Recovery of Rare Earth Elements (REEs) from Coal Ash with a Closed Loop Leaching Process

2017 Rare Earth Elements Portfolio Review

DE-FE-0027012

Period of Performance March 1, 2016 – August 31, 2017

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March 22, 2017

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Project Goals and Objectives

- Validate economic viability of recovering Rare Earth Elements (REE) from coal byproducts using Battelle's closed loop Acid Digestion Process
 - Analyze and assess potential sources of coal byproducts as process feedstocks
 - Perform a techno-economic assessment based upon preliminary laboratory testing and process modeling
- Design a bench scale closed loop process for REE recovery from coal byproducts



Presentation Outline





Project Overview

- Battelle proposed demonstration of a bench-scale technology to economically separate, extract, and concentrate mixed REEs from coal and coal byproducts (AOI 1)
 - Uses a closed loop acid leaching process incorporating the Acid Digestion Process for acid recovery
 - Targets coal sources with >300 ppm REE by weight
 - Target concentration of >2% by weight for recovered REEs
- Award No. DE-FE0027012
 - \$900,014 (\$710,000 Federal; \$190,014 OCDO)
 - PoP = March 1, 2016 to August 31,2017





Project Partners

- Battelle has identified project partners with the ability to support identification and procurement of high REE content coal samples
 - The Ohio Coal Development Office includes key strategic partners such as AEP and can provide access to coal ash samples and critical information for the economic feasibility assessment
 - Pennsylvania Bureau of Topographic and Geologic Survey will provide coal samples from their inventory for characterization
 - West Virginia Geological and Economic Survey will similarly provide coal samples from their inventory for characterization



– a Prospectivity Analysis



Technical Approach

- Proposed technology: concentration of rare earth oxides and recovery of leach acid
- Patented Acid Digestion Process previously demonstrated for metal leaching and acid recovery at 70 lb/hr
- Synergistic with current DOE/OCDO Project for direct coal liquefaction



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Sampling and Characterization Results



Three Coal Ash Sources were Investigated





PCC Fly Ash Tended to Have Higher REE+Y+Sc Concentrations than Bottom Ash







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Fluidized Bed Combustor Ash was Diluted in Calcium

	Calcium	Total	Total	HREE	LREE	HREE/L
		REE	REE+Y+Sc	+Y	+Sc	REE
	Weight Percent (%)	ppm	ppm	ppm	ppm	Ratio
Fluidized Bed Combustor Fly Ash	18.1%	147.5	188.1	38.6	149.5	0.26
Fluidized Bed Combustor Bottom Ash	13.7%	121.4	153.2	30.5	122.7	0.25

The low REE concentration in FBC is diluted in Limestone used to precipitate SO₂, Reduce NOx and improve the heat transfer



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Higher Temperatures Lead to Formation of Refractory Phases



Sample	Percent Crystallinity
Middle Kittanning Coal	5%
Liquefaction Residual	7%
800°C Ash	35%
1200°C Ash	42%



Battelle's Direct Coal To Liquids (CTL) Separates Organic and Mineral Material





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Coal Liquefaction Increased TREE Concentration Over the Feed Coal on an Ash Basis

■ Feed Coal ■ Low Density ■ High Density ■ >850 micron ■ 600-850 micron ■ 355-600 micron ■ <350 micron



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Scandium Represents the Bulk of Material Value in the Fly Ash







Preliminary laboratory testing



Leaching Efficiencies Appear to Decrease at High Nitric Acid Concentrations

	Starting Nitric Acid Concentration in PCC Fly Ash Leaches						
Element	17%	17%	17%	34%	51%	68%	34%
							(milled)
Sc	19.2%	20.8%	21.5%	21.5%	N/A	N/A	55.3%
Υ	24.6%	26.7%	28.0%	28.0%	14.9%	13.0%	46.9%
La	19.0%	19.3%	20.0%	19.0%	9.9%	8.2%	35.4%
Се	21.0%	21.5%	21.7%	27.0%	11.9%	9.9%	34.0%
Pr	20.3%	21.7%	22.4%	22.9%	11.6%	10.0%	36.3%
Nd	20.8%	22.6%	23.4%	23.9%	12.3%	10.5%	39.5%
Sm	22.5%	24.0%	25.0%	25.4%	13.7%	11.8%	40.5%
Eu	22.7%	24.5%	25.4%	26.4%	14.8%	12.7%	42.4%
Gd	25.0%	27.2%	28.5%	28.8%	15.7%	13.7%	45.2%
Tb	23.3%	25.5%	26.9%	28.1%	15.4%	13.4%	44.3%
Dy	24.1%	26.2%	27.6%	28.6%	15.5%	13.0%	41.9%
Но	24.6%	26.8%	28.0%	28.6%	15.2%	13.3%	41.8%
Er	23.8%	26.2%	27.5%	27.8%	14.8%	12.6%	43.8%
Tm	23.0%	25.2%	26.4%	26.9%	14.4%	12.0%	42.2%
Yb	21.2%	23.1%	24.7%	24.8%	12.9%	10.6%	36.3%
Lu	21.2%	22.6%	23.9%	24.3%	13.0%	10.2%	34.6%

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Serial Acid Contacts with Ash Decrease Leaching Rates





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Conceptualization of REE purification train.





Compressive Strength Testing of Concrete Suggests Leached Ash Pozzolan Does not Adversely Affect Concrete Strength.

Sample	Description	Cement (wt%)	Ash (wt%)	Sand (wt%)	Water/Cement weight ratio	Failure Pressure
	Concrete with post leached fly					
Α	ash	77	19	4	0.5	3,420 psi
В	Concrete with fly ash	77	19	4	0.5	3,420 psi
	Concrete without addition of fly					
C	ash	77	0	23	0.5	3,250 psi







Feasibility study



CHEMCAD Simulation Has Been Developed to Model Proposed Plant

- Model simulates a 30 tonne/day process.
 - Sized to reflect full capacity of a typical coal plant.
- Laboratory testing results incorporated into process model:
 - Species leaching efficiency
 - Acid loading capability
 - NOx generation rates
 - Acid roasting recovery estimates
- Model results used to inform equipment sizing parameters within TEA.





Approach to TEA follows EPRI's TAG® method





TEA results for FOAK and NOAK REE Recovery Plant

First-of-a-Kind Plant					
Cost Component	\$Million per year (2015)	\$/tonne Coal Ash Processed			
Annual Fixed Cost	\$5.4	\$29			
Annual Variable Cost	\$5.2	\$28			
Annualized Capital Cost	10.0	\$56			
By-Product Credits	\$(2.1)	(\$11)			
Total Annual Revenue Requirement	\$19.1	\$103			
Requirement					

Nth-of-a-Kind Plant					
Cost Component	\$Million per year (2015)	\$/tonne Coal Ash Processed			
Annual Fixed Cost	\$3.6	\$20			
Annual Variable Cost	\$5.2	\$28			
Annualized Capital Cost	4.7	\$26			
By-Product Credits	\$(2.0)	(\$11)			
Total Annual Revenue Requirement	\$11.6	\$62			



Revenue requirement for FOAK and NOAK plants compared favorably to value present in coal ash.





Next Steps

- Integrated Process Design
 - Generation of Laboratory Test Plan
 - Laboratory testing to inform process design
 - Generation of design package for 100 lb/day system





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Selection of Analytical Method for Sample Characterization



Mean Distance from NIST Value as Percentage

Comparison of Sodium Peroxide vs. Lithium Borate digestion using NIST sample

Sodium Peroxide digestion method gives a closer result to the NIST reference material than Lithium Borate digestion

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