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Economic Analysis of Rare Earth Elements Extraction from Clay Waste

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May 1, 2017
Dr. Robert Counce
Department of Chemical and Biomolecular Engineering
University of Tennessee
424 Dougherty Engineering Building
Knoxville, TN 37996

Dear Dr. Counce:

We are submitting the attached report entitled Economic Analysis of Rare Earth Elements Extraction from Clay Waste.

This report examines the results of designing a process and estimating the capital and manufacturing costs for the extraction of rare earth elements from the wet process for phosphoric acid production.

I hope you find this report satisfactory.

Sincerely,
Seth Archambault, Jacob Lockhart, Tiffany Onwu, Clifford Swanson

Honors Design in Green Engineering
University of Tennessee
Knoxville, Tennessee

Enclosure: Final Report

cc: Thomas Gaetjens

Economic Analysis of Rare Earth Elements Extraction from Clay Waste

CBE 488: Honors Design in Green Engineering

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Jacob Lockhart
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Submitted:
April 28, 2017

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1.0 Introduction

This report will document the results of the study to design a process and analyze its economics for the extraction of rare earth elements (REEs) from the wet process for phosphoric acid (H_3PO_4) production. In the wet process being analyzed, phosphoric acid is produced using concentrated sulfuric acid and multiple stirred reactors. The byproduct streams consist of sand, clay, phosphogypsum, and sludge. Each of these streams contain varying amounts of REEs. During the wet process for H_3PO_4 production, the ore is separated into phosphate rock and waste streams of clay and amine flotation tails (sand). Through many mining processes, large amounts of clay waste are produced. Since REEs are used for a variety of products, the recovery of REEs from phosphoric acid by product streams could potentially provide additional economic value in industry.

The process originated through efforts by Florida Industrial and Phosphate Research Institute (FIPR) to recover these valuable REEs and make the process profitable. The economic viability of this process will be estimated by determining the capital cost as function of equipment cost and manufacturing cost [9]. The process scale is 4 million tonnes of clay processed annually. The yearly profit will then be evaluated as a function of REE conversion from phosphate form to oxide form.

The primary design objective was to develop and compare flow sheets, capital costs, and the intermediate operating cost estimates for a process to recover REEs from the clay stream exiting the dissolution step of the wet phosphoric acid process. The purity of the product required low concentrations of non-Lanthanide (Ln) metals and low radioactivity. The Ln concentration of the product was required to be 50wt%. With a ChE index of 543, this report aims to focus on recovering rare earth elements from the clay waste stream. It will also display the approach that

involves analyzing the economics of REE extraction, estimating for the capital and operating costs, and making recommendations for REE profits.

Contributions to this study were provided by Critical Materials Institute (CMI; a US Department of Energy (DOE) energy innovation hub), Oak Ridge National Laboratory (ORNL), the University of Tennessee (UT), Idaho National Laboratory (INL), and the phosphoric acid industry, with support from FIPR.

2.0 Synthesis Information for Processes

2.1 Overall Process Design

The production of phosphoric acid is accomplished by processing mined phosphate rock. It is in the waste streams of the phosphate process that the recovery of REE's seems promising. The different waste streams are clay, amine flotation tailings, phosphogypsum, and sludge. The process begins with the mining of the ore followed by separation into three streams of phosphate rock, clay waste, and amine flotation tailings.

For the REE recovery from clay, evaluated the process shown below. The steps of this process are as follows: (1) separation of solids based on particle size, where the coarse fraction is fed to the leaching vessel and the fine fraction sent for reclamation, (2) leaching of the REE phosphates into REE sulfates and separation of the solids from the stream, (3) removal of the REE sulfates out of the leaching product stream, (4) concentration of the REEs into our final product. This is a basic look at the process design and each step involves more intermediate steps.

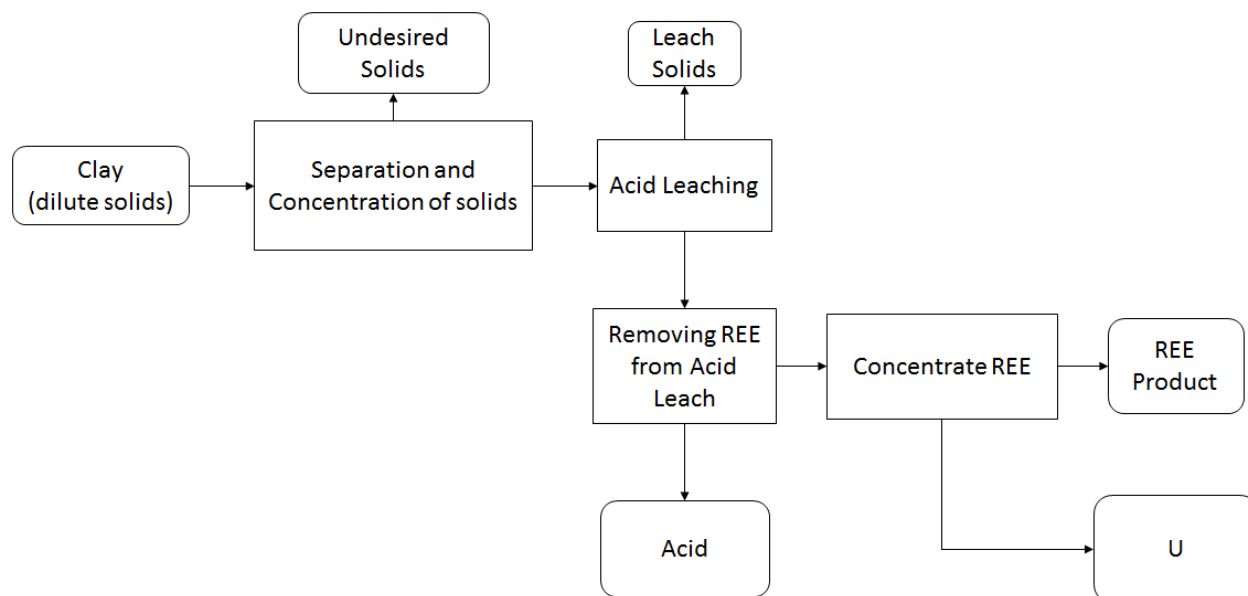
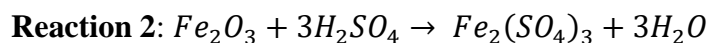
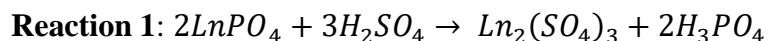


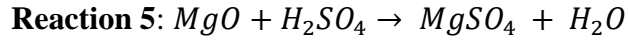
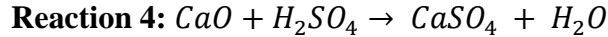
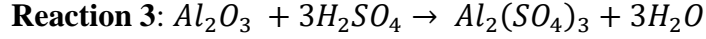
Figure 2.1: General Clay REE Recovery Process

2.2 Process Chemistry

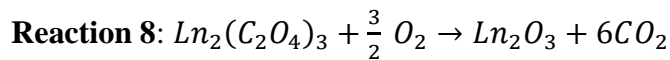
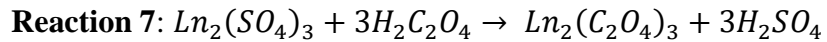
The production of phosphoric acid is carried out by the reaction below. The general process is that phosphate rock (represented by $\text{Ca}_5(\text{PO}_4)_3\text{X}$, where X is representative of OH, F, Cl, or Br) is reacted with sulfuric acid to produce phosphoric acid, phosphogypsum, and HX.

The waste stream we are processing is the clay waste, which is separated prior to the reaction described above. The reactions for our total process include the leaching reactions, the precipitation reaction, and the calcination reaction. The leaching reaction is essentially a reaction between sulfuric acid and REE phosphates (represented by Ln) to form phosphoric acid and REE sulfates. Similar reactions will also take place during the leaching process between sulfuric acid and Iron (III) oxide, aluminum oxide, magnesium oxide, and calcium oxide. Also, during this process a reaction occurs between phosphorus pentoxide and water to form phosphoric acid.





The leaching step is followed by the separation of $Ln_2(SO_4)_3$ from the metal sulfates. This step is carried out by a series of staged liquid-liquid extraction and stripping. The quantitative model for extraction and stripping is given by the Kremser equation from Geankoplis, where the distribution coefficients are given in Table E.2, in appendix E. This step is followed by precipitation and calcination to obtain the final product. Below are the reactions for precipitation and calcination. The precipitation reaction is a reaction between oxalic acid and the REE sulfates to form REE oxalate. This reaction is important because REE oxalate is insoluble in water and therefore can be separated out. The calcination then converts the REE oxalate into REE oxide using oxygen at a high temperature, releasing carbon dioxide as a byproduct. These reactions are shown below respectively.



2.3 Literature Summary

Rare earth elements (REEs) are a unique set elements that consist of fifteen lanthanide elements and yttrium, and are so called “rare” because most of them were originally isolated in the 18th and 19th centuries as oxides from rare minerals. Due to their reactivity, REEs are difficult to extract and refine to pure metals. Each of these elements have unique characteristics that make them useful for a variety of purposes. All the REEs were identified in the 20th century, and commercial markets for most of them arisen in the past fifty years.

REEs exist in multiple forms, such as halides, oxides, carbonates, and silicates. While most of these elements are not as uncommon as the name implies, they are simply uncommon in commercially viable concentrations. Cerium, for instance, is more abundant in the earth's crust than copper or lead. Furthermore, many REEs are more common than tin and molybdenum, and all but promethium are more common than silver or mercury. The element praseodymium is used to create strong metals for use in aircraft engines. Praseodymium is also a component of a special sort of glass, used to make visors to protect welders and glassmakers.

In addition to a world of increasing technological innovations, REEs also serve as components of efficient lighting and display panels. Lanthanum is used in camera and telescope lenses. The compounds containing lanthanum are used extensively in carbon lighting applications, such as studio lighting and cinema projection. With expected rise in cinematography and automotive industry, and as the future progresses, the demand for REEs will continue to increase.

2.4 Basic Process Economics

In this study, our main goal was to analyze the economic viability of REE recovery from the clay waste stream. We looked at many different scenarios based on the conversion of REEs into REE oxides. The variables used in our economic analysis include the overall REE phosphate conversion to REE oxide form (our final product), the design variables of the individual equipment cost variables (generally the cost of purchasing and operating the equipment), and the conversion of our sellable byproducts. The cost information for our products, byproducts, and raw materials is shown below in Table 2.1.

Cost Assumptions	
Material	Cost/Unit
REE Oxide	\$10.00/kg
Phosphoric Acid	\$0.50/kg
Sulfuric Acid	\$0.05/kg
Oxalic Acid	\$0.70/kg
Isopar L	\$2.00/kg
Ligand A	\$60.00/kg
Feed Oil	\$0.07/lb
Natural Gas	\$2.73/GJ

Table 2.1: Material Cost Data

3.0 Method of Approach

The following steps explain how the REEs are extracted from a clay stream. First, the clay stream is fed into the hydrocyclone, where it is separated into a fine stream and a coarse stream. The coarse stream is sent to a flotation tower, where air and wash water enter the tower and separate the coarse clay into a waste tailings stream and a concentrate stream that will be fed into the leaching vessel. During the leaching step, the sulfuric acid enters the leaching vessel and converts the REE phosphates into REE sulfates. This leachate is then filtered to remove insoluble impurities. Next, the filtered leachate is pumped into a solvent extractor to separate the REEs from the phosphoric acid produced via leaching. A second extractor is then used to strip the REEs from the organic solvent back into the aqueous phase. The REE sulfates are then sent to a precipitator where they are converted to insoluble REE oxalates. The REE oxalates are sent through a second filter to separate them from water. The REE oxalates are then fed into a calciner, where they react with air at high temperature to produce REE oxides.

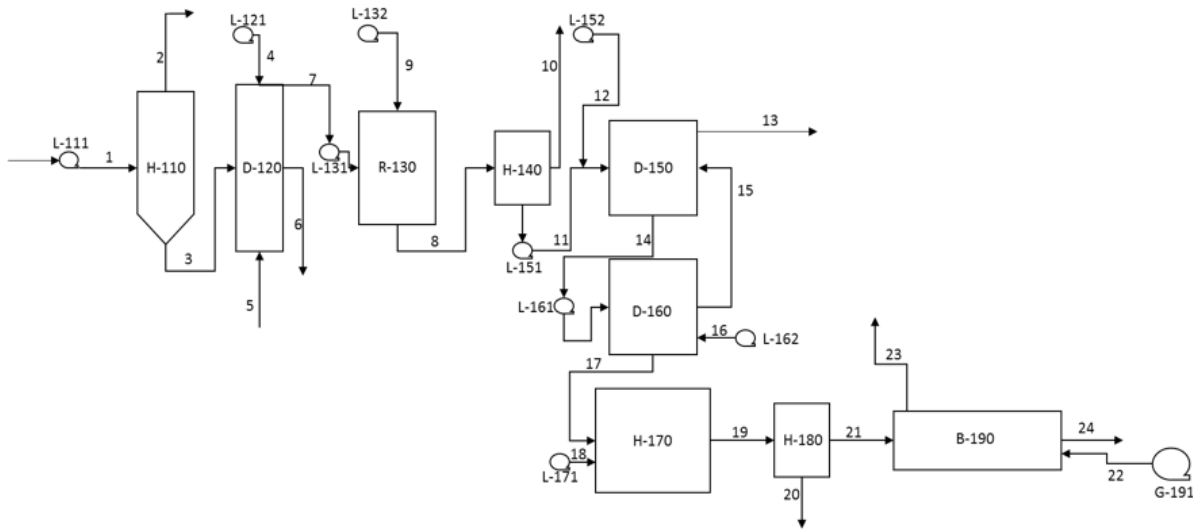


Figure 3.1: Flow diagram for REE extraction from clay stream.

3.2 Sustainability, Environmental, and Worker Safety

The plant design was made considering sustainability and environmental impact. The overall plant was expected to be a sustainable process. The reason is that the plant gets the raw materials from the waste of an existing plant, leading to the assumption that this plant will be sustainable for as long as the phosphoric acid plant is running. The process does produce a few waste streams, such as a slightly acidic waste stream from the second vacuum filter, a solid waste stream from the first vacuum filter, a clay waste stream from the hydrocyclone, and a tailings stream from the flotation tower. Despite there being several waste streams, there is not a large environmental impact that is not already present. This can be seen from the fact that every waste stream contains mining waste that is already being released by phosphate mining and the phosphoric acid plant. The only other possible source of environmental contamination is the effluent air from the calcination process due to some residual acid that gets vaporized and carbon dioxide production. An analysis showed that the annual acid release is less than 5kg/year. It is also determined that at our operating conditions, the plant produces less than 1000 tonnes of carbon

dioxide in an operating year. This is a very small environmental impact from the calciner, allowing us to assume that the proposed plant has a small environmental impact.

Another major consideration for designing this plant was worker safety. Looking at all the materials used in this plant the most dangerous aspects are the high amount of acids used and the temperature of operation for the leaching vessel and the calciner. Extra precaution is required for operators working with the calciner due to the extreme temperature it operates at. This may mean some special training and probably some warning system to keep the calciner separate from untrained persons. The rest of the plant will not be different than the existing phosphate plant, as it uses similar reagents as the proposed plant. This means that our proposed plant will adhere to the same safety measures as the phosphoric acid plant.

3.3 Product Quality

The next major design consideration was product quality. The quality is important as the better the quality of product and byproduct, the closer to selling prices from table 2.1 we can achieve. For product quality, we set a goal of greater than 50% by weight of elemental REEs. To go with this, we also had to consider the content of radioactive materials (i.e. the thorium and uranium content). The product had to contain less than 5kg of radioactive material for the production year. Alongside the product quality, we had to consider the byproduct quality, which was phosphoric acid. The major quality consideration was that the phosphoric acid must be at 50% concentration to be sold at our assumed phosphoric acid price. To meet these considerations the equipment had to be designed for specific separations, which affected their sizing and costing.

4.0 Results

A study of the economic potential of the process was performed to determine its viability. The economic potential was determined as a function of conversion of $\text{Ln}_2(\text{SO}_4)_3$ to Ln_2O_3 in four

different levels as described by Douglas in his paper. The level four economic potential study showed an economic potential range from \$37.6 million at 0% conversion of $\text{Ln}_2(\text{SO}_4)_3$ to \$39.7 million at the maximum possible conversion of $\text{Ln}_2(\text{SO}_4)_3$. The results of the economic potential study are shown below in Figure 4.1.

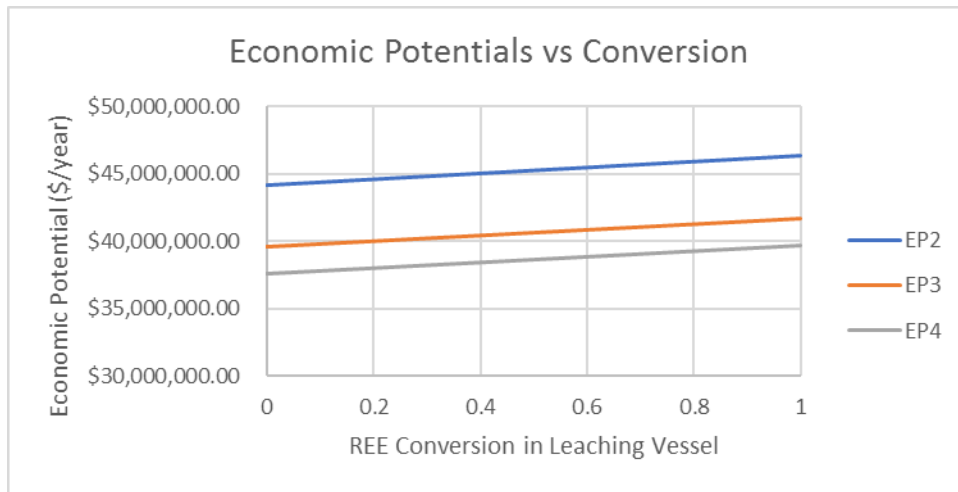


Figure 4.1. Economic potential vs conversion of $\text{Ln}_2(\text{SO}_4)_3$ to Ln_2O_3

4.1 Capital Cost Estimates

The equipment was sized based on the mass balance and the capital cost of each piece of equipment was determined based on the cost charts in Ulrich. Sample capital cost calculations can be found in Appendix D, and the capital cost of each piece of equipment can be found in the capital cost summary in Appendix C. The total capital cost of the plant was determined to be \$26,117,668. The capital cost distribution is shown below in Figure 4.2.

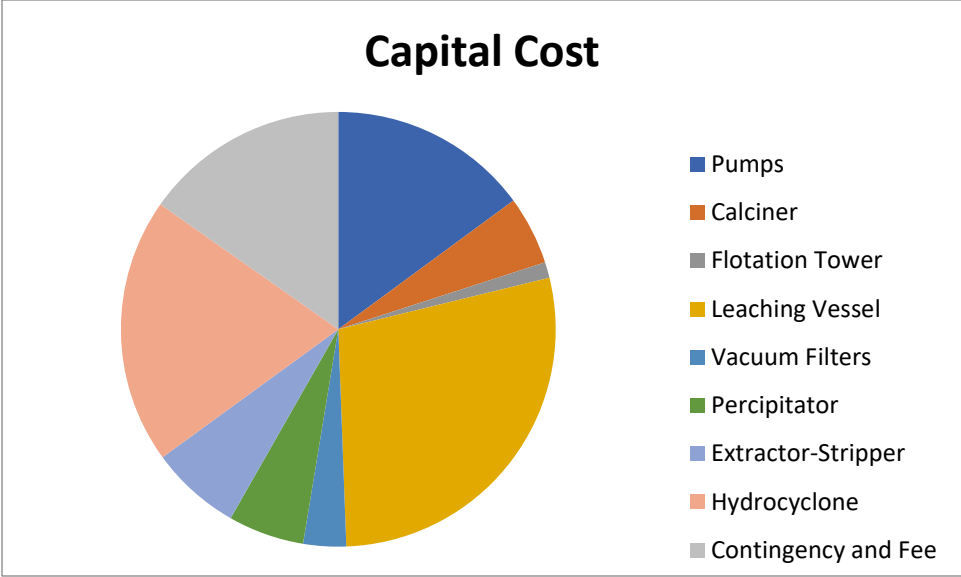


Figure 4.2. Yearly capital cost distribution.

4.2 Manufacturing Cost Estimates

The manufacturing cost was determined following the method outlined in Ulrich. The manufacturing expense estimates can be found in the manufacturing cost summary located in Appendix C. The total manufacturing cost was determined to be \$27,515,992.73 per year. The manufacturing cost distribution is shown below in Figure 4.3.

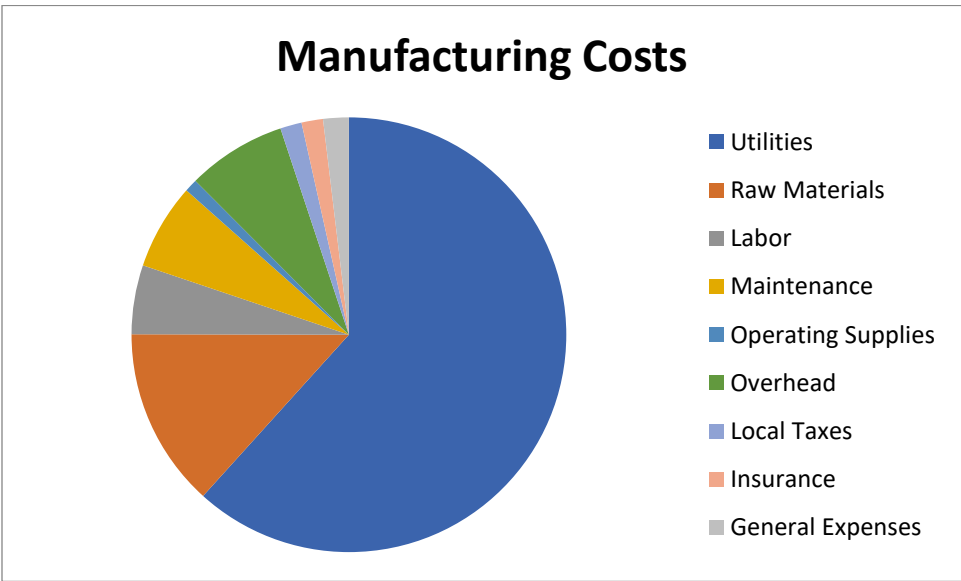


Figure 4.3. Yearly manufacturing expense distribution.

5.0 Discussion of Results

The recovery of REES from phosphoric acid was found to have a favorable economic potential. As shown, the level 4 economic potential has a \$40 million potential profit with the inclusion of raw materials and equipment, while excluding utilities and miscellaneous operating expenses. The net annual profit after taxes of the process was \$14.4 million, compared to the price of raw materials of \$3.3 million. The capital cost was about \$26 million, which is reasonable, since the most expensive piece of equipment was the leaching vessel, costing about \$7.37 million. The process appears to have significant potential given that we are achieving an annual rate of return after taxes of 72%.

6.0 Conclusions

Based on the level 4 economic analysis, a potential profit of about \$40 million can be seen. It was also shown that with our capital cost (\$26million), manufacturing cost (\$27.5million), REE revenue (\$1.8million), and byproduct revenue (\$47.5million) that we are able to achieve an annual rate of return after taxes of 72%. This tells us that the process has lots of potential for being a profitable, viable option for REE production. The big thing to note here is that the large majority of the profit is derived from phosphoric acid production, and that if it was not there, the process would lose millions of dollars a year. It can also be seen that there is still a significant amount of work left to be done as this study gave a base case. The study is not going to be very accurate as many price assumptions are all best-case scenarios since the product will not be entirely pure and the byproduct is not at merchant grade concentrations of 50%. The other large issue is that the product contains about 2% thorium and uranium, which yields a total radioactive production of 4,557 kg/year, well above the required 5 kg/year. This tells us that there is significant work that must be done to remove the remaining thorium and uranium from the product. Despite these areas that need to be considered, we have a lot of confidence in the economic viability of this process.

7.0 Recommendations

The first major recommendation is to implement an evaporative system that takes the phosphoric acid mixture from the solvent extraction process and increases the concentration to merchant grade. This change would bring our byproduct revenues to our estimated revenue given earlier. This process would help remove most of the soluble metals and potentially could separate the sulfuric acid from the phosphoric acid, making it easier to recycle. The equipment required is something that would need to be looked at, but is suspected to consist of a single evaporator or maybe a series of evaporators.

Something that needs to be worked on is the removal of radioactive material from the product, such as thorium and uranium. This process would potentially be very costly as this process would be very difficult. The problem is that thorium, and partially uranium, behave similarly to the REEs and therefore would be hard to separate. To do so would most likely require another section of extraction-stripping, but different reagents would be required to make the thorium and uranium leave the REEs. This is where most of the research would need to focus, the reagent for removing thorium and uranium.

A major percentage of the potential process is lost after the first separation stage, the hydrocyclone cluster. This stage results in the loss of approximately 60% of the REEs, but is critical in removing large portions of the excess water. Improvements in this field would greatly increase the profitability of this process. Alternative separation processes could be explored to remove the excess water, such as distillation, but since distillation is so costly to operate, it was not considered for this process. Improvements in the separation processes would generate greater yields and thus greater profits, if the cost of alternative separation were not costlier than the added profits from the increased yield.

The design of our plant does not incorporate any recycling schemes. Potential locations where recycling may improve the profitability of the plant would be the wastes from the vacuum filter after the leaching vessel and the stripped aqueous phase. Originally, we considered a second hydrocyclone cluster after the leaching vessel that allowed for an easy recycle stream, but a vacuum filter fit our needs more appropriately. By optimizing the recycle ratios around these two points, an increase in phosphoric acid and REE production may be seen, which would increase the economic viability of the plant. More experiments would need to be performed to determine if recycle streams are worthwhile and if they are, what the optimized ratio would be.

A large portion of the equipment had high bare module factors (F_{BM}) because many of the process are acidic in nature. While doing a more in-depth analysis, it would be advantageous to explore newer materials rather stainless steel or nickel-based alloys for process vessels. While this project did not explore piping costs, installing all stainless-steel pipes would be a costly venture, so most cost saving from using polymer lined materials would likely be seen here.

8.0 References and Acknowledgement

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Acknowledgements

- Dr. Counce, University of Tennessee: Guidance in Project
- Weir Minerals: For Hydrocyclone Design and Pricing
- Cytec Industries: For Mixer-Settler Design and Pricing
- Dr. Zhang and FIPR: Process Assumptions and Data

Appendices

Appendix A: Project Assumptions

The equipment assumptions include assumptions made about the separations the perform as given by the data of Dr. Zhang. This is given in table A.1

Equipment Assumptions		
Process	Assumption	Assumption Value
Hydrocyclone	Feed Composition	97wt% Water, 3wt% Clay
	Clay Separation	40wt% Clay recovery in Coase
	Coarse Composition	15wt% Clay, 85wt% Water
Flotation	Concentrate Recovery	30wt% Solid Recovery
		70wt% REE Recovery
		80wt% P ₂ O ₅ Recovery
	Fuel Oil Usage	1lb/Ton fed
Leaching	H ₂ SO ₄ :CaO	4:1 Molar Ratio
	REE Conversion	90%
	P ₂ O ₅ Conversion	95%
	Fe Conversion	10%
	Al Conversion	35%
	Mg Conversion	20%
	Operating Temp	230°C
	Residence Time	240minutes
Vacuum Filtration	Moisture Contenet in Solids	10wt%
Solvent Extraction	Organic:Aqueous	1:1 Mass Based
REE Recovery	Organic:Aqueous	1:1 Mass Based
Percipitation	Conversion	100%
	Oxalic Fed: Oxalic Needed	2:1 Molar Ratio
Calcination	Conversion	100%
	Temperature	900°C
	Thermal Efficiency	75%

Table A.1. Equipment assumptions

The economic assumptions range from CE plant cost index to operator pay. These assumptions are based on Ulrich's method for economic analysis or from various online source for economics. These are shown in the table below.

Economic Assumptions	
CE Plant Cost Index	543
Operator Salary	\$63,000/yr
Income Tax Rate	34%
Operators Required	20
Electricity Cost	\$0.10/kWh
Process Water	\$1.10/m ³
Steam	\$0.0122/kg

Table A.2: Some of our economic Assumptions

Appendix B: Mass and Energy Balance

One of the first major steps in this analysis was a full material balance and energy balance. The material balance followed a basic steady state format of input equals output, which can be shown in Equation B-1. The separation assumptions and conversions for each piece of equipment can be found in Table A.1. Using this equation with each stream we get the results in Table B.1a and Table B.1b.

Equation B.1: $F_{out} = F_{in} + Generation$

Component	MW (kg/kmole)	Streams (kg/hr)											
		1	2	3	4	5	6	7	8	9	10	11	12
Total*	NA	16911889	15558938	1352951	138544.2	848.5834	1299497	191997.9	235381.4	43394.87	47284.4	188097	202254.8
H ₂ O	18	16404532	15254524	1150008	138544.2	0	1150008	138544.2	139139.6	3037.641	3411.962	135727.6	14157.84
H ₂ SO ₄	98.076	0	0	0	0	0	0	0	35041.7	40357.23	859.2881	34182.42	188097
H ₃ PO ₄	97.998	0	0	0	0	0	0	0	12356.04	0	302.9932	12053.05	0
H ₂ C ₂ O ₄	90.038	0	0	0	0	0	0	0	0	0	0	0	0
P ₂ O ₅	141.948	29394.02	17636.41	11757.61	0	0	2351.522	9406.087	470.3043	0	470.3043	0	0
Organic**	NA	0	0	0	0	0	0	0	0	0	0	0	0
Air***	29	0	0	0	0	848.5834	0	0	0	0	0	0	0
Fe ₂ O ₃	159.69	10200.41	6120.245	4080.163	0	0	2856.114	1224.049	1101.644	0	1101.644	0	0
Fe ₂ (SO ₄) ₃	399.87	0	0	0	0	0	0	0	306.5066	0	7.516116	298.9905	0
Al ₂ O ₃	101.963	36317.56	21790.53	14527.02	0	0	10168.92	4358.107	2832.769	0	2832.769	0	0
Al ₂ (SO ₄) ₃	342.143	0	0	0	0	0	0	0	5118.362	0	125.5118	4992.85	0
MgO	40.305	11328.52	6797.113	4531.408	0	0	3171.986	1359.423	1087.538	0	1087.538	0	0
MgSO ₄	120.365	0	0	0	0	0	0	0	811.9434	0	19.91037	792.033	0
CaO	56.078	48073.95	28844.37	19229.58	0	0	13460.71	5768.875	5768.875	0	5768.875	0	0
LnPO ₄	221.817	145.9235	87.55411	58.3694	0	0	17.51082	40.85858	4.085858	0	4.085858	0	0
Ln ₂ (SO ₄) ₃	541.863	0	0	0	0	0	0	0	44.9148	0	1.101395	43.8134	0
Ln ₂ (C ₂ O ₄) ₃	516.259	0	0	0	0	0	0	0	0	0	0	0	0
Ln ₂ O ₃	300.193	0	0	0	0	0	0	0	0	0	0	0	0
Density (kg/m ³)		1000	1000	1000	1000	1.15	1000	1000	1169	1840	NA	1169	1781.2
Pressure (Bar)		19.7	10	10	10	10	10	40	10	40	10	10	10
Temperature (°C)		25	25	25	25	25	25	25	25	25	25	25	25

* Total may not equal the sum of everything shown. Th and U were neglected, as well as Unreactive soluble and insoluble

** Includes a mixture of Exxal 12, Ligand A, and a other components for aid in REE extraction

*** Air is assumed to be 79% Nitrogen and 21% Oxygen

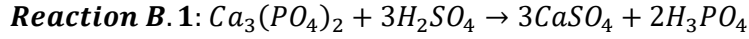
Table B.1a: Material Balance table for streams 1 through 12

Component	MW (kg/kmole)	Streams (kg/hr)											
		13	14	15	16	17	18	19	20	21	22	23	24
Total*	NA	390304.1	390399.6	390399.6	390399.6	390447.1	44.3067	390491.4	390445.5	45.89979	1413.717	2267.272	24.12086
H ₂ O	18	149885.4	0	0	390399.6	390399.6	1.329201	390400.9	390396.3	4.589401	0	85.52549	0
H ₂ SO ₄	98.076	222279.4	0	0	0	0	0	23.40713	23.40685	0.000275	0	0.000275	0
H ₃ PO ₄	97.998	12053.05	0	0	0	0	0	0	0	0	0	0	0
H ₂ C ₂ O ₄	90.038	0	0	0	0	0	42.9775	21.48875	21.4885	0.000253	0	0.000253	0
P ₂ O ₅	141.948	0	0	0	0	0	0	0	0	0	0	0	0
Organic**	NA	0	390351.8	390351.8	0	0	0	0	0	0	0	0	0
Air***	29	0	0	0	0	0	0	0	0	0	1413.717	2062.343	0
Fe ₂ O ₃	159.69	0	0	0	0	0	0	0	0	0	0	0	0
Fe ₂ (SO ₄) ₃	399.87	298.9905	0	0	0	0	0	0	0	0	0	0	0
Al ₂ O ₃	101.963	0	0	0	0	0	0	0	0	0	0	0	0
Al ₂ (SO ₄) ₃	342.143	4992.85	0	0	0	0	0	0	0	0	0	0	0
MgO	40.305	0	0	0	0	0	0	0	0	0	0	0	0
MgSO ₄	120.365	792.033	0	0	0	0	0	0	0	0	0	0	0
CaO	56.078	0	0	0	0	0	0	0	0	0	0	0	0
LnPO ₄	221.817	0	0	0	0	0	0	0	0	0	0	0	0
Ln ₂ (SO ₄) ₃	541.863	1.305888	42.50752	0.128354	0	42.37916	0	0	0	0	0	0	0
Ln ₂ (C ₂ O ₄) ₃	516.259	0	0	0	0	0	0	40.488	0	40.488	0	0	0
Ln ₂ O ₃	300.193	0	0	0	0	0	0	0	0	0	0	0	23.54287
Density (kg/m ³)		1501.7	791.8	791.8	1000	1000	1000	1000	1000	NA	1.18	1.15	NA
Pressure (Bar)		10	10	10	10	10	10	10	10	10	1	1	1
Temperature (°C)		25	25	25	25	25	25	25	25	25	25	900	900

Table B.1b: Second Half of Material Balance.

The next thing we were concerned with was the energy balance, specifically the energy balance around the leaching vessel to determine our heating needs, and the energy balance around the calciner for the natural gas needs. Starting with the leaching vessel the heat to raise the material temperature came from the reaction and a steam heater. This led to Equation B.2 shown below. The data for the steam comes from a large NIST steam table, where the steam is used at 45 barg from temperatures 900°C to 270°C. This is more than likely not realistic but it was a possible operational range that gave us a realistic steam need. The heat of reaction was determined based upon the chemical reaction given by Reaction B.1. This reaction we assumed represented the phosphate reaction, since this reaction occurred at a higher molar quantity than any of the other reactions. The specific heat of the fluid was assumed to be that of water since water was such a high weight percentage.

$$\text{Equation B. 2: } -\Delta H_{rxn} - \Delta H_{steam} = C_p * n * \Delta T$$



$$\begin{aligned} \Delta H_{rxn} &= n_{phosphate} * (h_{f,prod} - h_{f,reactants}) \\ &= \frac{62.95 \text{ kmoles}}{\text{hr}} \\ &* \left(2 * \left(-\frac{1271.7 \text{ kJ}}{\text{mol}} \right) + 3 * \left(-\frac{1434.5 \text{ kJ}}{\text{mole}} \right) - 3 * \left(-\frac{814 \text{ kJ}}{\text{mole}} \right) - \left(-\frac{4120.8 \text{ kJ}}{\text{mole}} \right) \right) \\ &* \frac{1000 \text{ mole}}{\text{kmole}} = -4967.9 \text{ kW} \end{aligned}$$

$$\Delta H_{steam} = m_{steam} * (H_f - H_o) = m_{steam} * (2846.7 - 4352.1) = -1505.4 * m_{steam}$$

$$C_p n * \Delta T = \left(\frac{75.3 \text{ kJ}}{\text{kmole} * K} \right) * \left(\frac{2.322 \text{ kmole}}{\text{sec}} \right) * (230^\circ C - 25^\circ C) = 35843.553 \text{ kW}$$

$$4967.9 \text{ kW} + 1505.4 m_{steam} = 35843.553 \text{ kW} \rightarrow m_{steam} = 20.5 \text{ kg/sec}$$

The other portion we looked at was the energy required to heat air from ambient temperature to 900°C required for the calcination process. The heat required is based on the specific heat, since it is air we used the temperature dependent function for specific heat given by Felder. The heat supply is based on the heat of reaction from natural gas, which we assumed was nearly pure methane, at a 75% efficiency. This lead to Equation B.3 given below.

Equation B.3:

$$\begin{aligned} -n_{NG} * \varepsilon_{thermal} * \Delta H_{rxn} &= n_{air} \int_{298K}^{1173K} C_p dT \\ n_{NG} * \varepsilon_{thermal} * \Delta H_{rxn} &= 0.75 * -\frac{802.3665 \text{ MJ}}{\text{kmole} CH_4} * n_{NG} = -601.775 n_{NG} \end{aligned}$$

$$\begin{aligned}
n_{air} \int_{298K}^{1173K} C_p dT &= \frac{992.26 \text{ kmole}}{\text{hr}} \\
&* \int_{298K}^{1173K} 0.02809 + 1.96 * 10^{-6} T + 4.799 * 10^{-9} T^2 - 1.965 * 10^{-12} T^4 dT \\
&= 1345.4 \text{ MJ/hr} \\
601.775 n_{NG} &= \frac{1345.4 \text{ MJ}}{\text{hr}} \rightarrow n_{NG} = 2.23 \text{ kmole/hr}
\end{aligned}$$

Appendix C: Economic Results and Analysis

The first major portion for the economic analysis would be the required fixed capital for the process. To determine this, we followed the method outlined by Ulrich. This method required us to first determine the actual capital of each piece of equipment, which is shown in Appendix C. The following step required the sum of the equipment capital with an assumption of two pumps, one for the process and one for a backup. The remaining amount of fixed capital for a non-grassroots plant was the capital related to contingency and fee, which is given by the following equation:

$$\text{Equation C.1: } C_C + C_F = 0.18 * C_{BM}$$

Where C_C and C_F are the associated capital of contingencies and fees and C_{BM} is the total equipment capital. From this, we can add the two together to get our non-grassroots plant fixed capital, which is given by C_{TM} in the table below to be about 26 million dollars.

CAPITAL COST SUMMARY
 Location: Florida
 Date to which estimate applies 2017

Job title: Rare Earth Element Recovery Plant
 Cost Index Type: CE Plant Cost Index
 Cost Index Value:

Equipment Identification	Number	Capacity or Size Specifications	(base material)		Actual Bare Module Factor, Fam	Actual Bare Module Cost, C _{BM}	Total
			Year 2004	Year 2017			
Gas-Solid Contactors							
Calcliner	B-190	Inside Volume is 1700m ³ , Diameter=4.5 meters	\$ 98,217.00	\$ 133,329.58	10.0	\$ 1,333,295.78	\$ 1,333,295.78
Total Gas-Solid Contactors							\$ 1,333,295.78
Process Vessels							
Flotation Tower	D-120	72m height, 3.5m diameter	\$ 38,000.00	\$ 51,585.00	5.00	\$ 257,925.00	\$ 257,925.00
Flotation Tower Trays		Stainless Steel Trays	\$ 16,000.00	\$ 21,720.00	2.20	\$ 47,784.00	\$ 47,784.00
Solvent Extractor	D-150	5 Stage Mixer Settler, with a settling area of 140m ²	\$ 596,267.94	\$ 809,433.73	1.00	\$ 809,433.73	\$ 809,433.73
REE Stripper	D-160	7 Stage Mixer Settler, with a settling area of 163m ²	\$ 699,603.50	\$ 949,711.75	1.00	\$ 949,711.75	\$ 949,711.75
Total Process Vessels							\$ 2,064,854.47
Reactors							
Leaching Vessel	R-130	Two, 403m ³ Tanks	\$ 320,000.00	\$ 434,400.00	16.0	\$ 6,950,400.00	\$ 6,950,400.00
Leaching Vessel Agitators		Two Agitators for leaching vessel, both 48.5 kW	\$ 124,000.00	\$ 168,330.00	2.5	\$ 420,825.00	\$ 420,825.00
Total Reactors							\$ 7,371,225.00
Separators							
Hydrocyclone	H-110	Used to separate the Clay, handles 4.7m ³ /sec	\$1,267,614.31	\$1,720,786.43	3.0	\$ 5,162,359.30	\$ 5,162,359.30
Vacuum Filter for Leachate	H-140	A belt filter, made to handle about 0.06m ³ /sec	\$ 70,000.00	\$ 95,025.00	3.6	\$ 342,090.00	\$ 342,090.00
Precipitator	H-170	A pressure vessel with a volume of about 728m ³	\$ 150,000.00	\$ 203,625.00	7.0	\$ 1,425,375.00	\$ 1,425,375.00
Precipitator Agitator		Agitator for precipitor, uses 9.6kW	\$ 15,000.00	\$ 20,362.50	2.5	\$ 50,906.25	\$ 50,906.25
Vacuum Filter for Calciner	H-180	A belt filter, made to handle about 0.1 m ³ /sec	\$ 100,000.00	\$ 135,750.00	3.6	\$ 488,700.00	\$ 488,700.00
Tower Total							\$ 7,469,430.55

The next step was determining the annual manufacturing cost, revenue from sales, and net annual profit after taxes. Most of the calculation assumptions are either given in the table below or in appendix A. A big thing to keep in mind is that byproduct revenue is calculated with direct manufacturing expenses, which indicates a negative overall manufacturing expense.

MANUFACTURING COST SUMMARY

Job Title Rare Earth Element Recovery Plant

Location Florida Annual Capacity (kg/yr) 4,000,000,000 Waste Clay/year

Effective Date to Which Estimate Applies 2017 Cost Index Type CE Plant Cost Index

Cost Index Value 543

Capital

Fixed capital, C_{FC}	\$	26,117,667.65
Working capital (15% of fixed capital), C_{WC}	\$	3,917,650.15
Total capital investment, C_{TC}	\$	30,035,317.79

		Annual Cost	
		\$/yr	\$/yr
Manufacturing Expenses			
Direct			
Raw materials		\$	2,671,294.59
By-product credits		\$	(47,506,839.12)
Catalysts and solvents		\$	598,520.32
Operating labor		\$	1,260,000.00
Supervisory and clerical labor (15% of operating labor)		\$	189,000.00
Utilities			
Steam	581,053,822.78 kg/y	45 barg @ 0.012195 \$/kg	\$ 7,086,126.38
Electricity	34,977,290.05 kWh @	0.1 \$/kWh	\$ 3,423,822.89
Process water	4,170,192.83 m ³ @	1.1 \$/m ³	\$ 4,584,448.63
Natural Gas	14,142.75 GJ @	2.73 \$/GJ	\$ 38,605.50
Maintenance and repairs (6% of fixed capital)		\$	1,567,060.06
Operating supplies (15% of maint & repairs)		\$	235,059.01
Laboratory charges (15% of operating labor)		\$	189,000.00
Patents and royalties (5% of total expense)		\$	714.25
		Total, A_{DME}	\$ (25,663,187.48) \$(25,663,187.48)
Indirect			
Overhead (payroll and plant), packaging, storage (60% of op. Labor+supervision+ maint.)		\$	1,809,636.04
Local taxes (1.5% of fixed capital)		\$	391,765.01
Insurance (1.5% of fixed capital)		\$	391,765.01
		Total, A_{IME}	\$ 2,593,166.06 \$ 2,593,166.06
Total manufacturing expense, $A_{ME}=A_{DME}+A_{IME}$		\$	(23,070,021.42) \$(23,070,021.42)
General Expenses			
Administrative costs (25% of overhead)		\$	452,409.01
Distribution and selling (10% of total expense)		\$	14,285.00
Research and development (5% of total expense)		\$	714.25
		Total general expense, A_{GE}	\$ 467,408.26 \$ 467,408.26
Depreciation (approximately 10% of fixed capital), A_{BD}		\$	2,611,766.76
		Total Expenses, A_{TE}	\$ (19,990,846.39) \$(19,990,846.39)
Revenue from Sales	185,101.53 kg/yr @ 10.00 \$/kg), A_S		\$ 1,851,015.33
Net annual profit, A_{NP}			\$ 21,841,861.73
Income taxes (net annual profit times the tax rate), A_{IT}			\$ 7,426,232.99
Net annual profit after taxes ($A_{NP}-A_{IT}$), A_{NNP}			\$ 14,415,628.74

Aftertax rate of return, $i = (1.5 A_{NNP}/C_{TC}) \times 100 = 71.99 \%$

Table C.2: Manufacturing Expense Calculation Table

Appendix D: Equipment Design and Costing

- Hydrocyclone (H-110):** The sizing and costing of the hydrocyclone was based on an estimation from Weir Minerals. The important thing here is that the capital for the hydrocyclone is based on flow input, not physical dimensions. This meant we could estimate the capital using Equations D.1 shown below. The capital is represented by C_p , which does not consider the material of construction, F_{bm} . The actual capital is given as C_{bm} , which is the product of C_p and F_{bm} . F_{bm} is determined to be a material factor, which for carbon steel construction was 3. The next portion was determining the cost and size of the feed pumps. Using figures 5.49, 5.50, and 5.51 from Ulrich, the cost of the pumps is based on input shaft power. Equations D.2 and D.3 show how shaft power and pump efficiency were determined. Also pump power would be required for the electricity consumption, which is shown below in Equation D.4. Through our initial design, it was determined that for a centrifugal pump the feed rate was far too much for one pump. We then determined that it would require 5 feed pump to meet our feed rate demand. The calculations below for shaft work and power are shown for one of these pump, the capital for the pump system is given for the whole set of feed pump. This is the only place we show an example calculation for the pumps as they all follow the same procedure.

$$C_C + C_F = 0.18 * C_{BM}$$

$$\text{Equation D. 1: } C_{p, hc} = \$200,000 * \frac{q}{\frac{546L}{sec}}$$

$$q = \frac{\dot{m}}{\rho}, \text{ where } \dot{m} \text{ is given as } \frac{16911889kg}{hr} \text{ given by mass balance}$$

$$q = \frac{16911.88m^3}{hr} = \frac{4697.7L}{sec}$$

$$C_{p, hc} = \$200,000 * \frac{\frac{4697.7L}{sec}}{\frac{546L}{sec}} = \$1,720,786.43$$

$$C_{bm} = F_{bm} * C_p = 3 * \$1,720,786.43 = \$5,162,359.30$$

Equation D. 2: $W_s = q * \frac{\Delta p}{\epsilon_i}$, where ϵ_i is the pump efficiency

Equation D. 3: $\epsilon_i = (1 - 0.12q^{-0.27})(1 - \mu^{0.8})$
 $= \left(1 - 0.12 * \left(\frac{0.94m^3}{s}\right)^{-0.27}\right) * (1 - 0.00089Pa * s^{0.8}) = 0.875$

$$W_s = \frac{0.94m^3}{sec} * \frac{197kPa}{0.875} = 211.5kW$$

Equation D. 4: $P = \frac{W_s}{\epsilon_d} = \frac{211.5kW}{0.9} = 235.1kW$

$$C_{bm} = C_p * F_{bm} * 543/400$$

$$C_{bm} = 5 * \left(\$33,000.00 * 4.25 * \frac{543}{400}\right) = \$951,946.88$$

2. Flotation Tower (D-120): For the costing of the flotation tower we determine, with the advisement of Dr. Counce, that it would be best to cost it as a pressure vessel with a gas sparger and two sieve trays per column. This lead to us needing to size the column, which a procedure for flotation columns was given by J.B. Yianatos, where equations are given below. Due to the amount of material being processed, we determined that two flotation columns would need to be used to meet limitations for costing given by Ulrich. The material of construction for the sieve trays were stainless steel and for the vessel it was carbon steel. We also needed to determine the power usage, which is given below for the gas sparger.

Equation D.5:

$$flot = q * res = 30minutes * \frac{22.549m^3}{sec} = 676.47m^3$$

$$\frac{H}{D} = 10; D = 3.5m, H = 35m$$

$$C_{bm, ves} = 2 * \left(\$19,000 * 5 * \frac{543}{400}\right) = \$257,925.00$$

$$C_{bm, svt} = 2 * (\$8,000 * 2.2 * 2 * 1.6 * 543/400) = \$152,908.80$$

$$C_{bm, flot} = \$257,925.00 + \$152,908.80 = \$410,833.80$$

$$\text{Power of Gas Sparger} = m_{air} * 0.6 * \frac{\Delta p}{\rho} = 0.6 * q_{air} * \Delta p$$

$$SGR = \frac{2cm^3}{\frac{cm^2}{sec}} = \frac{q_{air}}{Across} \rightarrow q_{air} = \frac{0.02m}{sec} * \pi * \left(\frac{3.5m}{2}\right)^2 = \frac{0.192m^3}{sec}$$

$$\text{Power of Gas Sparger} = 0.6 * \left(\frac{0.192m^3}{s}\right) * 167kPa = 28.92kW$$

3. Leaching Vessel (R-130): The costing for the leaching vessel was done using figures 5.42, 5.44, 5.45, and 5.46 from Ulrich. They give costing information for an agitator and a pressure vessel. The cost is heavily dependent on the volume and diameters. For the vessel, the requirements were a 240-minute residence time for a 90% conversion of REE phosphates. It also important to note that the leaching vessel needed to operate at 40barg due to the temperature being at 230°C. Using Equation D.5 from the flotation section the volume was determined. Again, due to limitations from the costing by Ulrich, we determined two vessels in series would be used. The capital is given below for both the agitator and vessel and then they are added together and multiplied by two to get the total leaching capital. For operating cost, we needed to know the power requirements for the agitator, which is given by Equation D.6 below.

$$V_{leacher} = q * Res = \frac{201.35m^3}{hr} * 240min * \left(\frac{1hr}{60min}\right) = 805.7m^3$$

$$H = \frac{V}{Across}; \text{Diameter} = 4m; H = 32m$$

Equation D.6: $\text{Power of Agitator} = 0.4 * V^{0.8} = 0.4 * \left(\frac{V_{tot}}{ntanks}\right)^{0.8} = 0.4 * \left(\frac{805.7m^3}{2}\right)^{0.8} = 48.53kW$

$$C_{bm, ag} = 2 * (C_{p, ag} * F_{bm} * 543/400) = 2 * (\$62,000 * 2.5 * 543/400) = \$420,825.00$$

$$C_{bm, ves} = 2 * (C_{p, ves} * F_{bm} * 543/400) = 2 * (16 * \$160,000.00 * 543/400) = \$6,950,400.00$$

$$C_{bm, Leacher} = \$420,825.00 + \$6,950,400.00 = \$7,371,225.00$$

4. Leachate Filter (H-140): The separation of solids and the aqueous is done by a belt vacuum filter. The costing of this filter is done using Ulrich's method, which is based on the filter area. This was determined using Equation D.7 shown below, which is given by Ulrich for a belt filter. The operating cost was based on power usage, which is given by Equation D.8 from Ulrich.

$$\text{Equation D.7: } \frac{q}{A_{filter}} = 0.01 \rightarrow A_{filter} = 0.01 * q = 0.01 * \frac{0.0559m^3}{s} = 5.59m^2$$

$$Power = A^{0.75} = 5.59m^2^{0.75} = 3.64kW$$

$$C_{bm} = C_p * F_{bm} * \frac{543}{400} = \$70,000 * 3.6 * \frac{543}{400} = \$342,090.00$$

5. Solvent Extractor (D-150): This equipment allowed us to take and remove all our REEs from the aqueous stream, which we were then getting a byproduct credit for the phosphoric acid produced. The method for determining the needed number of stages was using the Kremser equation as it was given in Geankopolis. The design equations for the Mixer settlers used were given by a representative of Cytec Industries and are shown below. In this was also costing information and power requirements for the mixer settlers.

$$Volume = N_{stages} * q_{tot} * restime = 5 * \frac{0.193m^3}{sec} * 2minutes * \frac{60sec}{min} = 115.78m^3$$

$$Total Power = \frac{0.03Hp}{gal} * \frac{264.172gal}{m^3} * \frac{1kW}{1.34HP} * 115.78m^3 = 943.3kW$$

$$Settling Area = \frac{694.7m^3}{\frac{5m}{hr}} = 138.9m^2$$

$$C_{bm} = \frac{\$5372}{m^2} * 138.9m^2 = \$727,964.58$$

- 6. REE Stripper (D-160):** This process had the purpose of removing the REEs from the organic phase into a water phase, this way we could recycle the organic for cheaper operation. The design equations are the same as above.

$$Volume = Nstages * qtot * restime = 7 * \frac{0.226m^3}{sec} * 2minutes * \frac{60sec}{min} = 190.188m^3$$

$$Total Power = \frac{0.03Hp}{gal} * \frac{264.172gal}{m^3} * \frac{1kW}{1.34HP} * 190.188m^3 = 1123.97kW$$

$$Settling Area = \frac{815.1m^3}{\frac{5m}{hr}} = 163m^2$$

$$Cbm = \frac{\$5372}{m^2} * 163m^2 = \$876,227.30$$

- 7. Precipitator (H-170):** The process of removing the REE from the aqueous phase started with a precipitator. This assumed to be done using an agitated process vessel. Costing, done by the Ulrich method, required the volume and height of the tank. This was done using Equation D.5 from above in the flotation section. The residence time used was picked so that we would not need multiple vessels as there was not an existing assumption. The power usage for the agitator was done using Equation D.6 from the leaching section.

$$V = qtot * Res = \frac{(360.23 + 0.041)m^3}{hr} * 50min * \left(\frac{1hr}{60min}\right) = 300.2m^3$$

$$D = 4m, \quad H = \frac{V}{\frac{\pi}{4} * D^2} = \frac{300.2m^3}{\frac{\pi}{4} * 4^2m^2} = 24m$$

$$Power of Agitator = 0.4 * V^{0.8} = 0.4 * 300.2m^3^{0.75} = 9.6kW$$

$$Cbm, tot = (Cp, ves * Fbm, ves + Cp, ag * Fbm, ag) * \frac{543}{400} = (\$130,000 * 7 + \$15,000 * 2.5) * \frac{543}{400} = \$1,286,231.25$$

- 8. Precipitation Filter (H-180):** This process removed the REE oxalates from the aqueous waste in the same fashion as the leachate filter, using a vacuum belt filter.

$$\frac{q}{Afilter} = 0.01 \rightarrow Afilter = 0.01 * q = 0.01 * \frac{0.1m^3}{s} = 10m^2$$

$$Power = A^{0.75} = 10m^{2 \cdot 0.75} = 5.63kW$$

$$Cbm = Cp * Fbm * \frac{543}{400} = \$100,000 * 3.6 * \frac{543}{400} = \$488,700.00$$

9. Rotary Calcination Kiln (B-190): The final equipment was the rotary calciner kiln. The costing was done using the Ulrich method for calciners, which required internal volume. This was done using the mass flow rate relationship to diameter and length. This equation is shown below as Equation D.9. The capital for the calciner is given below. The operating cost for the calciner included a built-in drive that rotated the kiln. The power required is given by Equation D.10 below.

Equation D.9: $m = 0.0015L * D^2 = \frac{45.9kg}{hr} * \frac{1hr}{3600s} \rightarrow L * D^2 = \frac{0.01275kg}{0.006} = 8.5m^3$

$$V = L * \pi * \frac{D^2}{4} = \frac{\pi}{4} * 8.5 = 6.67m^3$$

$$Cbm, calciner = Cp * Fbm * \frac{543}{400} = \$98,217.00 * 5 * \frac{543}{400} = \$666,647.89$$

Equation D.10: Power for Calciner=0.15*V_{internal}

Appendix E: Distribution Coefficients and Clay Composition

Clay Composition			
Substance	1	2	Average
P2O5	6.10	5.89	5.793561
P2O5	5.64	5.55	
Insol	51.04	51.75	51.39
MgO	2.33	2.13	2.23
Fe2O3	2.16	1.86	2.01
Al2O3	7.84	6.48	7.16
CaO	9.55	9.40	9.48
Pr ppm	2.55	2.63	2.59
Eu ppm	1.92	1.82	1.87
Tb ppm	1.02	0.95	0.98
Dy ppm	7.26	7.11	7.19
Ho ppm	0.55	0.80	0.68
Er ppm	4.77	4.49	4.63
Tm ppm	0.60	0.52	0.56
Yb ppm	4.41	4.27	4.34
Lu ppm	0.84	0.79	0.81
Sc ppm	6.05	6.16	6.10
Gd ppm	10.42	9.82	10.12
Sm ppm	6.07	6.47	6.27
Th ppm	5.24	6.61	5.92
U ppm	37.12	37.49	37.31
Ce ppm	85.65	86.28	85.97
Y ppm	61.00	59.35	60.18
La ppm	42.02	41.30	41.66
Nd ppm	54.16	53.19	53.67
ΣREE	289.30	285.93	287.62

Table E.1: Composition of Clay

Components	Solvent Extraction	REE Stripping
	D	D
Th ₂ (SO ₄) ₃	419.74	0.38503
U ₂ (SO ₄) ₃	0.934	0.28507
Pr ₂ (SO ₄) ₃	9.96933	0.14781
Eu ₂ (SO ₄) ₃	270.83	0.16906
Tb ₂ (SO ₄) ₃	146.1	0.19852
Dy ₂ (SO ₄) ₃	504.95	0.2847
Ho ₂ (SO ₄) ₃	252.6	0.4239
Er ₂ (SO ₄) ₃	1711.2	0.6416
Tm ₂ (SO ₄) ₃	1411	0.81596
Yb ₂ (SO ₄) ₃	2638.5	1.05371
Lu ₂ (SO ₄) ₃	1E-24	4.40611
Sc ₂ (SO ₄) ₃	9.9808	0.27927
Gd ₂ (SO ₄) ₃	229.63	0.17248
Sm ₂ (SO ₄) ₃	1E-28	0.17378
Ce ₂ (SO ₄) ₃	4.3064	0.17012
Y ₂ (SO ₄) ₃	1027.3	0.43814
La ₂ (SO ₄) ₃	1.5959	0.14032
Nd ₂ (SO ₄) ₃	26.516	0.14591

Table E.2: Extraction and Stripping Process Distribution Coefficients