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FINAL TECHNICAL REPORT

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RARE EARTH EXTRACTION AND CONCENTRATION AT PILOT-SCALE FROM NORTH DAKOTA COAL-RELATED FEEDSTOCKS

PRINCIPAL INVESTIGATOR

Nolan Theaker
Phone: 701-777-6298
nolan.theaker@und.edu

ADDITIONAL AUTHORS

Abdelmalek Bellal, PhD – University of North Dakota
Nicholas Dyrstad-Cincotta – University of North Dakota
Alexander Benson – Microbeam Technologies Inc.
Eric Kolb – Microbeam Technologies Inc.
David Stadem – Microbeam Technologies Inc.
Ryan Winburn – Rare Earth Salts LLC
Nick Sosalla – Barr Engineering
Ryder Shallbetter – Barr Engineering
Daniel Palo – Barr Engineering

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SUBMITTED BY

Center for Process Energy Research
College of Engineering and Mines
University of North Dakota
2844 Campus Road, Stop 8153
Grand Forks, ND 58202

WORK PERFORMED UNDER AGREEMENT

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1 EXECUTIVE SUMMARY

The University of North Dakota (UND), combined with partners Barr Engineering (Barr), Microbeam Technologies Inc. (MTI), Rare Earth Salts (RES), and other parties evaluated the technical and engineering feasibility of a process for extracting rare earth elements (REEs) and critical minerals (CMs) from North Dakota (ND) lignite. The project was divided into two budget periods; the first budget period saw the acquisition of the 300 ppm blended feedstocks, design of the pilot facility, and a preliminary feasibility assessment; the second period aimed at constructing, commissioning, and operating a 1,000 lb/hr pilot facility for UND's REE extraction process and evaluation of the refineability of UND's concentrates as well as the evaluation of process economics. In each economic analysis evaluated in this project, the most recent process data was utilized, even if this data was not likely to be equivalent to future data from testing due to coal weathering.

Feedstock was procured from two sources, one from a coal seam in the Rhame bed with the assistance of the North Dakota Geologic Survey (NDGS) of 44 tons with an average REE content of ~530 ppm, the second from the top of a seam in the Freedom Mine of ~120 tons with an average REE content of ~160 ppm. The blended material of a 60% Freedom Mine and 40% Rhame bed mixture was found to ensure a 300-ppm mixture (average of 310), and was chosen as the feedstock for the pilot testing in the second phase of the project. A design of the pilot was completed, including P&IDs, PFDs, a HAZOP review, and permits acquired. This included the completion of an air, water, and industrial fire permit for the site chosen in Grand Forks, ND and the expected conditions of the pilot facility. These documents and studies were supplied to the U.S. Department of Energy National Energy Technology Laboratory for review of a Go/No-Go decision, which was successfully passed by the project in September, 2020.

Construction of the pilot facility commenced in October of 2020, although was dramatically delayed due to the impacts of the COVID-19 pandemic on equipment availability, shipping delays, personnel safety policies, and quarantining policies. Due to these delays and significant cost increases, an amendment for both additional time and budget was requested and granted, permitting the complete attainment of the statement of project objectives originally proposed. Due to the delays discussed above in equipment arrival, additional efforts were employed to focus on automation and operator safety above the original design plans. The construction was finally completed in Q2 2023, with commissioning completed within another 6 months from this. The pilot facility was evaluated as able to operate in a mode capable of the nameplate capacity of 1,000 lbs/hr for an extended period, and the achievement of construction and commissioning goals was reached.

Pilot operation was completed processing over 100 tons of the 300-ppm blended feedstock, and produced REE concentrates as high as ~90% pure REEs. However, the CMs originally planned for extraction (Ge and Ga) were dramatically reduced in extraction, along with a mild reduction in the REEs due to the significant weathering of the coal feedstocks (stored for over 4 years prior to usage). Mineral changes in the coal are believed to be the primary result of these changes in extractability. These extraction percentages are believed to be below that of a system utilizing fresh feedstock (echoed by the higher extraction percentage achieved on the bench-scale system of the fresh feedstock in 2020).

Overall, the pilot was constructed and shown to be capable of utilizing UND's REE extraction technology to recover high-purity concentrates from lignite. The economics showed a potential beneficial case when paired with carbon products manufacturing, although this would be substantially improved using the prior bench-scale extraction data as compared with the pilot (only pilot data was used in the model for consistency). This project demonstrated the feasibility of the UND REE process for recovering REEs from lignites into refinable products, and paves the way for additional testing and technology commercialization to a realizable commercial demonstration.

2 TABLE OF CONTENTS

2.1 Table of Figures

2.2 Table of Tables

3 SCOPE OF WORK

The overall objective of this project was to demonstrate a high performance, economically feasible and environmentally benign technology to recover rare earth elements (REE) from North Dakota (ND) lignite coal feedstocks at the pilot scale. In order to meet the project objectives, the following specific objectives have been identified:

- Design and construct a pilot-scale system for continuous REE extraction from ND coal feedstocks capable of a minimum 0.5 tons/hr feed rate of physically beneficiated lignite coal based on previous laboratory and bench scale testing.
- Obtain a large sample (~300 tons) of high REE (>300 ppm following coal cleaning) from ND lignite to provide adequate material for shakedown and continuous testing in a pilot-scale demonstration facility, including extra feedstock for contingencies.
- Conduct initial parametric testing on samples of the ND lignite at bench-scale to cost-effectively identify optimal operating conditions and aid in pilot-scale system design.
- Commission the pilot facility using selected high REE containing coals from various regions in ND.
- Conduct continuous pilot-scale testing utilizing optimal conditions for REE extraction and concentration on at least 100 tons of the >300 ppm REE-containing ND lignite.
- Confirm the compatibility of REE concentrate generated during pilot-scale testing with commercial-scale REE refining.
- Conduct a Techno-Economic Analysis (TEA) and preliminary Front End Engineering Design (pre-FEED) study on a potential commercial facility based on results from the pilot testing campaign utilizing the ND lignite, in which an economic feasibility study and workforce assessment will be contained.
- Work with industry partners to develop a technology roadmap and commercial deployment plan.

A two-phase approach was planned to meet the project objectives, with a Go/No-Go decision following completion of the first. The primary goals of the first phase were to ensure the technology's profitability and feasibility through the completion of preliminary economic, feasibility, and commercialization studies. Additionally, gathering, preparing, and verifying sufficient REE concentrations in the chosen domestic coal feedstock(s) will be conducted, along with a conceptual design study of the pilot plant, including engineering drawings, to enable rapid construction and to develop quotes for all equipment required for continuous operation. Logistical planning for coal and process chemical requirements, storage, feeding, and replenishment were investigated during pilot plant design, with finalized plans for operation completed prior to the Go/No-Go Decision.

Following a favorable Go/No-Go decision, pilot plant procurement and construction began in tandem with bench-scale parametric testing and evaluation of the chosen domestic feedstock. REE concentrate results from this bench-scale testing were delivered to the project partner for preliminary lab-work to evaluate feasibility and optimal process conditions and pathways for the rapid refinement of more valuable components. Following the completion of pilot plant construction and suitable commissioning, testing of at least 100 tons of the feedstock was conducted. Finally, updates were made to economic, feasibility, and commercialization studies in the form of a pre-Front End Engineering Design (pre-FEED) study to evaluate the performance and economics of a potential commercial plant in detail.

3.1 Task Structure

Task 1.0 - Project Management and Planning

SubTask 1.1 – Project Management Plan

The Recipient shall manage and direct the project in accordance with a Project Management Plan to meet all technical, schedule and budget objectives and requirements. The Recipient will coordinate activities in order to effectively accomplish the work. The Recipient will ensure that project plans, results, and decisions are appropriately documented, and project reporting and briefing requirements are satisfied. The Recipient shall update the Project Management Plan 30 days after award and as necessary throughout the project to accurately reflect the current status of the project. Examples of when it may be appropriate to update the Project Management Plan include: (a) project management policy and procedural changes; (b) changes to the technical, cost, and/or schedule baseline for the project; (c) significant changes in scope, methods, or approaches; or (d) as otherwise required to ensure that the plan is the appropriate governing document for the work required to accomplish the project objectives.

Management of project risks will occur in accordance with the risk management methodology delineated in the Project Management Plan in order to identify, assess, monitor and mitigate technical uncertainties as well as schedule, budgetary and environmental risks associated with all aspects of the project. The results and status of the risk management process will be presented during project reviews and in quarterly progress reports with emphasis placed on the medium- and high-risk items.

SubTask 1.2 – Technology Maturation Plan

The Recipient will develop a Technology Maturation Plan (TMP) that describes the current technology readiness level (TRL) of the proposed technology/technologies, relates the proposed project work to maturation of the proposed technology, and describes known post-project work necessary to further increase the technology TRL level.

SubTask 1.3 Workforce Readiness Plan

The Recipient will prepare and maintain a Workforce Readiness Plan (WRP) related to the technology being researched under the project. The Plan must describe the skillset and availability of the workforce needed for future commercialization and deployment of the technology, including whether any related apprenticeships, certificates, certifications, or academic training are currently available. If a workforce with the required skills is not readily available, or if the technology is so new that a trained workforce does not yet exist, the Recipient’s plan shall detail how the needed workforce could be developed, for instance, through coordination with educational institutions such as community colleges, technical schools, and universities; company-led in-house training;

union training, etc. The Recipient will monitor and update its assessment of workforce availability and development plans throughout the life of the project.

Task 2.0 - Financial Plan for Commercialization

The Recipient will develop and periodically update a Financial Plan for Commercialization. At a minimum, the plan should explain the economic feasibility demonstrated by the Recipient's Excel financial spreadsheet model and include a description of the Recipient's proposed business plan for developing and commercializing their technology to economically produce salable REEs and Critical Materials (CMs) from U.S. coal and coal-based resources. Information to be included is an explanation of the hurdles and risks for factors such as: supply of process inputs; process and technology development; capital, operating, and maintenance cost; process operation factors; life-cycle environmental, permitting, and other regulatory factors; market demand and quantity/price points for output products; offtake agreements; downstream supply chain for refining products; international demand, supply, competition, and other considerations; etc.

The financial plan will consist of a written report with the key assumptions and data used for development being documented in the design basis. The results of the design basis and financial plan will be utilized and refined through subsequent tasks. A final financial plan will be developing using all data and experience gained through execution of the project and will be complete 30 days prior to award completion.

Task 3.0 – Techno-Economic Assessment

The Recipient will develop and provide NETL a Techno-Economic Assessment (TEA) based on testing and operation of the REE/CM recovery system. The Recipient will develop a detailed TEA that estimates the cost and performance for scale-up to a commercial demonstration.

The techno-economic analysis produced during DE-FE0027006 will be updated and refined utilizing the domestic feedstock chosen for the process and based upon the results from the previous tasks. During this task, the Recipient plans to utilize a commercial-scale location as the possible site for the TEA. This will be determined through assistance and discussion with project partners. Evaluation of the potential of opening an additional mine for targeted REE recovery, in addition to utilization of existing mine's coal seams, will be conducted. Included in this study will be the key components of a preliminary commercialization plan, such as resource assessments, plant scale, time to market, market analysis, equipment vendor discussions, identification/discussion with purchasers/refiners of the Recipient's 2wt% concentrate, and initial investment strategies. Evaluation of possible critical material economics will be evaluated for economic parity. The technical and economic feasibility study will be performed in collaboration with a qualified Architectural and Engineering (A&E) firm who has extensive experience in coal mining and handling, power generation, minerals exploration, and minerals processing and extractive metallurgy.

The TEA will include and be based on a mass and energy balance that identifies component concentrations and yields associated with each processing step. Conversion factors and units will be identified. A fully functional interactive Excel spreadsheet model with no locked or hidden cells will be included with the TEA. In addition to rare earth element prices supplied by NETL, future market prices for products based upon input from project partners Rare Earth Salts and the Critical Materials Institute will be used for the TEA. Revenue projections will be itemized for each rare earth compound/element and each product other than rare earths. Both capital and operating cost factors will be used to estimate the economic viability. The capital cost estimate will indicate the all-in costs for the facility, including infrastructure from the site fence line, interconnection to existing facilities, equipment costs, construction costs, construction indirect costs, and owner's costs.

The TEA will include a design estimate with adequate detail to be classified as an Association for the Advancement of Cost Engineering (AACE) Class 3 or better estimate. This estimate is intended to serve as a pre-Front-End Engineering and Design (FEED) level estimate for a future commercial scale demonstration project. In order to obtain the AACE Class 3 estimate, Task 3.0 will require the creation of a design basis, process model (including mass and energy balance and process flow diagram), proposed plant general arrangements, equipment specifications, and a Basis of Estimate. Task 3.0 will consist of the engineering documents outlined above for the completion of the cost estimate, the AACE cost estimate, and the full TEA interactive Excel model.

Task 4.0 – Provide Split Samples

The Recipient will provide NETL with a single sample that reflects the highest achieved REE concentration generated during conduct of project effort. The quantity of material to be provided to NETL shall be no less than 3 grams. Material Safety Data Sheets (MSDS) are required to accompany material supplied to NETL. NETL reserves the right for DOE/NETL employees or agents to witness the sampling and splitting. Results of any analysis arranged by DOE/NETL will be documented in a Publicly Releasable Report accessible on the NETL website.

BUDGET PERIOD 1

Task 5.0 – Feasibility Study

The Recipient will develop and provide NETL a Feasibility Study 30 days prior to the Go/No Go decision point. The Feasibility Study will provide NETL with information on, but not limited to, availability of the proposed feedstock; information on environmental impacts; process flow diagram(s); product yield and concentration; estimated system costs; etc.

At a minimum, the feasibility study should investigate and discuss the following: 1) information developed in the Sampling and Characterization Plan (if applicable) with regard to the available resource and REE and CM grade on a regional or national scale as required for eventual commercial recovery of REEs and CMs from the proposed feedstocks with the proposed technology; 2) expected waste management characterization and proposed processes to minimize or reduce environmental impacts; 3) future advanced manufacturing techniques; 4) a quantified process flow diagram showing REE and CM recovery process input and process flows, including feedstocks, reagents and other additives; 5) identification of process recovery, yield, final product concentration for REEs and CMs and other useful materials; 6) mass/water/energy balances; 7) capital, operating and maintenance, and process costs per unit of input and output; 8) the expected market demand and pricing for REEs and CMs and other useful recovered products on a regional basis; 9) a fully functional financial spreadsheet model with no hidden or locked cells (the model should clearly identify assumptions and include instructions for use by DOE); and 10) feasibility study conclusions and recommendations.

Information and conclusions will be evaluated and reported in a format similar to that used for mineral resource reporting according to Canadian National Instrument 43-101 (NI43-101). Task 5.0 will consist of a written feasibility study report containing but not limited to the information provided above as well as the information developed as part of Tasks 2.0 and 3.0.

Task 6.0 - Large Sample Collection and Preparation

This task will include the collection of a large-sample (>300 tons) of coal, and the physical preparation of the coal, including crushing, drying, and coal cleaning. The final product of the coal cleaning process will be taken as the feedstock to the process of >300 ppm and may be blended with additional higher REE coal (>600 ppm) to evaluate performance.

Subtask 6.1 – Large Sample Planning:

The Recipient will hold discussions with a coal supplier regarding the most effective methods of gathering the large samples for further processing. Mining concerns, particularly involving permitting and selective mining methods, will be re-evaluated for each coal elected for sample extraction.

Subtask 6.2 – Procurement and Preparation:

The coal supplier will work with the Recipient to mine and deliver >300 tons of ND coal (>300 ppm after coal cleaning) for physical processing and coal cleaning. Coal will be crushed to -10 mesh and screened to prepare for downstream operation. Sample analysis, including critical mineral content including but not limited to Ge, Ga, and Co, will be conducted to determine expected project production amounts.

Task 7 – Pilot Plant Design

This task includes pilot plant design, including finalizing site location, the sizing and planning of all equipment, necessary environmental controls, and staffing requirements. Design and permitting will ensure the UND pilot plant meets all current regulations and is recovering and producing REE/CM in an environmentally benign manner.

7.1 – Planning and Conceptual Design of the Pilot Plant

This subtask will consist of the major elements of pilot plant planning, including finalizing plant location, final equipment sizing, and plant layout. Sizing will be largely conducted through scale-up parameters associated with the data from the operating bench-scale equipment. Additionally, the plant location will be finalized, along with necessary utility requirements laid out for successful pilot operation. Control requirements for the pilot plant will be addressed and designed during this phase. A hazards analysis and review of the plant, in addition to the generation of Standard Operating Procedures, will be conducted during the task.

7.2 - Permitting and Logistics Planning

This subtask will include obtaining construction and environmental permits in addition to planning for logistical concerns with continuous pilot plant operation. Wastewater treatment and disposal systems will be developed as per local, state, and federal environmental requirements, along with management with local utilities to ensure effective integration into existing infrastructure. Any actinide management concerns will be addressed during this subtask.

Logistical concerns, particularly in the transport, processing and storage of materials required for seamless pilot plant operation, will be explored. Contact will be established with chemical vendors to confirm shipping regularity and total size for the chosen location, in addition to possible bulk chemical and coal storage on/off site.

GO/NO GO DECISION POINT AFTER TASK 7

The Recipient is NOT authorized to proceed beyond Task 7 without the DOE Contracting Officer's written approval of acceptable progress associated with the Go/No Go technical and economic criteria. The criteria that will be used to determine a Go/No Go decision includes:

- DOE approval of a Technology Readiness Plan, Workforce Readiness Plan, Financial Plan for Commercialization, and preliminary Techno-Economic Analysis and Feasibility Study.
- Copy of the lease agreement and/or host site agreement, as applicable.
- All permits received and the site approved.
- Verified availability of 300+ tons of coal with proven > 300 ppm REE on a cleaned, whole coal basis.
- A preliminary TEA that highlights scenarios with positive Net Present Value (NPV) using the global economic assumptions provided by DOE in Funding Opportunity Announcement (FOA) DE-FOA-0002003.

If the Recipient unilaterally decides to continue into the subsequent tasks prior to the DOE Contracting Officer's written approval, all costs are incurred at the Recipient's risk and no DOE funds may be utilized for such costs prior to the DOE Contracting Officer's written approval of the technical Go/No Go criteria.

BUDGET PERIOD 2:

Task 8 – Pilot Plant Procurement and Construction

Subtask 8.1 – Plant Component Procurement

The procurement of required pilot plant components will be initiated under this subtask. Multiple vendors have been contacted for bench-scale procurement with likely scaled versions of the equipment available. This task will be initiated upon completion of Subtask 7.1 and upon meeting the criterion for a “Go” decision.

Subtask 8.2 – Plant Construction

This subtask will incorporate multiple aspects of the pilot plant’s on-site construction phase, including fabrication of the equipment not supplied by vendors, equipment integration, control system development, and individual component testing to ensure adequate performance from vendors. The pilot plant will be constructed for the continuous and integrated processing of all major unit operations.

Subtask 8.3 – Plant Shakedown Testing

After the continuous pilot plant system is completed, the integrated system will undergo shakedown testing for adequate performance and training for operators, which will verify total system performance.

Task 9 – Bench-Scale Feedstock Testing

This task will perform preliminary parametric testing at the bench-scale for optimized operating conditions for the chosen feedstock. This testing will occur in a semi-continuous nature, where the optimized conditions found for the previous unit operation will be fed into the parametric testing associated with the next unit operation. Final testing of the complete process under optimized conditions will be conducted to ensure expected integrated performance. Testing of an actinide management system for the process (if required) will be evaluated at the bench scale. Determination of critical mineral extractability and expected concentrations in salable products will be evaluated during this task. Concentrations of the major CM expected for the project (Ge, Ga, Co, Al) are as of yet unknown in the feedstock and will be evaluated in Task 6; therefore, final product concentrations and recoveries will be determined in this task. At a minimum, concentration from feedstock to products should be on the order of 100x (i.e. 10 ppm in coal → 1000 ppm in products), with recovery amount from the coal expected >20% (~20% for Ge and Ga, ~50% for Co). Adding Al as a potential recoverable product is contingent upon profitable economics of the unit operation, with the economics determined thus far to be meager to negative.

The material generated during optimized condition testing will then be forwarded to a project partner for testing. Initial samples will be tested to ensure the feedstock performs within specifications utilizing commercially available separations technology. Following verification of performance, the remaining material will be used to optimize the flow stream through the RES process flow to maximize the number of REEs separated when moving to the pilot scale operation.

Task 10 – Pilot Plant Testing of Chosen Feedstock/Feedstock Blend

Subtask 10.1 – Abbreviated Parametric Testing Based on Bench-Scale Data for the Chosen Feedstock

Utilizing results from the bench-scale testing, abbreviated parametric tests around highest opportunity ranges identified through bench testing will be conducted. Each unit operation in use in the pilot plant will be tested sequentially. Economically optimal conditions for each prior unit operation will be the feed for further unit operations.

Subtask 10.2 – Continuous Operation Testing on at Least 100 tons of the Chosen Feedstock

Utilizing optimum parametric conditions for the chosen domestic feedstock, the Recipient will process at least 100 tons of chosen feedstock to generate a minimum of 10 kg of >2wt% REE concentrate. It is expected that REE and CM pre-concentrates of >65wt% will be produced on a rare earth oxide basis. As mentioned in Task 9, CM concentrations in products are expected to be >100x feedstock concentrations, with recoveries at least greater than 20% for all expected. If Al recovery for a secondary CM product is found to be profitable in this process, Al purity should be >60%, with recovery from the coal near 10-15%.

4 SAMPLE COLLECTION, PREPARATION AND ANALYSIS

4.1 Sample Identification and Preliminary Analysis

Coal samples were analyzed prior to and during consideration for award to identify possible coal sources that reach the requisite 300 ppm concentration of REEs, while also focusing on targeting possibly valuable seams for economic purposes. These sources included seams within active mining (in Freedom Mine, Beulah ND and Center Mine, Center ND, various other seams in Falkirk Mine, Underwood ND and Gascoyne Mine, Gascoyne ND), as well as uniquely enriched seams elsewhere in the state (Harmon Bed and Rhame Bed – Slope County ND). A blend was determined to be the most practical form of reaching the ppm threshold, given that the easily accessible sources (within active mining seams within active mines) could not reliably produce a >300 ppm sample over large areas; therefore, it was determined that these valuable, <300 ppm coals must be blended with a high, >500 ppm coal to reach the 300 ppm threshold required by the FOA.

4.1.1 Properties of Parent Coal Feedstocks

The properties of the parent coals used as feedstocks for the REE plant may impact the leaching behavior of REEs from lignites. The properties of the feedstocks are summarized in Table 1 for the REE contents, Table 2 for the CCSEM mineral analysis, and Figure 1 for the CCSEM-derived ternary composition plot of minerals. The key results established that:

- The Rhame Bed coal was higher in overall REE concentrations (477 ppm TREE dry basis) than the Freedom coals (139-243 ppm TREE dry basis), as shown in Table 1.

- The ratio of heavy REE to light REE was inversely proportional to total REE concentrations for all coals. Heavy REE tends to be more valuable and more extractable than light REE.

- The three Freedom coals exhibited differing concentrations of mineral phases, including quartz (16.5 – 32.3 wt.%), pyrite (1.7 – 18.8 wt.% all pyritic phases), gypsum (4.0 – 12.0 wt.%), and clays (7.0 – 12.1 wt.% all clay phases).

- Quartz (silicon dioxide) has a minimal impact on the ability to recover REE. Quartz can cause erosion in coal preparation and handling equipment as well as REE extraction and pumping systems.

- Clay minerals (kaolinite, montmorillonite, K-Al-Silicate (illite), Fe-Al-Silicate (nontronite), Ca-Al-Silicate, NaAl-Silicate, Aluminosilicate, and mixed Aluminosilicate) consist of a complex distribution of minerals. Levels of illite were relatively consistent in all samples. Nontronite (iron-montmorillonite) clay is more abundant in the Freedom North and South region compared to the Freedom West and Rhame coals. These minerals may be associated with the source rock for the REE elements that have accumulated in these coals.

- Pyrite (iron sulfide) and other iron rich sulfide minerals are abundant in the Freedom North and Rhame coals. The Rhame Bed coal was higher in pyritic materials (36.5 wt.% all pyritic phases) relative to the Freedom coals (1.7 – 18.8 wt.% all pyritic phases). Pyritic phases are typically not associated with high levels of REE in coals.
- The presence of gypsum in lignite coals is related to the degree of oxidation or weathering. The Rhame coal contained high levels of gypsum (13%), indicating the oxidation of pyrite producing sulfuric acid that reacted with organically associated calcium.
- Unclassified phases are mixed minerals where the mineral composition cannot be isolated because of mixing. The composition of the unclassified phases for the Freedom and Rhame coals is summarized in Table 3.
- The composition suggests that the mineral phases consist of mixtures of pyrite and clays, including illite clays. In addition, phosphorus is highest in the Freedom North coal, which is the richest in total REE of the three Freedom Mine coals. Phosphorus containing minerals can be rich in REE.

Table 1. Comparison of the REE Concentrations in the Freedom and Rhame Bed Coals**.**

	MTI 20-468 Freedom North	**MTI 20-474 Freedom South**	**MTI 20-500 Freedom West**	**MTI 20-288 Rhame Bed**
Ash, wt.% dry basis	50.0	44.8	27.1	32.3
REE + Sc + Y, ppm dry basis in coal				
Sc	8.9	6.8	5.1	21.5
Y	48.9	25.8	31.3	50.8
Light REE				
La	20.9	11.7	8.4	69.0
Ce	67.4	44.2	35.9	162.6
Pr	18.0	7.7	7.5	20.2
Nd	32.4	11.2	20.1	82.9
Sm	8.5	6.7	6.8	18.0
Eu	2.1	1.4	1.7	4.1
Heavy REE				
Gd	8.1	4.7	6.5	16.4
Tb	0.0	0.6	0.0	2.4
Dy	8.7	6.0	6.7	12.9
Ho	0.0	0.0	0.2	2.4
Er	15.0	9.8	7.8	6.6
Tm	0.0	0.0	0.0	0.9
Yb	3.7	2.3	2.9	5.6
Lu	0.7	0.5	0.5	0.8
Total REE	243.3	139.3	141.4	477.0
Heavy:Light Ratio	0.24	0.29	0.31	0.13

Table 2. Comparison of the CCSEM Analyses of the Freedom and Rhame Bed coals. Results Expressed as Weight Percent Mineral Basis.

MINERAL PHASE	MTI 20-468 Freedom North	MTI 20-474 Freedom South	MTI 20-500 Freedom West	MTI 20-288 Rhame Bed
QUARTZ	22.9	32.3	16.5	30.5
IRON OXIDE	0.6	0.9	0.3	3.7
RUTILE	0.5	0.8	0.1	0.1
ALUMINA	0.0	0.0	0.0	0.0
CALCITE	0.0	0.1	0.0	2.0
DOLOMITE	0.0	0.1	0.0	0.1
ANKERITE	0.2	0.0	0.0	0.0
KAOLINITE	0.2	0.6	0.4	0.1
MONTMORILLONITE	0.2	0.4	0.2	0.1
K AL-SILICATE	4.4	2.7	4.6	3.0
FE AL-SILICATE	3.5	3.9	1.0	0.2
CA AL-SILICATE	0.0	0.0	0.0	0.0
NA AL-SILICATE	0.2	0.5	0.3	0.0
ALUMINOSILICATE	0.3	0.8	0.0	0.0
MIXED AL-SILICATES	3.5	1.2	0.5	0.7
FE SILICATE	0.3	0.5	0.5	0.1
CA SILICATE	0.1	0.0	0.0	0.0
CA ALUMINATE	0.0	0.0	0.0	0.0
PYRITE	10.6	0.0	6.0	28.7
PYRRHOTITE	3.6	0.7	6.3	5.5
OXIDIZED PYRRHOTITE	4.6	1.0	4.3	2.3
GYPSUM	6.8	12.0	4.0	13.0
BARITE	0.0	0.1	0.0	0.3
GYPSUM/BARITE	0.0	0.0	0.0	0.0
GYPSUM/AL-SILICATE	0.0	0.1	0.1	0.0
SI-RICH	6.1	11.8	5.8	2.1
CA-RICH	0.1	0.1	0.2	0.1
CA-SI RICH	0.0	0.0	0.0	0.0
UNCLASSIFIED	31.4	29.2	48.8	7.4
TOTALS	100	100	100	100

Table 3. Comparison of the Unclassified Phase Composition of the Freedom and Rhame Bed Coals (Wt % equivalent oxide).

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	Fe_2O_3	BaO
MTI 20-468 Freedom North	1.3	2.2	12.1	49.5	2.4	13.5	0.2	2.4	0.9	2.2	11.4	2.0
MTI 20-474 Freedom South	2.0	3.4	10.3	43.2	1.9	13.2	0.3	1.8	2.5	3.4	15.4	2.8
MTI 20-500 Freedom West	2.7	2.3	9.0	32.6	1.4	29.9	0.3	0.9	1.6	2.5	13.6	3.1
MTI 20-288 Rhame Bed	4.3	2.5	7.7	26.1	1.4	37.7	0.9	2.9	3.0	1.0	11.1	1.4

The CCSEM data were used to create ternary diagrams to compare the distribution of mineral components in the coals. An example of ternary diagram data is listed for the Freedom coals compared to Rhame coal in Figure 1. The relative abundance of different mineral particle compositions are displayed in the 3D ternary diagrams. The 3D ternary diagrams show the variability of the composition of mineral observed, including clays and quartz (on the Al+Si endpoint), pyrites (on the Fe endpoint), and alkali/alkaline earth elements (on the Ca+Mg+Na+K endpoint). The results indicate that the Freedom North and South locations are dominant in aluminosilicates and quartz, while the Freedom West and Rhame Bed locations have high levels of aluminosilicates and pyrites. The relatively high-REE Freedom North coal, in particular, contains a peak just outside the Al+Si endpoint, corresponding to clays such as illite or mixed aluminosilicates that contain alkali/alkaline earth elements in the aluminosilicate structure. High REE in Freedom coals, such as the Freedom North coal, may be associated with or derived from these clays. In contrast, the high-REE Rhame Bed coal does not exhibit a second mixed aluminosilicate peak, indicating that the REE in the Rhame Bed coal may have migrated to the coal from a different source or originated from non-aluminosilicate sources.

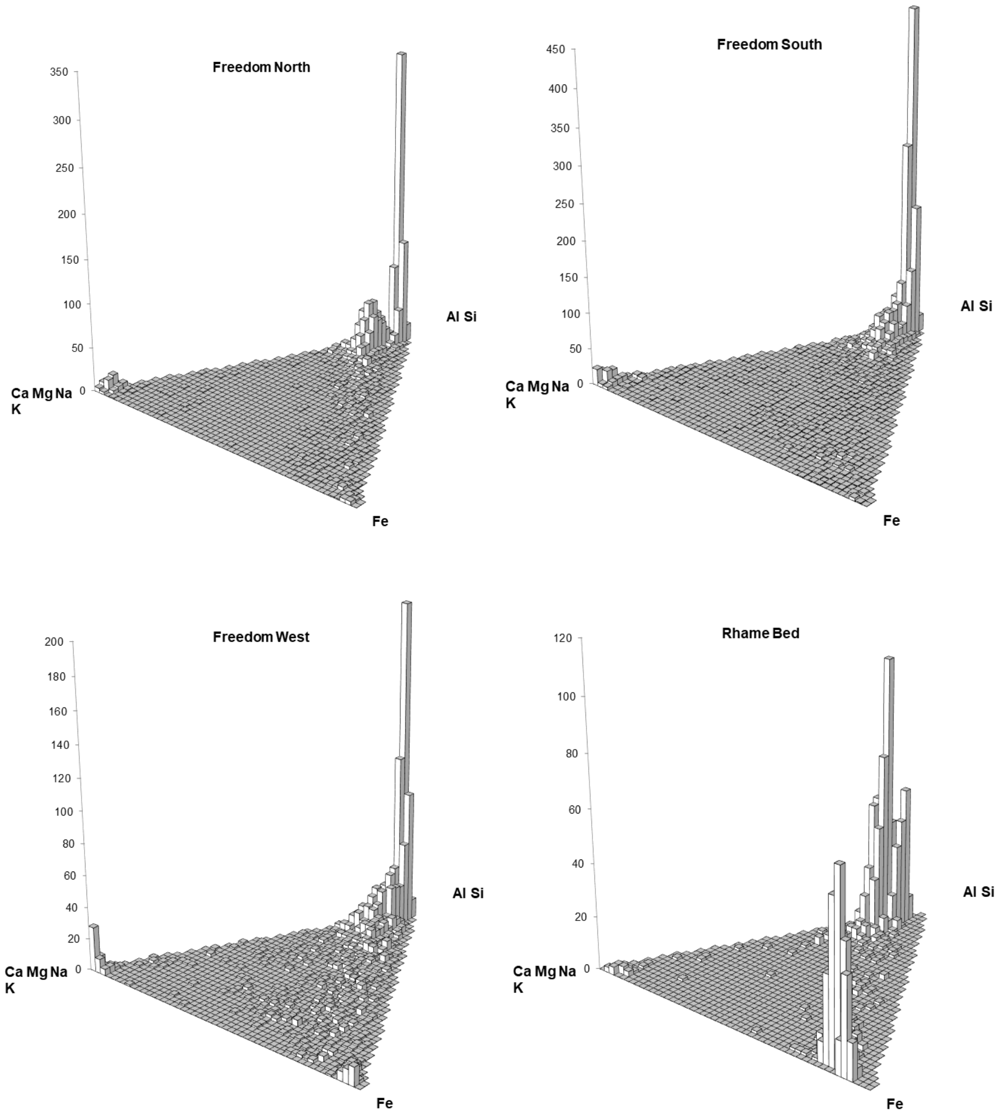

Figure 1. Ternary Diagrams for Freedom North, South, and West Compared to the Rhame Bed.

4.1.2 Properties of Density-Fractionated Coal

The pilot plant's original design included a density-based spiral separation system to concentrate REE-rich coal for the plant. The performance of the density-based separation system was assessed by analyzing different density fractions. Three sets of coal samples (North, South, and West) from the Freedom Mine were collected and subjected to float/sink separation analyses. The sample was separated into the following density groups, with units of specific gravity (g/mL): 1.5 FL (float), 1.5x1.7, 1.7x1.9, 1.9x2.1, and 2.1 SK (sink). The starting feed coal was analyzed along with each separated density group. Detailed results from this analysis are provided in Appendix A.

The key results include:

- The three coals exhibited differing density distributions. A density-based separation system may need to operate flexibly to accommodate these different types of feedstock coals.
- Mineral phases, including quartz, pyrite, gypsum and clays, tended to be enriched in the higher-density fractions, while unclassified/mixed inorganic materials were enriched in the lower-density fractions. Density-based separation systems may utilize 1.7 g/mL or 1.9 g/mL as an appropriate cutoff in order to segregate coals with lower levels of these mineral phases. The lower levels of mineral phases also come with higher levels of unclassified/mixed inorganic materials.
- For the coals tested, REEs were not consistently concentrated or depleted in any of the different density fractions. Density-based separation is not expected to result in a REE-enriched feedstock in downstream processes.

The three coal samples from the Freedom Mine (North, South, and West) exhibited differing density distributions, as shown in Table 4. The density distribution of the South and West coals showed a peak in the 1.5x1.7 density group (South: 30.4 wt.%, West: 68.7 wt.%). The North coal density distribution was mainly contained in the lowest density (25.6 wt.% <1.5 g/mL) and highest density (29.7 wt.% >2.1 g/mL) groups.

Table 4. Mass fractions of Density-Separated North, South, and West Coals.

	North	**South**	**West**
	Mass fraction, wt.%		
Density fraction (g/mL)			
1.5 FL (float)	25.6	15.0	4.2
1.5x1.7	15.4	30.4	68.7
1.7x1.9	19.2	14.4	7.4
1.9x2.1	10.1	13.0	6.9
2.1 SK (sink)	29.7	27.3	12.9

Mineralogy (CCSEM)

The CCSEM analyses of the three Freedom mine coals revealed that all coals had high amounts of unclassified phases, with the West sample having the most. The North coal had the highest quartz content and the highest pyritic phase content. The South coal had the highest gypsum and silicon-rich phase content and the least amount of pyritic minerals. The West coal had the least amount of quartz, clays, gypsum, and silicon-rich minerals.

- The CCSEM analysis of the coal float/sink density fractions revealed that the unclassified phases tend to concentrate in the **lower density groups** and decrease in concentration as the density increases.
- Quartz content tended to concentrate in the **higher density groups** for the North and South coals. The quartz distribution in the West coal was mostly in the 1.9x2.1 density group, with little in the other density groups.
- Pyrite tended to be concentrated in the **higher density groups** for the North and West coal samples. The South coal had most of the pyrite in the 1.5x1.7 density group.

- Gypsum tended to be concentrated in the **higher density groups** (1.9x2.1 and 2.1 SK (sink) groups) for all coals.
- The clays/silicates of the North coal were concentrated mostly in the **higher density groups** (1.9x2.1 and 2.1 SK (sink) groups) for all coals.

REE Analysis

The Freedom Mine coal samples (North, South, and West). along with their corresponding float/sink fractions. were analyzed for their Rare Earth Element (REE) concentrations using ICP-OES procedures. Detailed results from this analysis are presented in Table 5 through Table 7. The results indicate that the REE's are spread throughout all of the density fractions. The float/sink density separations did not tend to concentrate the total REE into any one or more density fractions (Figure 2).

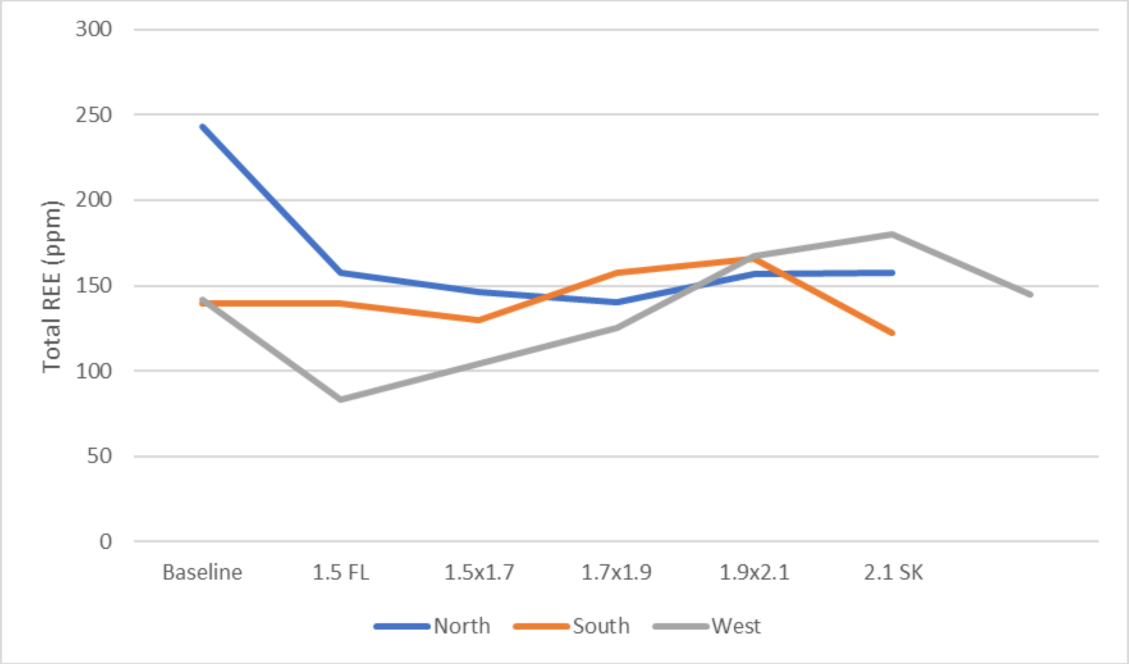

Figure 2. Distribution of Total REE Content Across Different Density Separations for the Float/Sink Samples.

Table 5. REE Analysis of Freedom Mine North Coal and Float/Sink Density Groups.

			ppm on a dry coal basis															
	Sample ID	Ash %	Sc	Y	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Total REE
20-468	North FC	50.02	8.94	48.93	20.9	67.41	18	32.38	8.46	2.05	8.09	0	8.7	0	14.98	0	3.74	0.72	243.3
20-463	North 1.5 FL	6.39	5.59	35.22	7.48	37.79	10.81	26.45	7.71	1.51	6.65	0	6.5	0.45	8.54	0	2.57	0.6	157.87
20-464	North 1.5x1.7	28.75	5.65	34.72	10.45	36.03	7.89	21.9	6.18	1.45	5.46	0	6.18	0.13	7.3	0	2.37	0.33	146.04
20-465	North 1.7x1.9	39.94	5.6	27.92	11.86	39.4	6.82	21.38	5.6	1.21	4.2	0	5.23	0	8.5	0	2.15	0.28	140.15
20-466	North 1.9x2.1	68.00	6.78	25.12	15.56	50.7	6.93	22.5	7.09	1.08	2.77	0	4.78	0	11.1	0	2	0.46	156.87
20-467	North 2.1 SK	76.61	5.27	21.25	17.98	59.22	15.44	9.81	5.09	1.09	1.27	0	5.45	0	13.99	0	1.63	0.18	157.67

Table 6. REE Analysis of Freedom Mine South Coal and Float/Sink Density Groups.

			ppm on a dry coal basis																
	Sample ID	Ash %	Sc	Y	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Total REE
20-474	South FC	44.84	6.77	25.84	11.74	44.24	7.67	11.17	6.66	1.35	4.74	0.56	5.98	0	9.82	0	2.26	0.45	139.25
20-469	South 1.5FL	12.37	5.35	25.93	6.28	37.9	7.21	26.39	7.91	1.51	5.12	0	5.81	0	7.79	0	2.09	0.35	139.64
20-470	South 1.5x1.7	23.83	6.61	25.64	7.99	32.54	7.65	21.96	5.92	1.44	5.58	0	5.29	0.17	6.61	0	2.24	0.4	130.04
20-471	South 1.7x1.9	37.17	7.64	29.33	11.64	42.21	9.06	25.15	6.4	1.51	5.42	0.36	5.95	0	9.69	0	2.58	0.44	157.38
20-472	South 1.9x2.1	59.60	7.45	25.02	15.19	52.57	7.6	22.19	8.34	1.34	3.57	0	6.11	0	13.4	0	2.38	0.6	165.76
20-473	South 2.1 SK	77.30	5.47	14.22	13.85	52.13	8.93	0	6.93	0.73	1.09	0	5.1	0	12.21	0	1.46	0.36	122.48

Table 7. REE Analysis of Freedom Mine West Coal and Float/Sink Density Groups.

									ppm on a coal basis									
	Sample ID	Ash %	Sc	Y	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Total REE
20-500	West FC	27.10	5.06	31.33	8.35	35.85	7.53	20.11	6.84	1.71	6.5	0	6.7	0.21	7.8	0	2.94	0.48	141.41
20-494	West 1.5 FL	3.21	2.35	21.55	3.2	17.82	2.87	14.1	4.44	1.11	4.83	0.07	4.51	0.39	3.4	0.07	2.02	0.39	83.12
20-495	West 1.5X1.6	7.45	3.38	25.53	4.73	23.43	3.75	17.35	5.33	1.35	6.01	0.23	5.41	0.45	4.13	0	2.48	0.45	104.01
20-496	West 1.6X1.7	14.54	4.68	30.57	6.22	26.99	5.56	20.19	5.92	1.68	7.24	0.8	6.44	0.44	4.97	0	3.07	0.59	125.36
20-497	West 1.7X1.9	36.88	6.37	36.14	11.61	44.95	5.06	25.28	6.37	1.69	7.87	1.31	7.68	0	8.61	0	3.56	0.75	167.25
20-498	West 1.9X2.1	64.87	6.52	31.96	17.94	61.96	6.85	19.89	5.22	1.3	5.54	0.33	7.17	0	11.74	0	2.94	0.98	180.34
20-499	West 2.1 SK	75.93	7.41	23.78	25.73	53.41	3.51	6.24	0	0.78	4.29	0	6.63	0	10.53	0	1.95	0.78	145.04

4.1.3 Properties of the Center Mine Resource

The Center mine was evaluated as a potential resource for processing in UND's pilot REE concentrate production facility. A total of eleven samples were collected from the Hagel A seam. Each sample was subjected to proximate, ultimate, ash composition, heating value, and CCSEM analyses.

The results revealed significant variations in feedstock properties, similar to the variations in the properties of the Freedom coal analyzed earlier. The Center coals are expected to be similar to the Freedom coals in terms of their properties and impacts on REE plant performance.

Proximate/Ultimate

The moisture levels for the samples ranged from 33.36 wt.% in sample 20-570 to 48.08 wt.% in sample 20-574. The "as-received" ash levels ranged from 3.25 wt.% in sample 20-569 to 13.49 wt.% in sample 20-574. Sample 20-567 had the highest heating value, at 7056 BTU/lb, while sample 20-575 had the lowest, at 6282 BTU/lb. Sulfur content was highest in sample 20-570, at 2.86 wt.%, and lowest in sample 20-571, at 0.34 wt.%.

Ash Composition

The ash composition varied quite widely from sample to sample, with samples 20-570, 20-569, 20-576, and 20-577 having low levels of silica and alumina. Samples 20-570 and 20-576 had high iron content, at 59.37 wt.% and 29.53 wt.% Fe_2O_3, respectively. Sodium in the ash ranged from 8.09 wt.% Na_2O in sample 20-567 to a low of 0.64 wt.% Na_2O in 20-570. The sulfur retained in ash samples 20-569, 20-572, 20-573, and 20-576 was high, ranging from 29.00 to 32.00 wt.% SO_3. Sample 20-574 had the lowest sulfur retained, at 8.38 wt.% SO_3.

CCSEM

Table 8 compares the CCSEM mineral phase analyses for the eleven coal samples. The unclassified phases in the CCSEM analyses were high (28 to 32 wt.%) for most samples. Samples 20-570, 20-575, and 20-576 contained lower levels of unclassified phases (6 to 24 wt.%), while samples 20-569, 20-571, and 20-577 contained higher levels of unclassified phases (39-46 wt.%). The samples with the lowest unclassified content also had the highest pyritic phase content and lowest quartz content. Clays and mixed silicates varied from 0 wt.% in 20-570 to 29.4 wt.% in 20-568 and 20-575. Sample 20-574 contained a high amount of gypsum (19.0 wt.%).

Table 8. Comparison of the CCSEM Analyses for the Eleven Coal Samples From the Center Mine, Hagel A Seam (wt % mineral basis).

MINERAL PHASE	20-567	20-568	20-569	20-570	20-571	20-572	20-573	20-574	20-575	20-576	20-577
QUARTZ	15.5	7.3	19.1	0.4	10.4	24.8	24.2	14.6	12.4	5.3	5.1
IRON OXIDE	0	0.4	4.4	0.7	0.8	0.8	2.7	2.5	0.9	0.6	1.2
PERICLASE	0	0	0	0	0	0	0	0	0	0	0
RUTILE	0	0.6	0.1	0	0	0	0.4	0	0	0	0.5
ALUMINA	0	0	0	0	0	0	0	0	0	0	0
CALCITE	2.4	0.7	4.2	0.8	7.0	0.4	4.3	7.0	0.4	3.1	3.5
DOLOMITE	0.2	0.1	0.5	0.1	1.8	0	0.3	0.3	0.5	0.6	0.2
ANKERITE	0.2	0	0.3	0	1.3	0.2	0.4	0.3	0.1	0.4	0.4
KAOLINITE	11.8	8.4	0.2	0	0.4	2.3	0.4	0.1	9.2	0.2	12.4
MONTMORILLONITE	1.8	0.5	0.4	0	0.3	3.4	6.1	0.3	3.8	0.0	1.1
K AL-SILICATE	0.7	10.5	3.9	0	2.7	0.5	1.8	4.3	7.0	1.0	1.0
FE AL-SILICATE	0.2	2.2	3.1	0	7.8	4.6	2.5	5.4	1.3	0.4	0.0
CA AL-SILICATE	5.1	2.0	0.7	0	1.8	8.8	1.1	0.2	0.6	0.2	1.7
NA AL-SILICATE	1.2	1.6	0.7	0	2.3	0.9	1.6	3.1	1.4	0.5	0.3
ALUMINOSILICATE	2.0	1.0	1.3	0	1.2	3.6	1.5	0.3	2.8	0.2	1.0
MIXED AL-SILICATES	4.5	4.2	2.9	0	2.0	3.3	3.3	2.5	3.3	0.6	2.0
FE SILICATE	0.2	0	0	0	0.1	0.2	0	2.1	0	0.1	0
CA SILICATE	1.0	0.6	0.5	0.1	2.1	0.4	4.2	0.6	5.4	1.7	1.0
CA ALUMINATE	0	0	0	0	0	0	0	0	0	0	0
PYRITE	8.9	11.9	3.5	73.3	3.6	2.2	4.3	0.5	14.5	38.2	3.5
PYRRHOTITE	1.9	4.3	0.7	13.3	2.0	0.7	4.1	0.2	3.4	9.8	0.7
OXIDIZED PYRRHOTITE	0.1	0.7	0.2	3.0	0	0.3	0.3	0.3	1.0	4.8	0.0
GYPSUM	1.5	2.0	0	0.7	4.4	0.7	2.2	19.0	5.5	2.2	5.9
BARITE	0.3	0	1.0	0	0	0	0	0	0	0.1	0
APATITE	0	0.2	0.2	0	0	0	0	0.6	0	0	0
CA AL-P	0	0	0	0	0	0	0	0	0	0	0
KCL	0	0	0	0	0	0	0	0	0	0	0
GYPSUM/BARITE	0	0	0	0	0	0	0	0	0	0	0
GYPSUM/AL-SILICATE	2.4	2.0	1.9	0.1	2.2	4.5	0.5	0.4	0.8	0.3	3.3
SI-RICH	3.1	8.1	5.3	0.0	2.1	3.8	4.2	3.6	6.2	1.7	1.3
CA-RICH	2.1	0.4	1.0	0.5	4.3	0.9	1.5	1.6	0.9	3.8	6.7
CA-SI RICH	0.1	0.1	0.8	0.0	0.6	0.5	0.3	0	0.7	0.4	0.8
UNCLASSIFIED	32.8	30.2	43.1	6.7	39.1	32.0	27.9	30.2	17.7	23.7	46.4
TOTALS	100	100	100	100	100	100	100	100	100	100	100

4.1.4 Properties of Parent and Leached Coals from Pilot Plant Operations

Analyses were performed on parent and leached coals from the pilot plant to understand changes in coal properties through the leaching process. A summary of the results is discussed in this report.

The key findings include:

- Ash is more variable and does not significantly decrease in the leached coals compared to the parent coals. The extraction of REE from the coal may be causing mineral segregation, leading to more variability in the leached coal ash content. Obtaining a representative sample from the filter cakes is challenging and likely the reason for the variability. Ash content for the 60% Freedom 40% Rhame-Bed parent coals ranged from 41.01 to 50.60% (dry basis). Ash ranged from 47.42 to 67.04% on a dry basis for most of the pilot leached samples.
- The ash composition (wt. %) of the leached coals consisted primarily of silicon dioxide (>60%), aluminum oxide (~10-13%), and iron oxide (~6.5-14.5%). Sodium oxide was low (<1%). In general, silicon dioxide and barium oxide were enriched in the leached coals, while magnesium oxide, sodium oxide, phosphorus pentoxide, and manganese dioxide were depleted in the ashes derived from leached coals compared to the parent coals. Iron oxide and sulfur trioxide were also slightly depleted in the leached coals. Decreased sodium and magnesium resulted from the removal of organically associated sodium and magnesium using the mineral acid leaching process.
- CCSEM mineral analyses of the leached coals revealed increased quartz content compared to the parent coals. The leached coals' minerals were comprised primarily of quartz, silica-rich, and Al-silicate minerals in varying quantities. The leached coals also contained 16.0-51.6% unclassified minerals. The unclassified minerals in the leached coals consisted mainly of silicon, iron, aluminum, and barium. Two of the leached coal samples contained high sulfur and calcium containing minerals. Quartz was enriched in the leached coals due to the removal of other minerals.

4.2 Sample Extraction Planning and Collection

A blend of two coals from the Freedom Mine and Rhame beds were decided on for collection based on the sampling and analysis results from the coals tested for large sample collection. Each sample posed separate challenges for planning and collections, and were achieved through two separate methods, with both delivered to UND prior to March 2020.

Freedom Mine Sample Collection

High-REE mine wastes from the top of a seam in the Freedom Mine were selected as the bulk of the total tonnage of sample collection, with the final collection conducted by North American Coal Corporation. This recovery was conducted during standard mining operations, with the target sample cleaned-to and removed from the coal in separate steps using available smaller equipment, with a total of ~80 acres of mine waste collected from the top of the target seam, resulting in ~ 400 tons initially collected from this seam.

demonstrates this process and final pit piling, prior to collection into super sacs and transported to UND's pilot facility.

Figure 3. Removal of Overburden and Cleaning of the Top Mine Waste Layer.

Figure 4. Mining Operation and Equipment from the Freedom Mine Sample Collection.

Figure 5. Piled Mine Waste Sample Prior to Bagging and Shipment.

The Freedom mine coal mine waste sample was shipped and delivered to UND in individual super sacs, and the coal was stored for facility construction and use.

Rhame Bed Excavation

The Rhame bed had been selected as the highest-likelihood blending coal because it significantly exceeded 300 ppm, reducing risks associated with bulk coal collection. A significant sample of this high-REE coal was extracted with the assistance of the North Dakota Geologic Survey and local landowners. Permits from the North Dakota Trust Lands Commission and the Department of Mineral Resources (state of ND, permit number row 8630) were acquired to mine a ~30-35-ton sample from the outcrop previously mined in Slope County, ND, through the assistance of the North Dakota Geological Survey and the local landowner. Preparations for mining were planned for January 2020, when the ground would be frozen and reduce the ecological impact of the mining trip.

Plans for extraction, including depth into the outcrop face, amount of overburden, and safe coal transportation to the roadside, were planned and executed prior to coal mining (Figure 6). Coal was planned to be extracted by use of two skid-steer equipment with various attachments and transported with a front end-loader to the roadside. An estimated 110 tons of overburden, comprised primarily of sandstone and mudstone, would need to be removed prior to coal excavation. Site selection was chosen due to the minimal expected impact from the nearest navigable road (Figure 7).

Figure 6. Site Satellite Picture with Expected Required Scoria Trail Layout (red line).

Figure 7. View of the Pit Mining Site from the Road (red line).

The team required for mining included the landowner, two equipment operators in the area, three personnel from the NDGS, and a mixed team of UND and MTI personnel. Expected mining time was predicted at five days after completion of the scoria road. Reclamation work for the site was planned to occur primarily in the spring/fall after the mining trip to reduce the slip risk of equipment and personnel on the snowy slopes. Permitting guidelines require reclamation efforts to be complete and satisfactory by December 31st, 2020. The mining process was conducted similarly to the Freedom Mine effort, with a removal of bulk overburden, a detailed cleaning of the coal seam, the extraction of the coal itself, and bagging into super sacks (Figure 8-10).

Figure 8. Removal of Overburden Overlaying the Target Coal Seam.

Figure 9. Cleaning of the Coal Seam and Coal Sample Collection.

Figure 10. Final Extraction of the Coal Seam and Bagging into Super Sacks.

The Rhame bed material was analyzed using grab samples from each bag and homogenizing across all bags containing ~44 tons, with an average concentration of TREE at 477 ppm, suitable for blending into the Freedom Mine sample, for an average of 300 ppm across at least 100 tons.

5 BENCH-SCALE MREC TESTING

The goal of bench-scale testing was to i) establish a baseline expectation for pilot performance from the chosen coal blend, and ii) produce meaningful extraction and processing data of fresh material. The latter was increasingly important due to the requirement for the resource available prior to pilot construction, causing weathering of the resource over the construction and commissioning period.

Leaching Testing

Leaching tests were conducted on the bench-system utilizing the above coal blended feedstock with a similar liquid/solid ratio as expected from the spirals operation of 3:1. Coal feed rates were set at 23 kg/hr, capped through ancillary equipment utilizing a higher L/S ratio than what was previously designed. As a necessity of the recycle stream present within the leaching circuit (the water washing the coal prior to disposal/sale was utilized as the feed liquid for leaching), developing the solutions to a steady state was required to be able to conduct quantitative testing on the effectiveness of the other processes. A singular pH target was used for the leaching and washing stages, and to accelerate the process of concentrating (and test whether the possibility may work at the pilot scale). The leachates from the first four tests were utilized as the feed liquid for the following tests. Coal washing ratios were designed to minimize excess water production, generating only the water required for leaching at the 3:1 ratio defined above.

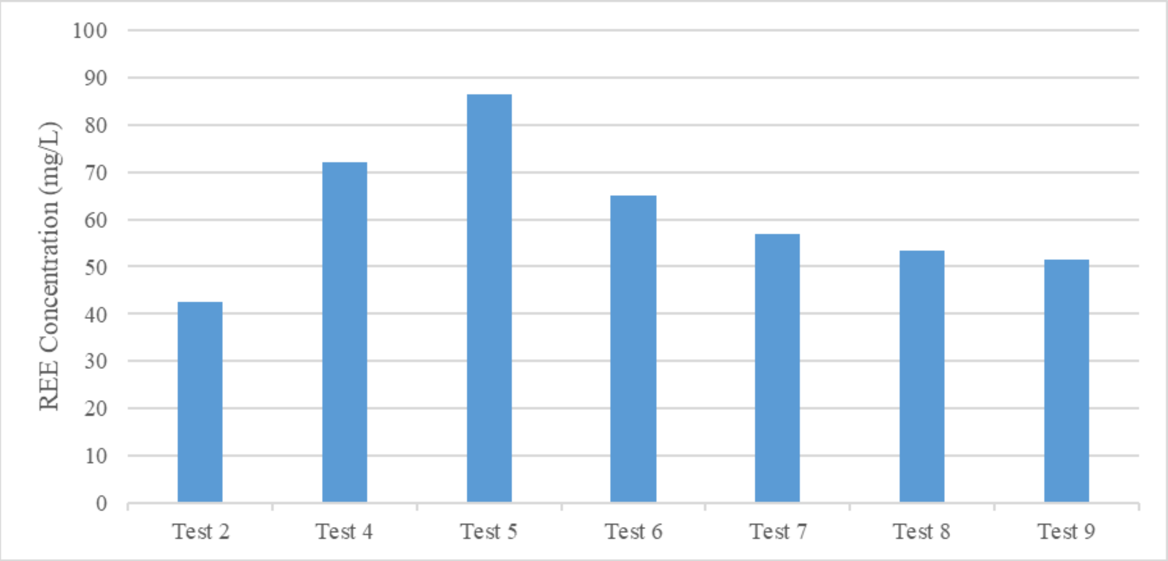

Figure 11. Total Concentration of the REE within the Bench-Scale Leachates as a Function of Time (tests).

The <10% change from tests 7→8, then 8→9 was taken as evidence of steady state behavior within the leachate and could be backed up with the washing liquid concentration (Figure 12). Again, a similar pattern of gradual leveling into the steady-state concentration may be shown and also used to identify the value in utilizing this washing stream, containing approximately 15-20% of the REE found within the leachates (permitting further recovery of potentially lost material).

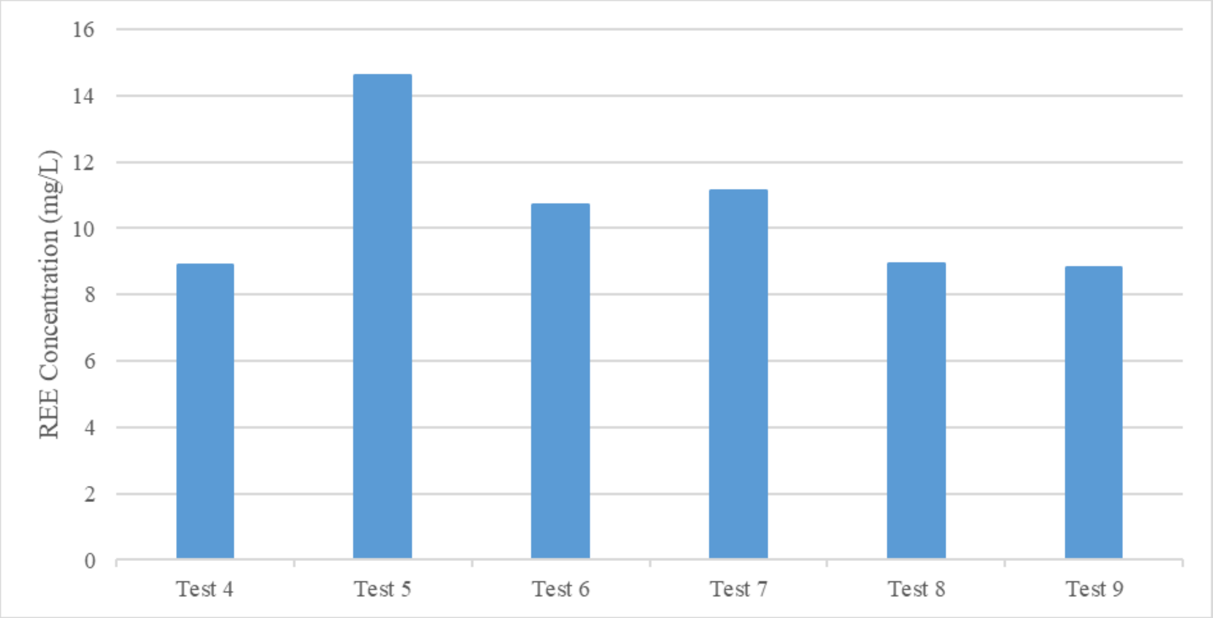

Figure 12. Total Concentration of the REE within the Washing Fluid as a Function of Time (testing).

UND moved forward with leaching testing after reaching an equilibrium in the leaching circuit, testing a wide range of equilibrium pH values at the bench scale to determine specific leaching conditions under which the highest economic position could be reached. Based upon the updated economic models, leaching efficiencies as a function of pH and coal type were the only parameters expected to be required in the determination of high-level economics; the other process parameters may be calculated for liquid conditions and concentrations.

Impurity Removal

A mixed leachate batch of 300 gallons was produced to determine impurity removal parameters at the bench scale in a semi-continuous fashion. The parameters of interest involved the use of a divalent base and a monovalent base, with a third test grouping using a divalent base and the addition of a monovalent salt to improve precipitation conditions. The divalent bases were expected to reduce processing costs if enough REE could be retained within the liquid. Each group was tested at two maintained pH levels each and analyzed for REE and Fe precipitation (Figure 6.)

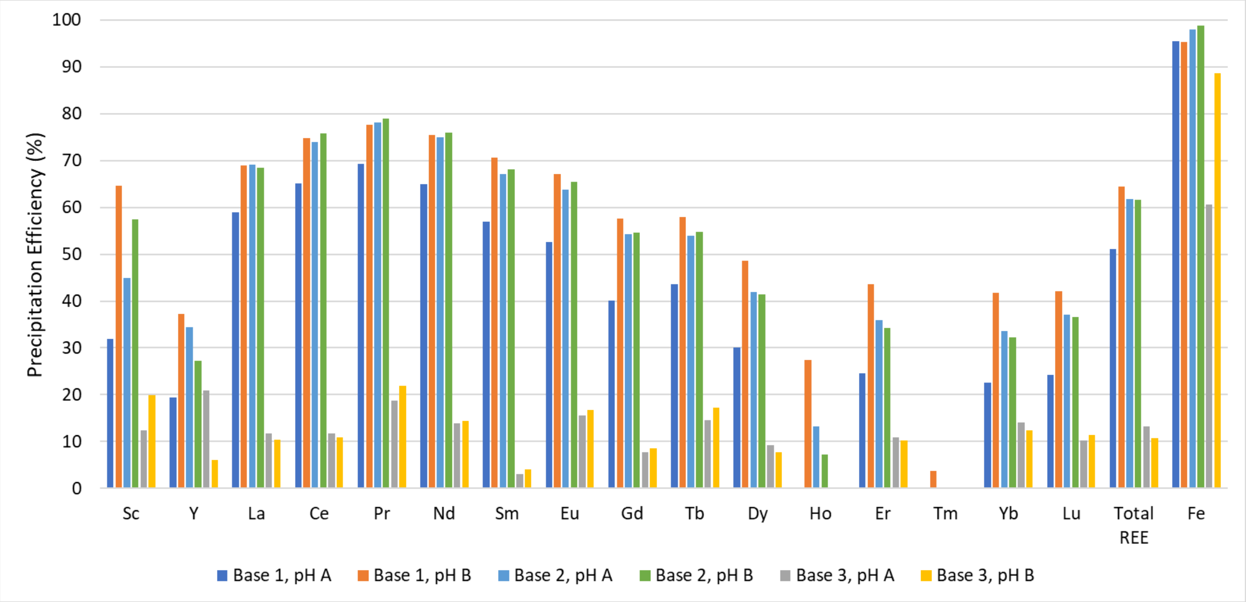

Figure 13. Impurity Removal Bench-Scale Testing. (Base 1 is a divalent base, Base 2 is a divalent with monovalent salt, and Base 3 is a monovalent base. As a note, Tm and Ho were near the limits of detection in the feed liquid, explaining the abnormal behavior of the elements).

Based on the results, the decision was made to continue using the monovalent base and the higher pH value (pH B in these tests) because the REE precipitation exceeded the cost savings of the cheaper bases. These tests were conducted with a residence time of two hours in the pH maintenance processing tank and contrasted previous experiments at similar conditions (from DE-FE0027006). As a result, future tests were targeted with a 2.5-hour residence time to ensure equilibrium conditions were met, typically reducing REE loss in prior testing. For the impurity removal testing conducted for the next parametric testing, total REE precipitation was 7%, down from 11% from previous testing.

REE Precipitation

Goals of identifying the minimum concentration of oxalic acid necessary to near-quantitatively precipitate the REEs (and therefore minimize Ca co-precipitation and improve overall concentrate purity) were met. Prior work had concluded that the oxalate ion could be bound in solution by other trivalents present in the liquid (Al and Fe as the majority); therefore, the target concentration is difficult to predict and has been most usually identified through testing. Four concentrations were tested, targeting the area where predictive calculations assumed where the region of precipitation would lie, varied each by 0.05 mole/L concentrations.

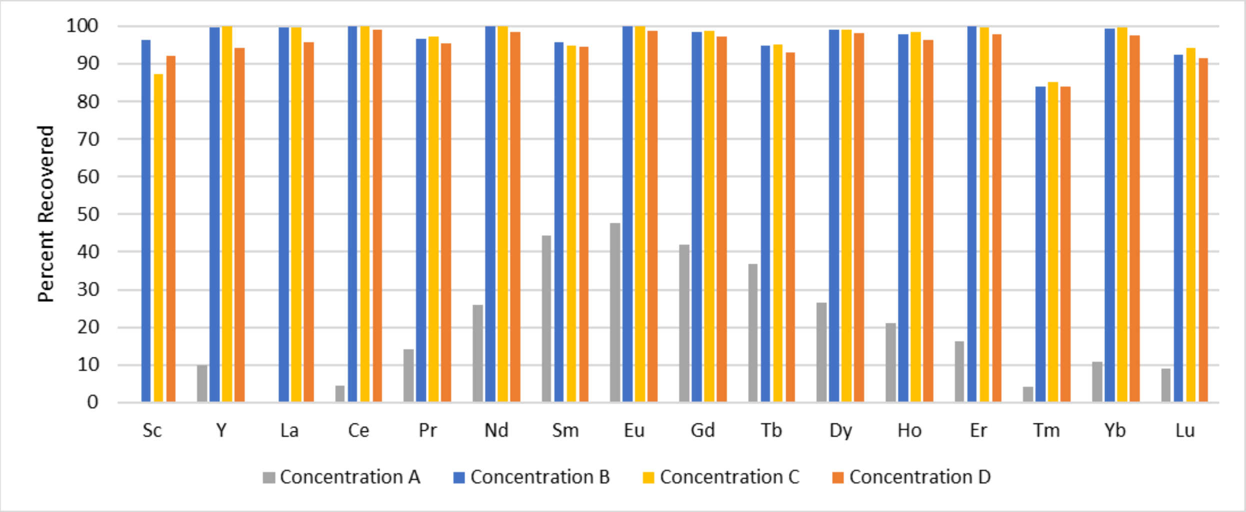

Figure 14. REE Precipitation as a Function of Oxalate Concentration (increasing from A → D).

Experimentation results revealed that complete precipitation occurred by the second tested instead of the first; therefore, an intermediate concentration between these values was chosen for further testing (to be completed early in the next reporting period). The produced solid purities were relatively poor for all solids (none exceeded 10% REO's), primarily because of poor REE precipitation in the first solid and too much precipitation of impurities by the second concentration tested.

Concentrate Upgrading and Refining

New methods of concentrating the final REE concentrate from <70% to >90%, while removing low-value elements from the blend, were developed. Calcining the REE concentrate from the oxide cake at an elevated temperature (>1000°C) permitted the near-complete exclusion of Ce and Fe from re-dissolution attempts. Both low-value elements are viewed as impurities from an economic point of view. Selective precipitation of the dissolved fluid resulted in similar concentrations (~75%) as previous, with Ca remaining the primary impurity. For this reason, solid-state ion exchange resins were investigated for REE/CM separation.

Multiple major resin types were investigated, including weak and strong cationic exchanges; however, two major front-runners were identified in preliminary testing. The first was an aminomethylphosphonic acid chelating resin (IRC 747 from DuPont - AMP), and the second was an iminodiacetic acid resin (TP-260 from Lanxess - IDA). Both resins exhibited selective absorption of the REE's and CM's compared to Na and Ca, while loading the resins below a pH of 4 and were viewed as promising candidates. Further elution testing on columns was conducted, with samples taken at each bed volume for stripping curve development.

Figure 15 presents the results when loading 126 mL of an IDA resin at 5 bed volumes (BV) per hr. The heavy rare earths began to break through, first starting around the 6th bed volume, which corresponds to an estimated operating capacity of 0.05 mEq/mL, 4% of the total capacity reported by the manufacturer. This result was deemed too low to be economically feasible and is likely due to high competition between the REEs and H^+ for the active sites at our operating pH.

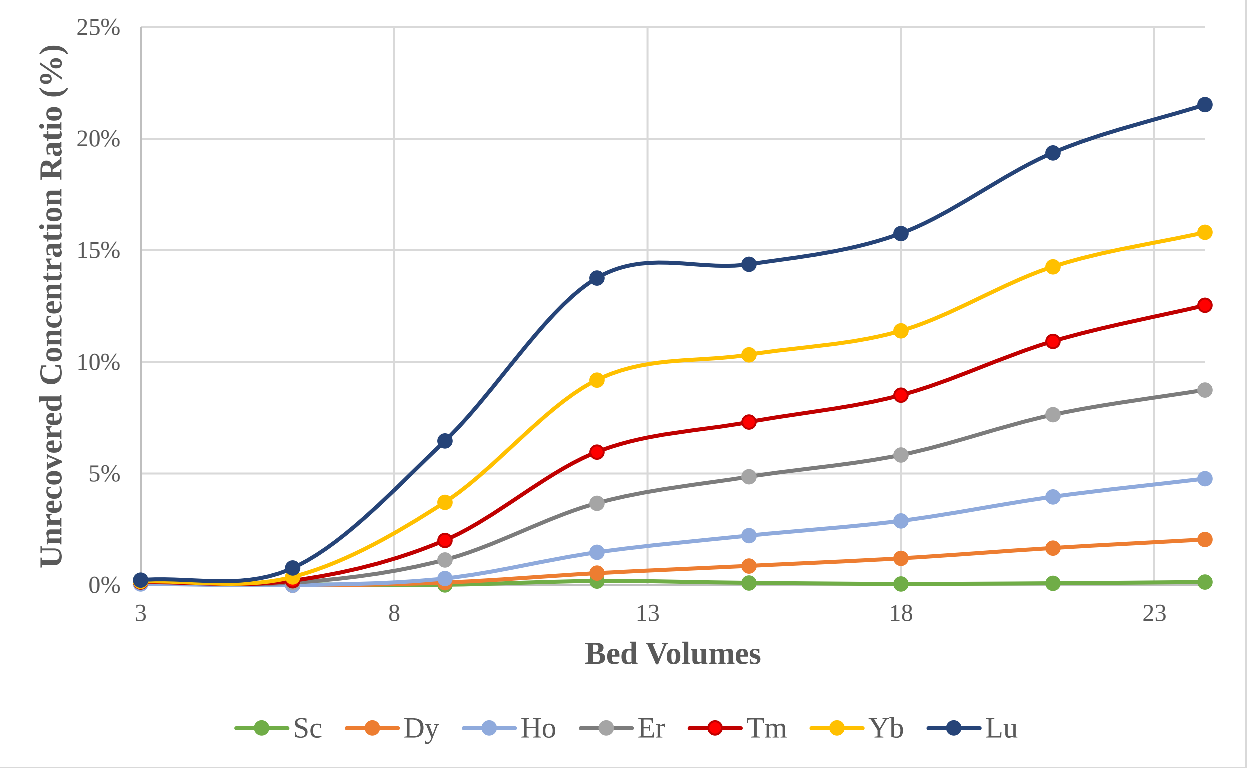

Figure 15. Loading Curve for IDA Resin in the H^+ Form.

Figure 16 presents the stripping curve for the IDA resin using 5% HCl at 3 BV/hr. Calcium co-loading onto the resin was very low and mostly eluted in the first bed volume. The rare earths all peaked in bed volume two and exhibited very little tailing. Even scandium, the strongest held rare earth based on the loading performance, easily elutes from the resin under these conditions.

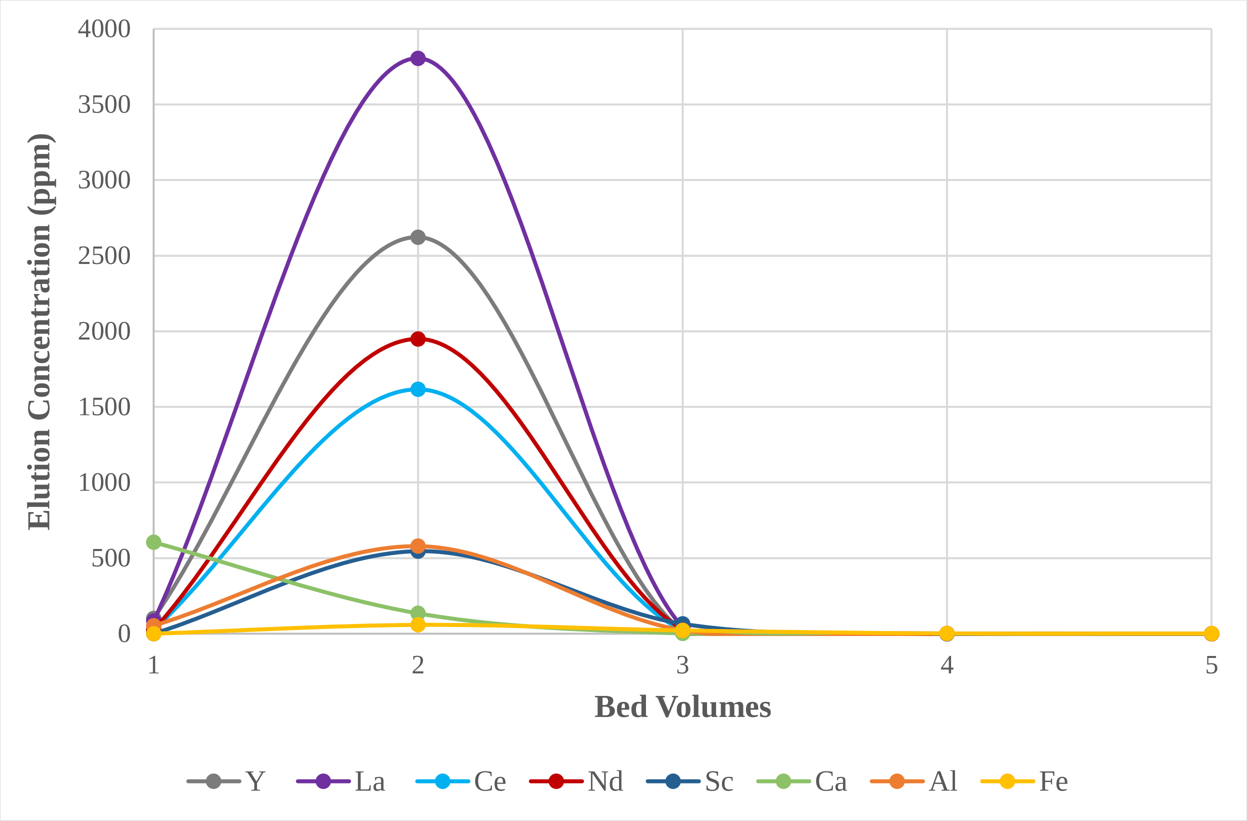

Figure 16. Stripping Curve for IDA Resin Using 5% HCl.

Figure 17 presents the loading curve for 119 mL of AMP resin in the H^+ form, with the solution fed at 5 BV/hr. This loading profile is much more favorable than the H^+-based resin, and the light rare earths do not begin to break through until the 40th bed volume. This result corresponds to an operating capacity of 0.50 mEq/mL, which is 21% of the reported total capacity. This capacity is lower than ideal; however, the breakthrough curve's shape is favorable and indicates that a loading time of eight to ten hours is possible.

Figure 18 presents the stripping curve for the AMP resin using 5% HCl. This curve is unfavorable for three reasons. First, calcium co-loading onto this resin is significant and is likely the cause for the low operating capacity. Second, the tailing of the curves is significant and there are significant REEs still coming off the resin even after 7 BV of acid. Finally, scandium does not elute under these conditions, which is a significant issue due to its high value; therefore, the literature was consulted to find a way to recover this metal. Fortunately, this issue is well-researched, and many authors have reported using a 200 g/L solution of sodium carbonate to recover scandium from AMP resins.

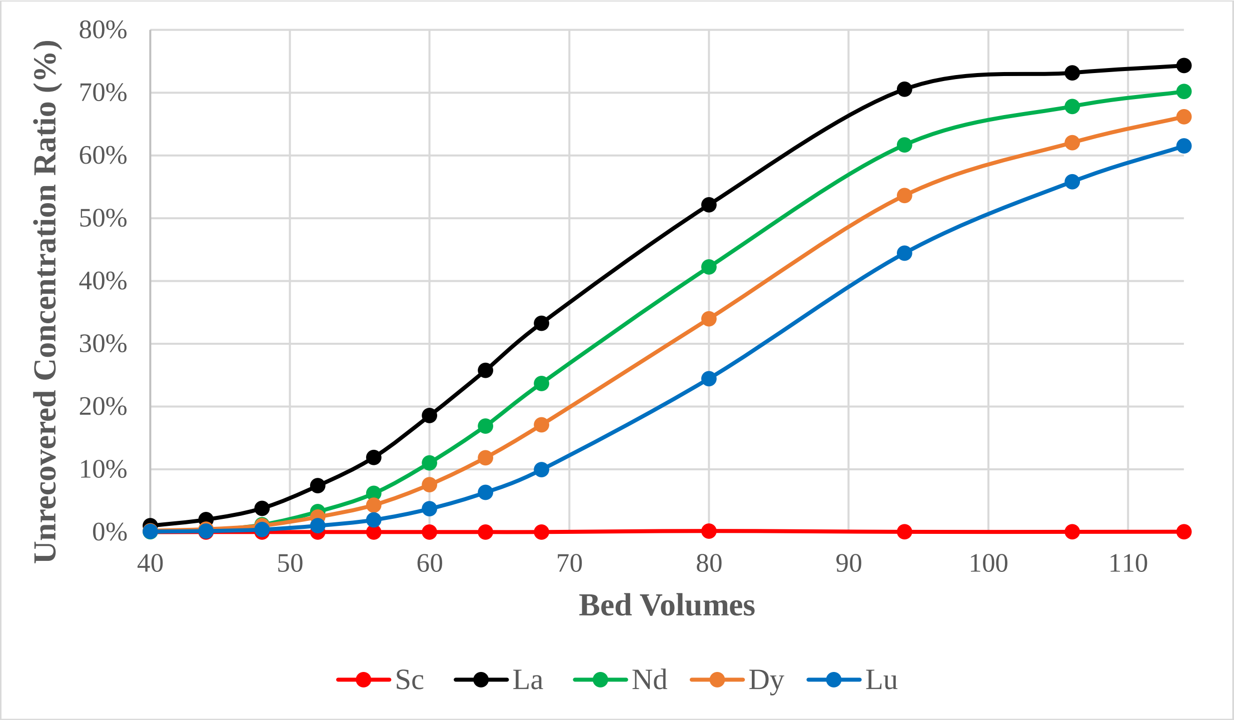

Figure 17. Loading Curve for AMP Resin in the H^+ Form.

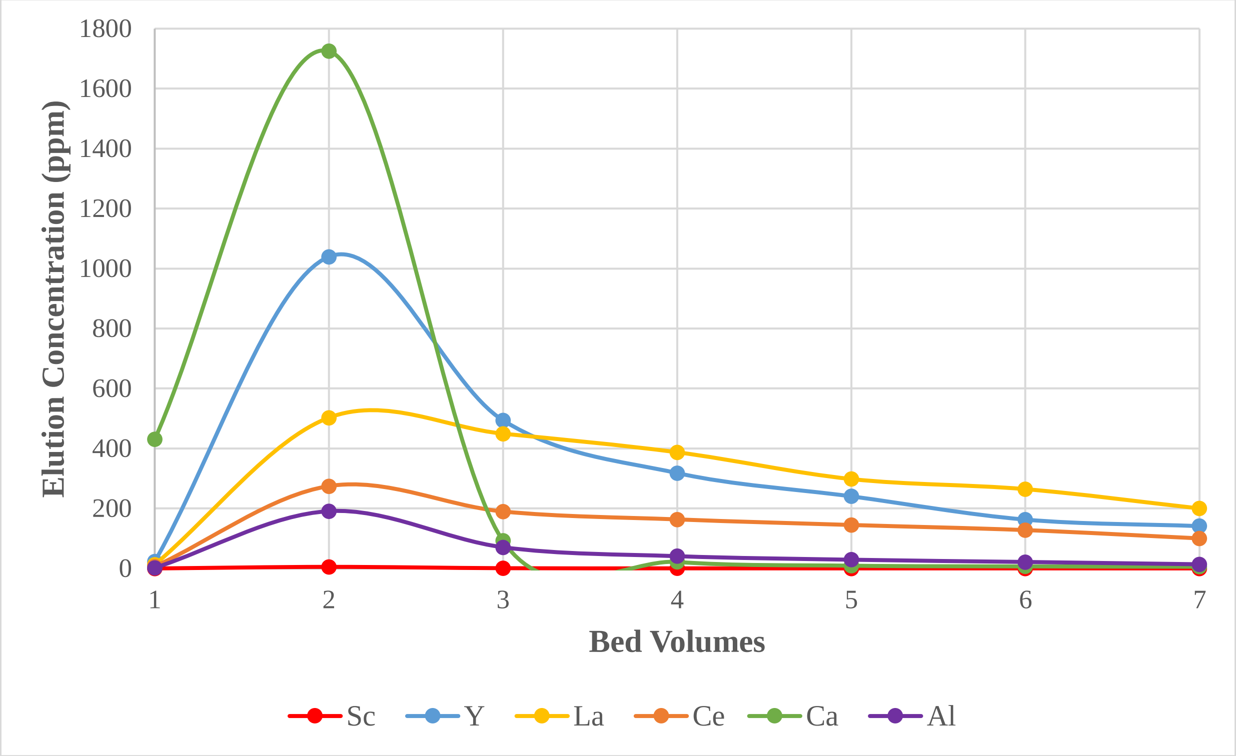

Figure 18. Stripping Curve for the AMP Resin Using 5% HCl.

Figure 19 presents the results from stripping a loaded AMP resin with 200 g/L Na_2CO_3 at 3 BV/hr. Scandium was recovered from the resin, but several challenges remain. First, calcium does not efficiently elute with this solution and would have to be recovered with acid in a second regeneration step to prevent its accumulation over time. Second, metals tailings remain an issue and 14 BVs of carbonate solution was not enough to completely regenerate the resin. This method would require upwards of 20 BV of a fairly concentrated base solution neutralized with acid to recover the metals, which would incur large reagent costs for this step.

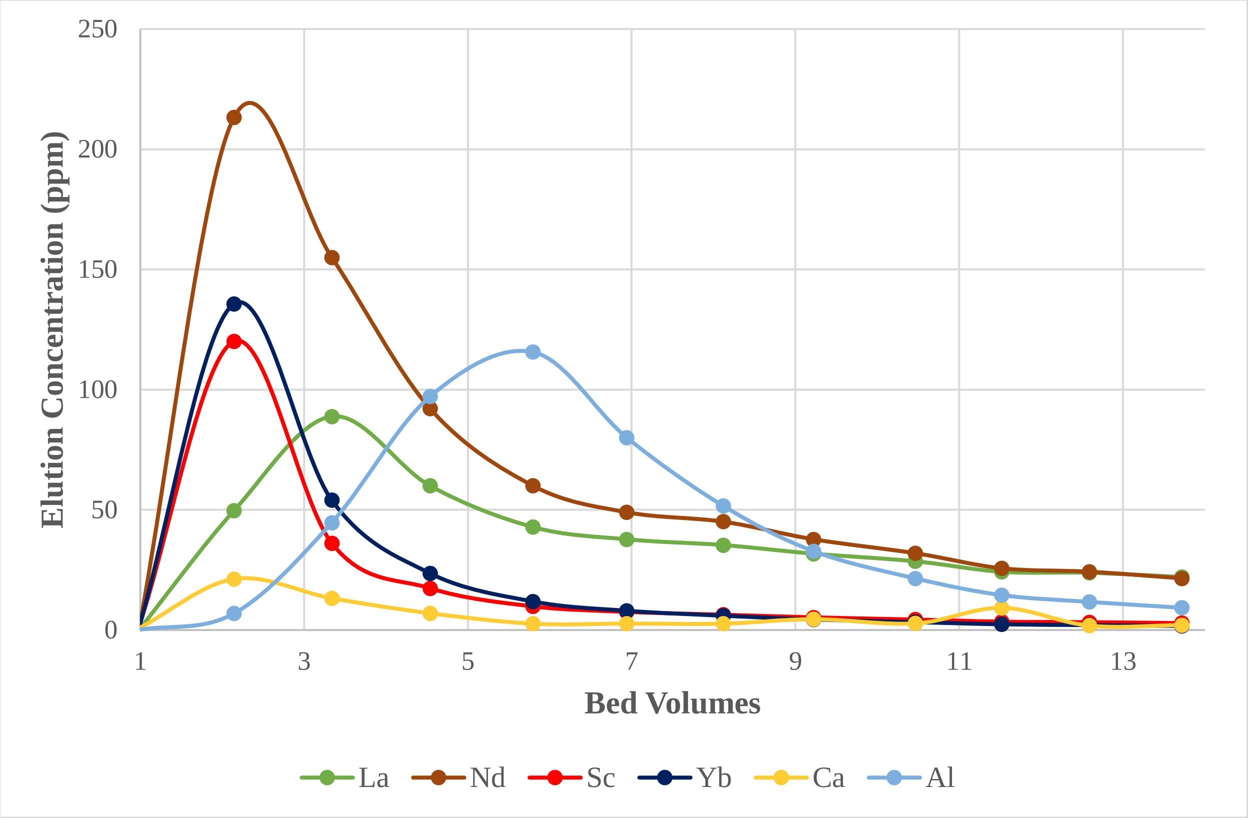

Figure 19. Stripping Curve for AMP Resin Using 200 g/l Sodium Carbonate.

The unfavorable regeneration of the AMP resins caused a re-evaluation of the IDA resin, now operating it in its Na^+ form, which would cause the effluent's pH to increase, and hopefully H^+ would no longer compete for the active sites with the REEs. Figure 20 presents the results from this experiment using 94 mL of resin, and it can be seen that this loading profile is significantly more favorable than the same resin in H^+ form.

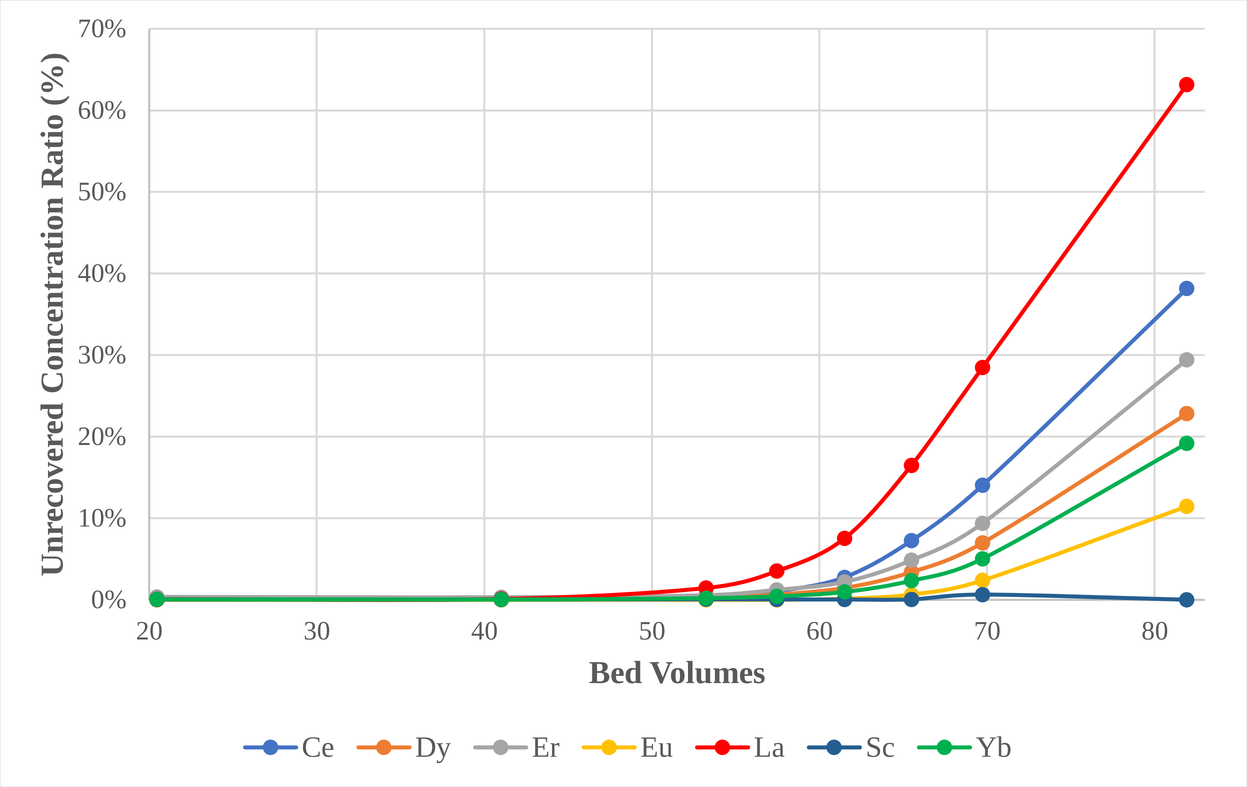

Figure 20. Loading Curve for IDA Resin in the Na^+ Form.

Interestingly, the breakthrough order of the REEs is opposite of that in Figure 15, and the lighter rare earths break through first. Based on these results, an operating capacity of 0.59 mEq/mL (corresponding to 44% of the total capacity) is attainable, which is significantly more favorable than IDA in the H^+ form and even outperforms the AMP resin. These results, combined with the favorable stripping behavior seen in Figure 16, make an IDA resin in the Na^+ form the most beneficial for achieving the rare earth-calcium separation.

6 PILOT DESIGN AND PLANNING

6.1 Pilot Equipment Design

The pilot facility was initially designed around the successful bench-testing apparatus from DE-FE0027006, a system capable of a semi-continuous 25 kg/hr of coal throughput for leaching. Specific process designs were based off of this system, with equipment aimed at attempting to replicate the processing methods at a larger scale, altering any based off of equipment requirements.

6.1.1 Process Area Definition

Within the pilot facility, control and placement of specific equipment was separated by process area based on the process flowsheet. These were divided into:

0. Coal Handling, Chemical Storage/Delivery, and Ancillary Equipment
1. Physical Separation (Spirals) and Associated Equipment
2. Leaching of Coals and Generation of Cleaned PLS
3. Preliminary Precipitation and Iron Removal
4. REE Precipitation 1
5. REE Precipitation 2
6. Wastewater Treatment

In these various process areas, these could be located on separate floors or significantly removed (>20 ft) from surrounding process areas. This allowed for PPE requirements to be defined on a per-process-area basis, rather than for the entire plant, as well as simplified operations planning and controls development. For instance, the process areas interacting with coal could be 0-2, whereas the only process areas possibly interacting with any form of oxalic acid (solid or liquid) are 4-6, limiting potential contamination and PPE needs throughout the facility. Additionally, this allows for a more rapid spill response clean-up in the event of spills, as spill response was dictated based on the possible liquids involved in a spill – all of which would be known on a process-area basis and could be rapidly treated.

6.1.2 Process Area Zero Design

Process Area 0 primarily addresses the water purification and ancillary equipment/chemical storage required for the plant. This includes mineral acid storage and metering to process areas; water purification to the specification to be identified in bench-scale testing (RO chosen); coal crushing, pneumatic transport, metering and storage in limited-size-hoppers.

Water Purification and Quality: Reverse Osmosis (RO)-quality water was determined as a viable water source (avoiding de-ionization) output of the bench-scale system water purity testing, and was modeled for the pilot facility. The performance of RO water filtration system decreases as the processing water temperature drops. Speculated efficiency drop for the system is approximately, 2% per 1°F below 77°F water inlet temperature, and amounts to a slower than rated flow and higher reject percentage. Grand Forks water mimics the ground temperature seasonally. Thus, water utilized for REE extraction process will vary from 35°F (during winter) – 85°F (during summer) depending upon the season. Designs allowed for buffering of the fluctuations in thermal properties by blending of a hot water stream during colder months for optimal RO efficiency.

The water is discharged from the purification system into a storage tank to meter to various process conditions and act as a buffer for high-flow incidents. As will be discussed below, the primary fresh water inputs were designed to filter presses, which would ideally utilize high volume and pressure flow for short periods of time over long cycles (10 minutes for every 2-hour cycle planned), and a storage vessel to dampen this effect to the purification system is needed. This also allows for thermal stabilization to building temperatures, preventing potential thermal effects in the mineral processing. As noted in Section 8, the washing of the coal in the filter presses was largely unsuccessful at any practical rate without potential safety risks from edge leaks at elevated pressure, and so water was directly mixed with mineral acid to form the leaching solution, bypassing the filter presses.

Coal Crushing and Storage: As demonstrated in previous work (DE-FE0027006), the coal particle size does not have a substantial impact on the leachability or kinetics of REE extraction. For this reason, the coal was designed to be crushed to a fairly coarse topsize of 4 US mesh (approximately 1/8 inch), chosen to reduce costs and allow the application of the cleaned-coal byproduct as feed to cyclone fired boilers which require a larger particle size than conventional pulverized coal boilers. A hammer crusher is currently on-site for this application, and a combined shaker table to recycle the coarse +4 mesh fraction back to the crusher is being installed. The correct size fraction material will be pneumatically transported to the coal hoppers (one each for the different coals) where a mixed cyclone/filter bag will separate the fluidizing gas and the solids. Crushing and transport have been oversized above the scale of the process (up to 10 tons/hr coal feed) to reduce potential labor requirements on all shifts (crushing only conducted during day shifts, and only for set 3-4- hour period). Coal storage hoppers have been designed for up to 30-hour capacity for the coals, with rotary metering valves and pneumatic transport to the coals' respective locations after the storage hopper.

The choice of day hoppers for semi-continuous crushing was made due to the ability of lignite to self-ignite when in insulated piles with air leakage, with the assumption that storage for less than 48 hours preventing the coal from generating enough heat to sustain combustion. The storage hoppers will be sealed and pressure tested to ensure no leakages upon installation.

The flow rate of Rhame coal into the process is set by the operator via XC-020 and the ratio of Rhame to Freedom Mine coal to achieve a 300ppm target is set by XC-022. The ratio is trimmed by manually entering the concentration of REEs into EEA-020 from lab analysis or eventual online measurements. Acid delivery, storage, and distribution: Concentrated mineral acid is required for the process for leaching, and will be contained within a double-walled PVDF tank of approximately 1500 gallons (week-long capacity of 24/7). The tank is materially-rated for long-term acid storage, and contains a 3” feed port for acid delivery to the plant, vent, and discharge port for acid delivery to the process. A multi-channel peristaltic pump is used, potentially discharging to R-101 in Process Area 1, as well as R-201 and D-202 in Process Area 2. The multi-channel peristaltic pump permits specific, non-contacting accurate flow control of acid in 3 separate applications. Each channel of the pump will be set to separate control logic for the respective tanks for pH maintenance, automatically dispensing acid as needed to reduce pH of the vessel to the setpoint target.

Utilities: Not shown on P&IDs is the utilities required for the pilot plant, including cooling water for pumps (as needed), air compressors for pneumatic valves and filter press air blowdown, and electric wiring from panels.

The pilot plant facility uses coal from two different coal seams from two regions of North Dakota. The Beulah seam comes from the Freedom mine in Mercer County. The Rhame seam comes from Slope County and was mined as a test pit for this project. Both coal seams were delivered to the pilot plant facility in supersacks.

The overall coal handling process continued to develop throughout the project but the general process steps for Process Area 0 (Coal Handling) are as follows:

- Receipt of delivered coals
- Storage of raw coals
- Crushing and particle sizing
- Transport to storage or transport to hopper
- Transport to Process Area 1

The facility utilized for this project was a building that housed a coal-fired power plant for the city of Grand Forks. The facility was not fully available to this project at the beginning of the project. As the project progressed, UND was able to acquire the full building, and the team was able to make improvements to the coal handling processes throughout.

The original plan for the project was to crush the Rhame coal and place it in storage to protect it from the environment until it was processed. The building has large steel concrete lined hoppers (Figure 21Figure 17) that were originally planned to be used for storage. These hoppers needed to be cleaned out and resealed. During the process of cleaning and making repairs of the hoppers, the team identified the risk that the coal may self-combust in these large hoppers. The project team determined it was too much of a risk to store coal long-term in the concrete-lined hoppers.

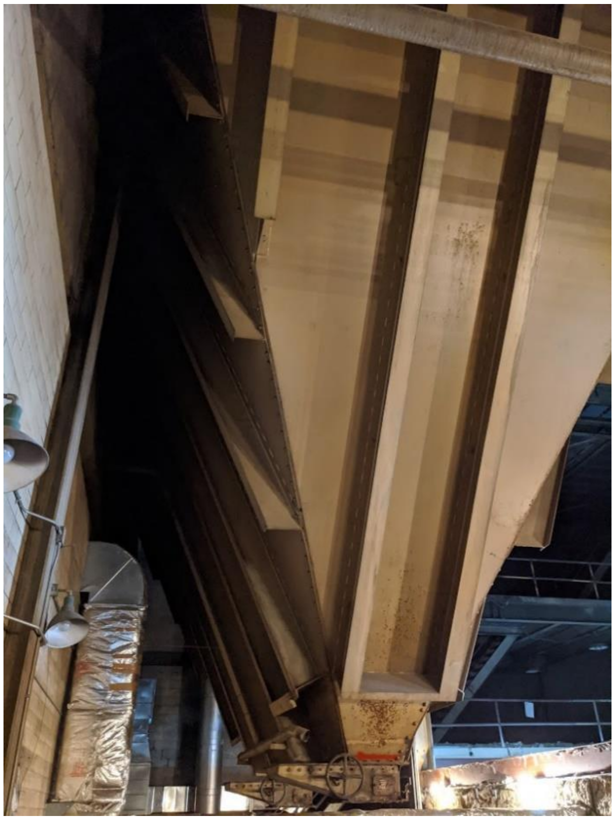

Figure 21. Large steel lined hoppers at the Pilot Plant facility.

The Rhame coal was delivered to the plant and initially stored indoors in super sacks. This caused the coal to dry out. To alleviate this issue, the Rhame coal was stored in 55-gallon drums to minimize lignite drying. The large storage hoppers for lignite allow for hot spots to form if lignite dries beyond a safe moisture content threshold.

At the beginning of the project, the coal handling process was limited to a 500 ft^2 room. A supersack unloading and crushing stand was fabricated to fit in this area. A large crusher was stacked on top of a particle sizer where the top crusher crushed coal to 1-inch minus and the particle sizer reduced 1 inch coal chunks to minus 0.22-inch particles. A stand was fabricated to hold a supersack (loaded onto the stand by forklift) with a conveyor underneath the stand to transport the coal to the crusher (Figure 22). With this setup, all of the Rhame coal was crushed and stored in 400 barrels. At this point in the project, the Freedom mine was not identified as high value so it was determined that it was acceptable to store supersacks of the Freedom Mine coal outside along the walls of the Pilot Plant facilities. The intention was to crush the Freedom Mine coal using this setup as needed for processing.

Figure 22. Stacked crushers and supersack unloading stand.

As the project progressed, the University of North Dakota acquired a new lease for the Pilot Plant facility which made the 15,000 ft^2 basement available. With the new available space the previously described crushing process was moved outside to support more efficient crusher loading and hopper feeding processes. Moving this process outside eliminated dust inside and limited the need for purchasing of additional equipment to transfer barrels or install an alternate transport process from the original coal handling room. A roll off dumpster was placed alongside the crushers as a staging area to dump the coal from the supersacks. This allowed for easier transporting of the coal from the bags to the crushers. A ramp was built leading up to the crushers so a skid steer could dump buckets of coal into the top of the crushers (Figure 23).

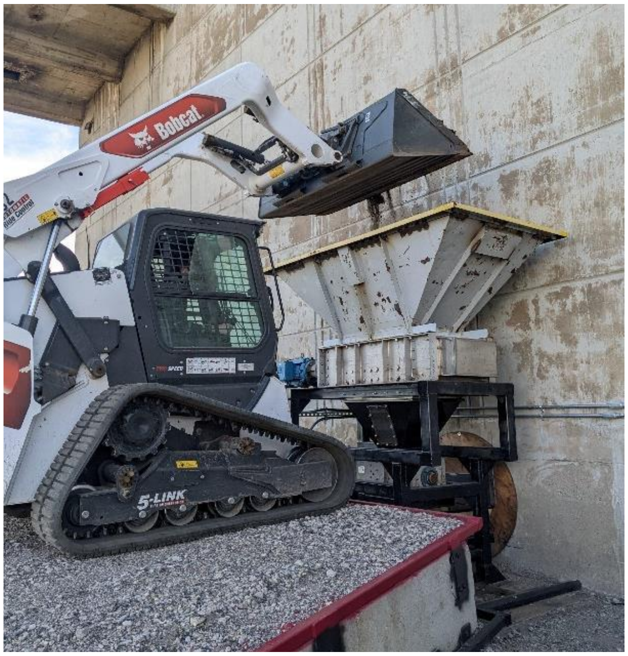

Figure 23. Skid steer dumping coal into crushers.

At the outlet of the second crusher a screw conveyor moved the coal through the wall into the top of a coal hopper located in the basement. Additionally, a mobile funnel was built to be able to dump the 400 Rhame barrels straight into the screw conveyor bypassing the crushers. Seven steel hoppers were acquired to hold the crushed coal for a campaign run of the Pilot Plant. The steel hoppers were only filled with coal when a processing run was about to begin to minimize any potential thermal runaway scenarios. Once a coal hopper was full, it was transported onto a large scale. An initial weight of the hopper was taken to document the amount of coal prior to being used for processing.

A rotary valve equipped with a scale was connected to the bottom of the hopper to meter the feed rate of coal. The coal was fed into a pneumatic transport line that transported the coal up three stories into a cyclone mounted on top of the process tanks. A mechanical vibrator was also added to the hopper to aid in feeding. Figure 24 shows the setup of the hopper on the scale, rotary valve, vibrator, and pneumatic transport line. A high-speed fan was selected to create a vacuum for pneumatic transport.

Downstream of the cyclone, a baghouse was used to collect fines that did not drop out in the cyclone. At the base of the cyclone, a double dump butterfly valve system was used to isolate the coal being transported to the process tank.

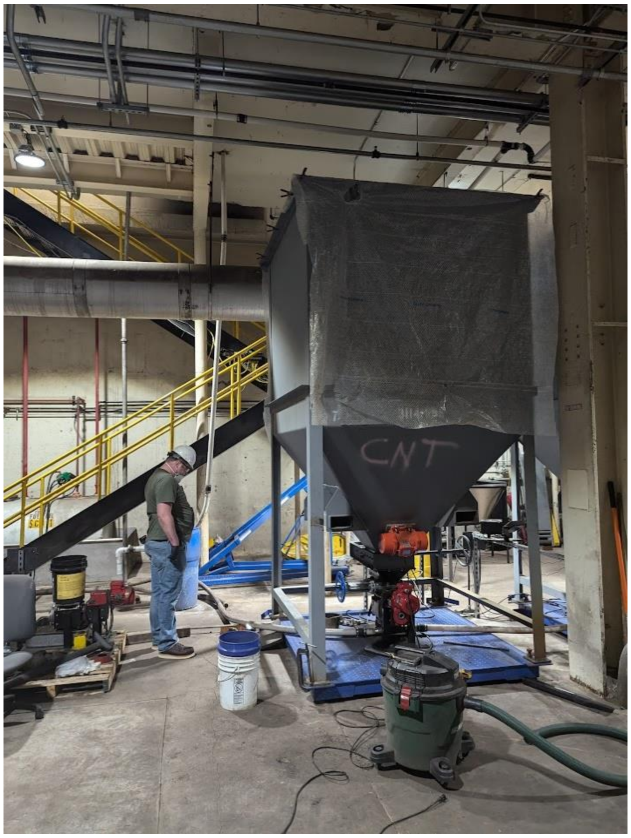

Figure 24. Operator checking coal feed out of a hopper.

Coal moisture is a critical part of this coal handling system. If the coal is too wet, the coal will have a difficult time being crushed and being fed out to the rotary valves due to the sticky nature of the wet coal. Since the Rhame was dry before it was stored in barrels, this was not a concern for this coal. The Freedom mine coal was stored outside so it would not be dry by the time of use. To help reduce moisture, the super sacks were split open in the roll off dumpsters and the coal was left to air dry a few days before being crushed.

The mineral acid was placed in a suitably-rated, double walled tank. This tank was placed in the corner of the pilot facility that is located next to a door for the chemical company to refill the tank, next to the leaching tanks that require acid, and out of the way from the forklift access to avoid damage. The tank was also placed inside a spill containment barrier. Pumps were installed on top of the tank to pull mineral acid out of the top instead of the bottom to prevent uncontainable leaks in the event of pump and/or plumbing failure – instead flowing back into the tank wherever possible.

The sodium carbonate and oxalic acid are both granular particles that were pneumatically transported into the processing tanks. Pneumatic transport was selected over screw conveying because pneumatic conveying allowed for a smaller footprint then screw conveying. There were two sodium carbonate hoppers and two oxalic acid hoppers that were installed on the plant floor next to process area 3, 4 and 5 tanks. One of the sodium carbonate hoppers for the process area 3 tank was designed to hold three times the amount of sodium carbonate than the other two sodium carbonate hoppers. The liquid coming into process area 3 tank was at a low pH and needed large amounts of sodium carbonate to bring it up to the correct pH.

The two oxalic acid hoppers were originally designed the same way as sodium carbonate hoppers to double dump out of the hopper and fall into the pneumatic transport line; however, during equipment commissioning, it was found that the oxalic acid would agglomerate together and plug the bottom of the hopper. To address this issue, a screw conveyor was installed with a mixer inside the screw conveyor's hopper. This allowed constant breakup of the oxalic acid, and it could be conveyed into the pneumatic transport line.

6.1.3 Process Area One Design

Preliminary testing from previous work (DE-FE0027006[1]) demonstrated that the REEs present in many lignites are found predominantly in organic-based fractions. With this in mind, and with the enrichment towards the tops of seams identified[2], the REE-rich lignites commonly have elevated ash as to more typical thermal-use lignites, allowing for a coal cleaning process to upcycle both the REEs and the coal in relative purity. Spiralization was chosen for the pilot given previous data from the effort (DE-FE0027006), with a system designed to spiral the lignite into two eventual fractions, a coal and REE-rich low-density fraction, and a mineral/clay-rich, REE-lean high-density fraction.

An 8-turn, triple outlet mineral spiral was acquired to serve as the separation media, with four supporting tanks (R-101 – slurrying of coal/water, D-201 – surge of coal concentrate, D-102 – surge of middlings, D-103 – surge of high-density tailings) were designed, with air-operated double-diaphragm (AODD) pumps used for transfer of slurry around the process system. This included lines to transfer the middlings to the tailings tank (for rejection if need be), and a pump to transport the tailings to a battery of two filter presses (F-101A and F-101B) for final filtration and removal.

6.1.4 Process Area Two Design

PA-2 focuses primarily on the leaching process, where rare earth elements bound to the organic matrix of the coal are leached at acidic conditions. The leaching tank (R-201, as depicted in Figure 25) is initially filled with a specific volume of reverse osmose (RO) water (Figure 25.Stream 1) and then dosed with acid (Figure 25.Stream 2) in a controlled manner to adjust the solvent's pH to

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[1] Mann, M; Theaker, N; Rew, B; Benson, S. A; Benson, A; Palo, D; Haugen C; Laudal, D. Investigation of Rare Earth Element Extraction from North Dakota Coal-Related Feedstocks. Final Technical Report submitted to the U.S. Department of Energy under contract DE-FE0027006.

[2] Moxness, L. D; Murphy, E. C; Kruger, N. W. Critical Minerals in the Fox Hills (Cretaceous), Hell Creek (Cretaceous) and Ludlow (Paleocene) Formations in North Dakota. Report of Investigation 130 submitted by the North Dakota Geologic Survey, 2022.

the required value. The tank is designed to hold 640 gallons of leaching volume. Peristaltic pumps with a flow capacity of 0.5 Liters/min are used to dose acid from the double-walled acid tank with a full volume capacity of 600 gallons. he peristaltic pumps are equipped with reversible flow functionality to allow draining of lines back into the acid tank if needed.

Coal is loaded from PA-0 into the leaching tank (Figure 25.Stream 3) using pneumatic conveying. The cyclone is connected to an induced draft blower (design specification: 160 cu.ft/min and -165 inH_2O). After the coal is loaded into the leaching tank, it is mixed with the solvent solution under monitored conditions. The separation of leached coal from the leachate solution is performed by filtering the slurry (Figure 25.Stream 4) using a 375 cu.ft filter press. 2-inch pipelines and an air-operated diaphragm pump (AODD) are installed to achieve the design pressure and flowrate required to fill the filter press.

The filtered coal (Figure 25.Stream 5), a by-product of the process, is collected in bins. The filtered leachate (Figure 25.Stream 6) is pumped to the temporary storage tank D201 (with 590 gallons of storage capacity). The leachate stored in D201 is then transferred to the further processing area PA-3 (Figure 25.Stream 7).

Before the coal is unloaded from the filter press, it undergoes an in-situ wash using acidic water. In PA-2, D203 serves as the storage tank for pumped acidic wash water through the filter press, while D202 serves as the storage tank for pumped RO wash water. This constitutes a loop cycle, where fresh RO water (Figure 25.Streams 8 and 9) is pumped to D202 and dosed with concentrated mineral acid under mixing. This solution is then used for the in-situ wash of coal inside the filter press (Figure 25.Stream 10). The resulting rich wash water containing REEs (Figure 25.Stream 11) is collected in D203 to be reused as makeup water for the next leaching batch (Figure 25.Stream 12). In this design, RO water is not fed directly to the leaching tank. Instead, it is first used to neutralize the coal in the filter press and is then pumped to D202 to serve as wash water for REE recovery from coal.

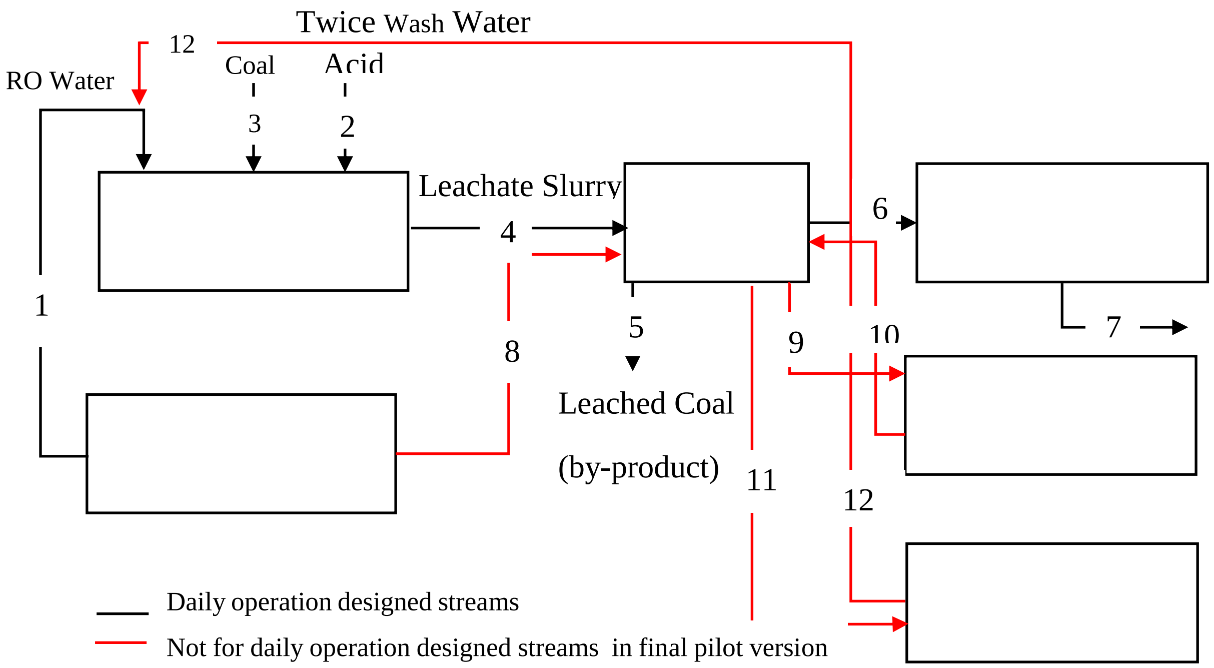

Figure 25. Design scheme of PA-2

6.1.5 Process Area 3 Design

Iron has previously been identified as an impurity of concern in prior work, primarily for the complexation of oxalic acid (and consequential additional oxalic acid consumption). Removal of Fe through increasing of the pH and precipitation of sodium jarosite is accomplished through the addition of solid sodium carbonate to a reaction tank holding the leachate to a pH of between 3 and 3.5. When held for over 2 hours, this was found to precipitate significant (>80%) of the Fe, while having minimal (<5%) REE co-precipitation.

Addition of a carbonate-based base, particularly to acidic solutions with potential organic constituents, often produces semi-stable foam, leading to sensor, flow, and mixing challenges within processes. Optional foam control mechanisms include the addition of a hot-air blowdown stream for raising the temperature of the foam and mechanical popping of bubbles, as well as addition of a de-foaming agent into the solution. The hot air blowdown is targeted as the ideal option, primarily as the chemical impacts of the de-foaming agent with REE precipitation are unknown, as well as the likely availability of warm air within any coal utilization process that may be co-located with the REE plant in future, commercial scenarios.

6.1.6 Process Area 4 and 5 Design

Process Areas 4 & 5 cover the entire precipitation of the REEs from solution using oxalic acid. These processes were designed to feed oxalic acid slowly and carefully into mixed tanks – using a total suspended solids (TSS) sensor to measure dissolution of chemicals and precipitation progress). The processes are aimed at taking advantage of the lower solubility of the REEs and some CM oxalates over Ca – the primary rival for precipitation. However, due to the extreme difference in concentration within solutions of these, and the presence of other species capable of complexing the oxalic acid but not forming precipitates (preventing stoichiometric-like additions from occurring), an extreme level of detail would be required to measure the small quantities of REE/CM precipitation prior to Ca precipitation. This would be followed by a greater precipitation, allowing for some Ca precipitation to occur to ensure quantitative REE/CM precipitation and recovery from solution. This progress using the cleaned solution from PA-3 (Figure 26.Stream.1) into a mixed, 900-gallon tank where oxalic acid (Figure 26.Stream.2) and sodium carbonate (Figure 26.Stream.3) are added gradually. The oxalic acid feed controls were designed for as little as 1's of grams per minute addition, to prevent potential overshoot with the oxalic acid concentration. The solution in R-401 and R-501 are continuously mixed and passed through a TSS sensor on a flow loop, allowing for real-time, accurate progression of precipitation. The slurry of the first concentrate and solution (Figure 26.Stream.4) is filtered through a filter press and/or cartridge (depending on size and quantity of material), where the concentrate (Figure 26.Stream.5) is removed, washed, and dried for further analysis. The remaining liquid then repeats the same process in process area 4 at a different oxalic acid loading (higher) to complete precipitation of all REEs/CMs (Figure 26.Streams.6-11). Finally, the barren solution containing no REE/CM is sent to PA-6 for wastewater treatment and disposal.

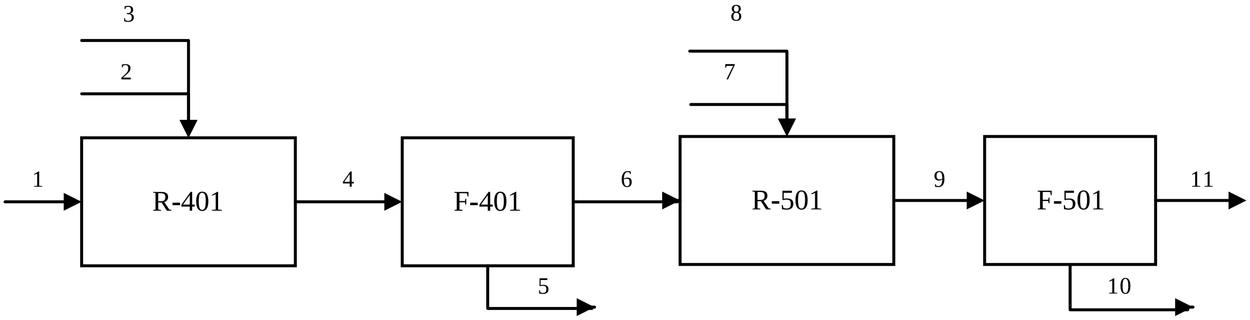

Figure 26. Process area 4 and 5 flowsheet.

6.1.7 Process Area 6 Design

Process Area-6 is designed to process ~1,000 gallons of wastewater per 8-hour shift, incorporating stages of wastewater neutralization, filtration, and ion exchange. A regeneration step is also integrated after 4,500 gallons of processed wastewater. Initially, the design included a second ion-exchange column for continuous processing during regeneration. However, the generated wastewater from scheduled operations does not require continuous processing, so the final design incorporates only a single ion-exchange column. This process area is equipped with minimal instrumentation and control, as it was developed during the course of plant operations.

The collected wastewater, stored in a 10,000-gallon tank, is introduced into the neutralization tank (250-gallon capacity) (Figure 27.Stream 1), where pH is adjusted manually with sodium carbonate

(Na_2CO_3) (Figure 27.Stream 2). The neutralization step optimizes the wastewater pH, which is crucial for the effective capture of copper, zinc, and nickel by the ion exchange column.

The wastewater slurry (Figure 27.Stream 3) is then pumped using an AODD pump to a 50 cu.ft filter press, where solids are captured. Iron sludge (Figure 27.Stream 4), a by-product, is removed for disposal, while the filtered wastewater (Figure 27.Stream 5) is pumped with a rotary gear pump at a controlled flow rate to the ion-exchange column. The treated wastewater (Figure 27.Stream 6) is suitable for discharge or reuse in the column washing cycle.

Regeneration of the ion-exchange column is triggered after 15 hours of processing time and is achieved using acid supplied from the acid tank (Figure 27.Streams 7-9). Acid make-up (Figure 27.Stream 9) replenishes the acid tank, while the spent acid, after two regeneration cycles, is purged as waste (Figure 27.Stream 10). RO water (Figure 27.Stream 11) is used for column washing and neutralization.

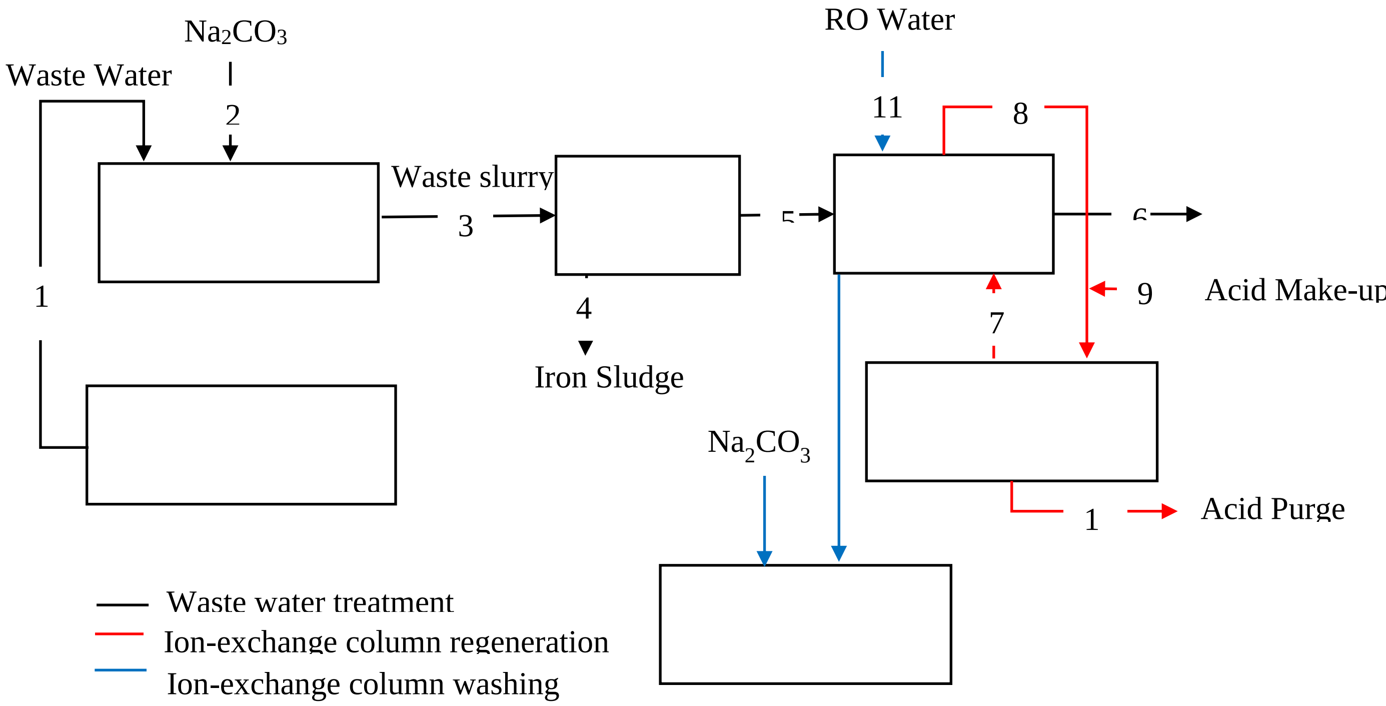

Figure 27. Designed flowsheet of PA-6

6.2 Pilot Controls and Safety

6.2.1 Pilot Control Philosophy

The pilot facility was designed around being able to test the UND REE extractive process from lignite developed in prior efforts in a manner most suitable for requisite data collection for commercial demonstration projects. With this in mind, a number of decisions were made early in the design phase to dictate equipment choice, control programming, and overall test planning for gathering this data. The focuses of the tests for the pilot were around:

1. Evaluating extraction and washing performance on relevant lignites – focusing on the exact washing and dewatering efficiencies and recoveries

2. Evaluate required filtration needs to prevent coal from moving downstream for capture

3. Evaluate UND's method of REE concentrate production, using state-of-the-art sensor packages to evaluate in-situ precipitation performance

4. Determine operability concerns with the as-designed process and identify areas of potential improvement

Each of these focus areas are discussed in further detail below.

1. The design of the coal washing systems were developed based upon prior bench-scale testing, and plans were to utilize the filter presses themselves as the washing sources, rather than discharging from a filter to re-slurry, before filtering again. At a bench-scale, testing had shown the feasibility of this plan with elevated water throughput when slurry pumping pressures were given a suitable deviation from maximum safe containment pressure. This washing cycle has shown promise in recovering additional REEs, recycling acid, and reducing the concentration of a number of deleterious species. With these design choices taken, equipment was focused on large, singular presses to be able to have a long discharge time to allow for washing cycles to complete.

2. As the filtration of the large amount of coal (largest potential solid stream by multiple orders of magnitude) was deemed to be a high potential cost and risk for commercial development, the pilot was designed to closely mimic the planned particle size (through specific crusher choice) and filters (primarily filter press, with bag filter back-ups) to be able to evaluate at which filter cloth size particulate was captured. This aimed at a ~1/8" size particle – forcing the sizing of a significant number of downstream equipment (to be able to handle solids in the 1/8" size range) and involved filters starting at ~50 µm and ending at ~2 µm to evaluate total capture.

3. The concentrate production from UND's process (directly from the leach solution with no solvent extraction) allowed for significant purity enhancement of REE's over other diluents (primarily Ca) mainly due to the solubility differences of the REEs and Ca. However, this precipitation required extreme control of oxalic acid dosing and evaluation of reaction progress – requiring some way of evaluating the precipitation in real-time. Top of the line TSS sensors were chosen for this task – with accuracy down to individual ppm (mg solid per L). These sensors dictated the eventual design of the flow and mixing systems, and forced a number of parameters around them for this reason (flow type and rate, rate of exchange, mixing speed, etc.)

4. Operability challenges discovered a pilot scale could have a reasonable chance to scale into a commercial scale, outside of any strange equipment configurations at the specific pilot scale. As such, identifying and correcting operability of the plant was a key priority, and did involve

compromising other designs in the end after commissioning to be able to operate the plant in a more reactive method, allowing for higher REE purities of products over time.

6.2.2 Controls System Design

To increase versatility and utilize the skillset of research staff at UND, the control system utilized was LabVIEW 2018. LabVIEW was used as the sole process control system & interface for the entirety of the Rare Earth Element Pilot Plant and incorporated hardware manufactured by National Instruments. LabVIEW, short for Laboratory Virtual Instrument Engineering Workbench, is a programming environment in which you create programs using a graphical notation (connecting functional nodes via wires through which the data flows). The resulting program is modular, visual based, and user-friendly. UND has the in-house capability of programming and making all modifications required to commission and operate the pilot scale facility, which was instrumental for research success.

National Instruments hardware is built to be plug and play with LabVIEW. By using LabVIEW as the host software and using national instruments hardware, we were able to quickly implement, test, and calibrate new sensors required for operation of the pilot research facility. The control system incorporates hundreds of sensors and electrical equipment of differing signal types and ranges. The pilot consists of over 400 pieces of instrumentation and controls equipment, the individual breakdown is shown below in Table 9Table 9. Controls and sensors overview.. The others column consists of miscellaneous sensors, indicators, and controls required to operate the facility. Every value is data-logged at an operator defined interval, typically 5 seconds.

Table 9. Controls and sensors overview.

Pneumatic Valves	Control Valves	VFD's	pH	Flow	Pressure	TSS	Level	Others
232	3	35	7	23	30	10	17	47

UND was responsible for wiring of the entire facility. All controls and instrumentation wired by UND researchers were 24VDC and under. A professional electrician who was dedicated to the building performed all high voltage wiring required for project completion. Figure 28 shows the control cabinet for the project, where all instrumentation and controls are fed and controlled from. The control cabinet includes several emergency indicators, as well as emergency shutoff switches that would enable the operator to quickly and safely shut down the plant to a safe state.

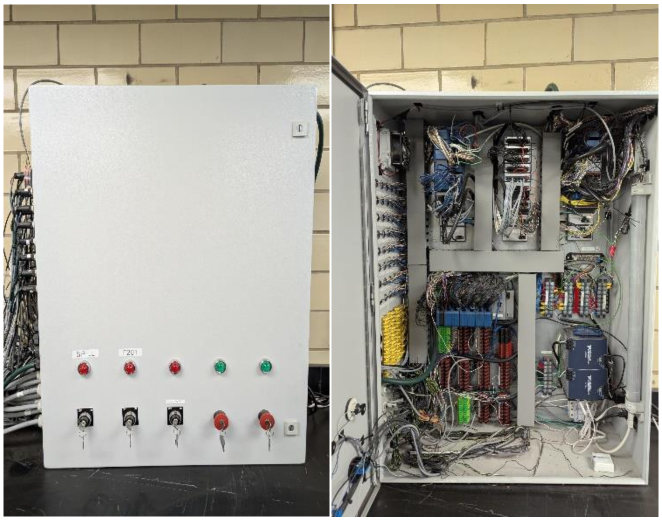

Figure 28. Control hardware and wiring.

Figure 29 represents the operator station where the entire project is monitored and controlled from. It consists of two screens of graphing, containing 17 graphs, datalogging, filter press controls, and the main control panel hosting the entire plant.

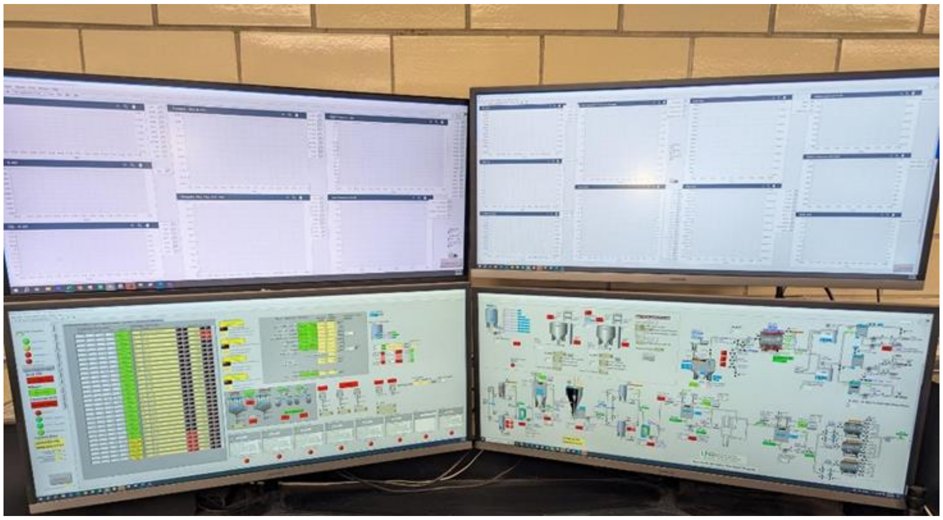

Figure 29. Operator control interface.

Safety measures were programmatically implemented before and throughout operation. Every safety condition in the system was tested under safe conditions with just water before transitioning to chemicals and coal. Every piece of instrumentation feedback was programmed with alarms to indicate low level and high-level failures. Additional preventative measures were programmed in eliminate possibilities of overflowing tanks and running pumps dry. Figure 30 shows the first of the control panels, with alarms and overrides for critical equipment (pumps, mixers, chemicals). The panel also incorporates error handling for the National Instruments hardware, which would indicate a problem with a damaged input/output variable.

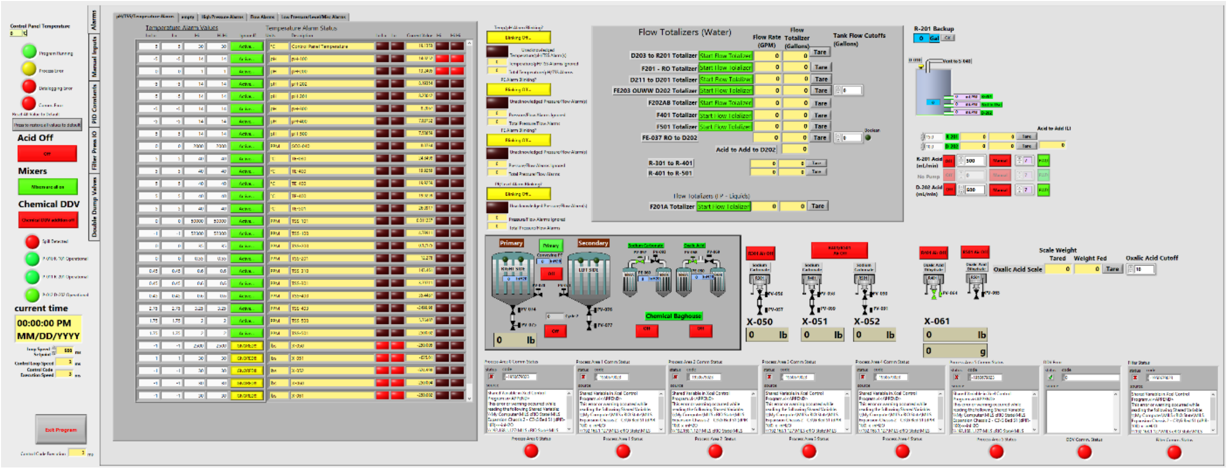

Figure 30. Main panel screen 1.

The second screen is the main process control panel, shown in Figure 31, is a 2D overview of the entire research facility. Every major control and indicator are hosted on this screen and provides means to quick feedback and adjustment of the process.

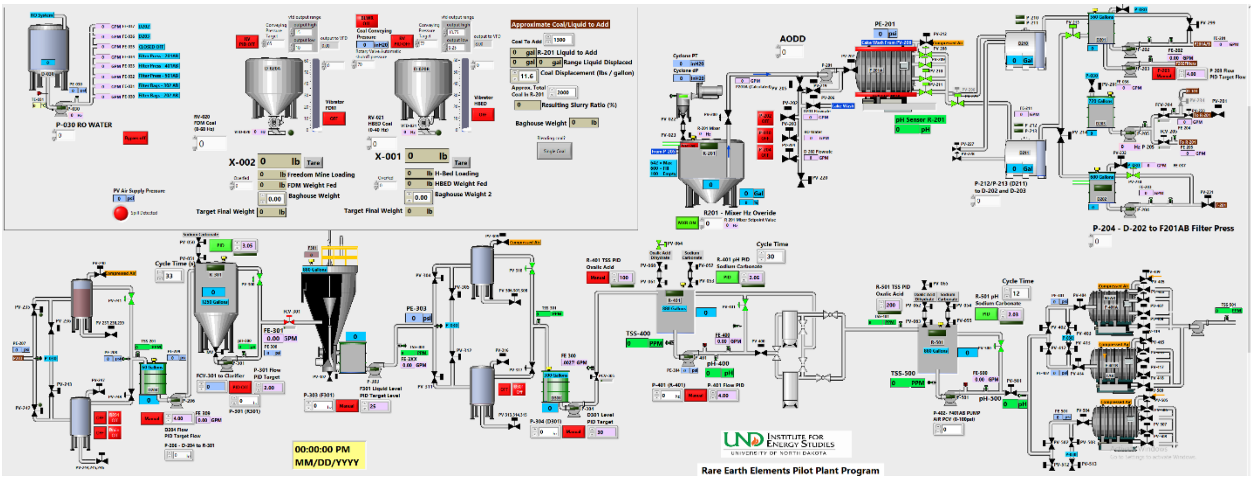

Figure 31. Main panel screen 2.

The pilot plant had total industrial sized filter presses which required programming controls as well. The vendor offered these services at a price point of $30,000 each. UND was able to successfully do the programming (Figure 32) of these major pieces of equipment internally, saving the project a significant amount of money – critical given the impacts of COVID 19 on budgets.

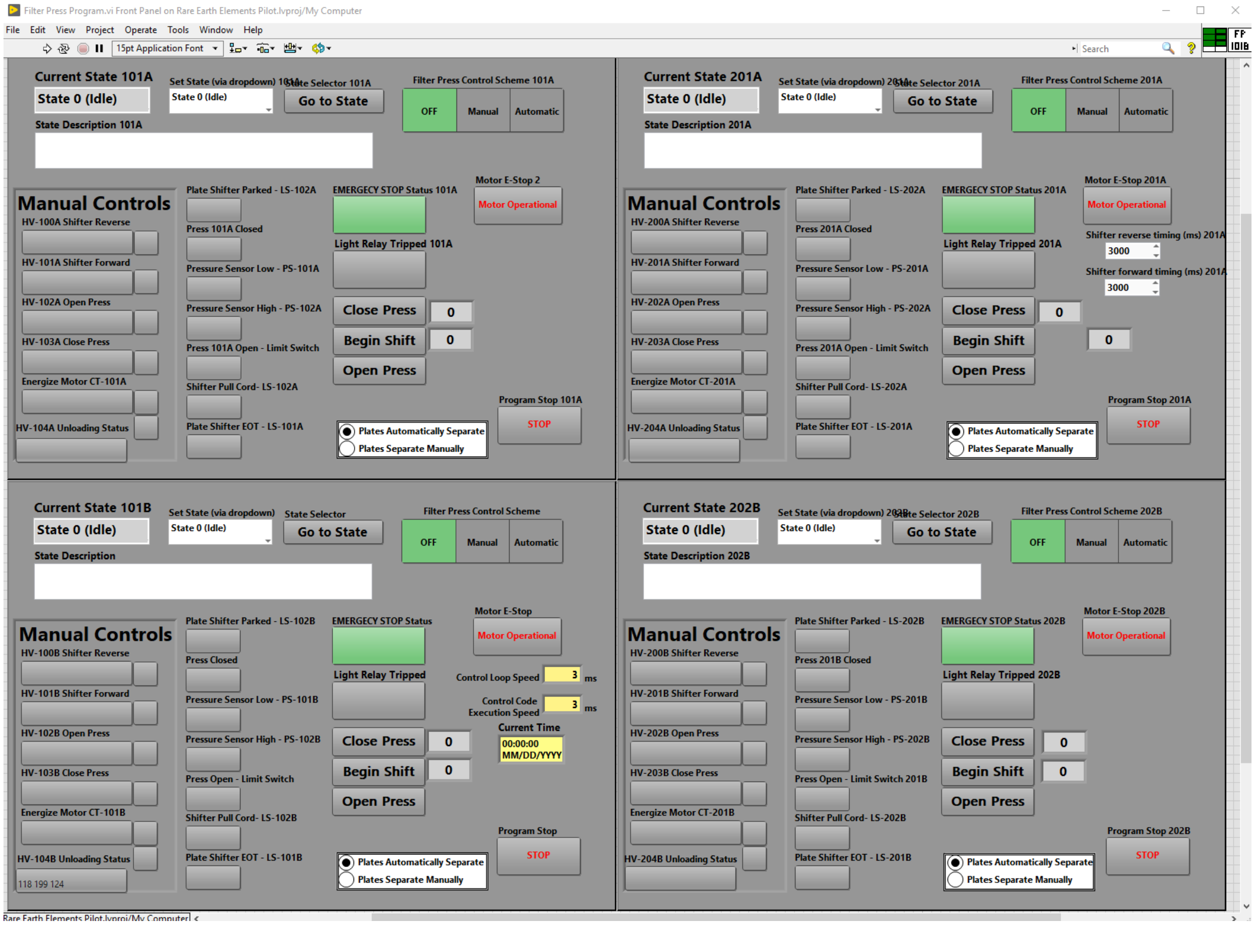

Figure 32. Filter press control panel

Pumps in the process were controlled via PID's, either based on level or flow (and in some cases cascaded - both). UND tuned all PID's based on our ideal process conditions for the smoothest control feasible. Mixer speeds were controlled automatically based on level, experimentally determining which speed gives the best flow output for certain solids loading. Solids chemical loading was accomplished with small double dump valves and adjusting the timing between opening and discharging. Limits were put into place to not exceed chemical dosing and pH conditions.

6.2.3 Hazards Analysis and Safety Review

6.2.3.1 Hazardous Substances

A number of hazardous substances to health and safety of the personnel working within the plant exist. These primarily include the following, each of which is discussed in greater detail with mitigation strategies to reduce risk of injury:

- Concentrated mineral Acid (stored and delivered by vendor)
- Dilute mineral Acid (process liquids)
- Concentrated Alkali Base (liquid NaOH solution, solids for pH adjustment)

- Oxalic Acid (target organ toxicity)

Concentrated Mineral Acid: The prime source potential injury/environmental release of this acid has been determined to be transfer to/from the mineral acid holding tank, as well as potential release from the tank. The tank has been specified to hold up to 1,550 gallons' capacity, and will remain within a secondary tank of 110% excess capacity (1,700 gallons minimum) to reduce likelihood of catastrophic spillage. Level of the primary holding tank will be electronically monitored and recorded to the plant control room, with alarms to sound if substantial level drops beyond expected values is identified. PPE including full chemical suits, full-face respiration, and specific concentrated-acid resistant gloves will be mandated for all events requiring acid exposure.

Additionally, transfer of the acid will occur through vendor delivery and pumping directly into the tank, with the fittings feeding the tank serviced each week in between deliveries. The pumping process is anticipated to be rapid, and level measurement and control is critical to ensure proper dosage. Areas surrounding all acid transfer equipment will be labeled and spill controls in place to ensure acid spills are diverted into an area for neutralization, rather than directed to a drain. Outputs from the acid tank will utilize PTFE contact to their designated areas for excellent acid resistance, and where feasible secondary channels will be developed under these lines. No concentrated acid delivery line to the process will be constructed above ground level, except for the final vertical segment for delivery into the tank.

Dilute Mineral Acid: The primary process liquid, and most concentrated within the leachate at <2% concentrated, the highest points of potential release/spills are leakage from the process, sample ports, or filter presses in the event of poor filtration and washing. Due to the volume of liquid involved, spill prevention and containment measures will be primarily focused on routine maintenance of equipment to ensure no leak points, use of flanges rather than threaded pipe for compression fittings, and flow channels in the ground to divert potential spills into a singular drain for the acidic liquid, after which they may be collected in a holding pit and neutralized. PPE required for staff handling these liquids at the small scale (samples or small leaks) include standard laboratory safety glasses, nitrile gloves, and nitrile boot covers. Large releases will be treated similarly as concentrated mineral acid PPE requirements.

Concentrated Alkali Base: Two bases will exist within the plant, a concentrated liquid NaOH stream for neutralization of materials for discharge, and a solid Na_2CO_3 section including 55 lb bags and hoppers contained on tanks. Solid sodium carbonate will be manually removed from 55 lb bags and poured into the operating hoppers as needed, and will be hoisted into position by the crane. Dust prevention, control, and safety measures will be of top priority in these areas, and will include local exhaust where possible and the requirement of full-face respirator equipment.

Oxalic Acid: Oxalic acid is used as the REE precipitation mechanism, and is a toxic chemical, specifically for the renal system. Oxalic acid will be delivered by 55 lb bags, and managed in a similar fashion as the sodium carbonate described above. Local ventilation and full-face respirators will be required for handling of the chemical, as well as long gloves. Oxalic acid itself is relatively non-corrosive, and full-chemical suits are not anticipated to be needed for the chemical. Spills will be managed through prompt cleaning of the area and spray with a lime solution (to neutralize the acid).

6.2.3.2 Industrial Safety

A number of potential industrial safety protocols will apply to the pilot plant, due to the size of the equipment, the necessity of heavy machinery for lifting (crane and forklift), as well as the potential fire hazards associated with the process (primarily fine coal dust). For these reasons, plant personnel and visitors will be required to wear steel-toed shoes, hard hats, and shatter-resistant safety glasses at all times when in the plant, and whenever outside if equipment is operating nearby. Fire suppression of pressurized water sprinklers is installed within the building, and is expected to be adequate for fire-fighting for the ground floor. Fires are not anticipated above or below these floors, due to the lack of combustible materials and ignition sources within these areas. Dust control measures will be specifically heavy in and around the coal crusher, the highest production source of fugitive coal dust, and weekly cleaning of the plant and surfaces to remove coal dust and prevent build-up will be conducted.

Processed coal and waste solids produced within the process will be disposed of weekly to prevent accumulation of materials, and may be removed more frequently as needed during long-term steady state testing.

6.2.4 Permitting

The pilot facility utilized existing UND industrial safety and air permits (including an air permit previously issued to the coal-fired steam plant on campus), but required the attainment of a wastewater discharge permit to the City of Grand Forks publicly operated treatment works (POTW). This was attained with the following criterion pollutants of interest to be measured during weekly discharge cycles of PA6 (Table 10).

Table 10. Table of criterion pollutants for UND’s pilot-scale REE extraction facility.

Pollutant/State	Disposal Limit	Pollutant/State	Disposal Limit
pH	5.5-11.0	Pb	0.69 mg/L
TSS	250 mg/L	Hg	0.014 mg/L
As	0.46 mg/L	Mo	0.5 mg/L
Cd	0.11 mg/L	Ni	3.57 mg/L
Cr	0.82 mg/L	Se	0.23 mg/L
Cr VI	0.82 mg/L	Ag	0.10 mg/L
Cu	1.77 mg/L	Zn	0.60 mg/L

Analysis for these pollutants was carried out an independent, third party certified laboratory for control purposes, and samples were sent as generated weekly during operations – with the exception of pH which was monitored in the pilot facility for each discharge event.

6.3 Pilot Logistics

6.3.1 Operational Planning

Pilot operations were planned to take place on an on-week/off-week (may be two off weeks) schedule of the chemical processing. This was designed both for coal preparation and wastewater treatment to occur primarily while the plant is not operational (allowing for a reduced labor force required) as well as to allow time for sample preparation, shipment, and analysis to be conducted on various streams during off-weeks. General off-week tasks included:

- Coal crushing and preparation into the week-hoppers for storage and use
- Sample preparation for shipment and/or analysis for all related process samples taken the previous week
- Limited data reduction and review about pilot trends
- Pilot debrief with all operations staff to review safety, operability, and sources of improvement
- Coal disposal of last week’s leached and washed coal
- Chemical inventory and intake as required for next operational week’s plans
- Scheduling of the running week to account for critical operations staff
- Review of next tests to be conducted with the pilot operations staff

The planned on/off schedule was anticipated to reduce the potential overall number of positions needed to operate the facility from approximately 8 per shift to a minimum of 5, significantly reducing the labor challenges caused by COVID 19.

6.3.2 Chemical Intake and Safety

Chemicals were purchased and delivered to the pilot facility by an outside vendor and supplied in industry-pure forms (not ACS or higher grade). Liquids were delivered by totes (commonly 250 gallons), with the mineral acids being directly injected into the storage tank by the vendor – limiting the potential hazards associated with replacing totes of acid from a contained area to trucks for off-site disposal. Solids were delivered in pallets of 50 lb bags, with an average pallet containing over 40 of such bags. All chemical handling and movement was conducted with appropriate PPE for all employee interacting or working in the environments of large chemical handling. For eventual solids distribution, bags were opened under areas surrounded with local air filtration and exhaust to limit dust migration, and transport systems for solids were designed and built to have ultra-fine particulate capture to prevent any possible leaks and/or exposures.

Chemical safety training was given to all employees following UND’s chemical hygiene plan, and detailed reviews of spill containment, emergency stop, and other critical first-response plans for major exposure hazards were reviewed in text and through practice by UND Safety. These plans changed as a function of changing design and construction as construction and commissioning progressed, and physical changes to the plant were made. Reporting procedures of all spills, injuries, and other events were developed based on response need, review, and severity of the event

– including near-miss events. Annual reviews with UND safety including walkthroughs were conducted to ensure chemical safety.

6.3.3 Coal Handling

Coal was originally planned for delivery and transfer to crushers in super sacks (cubic yard bags), and equipment for moving, holding, and opening these bags was developed and planned. However, due to lignite's unique ability to auto-ignite if allowed to dry with potential air-flow through large piles, the decision not to crush the coal into hoppers until the week before testing forced the coals (all extracted in either 2019 or January 2020) to largely remain in super sacks until the plant was operational (~3.5 years of waiting). Once crushing was able to begin to support near-term pilot operations in late 2023, the bags had failed due to stagnation, time, and freeze/thaw cycles – and new methods of removing the coal from bags and scooping the coal into the crusher were developed. Once the coal had been crushed in hoppers (see section 6.1.2 for details), the hoppers were moved into position on scales to monitor the weight of the hopper during discharge, and replace as required during operation.

6.3.4 Disposal Planning

Disposal of material at the pilot facility were categorized into three possible categories – liquid non-hazardous, solid non-hazardous, and hazardous – with the last minimized as much as possible. Treatment plans for the wastewater to reach city POTW compliance were developed alongside Barr Engineering and the City of Grand Forks Wastewater Treatment Plant, and primary solid disposal (coal and iron-rich precipitate) was evaluated for hazard classification. Coal and iron-precipitate materials, when mixed generated a material deemed non-hazardous in all categories except flammability (due to the coal), and eventual landfill options for the material were found to be limited. Trucking to an approved coal-disposal landfill was eventually setup, and the materials of each distinct solid mixture (any time any major change to the possible materials) were evaluated for pH, TCLP, and other RCRA testing for potential hazard classification. For hazardous materials, the pilot facility – due to being somewhat off campus (approximately 4 miles) required the issuance of its own hazardous waste generator, primary concentration, and shipping classifications with the state of North Dakota, and employees were trained for hazmat packing conditions for hazardous materials.

6.4 Impacts of COVID-19 on Final Design

Having designed the pilot in late 2019 and the very start of 2020 (including with the budget finalization of the cooperative agreement) and beginning construction in late 2020/early 2021 radically changed the eventual design and construction plans. Equipment quoted during prior design phases drastically ballooned in cost in many areas (most impactfully sensors and electronics, of which some increased by over 100% in 6 months), forcing a number of decisions to limit cost being made across the plant to produce an operable pilot, even if not in the final form planned. The main control philosophy of the plant was desired if at all possible, forcing other decisions to be made to cut costs where possible and practical to remain remotely within the scope of the original scope. As such, the plant built differs from the original designs, but aims to operate in the same general manner, with some areas (notably the coal washing circuit) being unable to operate at or near the nameplate capacity as of the end of the project.

Compounding this cost problem – especially with electronics and sensors, was the rapid need to reduce as much as feasibly possible the labor involvement in the pilot – due to COVID-19 restrictions and practicality of implementing these during operation. This forced the project team to automate areas not originally planned, and to design areas to be operated in batch (allowing shifting of resources to other areas of the plant at different times) to reduce labor wherever possible. This is most notable in the original pilot design plan of ~8-9 operators per shift, dropping to a final maximum of 4-5 to meet nameplate capacity.

7 PILOT CONSTRUCTION

7.1 Facility Location and Characteristics

The building is located in Grand Forks, North Dakota’s industrial park on the edge of town. The building is standalone in the lot, not connected to other facilities. There is a railyard, metal recycling, and metal/welding shops located nearby.

Figure 33. South side of building housing the REE pilot facility.

The building was the old power generation facility for Grand Forks and has several 100-ton coal hoppers, high bay space, and significant electrical infrastructure available to accommodate pilot scale projects. In addition, in contained both a 5-ton and 50-ton crane present in the building (needing certification and repair), but significantly reduced potential expenses in moving equipment as needed.

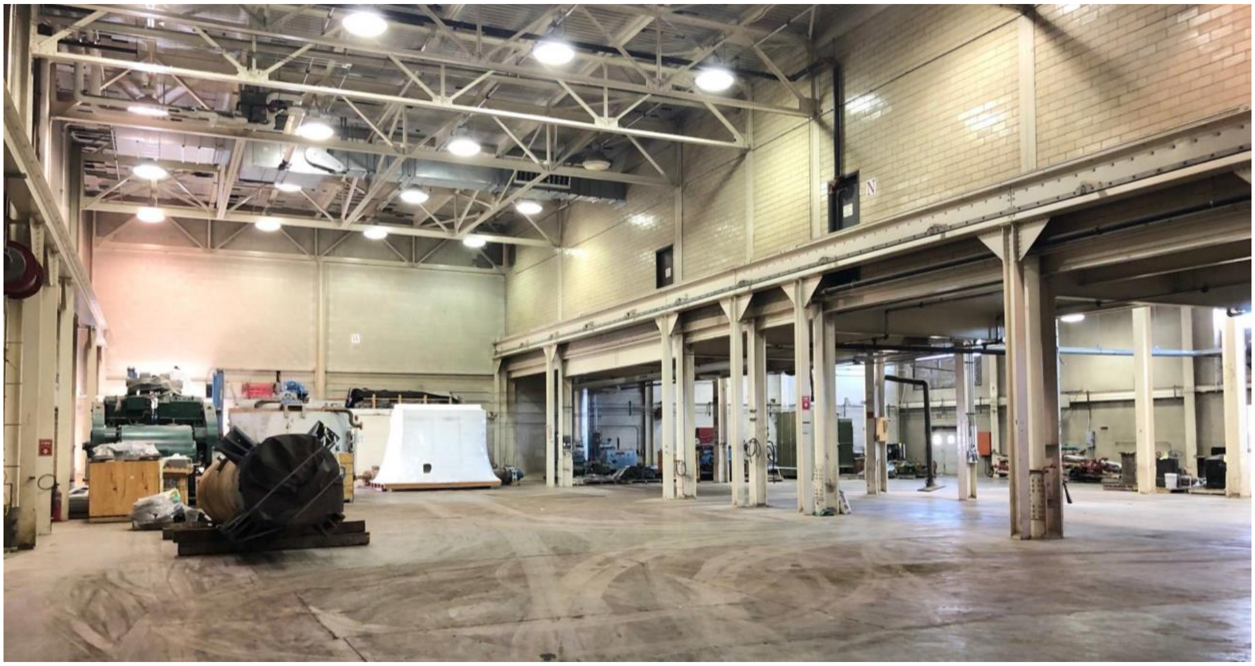

Figure 34. Side of the building allotted for the REE pilot facility.

7.2 Major Equipment Procurement

All major equipment (over $5,000 total) was procured through competitive bid processes, where the UND developed a list of specific technical and business criteria (including the relevant Buy America requirements) for each vendor to meet. Equipment procurement actions began following the Go/No-Go decision in late 2020, resulting in significant delays and cost increases over the designed equipment developed and quoted in 2019. Lead times originally quoted in 2019 at 5 weeks expanded as far as 13 months in 2020, causing drastic delays both in construction and eventual commissioning (any identified equipment changes caused months of delays).

7.3 Equipment Placement and Plumbing

The equipment placement and plumbing was largely determined by the building lease conditions at the time of acquisition. During the first year of the project’s second budget period, only the main floor of the building was leased and all other floors were considered unavailable and thus not considered when laying out the full process. Approximately 25% of the main floor was dedicated to the Rare Earth Element Pilot Project. It wasn’t until two years later that the full building lease was negotiated, giving UND access to all levels. At this point, an additional 25% of the basement level under the project was allocated for the pilot’s use. This space constraint forced original designs, and the bulk of the fixed (bolted down) major equipment had been installed prior to the availability of the basement. Coal feeding and crushing (the hoppers) were moved to the basement to make additional space on the main floor.

Figure 35. Original placement of major equipment.

Methodical placement of the equipment was required. Tanks were elevated and placed with enough room for walkways. Pumps were located under tanks and accessible for maintenance. Majority of the plant, with the exception of the solid's slurry lines, were plumbed with flexible pipe and cam and groove fittings for easy access and cleanouts. Floor drains were added across the entire plant to a controlled intermediary storage in case of spills and wash downs. All other liquid sources were plumbed to the 10,000-gallon waste water tank.

Plumbing size and material varied as a function of the plant, with primary liquid lines being either flexible tubing (secured with barbed fitting and hose clamps) or slurry lines being either rigid PVC or stainless steel. Mineral acid transfer lines were PTFE.

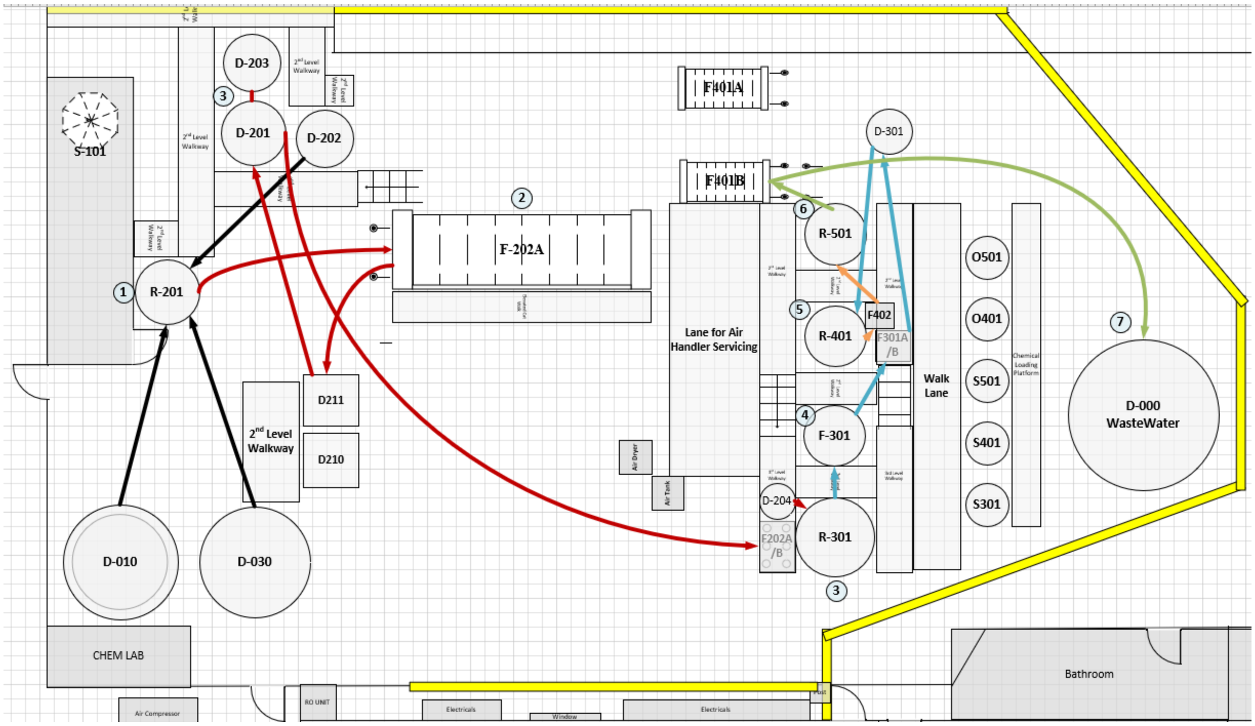

Figure 36: Most Common Operation Plumbing Layout

7.4 Electrical Wiring

Electrical wiring throughout the plant was completed by Northern Valley Electrical Service, contracted by UND to house a master electrician on-site for completion of all requisite high-voltage (>60V) electrical work. UND completed the low-voltage work, focusing on signaling and sensor wiring throughout the plant into large wire rack bays. All electrical equipment was determined to require spark-proof motors and housing due to the potential for coal dust in the surrounding environments in the event of coal containment loss.

All electrical motors over 120 volts were fed power through variable frequency drives – both to allow for power reductions to enable fine control of all motor operations, as well as to enable off/on control both locally (through button and/or switch to enable lock-out/tag-out procedures) and main operator control at the control station. These were housed each in water-resistant, ventilated cabinets away from the equipment in question (particularly in the event of possible splash risks), and were wired with quick-disconnects to allow for equipment movement during commission as required.

7.5 Safety Equipment and Needs

A variety of safety measures were taken throughput the plant's design and construction to educe potential exposure or injury risks throughout the plant. These included both physical barriers and equipment, sensors and instrumentation, and procedures and training. Each of these methods significantly contributed to the safety of operators and other in and around the plant.

7.5.1 Physical Barriers and Equipment Choice

The pilot facility, wherever possible, added physical barriers and equipment/piping choice to reduce chances and risks associated with exposure to chemicals, particularly potential liquid spray and/or concentrated chemical contact. These include:

- Adding a secondary containment tubing to concentrated acid lines in the event of a leak in any fitting and/or line breakage.
- Installing the acid transfer pump to draw from the top, rather than the bottom of the acid storage tank to reduce catastrophic leak chances.
- Adding a stainless-steel cover to the top and plastic sheeting to the sides of operational filter presses to reduce risks of filter press leaks – and to reduce spray hazards during cleaning cycles.
- Adding reinforcement at any turns in solid chemical transfer lines to reduce risks of failure by abrasion at sharp turns (avoided where possible).
- Selection of an air-operated double-diaphragm pump for slurry pumping into the filter presses to control potential outlet pressure (setting air setting for below rating of filter presses).

All pumps throughout the plant installed pressure relief valves to avoid over-pressurization, with the valves routing all over-pressure liquids to the tanks of origin of the fluids. All valves were evaluated on a case-by-case basis for safe power failure positions, focusing on first relieving any pressure through recycle lines, then to stop flow. This was most critical in evaluating any valves under pressure (such as those pumping to filters where back-pressure will exist) as well as any lines that transported hazardous materials.

For our operator intense tasks, such as coal cart handling and bulk chemical movement, an electric pallet jack was utilized. This allow safe lifting and transport of the large coal carts to a staging area where our forklift could then reach and remove it from the premises.

7.5.2 Sensors and Instrumentation

The team utilized numerous controls and sensors to ensure safety throughout the plant, particularly in regard to potential operator mistakes and in the cases of power failure and/or disasters. This was supported by the entire control system (computer and control cabinet) being powered through a battery back-up, allowing for operators to recognize any unsafe areas in the plant and make signal changes as a result of a potential power loss. In addition, multiple shut-off devices for each piece of equipment in the plant existed – including local on any larger powered equipment (anything over 120V), and distinct global shutoffs for high-voltage equipment, low-voltage equipment, and valves. Discussions of specific safety-based controls by process areas are below.

Plant-Wide Controls

Alarms were used throughout the process in many areas, and had three possible forms: i) visual control alarm – the values began flashing red on the operator screen to identify to the operator something was out of position; ii) visual and audio alarm on control screen – adding a noise to ensure operators took immediate action; and iii) cabinet visual and audio alarm – representing the most critical possible alarms to safety. This range allowed for operators to prioritize critical alarms for resolution, rather than all alarms (such as a faulty sensor in a currently un-used section of the plant) from alarming in the same fashion.

As all areas contained tanks, pumps, and other shared equipment (flowmeters, etc.) some controls were standardized across the entire plant. This included tank overflow/underfill controls and alarms – tied to specific pump actions and valve positions to prevent overflow in the involved tanks, to avoid unsafe conditions. Overflow conditions create obvious unsafe areas (chemical spill), but underfill conditions could result in some tanks with chemical dosing to contain fluids below measurement devices to track chemical dosing (such as pH meters), causing the potential for over-shoot and over-dosing of chemicals. These alarms were tied to radar-based level sensors capable of penetrating foam to ensure accurate and prompt alarming conditions at all times.

Pumps similarly were controlled with similar conditions, with all pumps with a pressure transducer attached in their line having critical pump function associated with that pressure. In the event of over-pressure (and below the safety release valve pressure), the pump's valve positions would switch to a recycle flow into the origin tank to prevent compounding of a leak and possible line rupture. Pump speed could then be slowed over time to a halt, if the fluid allowed for this (not a slurry that could plug pump internals if flow stopped in lines).

Filter presses also had relative uniform sensor packages to evaluate performance, with the large presses (F101 and F201 – all 30 cubic feet or larger) adding light curtains and pull cords that prevent press operation while tripped. In addition, the presses' control systems were built with specific manual lock-out procedures in place, where an operator working on the press could prevent the operator at the control station from acting on the presses while working in the area – preventing potential injury. All presses (F401 and F501 included) had pressure transducers to monitor incoming feed pressure, but were also fed by AODD pumps that restricted the maximum potential pressure of the fluid to the safe operating range of the presses. Additionally, this pressure measurement would alarm with audio in the event of a sudden pressure drop (apart from when selecting to open the press or during feed stoppage) as any sudden reduction in pressure was identified to be a containment leak from a plate – something that forced immediate action by the operator.

Process Area 0 Safety Controls

Controls focused on possible chemical leaks, coal leaks, and difficulty in feeding of materials. The acid storage tank had sensors deployed on the ground surrounding the double-walled tank (and within the berm region) to monitor for the presence of any liquids on the ground. Any liquid presence both lit up warning lights on the control cabinet and alarmed on the operator panel of the program. Additionally, the pump feeding the acid from storage to the distributed tanks was able to operate in reverse, allowing for the lines containing acid to be largely cleared at the end of operations – reducing the amount of acid present outside of triple-containment areas.

Coal alarms were focused on pressure of pneumatic feeding (under vacuum). High vacuum pressure reading identified potential plugs in the system, and alarmed for the operator – as well as controls to automatically reduce feed rates if pressure began rising during coal feeding. Low vacuum pressure implied a lack of coal in lines, and would commonly require operator action – running a vibrator attached to the hopper for a short period of time – to continue feeding. This was not done automatically, as incorrect vibrator force as well as duration could cause negative effects (further compaction of coal for long durations without feed, non-commensurate force causing physical damage to equipment).

Process Area 1 Safety Control

Splash guard were installed surrounding the spiral to prevent splash and spray of potential coal/water slurries, and all relevant plant-wide controls were used.

Process Area 2 Safety Control

All plant-wide controls were utilized, including critical underfill alarms and automatic acid pump stoppage controls associated with underfill in relevant acid-dosing tanks. Additionally, sensors tracking the solids loading of the slurry (TSS) visually alarmed if the value was significantly deviating from expected levels – allowing an operator to prevent either incomplete filter press filling (if too low of a slurry) or possible blockages and pump failure (if too high of a slurry) during operation.

Process Area 3 Safety Control

The iron precipitation tank had unique controls, given the large quantity of foam that could be generated during the initial pH increase with sodium carbonate. While the overflow and underfill alarms for the tank were with a foam-penetrating level sensor, an additional level sensor that was not (and would thus measure the foam itself) was needed to ensure manageable foam and prevent potential overflow of foam (causing wetting of surfaces that fed solid chemicals – eventually leading to a blockage in those lines).

Process Area 4 and 5 Safety Controls

These process areas utilized the plant-wide controls, as well as specific safety controls surrounding the solid oxalic acid feed (and specific measurement and tracking of flow and pressure of pneumatic feeding). In the event of any non-operator caused (manual intervention) change in expected pressure, an audible alarm would sound to prevent continued feeding – as a potential leak risk may exist and needed immediate addressment. Similar systems were employed with sodium carbonate feeding to reduce risks.

Process Area 6 Safety Controls

Wastewater treatment was located too far from the acid storage tank for use with the pumped mineral acid without significant concern as to the range of tubing, so local supplies of acid were used (purchased and used in gallon bottles). As such, safety controls were not sensor-based but were training-based for spill risks and clean-up. Otherwise, management of all other plant-wide sensors were conducted on tanks and pumps.

7.5.3 Operator Procedure and Training

Personal Protective Equipment was provided to all team members and strictly enforced. PPE requirements. Any task specifically requiring PPE required training with a senior team member and signatures from that team member noting that the person had received and passed training. These signatures and procedures were evaluated by UND Safety for appropriateness, and records kept of all trained employees – restricting tasks to be complete by un-trained persons.

In case of spills, several large industrial spill response kits were located at key locations across the plant. These kits consisted of neutralizing absorbent pads, pillows, and snakes. In addition to large spill response kits, there are also two large drums of acid/base neutralizing medium. Additional spill dams were purchased for isolation of spills and wash down fluids. Several safety showers and eye wash stations were stationed throughout the plant, routinely tested, and readily accessible. Training was provided to all team members on locations and usage of all safety equipment.

Figure 37. Spill kit locations and contents.

8 PILOT COMMISSIONING

Pilot commissioning was achieved through a steady process of: i) verification of equipment design operational parameters, ii) leak-testing of all plumbing and equipment, iii) electrical testing to ensure safe operation, iv) testing of equipment and filtration with relevant process liquids (slurries where applicable) in water (no chemical addition), and v) final process testing with chemicals in the system. Throughout the commissioning effort, a number of equipment, process, and operational changes were made to improve operational safety and operability of the pilot system. These amended designs made in Section 7,

8.1 Equipment Commissioning and Coal/Water Testing

Equipment was commissioned by adding coal and water to the equipment and evaluating performance.

Upon delivery of the pilot plant tanks, valves, sensors, motors, and pumps were installed in their intended operation location with supporting infrastructure. Electricity was provided to motor controllers and motors, LabVIEW control wires to sensors and electric motor controllers, PVC or stainless-steel piping between tanks and pumps, and air lines to valves to be able to open and close. Commissioning of all this equipment and supporting infrastructure involved bump testing with electricity, flowing water through equipment to determine leak points, trial and error testing of the LabVIEW computer control of equipment, and coal and water testing to stress test equipment.

There were six process areas that needed to be commissioned before adding chemicals in the system. Adding acid was the final point of when the project team would be confident in generating leachate, and the equipment needed to be checked and commissioned to ensure operators understood how equipment behaves, and no failure points existed before adding acid. Below is a general list of equipment per process area that was commissioned.

- Process area 0 – Coal Handling and Chemical Handling
    - Coal crusher
    - Particle sizer
    - Screw conveyor
    - Hoppers
    - Scales
    - Rotary valves
    - Pneumatic transport lines and fan
    - Cyclones
    - Double dump valves
    - Baghouses
    - Mineral Acid Tank
- Process area 1 – Spiraling of Coal
    - Pre-leach tank
    - Spiral
    - 3 size fractionated split holding tanks

    - Pumps
    - 2 Filter presses
    - 2 holding tanks for wash water
    - Flowmeters
    - Control valves and open/close valves
- Process area 2 – REE Leaching
    - Leach tank
    - 2 Filter presses
    - 3 leachate and wash water tanks
    - 6 filter bags
    - Surge drums
    - Pumps
    - Open/close valves
    - Flowmeters
    - pH sensor
- Process area 3 – Iron Separation
    - Reaction tank
    - Clarifier
    - Surge drums
    - 6 filter bags
    - Pumps
    - Flowmeters
    - Open/close valves
- Process area 4 – REE Concentrate Production 1
    - Reaction Tank
    - 2 Filter bags
    - Pumps
    - Total Suspended Solids sensor
    - Flowmeters
    - pH sensors
- Process area 5 – REE Concentrate Production 2
    - Reaction Tank
    - 2 Filter presses
    - Pumps
    - Total Suspended Solids sensor
    - Flowmeters
    - pH sensors
- Process Area 6 – Wastewater
    - Wastewater storage tank
    - Mixing tank
    - Filter press
    - Pumps
    - Resin column
    - Surge drum

During pilot plant commissioning, the team was able to isolate individual pieces of equipment to commission small areas while others were still under construction. A single tank's pump, flowmeter, and valves could be rerouted to flow back into the tank to water test and determine functionality of LabVIEW controls and ensure sensors were providing data to the control computer. This also included failure position checks – ensuring that in the event of power or air loss, sections of pipeline would not remain pressurized or open to atmosphere while transporting acidic solutions or slurries.

Challenges were overcome during commissioning where the project team found that when crushed coal was added to the first tank, the slurry pumps were undersized for the output of the crusher's particle size. This resulted in multiple pumping tests with different various pumps and particle sizes. The pilot plant team addressed this challenge with the use of a double diaphragm pump that had flap valve check valves. The original pump that was purchased had check balls inside the pump and the gap between the balls and the pump walls was not large enough for oversize (particulate larger than the crusher nominal crush size) coal particles to pass through. Although this problem was solved, it uncovered other problems with slurry pumping and pumping slurry into a filter press that were addressed:

- The mixers inside tanks were undersized and required alternate blade design.
- Slurry required a minimum pumping flowrate higher than originally predicted. Testing showed slurry was settling out in lines at the process design flowrate with the particle size present.
- Feeding a pump from the bottom of a tank (where any settling could occur) resulted in line blockages

After the pilot team was able to successfully pump the slurry in a recycle loop from the tank to the pump and back into the tank with the correct pump, the team began commissioning the filter press. Challenges observed and addressed were:

- The low flow rates that were required of continuous pumping resulted in poor filtration.
- Conveyor belts and screw conveyors for removing filtered coal were not adequate for removing wet coal at the rates required.

The pilot team tried multiple variations of pumping speeds, different styles of pumps, changes in filter press chamber size, adjusting mixing speeds in the tank, air blow down time lengths and programing of filter press controls. In conclusion of troubleshooting, the filter press purchased required high amounts of slurry pumped in to fill the chambers from back of the press to the front of press. A double diaphragm pump was installed at the inlet of filter press to be able to transport the slurry to the back of the press at an initial flow rate of approximately 100 gallons per minute. The pump inlet was changed to a suction line that pulled coal slurry from the top of the tank instead of from the bottom of the tank as originally designed. This allowed both easier pump isolation, as well as reduced the likelihood of feeding a non-homogenous, denser slurry into the pump.

With the change from continuous flow to a batch filling mode, the full filter press capacity was significantly larger than the potential coal capacity of the leaching tank. As a result, the filter press was shortened to ensure the amount of slurry in the leaching tank was able to fill the press.

PA-2: Complete filling of the filter press is essential to enhance both the overall throughput as well as reduce hazards with trapped acidic solutions in the filter press which discharges with the cake. Optimal coal concentration in the slurry for the leaching process is determined by filter press filling performances. Figure 38 shows the results of preliminary tests conducted on water-freedom mine coal slurries before chemical usage, using different slurry densities of coal and varying filter press capacities. At the first test, filter press capacity is set to 25 cubic feet (equivalent to 22 chambers) and coal slurry density of 36 wt.%. At these conditions, approximately 1,800 lbs were able to be loaded into the filter press prior to cessation of pumping. This corresponds to approximately 81 lbs/chamber for the Freedom Mine material. A second test was conducted at the same coal slurry density, but with a lower filter press capacity of 20 cubic feet (equivalent of 18 chambers) resulted in a total coal loading of approximately 1,400 lbs, resulting in a nearly equivalent coal loading of 77 lbs/chamber. Using this average of approximately 79 lbs/chamber, this expected ratio was utilized to maximize the potential filter press size while ensuring complete filling within the confines of the storage capacity at a 36% slurry density of the leaching tank.

A third test was conducted to evaluate the effect of increasing coal slurry densities from 36wt.% to 44wt.% while maintaining the same filter press capacity of 206 cubic feet. The resulted coal loading was 64 lbs/chamber, which is 21% lower than the 36wt.% under similar conditions. The loss in capacity per chamber was believed to result from the earlier stoppage of pumping due to the challenges at pumping the thicker slurry, resulting in an incomplete fill of the press.

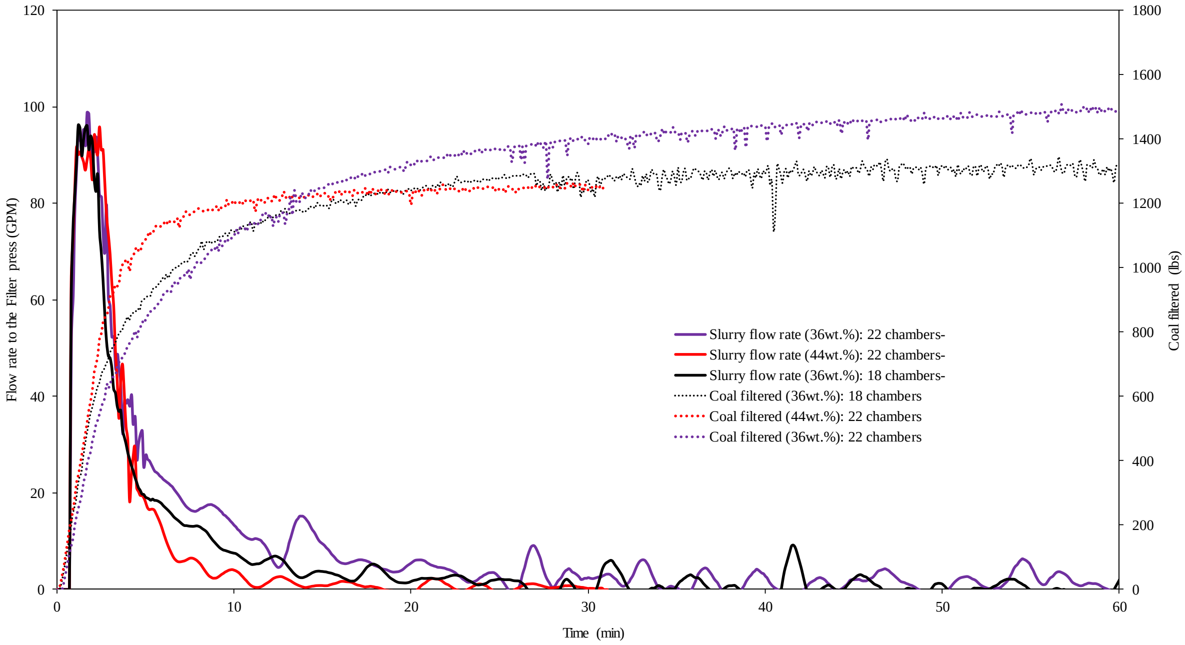

Figure 38. Effect of coal slurry density on pumping and the mass of coal filtered per filter press capacity.

However, the filling performance of the filter press depends not only on the pumping performance and the concentration of the coal in the slurry but also on the coal density. Loading 40% (mass) of Rhame bed with 60% (mass) of Freedom Mine coal instead of pure Freedom Mine will result in reduction in the overall density of coal. To avoid misconception around filler press filling and determine the correct coal concentration that aligns with a desirable filter press filling, an estimation of R-201 capacity of coal was plotted against the expected range of variation in the overall coal density. Rhame bed density is averaged at 10.3 lbs/gallon and Rhame bed density varies based on moisture content, typically 13.5 lbs/gallon, additional moisture can be absorbed by the coal without a noticeable volume change. Thus, the target coal slurry density was limited to 37wt.% to ensure that complete filling is possible without the interference of thick slurry consistency. The full capacity of R-201 is 630 gallons, and variability of the coal quantity in the tank requires that a lower potential filling capacity be used in the event of either a slightly leaner coal slurry than expected, as well as a potentially lower-density coal (due to moisture variance, etc.). This was represented by Figure 39 which indicates that with pure Freedom loading in R-201 the optimal number of chambers that can be filled at 36 wt.% of coal concentration are 24 chambers. If 26 chambers are chosen instead of 24 chambers, there is not expected to be enough coal in R-201 to satisfy a “packed” filling. Furthermore, if coal concentration is reduced from 36wt.% to 34wt.% (a change realistically within the range of measurement error), it becomes safer to go with 22 chambers instead of 24 to grant a packed filling of the filter press. This is also applicable to the change in coal density, mixing Freedom Mine with H-B-Bed results in greater coal loading in R-201 than solely the Freedom Mine material due to the Rhame bed density being greater than that of Freedom Mine, chiefly due to the dryness of the material (Rhame bed being stored indoors as compared to Freedom largely stored outdoors). The density of the mixture falls between 10.3 lbs/gal and 13.5 lbs/gal. Using the planned average of 11.5 lbs/gal, there would be sufficient coal in R-201 at coal concentration of 36wt.% to fill 26 chambers.

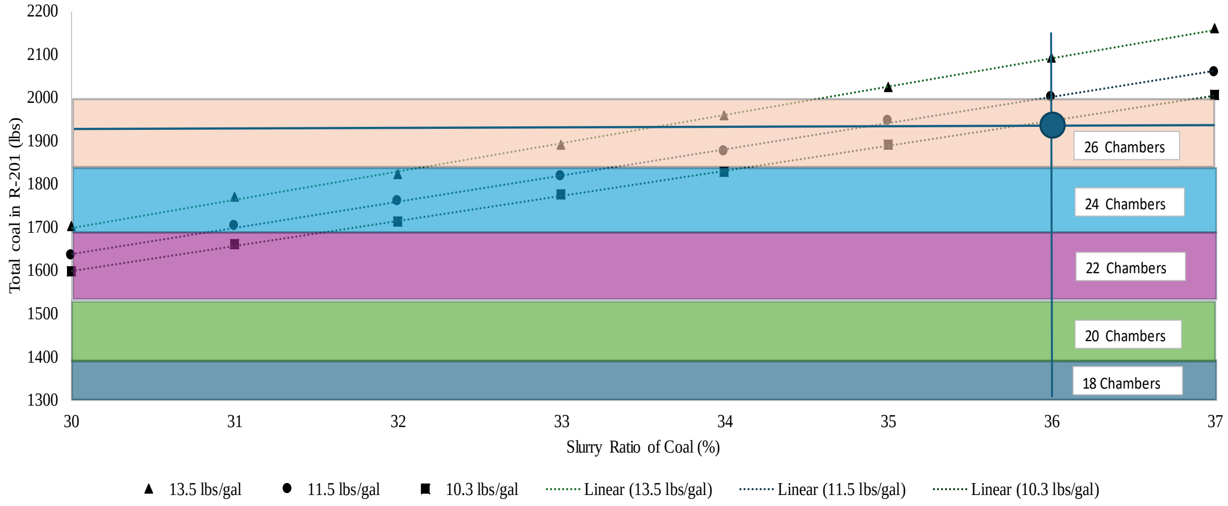

Figure 39. Effect of coal density on the on R-201 coal holding capacity and defined filling of the filter press

Figure 40 illustrates the change in coal loading in R-201 along 47 conducted leaching batches. Each batch was conducted under a fixed filter press capacity of 24 chambers and maintained slurry concentration at 36 wt.% where possible. As demonstrated, the variability of slurry density in R-201 was inconsistent and was found difficult to accurately measure and track from batch to batch. High ending level tank is an indicative of poor filter press filling which is translated into thicker slurry. Based on these observations, the coal loading to R-201 are adjusted for the upcoming batch. Corrections from batches were conducted by visualizing and analyzing data from previous batches. For future efforts, real time monitoring of coal density is key to track the coal slurry density in the leaching tanks and ensure a near-constant value. This ensures effective filter press filling and subsequent filtration, key to allowing for effective leachate recovery and minimizing safety hazards.

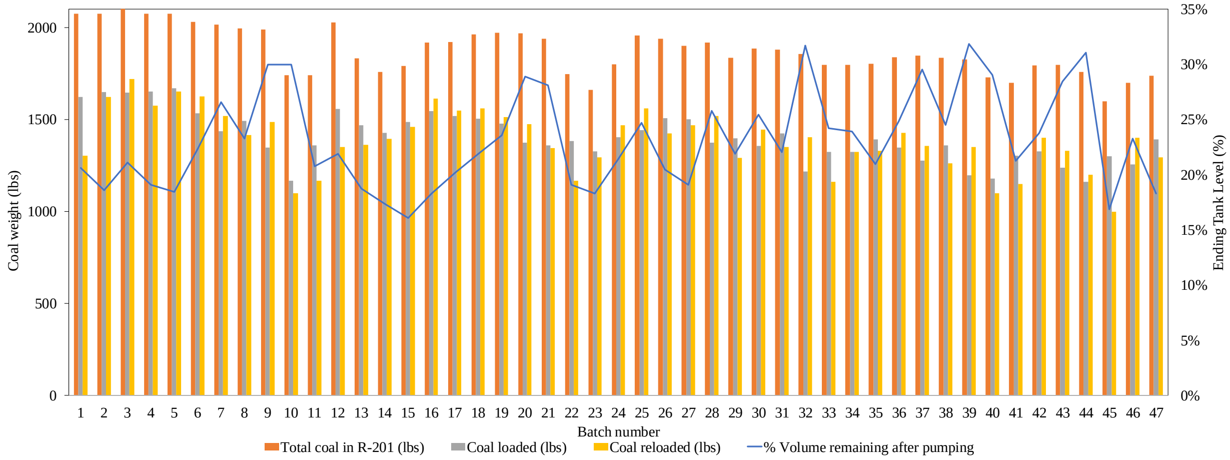

Figure 40. Coal loading in leaching tanks and subsequent filter press filling over the period of testing.

Another process variable that was critical towards the success and completion of the testing for PA-2 is the control of mixing speed. As shown in Figure 41, the mixing speed was adjusted and optimized to maximize the slurry flowrate (represented by level change of R-201). In the original case the mixing speed is adjusted to follow a linear decrease correlated to the level change, with a minimum mixing speed of 1/3 of maximum speed. This was done to prevent from losing the pump prime caused by turbulent mixing and introduction of air into the slurry mixture. However, this still resulted in poor mixing and required adjustment. A regimen of discrete mixer frequency variation at specific tank levels corresponding to the level of the first and second mixing impellers were applied as additional conditions to the original correlation. From Figure 41, this adjustment enhanced the overall flow rate and resulted in better pumping performances compared to the original case. This better mixing profile ensure that during the periods of passing the first two impellers while draining the tank, significantly turbulent mixing and introduction of air into the slurry was avoided wherever possible. Once passing the first two mixing impellers, the mixing speed of the final kick impeller (at the base of the mixing shaft) was increased to the maximum to keep the slurry entrained.

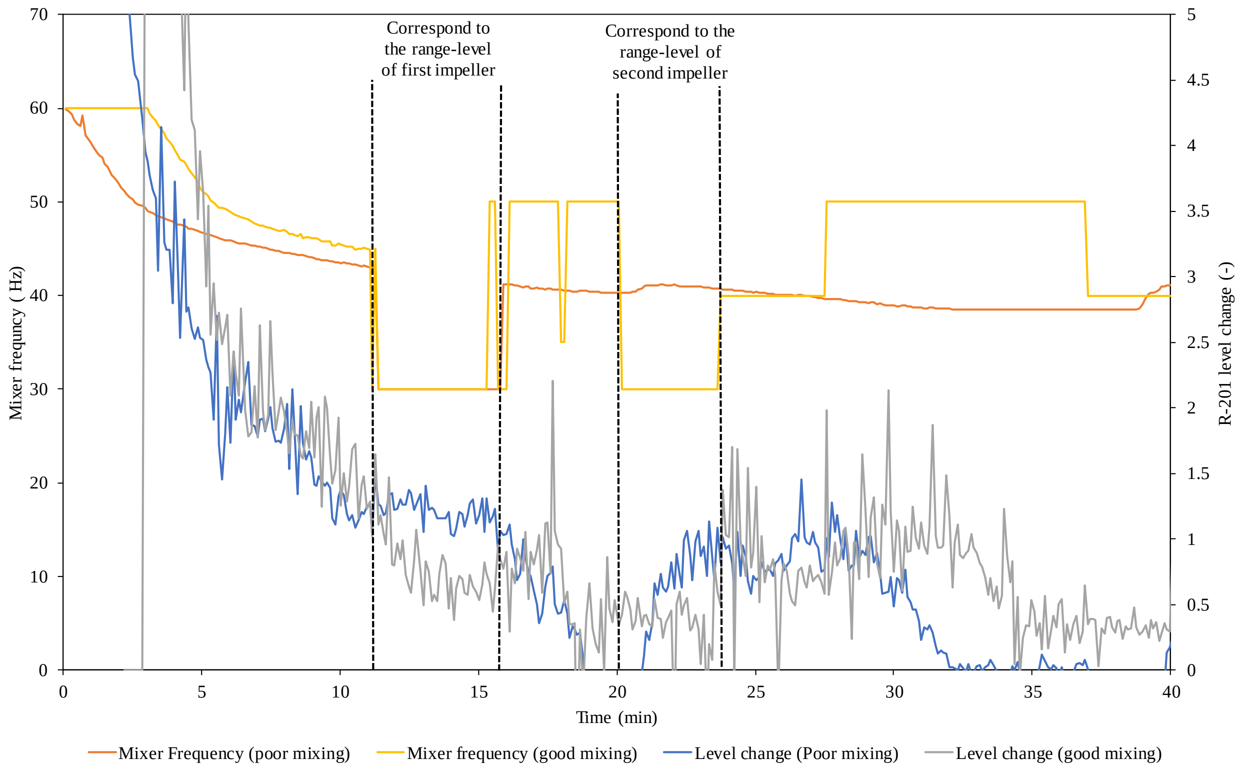

Figure 41. Effect of mixing speed control on the slurry pumping performances

8.2 Chemical Safety Training and Planning

The chemical safety training for the project was guided by the UND Office of Safety as well as UND Energy and Environmental Research Center (EERC) EHS. Several comprehensive HAZOPS were conducted by both safety offices and cautionary measures were taken to protect personnel as much as possible. Before delivery of chemicals to the facility, everyone on the team was required to go through online training for lab safety awareness and chemical handling. Additional onsite chemical training and lab safety awareness training was provided to all team members to prepare them for research operations. Safety training was repeated on a semi-annual basis and documented. Training was provided on handling of all unique chemicals present, as well as spill response procedures for them. All safety training materials, MSDS, and manuals were made available offline locally in the control room office. In addition, dedicated personnel were trained in HAZMAT packing procedures to ensure safe shipment of all samples and materials produced from the facility. Additional discussion of safety equipment procured and developed processes may be found in Section 6.2.

8.3 Complete Process Testing

8.3.1 Complete Process Testing

The entire end-to-end process was tested under controlled conditions, simulating results obtained from bench-scale operations. This included testing process parameters such as slurry density, residence time, flow rates, and chemical conditions (pH, concentration of additives, etc.) for each area of the process. Additionally, troubleshooting and optimization efforts were conducted for each unit operation to achieve desirable performance and operability. Operational data and product distribution of REEs were analyzed under proposed and modified operating conditions to validate the design and ensure that all components were aligned for optimal performance to achieve the desired product quality.

Throughout the testing, the key outcomes from the complete process testing include:

8.3.1.1 Coal Handling

Pneumatic conveying was optimized for both dry and wet conditions and integrated with a PID control system. A stable coal feed rate of 2100 lbs/hr was achieved for relatively dry Rhame bed coal (25 wt.% moisture content) and 1300 lbs/hr for relatively wet Freedom Mine coal (35 wt.% moisture content). Feeding at above these levels was possible, but risked forming blockages at fittings and turning points in the line due to the moisture content and stickiness of the material.

In addition to moisture content, the coal loading rate depends on coal characteristics such as particle size. Larger particles improved separation at the cyclone and allowed for greater flow. However, the maximum loading rate is not limited by blower performance but is instead tied to the throughput capacity of the cyclone and the coal characteristics. The cyclone and subsequent pneumatic valves loading coal into the leaching tank were found to have a theoretical capacity of up to 2,500 lbs/hr, but elevated moisture content combined with a smaller particle size reduced this. The coal was found to occasionally build on the transport surfaces of the cyclone, effectively increasing velocity and resulting in worse separation.

For control of the pneumatic conveying system, operators had continuous, remote readings of vacuum pressure at the blower inlet and scale readings of the coal hoppers. Variance from target vacuum pressures were indicative of challenges (lower pressure noting a lack of feed from hoppers, higher representing a potential blockage in the flow path). Control systems were designed to operate near-autonomously to control for specific vacuum pressures (with control variables being feeding speed through rotary valves), with only hopper vibration being a sole manual control. Manual control of vibrations was necessary, due to the potential for hopper movement on the scale when near-empty (also when vibrations were commonly most required).

8.3.1.2 Leaching

Commissioning tests indicated that a solid concentration of ~36 wt.% in the slurry was optimal to ensure desirable flow rates during pumping while preventing solids from settling or causing line blockages. To achieve this solid concentration, each batch requires approximately 350 gallons of fresh leaching fluid and 1,600 lbs of coal, added to the remainder of the prior batch (with variability needed based on final tank level of prior batch).

After coal is loaded into the leaching tank, it is mixed with the acidic solution at a controlled pH for a duration of 45 minutes, controlled through continuous pH monitoring and acid addition. Testing demonstrated that this leaching duration achieves an effective maximum REE recovery of approximately 44% with the feedstock selected while minimizing potential leaching of non-valuable, deleterious components. Extending the leaching time was found to result in slight additional REE recoveries (~1-2% for 2 hours) with significant additional impurity element extractions (Fe increased by 7%, Al increased by 4%). The pH target chosen was selected based off of the known extraction profile of elements within the coal to reach a maximum of REE extraction while minimizing deleterious element recovery (most notably Th and U). A reduction in pH by 0.1 units resulted in a 3% REE recovery increase paired with a >10% additional recovery of the actinides, as well as additional trivalent impurity extraction (Fe and Al). The potential implications for processing and treatment of the actinides informed the decision to operate at a position of lower Th and U extraction.

However, pH measurement in the leaching tank emerged as a challenge during testing. Submerged industrial pH sensors were prone to exhibit sensor drift immersed in the metal-containing solution, and this drift could either manifest as masking the pH change (drifting downwards while pH rises) or exacerbating this change (drifting in the same direction to force over-leaching). To address this, a time-dependent sampling approach was implemented: samples were periodically extracted via a newly installed sampling port and analyzed in a local laboratory for precise pH measurements with more routine calibrations that the tank ports. This method allowed for accurate corrections to acid dosage, and enabled a more accurate pH leaching environment.

The installed TSS sensor, mounted on the tank wall, proved unreliable as it provided localized readings that were dependent on the mixing conditions in the sensor's immediate vicinity. As an alternative, an estimation of concentration based on coal density, feed rates, and moisture content was employed, eliminating the need for additional recirculation lines and possible points of blockage during unmixed periods. This calculation was evaluated to be accurate to within 2% of slurry density during prolonged testing and measurement, allowing for a guaranteed avoidance of slurry densities over 38%.

Separation of leached coal from the leachate solution is performed using a 375 cu.ft filter press. The slurry is pumped at an average flow rate of 16 gpm using an air-operated diaphragm pump (AODD). The AODD pump is designed with a maximum capacity of 120 gpm at low discharge pressure (close to atmospheric) and 3 gpm at high discharge pressure (95 psi). The discharge pressure is determined by the backpressure generated during filter press filling. The pumping cycle was tested to take an average of 30 minutes to fill the filter press, initiated after the residence time for leaching had lapsed. The filter press is rated for a maximum pressure of 100 psi, and the discharge pressure of the AODD pump is maintained at this maximum allowable pressure throughout the pumping cycle to avoid over-pressurization (and subsequent leakage or damage) of the filter press.

8.3.2 Iron Precipitation Performance

In process area 3, iron precipitation was operated in both continuous or batch modes. The chemical feed of sodium carbonate was controlled through a PID control loop to condition the incoming, filtered leachate to the desired pH of 3.5 continuously. Sodium carbonate was added from bags to a hopper stored beside the processing tank (S-301 on Figure 36), dropped from the hopper using double-dump valves for gas isolation, and pneumatically transported using a vacuum conveyor to a cyclone on top of the tank, separating the chemical into the tank. The gas return was plumbed to a baghouse surrounded with local ventilation to avoid any dust discharge in the area. The double dump valves were controlled via a PID system, allowing for autonomous adjustment of the sodium carbonate feed in response to the inlet processing flow. Flow into the tank was controlled tightly. In batch mode, this is due to foaming in the tank at high sodium carbonate addition rates, and in continuous mode, it results in poor separation of the iron solids in the clarifier due to a shortened residence time.

In continuous operation, the reaction time is controlled by the residence time in the tank. However, significant challenges were encountered in this process area, particularly with the separation of the formed iron solids. The integrated clarifier was found to be inefficient except at either very slow flow rates (below 2 gallons per minute), or when the clarifier had any appreciably quantity of solids contained (where discharge of the solids would discharge a thinner slurry due to the lack of a thick layer present) and the separation achieved in continuous mode was found to be inadequate at the conditions tested. Large 1-micron filter bags downstream of the clarifier – meant for only polishing actions - were unable to handle the high load of fine iron particles and were prone to rapid blinding on either condition stated above.

To address these challenges, PA-03 was operated in batch mode, where the clarifier was filled with processed leachate from the iron precipitation tank. The system allows sufficient residence time for solid precipitation, and clear liquid is subsequently drawn from the top of the clarifier. Although this approach extends the overall processing time for the leachate, it successfully resolves the inefficiencies associated with the original continuous mode design with minimal actual reduction in throughput when scheduled properly with shifts.

8.3.3 REEs Precipitation Performance

For the REE precipitation circuits, TSS and pH are identified as critical parameters for defining the quality and efficiency of REE precipitation. The design aimed for a two-stage precipitation operation, first to precipitate a REE-rich, Sc-lean mixed oxalate concentrate using free oxalate concentration as the primary distribution method. As such, the first precipitation action occurring was found to be the REEs, with a slight delay prior to the precipitation of Ca and Sc oxalates. Careful monitoring and control of both TSS and pH are essential for the success of the precipitation split. To determine optimal operational conditions, multiple tests were conducted in the pilot plant, accompanied by rigorous product characterization.

Initial tests achieved low REE purity due to slow reaction kinetics due to slow oxalic acid dissolution rates at the pH's employed (below 2). This limited the rate of precipitation and enabled overdosage of oxalic acid, leading to co-precipitation of calcium with oxalic acid. To remedy this slow dissolution and to enhance the responsiveness of the REE precipitation (occurring only effectively above pH 1.75), a pH value of 2.25 was chosen to be maintained autonomously through a similar PID loop controlling process area 3. At this pH, oxalic acid was dosed slowly while observing the response in TSS. A solid precipitate concentration of 100-130 ppm was identified as the range required to achieve high-purity REEs for the leachate in question.

PA-04 and PA-05 were tested in continuous operation mode. However, due to limitations in the iron clarifier (from PA-03) which could not be continuously operated in a stable manner, REE precipitation was also shifted to batch mode for better process control and performance. The method of control planned for PA-04, and what could be envisioned in a commercial process, would be the maintenance of a specific oxalic acid dosing concentration in the tank, with a periodic decline in concentration to observe a similar small decline in TSS response. This would indicate that the tank is still operating on the lower edge of the plateau between REE and Ca precipitation, and avoid the potential for overdosage.

In the event of overdosage into PA-04, the TSS would dramatically increase as Ca oxalate formed, and this precipitate was found to be stable in solution except when the pH is lowered below 1, something not easily achieved in the pilot. Oxalic acid could not be used for this lowering, as the subsequent free oxalate concentration at higher pH values when restored would be well into the Ca oxalate precipitation window, and repeat the challenges of before.

For PA-05, this process was designed to precipitate solids to a threshold TSS (where anything above this would be acceptable, but non-preferred). This was the end of the Ca oxalate solubility, guaranteeing all REEs and Sc (more insoluble oxalates than Ca) would be precipitated from solution. In practice with better control, a more favorable value may be found where incomplete Ca and complete REE precipitation could occur, although this was not tested to this degree in this piloting study.

8.3.4 REEs Filtration Performance

Initially, filter presses were employed to filter high-purity grade REEs (c) precipitated in PA-04. However, the filtration efficiency was only 75%, which resulted in significant losses that negatively impacted the REE recovery of the process. This inefficiency can be attributed to the relatively low concentration of solids collected in PA-04, measured at 1 gram/gallon of leachate, as well as the small size of particulate (estimated at below 15 µm).

To address this issue, 1-micron filter cartridges were installed downstream of PA-04 to improve recovery efficiency. These cartridges successfully captured up to 99% of the REEs. However, the main limitation of the 1-micron cartridges is their lower capture capacity, which is approximately 10 times smaller than that of the filter press. As a result, the cartridges require more frequent cleaning and replacement, which increases operational downtime and maintenance requirements. However, this did result in more frequent REE concentrate 1 samples to be taken, allowing a more granular evaluation of purity enhance mechanisms.

For the low-grade REEs (concentrate 2 – mixed with Ca), the larger volume of solids and the larger solids (from the presence of larger Ca oxalate crystals) allowed for the use of the filter press for filtration. A filter press of a volumetric capacity reduced to 0.5 cubic feet was employed for this testing, and was not opened in between batches (only at ends of operating days and/or weeks).

8.3.5 Wastewater Treatment Performance

During the neutralization phase, the wastewater undergoes chemical treatment to adjust its pH to 6, facilitating the precipitation of metal hydroxides, primarily iron and minor amounts of aluminum. After 30 minutes of mixing, a 2% w/w flocculant is introduced to coagulate these fine solids, thereby enhancing filtration efficiency. Filtration into a 3.5 cu ft filter press was effective in capturing these flocculated particulate, primarily Fe and Al based.

The ion-exchange column is engineered to effectively remove copper, nickel, and zinc, ensuring their concentrations in the effluent remain below the Environmental Protection Agency (EPA) discharge limits defined for the Grand Forks Public Wastewater Treatment Facility: 1.7 parts per million (ppm) for copper, 3.57 ppm for nickel, and 0.6 ppm for zinc. This performance is achieved using AmberSep™ IRC748 resin, which has demonstrated high selectivity for heavy metal cations, including copper, nickel, and zinc. The resin's operating capacity for these metals (and low capacity at these conditions for the major element in solution – Na) and excellent physical stability make it particularly suitable for such applications.

To achieve the desired metal removal, a resin volume of 3.5 cubic feet is utilized at a flow rate of 5 gallons per minute (gpm). The column's diameter is optimized to maintain a linear velocity greater than 10 meters per hour, preventing reversible de-ionization and ensuring efficient ion exchange.

For column washing and neutralization, 41% of the treated water is recirculated. To reduce the volume of wash water, it is neutralized and recycled through the column after the initial 300 gallons, which removes iron, addressing the inefficiency observed in the filtration of iron during wastewater treatment.

8.4 Establishment of Final Process Operational Schemes

With commissioning complete, the following operational schemes were developed.

Process Area 0: Coal was crushed to a nominal topsize of 0.25”, with filling of four hoppers conducted prior to each operational week – 2 each of the Freedom Mine lignite as well as the Rhame bed material. These were kept distinct, and fed independently into R-201 for direct leaching at separate time intervals, while the acid solution was pumped into R-201 from a separate tank. The acid was fed at a maximum rate of ~1.1 liters per minute into the leaching tank when required, and also into a separate tank for pre-dosing the leaching solution to the correct pH of leaching prior to testing.

Process Area 1: This process area was not operated during the scope of this project, and was bypassed in favor of gathering critical data from the rest of the facility for ongoing engineering work.

Process Area 2: Once the leaching solution and coal were dosed into R-201, a mixed leaching time of 40 minutes was established from prior bench-scale data, and acid was fed as required during this period based upon the planned test in question. Samples were taken at two points within this period for each batch, once within 5 minutes of testing (for pH back-up analysis against the installed probe) and again at the end of testing to produce samples. Once the 40-minute time had elapsed, pumping of the leaching solution began using the installed AODD pump suctioning material from the tank and pumping this into the filter press. Flow would be continued until one of three possible parameters were reached: i) Pumping speed dropped to an average of less than 4 gallons per minute – measured both by level and flow sensors as well as counts of strokes, ii) total elapsed time of pumping exceeding a target threshold (typically 40 minutes), or iii) individual pump strokes beginning to occur over 2 seconds.

The cake would then be blown down with pressurized air at 40 psig for 15 minutes to remove remaining contained acidic solution, and the cake discharged through opening of the press. This pumping and cake procedure was found to produce reliable, dry and low-moisture cakes with an average of less than 3 gallons of free moisture upon discharge (making up less than 2% of total filtered volume), reducing potential safety risks associated with filter press clean-out as much as practical. Additionally, coal discharge moisture contents were measured at between 35-38%, effectively equivalent to in-ground moisture contents for lignites. For the tank, the solution, coal, and acid would be replenished for another batch – with total batch times for the tank estimated at about three hours for standard operation (100 minutes for coal and solution feeding, 40 minutes for leaching, 40 minutes for pumping). For the press, a two-step cycle of cleaning was instituted on each batch, first a quick-clean designed to remove cakes of solids wherever possible and scrape any materials remaining followed by a pressure-wash cycle to ensure clean sealing surfaces of all plates – found necessary to reduce the possibility of potential side leakage.

The filtered leachate was stored in a holding tank for downstream use, and was passed through a battery of filter bags (ranging from 10 µm to 1 µm) prior to flow into process area 3. The discharged coal cakes would then be wetted and neutralized using available caustic materials to reduce the hazards associated with the acidic-leached coal, followed by stockpiling for final collection and discharge for disposal.

Process Area 3: Twice-filtered leachate would be pumped into R-301 at a consistent rate of approximately 2 gallons per minute (rough flow that allowed for acceptable performance and equivalent to leachate production rate), with sodium carbonate consistently fed to maintain a pH at a target level of 3.5. This was utilizing the findings from the bench-scale effort, that showed a pH of ~3.5 was effective in removing considerable (>80%) of the Fe without noticeable REE loss. With continuous feeding of liquid and solids, foam production was considerably lower than during initial filling and batch processing. This material was pumped into the clarifier (F-301) with an expected residence time of approximately 8 hours, where the overflow was collected into a surge drum and measured with a TSS sensor to ensure effective solids removal. Iron-sludge slowly built in the bottom of the clarifier, and was removed once or twice per operational week, depending on the level (known through separate liquid and sludge level sensors installed in the tank). The solid-lean overflow passed through more filter bags at ~1 µm in size, before reaching process area 4. Samples of the cleaned liquid was collected at intervals of approximately twice per shift, with the solids sampled as produced from the clarifier bottoms discharge.

Process Area 4: Processing of the REE concentrate 1 began with a batch charge of R-401, followed by an initial measurement of existing TSS levels through flowing past the in-line TSS sensor. Any level above 50 ppm necessitated a pre-filter prior to precipitation, which was achieved through flowing through a 1 µm cartridge filter to capture any remaining solids in suspension. Once preliminary TSS levels were determined low enough, addition of oxalic acid in steps – paired with additions of sodium carbonate to maintain a target pH of 2.25 were performed. In these, once a likely point of plateau was discovered at a specific concentration of oxalic acid in the solution, this was used as a baseline for future additions – beginning slightly before this concentration. Once the TSS had risen to the desired level (calculated based on the expected REE concentrations in the solution based on prior data), the slurry would be pumped and captured on filters before passing to process area 5. Due to the low level of concentrate in the solution (commonly ~1-5 grams per gallon of fluid), significant build-up of concentrate was desired both to improve filtration (through formation of a cake) as well as for a sizable layer to build up to avoid sampling error.

Process Area 5: Precipitation of the remaining REEs was accomplished through addition of the remaining oxalic acid required to achieve the target 1.5 times stoichiometric needs of the solution (accounting for expected consumption from dissolved metals not precipitating in solution – Fe and Al) or TSS target (whichever arrived first). Commonly, this only required an additional concentration addition of 0.01-0.02 molar of the oxalic acid, highlighting the difficulty in achieving the REE-speciation through the use of concentration. In the pilot system with batches of testing of 600 gallons or more, this amounted in cases to additions in the second tank of less than 2-5 pounds.

This oxalic acid was mixed into liquid programmatically controlled to a pH value of 2.25 to ensure prompt dissolution and precipitation. Mixing of this solution once the oxalic acid dosing was complete continued for 30 minutes, prior to filtration through a filter press (effective capacity of 0.5 cu ft). The second concentrate was found to generally produce larger particulate than the first, noted by the lack of bypass through the 10 µm effective capture radius cloth of the press with the second precipitate as compared to the 30% bypass of the first concentrate solids. Once the solid cake was formed and the liquid fully filtered, the cake was washed with RO water to remove residual PLS liquids from the cake, followed by blowdown to dry the cake to a workable amount (20% moisture) and discharge.

Process Area 6: The final process area, the wastewater processing system, follows a precipitation and ion-exchange process to reduce all permitted metal concentrations to the necessary levels for sanitary sewer discharge (per permit #14 from Grand Forks City Public Works Wastewater Treatment Plant). The depleted leachate is pH-adjusted to 6.5, filtered of contained solids, and passed by resin columns containing AmberSep 748 cation exchange resins to capture residual transition metals, pH monitored (pH of resultant liquid does not fall below 6 during normal operation, and pH of below 5.75 is indicative of possible breakthrough of resin during laboratory experimentation) and discharged. The AmberSep resin is regenerated with a 5% sulfuric acid solution. The wastewater processing (pH-adjustment and resin pass) is conducted batch-wise on batches of produced wastewater (wastewater being produced in batches of ~10,000 gallons from upstream processing and stored for downstream treatment and disposal). Previous designs of the resin column allowed for floating of the resin – which was determined in early testing to result in insufficient metal capture (primarily Zn). This column design was changed from a upward flow to a downward packed and flooded flow to prevent liquid bypass around the resin. For regeneration fluid treatment (to prevent hazardous waste generation at the facility), the regeneration fluids for the resin columns are treated with flocculant and sodium carbonate to a pH of ~8, with flocculant addition added to achieve a 1% mixture with the solids precipitated. This takes place in the same precipitation tank as the wastewater treatment during periods of extended down-time of the pilot.

9 PILOT OPERATIONS

9.1 Preliminary Continuous Testing

9.1.1 Preliminary Operations

Once the system has been fully constructed and commissioned under relevant process conditions, the first two months of operation (non-continuous) were

9.1.2 Operational Challenges

The following summarize the operation challenges accounted during the processing of 100,000 lbs of coal.

a. PA-0:
   The coal cyclone experienced clogging at the inlet, leading to inconsistent feed and bypassing. To address this, routine maintenance and cleaning were scheduled.
   Wet coal limited the feed rate, slowing down the process.
   Accumulation of solids in the hopper blocks the inlet and causes issues with the screw feeder. Implementing a hopper vibrator can help level the solids.
b. PA-2:
   Air entrained by mixing turbulence limited slurry pump performance. Efforts were made to monitor level disturbance and program in mixer speed changes for simplifying operability.
   In-situ washing of coal in the filter press was not feasible with the current design of the plate and different design.
c. PA-3:
   Iron solids settled into the pump casing, causing the centrifugal pump to struggle at low flow rates. A minimum flow recirculation line with flowmeters was installed to resolve this issue.
   The clarifier was not efficient in continuous mode of operation.
d. PA-4:
   controlling TSS in R-401 proved to be very challenging and time-consuming. It was difficult to control the reaction without performing very small increments of chemical dosage over a prolonged reaction time.
   Oxalic acid compacted under its own weight, forming large chunks that could not be fed by pneumatic conveying, necessitating the design of a screw feeder equipped with a mixer to break the particles.

9.2 Production Testing

With a finalized process and preliminary analysis on the operations of the pilot, the final 6 weeks of pilot testing were conducted at largely continuous conditions, excepting the last week where testing to evaluate the potential of leachate recirculation for the purposes of reducing chemical usage were considered. This data will be broken into a discussion of the average performance of the first 5 weeks of scaled, consistent testing, followed by analysis of the alternative leaching circuit.

Starting with the high-purity concentrate production occurring in June, 2024, future testing was focused on the specific conditions utilized and achieved during those testing runs. Replication of these runs produced relatively stable results across the weeks of testing, both in overall chemical usage, relative REE purity of initial precipitates, and general operability of the pilot facility.

9.2.1 Coal Slurrying, Leaching and Filtration

As discussed in Section 8.1, an optimal level of coal/water slurry had been identified at approximately 35% solids by weight – a level that both maximized the solids concentration in the slurry while remaining a pumpable fluid for filtration purposes. Production testing focused on maintaining this slurry density wherever possible, achieved through first filling R-201 with acidic liquid to reach a threshold level, followed by addition of the mixture of coal through the pneumatic conveying system (weight fed measured by loss of weight of hoppers on scales), where slurrying would begin. However, testing with both the 300-ppm coal blend and separate coal materials identified a variable volume change as a function of weight, most notably affected by drying and coal weathering. As such, preliminary lab testing was completed to evaluate the current moisture content and the saturated moisture content of coals tested to reduce the likelihood of a lower coal/water slurry (preventing overflow in coals with a higher volume change/mass ratio than average).

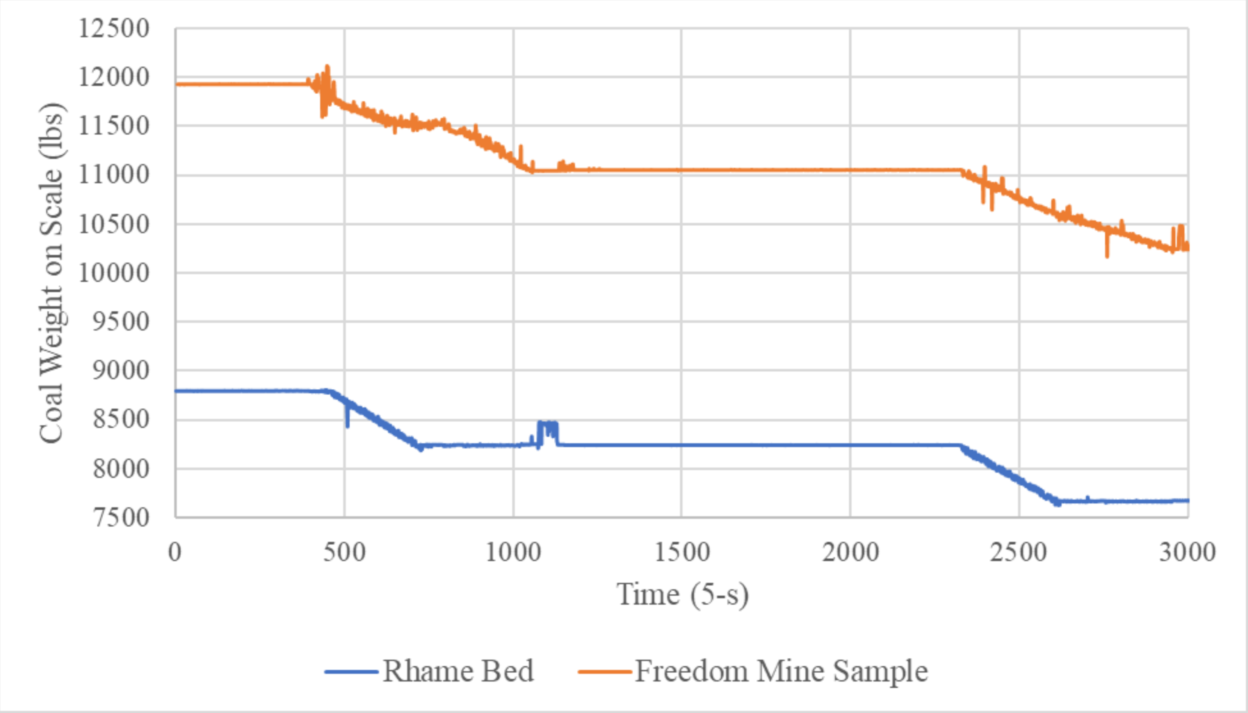

Figure 42. Coal mass feeding rate (constant volumetric) as a function of time between both blended coal feedstocks.

During leaching, a sample of solution was taken from the tank at both the first minute of full coal/liquid slurrying, as well as 20 minutes to both evaluate current solution conditions and for analysis. Table 11 includes an example analysis of a sample taken during filtration (at the conclusion of leaching.

Table 11. Example REE analysis of leachate PLS.

Element	Concentration (ppm)	Element	Concentration (ppm)
Sc	1.38	Gd	1.76
Y	7.17	Tb	0.27
La	5.00	Dy	1.48
Ce	13.5	Ho	0.27
Pr	1.85	Er	0.73
Nd	7.72	Tm	0.09
Sm	1.71	Yb	0.59
Eu	0.44	Lu	0.08

Primary filtration proceeded along guidelines described in Section 8.4 for the filter press, with an average recovery of ~300 gallons of leachate (approximately 75% of the fed liquid, with the residual remaining in the coal. In coals with higher original moisture contents (including one near-saturated due to weather), higher percentages of liquid recovery were achieved, up to 95%. Secondary filtration through the 10, 5, and 1-micron bag filters was conducted in a more continuous method to allow for continuous flow into PA-3. Limited coal fines were detected in these filters, amounting to less than 0.01% of all solids fed. As such, bags were only replaced either due to reaching a maximum pressure differential, or quarterly, depending on the time period elapsed.

9.2.2 Impurity Removal and Liquid Processing

Following coal filtration, iron precipitation was completed through addition of sodium carbonate in a semi-continuous fashion, utilizing pneumatically fed solid sodium carbonate as the basic material. Addition of the sodium carbonate to the PLS did produce significant foaming, manageable during "continuous" (slow liquid and chemical addition) operations but challenging on start-up without a solution of the equivalent pH and concentration present in the tank. Start-up operations in this area were managed over a 2-day period to limit the magnitude of foaming to within the tank (foaming of up to 300% of the liquid volume was possible during this period as compared to <20% of liquid volume during "continuous" operation). This smaller level of foam did not interfere with solid base addition (pH was responsive in seconds) as compared to the larger foam during start-up (pH unresponsive in minutes with base addition). During continuous operations, this is not expected to present a challenge in scaled testing/production, although may present a challenge for start-up operations.

Precipitation of the iron material was found to be near-complete after 2.5 hours of a mixed period, with the precipitate found to be between 1 and 10 µm. With these ultra-fine precipitates (combined with the high moisture content), filtration success was limited, with the goals of the system aiming to settle the material instead of rapid changing and cleaning of filters. Settling was originally controlled through a residence time and flowrate through the clarifier designed, although this was found to result in small amounts of particulate to overflow into downstream polishing filters if clarifier sludge volumes exceeded 5%. Due to the difficulty of controlling this low level and flow out of the clarifier, the team elected to transition the clarifier into a batch operation of a 10-14 hr settling time, with clarified liquid removed from the top and pumped forward. Residual sludge was removed periodically (roughly weekly) as the level increased in the tank. This method, while effective in producing a clearer filtrate (critical for downstream REE processing) did result in lower liquid retention, resulting in a wetter iron-based sludge produced. Liquids were sampled from post-polishing filtration for liquid analysis, while the sludge was periodically sampled for solids analysis (not run-dependent, due to the weekly discharge). The precipitation rate of relevant species is presented in Figure 43.

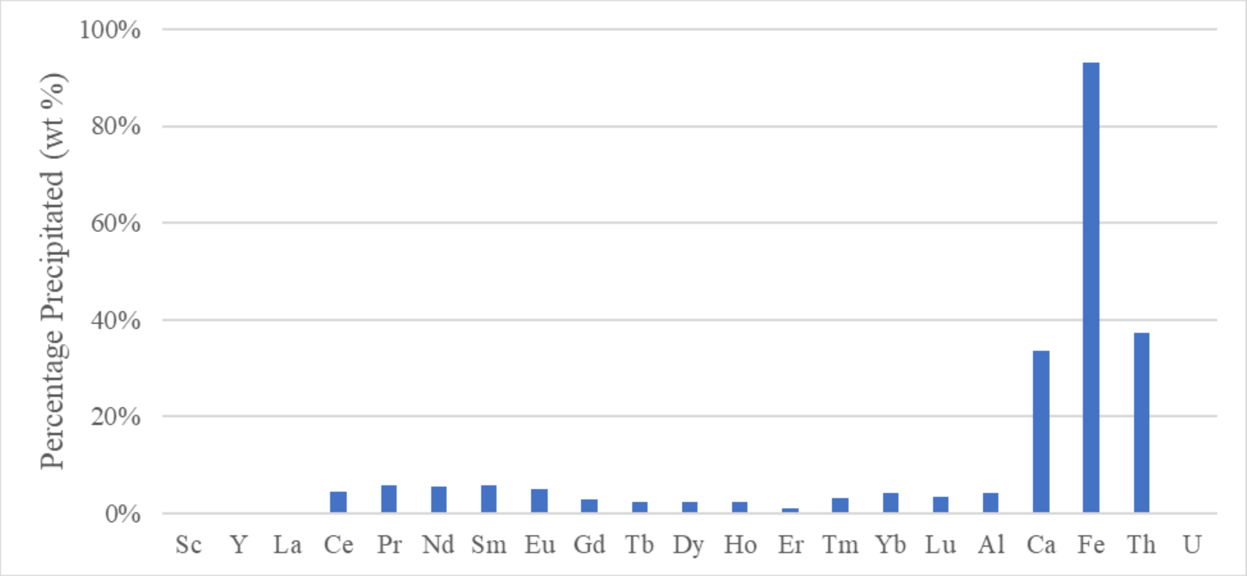

Figure 43. Precipitation efficiency of process-relevant species in PA-3.

9.2.3 <u>Production of High-Purity Direct Precipitation Concentrates</u>

Rare earth precipitation was designed process-wise as a two-stage precipitation process, utilizing the variable solubilities of calcium and the REE oxalates using oxalate concentrations as the separation factor. The first stage aimed to precipitate a largely REE-pure precipitate (avoiding Ca and other diluent admixture) through tight control on free oxalate concentrations, most cost-effectively managed through variable oxalic acid dosing rather than pH. Identifying the region of selective REE precipitation was achieved through continuous monitoring of total suspended solids (TSS) – measured through a continuous pumped loop from the tank through a calibrated section of pipe with the sensor. Precipitation was determined to effectively occur at pH values of 1.9 or above, of which the process aimed to keep the PLS solution at a pH of approximately 2.25 during oxalic acid feeding to promote rapid dissolution of the acid as well as ensure prompt TSS response with slow oxalic acid addition (Figure 44). Note: the TSS sensor's upper range of 300,000 mg/L resulted in noise at very low levels of measurement (less than 0.1% of range).

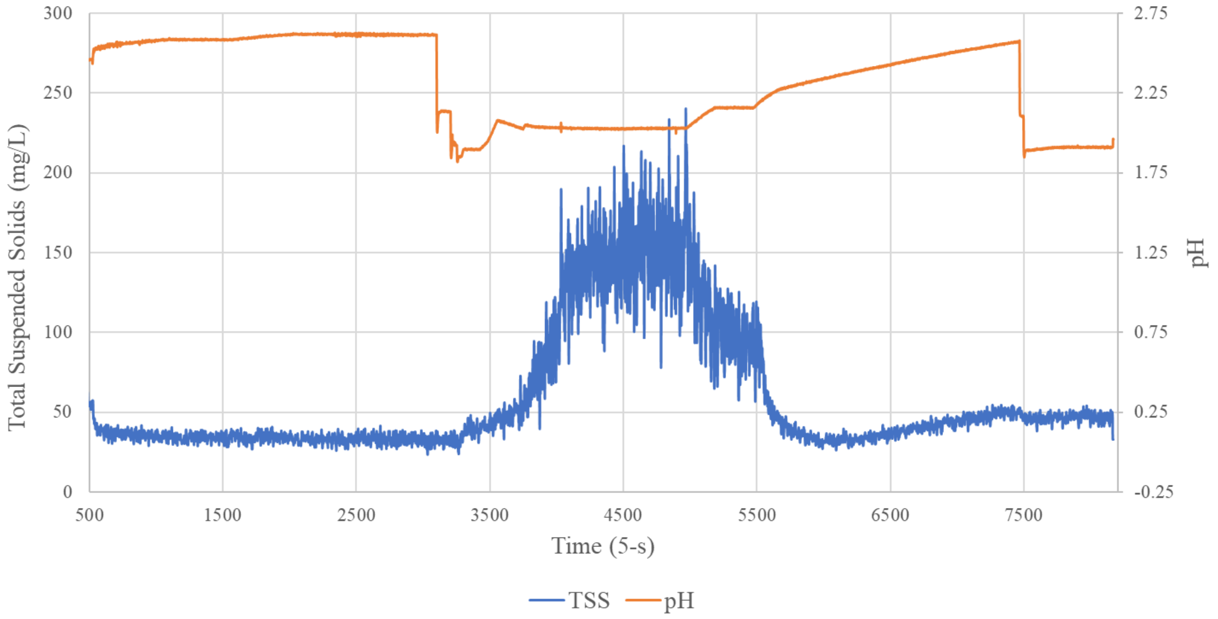

Figure 44. Precipitation profile and subsequent filtration of the first REE precipitate during successful speciation.

With successful speciation of the REEs using the variable oxalic acid concentration profile, significant concentration improvements in the REEs could be achieved as compared with direct precipitation of all materials. This speciation allowed purities of >90% as compared with the Ca to be achieved, while direct complete precipitation yielded no higher than 6% REE purity. While this speciation did require testing to be able to be achieved (Figure 47), eventual direct precipitation purities of greater than 80% were achieved, exceeding the original project goal of 65% purity for these products. Notably, the Sc content was designed to be lower in these precipitates so at so produce a partitioning effect into the lower-REE yield second precipitation product, allowing for a significant relative enrichment of Sc in these materials.

9.2.4 Recovery of Residual REEs and Concentrate Processing

Remaining REEs and CMs of interest were recovered during a secondary oxalic acid precipitation, including the significant majority of the Sc contained within the original PLS. To achieve this precipitation, additional oxalic acid was added to reach a pre-determined concentration (one identified to precipitate all REEs, roughly 1.2x stoichiometric levels for REEs, Al, Fe). TSS sharply rose during this addition, evident of the Ca oxalate precipitation (Figure 45). In this precipitate, a low immediate REE concentration was expected (most REEs are precipitated in the first step, with the remainder precipitating during Ca oxalate precipitation, significantly diluting this material - Table 12). However, this division of concentrates did show marked Sc enrichment in this product, with relative Sc percentages ranging from 5-10% (from an initial feed of less than 2.5%). This 2-4x concentrating factor achievable with no additional chemical requirements (another tank and filter only) is expected to add value to compensate for the additional capital and minor operating expenses associated with this, as well as reduce risks of downstream yield associated with further processing of concentrate (majority of the REE do not require additional processing to a refinable purity if first precipitation step is carried out properly).

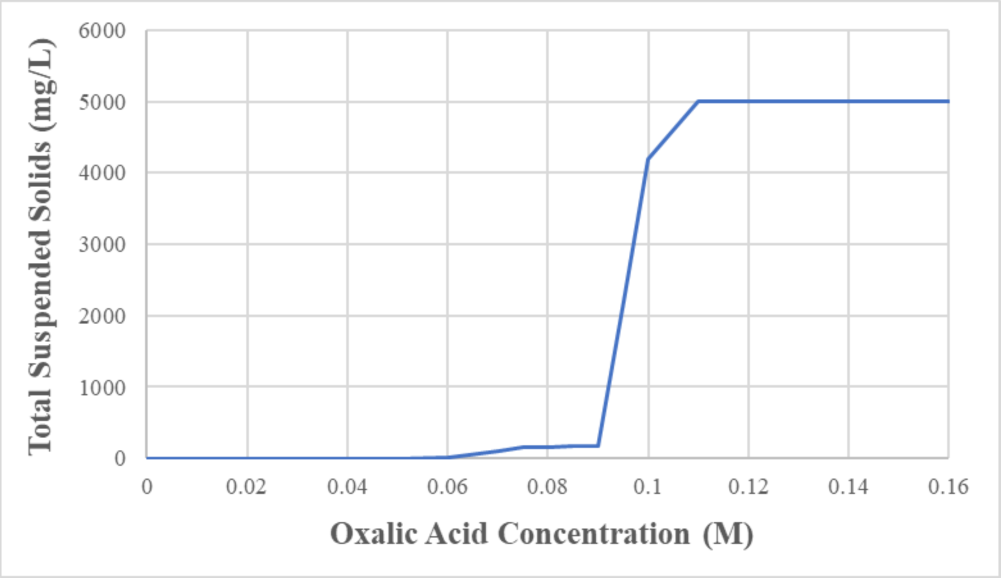

Figure 45. Expected TSS profile with oxalic acid addition.

Table 12. Concentration profile of an example directly-precipitated second mixed REE product.

Element	REE Concentration (ppm)	Element	REE Concentration (ppm)
Sc	1548	Gd	866
Y	6277	Tb	139
La	4482	Dy	909
Ce	10328	Ho	174
Pr	1136	Er	542
Nd	4071	Tm	70
Sm	745	Yb	482
Eu	189	Lu	64

[*] TREE content of 3.20% oxide basis as-precipitated.

To reach salable qualities of REEs, this material would be processed through the calcining and dissolution/ion-exchange system described in Section 5 of this report, with evidence of reaching purities of up to 90% determined on this equipment. Schedule and budget constraints prevented effective processing of this material at the pilot scale during this effort.

9.2.5 Analysis of Recirculated Leachate on Performance

Preliminary analysis of the effect of recirculation of the original leachate into the original leaching circuit has been identified as a potential path for relative chemical reduction as compared to REE yield, a significant potential cost-saving method. This was theorized to relatively enrich the REE in the PLS as compared to primary diluent impurities (Ca, NORM) due to the selective leaching process and the reversibility of the leaching from the ore. This would in general reduce the total chemicals required downstream on a per-kg-REE basis, aiming to reduce high operating costs. However, a negative expected from this recirculation would be a decreased overall REE recovery, due to the higher REE content in liquid being trapped within the porous matrix of the coal (already a notable source of REE loss).

The final testing of this project utilized a 30% recycle of the leachate generated in successive tests over a week period to selectively enrich the REEs as compared to other deleterious and diluent elements in the PLS. For these tests, physical modification to allow downstream filtered leachate to be pumped into the leaching tank (R-201) was completed.

Table 13. Example REE content in recirculated PLS.

Element	Concentration (ppm)	Element	Concentration (ppm)
Sc	1.82	Gd	2.21
Y	9.24	Tb	0.331
La	5.89	Dy	1.88
Ce	16.2	Ho	0.342
Pr	2.31	Er	0.934
Nd	9.79	Tm	0.120
Sm	2.14	Yb	0.753
Eu	0.531	Lu	0.105

This increased REE concentration did show a reduction in REE recovery of between 1 and 8% (with highest reduction in recovery of La, Ce, and Eu) as expected due to the increased concentration in the leachate being trapped in the porous lignite during filtration. These percentage changes are percent's of the original percentage, not percentage points (i.e. a 5% reduction of 40% results in 38%, not 35%). This behavior would be expected to be less pronounced (with a smaller reduction) in systems where the detailed coal washing would be able to be employed.

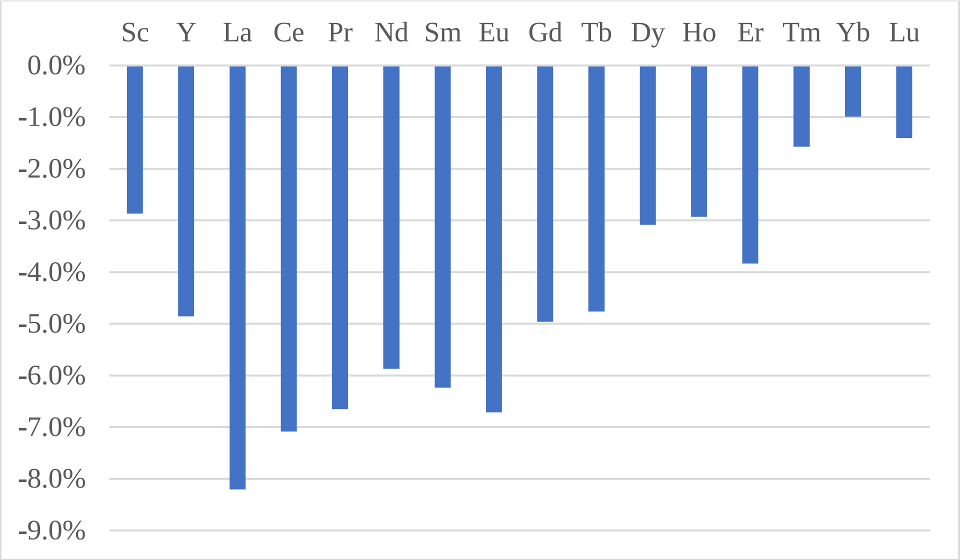

Figure 46. Percentage reduction in leaching efficiency as a result of a 30% leachate recycle.

The increased REE concentration (and Al concentration) in the PLS did cause upsets in the REE precipitation steps (as the change in both of these concentrations in solution changed the expected oxalic acid concentration required for the REE-rich concentrate split). As such, the overall REE concentration in products decreased, cut would be expected to increase in a stable system where experience with the precipitation with this specific PLS would enable more accurate precipitation targets. In addition, the previous week's data showed a lower concentration due to the operation of the pilot with other coal sources intermediately between the piloting for this project (utilized a lower concentration feedstock, and required some re-benchmarking of the piloting team for the new feedstock testing).

Table 14. Concentration profile of REE-rich concentrates from week before and during PLS recirculation (all in oxide basis).

Element	Conc. of REE 1 (wt %, standard)	Conc. of REE 1 (wt %, recycle)	Element	Conc. of REE 1 (wt %, standard)	Conc. of REE 1 (wt %, recycle)
Sc	0.13%	0.16%	Gd	3.26%	2.63%
Y	9.06%	7.59%	Tb	0.45%	0.37%
La	3.65%	2.54%	Dy	2.28%	1.95%
Ce	15.41%	13.01%	Ho	0.37%	0.32%
Pr	2.54%	2.04%	Er	0.94%	0.82%
Nd	12.70%	9.99%	Tm	0.10%	0.10%
Sm	3.10%	2.45%	Yb	0.60%	0.55%
Eu	0.79%	0.62%	Lu	0.09%	0.08%

[*]TREE contents of standard and recycle at 55.47% and 45.23%, respectively.

With these changes in the PLS, chemical reductions were achieved throughout the process (Table 15). This average reduction would be expected to result in a more economic facility, although more testing to find specific points of highest benefit remain to be evaluated. Effects of this process on CMs such as Ga and Ge (both unable to be leached appreciably from this coal as described in Section 9.2.6) are unknown, and would need to be tested in more detail in future efforts.

Table 15. Chemical reductions achieved throughout the UND REE process while utilizing a 30% PLS recycle.

Chemical	Area of Reduction	Percentage Reduction (per-kg-REE basis)
Mineral Acid	Leaching	18%
Sodium Carbonate	Impurity Removal	14%
Oxalic Acid	Both REE Precipitations	21%
Sodium Carbonate	Both REE Precipitation	7%

9.2.6 Impacts of Weathering on Piloting and Data

The data produced from the pilot testing, while valuable in the ability to produce higher-purity concentrates directly from precipitation of the PLS and at pilot scales, does not reflect the expected potential of the commercial technology due to the state of the feedstock by the finalization of testing. The feedstock – procured prior to the Go/No-Go decision and allowance for pilot c4447onstruction to begin due to project risks associated with the procurement – had been extracted from the ground more than 4 years at the completion of testing, causing drastic changes in the coal matrix and very likely some degree of critical minerals involved. This is most notable in a comparison of the extraction ratios of the exact same material at the bench scale 4 years prior (while the material was fresh), found in Table 16.

Table 16. Variability in extraction from bench-scale to pilot-scale testing.

Element	**Extraction (Bench)**	**Extraction (Pilot)**	**Element**	**Extraction (Bench)**	**Extraction (Pilot)**
Sc	33.34%	21.92%	Tb	54.79%	50.16%
Y	57.28%	32.81%	Dy	55.26%	48.73%
La	42.42%	40.18%	Ho	56.34%	47.93%
Ce	45.53%	46.40%	Er	53.16%	46.87%
Pr	46.28%	46.05%	Tm	51.02%	46.32%
Nd	46.71%	45.06%	Yb	49.22%	45.44%
Sm	46.02%	47.50%	Lu	47.83%	50.19%
Eu	48.85%	48.61%	Ga	30.48%	2.16%
Gd	52.38%	38.23%	Ge	28.57%	10.15%

This variability is stark when considering the value proposition of the produced concentrates come over 70% from the combined Sc, Ga, and Ge elements – all of which were dramatically reduced in extractability. This is also with a system known to have better dewatering efficiencies (and thus expected improved recoveries) of the leachate from the coal as compared with the bench system, as well as other notable improvements downstream of this, increasing the change occurred from this. To describe the extreme weathering that had occurred over the prolonged construction and commissioning period, the dry-basis BTU value of one of the coals utilized decreased from ~6,200 to ~1,200 – evidence of dramatic change to the material. This oxidation was expected and was not likely to affect the believed REE-coal bonds (as these are already oxidized bonds and are reasonably stable), but were found to have changed with the CM-lignite bonds affected greatly by this weathering. While not in the scope of this effort, other fresh coals have been tested in the pilot system and show comparable or improved performance as compared with the bench-scale extractive efforts, an expected result of the equipment and design choices made in the pilot system.

10 MREC EVALUATION FOR REFINING CAPABILITY

Rare Earth Salts (RES) received samples from UND at various times over the course of the past year and a half. The samples ranged in size from 0.25 g to approximately 2 kg. Samples for analysis were prepared using Trace Metal Grade acids (nitric and/or hydrochloric) and Type I water (18 Mohm). Samples were analyzed utilizing an Agilent 5800 ICP-OES and standards purchased from Inorganic Ventures that were brought to concentration using sequential dilution.

The first samples of bench-scale concentrates received ranged from 0.25 g to 1.0 g. These samples were from benchtop experiments and were used to give RES an idea of the MREC composition that was expected to be received, so that RES could begin testing process flows on similar systems. The results (Table 17) are presented as both percentage of rare earths versus total metal content and percentage of rare earths versus rare earths plus calcium content (on a metals basis). Sulfur content was also analyzed, but was not an issue in the initial samples. Table 18 lists the average composition of the samples (minus Sc, which was not analyzed for these samples) along with the standard deviation.

Table 17. Results for Initial Benchtop Samples Received at Rare Earth Salts (RES)

Sample	% RE/total metals	% RE/(RE+Ca)	Other major impurities
REE 1.0S.2	3.4	3.7	
ox1 solid 4/13/21	11.0	12.3	Ca (2%)
REE 6.0S.1	56.8	91.8	Ca (14%)
REE 6.1S.2 REEPBPT	3.5	4.4	Fe (2%), Ca (1%)
REE 5.0S.1	54.5	72.5	Na (6%), Ca (18%)
REE 7.0S.1	59.6	94.8	Na (6%), Ca (20%)
ox2 solid 4/13/21	0.5	0.6	Na (6%)

Table 18. Average Composition (%) of the Rare Earth Elements in the Initial Samples

Element	Average Percentage	Standard Deviation
La	12.1	6.2
Ce	32.7	4.7
Pr	3.1	2.1
Nd	18.9	6.6
Sm	2.6	3.2
Eu	1.1	0.7
Gd	4.3	1.8
Tb	0.9	0.4
Dy	3.0	1.2

Ho	0.8	0.2
Er	1.1	0.7
Tm	0.4	0.2
Yb	1.5	0.2
Lu	0.2	0.1
Y	16.6	3.1

Later testing of UND's pilot produced samples showed an initial variable REE concentration produced from concentrate 1, which was able to stabilize and reach refinable levels in June, 2024 (Figure 47). Later concentrates (particularly runs 11-15, representing the week of June 10th, 2024) showed promise for refining and were deemed acceptable for processing using RES's existing processes for refining of the REEs. Separated REEs from a blend of these concentrates was produced during the project.

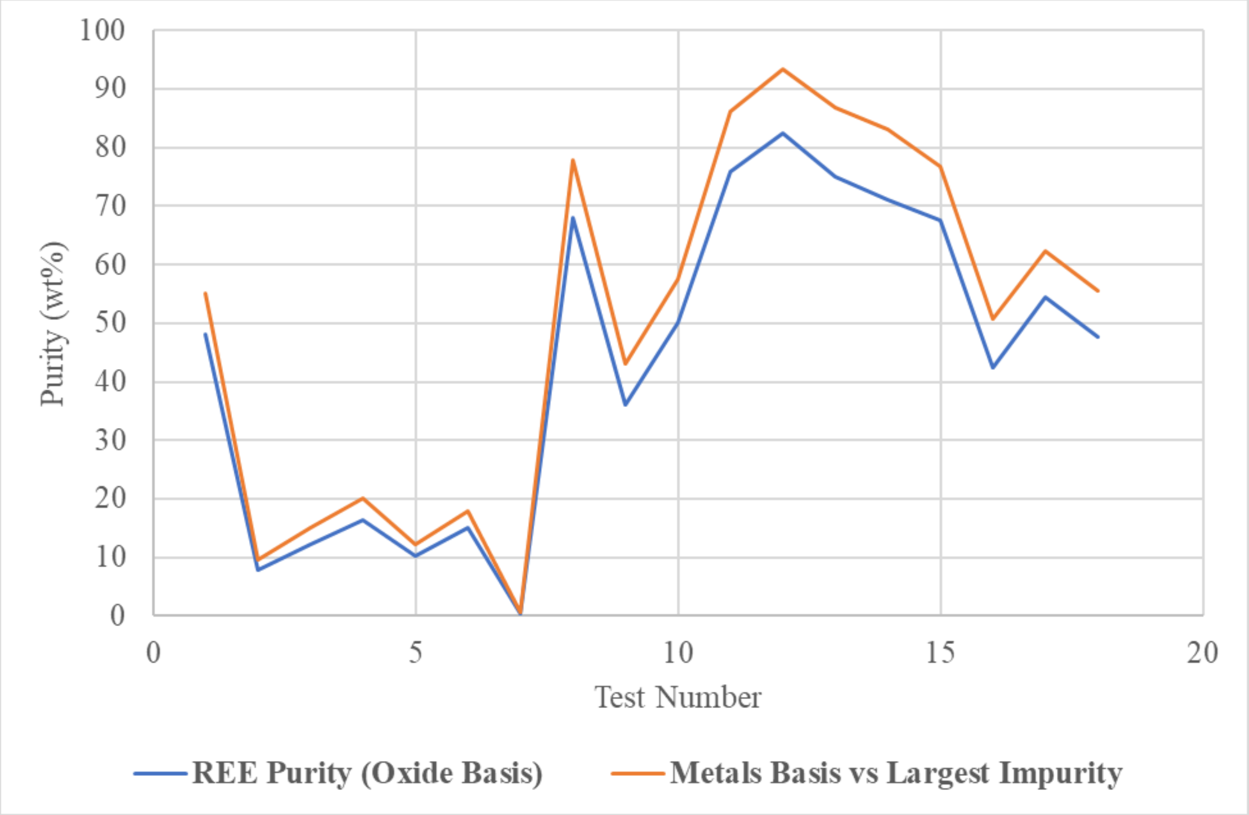

Figure 47. Produced concentrate 1 purity delivered to RES.

RES had access to a mineral concentrate that contained more La and less Y than the provided samples, along with lower concentrations of the heavy elements (beyond Gd). While not containing as high a concentration of the heavy rare earths, it allowed RES to test and verify that our process was feasible for such systems. RES was able to produce purified La, Ce, and didymium (NdPr) from this alternate concentrate at amounts up to hundreds of kilograms.

11 PROCESS EVALUATIONS

11.1 Techno-Economic Analysis

The TEA produced previously during DE-FE-27006 and 89243320RFE000032 was updated and refined utilizing the feedstock chosen for this pilot project and the updated results from the pilot testing. Main components included in the TEA are capital expenses, operating expenses, payable revenue, and payback analysis. The overall target of the TEA is to determine the required upgraded coal (post-processing) sale price in order to result in a net-present value (NPV) of $0 after 10 years. This TEA does utilize the weathered coal testing from the pilot as the basis, and is not expected to be fully representative of a final commercial demonstration case, but is included to acknowledge the exact pilot testing data produced. A discussion of the alternative case using the fresh feedstocks that underwent bench testing within 3 months of extraction is contained below in Section 11.1.3.

11.1.1 Base Case, Additional Scenarios, and Sensitivity Analysis

11.1.1.1 Base Case

The TEA analyzed a base case scenario including the following process considerations:

1. Coal crushing
2. Acid leaching
3. Leached coal filtering
4. Coal washing
5. Washed coal dewatering
   a. Filter Press
   b. Drying
6. Impurity removal (iron)
7. Iron filtering
8. REE precipitation
9. REE filtering

The Base Case CAPEX was generated using a developed equipment list from preliminary process flow diagrams. The OPEX was generated assuming operating and maintenance rates for each equipment item and using the H Bed and Freedom blend bench-scale and pilot-scale testing results and a dry coal mass flow rate of 110,230 lb/hr.

A payable revenue for the REE oxides and select target elements was estimated using published market values. Additional payable revenue is generated from the sale of the upgraded lignite coal. The payback analysis uses each of these components to estimate a simple payback and an IRR, ROI and NPV over 10 and 20 years.

11.1.1.2 Additional Scenarios

In addition to the base case described above, several other cases were considered to determine the impacts of various factors on the economics of the plant. These additional cases included:

- Base Case with total CAPEX decreased by 50%, simulating government (Federal or Local) investment and assistance

- Base Case with updated CAPEX assuming deep well injection for wastewater disposal
  - The CAPEX for the deep well injection was based on very preliminary estimates and requires additional scoping before consideration of implementation at full scale.

- Base case with deep well injection and total CAPEX decreased by 50%, simulating government (Federal or Local) investment and assistance

11.1.1.3 Sensitivity Analysis

Sensitivity analysis was built into the TEA and ran for the base case and each of the various scenarios. The key parameters that were included in the analysis were:

- Base Case CAPEX increased by +/- 25%
  - Overall CAPEX increased/decreased by +/- 25% for all equipment
- Upgraded Coal Price increased by +/- 10%
- Increased REE and CM recovery by +/- 10%
  - Improvement in recovery of REE/target metals by +/- 10% based upon improving dewatering efficiency of coal
- REE Price increased by +/- 10%
  - Overall REE price increase by +/- 10% from utilized values
- Increased REE-processing OPEX cost by +/- 25%
  - Processing costs for REE extraction and concentrate production increased by +/- 25% (not associated activated carbon & REE refining costs)

The economic/sensitivity analysis indicates that the two most impactful metrics for profitability of the combined REE/carbon processing plant include the price of the upgraded lignite coal and the OPEX of the REE processing facility.

11.1.2 TEA Summary and Conclusions

As stated earlier in this section, a full summary of input parameters and payback table results can be found within the attached standalone TEA summary report found in Appendix A. According to this TEA, the project is profitable after 10 years when the sale price of upgraded coal is between \$133-176/dry ton. The following summarize the main conclusions from the TEA:

- The pilot-testing results indicate that a 93% REE concentrate (oxide basis) is achievable using a dilute mineral acid leach, but requires additional impurity removal (i.e., iron removal via pH adjustment)
- Approximately \$62.4 million in sales for upgraded lignite coal (110,230 lb/hr feed basis)
- REE/target element oxides and base metals also contribute a good portion in sales; approximately \$10.5 million

  - Assumes the final product is REE/target element oxides

- The 10-year and 20-year NPV is $0 million and $29.5 million respectively assuming 12% discount rate
- A mixture of the Rhame bed and Freedom mine can be profitable, however the Rhame bed with its higher REE content would lead to more profit at identical flows when operating as a standalone feedstock.

Within this analysis. the following were identified as the primary technical and economic drivers of the project.

- Technical drivers
    - Overall REE/valuable element recovery
    - REE content and distribution of elements in the feedstock
    - Complexity/number of processing steps
- Economic drivers
    - CAPEX/OPEX
    - Upgraded carbon sales price
    - REE/target element price and price volatility

11.1.3 Alternative Case Associated with Bench-Testing Data

As a note to the produced and discussed TEA – mass balances are based off of the final pilot testing data available at the end of the project. However, this pilot testing utilized coals significantly weathered due to their extraction more than 4 years prior to the final testing conducted. Based off of previous bench testing as compared with the pilot, efficiencies of extraction of these elements were significantly lower than found at the pilot scale, including some high-value elements extracted at below 3% efficiency, when extracted previously at the bench-scale at far higher efficiencies (see Section 9.2.2 for more discussion).

When a similar economic scenario is developed for the extraction results attained at the bench scale, a 10-year NPV breakeven coal sale price of ~$80 is required, a price fully attainable by sale of the coal into carbon processing and manufacturing markets rather than for combustion (sale price currently estimated at ~$120/ton for carbon processing purposes). In this case, a plant size based around matching an equivalent carbon manufacturing facility presents a plausible and economically feasible concept, and warrants further investigation upon testing of new, fresh materials in UND's pilot facility.

11.2 Workforce Readiness Plan

Previously completed under agreement DE-FE0031835, a workforce readiness plan was reanalyzed with consideration to results produced from the pilot plant test program described elsewhere in this report. Upon review, it was determined that the majority of the labor analysis could still be assumed to be valid.

In summary, the University of North Dakota (UND) Institute for Energy Studies (IES) developed a process layout for a full-scale commercial plant for its rare earth and critical material (RECM) extraction plant. A large-scale (50 to 100 ton/hr) stand-alone RECM extraction plant is expected to need a staff of 50 to 60 people to be fully functional. There is no specialized equipment in the commercial plant, and operation personnel with a normal level of training and/or experience can meet the needs of the plant. Energy is one of the top two industries in the State of North Dakota, with approximately 20,000 employees working directly in this industry sector. Another 30,000 are working in construction and other directly related industries. The North Dakota University System (NDUS), comprised of 11 institutions provides workforce development and career training at all levels in fields directly related to the energy industry, and representing the needs of a RECM plant. Therefore, it is anticipated that due to the small number of people required in relation to the existing large industry in the state, there will be no issues in meeting the workforce requirements to fully commercialize this technology. Total plant personnel for the plant were assumed to be spread across the following categories:

- Operations, including
    - Supervisors and operators
- Maintenance, including
    - Supervisors and general maintenance technicians
- Quality Control, including
    - Chemists and lab technicians
- Overhead, including
    - Management, administration, human resources, distribution and marketing, and engineering

12 RECOMMENDATIONS FOR FUTURE EFFORTS

Two main areas of future efforts were identified in the completion of this project: pilot modifications and technology validation/progression. For the technology progression, additional testing of *freshly mined* resources in the pilot configuration with an emphasis in evaluating steady-state performance of a variety of leachate recirculation schemes would be valuable in improving the cost-competitiveness and the final expected commercial conditions recommended for construction of a commercial demonstration. These include material handling testing (crushing, spiralizing, filtration, etc.) as well as chemical process testing. Of key importance in the chemical process testing would be methods of increasing the selectivity of the REE precipitations steps, thus reducing the oxalate consumed by Ca as well as improving purities, possibly to points of avoiding any subsequent purification until refining. Methods including dopant testing of seed particulate of REE oxalate crystals (a different crystal structure to that of Ca oxalate) as well as effects of temperature or ionic strength would be employed.

A number of modifications to the final system tested during this project were identified for improving the throughput, operability, and a more accurate representation of the envisioned process in separate ways.

In improving throughput of the pilot, two main bottlenecks exist in the pilot processing of UND's pilot system, leaching throughput and impurity removal solid separation efficiency. To improve the leaching throughput, the only modification anticipated to be required would be a larger holding tank for the leaching and/or filtration preparation, as the current tank was not designed for a full filling of one of the 50 cu-ft filter presses present on-site. Due to this, artificial shrinkage of the filter press was required to develop adequately de-watered cakes – something that could be avoided with a larger tank size. This would not expect to require significant layout modification or pump changes for this. For impurity removal solid separation, laboratory tests of moderate-to-high temperature precipitation of this material was shown to develop far larger and denser particulate, enabling both rapid settling and improved filtration performance. For the pilot, this would involve replacing the existing plastic R-301 with a stainless steel, heated tank (likely electric immersion heaters), combined with a heat exchanger for cooling the post-reaction fluids and pre-heating fluids entering the tank.

Operability of the plant would be improved with the previous changes (reduces frequency of filter press exchange, and reduces challenges associated with iron precipitate contamination in downstream oxalate processing tanks), and could also be improved through additional automation, particularly surrounding the column(s) for REE cleaning and wastewater treatment (columns a largely manually operated as of now for volumetric switching, flow checks, sampling, and others). Additionally, improving the reliability of installed pH probes (either through automated cleaning and calibration cycles or through more resilient probes) would enable a more robust and less manually-intense operation.

Finally, the overall process (not pilot) is designed around a detailed coal washing regimen to both capture contained REEs and acid, as well as wash the coal thoroughly. With the installed equipment, only one of these (recovery or cleaning) is achievable as of now, and with significant manual requirements to conduct. Amending the filter press plates to a plate more easily able to be pressurized multiple times with a cake present (such as membrane-based plates) would enable this washing, or to switch a belt-filter style filtration with in-line washing. This change would both reduce the effort required to generate cleaned coal from the pilot, as well as be able to fully integrate the coal washing regimen into the pilot processing plant.

Appendix A. Technical and Economic Analysis

TECHNICAL AND ECONOMIC FEASIBILITY STUDY

SUBMITTED TO

U.S. Department of Energy
National Energy Technology Laboratory

SUBMITTED UNDER FUNDING OPPORTUNITY ANNOUNCEMENT

DE-FOA-0002003

RARE EARTH ELEMENT EXTRACTION AND CONCENTRATION AT PILOT-SCALE FROM NORTH DAKOTA COAL-RELATED FEEDSTOCKS

PRINCIPAL INVESTIGATOR

Nolan Theaker
Phone:
Fax: (701)777-6298
Nolan.theaker@und.edu

Date Submitted: September 29th, 2024

DUNS # 10-228-0781

SUBMITTED BY

Center for Process Engineering Research
College of Engineering and Mines Research Institute
University of North Dakota
2844 Campus Road, Stop 8153
Grand Forks, ND 58202-8153

WORK PERFORMED UNDER AGREEMENT

DE-FE0031835

Signature of Submitting Official: Nolan Theaker
Nolan Theaker

Rare Earth Element Extraction and Concentration at Pilot-Scale from North Dakota Coal-Related Resources

Technical and Economic Feasibility Study

Prepared for
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September 29th, 2024

3128 14th Avenue E
Hibbing, MN 55746
218.262.8600
www.barr.com

Rare Earth Element Extraction and Concentration at Pilot-Scale from North Dakota Coal-Related Resources

Technical and Economic Feasibility Study

Contents

List of Tables

List of Figures

Acronyms

Acronym	**Description**
AACE	Association for the Advancement of Cost Engineering International
AC	Activated Carbon
AOI	Area of Interest
Barr	Barr Engineering Co.
CAPEX	Capital Expenses
DOE	Department of Energy
FOA	Funding Opportunity Announcement
HA	Humic Acid
IRR	Internal Rate of Return
NDSU	North Dakota State University
NDUS	North Dakota University System
OPEX	Operating Expenses
PFD	Process Flow Diagram
PLS	Pregnant Leach Solution
PPMW	Parts per Million Weight
REE	Rare Earth Element
RES	Rare Earth Salts
ROI	Return on Investment
TEA	Technical Economic Analysis
UND	University of North Dakota

I. Executive Summary

The University of North Dakota (UND) has teamed with Barr Engineering Co. (Barr), Microbeam Technologies Inc. (MTI), Rare Earth Salts (RES), and MLJ Consulting to determine the technical and economic feasibility of extracting and concentrating rare earth elements from North Dakota lignite coal-related feedstocks, for a project submitted under US Department of Energy Funding Opportunity Announcement DE-FOA-0002003 in Area of Interest (AOI) 2. The project is supported by cost-share partners North American Coal Corporation, Great River Energy, Minnkota Power Cooperative, BNI Energy, MTI, UND, and the North Dakota Industrial Commission/Lignite Research Council.

The work described in this report identifies conditions for an economically viable and environmentally benign process to produce an REE concentrate at a stand-alone REE processing facility. The effort includes feedstock identification and procurement, construction and testing of a pilot-scale system, separation and concentration methods development and testing, production of an REE concentrate product, and technical and economic feasibility evaluations of the complete system. Background and introductory information can be found in Section 1.0 of this report.

Section 2.0 of this report summarizes the following considerations regarding the design basis and approach:

Table EX-1. Summary of the REE Extraction process modeled within this TEA.

Feedstock	**Rhame (H-bed) and Freedom Mine, Rider Seam (blend)**
Plant Size	Upgraded Coal-110,230 lb./hr. (50 tonne/hr)
Lignite Feedstock REE concentration	• 300 ppm REE+Y and Sc, (dry coal basis), 443 ppm REE+Y and Sc, (ash basis)
Process and Concentrating Sequence	• **Crushing -** (to -4 mesh) • **Spiraling –** Remove high ash material • **Leaching -** (dilute mineral acid) • **Separate** – Residual coal for Activated Carbon or Humic Acid Processing • **Remove Fe Impurity** – pH Adjustment • **Recover REE –** Selective Precipitation (oxalic acid) • **Transport** REE concentrate to processor/buyer
Concentration Results (dry solid oxide basis)	**Primary – 54 wt% REE oxide** **Secondary – 7.5 wt% REE oxide**
Environmental Considerations	• Ambient temperature and mild leaching solution w/ zero acid discharge methodology • Waste heat used to reduce energy of drying materials.

Section 3.0 - Process Design and PFDs: This section describes the systems to be considered for full scale production. Section 4.0 discusses the evaluation of the mass and energy balances of the total system used in the economic evaluation. Section 5.0 discusses the technical and economic evaluation results. These Class 4 estimate results are based on mining and minerals processing industries and are summarized below:

Table EX-2. Summarized Base Case Economics

Item	Base Case
Coal Feed Rate (dry lbs/hr)	110,230
CAPEX	($105,463,282)
OPEX	($45,900,000)
REE and Base Metal Payable Amount per year	$10,500,000
Upgraded Coal Payable Amount	$63,700,000
Net Annual Revenue per year	$18,700,000
Simple Payback (years)	5.6
IRR (10 years)	12%
ROI (10 years)	8%
NPV (10 years) @12% discount rate	$0
IRR (20 years)	17%
ROI (20 years)	13%
NPV (20 years) @12% discount rate	$34,000,000
CAPEX/annual dry ton feed	$200
OPEX/annual dry ton feed	$100
Net Revenue/annual dry ton feed	$40
Coal Dry Ton Feed Purchase Price ($/ton)	$25
Upgraded Coal Sale Price to NPV $0 at 10 years ($/ton)	$180

1.0 Introduction

Background of Funding Opportunity Announcement DE-FOA-0002003

This project addresses the technical area of interest as defined by DOE in DE-FOA-0002003: AOI 2 – Production of REEs and CMs – Transitioning, Scale-up and Validation of Conventional Extraction/Separation Processes from Bench-Scale to Pilot-Scale Facility REE/CM Production.

The DOE program objective for this FOA is as follows:

- Further develop/scale bench-scale, conventional, REE recovery processes to produce REEs and CMs in pilot-scale facilities.
- Address process optimization and efficiency improvements for the design, construction, and operation of pilot-scale research extraction/separation facilities, for recovery of REEs and CMs.
- At a minimum, produce >2wt % (>20,000 ppm) REE and CM pre-concentrates, but also produce higher purity individual REOs and CMs if a facility has the ability to do so.
- Implement, test, and operate the REE/CM recovery and production technology in an environmentally benign manner.

The specific objectives of this project included: feedstock identification and procurement, construction and testing of a pilot-scale system, development/testing of concentrating methods, technical and economic evaluation of the concentrating process, production of an REE concentrate product, and development of a strategy for commercialization.

Project Team

The University of North Dakota (UND) has teamed with Barr Engineering Co. (Barr), Microbeam Technologies Inc. (MTI), Rare Earth Salts (RES), and MLJ Consulting to determine the technical and economic feasibility of extracting and concentrating rare earth elements from North Dakota lignite coal-related feedstocks. The project is supported by cost-share partners North American Coal Corporation, Great River Energy, Minnkota Power Cooperative, BNI Energy, MTI, UND, and the North Dakota Industrial Commission/Lignite Research Council.

1.1 Purpose

The overall goal of the project was to demonstrate at the pilot-scale, a high performance, economically viable, and environmentally benign technology to recover rare earth elements from local lignite coal or lignite-related feedstocks.

To meet the goal, the following are specific objectives:

- Design and construct a pilot-scale system for continuous REE extraction from ND coal feedstocks capable of a minimum 0.25 tons/hr feed rate of physically beneficiated lignite coal based on previous laboratory and bench scale testing at UND.
- Leverage prior REE project experience, obtain large sample (~300 tons) of high REE (>300 ppm following coal cleaning) from the Freedom mine in North Dakota for testing in the pilot-scale demonstration facility
- Conduction initial parametric testing of a sample of the Freedom lignite, leveraging bench-scale equipment from previous projects, to cost-effectively identify optimal operating conditions and aid in the design of the pilot-scale system
- Commission the pilot facility using selected high REE containing coals from various regions in ND.
- Conduct a continuous pilot-scale testing utilizing optimal conditions for Ree extraction and concentration on at least 100 tons of the >300 oon REE-containing Freedom lignite.
- Confirm compatibility of REE concentration generated during pilot-scale testing with commercial scale REE-refining
- Based on results from the pilot testing campaign utiliting the Freedom lignite, conduct a preliminary Front End Engineering Design (pre-Feed) study on a potential commercial facility, in which an economic feasibility study and workforce assessment will be contained.
- Work with industry partners to develop a technology roadmap and a commercial deployment plan.

1.2 Scope and Technical Basis

The scope and basis for the technical and economic feasibility analysis is derived from the results of testing and analysis conducted in this project. Sampling and analysis conducted in previous phases of this project indicated that the bulk of the REE in the lignite coals are organically associated, loosely bound to the clays, and in mineral grains less than 10 µm. A majority of the REE forms are present as weakly bonded and are extractable using a mild acid leaching process.

The UND REE extraction process involves a mild acid leach at ambient temperature of the pre-combustion coal to extract the REE, Y and Sc, and other valuable elements. The mild leach is made possible by the weak association of the REE within the lignite coals. The resulting residual coal has a lower ash content and higher heating value and can be beneficially utilized in other processes such as activated carbon manufacturing or humic acid production. The pregnant leach solution (PLS) is pH adjusted to remove impurities (i.e. iron) before the REE extraction step where REE's and critical minerals (CM) are precipitated using oxalic acid and sodium carbonate. The remaining PLS may then be processed to recover aluminum by base addition to the barren solution.

The overall concept for commercial implementation of this REE recovery process has several pathways that may combine the REE recovery with an additional value-added process, which utilizes the residual coal, such as activated carbon production co-located at a combined heat and power plant, or at a purified humic acid production facility. However, for the scope of this TEA, sale of the residual coal was the only case considered. The residual coal resulting from the leaching processes, with lower ash content/higher value than the feed coal, can be utilized in an array of value-added coal utilization processes.

For this TEA, the scope of economic modeling is major assumptions include:

- Mined coal from the Rhame (H-bed) and Freedom mine in North Dakota is purchased and blended (additional locations for selective mining have been identified, but blended coal is being used as the basis of this evaluation)
- Iron precipitates generated in the process is sold for the cost of transportation (no revenue or cost)
- Concentrated REE solid is sold to a commercial processor for final separation

The above concept has numerous benefits that are summarized as follows:

- Uses smaller quantity of fuel than large-scale power plant (i.e., 1100 MW Coal Creek Station), which will enable selective mining to prevent dilution of the REE content in the feed coal.
- Extraction of other valuable minerals such as cobalt, copper, gallium, germanium, manganese, and zinc.
- Technical, environmental and economic benefits of the REE extraction/concentration process include: (i) ambient temperature and mild leaching of the raw coal, (ii) minimal NORM extraction from the coal, (iii) minimal production of waste, and (iv) simple, highly effective and industrially proven processing steps. Additional details are in Section 2.4.

2.0 Design Basis/Approach

2.1 Feedstock Identification

The feedstock for the pilot testing was taken from the Freedom Mine in Beulah, ND owned and operated by project partner North American Coal Corporation (NACC). This site was selected due to the elevated levels of REEs and the availability to collect the quantities needed for testing. In total ~250 tons of 130-270 ppm REE coal was recovered from the mine-site. Additionally, to evaluate the potential for blended feed performance a 45 tons of high REE H-Bed coal from the Harmon-Hansen coal region of southwestern ND was collected. The final blend was 35/65 H-bed to Freedom coal to reach the project threshold of 300 ppm, with 122 tons available for pilot testing.

2.2 Concentrating Methods

Figure 2-1 displays a simplified block flow diagram of the concentration process. Due to unique association of REEs with ND lignite coals, a mild leaching process has been developed that extracts REEs directly from the raw coal with recovery efficiencies of about 35 to 50% for the REE and Y and about 28% for Sc. The overall process is summarized as follows:

- Spirals are used to remove the mineral fraction from the coal
- The coal is then slurried with dilute mineral acid while maintaining pH to extract REEs
- The coal is washed to remove residual acid
- The final residual coal (with reduced inorganic content) can be dewatered and utilized as fuel or feedstock
- The pregnant leach solution (PLS) containing the REEs is purified to remove iron via sodium carbonate addition to adjust the pH
- REE extracted from solution by mixing the PLS with oxalic acid and sodium carbonate
- The final REE concentrates are sent for separation at an offsite refinery

Mass balances used in this evaluation are based on bench-scale and pilot-scale testing performed at UND with the following set of conditions:

- Blended coal is from the Freedom mine and H Bed seam with known REE and moisture content
- Liquid to dry coal ratio of 2:1 for leaching with dilute mineral acid
- Coal/leaching solution stirred with residence time of 1 hour while maintaining the pH through addition of acid
- After filtering coal from the PLS, coal is washed with water in a 2:1 liquid to solid ratio
- Amount of sodium carbonate added for all steps based on maintenance of a specific pH value

- A 90-minute residence time during impurities removal step, to maximize precipitation of iron
- REE precipitation performed by adding oxalic acid in a specific ratio before sodium carbonate addition to increase pH
- Residence time of 2 hours for REE precipitation
- All solids filtered, washed, dried, and analyzed for REE, other target elements and impurities content
- All liquids filtered and analyzed for REE, other target elements and impurities content
- Mass balances determined based on analysis results from liquid streams and solid products

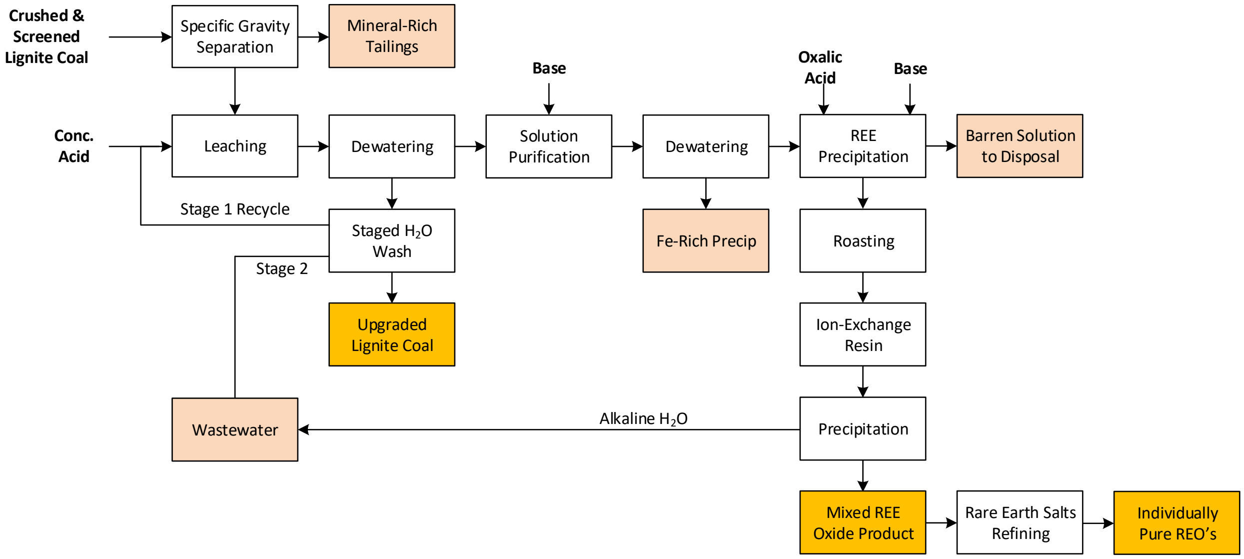

Figure 2-1 Simplified diagram of the REE concentrating process

Figure 2-1 includes the simplified diagram of the REE concentrating process evaluated in the TEA. The concentrated REE product is sent for final separation to the RES refining. For this study, it was assumed a commercial buyer would purchase the concentrated REE product.

2.3 Development / Testing

Initial parametric testing was completed on the bench-scale system with the Freedom coal to optimize the process conditions prior to the pilot-testing startup. Each of the process unit operations were evaluated over a range of conditions to optimize performance. This testing was completed sequentially such that the first unit operation was optimized prior to testing of the next. Process conditions used during production testing were chosen based on the following:

- The pH for leaching chosen to achieve maximum REE extraction from the coal with the least amount of acid to minimize processing costs

- Leaching residence time based on laboratory kinetics testing results which showed majority of REE extraction from the coal occurred in within the first 30 to 40 minutes
- The pH for the impurity removal step was chosen to maximize iron removal and minimize REE loss
- Oxalic acid concentrations chosen to maximize purity of REE in the primary product and maximize recovery of REE in the secondary product
    - This was monitored by TESS sensors to evaluate the completion and kinetics of the REE product precipitation

Assumptions for the TEA

- Process conditions and residence times will be the same as during pilot-scale testing
- Mass balance data used for the TEA is based upon the highest fidelity (least experimental challenges) obtained in the pilot-scale testing.
- Assume size reduction of coal feedstock for leaching at -4 US mesh particle size; bench-scale tests performed using this size
- Ambient temperature (~25°C or room temperature) operation.

Baseline leaching data for the blended feedstock is listed in Table 2-1. This study will focus on the blended feedstock with a coal feed rate of 110,230 lb/hr for the upgraded lignite coal production. This production rate was based on the sizing of available feedstock at the mine-sites.

Table 2-1 Total Recovery into Product Results of the H-Bed/Freedom Lignite Testing

H Bed Feedstock		
Element	**Initial ppm**	**Percent Recovered (based on total dry coal feed)**
Sc	13.4	22.79%
Y	46.8	33.43%
La	37.4	46.4%
Ce	90.5	40.18%
Pr	12.0	46.05%
Nd	51.2	45.06%
Sm	11.3	47.5%
Eu	2.7	48.61%
Gd	11.2	39.99%
Tb	1.7	50.16%
Dy	9.5	48.73%

H Bed Feedstock		
Element	**Initial ppm**	**Percent Recovered (based on total dry coal feed)**
Ho	1.8	47.92%
Er	5.0	46.87%
Tm	0.7	46.32%
Yb	4.2	45.44%
Lu	0.6	50.19%
Co	13.5	55.88%
Ga	14.0	2.16%
Ge	14.4	10.15%
Th	14.3	14.20%
U	18.3	7.68%
Al	20200	0%
Fe	25600	0%
Ca	2450	0%

*Recoveries obtained from piloting data

2.4 Environmental Considerations

The environmental impact was considered during the approach of the system configuration. Many of the gases and byproduct solids are processed to be a saleable product thus reducing waste streams discharging from the plant. The following considerations are discussed to better understand how the proposed system was designed to optimize waste streams and energy usage to approach an environmentally benign process.

The weak association of REEs in ND lignite coal presents a unique opportunity that, based on a thorough literature review of the modes of occurrence of REEs in coal, is not likely possible for higher rank coal types (i.e., bituminous) or for most coal-related byproducts (i.e., roof/floor sediments or combustion flyash).[1] [2] [3] [4] [5] [6] The process is simple, environmentally benign, and low cost, and is based on industrially proven mineral extraction methods widely utilized (including at the Mountain Pass Mine USA and in China for REE recovery). Some of the advantages of this technology are summarized below:

- Mild leaching of the raw coal at low/ambient temperature – environmentally benign processing
    - ✓ Hard rock ores or coal flyash require highly concentrated (i.e., >80wt% acid) acid baking at high temperatures (200-300°C) to achieve reasonable extraction of the REE. This is expensive and makes environmentally friendly processing challenging.
- Minimal NORM extraction from the coal
    - ✓ Concentration of thorium and uranium in all streams and products in this REE recovery process significantly less than regulations for thorium and uranium in rare earth materials listed in Title 10 Code of Federal Regulations Part 40, Section 40.13 (c) (1) (vi)[7].
- Minimal production of waste
    - ✓ Water used to wash coal is recycled within the process and reused in the leaching step. Recycling of the wash water cuts down on acid costs and increases total REE recovery.

---

[1] Dai, S., Ren, D., Chou, C.-L., Finkelman, R.B., Seredin, V.V., Zhou, Y., Geochemistry of trace elements in Chinese coals: a review of abundances, genetic types, impacts on human health, and industrial utilization. International Journal of Coal Geology. doi:10.1016/j.coal.2011.02.003, 2011a.

[2] Dai, S., Wang, X., Zhou, Y., Hower, J.C., Li, D., Chen, W., Zhu, X., Chemical and mineralogical compositions of silicic, mafic, and alkali tonsteins in the late Permian coals from the Songzao Coalfield, Chongqing, Southwest China. Chemical Geology, 282, 29–44, 2011b

[3] Bouska, V., and Pesek, J., Quality parameters of lignite of the North Bohemian Basin in the Czech Republic in comparison with the world average lignite, International Journal of Coal Geology 40, 211–235, 1999.

[4] Hower, J.C., Granite, E.J., Mayfield, D.B., Lewis, A.S., and Finkelman, R.B., Notes on Contributions to the Science of Rare Earth Element Enrichment in Coal and Coal Combustion Byproducts, Minerals 2016, 6, 32; doi:10.3390/.

[5] Eskenazy, G.M., Rare earth elements in a sampled coal from the Pirin deposit, Bulgaria. Int. J. Coal Geol. 7, 301–314, 1987.

[6] Seredin, V.V. and Dai, S., Coal Deposits as potential alternative sources for lanthanides and yttrium, International Journal of Coal Geology, 94 (2012)87-93.

[7] Commission, U.S. Nuclear Regulatory. *Title 10 Code of Federal Regulations Part 40.*

    - ✓ The only waste streams produced in this process are the tailings from coal spiraling and the barren solution after the PLS has been processed.

- <u>REEs can be leached directly from the raw coal using simple, highly effective, industrially proven processes – a highly economical approach</u>

    - ✓ The resulting residual coal has decreased ash content, and thus a higher value, and can be utilized in an array of coal conversion processes to augment economics

    - ✓ Other valuable elements, such as germanium, gallium (and others) are extracted with high efficiency along with the REEs, and can be recovered/purified to improve economics

    - ✓ Silica impurity is not extracted with the REEs, a significant benefit because it is known to cause technical issues for purification processes

2.4.1 Acid Gases from Leachate Process

Acid gases from the leaching process are anticipated. A scrubber has been considered in the capital cost that will remove the acid vapors from the gases. Water can scrub the acid gas and form acid that can be reused back into the acid leaching system. No additional environmental equipment is anticipated during the leaching process. To the extent possible, zero acid discharge methodology has been included.

2.4.2 PLS Processing

Addition of sodium carbonate during processing of PLS for impurity removal, REE precipitation, and aluminum recovery is anticipated to off-gas CO_2. The CO_2 can be combined with the gases from the power plant before being sent to the CO_2 capture system.

3.0 Process Design

This section presents the process design for achieving the REE concentrate. This design is based on bench-scale and pilot-scale testing results.

3.1 Coal Preparation

It is assumed that the coal 'as received' is large in size and will require crushing and pulverizing to achieve an acceptable particle size for leaching and further processing. The crusher and pulverizer will reduce the coal to about 4 US mesh (4.76 mm) prior to the spiraling process. The coal will then be sent to the spirals as a density separation process. The high density, high ash material will be separated from the coal and the overall ash content of the coal will be reduced. The following list summarizes the major pieces of equipment:

- Raw Coal Storage
- Primary Crusher & Coal Bin
- Coal Pulverizer & Fine Coal Feed Bin
- Dust Collection Baghouse
- Primary Spirals
- Scavenger Spirals

3.2 Leaching

After spiraling, the coal is conveyed to the acid leaching tank where it is mixed with a dilute mineral acid for a residence time of approximately 1 hour. Additional concentrated acid is added to the tank to maintain the pH of the mixture. The pH value is chosen based on achieving the maximum amount of leaching of the REEs with the least amount of acid to minimize processing cost.

After leaching, the slurry is sent to a filter press where the coal is collected and the PLS is separated from the coal. The coal is washed with water to remove any residual acid from the coal and to collect REE remaining in the pore water of the coal within the prior filter press (without discharging). This water is then recycled into the leaching process to reduce acid consumption and improve REE recovery. The washed coal is dried prior to any additional processing, which is discussed further in Sections 3.4. The following list summarizes the major pieces of equipment:

- Acid leach tank
- Acid scrubber
- Leached coal filter press
- Primary coal wash tank
- Primary coal wash filter press

- Secondary coal wash tank
- Secondary coal wash filter press
- Rotary kiln dryer

3.3 Impurity Removal

After the PLS is separated from the coal, it is sent to the iron precipitation unit where the pH is adjusted using sodium carbonate to precipitate the iron and other impurities. The solution reports to the clarifier to drop out the iron solids. The solids are periodically removed with a sludge pump and sent to a filter dewatering step where the iron is separated, and the filtrate retained for further processing. The following list summarizes the major pieces of equipment:

- Iron precipitation tank
- Clarifier
- Iron byproduct storage bin

3.4 REE Precipitation

The REE PLS is then sent to the REE precipitation unit where oxalic acid is added along with sodium carbonate to adjust the pH and precipitate REE. The solution is pumped to a filter press where the REE solids will be separated from the liquid. This REE precipitation step can be repeated to produce a secondary REE product. The purpose of the primary and secondary REE precipitation step is to produce a primary product, which is higher in purity of REE, and a secondary product, which has slightly lower REE concentration, but which allows for the extraction of nearly all of the REE from the PLS.

From the filter press, the REE solids are roasted to form rare earth oxides before being sent to a tank where they are mixed with water to dissolve calcium and increase the purity of the product. A filter press is used to separate the solids from the water. The REE product will be sent to an offsite to the RES site for processing and separation. The RES is not defined as part of this project. The following list summarizes the major pieces of equipment:

- REE precipitation tank
- Precipitated REE filter press
- Indirect rotary kiln
- Ca separation tank
- Purified REE filter press
- Final REE product storage bin

4.0 Mass and Energy Balances

Mass and energy balances in this section are based on the size of a 110,230 lb/hr (50 tonne/hr) dry-coal feed facility using ND lignite feedstock.

4.1 Leaching Requirements

As discussed in Section 2.0 Design Basis/Approach, a dilute mineral acid was selected for this process.

- Using 110,230 lb/hr of coal calls for approximately 71.4 ft^3/hr of concentrated mineral acid, which will be used to make the dilute acid based on scaled pilot system results. This may vary slightly as this step is pH controlled, and acid consumption may vary with changes in coal composition.

4.2 Iron Precipitation

As discussed in Section 2.0 Design Basis/Approach, iron is the major impurity causing the REE concentration of the leachate to be reduced. From bench-scale testing results, it has been found that nearly 80% Fe removal from the PLS can be achieved, with a loss of only 0 to 2% REE in the solution through pH adjustment by sodium carbonate addition.

- Based on scaling of bench system testing results, sodium carbonate will be fed at a rate of 7840 lb/hr. This may vary slightly with potential changes in PLS composition.

4.3 REE Concentration

REE concentration, both in the feedstock and in the final product, is a major factor in measuring the success and viability of the proposed system, as a higher concentration will decrease costs of downstream separation. According to DOE guidelines for this program, the REE concentrations in the feed need to exceed 300 ppm (whole sample basis) for feasible commercial deployment whether in ash or raw coal feed. H Bed and Freedom mine coal were blended at a 35/65 ratio in this stud which resulted in REE+Y and Sc concentration of about 300 ppm on a whole coal basis. As discussed in Section 2.0 Design Basis/Approach, oxalic acid and sodium carbonate are used to precipitate REE from solution.

- Oxalic acid will be added at a rate of 1740 lb/hr

- The feed rate for sodium carbonate will be approximately 1740 lb/hr. This rate may vary slightly with changes in PLS composition.

5.0 Technical and Economic Analysis

A Technical and Economic Analysis (TEA) for this phase was prepared and is described in the following sections. The TEA produced during DE-FE-27006, and 89243320RFE000032 was updated and refined utilizing the feedstock chosen for this pilot-project and the updated results from the pilot-testing. Main components included are capital expenses, operating expenses, payable revenue, and the payback analysis. The overall target of this TEA is to determine what the required upgraded coal (post-processing) sale price is to result in net-present value (NPV) of $0 after 10 years.

5.1 Technical and Economic Analysis Background

The TEA focuses on evaluating the cost of leaching, impurity removal steps, and extraction and concentration of REEs and other target elements from coal. In conducting the TEA, the principal process drivers considered include: material price, resource composition/ concentration, leaching efficiency, and operating and maintenance expenses.

The TEA accounts for the major variables affecting both capital (CAPEX) and operating (OPEX) expenses for mineral recovery and processing in conjunction with an offsite refinery to further process and separate the REE concentrate into the respective rare earth oxides and other target element products. Since there are no published data for polymetallic concentrates (as would be produced by our process), we evaluated the process on an end-to-end basis that accounted for all the costs to achieve saleable REE/element oxide products, including an offsite RES facility. By modeling the all-in costs to achieve saleable products, we do not need to know the sales price of the REE concentrate, which is the product of our processing plant. Instead, that value is wrapped into the larger calculation of net profit. We address the cost of the offsite refinery by assigning a processing cost per ton of element produced which includes the refiner's operating cost-plus profit. Since the refiner would process our concentrate on a contract basis, we have not included any capital expense for the refinery – it is assumed to be built and available for contract processing. This approach allows us to use published market prices for the REE/element oxide products to calculate gross revenue for the project, subtract refinery costs, and estimate the net profit of the recovery effort that is within our scope. This provides a net revenue stream (after accounting for internal OPEX) that can be used, along with the estimated extraction plant CAPEX to determine IRR, ROI, or other relevant economic parameters.

5.1.1 Market Impacts

The following section briefly discusses the market implications of introducing new sources of domestic production of REE+Y and Sc, as well as other metals that have a large impact on the economics of the proposed plant. Data is obtained from the 2024 USGS Mineral Commodities Report[8]. It is important to note that these discussions are based on production rates from the proposed plants. With installations at additional and larger facilities, impacts are likely to be different.

According to the USGS Mineral Commodities Report, total estimated consumption of REE+Y in the United States has decreased from 11,800 to 8,800 metric tons from 2019 to 2023. The U.S. is a net importer with about 8,800 metric tons imported in 2023. Total US sources produced 43,000 metric tons of REE mineral concentrate in 2023, which was a majority (40,000) was exported to China. Total domestic consumption of Yttrium was about 200 metric tons in 2023, all of which was imported. Approximately 65 tons per year of REE oxides are produced from the plant evaluated in this study after final purification. Due to the very small fraction of overall domestic consumption, besides reducing reliance on imports, it is not expected that any significant impact would result from introduction of this new domestic production source of REE.

Production of approximately 2.2 ton/year of scandium oxide for the plant, on the other hand, is likely to have an impact on the market, as according to the USGS Mineral Commodities Report, total *global* consumption of scandium was approximately 30 to 40 tons in 2023. Because the current market price of Scandium is very high, while the market consumption is very small, introduction of new resources for scandium, such as coal, is likely to impact the market price. However, upon increase of the supply, additional interest may be garnered from the auto and aerospace industries (for aluminum-scandium alloys) to expand the market utilization.

Another major contributor to sales from the proposed plant is germanium oxide, with a production rate of about 3.7 ton/year from the plant. According to the USGS Mineral Commodities Report, total imports for consumption of germanium was about 38 metric tons in 2023. The U.S. did export germanium at about 6.4 metric tons in 2023. Although not to the same extent as scandium, it is expected that germanium production from new coal resources would have an impact on the market. Currently used primarily in fiber and infrared optics (~50%), germanium also has use in solar cells, which may be a growth market in the event of larger domestic supply.

5.2 TEA Assumptions & Conditions

Our TEA makes the following assumptions/conditions:

1. The processing facility that receives the REE concentrate is located nearby to minimize shipping costs and/or the cost of shipping is built into the refiner's processing costs.

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[8] U.S. Geological Survey, 2024, Mineral commodity summaries 2024: U.S. Geological Survey, 212 p., https://doi.org/10.3133/mcs2024.

2. REE and other target element separation costs, including 20% refiner's profit for toll refiner, are estimated at present prices to be:
    a. $500 per dry ton of contained REE oxide (per element) – includes REE, Y, Sc, Ga, Ge
        i. Based on industry experience and considered conservative to reduce risk
        ii. Accounts for shipping cost assuming the refiner is not co-located
    b. $500 per dry ton of contained base metal (per element) – includes Co, Cu, Zn, Mn
        i. Based on industry experience and considered conservative to reduce risk
        ii. Accounts for shipping cost assuming the refiner is not co-located
3. Capital cost was calculated using cost estimate resources, equipment vendor quotes, and engineering judgement based on industry experience
4. Leaching/processing cost factors were estimated at:
    a. Raw coal cost = $25 per ton
        i. Includes mining of coal and shipping to facility via truck
    b. Electricity cost = $0.061 per kWh
    c. Natural gas cost = $3.50 per MMBtu
        i. Based on similar projects completed by Barr and typical natural gas cost in industry
    d. Water cost = $0 per 1000 gallons
        i. Water would be pulled from source at project site
    e. Mineral acid cost = $0.055 per lb (assumed 100% makeup rate)
        i. Used Intratec commodity prices
    f. Yearly operator cost
        i. Based on assuming 9 operators at $54,000 per shift
        ii. 25% fringe & overhead rate
        iii. Four shifts (24/7 operation)
    g. 5% added to overall process cost for miscellaneous items
        i. Typical factor for cost estimates at this level of detail – engineering judgement

The opinion of probable cost provided in this report is made on the basis of Barr's experience and qualifications and represents our best judgment as experienced and qualified professionals familiar with the project. The cost opinion is based on project-related information available to Barr at this time and includes a conceptual design of the project. The opinion of cost may change as more information becomes available, further design is completed, etc. In addition, since we have no control over the cost of labor, materials, equipment, or services furnished by others, or over the contractor's methods of determining prices, or over competitive bidding or market conditions, Barr cannot and does not guarantee that proposals, bids, or actual costs will not vary from the opinion of probable cost prepared by Barr. If greater assurance as to probable cost is required, Barr recommends to collect further information including pilot-scale testing results and complete further design to provide a higher accuracy cost estimate. Table 5-1 below shows the AACE Class 4 accuracy range and level of project to further clarify the boundaries of the cost estimate provided in this report.

Table 5-1 Description of Classification Values and Methods for Generic Cost Estimate

	Primary Characteristic	Secondary Characteristics			
	Level of Project Definition	End Usage	Methodology	Accuracy Range	Preparation Effort
Estimate Class	Expressed as % of complete project definition	Typical purpose of estimate	Typical estimating method	Typical +/- range relative to best range index of 1[a]	Typical degree of effort relative to least cost index of 1[b]
Class 5	0% to 2%	Screening or feasibility	Stochastic or judgment	10 to 20	1
Class 4	1% to 5%	Concept study or feasibility	Primarily stochastic	5 to 10	2 to 4
Class 3	10% to 40%	Budget, authorization or control	Mixed but primarily stochastic	3 to 6	3 to 10
Class 2	30% to 60%	Preliminary estimate	Primarily deterministic	2 to 3	5 to 20
Class 1	50% to 100%	Check estimate or engineer's estimate	Deterministic	1	10 to 100

Notes:
[a] If the range index value of "1" represents +10/-5%, then an index value of 10 represents +100/-50%.
[b] If the cost index value of "1" represents 0.005% of project cost, then an index value of 100 represents 0.5% of project cost.

Because the technology is still under development, some of the assumptions and numbers listed above will require refinement as the process matures through additional testing. However, where possible, we have incorporated cost data from similar processing systems in order to estimate the system CAPEX and some of the system OPEX.

5.3 Base Case TEA

The Base Case TEA includes the following process steps:

1. Coal crushing
2. Acid leaching

3. Leached coal filtering
4. Coal washing
5. Washed coal dewatering
    h. Filter Press
    i. Drying
6. Impurity removal (iron)
7. Iron filtering
8. REE precipitation
9. REE filtering

The Base Case CAPEX was generated using a developed equipment list from preliminary process flow diagrams. The OPEX was generated assuming operating and maintenance rates for each equipment item and using the H Bed and Freedom blend bench-scale and pilot-scale testing results and a dry coal mass flow rate of 110,230 lb/hr.

The Base Case TEA uses the results of the H Bed and Freedom coal blend bench-scale and pilot-scale testing as discussed in Section 2.3. Using those results, a payable revenue for the REE oxides and select target elements was estimated using published market values.[8] Additional payable revenue is generated from the sale of the upgraded lignite coal. The payback analysis uses each of these components to estimate a simple payback and an IRR, ROI and NPV over 10 and 20 years. These are all further discussed in the following sections.

5.3.1 Capital Expenses

The capital expense (CAPEX) for the proposed process is considered a Class 4 estimate, as described by the AACE International, Cost Estimate Classification System with an expected accuracy range of -15% to -30% (low) and +20% to +50% (high) (AACE, 2005).[9] The equipment was sized assuming a leaching processing rate of 110,230 dry lb/hr of coal.

The CAPEX estimate includes the following items:

- Raw Coal Storage Bin
- Belt Conveyors
- Primary Crusher
- Crushed Coal Storage Bin
- Coal Pulverizer

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[9] AACE International, 2005. Cost Estimate Classification System – As Applied in Engineering, Procurement, and Construction for the Process Industries, 2005, p. 1 – 9. http://www.aacei.org/toc/toc_18R-97.pdf

- Fine Coal Bin
- Dust Collection Baghouse
- Deslime Screen
- Primary, Scavenger Spirals
- Coal Feed Belt
- Acid Leach Tank, Mixer, Pump
- Acid Scrubber
- Filter Press (Leached Coal Dewatering)
- Primary Coal Wash Tank and Pump
- Filter Press (Primary Coal Wash Dewatering)
- Wash Water Recycling Storage Tank and Pump
- Secondary Coal Wash Tank and Pump
- Leached Coal Dryer
- Upgraded Coal Storage Bin
- Iron Precipitation Tank, Mixer, Pump, Feeder
- Iron Clarifier and Pump
- Iron Product Storage Bin
- REE Precipitation Tank, Mixer, Pump, Feeders
- Filter Press (REE Solids Dewatering)
- Aluminum Precipitation Tank, Mixer, Pump, Feeders
- Filter Press (Aluminum Solids Dewatering)
- Aluminum Product Storage Bin
- Barren Liquor Solution Tank, Mixer, Pump
- Indirect Fired Rotary Kiln (REE Solids Roasting)
- Calcium Dissolution Tank, Mixer, Pump
- Final REE Product Storage Bin
- Building
- Ductwork and Piping
- Wastewater Treatment

The total estimated project cost for the base case plant is $105.5 million. This includes additional cost for installation, site work, and site electrical and controls as a percentage of the total equipment cost. Also, an engineering, legal, and administrative amount is included as a percentage of the total construction cost.

5.3.2 Operating Expenses

Operating Expenses (OPEX) include power, fuel, maintenance, raw material cost, and operator cost. The total power cost was estimated using an assumed power requirement for the equipment. The OPEX includes the following items:

- Coal Cost = $25 per ton, delivered cost
- Electricity, Natural Gas, and Maintenance Expense
- Steam Cost
- Water Cost
- Operator Cost
- Acid Cost
- REE Processing Cost
- Base Processing Cost
- Wastewater treatment
- Adder of 5% of total processing cost to account for miscellaneous items

Sewer, waste disposal, precipitation, additional unknown items

The total estimated operating cost is $45.9 million per year.

5.3.3 CAPEX, OPEX & Maintenance Expense Assumptions

The project team leveraged data from previous projects DE-FE-27006, and 89243320RFE000032 and scaled where applicable to the values that related to the plant size specified in this report.

Listed below are the assumptions specific to the CAPEX, OPEX, and Maintenance Expense portions of the TEA:

- Sources for capital equipment cost data:

Mine and Mill Equipment Costs, An Estimator's Guide", InfoMine USA Inc., 2019

Quotes for similar pieces of equipment obtained by Barr for other projects in the recent past (and updated to 2024 USD)

These were escalated or de-escalated using the 6/10 rule for different equipment sizes:

$$Final\ Cost = Cost\ from\ Quote * \left(\frac{Equipment\ Size\ from\ Quote}{Equipment\ Size\ for\ Final\ Cost}\right)^{0.6}$$

New budgetary quotes for certain pieces of equipment requested from vendors

- Operating and Maintenance costs for equipment were taken for similar pieces of equipment from Mine and Mill Equipment Costs, An Estimator's Guide", InfoMine USA Inc., 2019

Repair Labor $41.30 per hour
Lubricants $28.39 per gallon
Natural Gas $3.50 per MMBTU
20,000 Btu/lb

- Electric Power $0.061 per kWh
- Natural gas consumption rate for kilns is process maximum assuming no credit for recycled waste heat
- Tank sizing calculated based on residence times and flow rates
- All conveyors were assumed to be 100 feet in length, since there is no general arrangement for a study at this level of detail
- Dust collection requirements were assumed to be 10,900 cfm for fine coal dust

Based on available historical quote

- Primary Crusher is assumed to be a hammer mill type crusher
- Fine Coal Feed Bin assumed to be 30-ton capacity
- Raw Coal Storage assumed to be 200-ton capacity

5.3.4 Payable Revenue

For the Base Case process two payable revenue streams have been identified:

1. REEs and Base Metals
2. Upgraded thermal coal

REE and Base Metals

The market prices used assume the final sale product to be REE/target element oxides. The lab results are elements and not the oxides. To account for the total mass of oxides as the final product the molar mass of the oxide and the element was used to calculate the total pounds of each element. This calculation was completed within the detailed economic analysis and accounted for in the total lb/yr calculation and total payable revenue for the product oxides.

This study has evaluated the economics of producing/purifying all of the REEs, plus Y, Sc, Ga, Ge and select base metals (Co, Cu, Mn, Zn). However, in an actual application, it may be beneficial to target only specific REEs or base metals, namely the higher price/ critical REEs that provide highest incremental value above the refiner’s processing cost.

The coal blend projects a total of 139,000 lb/yr of REE/target element oxides and about 7,000 lb/yr of base metals assuming 100% recovery during the refiner processing (i.e., overall recovery on dry whole coal basis is: leaching extraction recovery X recovery from PLS X refining recovery).

The calculated value of the finished REE and other element oxides products produced at the refinery is deduced by assuming a refining cost per ton of REE/target element and base metal produced. These were set at $500 per ton of REE, Y, Sc, Ga, Ge (as oxide) and $500 per ton of base metal (Co, Cu, Mn, Zn). These costs are on a per element basis.

5.3.5 Payback Analysis

As stated previous the objective of this TEA was to determine what the saleable value of the upgraded lignite coal must be to reach a NPV of $0 after 10 years. Each cost and revenue component discussed in the previous sections is used to estimate ROI, IRR, and simple payback. Table 5-2 provides the analysis.

Table 5-2 Payback Analysis Summary

Item	Base Case
Coal feed rate (dry lbs/hr)	110,230
CAPEX	($105,463,282)
OPEX	($45,900,000)
REE and Base Metal Payable Amount per year	$10,500,000
Upgraded Coal Payable Amount	$63,700,000
Net Annual Revenue per year	$18,700,000
Simple Payback (years)	5.6
IRR (10 years)	12%
ROI (10 years)	8%
NPV (10 years) @12% discount rate	$0
IRR (20 years)	17%
ROI (20 years)	13%
NPV (20 years) @12% discount rate	$34,000,000
CAPEX/annual dry ton feed	$200
OPEX/annual dry ton feed	$100
Net Revenue/annual dry ton feed	$40
Coal Dry Ton Feed Purchase Price ($/ton)	$25
Upgraded Coal Sale Price to NPV $0 at 10 years ($/ton)	**$180**

5.4 Additional Cases/Scenarios

In addition to the base case described above, several other cases were considered to determine the impacts of various factors on the economics of the plant. These additional cases included:

- Base Case with total CAPEX decreased by 50%, simulating government (Federal or Local) investment and assistance
- Base Case with updated CAPEX assuming deep well injection for wastewater disposal

    - The CAPEX for the deep well injection was based on very preliminary estimates and requires additional scoping before consideration implementation at full scale.
- Base case with deep well injection and total CAPEX decreased by 50%, simulating government (Federal or Local) investment and assistance

The results of ease of these cases may be found in Table 5-3 below. The total required sale price of the coal to meet the NPV of $0 after 10 years ranges from $131-180.

Table 5-3 Payback table results for additional cases

Item	Base Case	Base Case 50% CAPEX	Case Well Injection	Case Well Injection 50% CAPEX
Coal feed rate (dry lbs/hr)	110,230	110,230	110,230	110,230
CAPEX	($105,463,282)	($52,731,641)	($96,509,259)	($48,254,629)
OPEX	($45,900,000)	($45,100,000)	($44,700,000)	($44,000,000)
REE and Base Metal Payable Amount per year	$10,500,000	$10,500,000	$10,500,000	$10,500,000
Upgraded Coal Payable Amount	$63,700,000	$48,700,000	$60,000,000	$46,400,000
Net Annual Revenue per year	$18,700,000	$9,300,000	$17,100,000	$8,500,000
Simple Payback (years)	5.6	5.6	5.7	5.6
IRR (10 years)	12%	12%	12%	12%
ROI (10 years)	8%	8%	8%	8%
NPV (10 years) @12% discount rate	$0	$0	$0	$0
IRR (20 years)	17%	17%	17%	17%
ROI (20 years)	13%	13%	13%	13%
NPV (20 years) @12% discount rate	$34,000,000	$17,000,000	$31,000,000	$15,600,000
CAPEX/annual dry ton feed	$200	$100	$200	$100
OPEX/annual dry ton feed	$100	$100	$100	$100
Net Revenue/annual dry ton feed	$40	$20	$37	$18
Coal Dry Ton Feed Purchase Price ($/ton)	$25	$25	$25	$25
Upgraded Coal Sale Price to NPV $0 at 10 years ($/ton)	**$180**	**$138**	**$170**	**$131**

5.5 Sensitivity Analysis

Sensitivity Analysis was built into the TEA and ran for the base case and each of the various scenarios. The key parameters that were included in the analysis were:

- Base Case CAPEX increased by +/- 25%
    - Overall CAPEX increased/decreased by +/- 25% for all equipment

- Upgraded Coal Price increased by +/- 10%

- Increased REE and CM recovery by +/- 10%
    - Improvement in recovery of REE/target metals by +/- 10% based upon improving dewatering efficiency of coal

- REE Price increased by +/- 10%
    - Overall REE price increase by +/- 10% from utilized values

- Increased REE-processing OPEX cost by +/- 25%
    - Processing costs for REE extraction and concentrate production increased by +/- 25% (not associated activated carbon & REE refining costs)

The economic/sensitivity analysis indicates that the two most impactful metrics for profitability of the combined REE/carbon processing plant include the price of the upgraded lignite coal and the OPEX of the REE processing facility.

6.0 Summary & Conclusions

According to this TEA the project is profitable after 10 years when the sale price of upgraded coal is between $131-180/dry ton. The following summarize the main conclusions from the TEA:

- The pilot-testing results indicate that a 54% REE concentrate (oxide basis) is achievable using a dilute mineral acid leach, but requires additional impurity removal (i.e., iron removal via pH adjustment)

Approximately $63.7 million in sales for upgraded lignite coal (110,230 lb/hr feed basis)

- REE/target element oxides and base metals also contribute a good portion in sales; approximately $10.5 million

Assumes the final product is REE/target element oxides

- The 10-year and 20-year NPV is $0 million and $34 million respectively assuming 12% discount rate
- A mixture of the H-bed and Freedom mine can be profitable, however the H-bed with its higher REE content would lead to more profit at identical flows when operating as a standalone feedstock.

Additional discussion regarding different scenarios and next phases of study are provided in the last few sections.

6.1 Discussion of Worst, Likely and Best-Case Scenarios

The technical and economic data presented in this report are developed based upon the bench-scale and pilot-scale data and quoted/discovered pricing for all elements and reagents, and are purposefully conservative, with the economics tending towards the 'low REE recovery' scenarios. However, additional 'likely' scenarios will be discussed below from a technical and economic standpoint to give insight into possible outcomes.

The technical and economic data presented in this report are purposefully conservative, and thus the economics are likely to be more towards the 'worst-case' scenario. However, based on the request from DOE, below we discuss these three scenarios both from a technical and economic standpoint to give indication of the spectrum of possible outcomes.

Technical Scenarios

The primary technical drivers dictating the success of the technology are: i) overall REE/valuable element recovery, ii) REE content and distribution of elements in the feedstock, and iii) complexity/number of processing steps. Each of these categories are discussed below.

REE Recovery Efficiency: Since the REE recovery efficiency presented in this study is based on bench-scale testing data performed by UND, we classify these results as 'likely'. As additional process optimization is accomplished in subsequent testing, there may be some tradeoffs between overall recovery and process simplicity. For instance, it may be economically beneficial to reduce leaching contact time to reduce equipment sizes or increase throughput, at the same time potentially reducing REE recovery. On the other hand, improvement of the leaching processes via a better understanding of the modes of occurrence of the REE in the feedstocks may improve overall REE recovery. It is also possible that the extraction could be optimized to best accommodate downstream separation processes (i.e., increase recovery efficiency from selective extraction steps). Increased recovery is also expected as dewatering efficiency is increased. REE recovery efficiency also depends on the feedstock chemistry. Extraction efficiencies of elements such as Scandium and Germanium have a large impact on overall economics as well because of their high market prices. We would classify the best and worst-case scenarios at ±10% recovery in the leaching step, making the worst-case scenario 10-40% recovery and the best-case scenario 30-60% recovery (variation of Table 2-1).

Increased Operating Costs: We have evaluated one coal type, with its impurity and REE distribution. Some lignites previously identified with REE > 300 ppm on a whole coal basis also contain significantly higher organic-based impurities (such as Fe and Al) which are anticipated to significantly increase operating costs of the REE plant (with the upgraded carbon plant buffered against these adjustments). The operating costs anticipated to increase include the acid costs (due to higher acid consumption), general base costs (higher precipitating material concentrations, such as Fe), and higher oxalic acid costs (due to increased aluminum complexation). Further, higher potential labor costs may be assumed in such a case, likely due to potential variability and control of the feedstock (requirement of higher-skilled labor). Additionally, this scenario may be used to define a lower REE-bearing coal (by 25%), with all costs reflected of this.

Complexity/Number of Processing Steps: This study has presented 13 main processing steps as outlined in Section 4.3 including the production of either activated carbon or humic acid byproducts. The processing steps involved in this REE extraction process are all simple, highly effective, and industrially proven. However, if higher purities/more impure feedstock are utilized, the complexity of generating higher-purity materials, including the addition of hydrometallurgical cleaning steps (such as solvent extraction or ion-exchange), would increase both capital and operating costs.

Economic Scenarios

The primary economic drivers are: (i) CAPEX/OPEX, (ii) upgraded carbon sales price, and (iii) REE/target element price. Each are discussed briefly below.

CAPEX/OPEX: This study has made conservative assumptions for capital and operating expenses to account for the early stage of technical development, and thus we believe the data presented to be the likely case, but erring slightly towards the worst-case. The main driver dictating capital and operating expenses is process complexity. Our sensitivity analyses have evaluated a ±25% change in both the CAPEX and OPEX. This includes evaluation of a more complex plant, lower REE purity, and/or additional extractable impurities feedstocks.

REE/Target Metal Sales Volatility: Sales prices for the REE/target elements are based on current market prices, and thus are considered the likely case. However, REEs have experienced a huge price range in recent years, with spikes from 2019 to 2024. Our sensitivity analyses have evaluated a ±25% fluctuation in the current prices to understand the impact.

Results

The data presented in this document can reasonably be assumed as the likely or average scenario, accounting for conservative assumptions. REE-specific best-case scenarios would involve: (i) improvement in REE recovery from the feedstock, (ii) increases in REE/target element sales prices, (iii) reduced process complexity through beneficial leaching, and (iv) increased upgraded carbon price. Worst-case scenarios would include: (i) reduction in REE/target element content in feedstocks, (ii) reductions in REE/target element recovery from the feedstock, (iii) increased process complexity, and (iv) reductions in overall REE/target element/upgraded carbon sales prices.

The worst-case scenario could be estimated as the following combined sensitivity case:

- Increase in OPEX by 25%
- Reduction in REE Recovery by 5%
- Decrease in REE/target element sales price by 10%
- Decrease in upgraded carbon sales price by 10%

When combining the above, worst-case economic metrics are as follows:

- 20-year IRR: -1%
- 20-year ROI: 0%
- Simple Payback: 21.2 years
- 20-year NPV (12% discount rate): ($85,300,000)

The best-case scenario could be estimated as the following combined sensitivity case:

- Increase in REE recovery from feedstock by 5%
- Increase in REE/target element sales price by 10%
- Decrease in CAPEX/OPEX by 10%
- Increase in upgraded carbon sales price by 10%

When combining the above, best-case economic metrics are as follows:

- 20-year IRR: 29%
- 20-year ROI: 34%
- Simple Payback: 2.54 years
- 20-year NPV (12% discount rate): $153,500,000