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

DE-FE0031835

Nolan Theaker

UNIVERSITY OF NORTH DAKOTA
LEADERS IN ACTION

Proposed Team

Project Team Members

- UND Institute for Energy Studies
- Microbeam Technologies
- Barr Engineering
- Rare Earth Salts
- MLJ Consulting
- NDGS

Project Sponsor Representatives/Executive Advisory Team

- DOE-NETL
- Lignite Research Program
- North American Coal
- Great River Energy
- Minnkota Power Cooperative
- BNI Energy
- Critical Materials Institute

Project Overview

- Budget – Overall $5,608,555

- Project Performance
  - BP1: 09/19 – 09/20
  - BP2: 10/20 – 06/23

Support Source	Funding	% of Project
DOE-NETL	$4,989,255	76.7%
NDIC	$900,000	13.8%
NACC	$125,000	1.9%
GRE	$125,000	1.9%
MPC	$125,000	1.9%
BNI	$120,000	1.8%
MTI	$34,300	0.5%
UND	$90,000	1.4%

Goals and Objectives

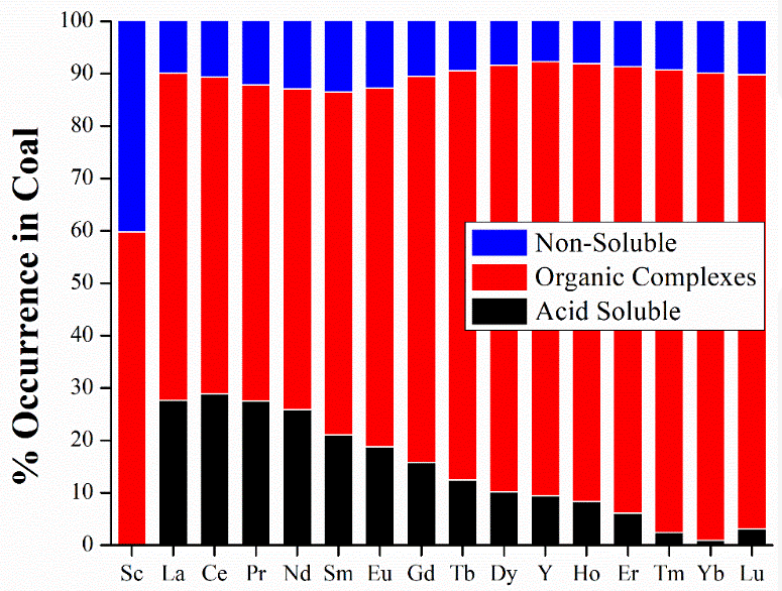
- Construct and test UND's extraction and concentration process at a 0.5 ton/hr scale
  - Develop a modular approach for ease of process adjustment and reconfiguration
  - Determine optimal equipment configurations for the process, including process intensification efforts
- Obtain and test a large sample of high-REE, ND lignite for REE extraction using the pilot facility
  - Determine scalability of the process, optimal operating points, and potential cost-reduction measures for commercial operation

Goals and Objectives (cont)

- Evaluate the economics of the UND processing technology
  - Utilize commercial quotes and pricing wherever possible, and define the scale/location of a REE extraction plant in ND
- Evaluate the technology and resource in a pre-feasibility and pre-FEED mechanism
  - Develop correlations for extraction, concentration, and potential economics based upon resource characterizations
- Develop a technology and commercialization roadmap and business plan
  - Include costs of commercialization phases, detailed business plans and structuring, and potential vendors/customers for the plant

Technology Background

- Extracts REE from low rank coals (LRCs) utilizing weak acids
  - Weak organic associations, rather than mineralized forms (carboxylic acid)
- Utilizes the *pre-combustion* coal for the feedstock
  - Generates a reduced-ash unique byproduct
    - Usable for many CBP
    - Low fouling ash for boilers



Lignite Chemistry

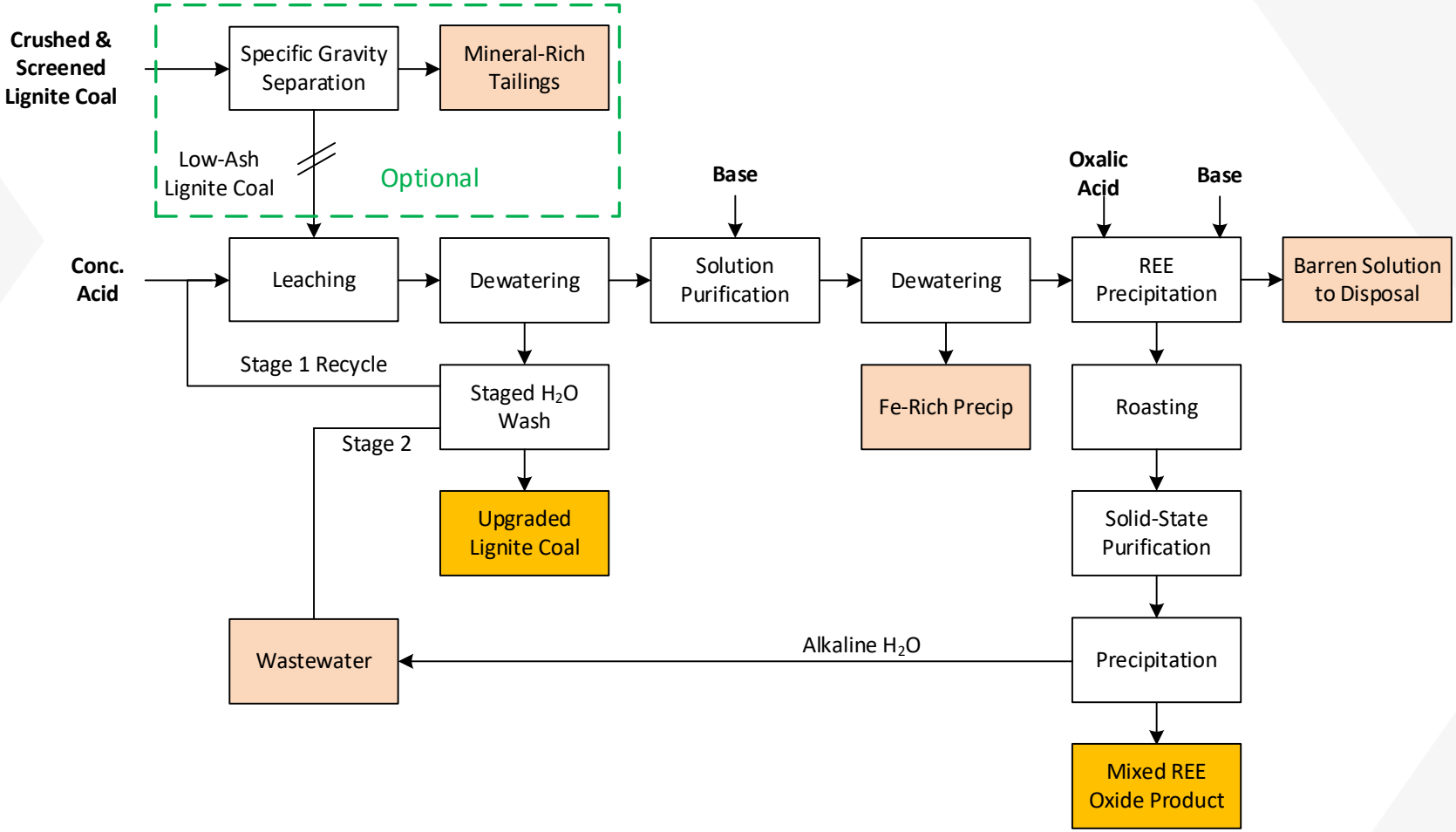
- Organic functional groups exist *in abundance* within the young lignite – terminal OH bonds
  - OH bonding allows for H atom replacement – with suitably acidic materials

			High volatile bituminous			Bituminous		
	Lignite	Subbitu-minous	C	B	A	Medium volatile	Low volatile	Anthracite
% C (min. matter free)	***65-72***	72-76	76-78	78-80	80-87	89	90	93
% H	***4.5***	5-4	5.5	5.5	5.5	4.5	3.5	2.5
% O	***30***	18	13	10	10-4	4-3	3	2
% O as COOH	***13-10***	***5-2***	***0***	***0***	***0***	***0***	***0***	***0***
% O as OH	***15-10***	12-10	9	?	7-3	2-1	1-0	0
Aromatic C atoms % of total C	***50***	65	?	?	75	80-85	85-90	90-95
Avg. no. benzene rings/layer	***1-2***	?			2-3		5?	>25?
Volatile matter (%)	***40-50***	35-50	35-45	?	31-40	31-20	20-10	<10
Reflectance (%) of vitrinite	***0.2-0.3***	0.3-0.4	0.5	0.6	0.6-1.0	1.4	1.8	4

Benefits of Organic Association

- No discrete minerals to dissolve
  - Extremely rapid kinetics, acid substitution process
- Selectivity of organic groups
  - Can purposefully leave higher valence elements attached to the organic structure
    - Very low NORM leaching
- Size reduction largely irrelevant for leaching
  - Lignite is highly porous – utilize pore structure for transport of acid/REE from groups

Flowchart


Crushed & Screened Lignite Coal
Specific Gravity Separation
Mineral-Rich Tailings
Low-Ash Lignite Coal
Optional
Conc. Acid
Leaching
Dewatering
Base
Solution Purification
Dewatering
Oxalic Acid
Base
REE Precipitation
Barren Solution to Disposal
Stage 1 Recycle
Staged H_2O Wash
Stage 2
Upgraded Lignite Coal
Fe-Rich Precip
Roasting
Solid-State Purification
Wastewater
Alkaline H_2O
Precipitation
Mixed REE Oxide Product

Challenges of Approach

- Pre-combustion material lower feedstock “grade” than coal ash
  - More feedstock to process = more material to slurry/process
- Lignite is not easy to handle
  - Wet feedstock (35-40% moisture)
  - Autoignition
- Wet process = even more moisture in product coal

Scope of Work

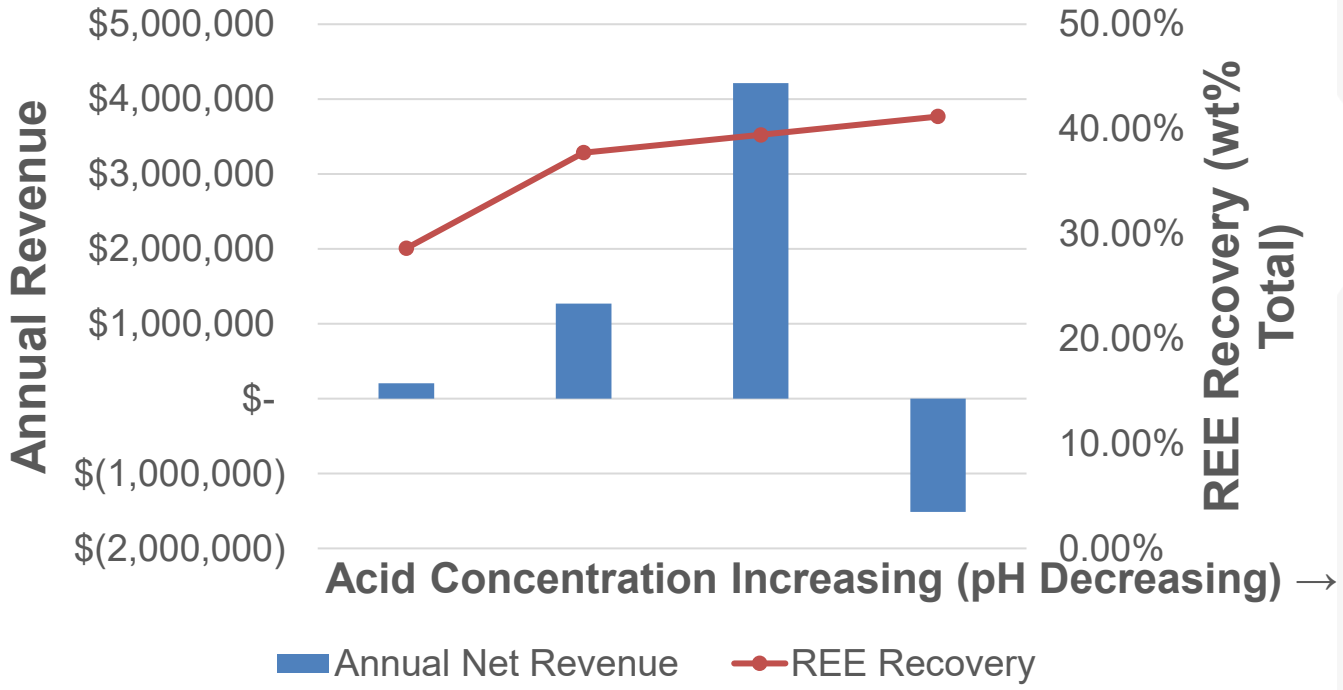
- Task 1 – Project Management & Planning
- Task 2 – Financial Plan for Commercialization
- Task 3 – Techno-Economic Assessment
- Task 4 – Split Sample Analysis
- Task 5 – Feasibility Study
- Task 6 – Large Sample Collection and Prep.
- Task 7 – Pilot Plant Design
- Task 8 – Pilot Plant Construction
- Task 9 – Bench-Scale Feedstock Testing
- Task 10 – Pilot Plant Testing of Feedstock

Processing – Bench-Scale

- Process steps to test:
  - Leaching
    - pH, slurry density, contact time
  - Impurity removal via base
    - pH, residence time, mixing speed, settling time
  - REE Precipitation
    - Oxalate concentration, mixing speed/time
  - Purification of concentrates
    - Resin type, loading/unloading curves

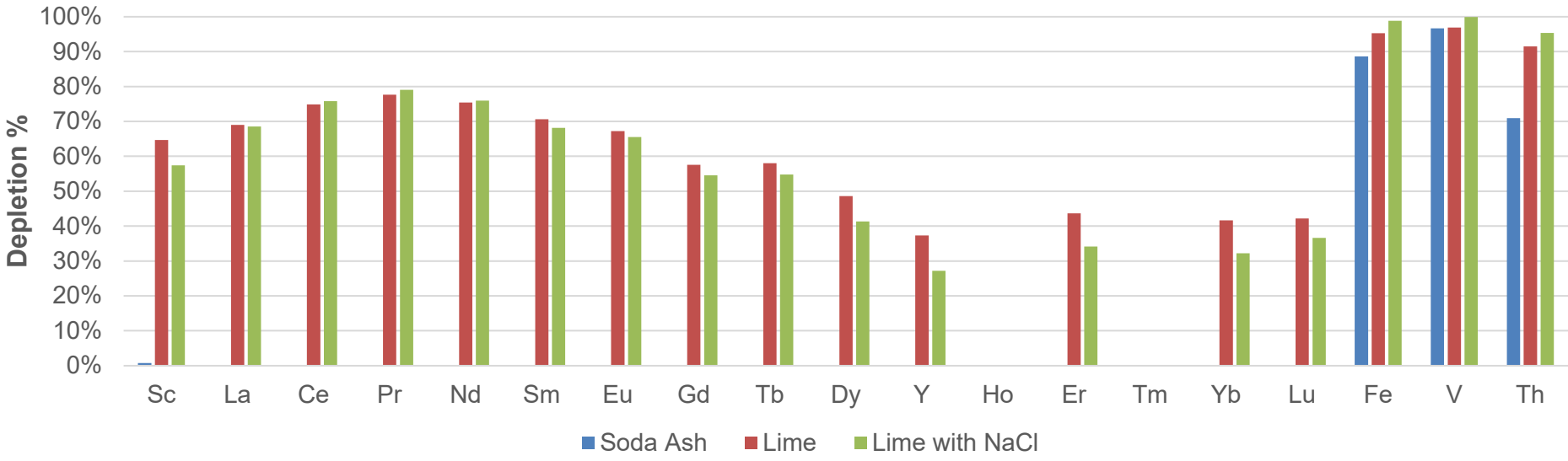
Bench-Scale: REE Leaching

- Tested REE/CM leaching at multiple held pH values
  - Total REE recovery from process, not leaching (including planned Ce removal)



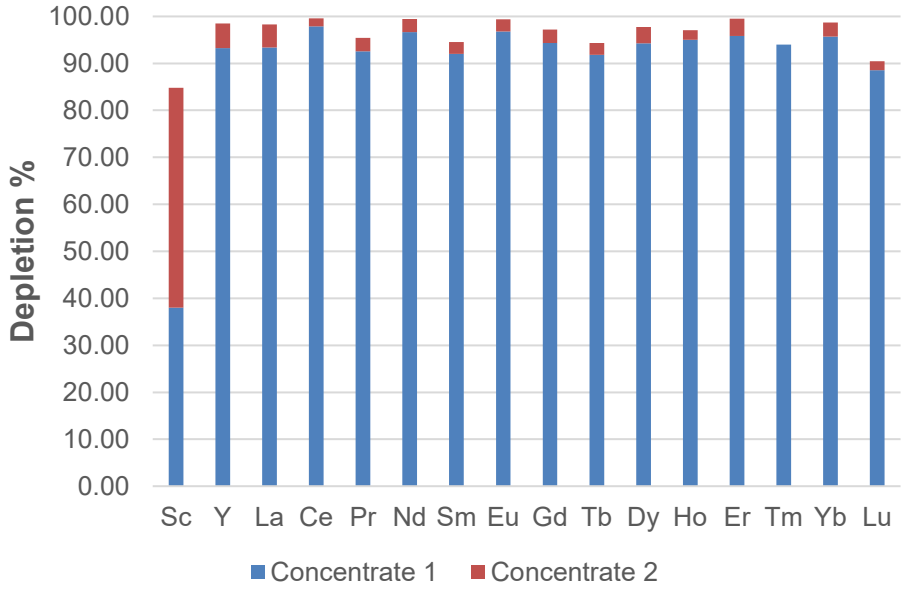
Bench-Scale: Impurity Removal

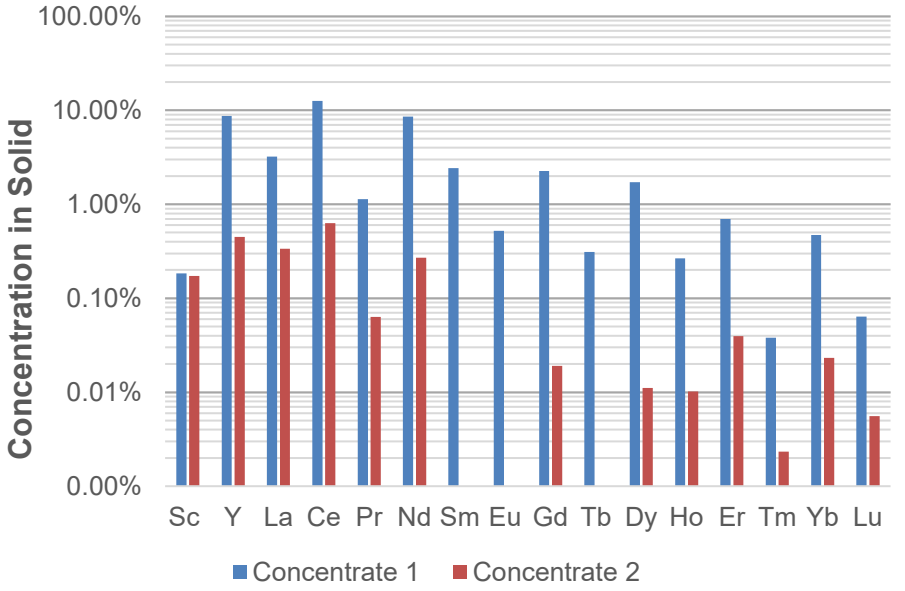
- Tested 3 base combinations at 2 pH values
  - pH 3.5 showed consistently best performance
- NaCl theorized to assist Fe/V precipitation
  - Poor REE retention, Na_2CO_3 chosen moving forward



Bench-Scale: REE Precipitates

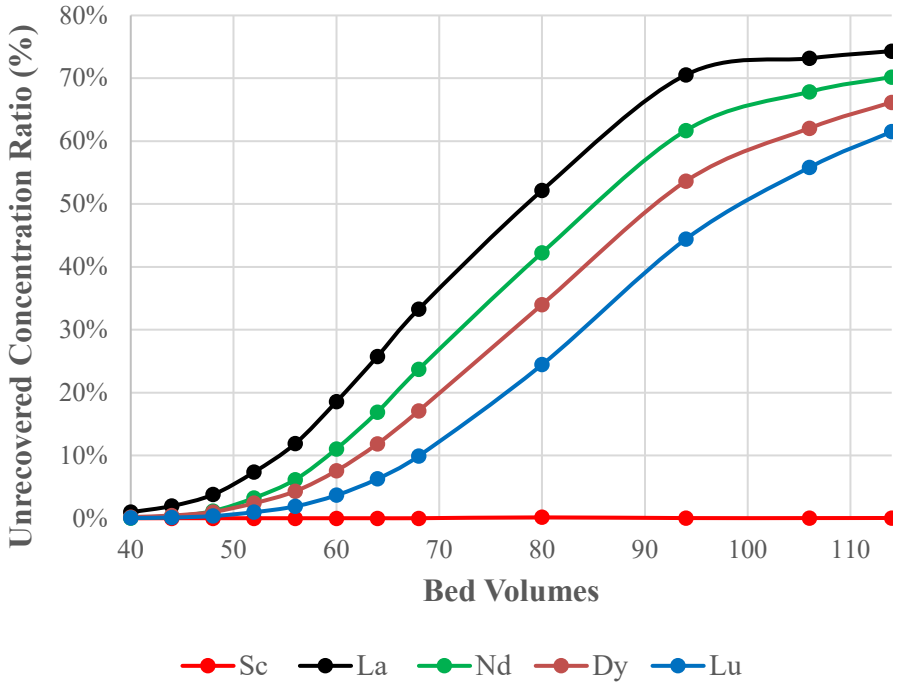
- Two concentrates formed – variable oxalate concentration
  - Sc enrichment into second product (vs REE), REE enrichment in first (vs Ca, Al)

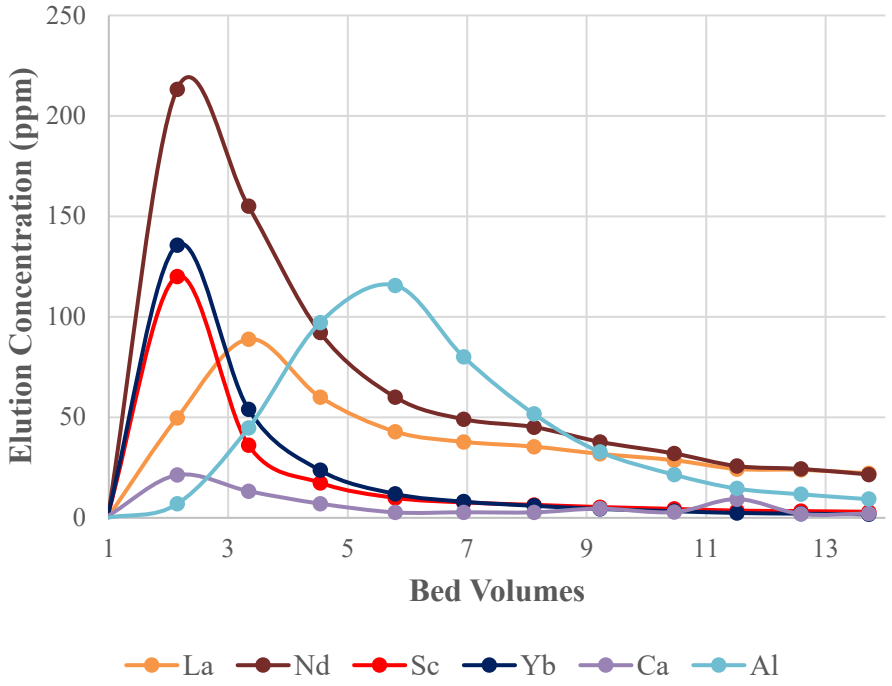




MREO Concentrate Purification

- Firing, dissolving, and separating impurities from ~65% pure concentrate using ion exchange resins
  - **IDA** and AMP resins chosen from ~10 options
  - Can produce >85% pure (cation basis) REO concentrate
  - Ce excluded by firing to tetravalent form, insoluble in dissolution step





Pilot Construction Updates

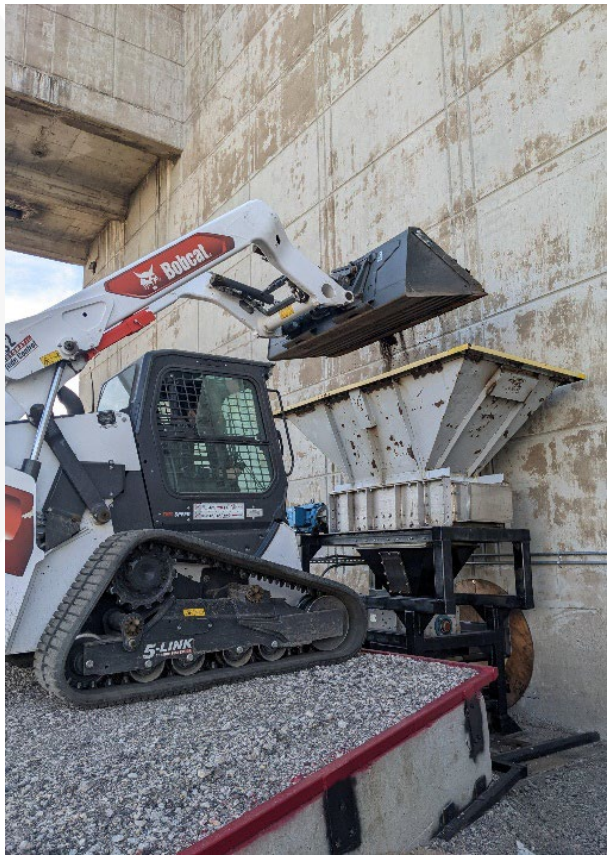
Pilot Construction Updates

- Pilot plant construction nearly complete
- In main process areas all equipment, piping, valves, and sensors have been installed
- All equipment, valves, and sensors are wired for feedback and control in controls program
- Developed control program to operate filter presses
  - Open/close, plate shifting, safety controls associated with operation and operator proximity

Filter Presses

- Needed to develop a control program for operation of filter presses
- The main program requirements included:
  - Opening and closing the filter press remotely
  - Shifting filter press plates with the plate shifter
    - Plate shifting can either be done automatically or semi-automatically
      - Automatic: all plates are shifted without stopping
      - Semi-automatically: a pull cord is used to shift one plate at a time
  - Integration of safety features into the control program
    - Light curtains, which would stop motion of filter press if tripped
    - Emergency stop pull cords

Coal Handling System



- Coal crushing and handling system operational and undergoing commissioning
- Coal is fed into staged, low-impact crushers, chuted into large 14-hr hoppers

Pilot Shakedown Testing

- Leak checking all piping – 99% complete
- All pumps, mixers, sensors, and valves working and tied into LabVIEW program
- VFDs have been programed, and are able to be controlled remotely in the LabVIEW program for pumps and mixers
- Working on sensor calibration for pH and total solids sensors
- Testing filter press control logic – confirming timing and process variable compliance
- Tuning PID control loops

Next Steps - Project

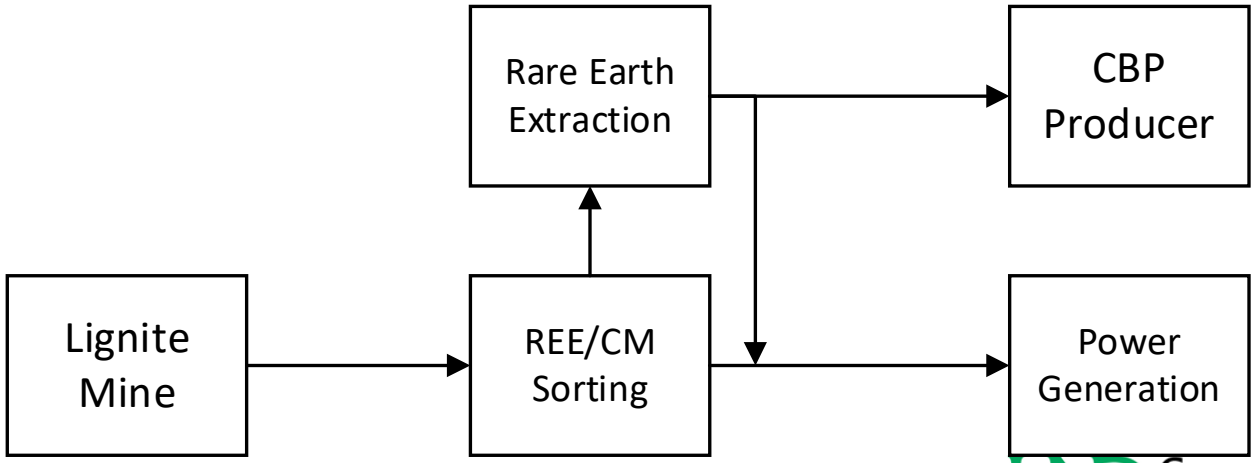
- Complete commissioning of pilot
- Complete preliminary testing (5-20 tons)
  - Send concentrate to RES for flowsheet development
- Conduct steady-state testing (75-125 tons)
  - Divide concentrate for RES and stockpiling
- Update Feasibility study to reflect pilot-scale testing and economics

Next Steps - Technology

- Resource characterization – drilling+
  - Identify high-concentration seams/sub-seams in active mines
  - Develop x-y-z model for contained REEs within coal seam/sub-seam
- Engineering/FEED Study
  - Design/engineering of commercial facility
    - Purification of CM's
  - Business development
    - Off-takes for byproducts (upgraded coal, CMs)

Commercialization Plans

- Co-locate with active mine/power facility
  - All lignite mines have a mine-mouth power station in ND
    - Optional CBP off-take, preferred for value-generation
  - Minimizes travel distance of high-volume feedstocks/products



Workforce Development

- Student Inclusion
  - Involved three graduate students in the project, and graduated one
    - Engineering, Geology
  - Developed engineering coop opportunities for undergraduate students
    - 2 students over 2 semesters
  - Hourly student employees
    - 3 undergraduate students

Public Outreach

- Conducted a radio interview for a Bismarck, ND-based radio station
  - Proposed site selections for commercial plants within range of radio station

- Three news releases to local/state newspaper columns

Summary

- Lignite offers major advantages as a REE ore, primarily involving the organic character
  - More selective, easier extraction
- Parameters identified and determined for planned pilot testing
- Pilot construction ongoing – 500 kg/hr lignite feedstock input
  - Producing 5-10 kg of >85% MREO concentrate per operating week
  - Deep into commissioning – operational goals of November

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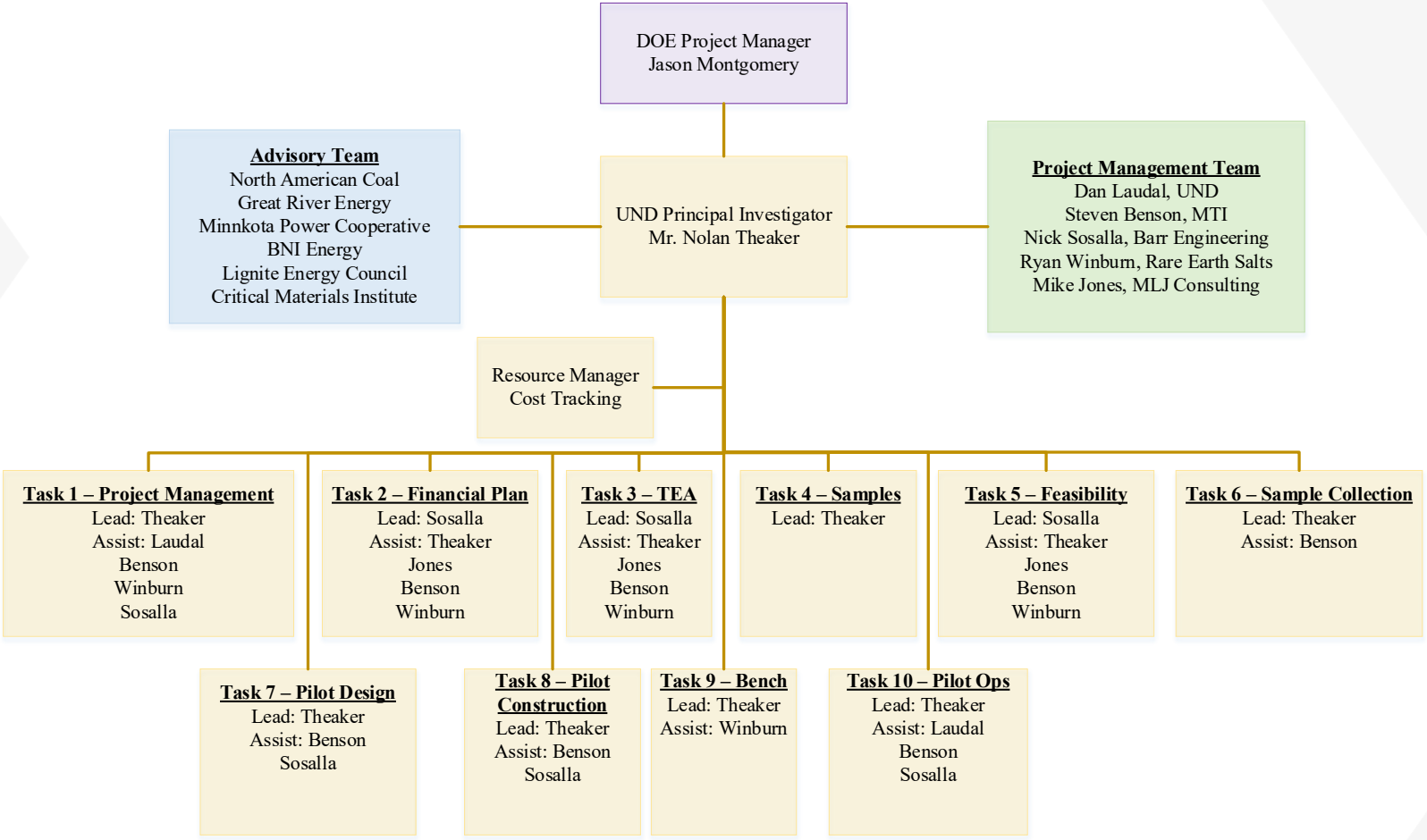
Organization Chart



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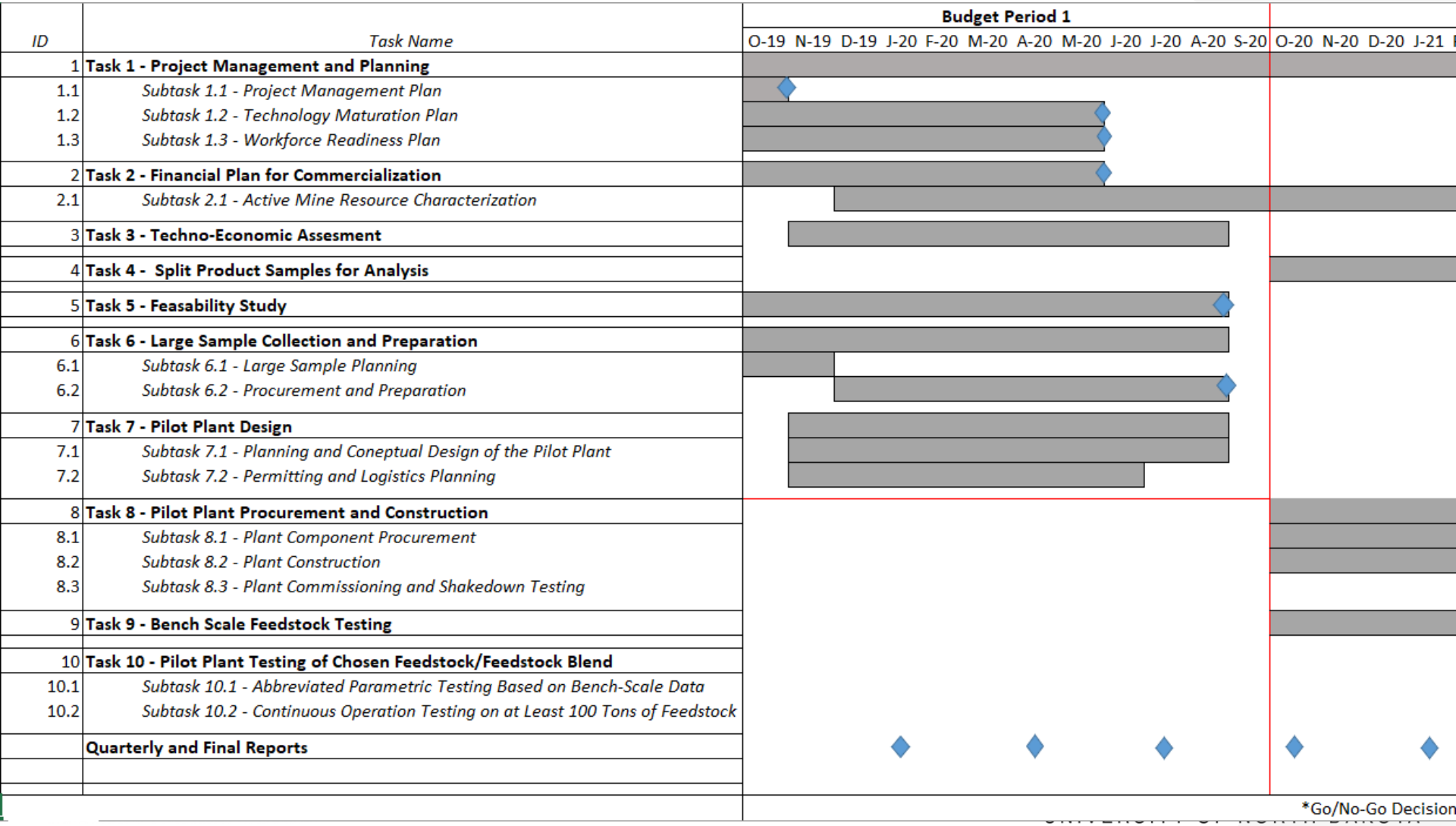
Gantt Chart (1)



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1.1	*Subtask 1.1 - Project Management Plan*
1.2	*Subtask 1.2 - Technology Maturation Plan*
1.3	*Subtask 1.3 - Workforce Readiness Plan*
2	**Task 2 - Financial Plan for Commercialization**
2.1	*Subtask 2.1 - Active Mine Resource Characterization*
3	**Task 3 - Techno-Economic Assesment**
4	**Task 4 - Split Product Samples for Analysis**
5	**Task 5 - Feasability Study**
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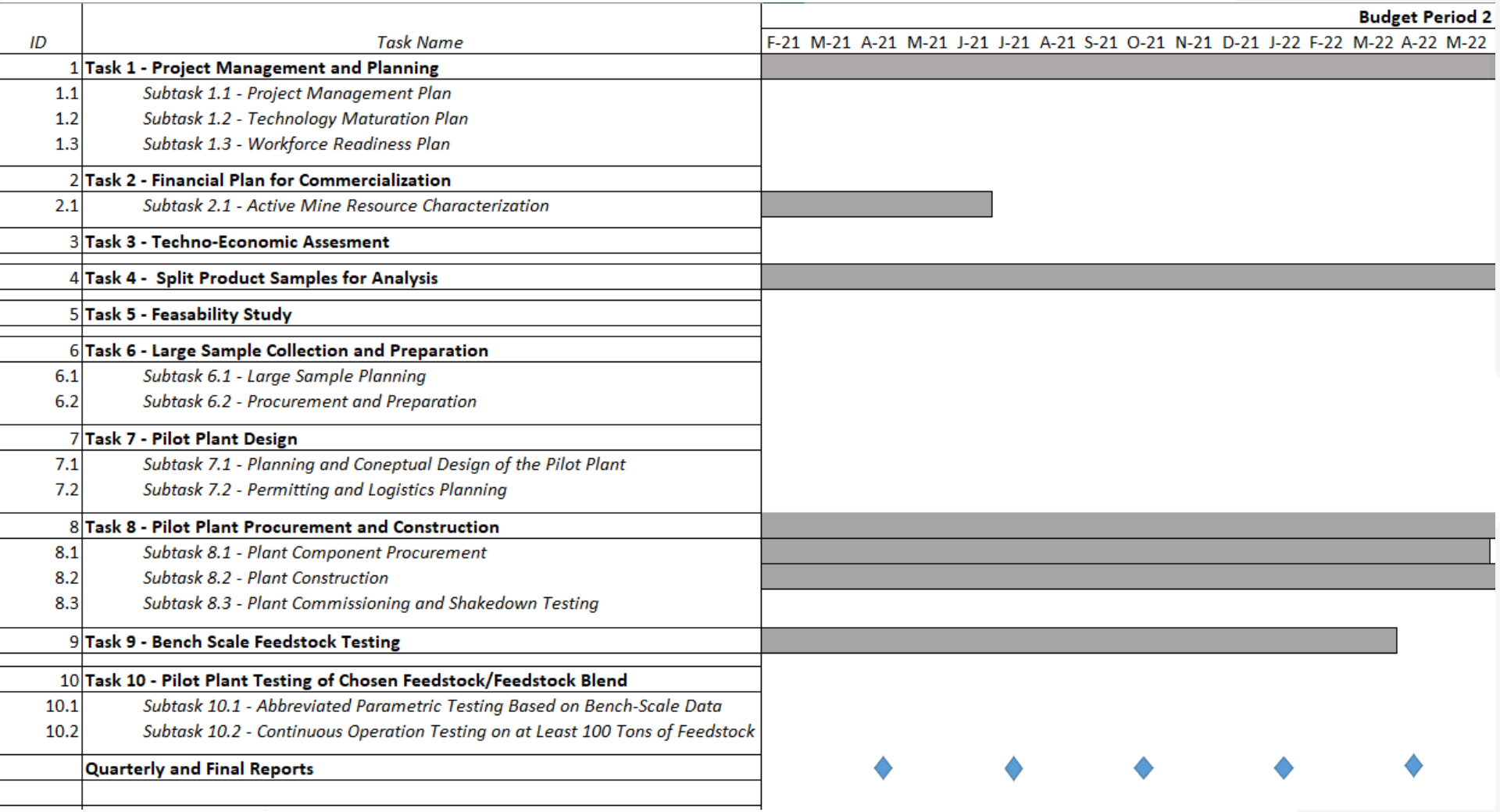
9	**Task 9 - Bench Scale Feedstock Testing**
10	**Task 10 - Pilot Plant Testing of Chosen Feedstock/Feedstock Blend**
10.1	*Subtask 10.1 - Abbreviated Parametric Testing Based on Bench-Scale Data*
10.2	*Subtask 10.2 - Continuous Operation Testing on at Least 100 Tons of Feedstock*
	Quarterly and Final Reports

Gantt Chart (2)



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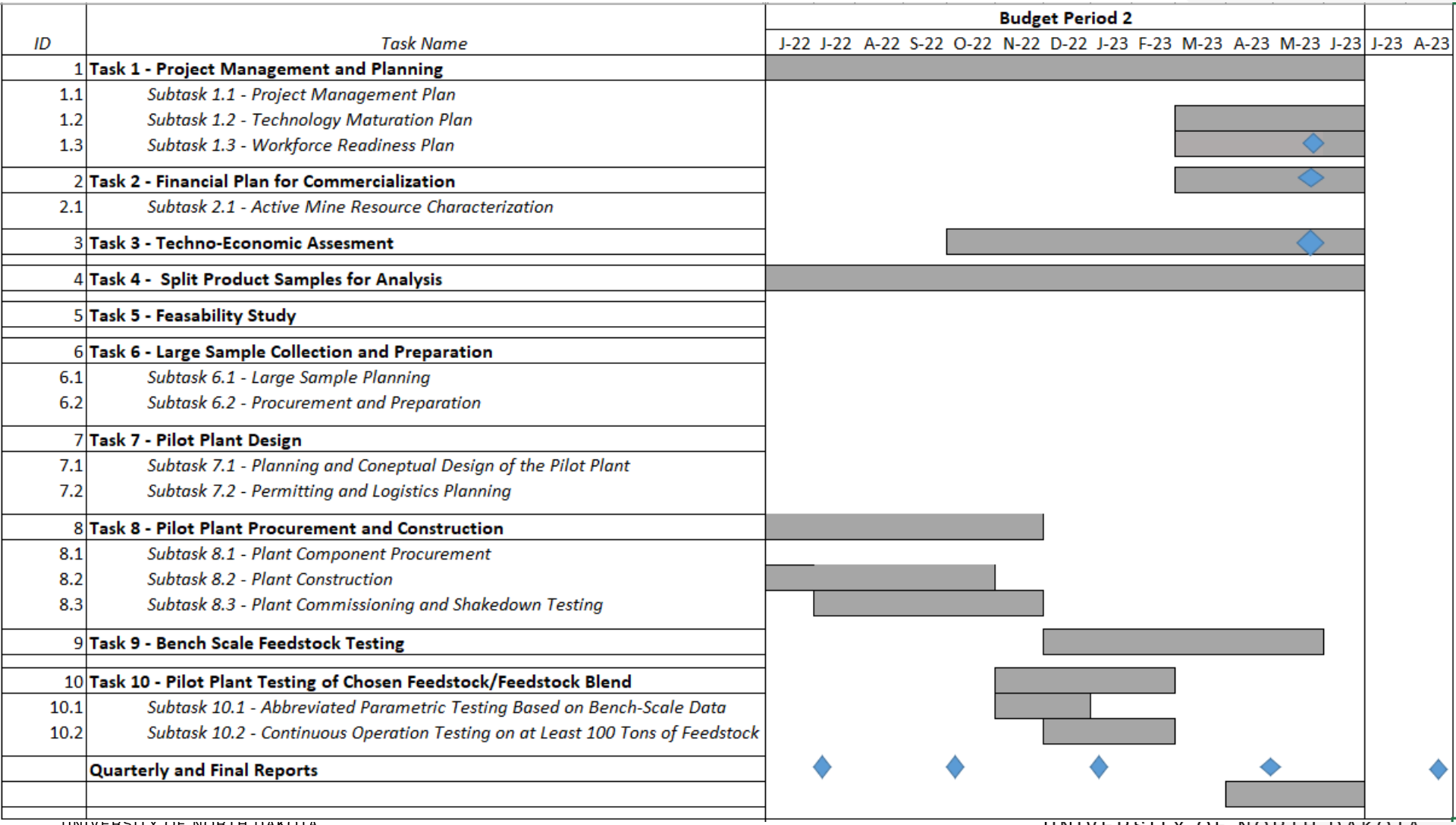
Gantt Chart (3)



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