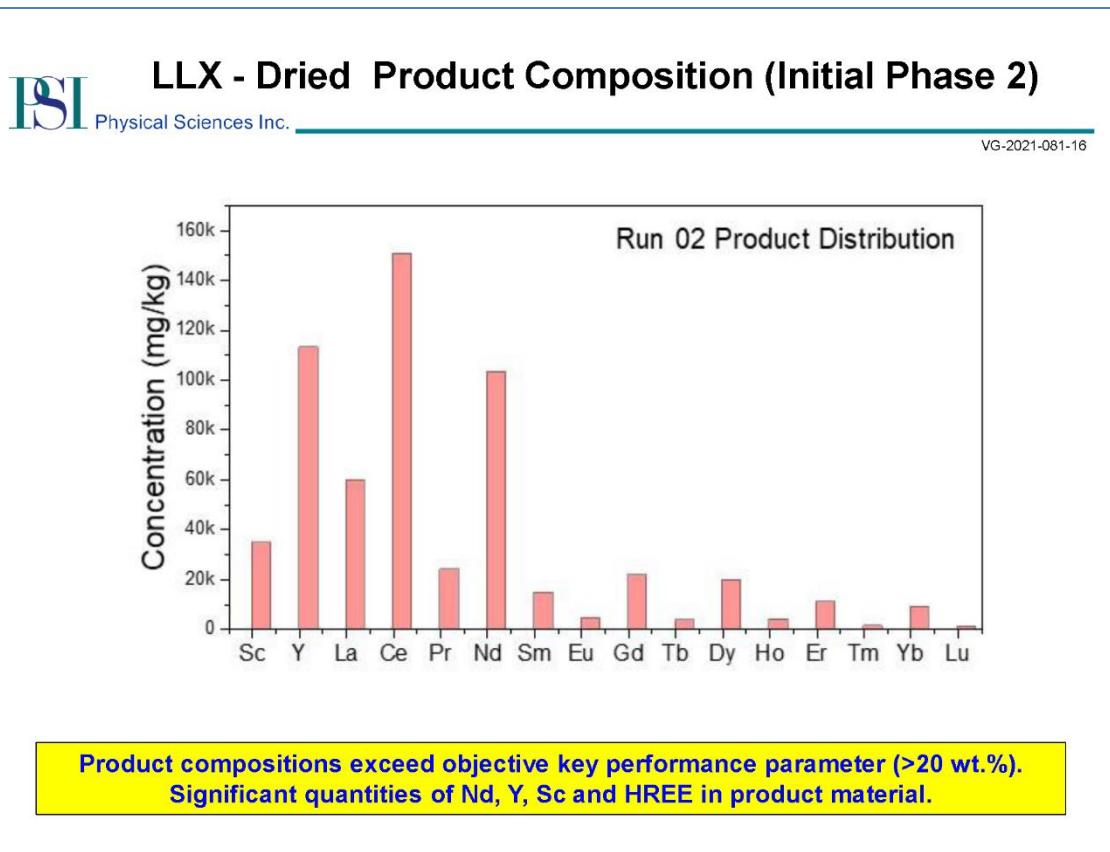
Hot Side Operation



Cold Side Operation

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



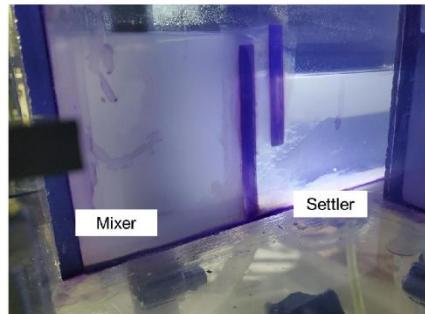
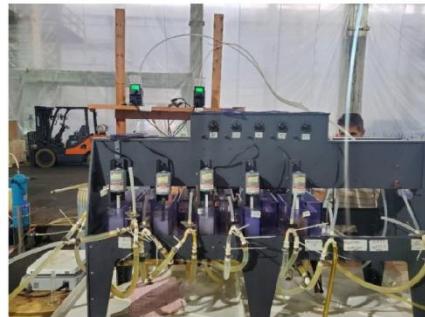
## LLX Optimization Approach



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VG-2021-081-18

- **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



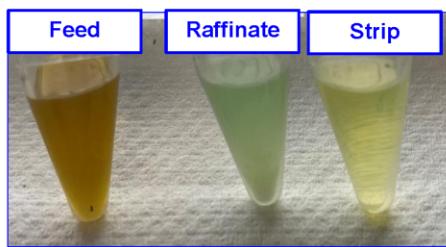
## Sc Recovery Summary

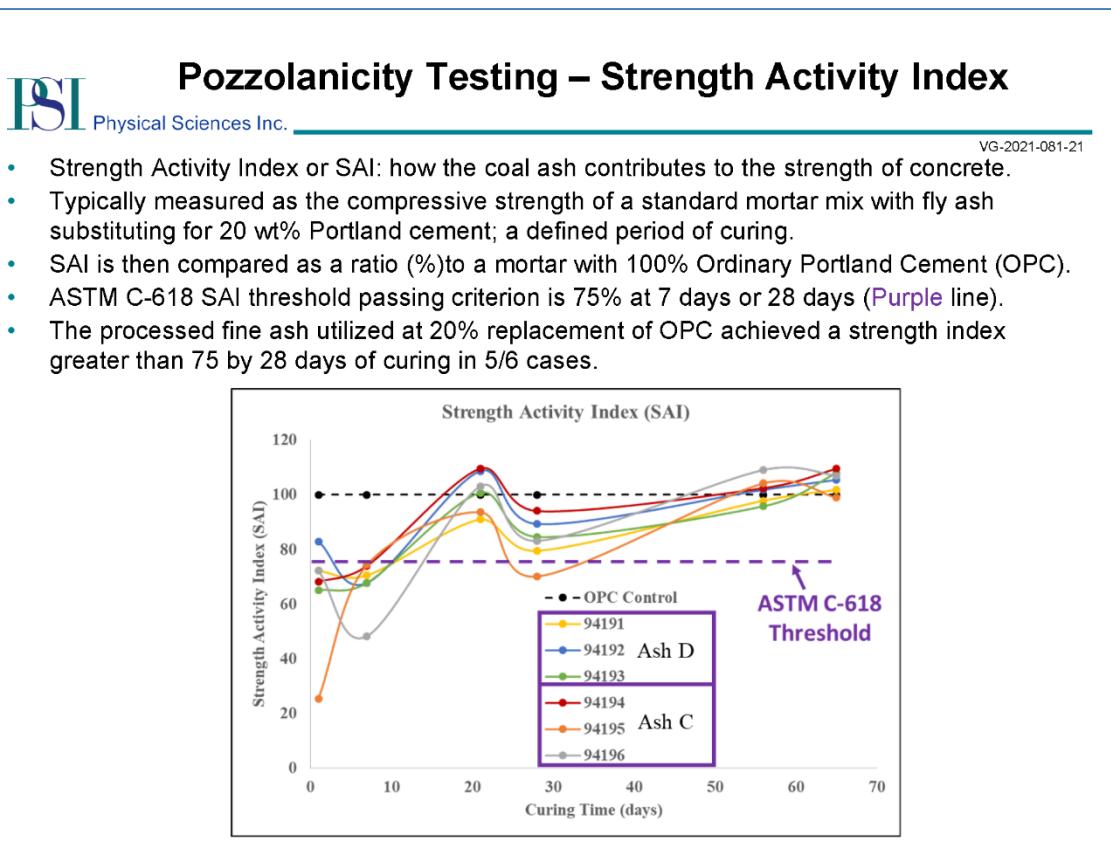
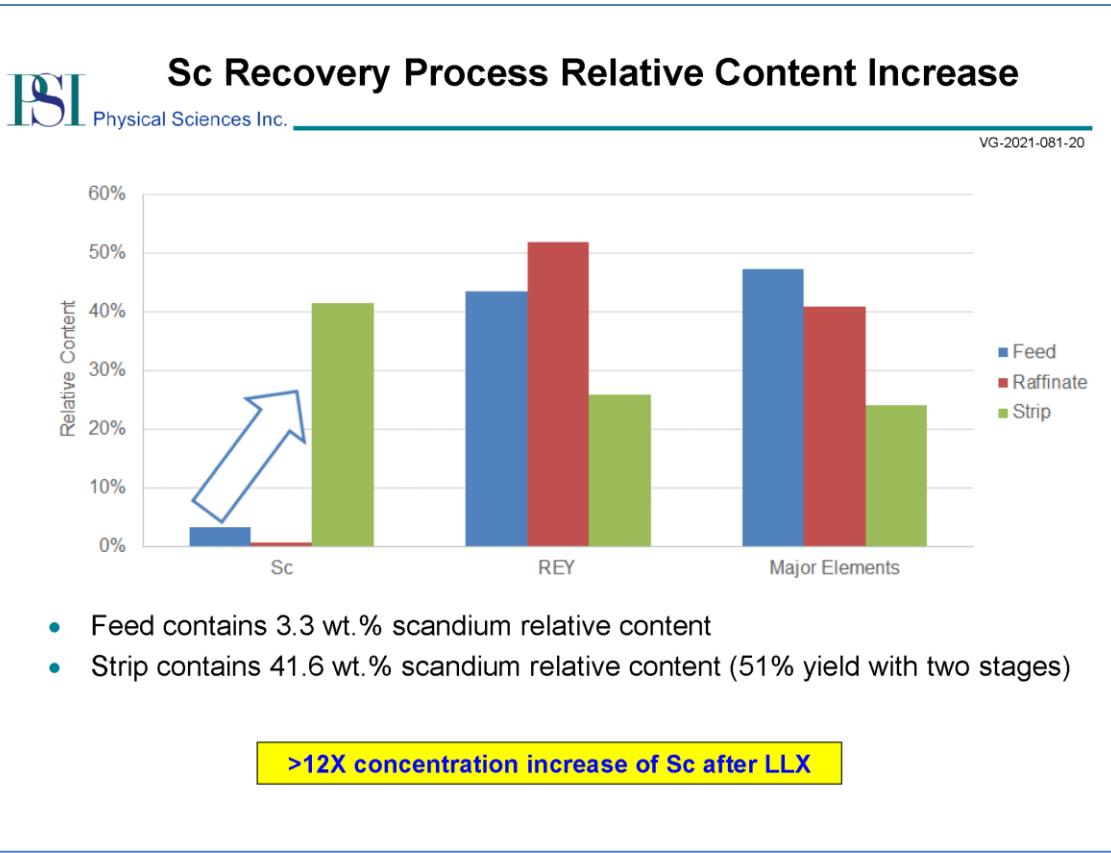


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VG-2021-081-19

- **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





## Roadmap: Chemical Plant Development Path

VG-2021-081-22

L-6146

- **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

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VG-2021-081-23

- **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:

Component	Quantity Produced*	Portion of Revenue (%)		Worldwide Market tons/year	Market Application
		2018 REE Pricing	2011 REE Pricing		
REEs	38.2	<b>1.0</b>	<b>10.8</b>	170K	Batteries, Magnets, Alloys, Catalysts
Scandium	5.8 <sup>†</sup>	<b>26.1</b>	<b>35.0</b>	10-15	Alloys, Catalysts
Carbon	96K	<b>6.2</b>	<b>4.6</b>		Low-grade Fuel
Magnetic	20K	<b>5.4</b>	<b>4.0</b>		Magnetite Substitute
Non-Magnetic >200 Mesh	48K	<b>1.0</b>	<b>0.8</b>		Geopolymer Feed
Non-Magnetic <200 Mesh	186K	<b>23.9</b>	<b>17.8</b>	71.8M	Cement Substitute (Pozzolan)
Cenosphere Product	2K	<b>36.4</b>	<b>27.1</b>	~51K	Concrete Additive

<sup>†</sup>Sc<sub>2</sub>O<sub>3</sub> market demand expected to reach 25,000 kg by 2023

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



Physical Sciences Inc.

## Preparing Project for Next Steps

VG-2021-081-24

- 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.

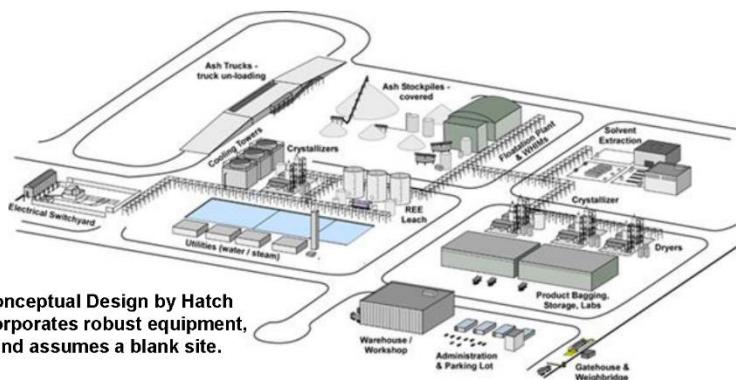
Project will result in a technology that will domestic supply of REY-rich concentrate for downstream separation and refinement into individual REE, as well as critical materials (Sc, Al)

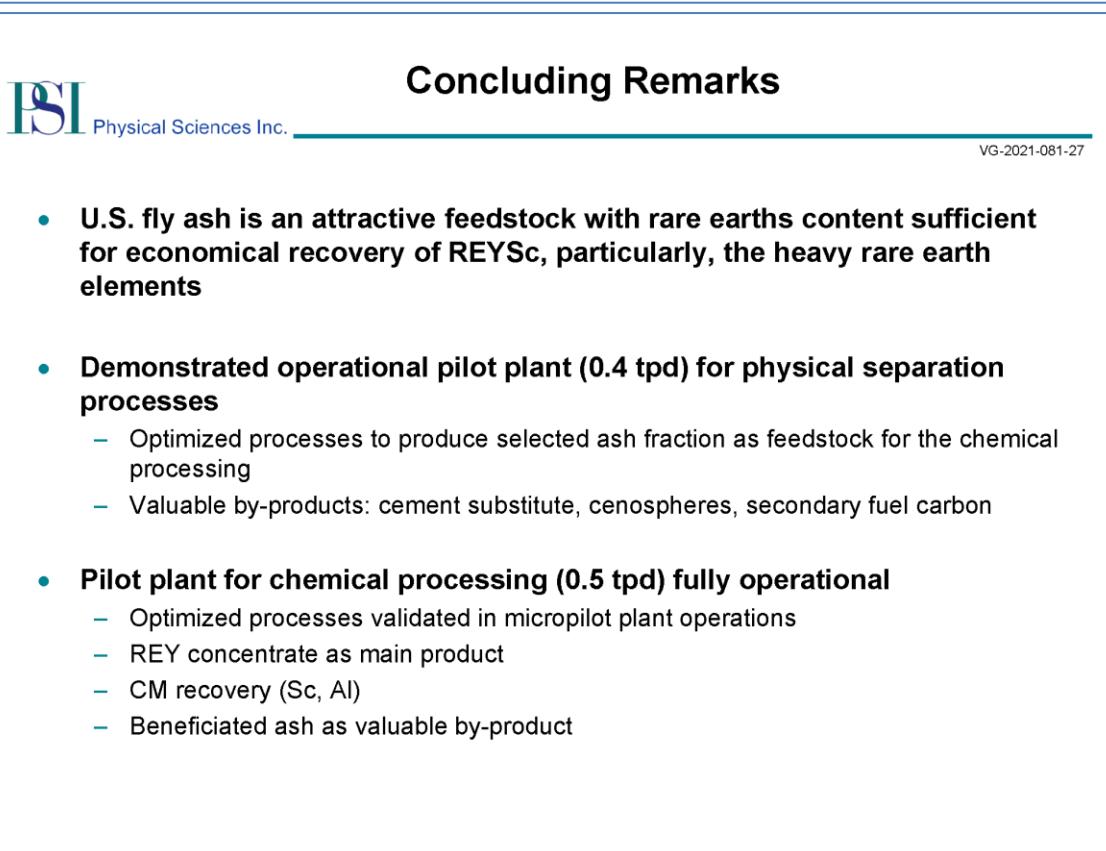
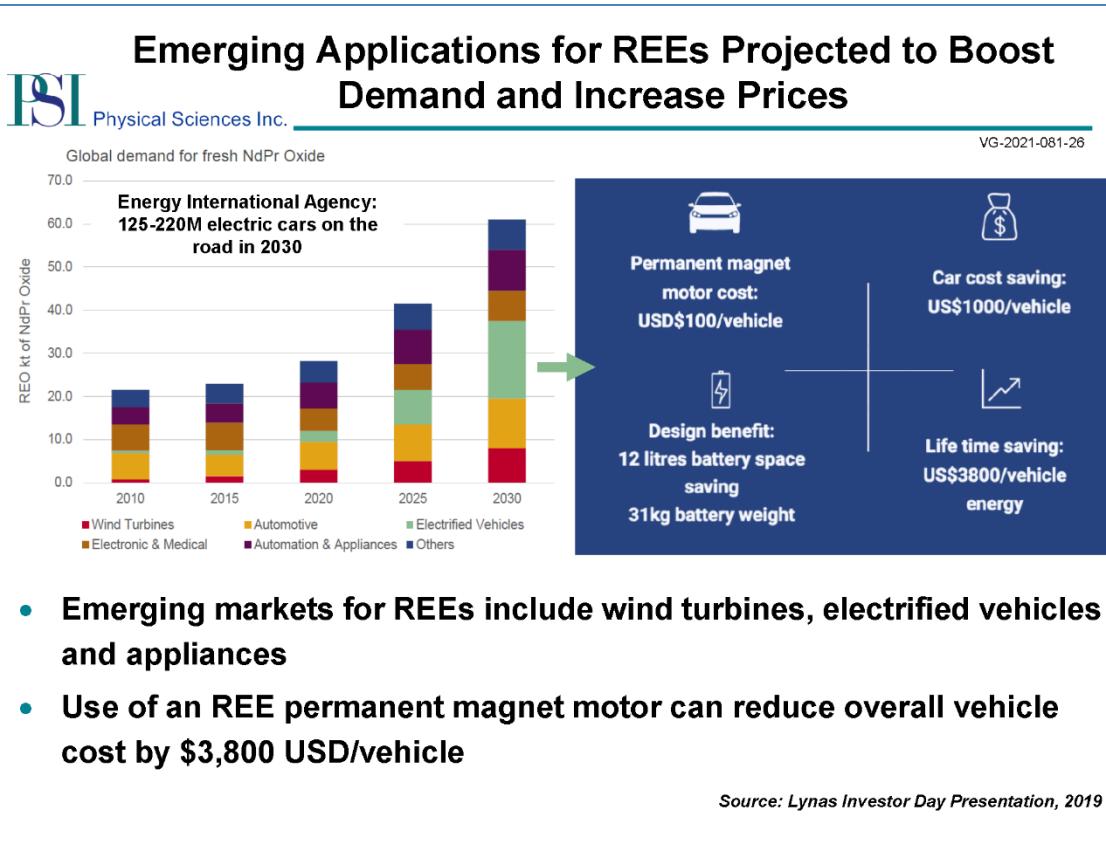


## REE Recovery from Coal Ash Plant Conceptual Design for Commercial Scale REE Concentrate Production

VG-2021-081-25

- 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







**Thank You!**

VG-2021-081-28

