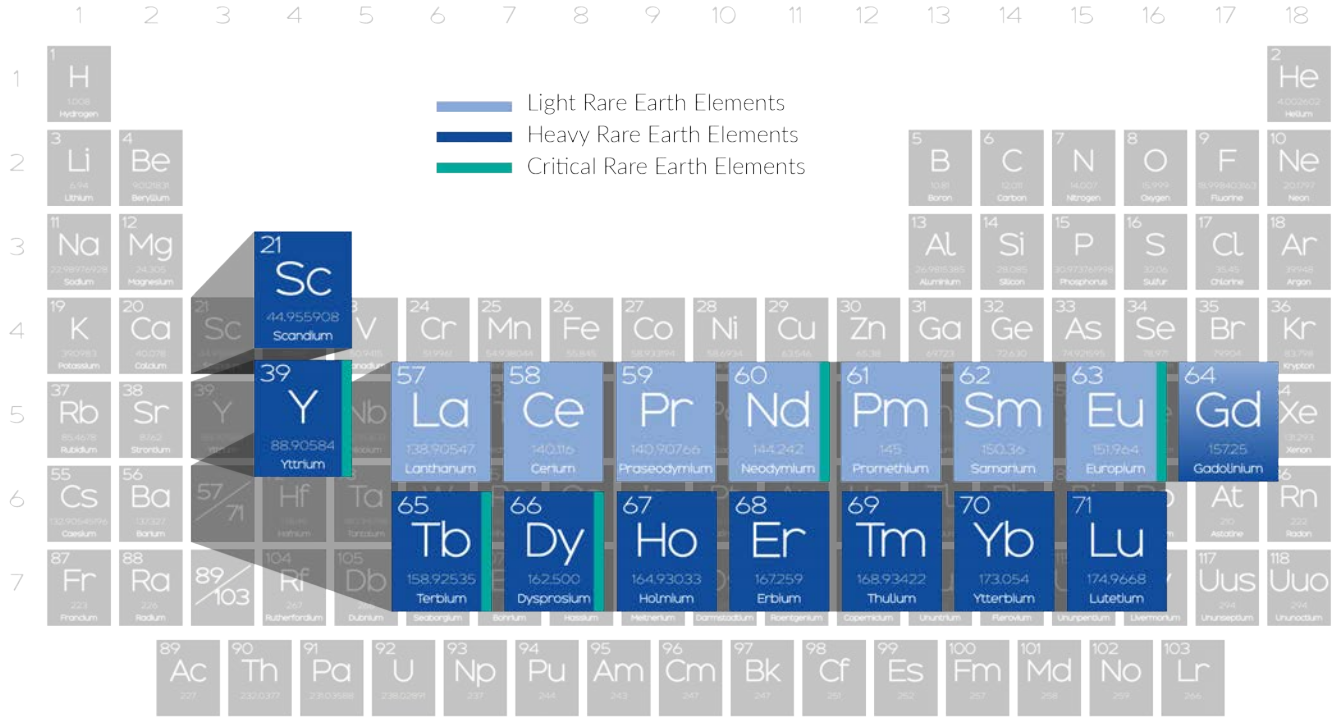
NATIONAL ENERGY TECHNOLOGY LABORATORY

RARE EARTH ELEMENTS



2019 PROJECT PORTFOLIO

U.S. DEPARTMENT OF ENERGY | the **ENERGY** lab
National Energy Technology Laboratory

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Contents

Introduction

BACKGROUND

The U.S. Department of Energy (DOE) Office of Fossil Energy conducts programs to ensure the availability of ultraclean (near-zero emissions), abundant, low-cost domestic energy from coal. These efforts are designed to fuel economic prosperity, strengthen energy independence, and enhance environmental quality. As the Office of Fossil Energy's research laboratory, the National Energy Technology Laboratory (NETL) is engaged in research, development, and demonstration (RD&D) activities to create technology and technology-based policy options for public benefit.

As part of its RD&D technology portfolio, NETL has initiated the Rare Earth Elements from Coal and Coal By-Products RD&D Program, which focuses on developing rare earth element (REE) separation and recovery technologies, addressing the current global REE separations market and process economics, and demonstrating environmentally benign REE separation processing capabilities. The quantities of REEs in our nation's vast coal resources offer the potential to reduce U.S. dependence on foreign sources for these critical materials, and to create new industries.

PROGRAM OBJECTIVES

The overall objectives of NETL's REE program are to demonstrate the techno-economic feasibility and performance of existing commercial or newly developed REE separation technologies. These technologies are focused on separating and recovering REEs from coal and coal by-products containing a minimum of 300 ppm total REEs, and concentrating the REEs to levels greater than or equal to 2 percent by weight, tentatively producing 90 to 99.99 percent high-purity, salable, individual rare earth metal oxides. These objectives will be accomplished through laboratory REE separation projects and demonstration of concept feasibility at bench- scale through pilot-scale facilities and integrated processing systems. Ultimately, these efforts will ready REE separations technology for commercial deployment. Key success factors for this program include co-production of materials and critical elements, successful demonstration of environmentally benign processing, and competitive economics.

KEY TECHNOLOGY AREAS

The REE Program consists of three key technology areas: Enabling Technologies, Separations Technologies, and Process Systems. The figure on page 7 shows how the key technology areas relate to the program efforts.

1. Enabling Technologies include resource identification, sampling, and characterization; techno-economic analysis; and field/process sensor development.

Significant progress in locating field sites and assessing the composition of potential coal and coal by-product materials containing REEs has been made. Efforts continue to focus on identifying the best source of materials to support future commercial REE production. Chemical and physical characterization efforts to determine REE elemental concentrations and phase compositions in coal and coal by-product resources are essential for developing viable REE separation processes.

Techno-economic analyses are being conducted to evaluate the international REE market demand and to assess the economics of commercially producing REEs from conventional separation

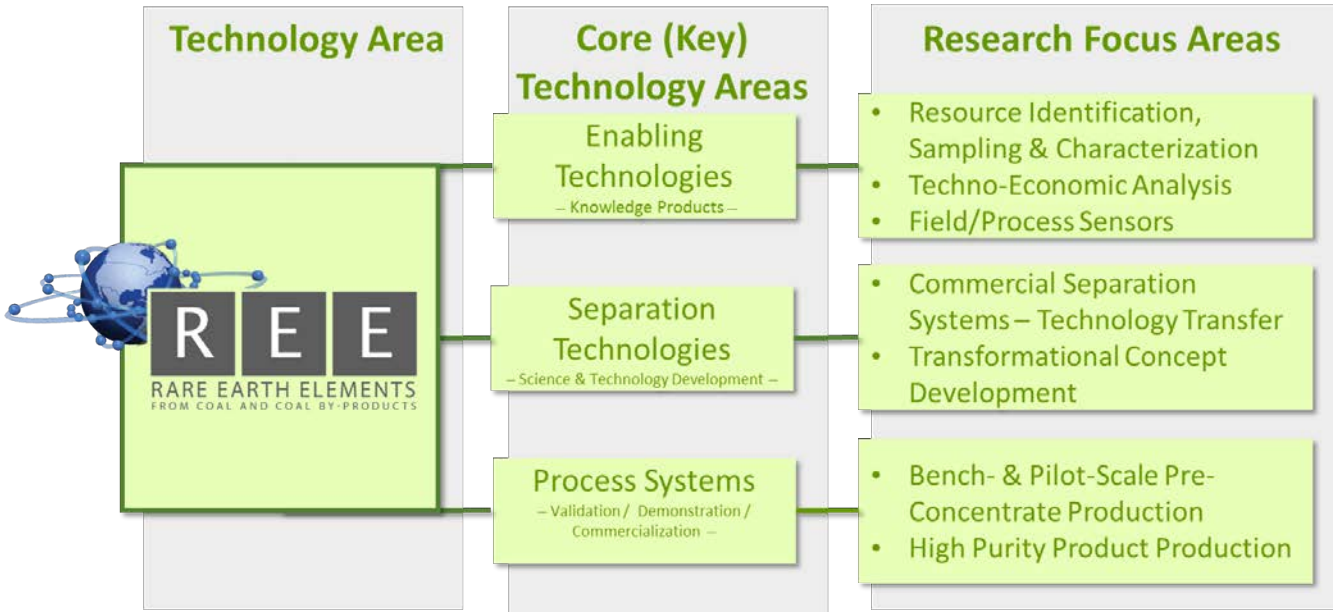
processes as well as potentially new and advanced transformational separation processes. A preliminary high-level REE jobs analysis is being developed that includes an estimation of the economic impacts of constructing and operating a REE separations and processing facility (or facilities) in the United States.

Portable sensors for identifying promising coal-based REE resources in the field, as well as devices for determining REE concentrations in process separation flow streams, are being developed and evaluated. These technologies are tentatively scheduled to be field-tested at bench-scale separations test facilities and validated to commercial-ready status during use in pilot-scale demonstration projects. While significant progress has been made in identifying field site locations and assessing the composition of potential REE-containing coal and coal by-product materials, continued effort is essential to identify the best source of materials to support future commercial REE production. Chemical and physical characterization efforts addressing REE elemental concentrations and phase compositions in the coal and coal by-product resources are essential for developing viable REE separation processes.

2. Separation Technologies include utilization or modification of current commercial physical separation systems (i.e., beneficiation via size, density, froth flotation, magnetic, ultrasound), hydrometallurgy and solvent extraction/digestion processes, and pyrometallurgy techniques (e.g., electro-slag refining, acid roasting) to separate and concentrate REEs from coal-based resources such as coal, coal refuse, clay/sandstone over/under-burden materials, aqueous effluents, acid mine drainage sludge, and power generation ash. Advanced or new transformational REE separation concepts such as physical, chemical, electrical, and thermal extraction, acid/base leaching, and ion exchange; reactive grinding; photochemical, ultrasonic-assisted, microwave-aided, photophoretic, plasma, and supercritical CO_2 separation; and advanced sorbents and membrane systems are being considered to further enhance REE separation.

3. Process Systems development, demonstration, and commercialization include validating the performance of bench- and pilot-scale technologies as well as determining the economic feasibility of separating REEs from coal and coal-related resources containing a minimum of 300 ppm REEs and concentrating them to an initial pre-concentrate matrix of greater than or equal to 2 percent by weight, with further integration of additional extraction systems that could help generate high-purity, salable rare earth compounds (such as oxides and/or metals, carbonates, etc.).

Ultimately, domestic generation of high-purity REEs may lead to the manufacture of REE-containing products in the United States, thus enhancing our national security and stimulating economic growth. Domestic production of REEs will provide secure access for industries involved in defense and national security, energy, medicine, communications, consumer electronics, and others. In addition, an available domestic supply of REEs may lead to commercialization of new and advanced materials and equipment.



REE Program Structure: Core Technology Areas

Rare Earth Elements – Small Pilot, High Purity, REE Separation Systems

Products from Coal and Coal Byproducts in the U.S. Using Advanced Separation Processes

Technology Partner: Marshall Miller & Associates, Inc.

Award Number: FE0029956

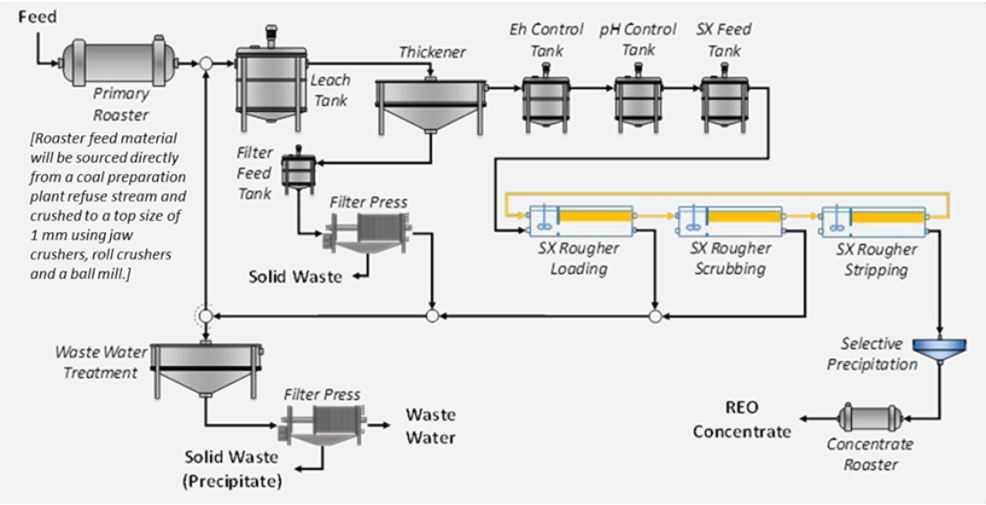
Project Duration: 09/01/2017 – 05/31/2019

Total Project Value: $1,250,000

Key Technology Area: Process Systems

Project Partners: Cumberland Mine Services, Kentucky River Properties, Minerals Refining Company, Outotec USA, University of Kentucky, and Virginia Polytechnic Institute and State University

This Phase 1 project will identify and characterize coal-related materials and design and perform a techno-economic analysis for a commercially viable technology to produce salable quantities and purities of rare earth elements (REEs) from coal-based feedstocks. The facility, which will be physically located in Appalachia, will utilize interchangeable modules that can be easily reconfigured to accommodate changing feedstocks and technology upgrades. The process includes both physical and chemical extraction processes and is expected to produce at least 10 pounds per day of rare earth oxides at purities of 90-99 percent. As part of the feasibility analysis, laboratory testing will be performed, including crushing/grinding, magnetic concentration, flotation release analysis, selective agglomeration, leachability/ion exchangeability, solvent extraction, mechanical dewatering, and solids thickening. Data obtained from the laboratory testing will be used to design a pilot facility and optimize REE recovery and process costs. Overall, this feasibility study will provide critical insight into the economic parameters and commercial viability of the integrated process system. In addition to the REE products, the facility will also co-produce a salable ultraclean coal by-product that can help to offset the cost of the facility and enhance long-term economic viability. If successful, this Phase 1 project may continue into the next stage of development, which will include installation, field-testing, and evaluation of the REE recovery technology. Successful completion of this project is expected to provide a first-generation production facility for recovering rare earth products from coal and coal by-product streams in a manner that is cost-effective and environmentally acceptable. This outcome will provide a secure domestic feedstock supply of REEs and maintain or create additional coal-related jobs.



Process block diagram for the REE recovery facility.

Recovery of Rare Earth Elements from Coal Mining Waste Materials

Technology Partner: Inventure Renewables, Inc.

Award Number: FE0030146

Project Duration: 09/01/2017 – 05/31/2019

Total Project Value: $1,250,000

Key Technology Area: Process Systems

Project Partners: K-Technologies, Inc.; Pennsylvania State University; and Texas Minerals Resources Corporation

This Phase 1 project will identify and characterize coal-related materials, and design and perform a techno-economic analysis for a self-contained, modular, and portable continuous ion exchange/continuous ion chromatography (CIX/CIC) pilot plant capable of processing and purifying rare earth elements (REEs). The process will combine chemical processing with physical beneficiation processes for providing a concentrated REEs leach liquor stream derived from clay-rich, co-produced coal materials such as those associated with overburden, and clay layers under or within a coal seam. After concentration, the REEs will be separated and purified into their individual elements using CIX/CIC techniques. The project will also determine the economic viability of mining and processing REEs associated with Appalachian coal deposits. If successful, this Phase 1 project may continue into the next stage of development, which includes installation, field-testing, and evaluation of the REE recovery technology. The expected outcomes of the project are (1) the production of REEs that are salable at an output of 10 pounds per day from coal by-products, (2) the development of a technology that is versatile and modular, (3) the preservation of mining jobs, and (4) a pathway to economic feasibility.

Continuous ion exchange pilot system.

Rare Earth Elements – Conventional REE Separation Systems

Recovery of Rare Earth Elements from Coal Mine Drainage

Technology Partner: West Virginia University Research Corporation

Award Number: FE0026927

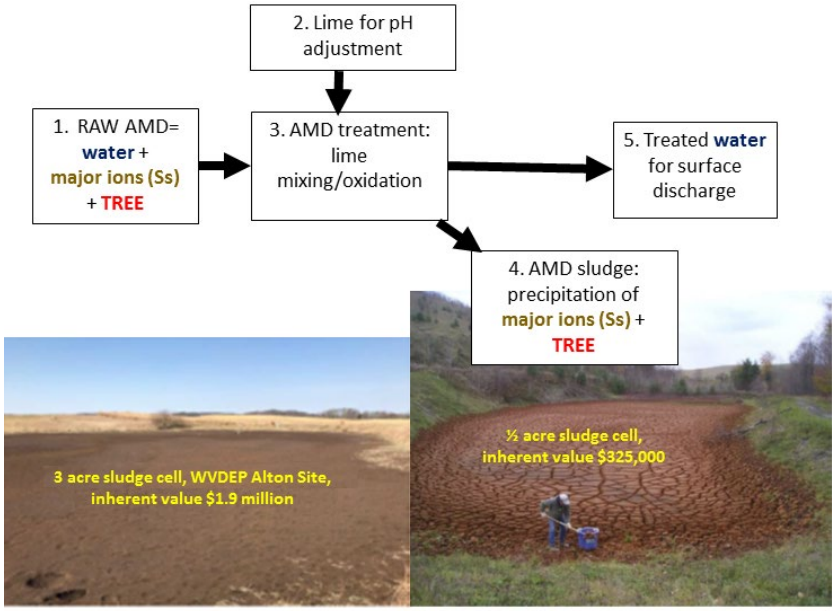
Project Duration: 03/01/2016 – 06/30/2019

Total Project Value: $4,339,414

Key Technology Area: Process Systems

Project Partners: Rockwell Automation; Tetra Tech, Inc.; and Virginia Polytechnic Institute and State University

In this Phase 2 project, West Virginia University and its partners will develop a cost-effective and environmentally benign process to recover rare earth elements (REEs) from solid residues (sludge) generated during treatment of acid coal mine drainage (AMD). This project will take advantage of autogenous processes that occur in coal mines and associated tailings which liberate, then concentrate, REEs. Also, during Phase 2, a continuously operating bench-scale unit is being constructed and operated, yielding 3 grams per hour of REE concentrate. Phase 1 findings showed elevated concentrations of REEs, particularly in low-pH AMD, and nearly all precipitating with more plentiful transition metals in the AMD sludge. REE extraction using hydrometallurgical methods produced a concentrate with 4.6 percent total REE content. A techno-economic analysis also found that REE extraction from AMD sludge is economically attractive with a refining facility projected to generate positive cash flow within five years. Also, in Phase 1, AMD treatment residues were identified as a domestic source of REE feedstock. In addition, an economically attractive extraction and refining process was identified with the potential to generate significant income for operators of AMD treatment sites and relieve the U.S. manufacturing industry's reliance on foreign REE supplies. Estimates based on the volume of AMD generated in Pennsylvania and West Virginia suggest that their AMD sludges represent approximately 610 to 2,700 tons of REEs per year—roughly sufficient to supply the 800 tons that the U.S. defense industry needs each year. In addition, a survey of 154 AMD treatment facilities found 225 tons of sludge (dry weight basis) in surface storage, with an inherent REE value of $122 million.



AMD treatment and sludge storage. TREE: total rare earth elements. Ss: sludge solids. Major ions: Ca, SO_4, Si, Fe, Mg, Mn.

Investigation of Rare Earth Element Extraction from North Dakota Coal-Related Feedstocks

Technology Partner: University of North Dakota

Award Number: FE0027006

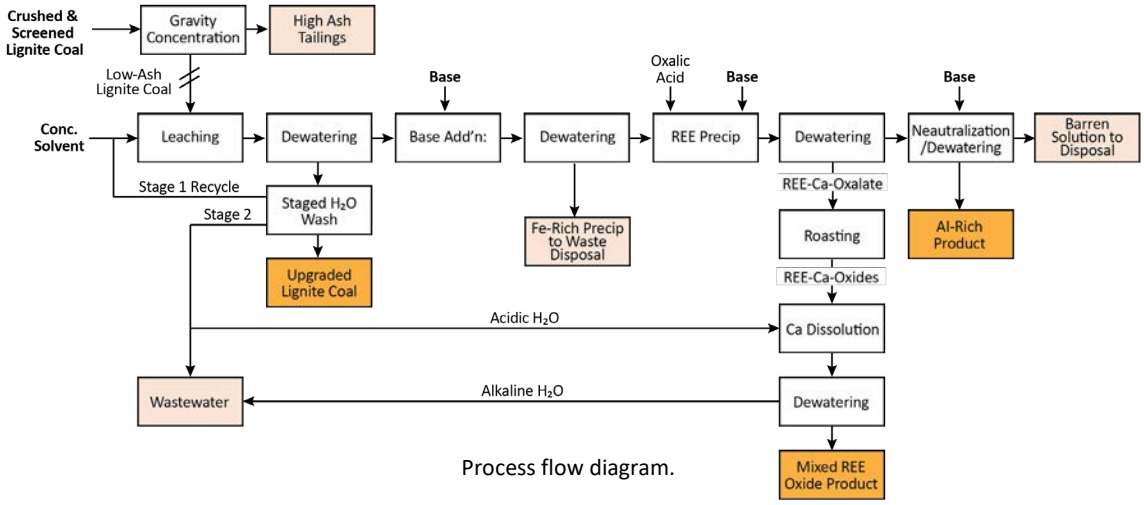
Project Duration: 03/01/2016 – 12/31/2019

Total Project Value: $4,464,347

Key Technology Area: Process Systems

Project Partners: Barr Engineering, Great Northern Properties, Great River Energy, Lignite Research Program of the North Dakota Industrial Commission, Microbeam Technologies, Minnkota Power Cooperative, MLJ Consulting, North American Coal Corporation, North Dakota University System, Pacific Northwest National Laboratory, University of North Dakota

In Phase 1 of this project, the University of North Dakota (UND) project team identified locations in North Dakota with coal-related feed stocks having exceptionally high rare earth elements (REE) content and developed a simple, highly effective, and low-cost method to concentrate the REEs in the lignite feed stocks using a novel technology that takes advantage of the unique properties of lignite. In laboratory experiments, UND achieved greater than two percent concentration of rare earths in the mixed rare earth concentrate while recovering up to 35 percent of the rare earths from the incoming feedstock. In Phase 2, the University is partnering with Microbeam Technologies, Barr Engineering, Pacific Northwest National Laboratory, and MLJ Consulting to investigate the feasibility of recovering REEs from North Dakota lignite and lignite-related feedstocks. The team will scale up the technology and demonstrate it at a scale of 10-20 kilograms per hour feedstock throughput and evaluate the economics for a commercial-scale, rare-earths-concentrating facility in North Dakota. The project also includes development of a commercialization plan and market assessment. The Lignite Research Program of the North Dakota Industrial Commission, North American Coal Corporation, Great River Energy, Minnkota Power Cooperative, Great Northern Properties, the University of North Dakota, and the North Dakota University System are cost-sharing this project. The recovery of REEs from lignite and related materials could lead to a significant new industry for maintaining existing and creating new jobs, and create new opportunities for marketable use of North Dakota lignite coal. Multiple high-value by-products that will further enhance the lignite industry will also result from the process. The potential to significantly or completely offset current imported REE and REE products by providing a reliable domestic resource and novel low-cost technology focused on its development also exists. The ultimate significance of this research is development of a high-performance, environmentally benign, and economically viable technology for REE production from an alternative resource that will reduce dependence on foreign supplies and strengthen the economic and national security of the United States. This project will enable transition to pilot scale, a foundation for subsequent larger demonstrations and eventual commercial deployment.



Process flow diagram.

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

Technology Partner: Physical Sciences, Inc.

Award Number: FE0027167

Project Duration: 03/01/2016 – 03/31/2020

Total Project Value: $8,750,166

Key Technology Area: Process Systems

Project Partners: University of Kentucky Center for Applied Energy Research and Winner Water Services

In this Phase 2 project, the team of Physical Sciences, Inc. (PSI), University of Kentucky/Center for Applied Energy Research, and Winner Water Services will develop and demonstrate a pilot scale plant to economically produce salable rare earth element (REE)-rich concentrates including yttrium and scandium (REYSc) and commercially viable co-products from coal ash feedstock using environmentally safe and high-yield physical and chemical enrichment/recovery processes. The pilot plant will operate at the scale of approximately 0.4-1 tons per day (tpd) ash throughput for physical processing and about 0.5 tpd for chemical processing, producing at least 50 grams (g) of dry REYSc nitrates concentrate containing more than 10 percent by weight of REYSc, and targeting 500 g of dry REYSc nitrates concentrate containing more than 20 percent REYSc by weight. The ash feedstock will come from Dale power plant in Ford, Kentucky, with at least 300 parts per million (ppm) of REYSc content, though more than 500 ppm is anticipated. The data obtained from the pilot plant operations will be used to enhance and validate the techno-economic analysis that was completed for both the physical and chemical processing plants at a scale of 600 tpd in Phase 1, and use it to design a commercial scale plant (hundreds of tpd throughput) with return on investment less than seven years. Development and demonstration of modular, transportable pilot-scale physical and chemical plants for REYSc recovery from coal ash, including enhancement and validation of a techno-economic model of pilot plant operations data, will enable the design of a commercial-scale (approximately hundreds of tpd) REYSc plant. Demonstration of PSI's technology to recover REYSc from coal ash will enable utilization of coal mining/coal combustion waste product in environmentally benign ways to produce REEs of strategic importance for the United States while generating jobs and economic growth in economically-depressed regions of this country.

Micropilot plant.

Pilot-Scale Testing of An Integrated Circuit for the Extraction of Rare Earth Minerals and Elements from Coal and Coal By-products Using Advanced Separation Technologies

Technology Partner: University of Kentucky

Award Number: FE0027035

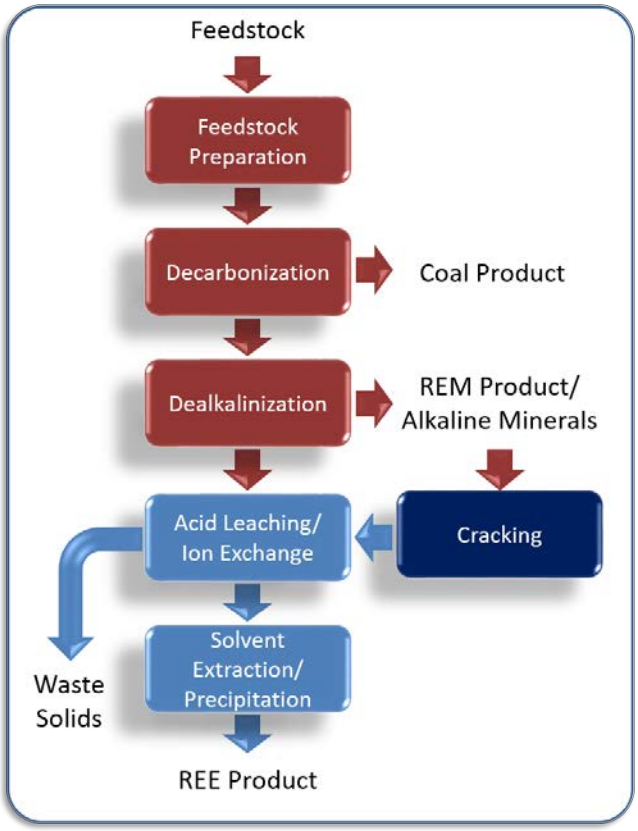
Project Duration: 03/01/2016 – 03/31/2020

Total Project Value: $8,820,009

Key Technology Area: Process Systems

Project Partners: Alliance Resources, Blackhawk Mining, Minerals Refining Company, Mineral Separation Technologies, Virginia Polytechnic Institute and State University, and West Virginia University

In Phase 1 of this project, the University of Kentucky (UK) identified two bituminous coal-related feedstocks qualified as having ample supply with high rare earth element (REE) content (above 300 parts per million) and developed a preliminary design for a mobile pilot plant to recover REEs from those feedstocks. In laboratory experiments, UK achieved greater than 80 percent concentration of rare earths in the mixed rare earth concentrate while recovering greater than 75 percent of the rare earths from the incoming feedstock. In Phase 2, the University is testing a one-fourth ton/hour pilot-scale plant for the extraction of REEs from Central Appalachian and Illinois Basin bituminous coal preparation plant refuse materials. The system integrates both physical and chemical (ion exchange and solvent extraction) separation processes that are commercially available and environmentally acceptable. The innovative enabling technology utilized in the system includes an advanced froth flotation process and a novel hydrophobic-hydrophilic separation process. The project is expected to develop critical technology, conditions and data necessary to design, construct and operate a state-of-the-art processing facility for the production of REEs from various coal and coal by-product streams.



Schematic of the University of Kentucky's process.

Rare Earth Elements – Novel REE Separation & Advanced Sensor Development

Evaluation of Novel Strategies and Processes for Separation of REE from Coal-Related Materials

Technology Partner: Los Alamos National Laboratory

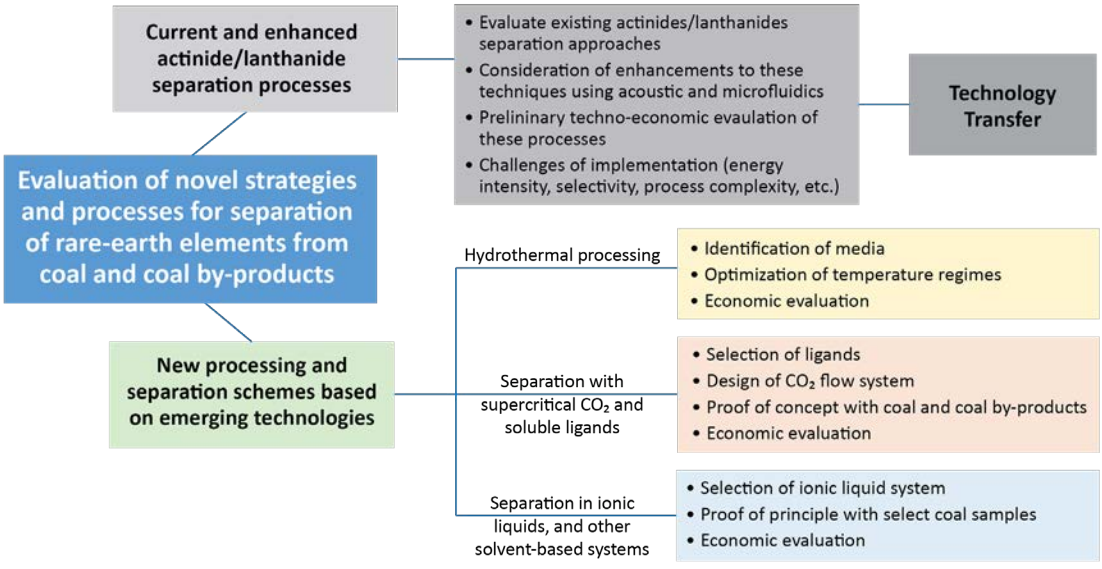
Award Number: FWP-FE-810-17-FY17

Project Duration: 08/01/2017 – 09/30/2020

Total Project Value: $1,000,000

Key Technology Area: Separation Technologies

Los Alamos National Laboratory (LANL) has a long history in the chemistry and separation of *f* elements (i.e., the lanthanide and actinide groups), as needed to support its core national-security mission. This history has resulted in unique experience in both process innovation and process implementation at a range of scales. LANL will use this unique expertise in separation of *f* elements to evaluate new innovative processes in rare earth element (REE) separation from coal and coal by-products. This effort comprises two complementary tasks. The first task will evaluate current and enhanced actinide/lanthanide separation processes relative to identifying potential processes and strategies for REE separation from coal and coal by-products. This first task will evaluate existing actinides/ lanthanides separation approaches developed for nuclear materials and their potential application for REEs extraction; included in this will be a consideration of techno-economic evaluation of these processes and challenges related to energy intensity, selectivity, and process complexity in the context of application to separation of lanthanides from coal-related materials. The second task will evaluate the potential of developing new processing and separation schemes based on emerging technologies. The initial phase of the project will screen the following processing approaches: (1) processing under hydrothermal conditions; an effort that builds on previous work that suggests that a better control of lanthanides speciation at elevated temperature can be exploited to perform efficient REEs separation, (2) REE-selective extraction using supercritical carbon dioxide (CO_2) and soluble ligands; an effort that builds on initial proof-of-concept studies on supercritical CO_2 that show great promise for simple and effective separation of REE from oxide materials, and (3) separation of REEs using ionic liquids and other solvent-based systems, an effort that builds on current efforts at LANL to develop actinide/lanthanide separation schemes in ionic liquids.



Schematic representation of the project.

Evaluation of Laser-Based Analysis of REE in Coal-Related Materials

Technology Partner: Los Alamos National Laboratory

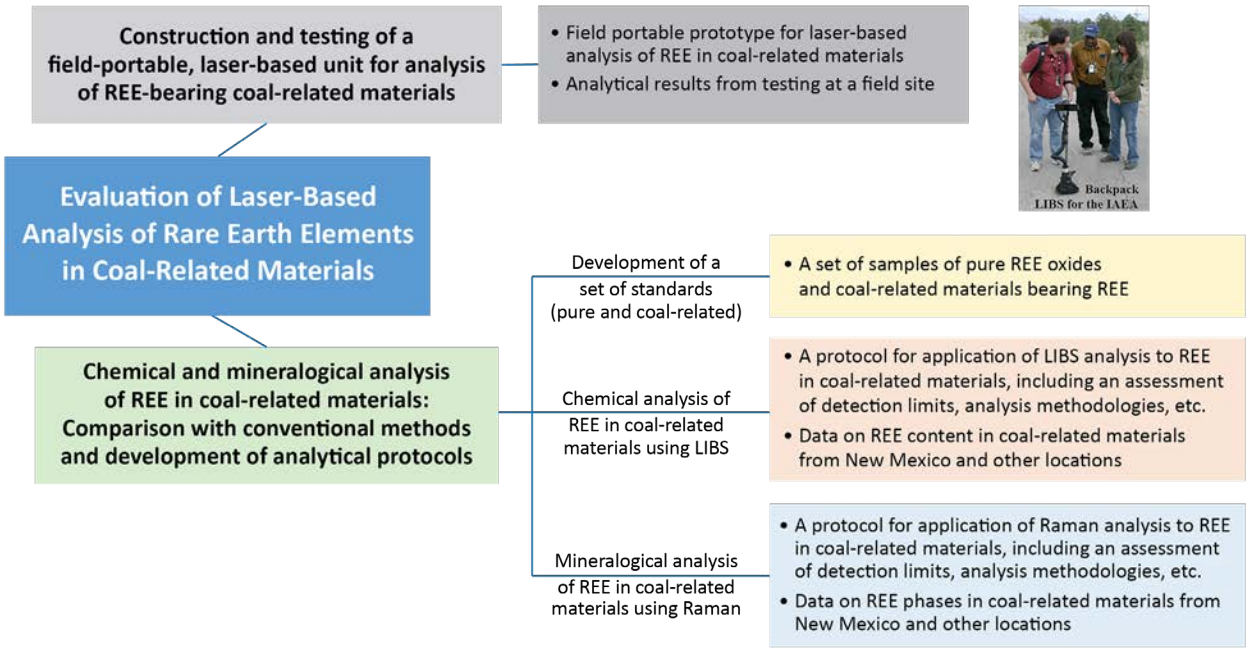
Award Number: FWP-FE-781-16-FY17

Project Duration: 10/25/2017 – 10/24/2020

Total Project Value: $1,000,000

Key Technology Area: Enabling Technologies

The primary objectives of this effort are to (1) develop and test analytical protocols for analyzing the concentration and mineralogy of rare earth elements (REEs) in coal-related materials using laser-induced breakdown spectroscopy (LIBS) and Raman spectroscopy; (2) develop a field-portable system for LIBS and Raman analysis of REE in coal-related material; and (3) analyze the concentration and mineralogy of REE in a variety of coal-related materials principally from New Mexico coal deposits. The project is divided into two tasks. In task 1, the team will construct a field-portable prototype unit for LIBS/Raman analysis of REEs in coal-related materials; field test and demonstrate the unit at New Mexico field sites; and revise analytical protocols as applicable. Researchers will take advantage of the extensive experience of Los Alamos National Laboratory (LANL) in developing LIBS for quantitative analysis of elements in various matrices and will use this expertise to develop protocols for analysis of REEs in coal-related materials. LANL has made recent institutional investments (through laboratory directed research and development) to develop a combined LIBS plus Raman system that can determine both chemistry (LIBS) and physical form (Raman). Researchers will use this new system to explore simultaneous chemical and mineralogical analysis of REEs in coal-related materials. In task 2, the team will develop analytical methods specific to the quantification of REE in various coal-related materials, thereby developing a broader database on REE concentrations and physical forms in a variety of coal-related materials. This step is necessary in the development of protocols for quantitative analysis of REE using LIBS. Interpretation of the data requires determining calibration curves for REEs in specific matrices, because the efficiency of plasma generation (a first step in LIBS analysis) can be impacted both by physical form and overall chemistry.



Schematic representation of the project.

New Sensing Mechanisms for REE Detection in Coal and Coal Byproducts

Technology Partner: Idaho National Laboratory

Award Number: FWP-INEL-B000-17-015

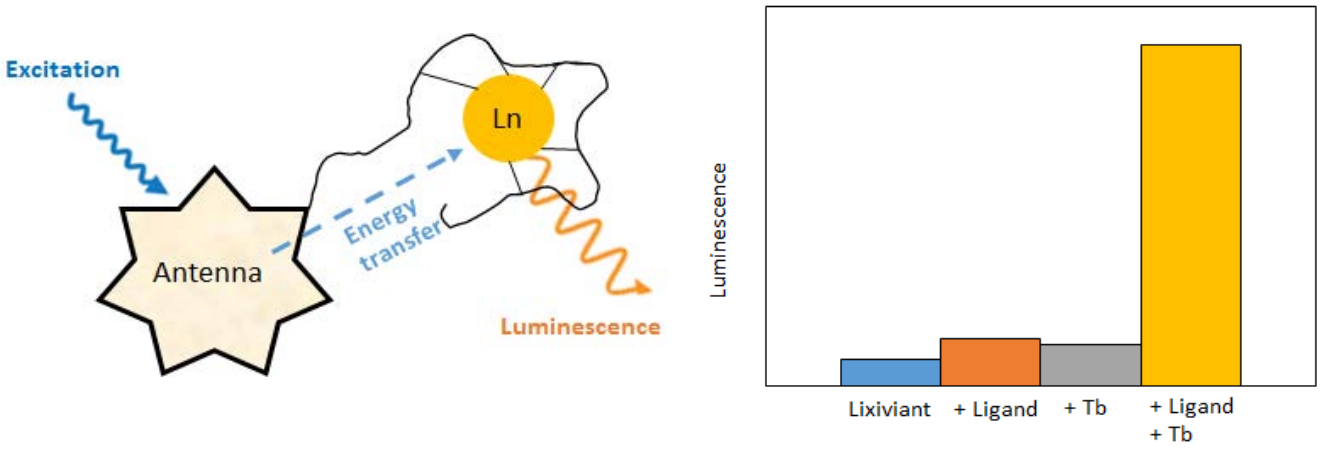
Project Duration: 08/31/2017 – 09/30/2020

Total Project Value: $1,000,000

Key Technology Area: Enabling Technologies

Project Partners: Lawrence Livermore National Laboratory; Rutgers–The State University of New Jersey; and University of California, Davis

The objective of this project is to evaluate novel complexation chemistries for the development of innovative sensing technologies for rare earth elements (REEs). Complexation of lanthanides by peptides, coupled with the unique spectroscopic properties of lanthanides, is the underpinning for luminescent applications of lanthanide binding tags (LBTs), originally invented as biochemical tools for the study of proteins. A peptide sequence specifically designed to bind lanthanides includes amino acids which have chromophore side-chains (tyrosine or tryptophan), and upon lanthanide binding the complex exhibits unique luminescence properties, enabling detection and visualization. In coal and coal by-products, although specific lanthanide enrichments may vary by provenance, generally the whole lanthanide series is present. A positive signal generated upon exposure of a sample to the specialized REE ligand would imply that the sample is enriched with REEs and is worthy of further examination. Specifically, Idaho National Laboratory will evaluate whether the chemistry of LBTs or other novel chromophore ligands can serve as the foundation for tools enabling rapid screening of REE-containing materials in the field. This work is at the discovery scale, but if successful, it could lead to the development of field-deployable sensors or field test kits for detection of REEs in coal or coal by-products. Such sensors could be useful for myriad purposes beyond REE detection in coal, such as monitoring of REE content in process streams, detection of upsets during industrial processing, and rapid testing and validation of new extraction or separations techniques. In addition, the modeling and laboratory studies of the REE-ligand associations will contribute to fundamental understanding of the complexation behavior of lanthanides.



Luminescence is enhanced when a lanthanide binds to a ligand that has an attached antenna chromophore.

Application of Biosorption of REE Recovery from Coal By-Products

Technology Partner: Lawrence Livermore National Laboratory

Award Number: FWP-LLNL-18-FEW0239

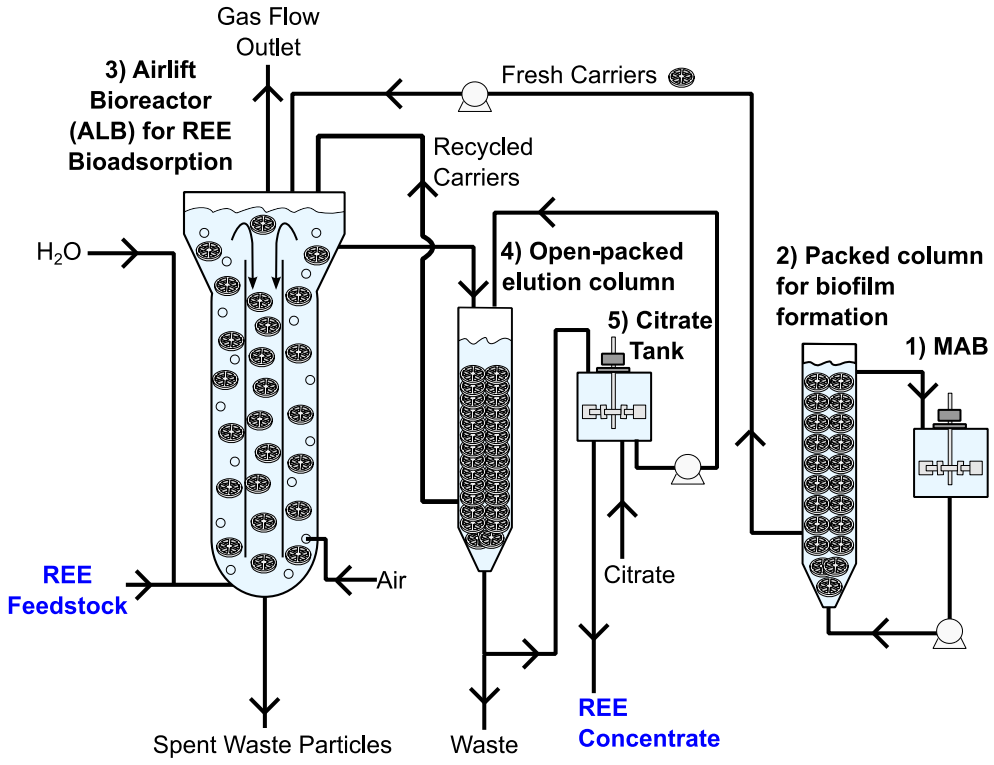
Project Duration: 03/01/2018 – 02/28/2021

Total Project Value: $950,000

Key Technology Area: Separation Technologies

Project Partners: Duke University and University of Arizona

The objective of this research is to determine whether biosorption can be used as an inexpensive and cost-effective means for rare earth element (REE) recovery from leachate of pre-combustion and post-combustion coal by-products. This project will develop a biofilm-based continuous flow-through system in an airlift bioreactor. This effort will also improve the biofilm stability of *E. coli* using a bioengineering approach to incorporate surface binding tags. These surface binding peptides are known to improve adhesion and increase biofilm stability under high shear force. The *Caulobacter* biofilm formation activity was previously completed. A single bacterial platform for the application of airlift bioreactor will be down-selected based on several evaluation criteria, including (1) biofilm density (number of cells per unit area), (2) biofilm forming efficiency (proportion of cells that attach to the surface in a cell population), (3) biofilm stability, (4) REE-binding ligand density (number of REE-binding ligands incorporated per cell), and (5) REE adsorption capacity per unit area of the biofilm. At conclusion, the project is expected to deliver a bench-scale demonstration of an airlift bioreactor for REE recovery and demonstrate its technical feasibility with preliminary economic viability analysis and a plan for commercialization.



Airlift bioreactor design and process flow diagram. The proposed system consists of a two-stage semicontinuous process including (1) a closed mechanically agitated bioreactor (MAB) used to grow the microbes, (2) an open cylindrical container as a packed column for biofilm formation on carrier disks, (3) an airlift bioreactor (ALB) for adsorption of REE onto biofilm carrier disks, and (4) an open-packed elution column (EC) with (5) circulating citrate from a citrate tank for REE desorption and recovery.

Rare Earth Elements – Transformational REE Separation

Low Temperature Plasma Treatment for Enhanced Recovery of Highly Valued Critical REEs from Coal

Technology Partner: University of Kentucky

Award Number: FE0031525

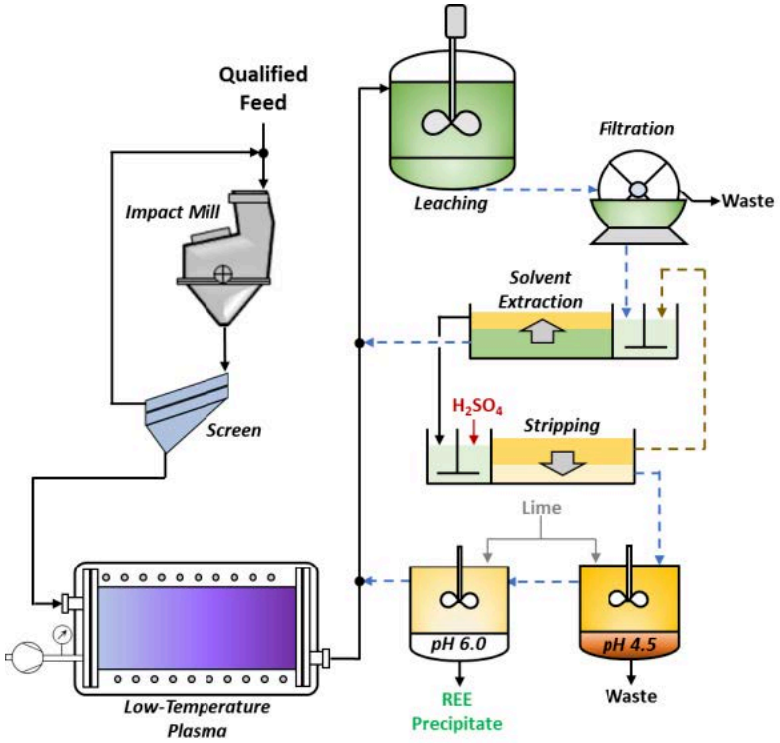
Project Duration: 11/16/2017 – 05/15/2019

Total Project Value: $404,969

Key Technology Area: Separation Technologies

Project Partners: Virginia Polytechnic Institute and State University

The principal objective of this project is to develop a novel process using low-temperature plasma treatment integrated with hydrometallurgical processes to recover rare earth elements (REEs), especially highly valued REEs (e.g., scandium and critical REEs), from coal and coal by-products. The project will initially evaluate the mineralogy, leachability, and effect of plasma pretreatment for the various segments of selected feedstocks containing greater than 300 parts per million of total REEs on a dry, whole mass basis. A laboratory low-temperature oxygen plasma unit with the ability to control test conditions will be used to optimize the operating parameters of the plasma treatment process (e.g., power, temperature, treatment time, etc.) with respect to feedstock characteristics, such as the exposed surface area, pore size, microstructure, degree of oxidation, etc. After optimizing the process, plasma treatment will be integrated with leaching, solvent extraction, and precipitation processes to produce REE concentrates at improved recovery levels and grades higher than 10 percent total REEs on a dry, whole mass basis. A techno-economic feasibility analysis will be conducted of plasma treatment integrated into an overall REE recovery system. The project, conducted at laboratory scale, has the benefit of providing key information needed to overcome challenges in larger-scale operations and further maturation of the method.



Flowsheet to produce greater than 2 percent REE concentrate with assist of low temperature plasma treatment technique.

Development of a Cost-Effective Extraction Process for the Recovery of Heavy and Critical REEs from the Clays and Shales Associated with Coal

Technology Partner: Virginia Polytechnic Institute and State University

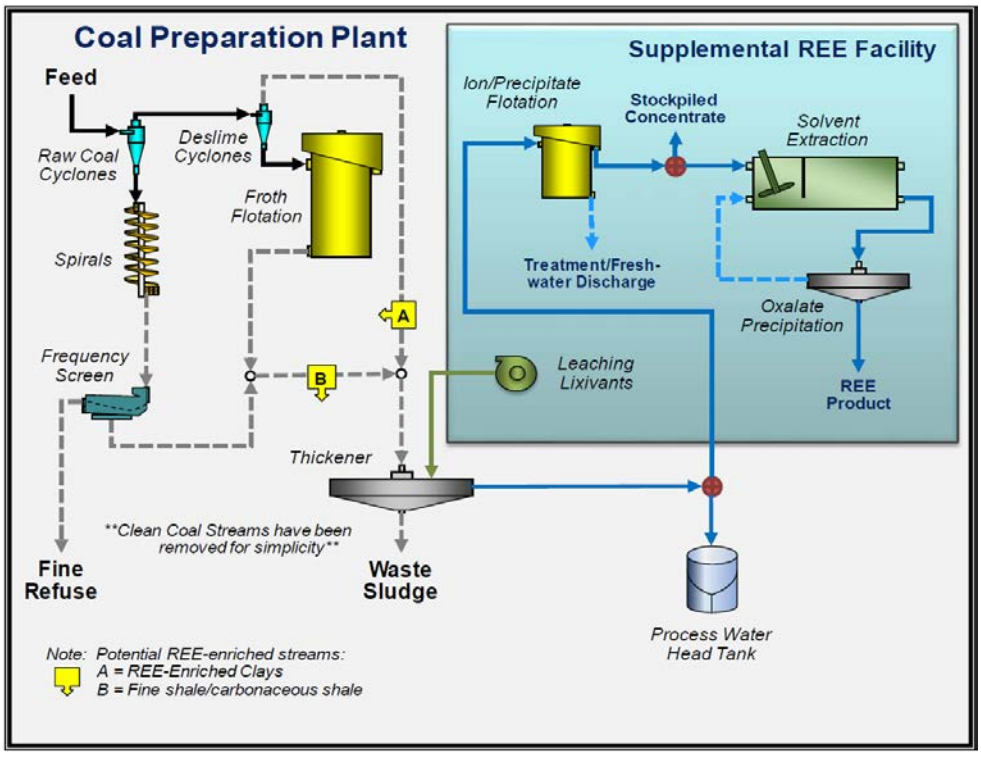
Award Number: FE0031523

Project Duration: 11/16/2017 – 05/15/2019

Total Project Value: $500,000

Key Technology Area: Separation Technologies

The project will investigate ion-exchange leaching and concentration technologies that can extract and enrich rare earth elements (REEs) derived from coal resources, specifically clay and shale. Work performed under this project will include field sample collection, thermodynamic assessments, routine laboratory testing, and engineering analyses. Initial efforts will focus on identifying, collecting, and characterizing at least three distinct feedstock samples that approach or exceed 300 parts per million total REE on a whole sample basis. Next, experimental efforts will focus on two distinct process operations, including (1) ion-exchange leaching and (2) ion/precipitate flotation. A limited number of solvent extraction tests will be performed for comparative purposes only. These experimental efforts will be supported by ongoing thermodynamic assessments, which will provide a fundamental basis for selection and dosing requirements of lixiviant (liquid used for leaching a metal from its ore or mineral). During the final tasks, the results from the experimental program will be used to generate a preliminary system design, and cost/revenue modeling will be used to perform a rigorous techno-economic assessment. The result will be a low-cost, environmentally benign process that can be readily implemented at many of the U.S. domestic coal production facilities. Successful development of the technology may provide a pathway to commercial extraction of the ion-adsorbed REE reserve in U.S. coals.



Process flowsheet integrated with existing process circuitry.

Low Cost REE Recovery from Acid Mine Drainage Sludge

Technology Partner: Research Triangle Institute

Award Number: FE0031483

Project Duration: 11/20/2017 – 05/19/2019

Total Project Value: $500,000

Key Technology Area: Separation Technologies

Project Partners: Cerahelix and Veolia Water Technologies

This project will develop a membrane-based, bench-scale system to extract strategic minerals such as rare earth elements (REEs) and other critical minerals from acid mine drainage (AMD) sludge generated as part of coal mining activities. The effort will use a staged, membrane-based treatment approach to separate, concentrate, and ultimately recover REEs from AMD. Initial work will take water samples from a potential AMD site (or sites) and characterize them for REE concentration, dissolved metals concentration, and key water-quality characteristics. Each individual process component will be tested with water samples to optimize performance. The work will initially include proof-of-concept experiments at the bench scale with the aim of varying process parameters such as water chemistry, nanofiltration membrane performance in monovalent/multivalent separation, affinity media chemistry, and solvent recovery of REE. From these experiments, separation conditions that can be reasonably transitioned to flow-through systems and larger prototype scales for further techno-economic analysis will be selected so that the economic performance of a continuously-fed AMD fluid process for REE recovery can be evaluated. The process has the potential to expand the United States' sources of strategic elements by exploiting a domestic resource such as AMD sludge, thereby reducing U.S. dependence on foreign countries for these critical elements. The process would enable energy-efficient, cost-effective recovery of REEs to two percent by weight REE pre-concentrate. The steps comprising the process are inherently low-cost approaches.

Cerahelix ceramic tubular membranes for monovalent/multivalent separation.

Economic Extraction and Recovery of REEs and Production of Clean Value-Added Products from Low-Rank Coal Fly Ash

Technology Partner: University of North Dakota

Award Number: FE0031490

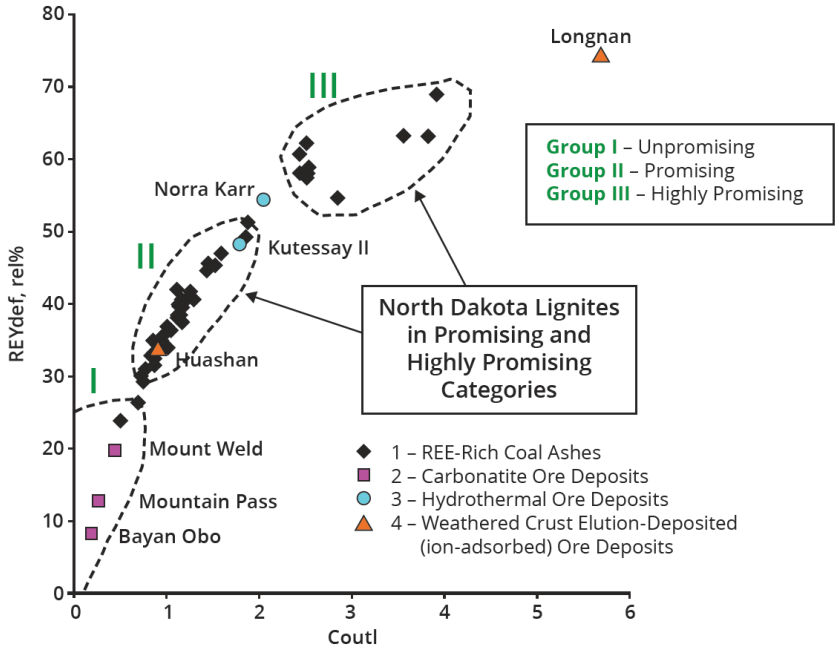
Project Duration: 11/16/2017 – 05/15/2019

Total Project Value: $508,812

Key Technology Area: Separation Technologies

Project Partners: Pacific Northwest National Laboratory

The project's objective is to develop an economically viable and tailorable rare-earth element (REE) extraction and concentration method for low-rank coal fly ash and bottom ash that will produce a concentrate containing greater than or equal to two percent by weight total REE. The project will focus on low-rank (lignite and subbituminous) coal combustion/ gasification ashes. Ash samples will be collected from industry partner facilities as well as from the existing sample database for North Dakota lignites at the University of North Dakota. The characterization to be performed will fully elucidate the abundance, form, and association of the REEs, both in the feed coals that produced the ash, and in the ashes. Additionally, the chemical composition, mineralogy, and morphology of the ash will be determined. Based on the characterization results, two ash samples will be downselected for laboratory-scale REE extraction and concentration testing. The laboratory-scale testing will involve evaluation of ash pretreatment methods, dilute acid leaching, and solvent extraction testing, which will be followed by REE concentration testing at downselected conditions and materials. The project will also evaluate a novel method of value-added beneficiation of the clean fly ash. Finally, based on the experimental testing, a preliminary technical and economic analysis will be completed to estimate capital and operating expenses and product revenues. The project benefits are the development of a high-performance, environmentally benign, and economically viable technology for REE production from an alternative resource that will limit dependence on foreign supplies and strengthen the economic and national security of the United States.



Classification of coal fly ash (black diamonds) REE distribution in comparison with selected conventional REE deposits. Seredin, V.V.; Dai, S. Coal Deposits as Potential Alternative Sources for Lanthanides and Yttrium. International Journal of Coal Geology 2012, 94, 67-93.

Concentrating Rare Earth Elements in Acid Mine Drainage Using Coal Combustion By-Products Through Abandoned Mine Land Reclamation

Technology Partner: Ohio State University

Award Number: FE0031566

Project Duration: 12/01/2017 – 04/30/2019

Total Project Value: $528,700

Key Technology Area: Separation Technologies

The project team will develop an integrated process that first uses stabilized flue gas desulfurization material (sFGD) to recover rare earth elements (REEs) from acid mine drainage (AMD) and a sequential extraction procedure to produce a rare earth feedstock with above two percent by weight REE. The objectives are to (1) validate the effectiveness and feasibility of the integrated REE recovery/concentrating process, (2) determine mechanisms controlling the rare earth recovery process, (3) quantify the associated economic and environmental benefits, and (4) evaluate the full-scale application of the process. To achieve these objectives, tasks will be carried out in four phases. In the first phase, the research team will collaborate with state agencies to carry out field investigations designed to screen and evaluate the seasonal changes of REEs from AMD discharges that have high recovery potential. In the second phase, laboratory-scale tests will be carried out to study the recovery process under a range of percolation conditions using AMD and sFGDs from selected sources. The associated water quality changes will be monitored. Advanced analytical techniques, including synchrotron-based X-ray methods, will be used to identify the mineral forms of retained REEs. In the third phase, a highly selective sequential extraction procedure will be used to concentrate the REEs. In the fourth and final phase, techno-economic analysis and life-cycle assessment to evaluate the economic and environmental benefits will be performed. The rare earth recovery and concentrating process can be integrated with abandoned mine land (AML) reclamation to create an approach that can (1) add economic incentives for AML reclamation, (2) remediate AMD discharge, (3) provide a long-term, high-volume beneficial use for coal combustion by-products, which otherwise need to be disposed of in a landfill, and (4) eliminate public safety hazards and threats to local environment and ecological systems posed by AMLs.

Bench scale testing setup.

Economic Extraction, Recovery, and Upgrading of Rare Earth Elements from Coal-Based Resources

Technology Partner: University of Utah

Award Number: FE0031526

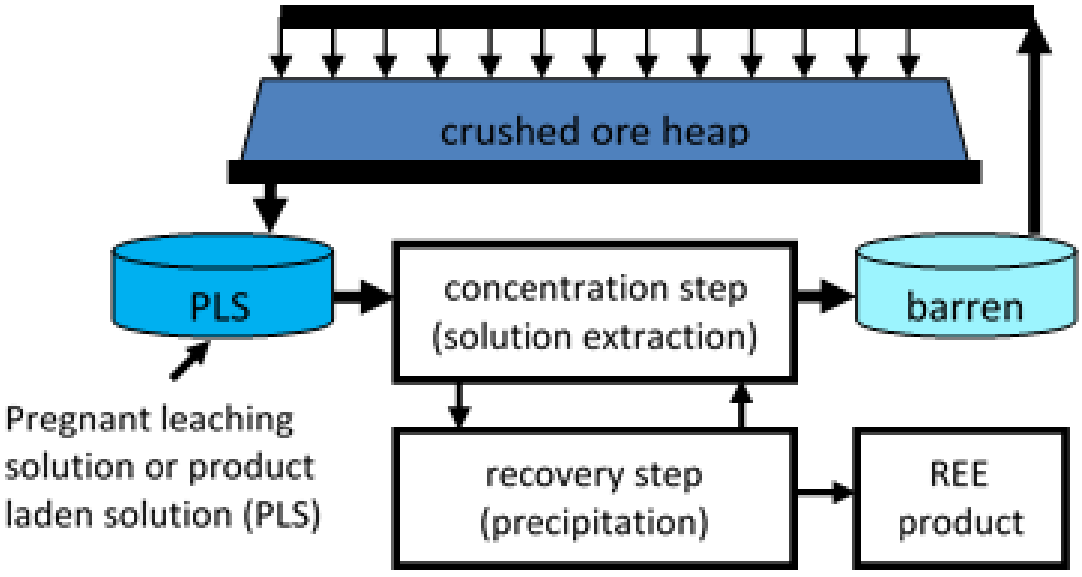
Project Duration: 11/16/2017 – 05/15/2019

Total Project Value: $499,000

Key Technology Area: Separation Technologies

Project Partners: Virginia Polytechnic Institute and State University

The objective of this project is to demonstrate and improve methods that can economically extract, recover, and upgrade the rare earth elements (REEs) contents from coal-based resources using integrated modeling, coal preparation, bio-oxidation, solution conditioning, heap leaching, solvent extraction, and precipitation technologies to cleanly and cost-effectively produce rare-earth-bearing products with more than 8 percent by weight REE. The project encompasses a range of technologies currently in industrial practice to produce REE products from coal-based sources. Advanced coal processing technology will deliver clean coal to the market as well as REE-bearing coarse refuse (non-coal rock) that is the correct size for heap leaching applications. In addition, the technology provides concentrated sulfide minerals (for mid-to-high-sulfur coals) for cleaner coal and enhanced biooxidation to accelerate leaching of REEs from the coarse refuse. The removal of the sulfide minerals cleans the coal, accelerates subsequent REE extraction, and eliminates the potential for most acid rock drainage. The processing method also utilizes biooxidation to increase ferric ion production to enhance leaching, while consuming the sulfide mineral and its associated environmental liability. The solution-conditioning processing method removes iron and controls the pH of the leaching solution to mitigate undesirable extraction of thorium, which often occurs below pH 1.5; the heap leaching portion will be modeled and designed to maximize extraction of REEs while minimizing thorium extraction. The solvent extraction portion of the project involves optimizing extraction and stripping for REE recovery in solution that will be precipitated as a final product in the last stage of precipitation processing. This project will provide the technology and data needed to facilitate large-scale implementation that can provide a domestic REE supply that is low in thorium, create revenue and jobs for the coal industry, and mitigate future acid rock drainage issues.



Flow sheet diagram for low-cost extraction, concentration, and recovery of REE product.

Coupled Hydrothermal Extraction and Ligand-Associated Organosilica Media Recovery of REEs from Coal Fly Ash

Technology Partner: Wayne State University

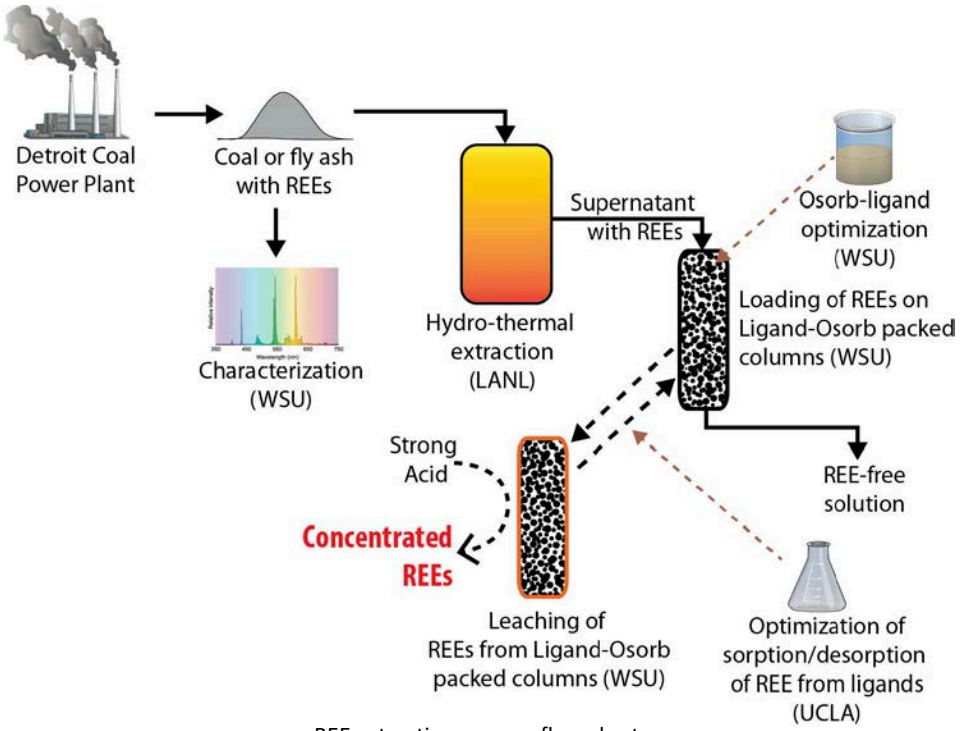
Award Number: FE0031565

Project Duration: 03/15/2018 – 09/14/2019

Total Project Value: $518,849

Key Technology Area: Separation Technologies

This project will investigate the use of hydrothermal leaching under alkaline conditions to rapidly dissolve rare earth elements (REEs) from coal ash solids to an aqueous solution; transfer REEs from the solution to an Osorb platform (commercially available swellable organically modified silica) functionalized with the appropriate ligand system; and extract the REEs to an acidic aqueous system with high concentrations of the targeted REEs (2–10 percent by weight). The project will also examine the potential to couple hydrothermal leaching of coal fly ash with a custom-engineered, ligand-associated medium to provide an organic solvent-free method of extracting and recovering REEs. The fly ash feedstock will be collected from Detroit-area coal-fired power plants, characterized for composition, and subjected to a bench-top batch hydrothermal leaching/dissolution process inside custom-built pressure reactors. Ligands known to be effective in lanthanide/actinide separation will be evaluated based on a series of criteria, such as selectivity and association to Osorb through hydrophobicity. Batch experiments will then be conducted to establish the best conditions (e.g., pH, eluent, etc.) and best ligand systems for effectively extracting REEs from the alkaline feed solution obtained from the hydrothermal leaching process. Once the Osorb-ligand extraction system is optimized for REE concentration, an aqueous solution adjusted to the appropriate pH will be used to back-extract and concentrate the REEs into a heavily REE-laden solution. This project is expected to result in a solvent-free extraction process that is more environmentally benign than many liquid-liquid extractions that typically incorporate organic solvents. Successful completion of this project could lead to future expansion to both bench and pilot scale.



REE extraction process flow chart.

Recovery of High Purity Rare Earth Elements (REEs) from Coal Ash via a Novel Electrowinning Process

Technology Partner: Battelle Memorial Institute

Award Number: FE0031529

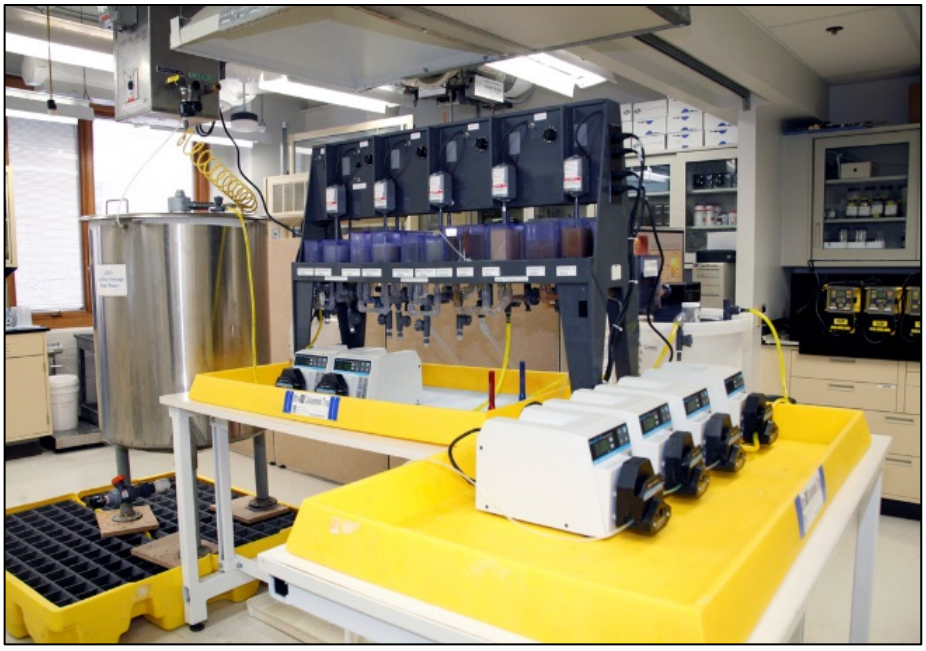
Project Duration: 11/16/2017 – 05/15/2019

Total Project Value: $874,940

Key Technology Area: Separation Technologies

Project Partners: Rare Earth Salts

The main objective of this project is to advance development and integration of the novel electrowinning separation and purification process developed by Rare Earth Salts (RES) and Battelle's acid digestion process, and validate that they can generate environmentally benign and economically sustainable rare earth element (REE) products from domestic coal ash sources at purities above 90 percent. Another objective is to enable domestic REE sources and new outlets for coal products by demonstrating advancements in these purification technologies on coal-based feedstock. These objectives will be accomplished by testing at laboratory scale. Battelle's acid digestion process will be used to upgrade the REE concentrate via solvent extraction, eliminating less-valuable elements such as iron, aluminum, sodium, and calcium, and carefully avoiding enrichment of contaminants such as uranium and thorium. REE products will then be separated and purified from this upgraded solution using RES's novel electrowinning process—a purification process that can reduce the number of stages and cost of REE purification compared to traditional solvent extraction circuits. The project team will perform a preliminary design of a commercial-scale system, using data obtained during lab testing, to generate preliminary capital and operating costs for the process. The outcome of this project will advance Battelle's solvent extraction REE upgrading process, RES's process, and their application to domestic coal-derived feedstocks by validating their feasibility in both technical and economic terms. A high-purity (greater than 99 percent) product of separated REE will be generated from fly ash from an operating pulverized coal combustion plant.



Solvent extraction upgrading of coal ash pregnant leach solution.

At-Source Recovery of Rare Earth Elements from Coal Mine Drainage

Technology Partner: West Virginia University Research Corporation

Award Number: FE0031524

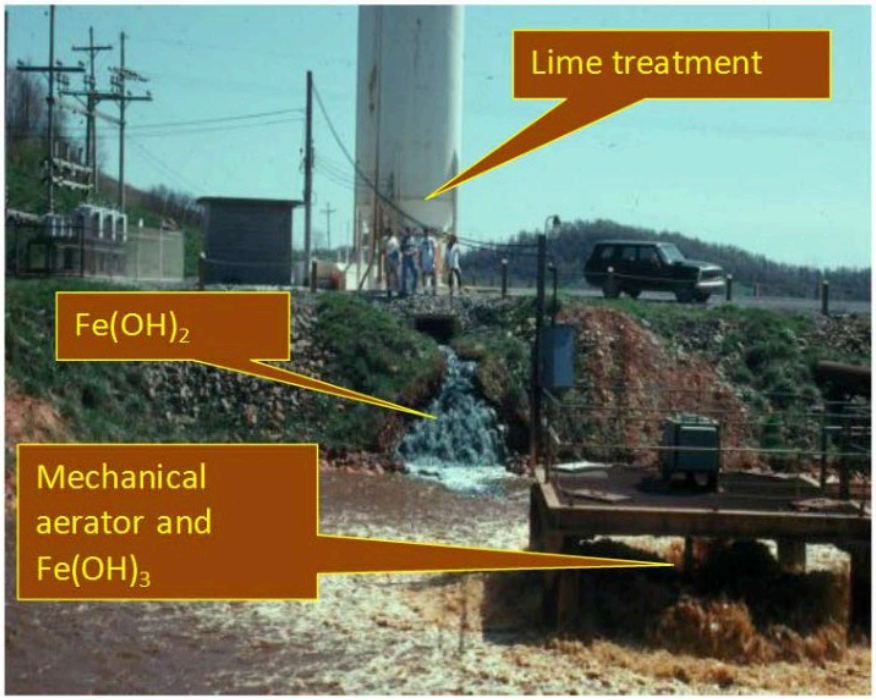
Project Duration: 11/16/2017 – 05/15/2019

Total Project Value: $864,258

Key Technology Area: Separation Technologies

Project Partners: Virginia Polytechnic Institute and State University

The project objective is to develop a process to extract an enriched, mixed rare earth element (REE) product from acid mine drainage (AMD) at the site of production, upstream of conventional AMD treatment. Two AMD cases— net acid and net alkaline (Cases A and B, respectively)—will be explored. The products will be processed through an acid leaching/solvent extraction (ALSX) plant to compare performance with ongoing ALSX trials using conventional AMD sludge feedstock. The project team will evaluate the benefits of separating REEs from the AMD stream under reducing conditions such that iron and manganese will remain in their reduced (Fe^{2+}, Mn^{2+}) states. As reduced species, they will bypass the REE extraction process, improving overall process economics. In Case A, the pH is raised to just pH 4, which will precipitate REEs but not Al, Fe^{2+}, or Mn^{2+}, metals that would otherwise need to be separated to achieve high REE purities. In Case B, the team will explore the application of an electrochemically stimulated supported liquid membrane strategy to separate REEs from ferrous ion. The project will demonstrate that significant improvements in REE extraction efficiency can be obtained through separation of REEs from aqueous phase AMD, upstream of conventional AMD treatment, by (1) creating an enriched REE feedstock, (2) producing a more consistent feedstock, (3) reducing transportation costs to a REE refinery, (4) reducing acid consumption in the acid leaching step, and (5) reducing the volume of waste produced at the ALSX plant.



Typical AMD treatment facility showing the lime treatment unit, reduced $Fe(OH)_2$ sludge (blue), the mechanical aerator, and resulting $Fe(OH)_3$ oxidized sludge.

Rare Earth Elements – Field Sampling, Process Economics & Embedded REE Demand

Sampling, Characterization and Round Robin Analyses of Domestic U.S. Coal Based Resources Containing High Rare Earth Element (REE) Concentrations

Technology Partner: University of North Dakota

Award Number: FE0029007

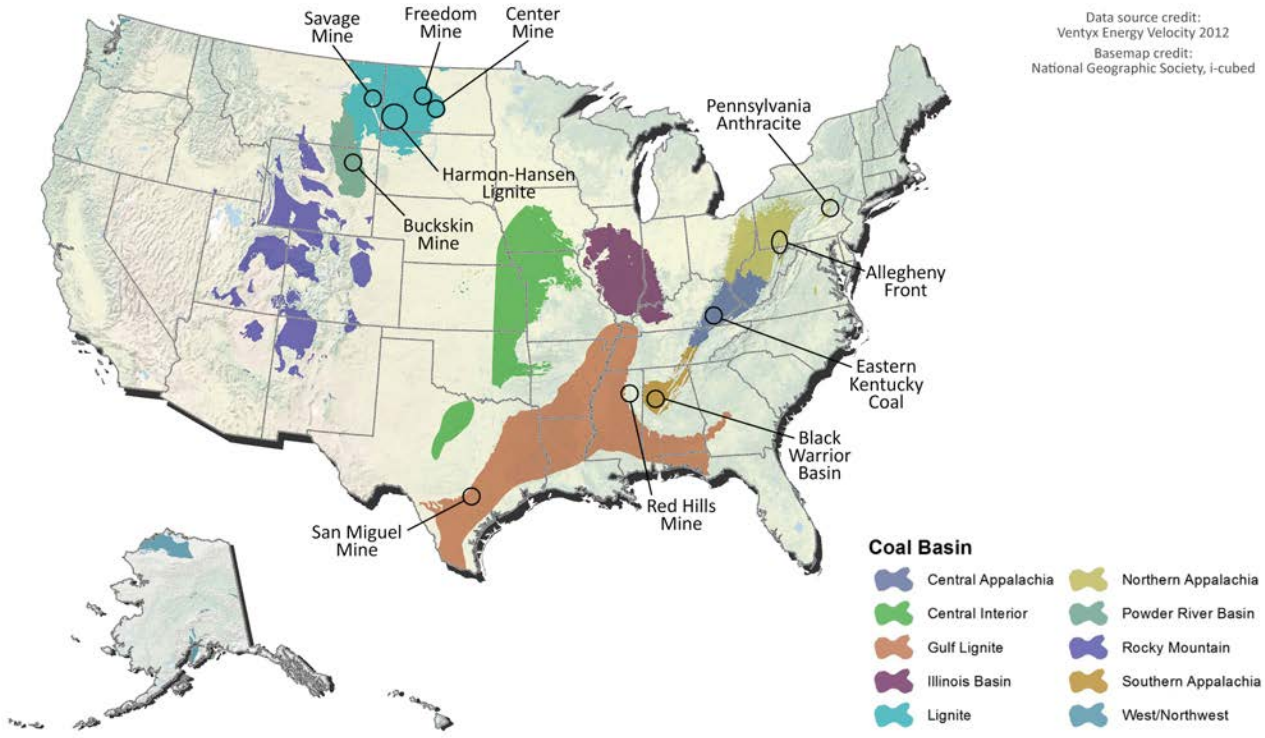
Project Duration: 10/01/2017 – 04/30/2019

Total Project Value: $ 1,499,580

Key Technology Area: Enabling Technologies

Project Partners: University of Kentucky

NETL is partnering with the University of North Dakota to collect and analyze samples of primarily lignite-based coal; and also some bituminous coals from northern, central, and southern Appalachian regions, and coal-related materials (run-of-mine coal; roof rock; overburden clay; shale interlay formations; mine seam underlays; coal preparation refuse; ash from coal combustion, gasification, and liquefaction) that have a minimum concentration of rare earth elements (REE) of 300 parts per million as the material is removed from the ground, with no processing other than drying. This sample analysis for REEs will be done by inductively coupled plasma mass spectroscopy. A round-robin analysis study will also be performed to quantify the within-lab and inter-lab variation of the most commonly used methods for analyzing REE content in ores and coal bearing materials. The project will provide a more quantitative indication of the location of coal and coal-related materials that have high REE content for economic recovery.



Proposed sampling locations.

Rare Earth Elements (REE) from Coal and Coal By-Products - Techno-Economic & Embedded Demand Analysis

Technology Partner: National Energy Technology Laboratory – Research and Innovation Center

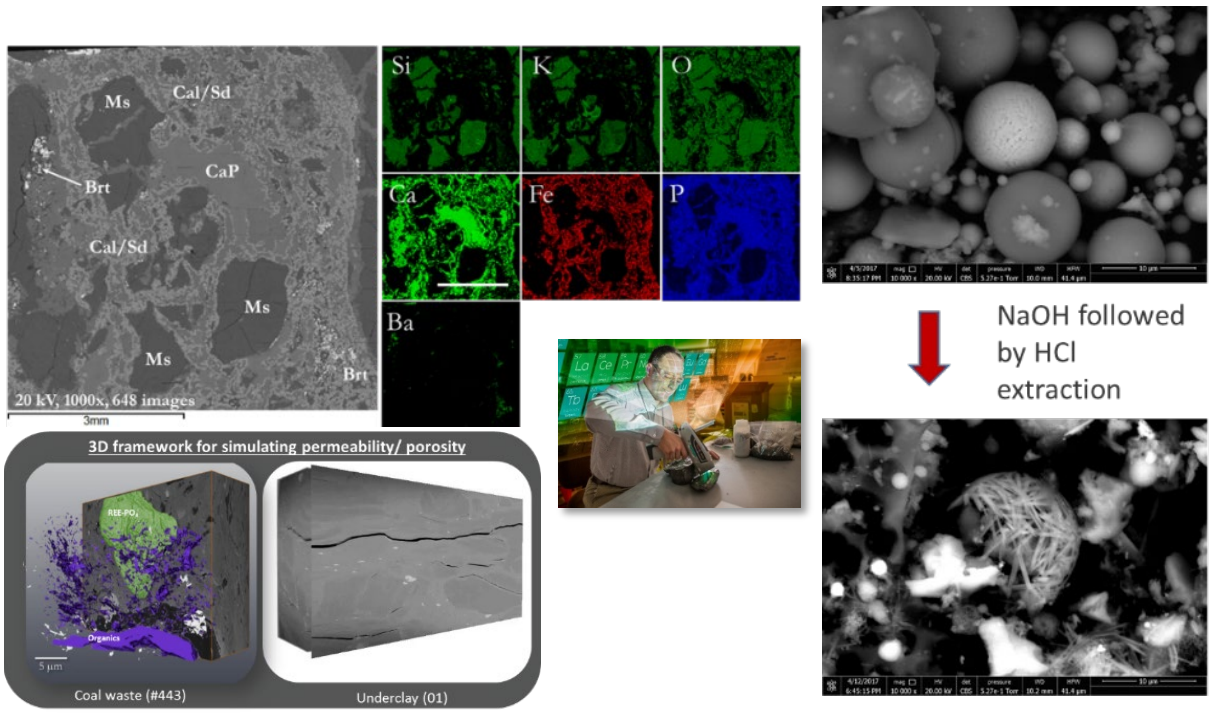
Award Number: FWP-RIC REE FY2016-2020

Project Duration: 10/01/2015 – 03/31/2021

Total Project Value: $11,868,856

Key Technology Area: Enabling Technologies, Separation Technologies, and Process Systems

The National Energy Technology Laboratory (NETL) is conducting a collaborative Field Work Proposal (FWP) focused on developing methods to locate coal-related reserves with high concentrations of rare earth elements (REEs); developing and testing technologies to extract and concentrate REE from coal and coal-related products with emphasis on pathways that are environmentally benign; and reducing technology commercialization risk through the use of modeling and analysis for process optimization and scale-up. The FWP builds upon past NETL research successes and is designed to complement extramural research efforts. Past efforts were focused on both the development of novel REE recovery technologies and characterization techniques, and understanding the relative distribution of REEs, their chemical forms, and association with other phases or minerals in the REE source materials. Current work is focused on (1) intelligent prospecting methods to predict high concentrations of coal-related REE reserves and REE forms which are easily extractable; (2) maturing several promising separation pathways; (3) computational fluid dynamic modeling to enhance separation process development; and (4) techno-economic modeling to understand market opportunities, environmental benefits, and process bottlenecks. Each research area is designed to support the portfolio as a whole and to address key knowledge gaps that are not being investigated by industry. Additional research areas include advanced characterization work in support of separations research, novel and exploratory separations research, and the development of prototype devices for real-time REE quantification in liquids and solids for prospecting and process control of separation processes. Novel separations research will focus on high-risk, high-reward recovery technologies and areas of NETL core competencies.



Rare Earth Elements – Posters

Coal Ash Beneficiation Through Critical Material Extraction and Recovery

Technology Partner: Anactisis, Inc.

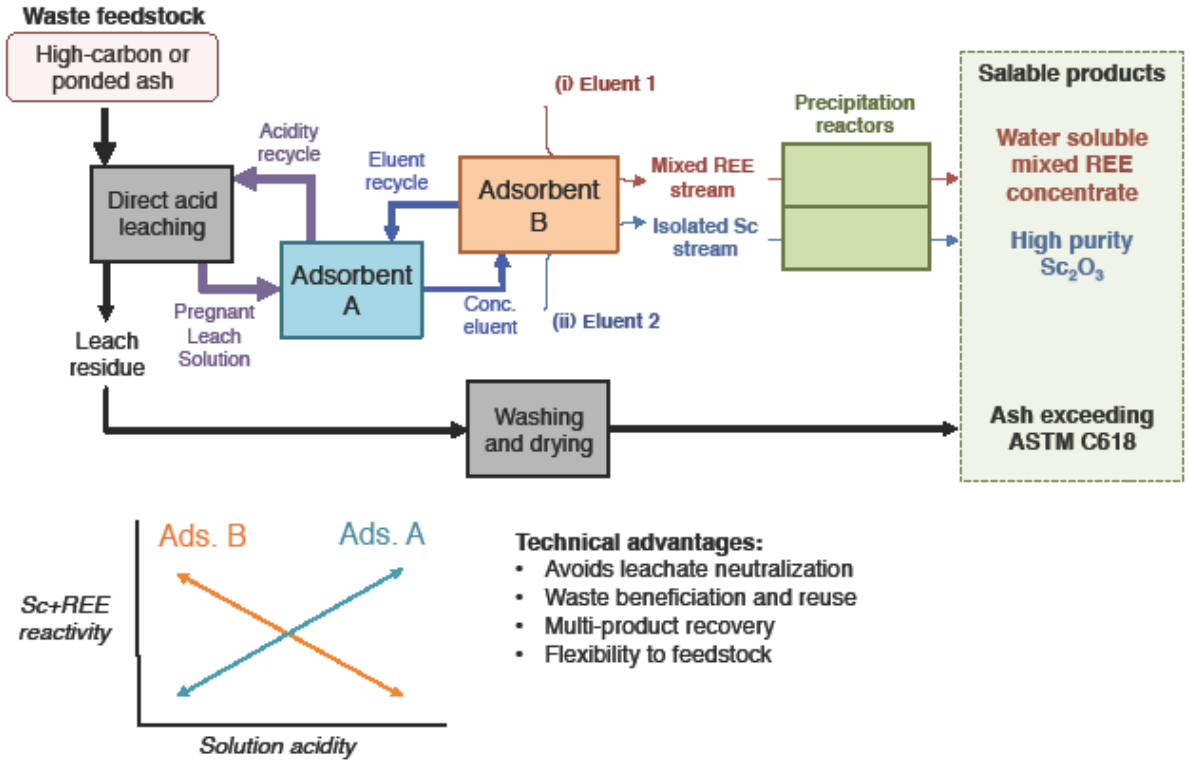
Award Number: SC0018520

Project Duration: 04/09/2018 – 04/08/2019

Total Project Value: $150,000

Key Technology Area: Separation Technologies

A great number of technological advancements have been spurred by the unique properties of the rare earth elements (REE). This widespread application has made REEs absolute necessities for sustaining healthy technological, scientific, and economic growth. In nearly all of these technologies, there are currently no viable substitutes for the REE with equivalent performance. Processes based on conventional mining and metallurgy—which are expensive and laborious with a significant environmental burden—are currently the only economical way of extracting and harvesting these elements. At the same time, global demand outstrips production, and previously mined stocks currently cover the difference via reprocessing. Moreover, there is a growing reliance on Chinese imports for the past 15 years. The proposed technology addresses technical and commercial limitations that have hindered previous attempts to extract metals from industrial wastes. Anactisis has developed a suite of specialized adsorbent media, selective to scandium and the rare earth elements, which enable recovery of these high-value, critical materials from low-grade or waste resources, such as coal combustion residuals. This core technology can dramatically disrupt conventional approaches to industrial waste management. Additional economic and environmental improvements to this process are investigated in this project.



Anactisis, Inc.'s process diagram.

Rare Earth Extraction from Coal Fly Ash

Technology Partner: Skyhaven Systems, LLC

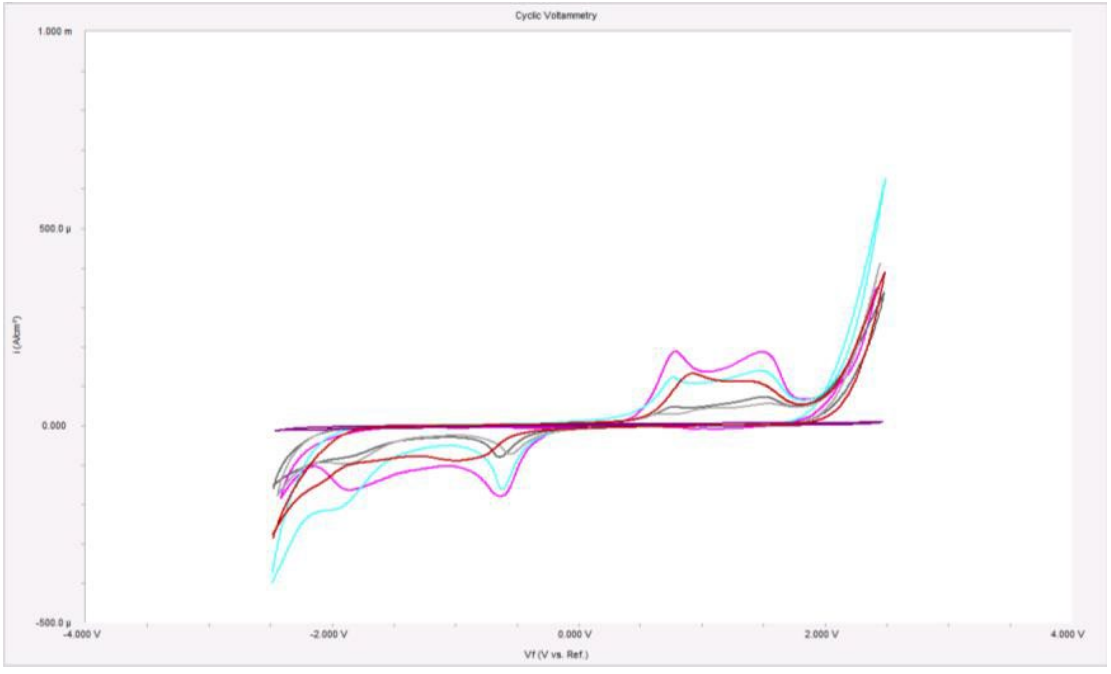
Award Number: SC0018528

Project Duration: 04/09/2018 – 01/08/2019

Total Project Value: $149,978

Key Technology Area: Separation Technologies

Skyhaven Systems, LLC is researching an electrochemical method for extracting and separating rare earth metals from coal fly ash by selective electrodeposition in an ionic liquid solution (IL). The approach will exploit the unique solvating capability of ionic liquids, acid, and water to dissolve stoichiometric amounts of rare earth metal precursors directly into the IL solution. Electrochemical methods will be employed using this same IL solution to recover these metals in a pure form. The overall goal of the work plan is to develop a method to dissolve fly ash directly into the ionic liquid solution, forming lanthanide complexes in-situ, and utilize secondary complexing agents to selectively bind the rare earths, eliminating the need for acid leaching. Selective electrodeposition at a controlled potential will then be performed on the same solution to recover individual rare earths as well as separate them from other metals. In addition, the complexing agents will increase differences in reduction potentials to allow greater separation and individual recovery of rare-earth species from mixtures.



Overlayed Cyclic Voltammograms of Individual Rare Earth Species in Ionic liquid IL-3. Voltage is on the x-axis and current is on the y-axis. IL-3 Baseline *(purple)* (zero current at all voltages), IL-3 with Lanthanum(III) Oxide *(gray),* IL-3 with Neodymium(III) Oxide *(black),* IL-3 with Samarium(III) Oxide *(teal)*, IL-3 with Cerium(IV) Oxide *(pink)*,IL-3 with Praseodymium(III) Oxide *(red)*. At the lower voltages (left side) the rare earths are being electrodeposited (reduced) and at the higher voltages (right side) they are being oxidized. This data was taken without complexing the rare earth species. Distinct oxidation peaks can be seen, however there is significant overlap in some cases. Complexing agents are being investigated to shift the relative reduction potentials enabling greater separation.

Ionic Liquids as Advanced Solvents for the Extraction of Rare Earth Elements from Coal Products

Technology Partner: Wyonics, LLC

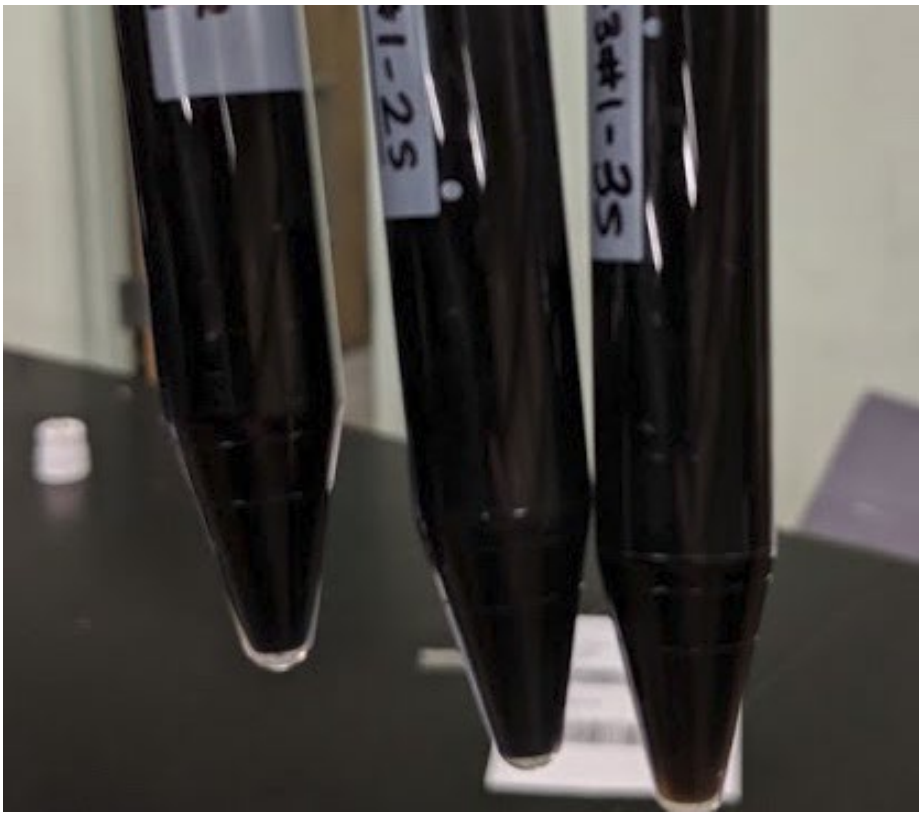
Award Number: SC0018561

Project Duration: 04/09/2018 – 01/08/2019

Total Project Value: $150,000

Key Technology Area: Separation Technologies

The project team will demonstrate the dissolution of coal in specially designed ionic liquids (ILs) for the liberation of rare earths, identifying the candidates which show the best rare earth liberation and coal dissolution. Chitin-based sorbents will be surface-modified with specific ligands to remove rare earth elements (REEs) from the coal solution. The REEs will then be stripped into successive electrochemically stable ILs for electrodeposition of the mineral. The ILs and sorbents that allow for the most efficient rare earth extraction and recovery will be identified. This technology has the potential to strengthen the coal industry as well as domestic manufacturing of automobiles, electronics, military technology, and many other high-tech devices which are dependent on rare earths. In addition, this technology is also expected to reduce the potential environmental impact of rare earth processing.



Three sequential dissolutions of the same coal sample with fresh IL (1, 2, 3: left to right).

Abbreviations

ALB ..... airlift bioreactor

ALSX ..... acid leaching/solvent extraction

AMD ..... acid mine drainage

AML ..... abandoned mine land

Ca ..... calcium

CIC ..... continuous ion chromatography

CIX ..... continuous ion exchange

CO_2 ..... carbon dioxide

DOE ..... Department of Energy

EC ..... elution column

Fe ..... iron

FWP ..... Field Work Proposal

g ..... grams

IL ..... ionic liquid

LANL ..... Los Alamos National Laboratory

LBT ..... lanthanide binding tag

LIBS ..... laser-induced breakdown spectroscopy

MAB ..... mechanically agitated bioreactor

Mg ..... magnesium

Mn ..... manganese

NETL ..... National Energy Technology Laboratory

pH ..... potential of hydrogen

PLS ..... product laden solution

ppm ..... parts per million

PSI ..... Physical Sciences, Inc.

RD&D ..... research, development, and demonstration

REE ..... rare earth element

RES ..... Rare Earth Salts

REYSc ..... REE-rich concentrates including yttrium and scandium

sFGD ..... stabilized flue gas desulfurization material

Si ..... silicon

SO_4 ..... sulfate

Ss ..... sludge solids

tpd ..... tons per day

TREE ..... total rare earth elements

U.S. ..... United States

UK ..... University of Kentucky

UND ..... University of North Dakota

Notes

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Notes

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http://edx.netl.doe.gov/ree

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