

AOI 3: At-source Recovery of Rare Earth Elements from Coal Mine Drainage



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NETL REE Review
Pittsburgh PA
10apr19
1430h

Project Objective



ETD 53 DE FE0031524

Develop and test a process to extract an enriched REE feedstock from raw Coal Mine Drainage

Sub-objective 1. Extraction strategy Case A, low pH AMD: Maintain reducing conditions in feed water. Determine the effect of titrating pH from 3.0 to 4.5. Collect precipitate for analysis.

Sub-objective 2. Extraction strategy Case B, net alkaline AMD: Maintain reducing conditions in feed water, explore electro-membrane extraction methods to separate REEs from matrix.

Sub-objective 3. Process resulting precipitates through our Solvent Extraction facility to a target REE oxide purity in excess of 90% and evaluate performance versus the AMD sludge feedstock pathway.

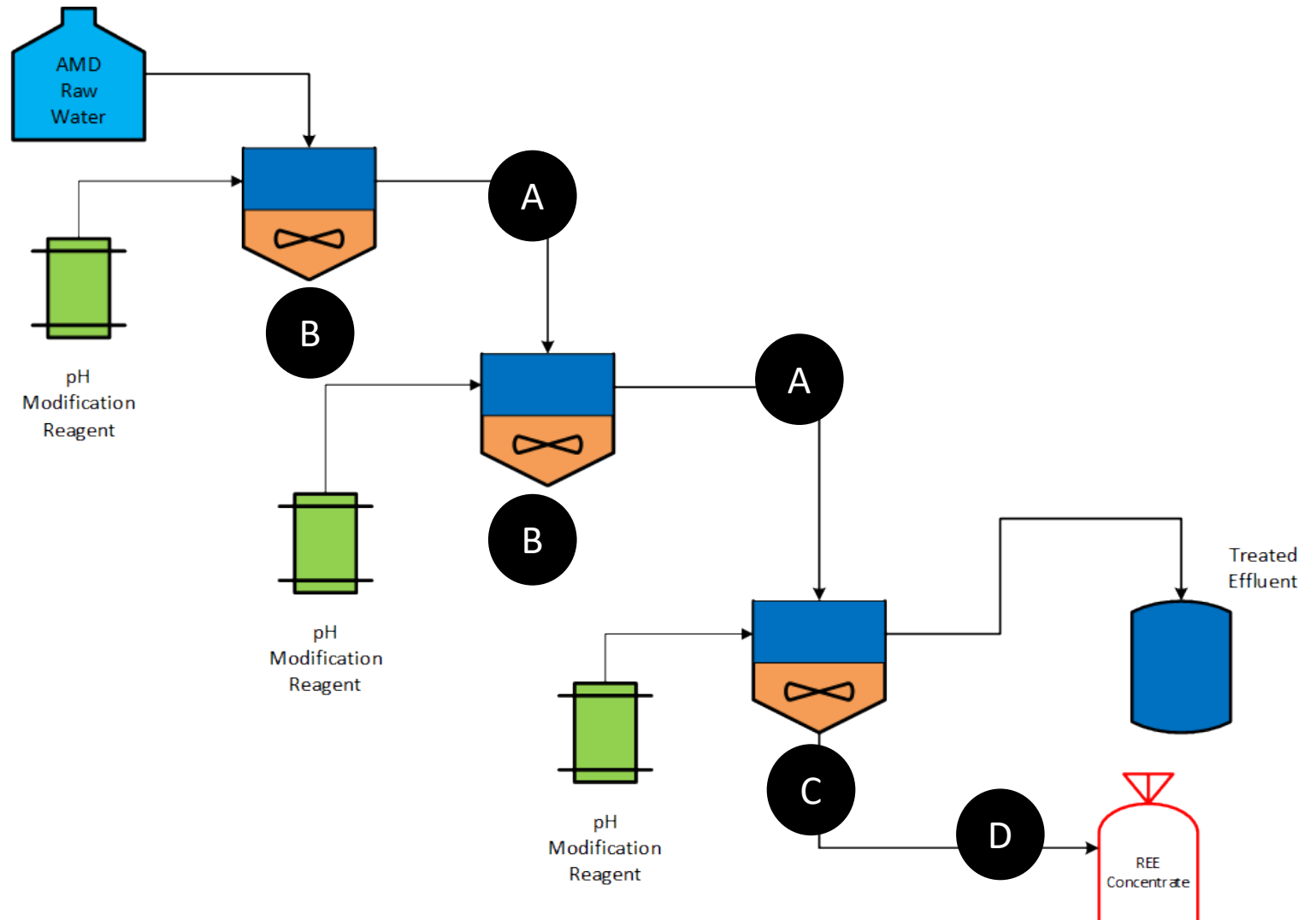
Sub-objective 4. Evaluate technical and economic advantages over at-source REE recovery versus the AMD sludge feedstock pathway.

At-source REE concentration recovery

Mobile plant

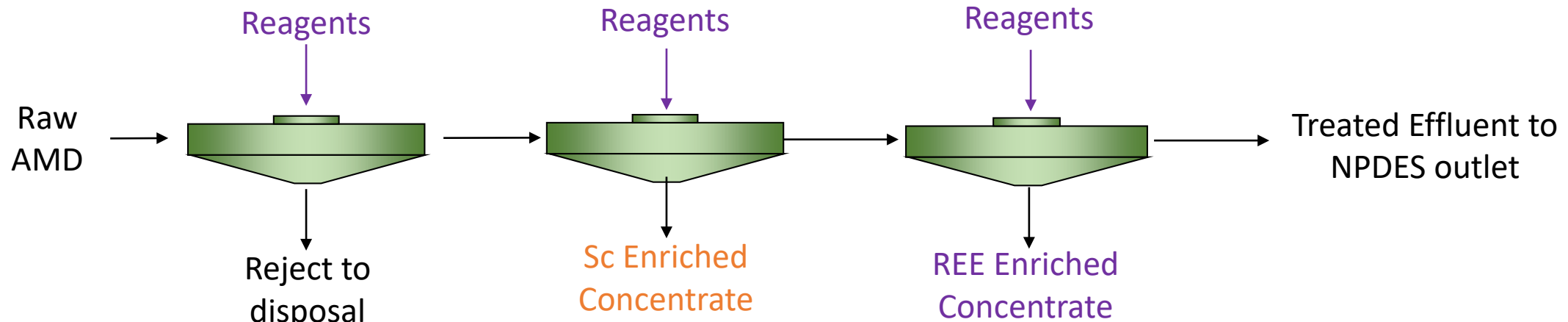
ETD53-AMD Concentrate Preparation

- A. Particulate removal (P&F)
- B. Gangue removal
- C. REE precipitation
- D. Dewatering



Integration with AMD treatment Plant

Process currently in development



Concentrate dewatering

→ Solids to Solvent Extraction

WVDEP's Omega AMD treatment plant

Treatment plant with clarifier

Dewatering unit



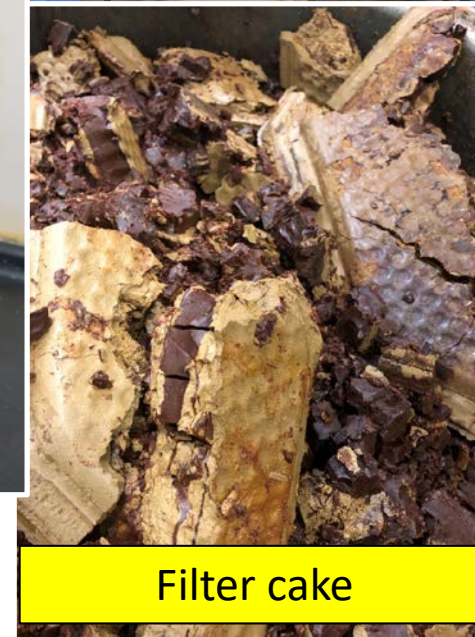
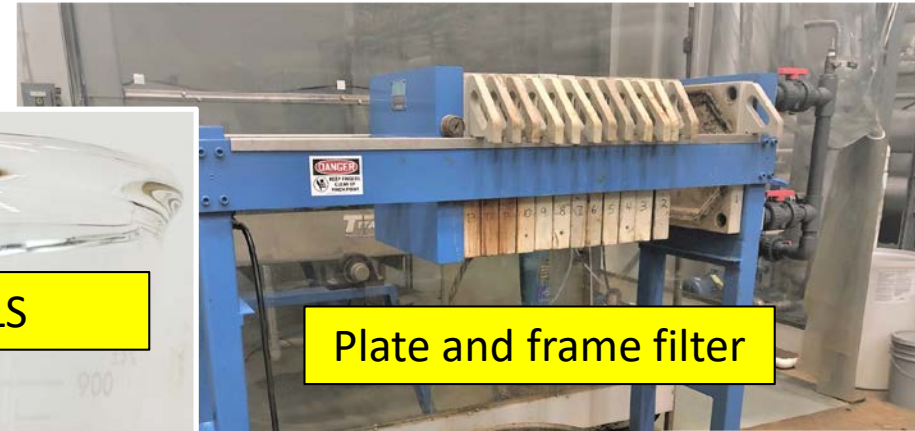
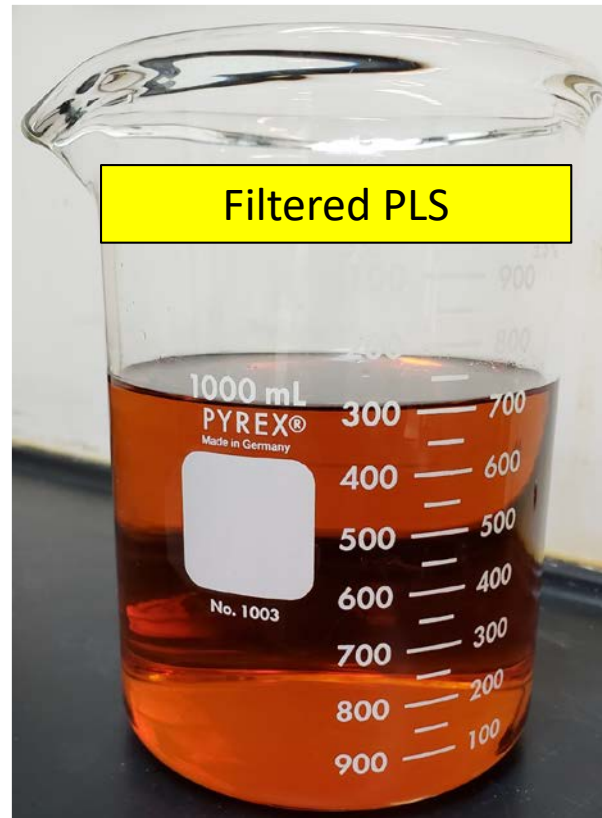
Acid Leaching

Filtration

PLS generation and filtering are becoming routine and we are comfortable with the process.

Filtering was initially an issue; however, the pressure filter has solved 95% of filtering issues.

Continuing work to maximize REE recovery in the leaching process while rejecting gangue metals.

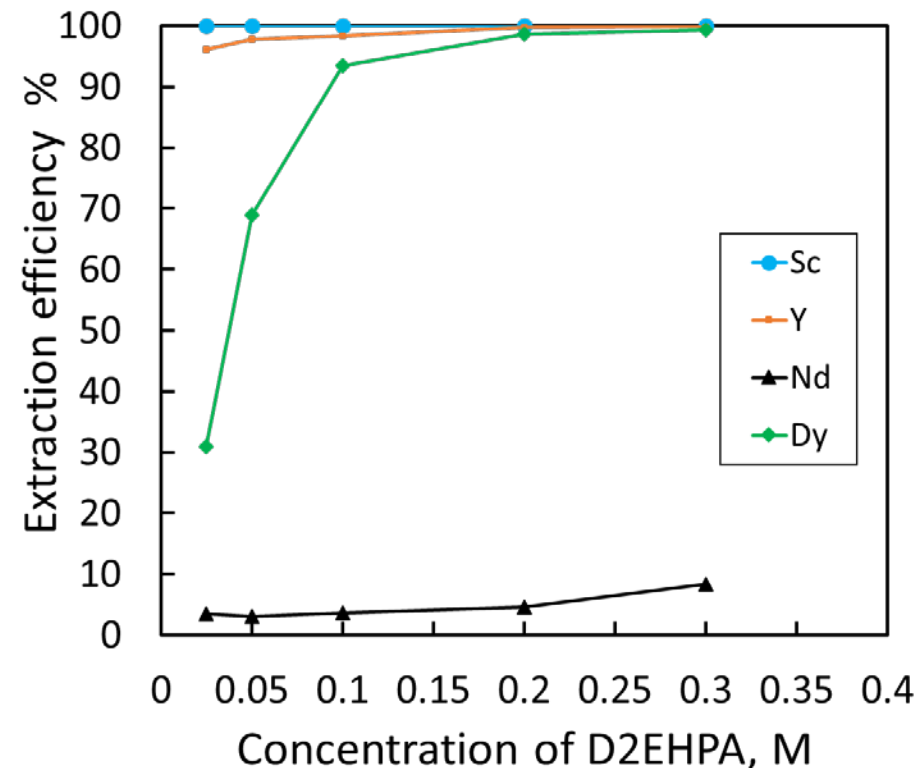


Parametric SX Study

Shushu Liu and Aaron Noble, Virginia Tech

- 1) extraction of the four REEs with D2EHPA follows the order of $\text{Nd(III)} < \text{Dy(III)} < \text{Y(III)} < \text{Sc(III)}$, which is consistent with their order of basicity [1].
- 2) extraction efficiency increases with increasing extractant concentration and the initial pH.
- 3) The addition of the modifier facilitates the extraction of REEs significantly without changing the extraction order of the four REEs with D2EHPA.

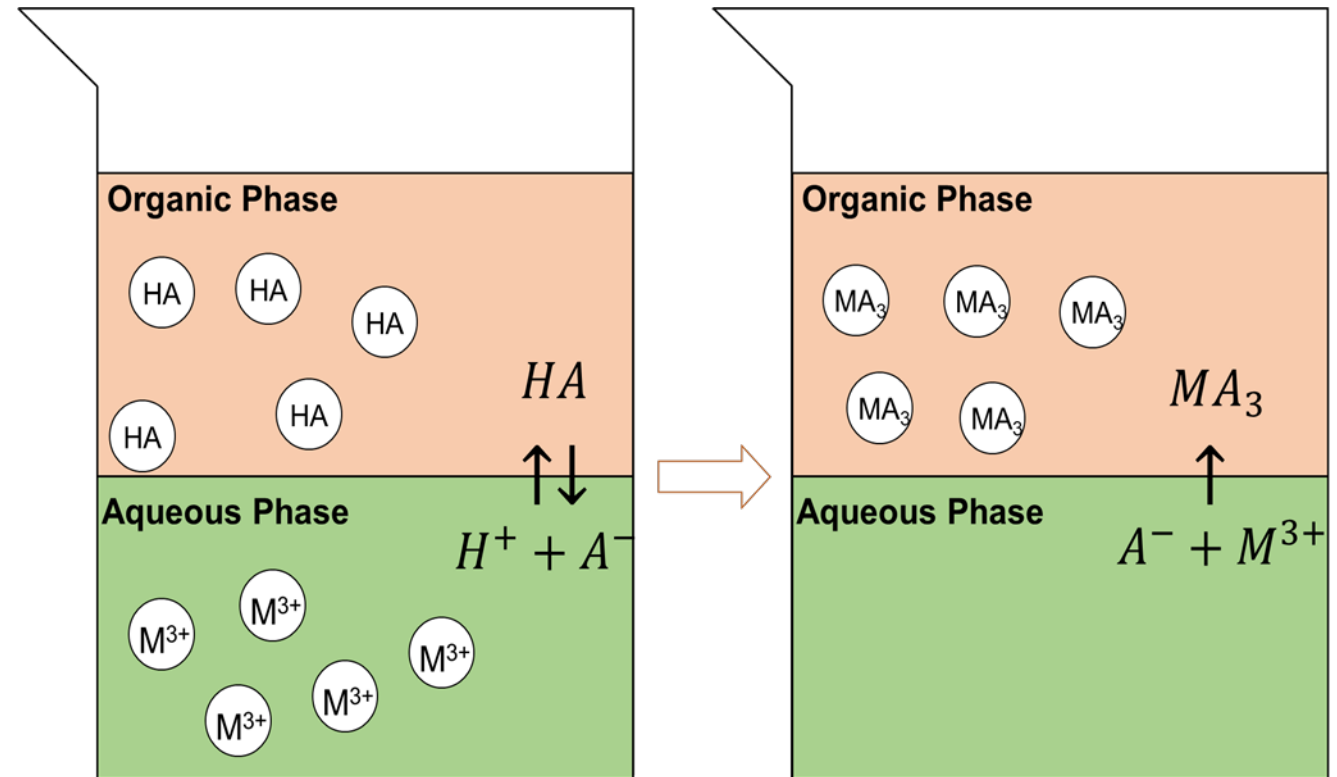
The optimum solvent extraction conditions were identified. While the study identifies a promising method for screening extraction conditions, further study with complex matrices will be undertaken to test the method's validity under realistic operating conditions.



Downstream Processing

Solvent Extraction

- HA = D2EHPA
- M^{3+} = REE ion
- MA_3 = D2EHPA/REE complex



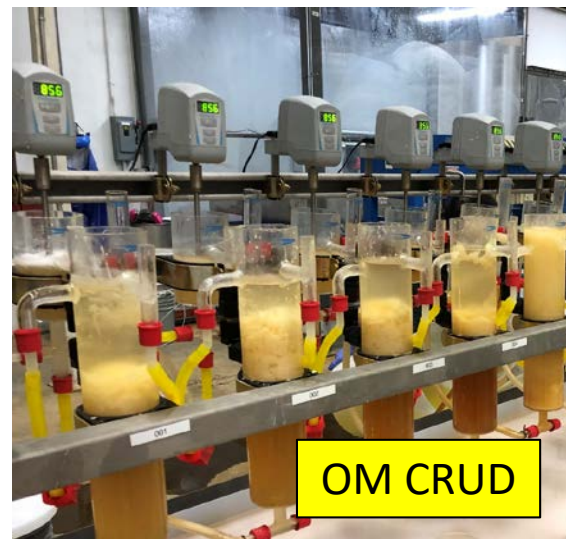
Solvent Extraction

Crud=Chalk River Unidentified Deposit
origins in actinide separation

Conducted shakedown testing for
all 3 feedstocks.

DLM & OM cause significant
buildup of CRUD in the ALSX
system resulting in poor mass
balances and organic loss.

RS feedstock runs relatively CRUD-
free.

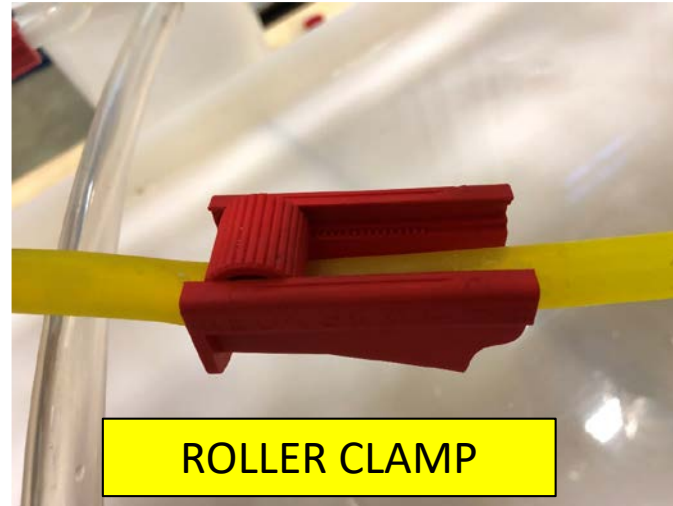


Solvent Extraction

Automation of Mixing O:A Ratio

Currently, the O:A in the mixing chambers is controlled by a roller clamp that recycles fluid from the settler back to the mixer. The O:A ratio in the mixer is important to ensure proper mass transfer. This method is functional, but labor intensive and relies on analog adjustments.

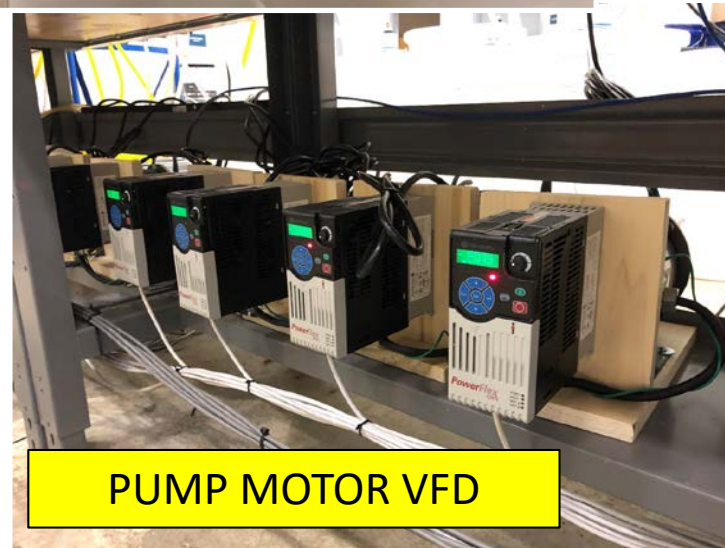
Rockwell is working to install torque sensors on the mixers. This data will allow the roller clamps to be replaced by VFD controlled peristaltic pumps so the mixing ratio will be continually optimized.



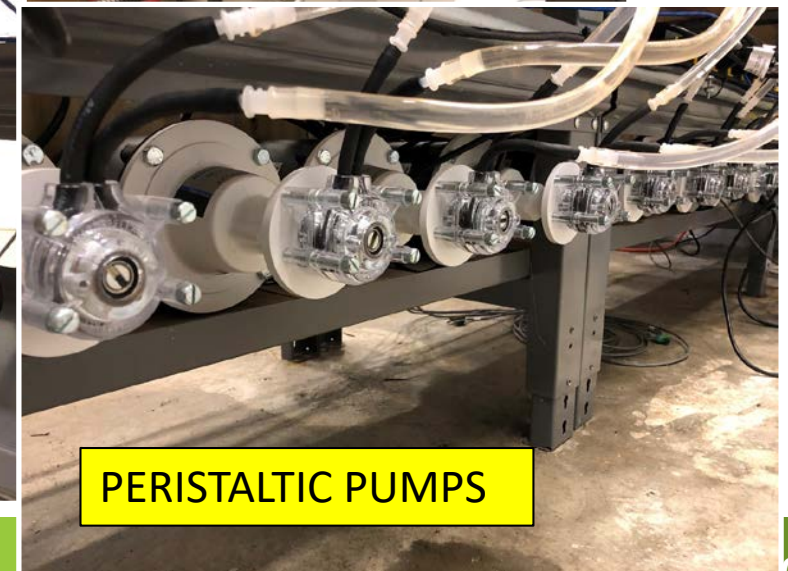
ROLLER CLAMP



MIXER AND AMPERAGE SENSOR



PUMP MOTOR VFD



PERISTALTIC PUMPS

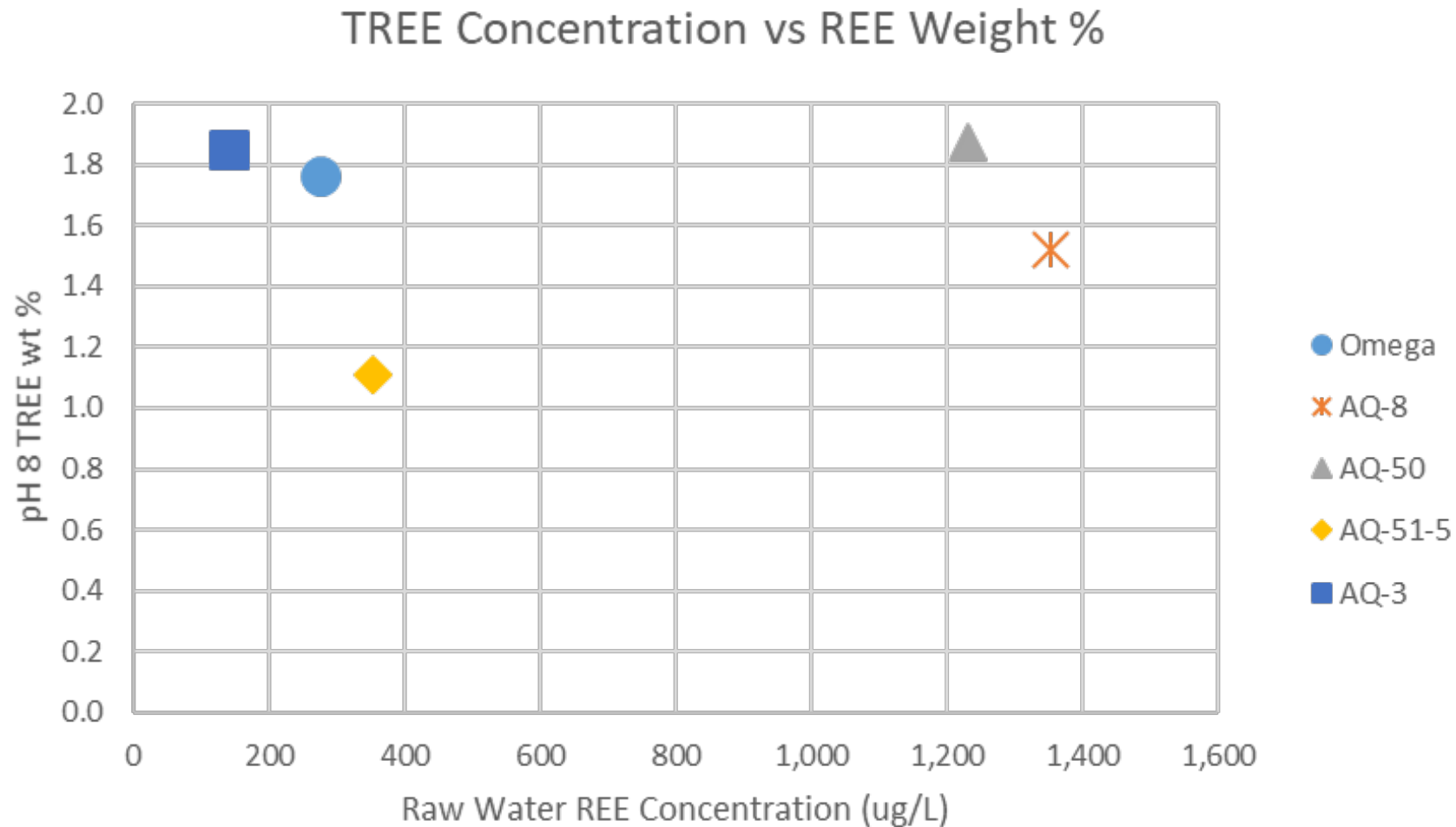
Concentration over multiple sites

Laboratory studies

site	raw	raw	MREO grade		sludge enrichment factor
	AMD μg/L	Sludge g/t	after treatment g/t	%	
AQ 2 1	352	513	20,772	2.08	40
AQ 8 1	2353	405	31,594	3.16	78
AQ 50 1	2119	1471	22,010	2.20	15
AQ 51 5	738	2138	13,030	1.30	6
	1391	1131	21,851	2.19	19

Effect of raw water REE concentration

Little effect on concentrate grade



Integration with AMD treatment

Addition of flocculent increases recovery, decreases grade

- 2 Step pH adjustment vs 3 step
 - 3 step showed slight decrease in Al and large decrease in Fe concentrations
- Floc effect
 - Increase in REE recovery (97% vs 83%)
 - Decrease in grade 1.77% vs. 0.56%

Solvent Extraction

Precipitation

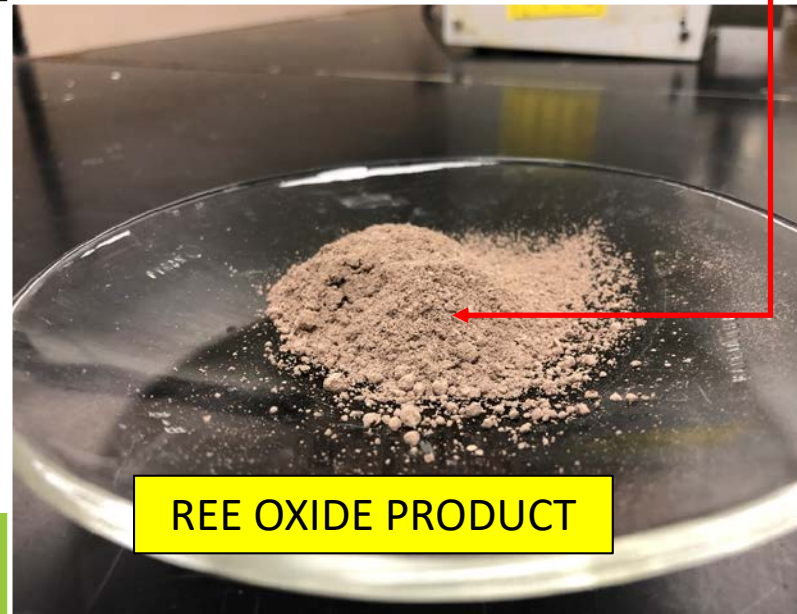
3 products have been produced from the 3 initial shakedown tests.

DLM @ 80% TREO

OM @ 47% TREO

RS @ Analysis Pending

Working on precipitation procedures to standardize and maximize recovery and grade of REE product.



Concentrates/Dewatering

0.59% MREO

West Virginia University: DE FE 0031524

Solid product from staged precipitation trial

	Elemental conc.		Rare earth oxide	
	REE g/t	TREE %	REO g/t	TREO %
Sc	70.4		82.9	
Y	1,521.1		1,789.6	
La	243.7		286.7	
Ce	903.8		1,063.2	
Pr	147.0		172.9	
Nd	711.5		837.0	
Sm	207.5		244.1	
Eu	53.9		63.4	
Gd	324.5		381.7	
Tb	59.1		69.5	
Dy	355.5		418.3	
Ho	69.6		81.9	
Er	188.5		221.7	
Tm	25.3		29.7	
Yb	141.5		166.4	
Lu	21.0		24.8	
total	5,043.9	0.50%	5,934.0	0.59%



Scandium recovery

Modifier transfers Sc to solid phase

Without SX Modifier



With SX Modifier



Scandium Stripping Tests Sc Precipitate

	Distribution of <u>Sc(III)</u> , %		
	Organic	Emulsion	Aqueous or Precipitate
w/o Modifier	14.85	61.15	23.99
w/ Modifier	24.96	--	75.04

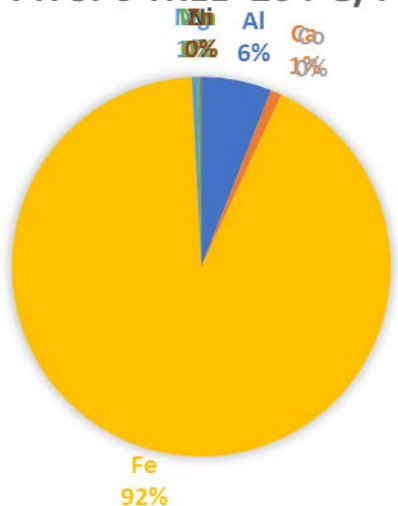
- The addition of a simple modifier resolved BOTH the scandium stripping issue and an emulsion issue.

Gangue rejection

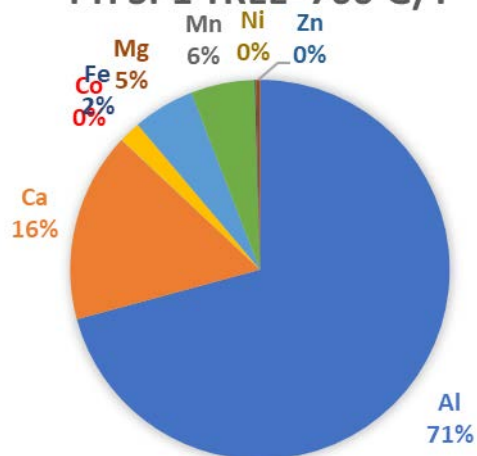
Major metals

REEs

PH SP0 TREE=204 G/T

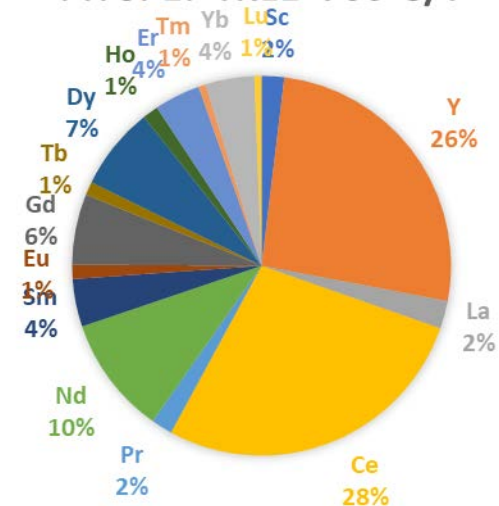


PH SP1 TREE=766 G/T

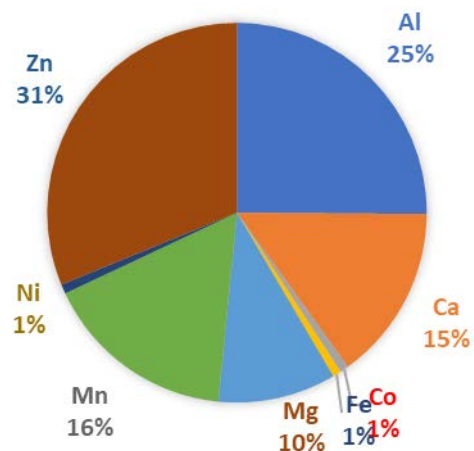


Sc precipitates at pH sp 1

PH SP1: TREE=766 G/T

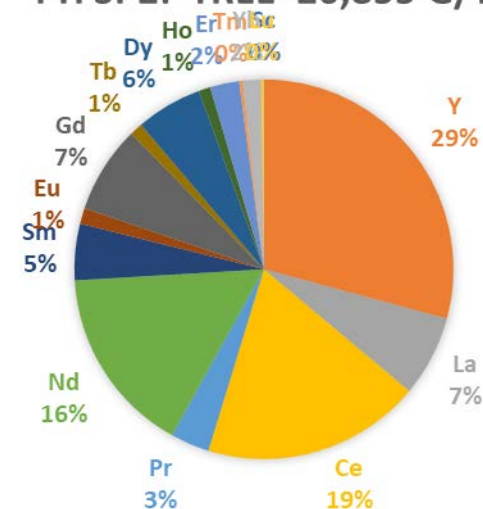


PH SP2: TREE=26,855 G/T



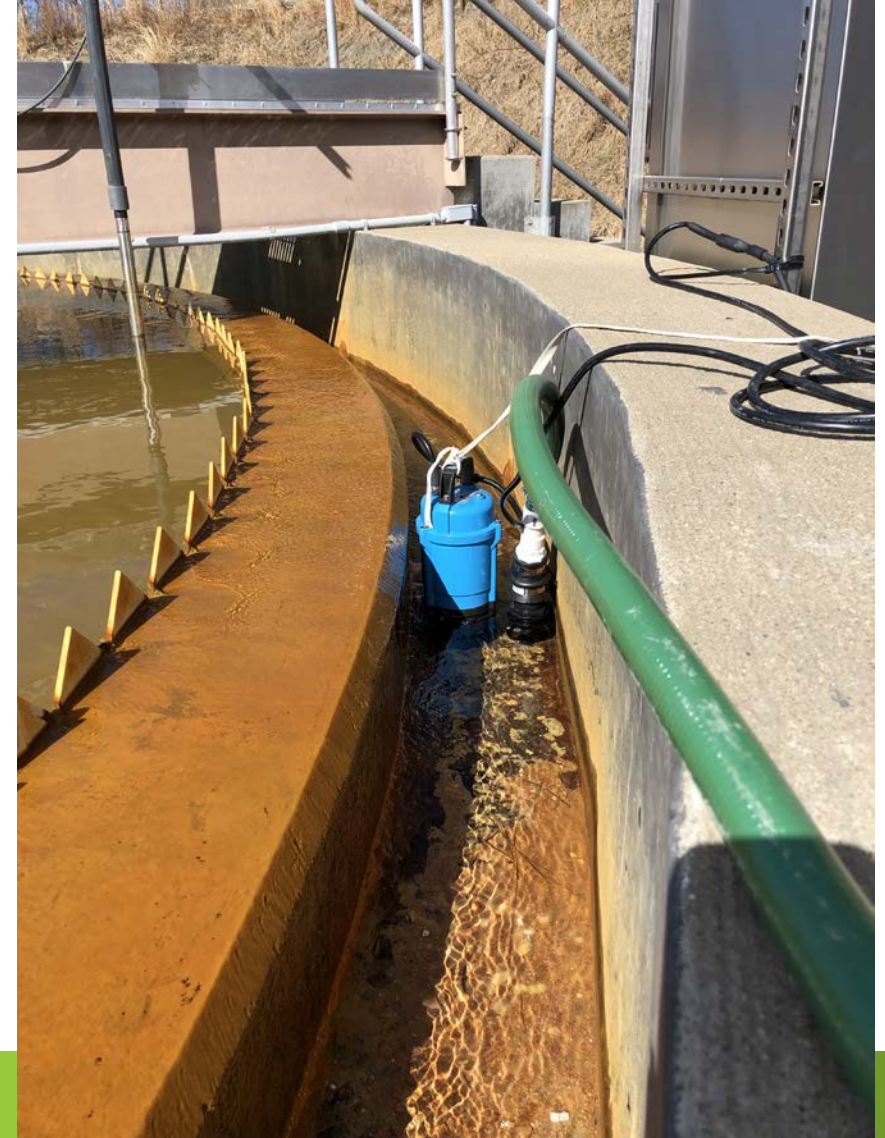
Zn dominates gangue at pH sp 2

PH SP2: TREE=26,855 G/T



Mobile plant for field extraction

Integrated with WVDEP's Omega AMD treatment plant



Interior of mobile plant

Two plate and frame presses



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Process train developed and under testing

Sub-objective 2. Extraction strategy Case B, net alkaline AMD: Maintain reducing conditions in feed water, explore electro-membrane extraction methods to separate REEs from matrix.

Insufficient throughput with SLMs or adequate separation via redox/pH control: terminated

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Process train developed and under testing

Summary of progress to date

Transition from laboratory to field scale studies

- A process pathway has been identified for the treatment of net-acid AMD. The process isolates and concentrates REEs and naturally captures scandium in a separate stream.
- Process has been tested at four AMD sites. Results range from 1.1 to 2.7% REE DW.
- The products from this process will be separated using solvent extraction (SX). Laboratory testing is now refining those SX processes.

Risk: REO, SREO

High confidence

- Regional/local resource dimension
- Prediction based on site conditions
- REE distribution
- MREO grade > 80%

Low confidence

- Elemental separability through ALSX
- REO distribution through ALSX
- Processing costs
- Market
- Valuation

Latest Quandaries

OM precipitated from strip solution (elemental)



- Sc, Co have high affinity for our extractant
- How to liberate
- What is the unknown?

	g/t
Sc	-
Y	187,055
La	7,977
Ce	41,011
Pr	5,916
Nd	30,172
Sm	12,922
Eu	3,839
Gd	24,010
Tb	5,235
Dy	35,023
Ho	7,116
Er	21,652
Tm	3,246
Yb	17,336
Lu	2,141
TREE	404,649
MREO	476,058

	g/t
Al	17,728
Ca	91,613
Co	127
Fe	41,559
Mg	136
Mn	409
Na	-
SO4	-
Si	3,980
	155,552
Th	14
U	62
	76
known	560,278
unknown	439,722

Questions?

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Quandary
Canyon