Low-Cost and Recyclable Oxygen Carrier and Novel Process for Chemical Looping Combustion
DE-FE0031534

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May 11, 2021

U.S. DEPARTMENT OF ENERGY

UNIVERSITY OF NORTH DAKOTA
LEADERS IN ACTION

NATIONAL ENERGY TECHNOLOGY LABORATORY

Project Partners

1. University of North Dakota (Prime)
2. Envergex LLC
3. Carbontec Energy Corporation
4. Microbeam Technologies, Inc.
5. BARR Engineering

Presentation Overview

- Background – Chemical Looping Combustion
- Project Objectives
- Current Status of Project
  - Bench Unit OC Manufacture; Bench Testing; OC-Ash Interactions; Preliminary OC Manufacturing TEA
- Future Work and Concluding Remarks
- Questions and Discussions

Main Project Objectives

Funding Objective: Advance CLC technologies towards meeting 90% CO_2 capture and 99% carbon conversion.

Project Objectives:

- Develop low cost, low attrition and “recyclable” oxygen-carrier
- Develop a 10 kW unit that:
  - Uses unique hydrodynamics of spouted fluid bed (SFB) to improve coal char reduction
  - Incorporates particle char separator (PCS) technology to improve char conversion
  - 90% CO2 Capture (90% fuel conversion)

Current Project Status / Highlights

- Evaluated **> 40 OC formulations** using a **mechanical mixing method**
- Best performers benchmarked against **ilmenite**
- Down-selected **one engineered OC (FEL3)** and one alternate (FEH31), reactivity **up to 8 times** better than baseline ilmenite
- Integrated CLC system – modifications ongoing to meet operating parameters
- Bench unit OC manufacturing – Methodology set, only waiting for material
- Established Ash-OC interaction methodology
- TEA study in progress – insightful data from preliminary investigation (ash recycling)
- One year project extension requested to accommodate delays in execution.

PROJECT PROGRESS

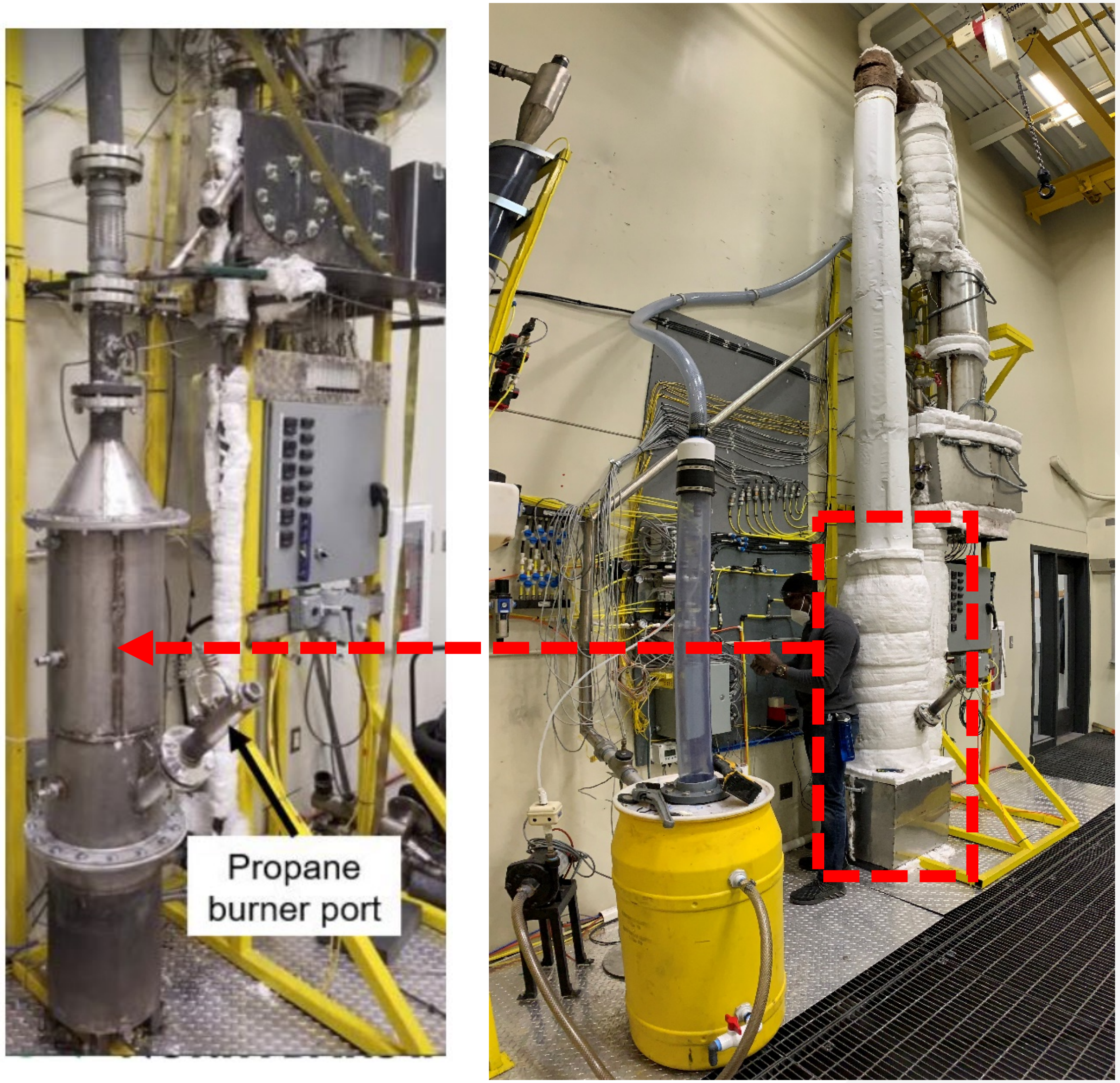
1. OC SCALED-UP MANUFACTURING
2. 10 KW TESTING

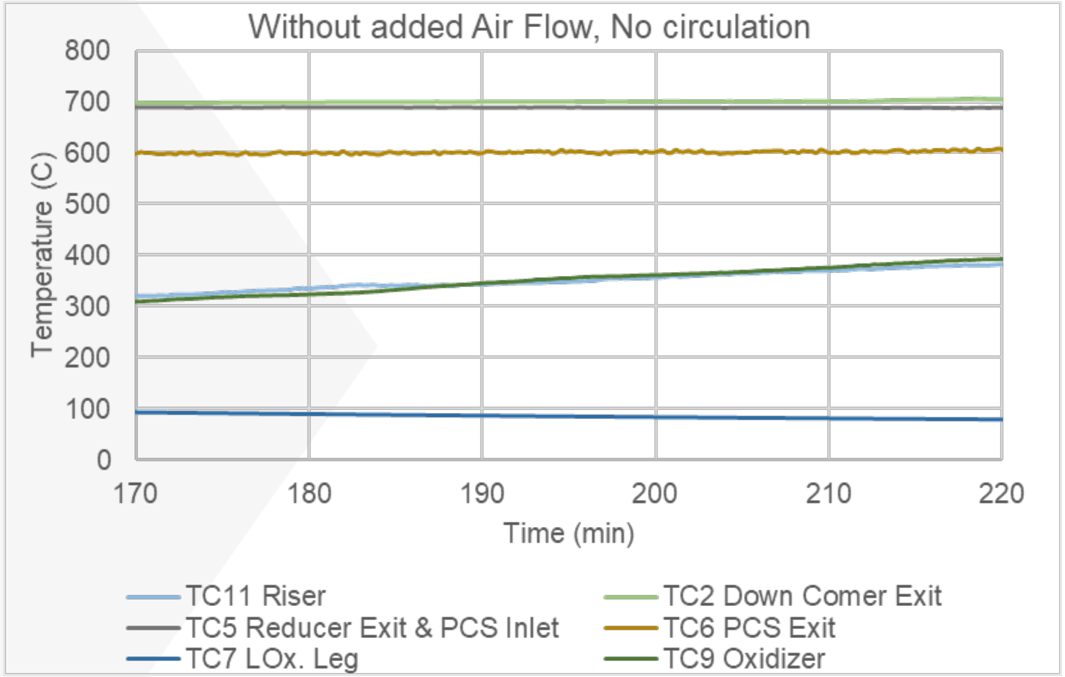
Bench Unit OC Manufacturing

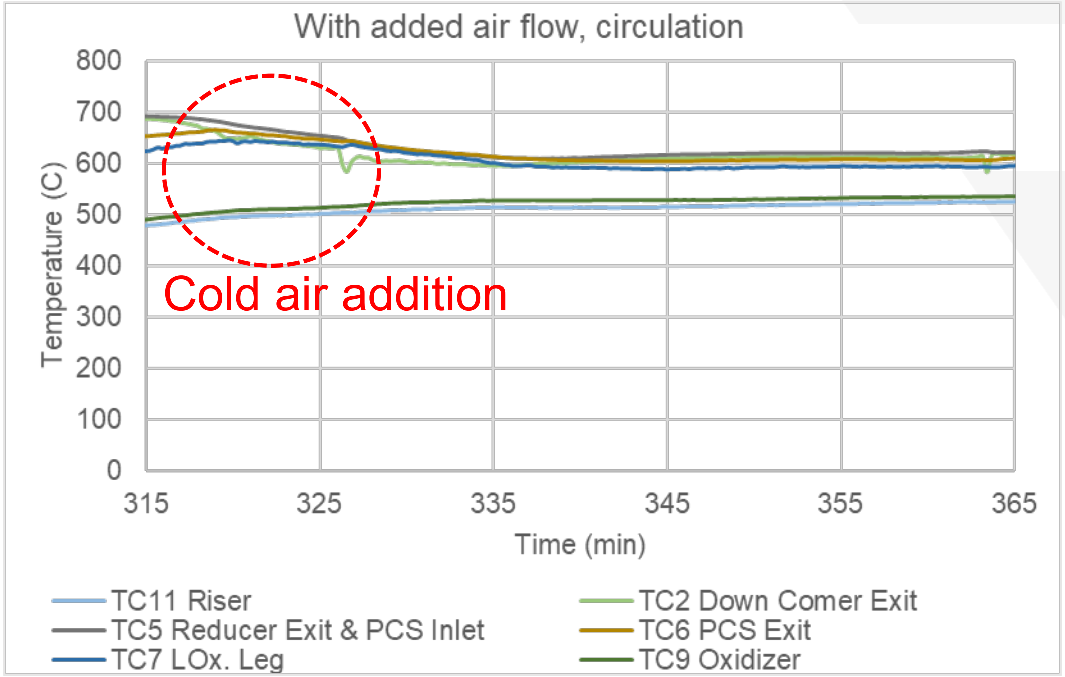
- **Scaled-up manufacturing (Selected OC Candidate - FEL3)**
  - Target production **1000 lbs**
  - Infrastructure arranged (jet-mill, micro-pelletizer, kiln)
  - Active looping ingredient procurement delay; discussing with a different steel mill for waste
  - Alternative looping ingredient ready for OC preparation (contingency)

Bench Testing

- Re-designed oxidizer unit, resulted in new operating paradigm
- Continuing work to achieve temperature in oxidizer needed for testing (>800˚C in oxidizer)
- Challenges have been in coupling material circulation with overall system heating







- Bulk of air flow in combustor (optimal heat balance, fastest ramp temperatures).
- Unable to circulate through system, due to low fluidization in the oxidizer bed
- With combustor, able to heat oxidizer dT: 100°C in 50min

- Circulating material in system (oxidizer -> reducer -> oxidizer) under the addition of “cold air” to the material bed through bed lance
- Observed cooling effect with this air addition
- With Lance and combustor air going to heat oxidizer dT: 30°C in 50min

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Bench Testing Next Steps…

- Challenges with circulating solids in system: Added in bed lance to deliver additional fluidization air to bed material
- Air addition = overall cooling effect on system *but was able to circulate material*
- Solution not feasible long term due to high propane / excess air requirements exceeding design velocities
- Solution: Modify oxidizer and adopt electrical heaters

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PROJECT PROGRESS

3. OC-ASH INTERACTIONS

OC-Ash Interactions

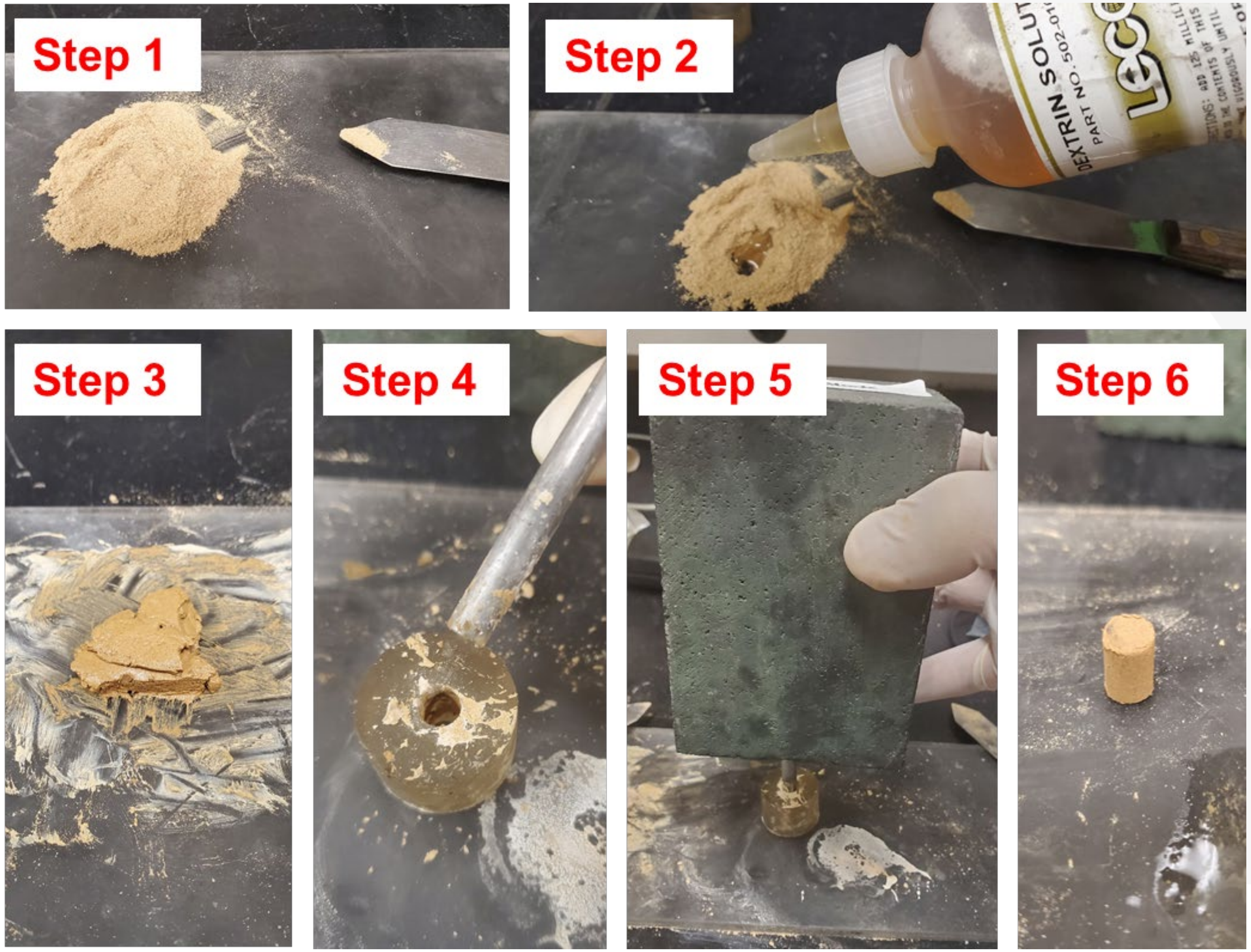
- **Interaction effects***
  - Agglomeration
  - Different oxidation/reduction rates
  - Catalytic (gasification)
  - Oxygen transport capacity

- **Study effects with target OC (FEL3) and fuel (Sub-bituminous, absaloka) using modeling and experimental procedures**

*Staničić, I., Hanning, M., Deniz, R., Mattisson, T., Backman, R. and Leion, H., 2020. Interaction of oxygen carriers with common biomass ash components. *Fuel Processing Technology*, *200*, p.106313.

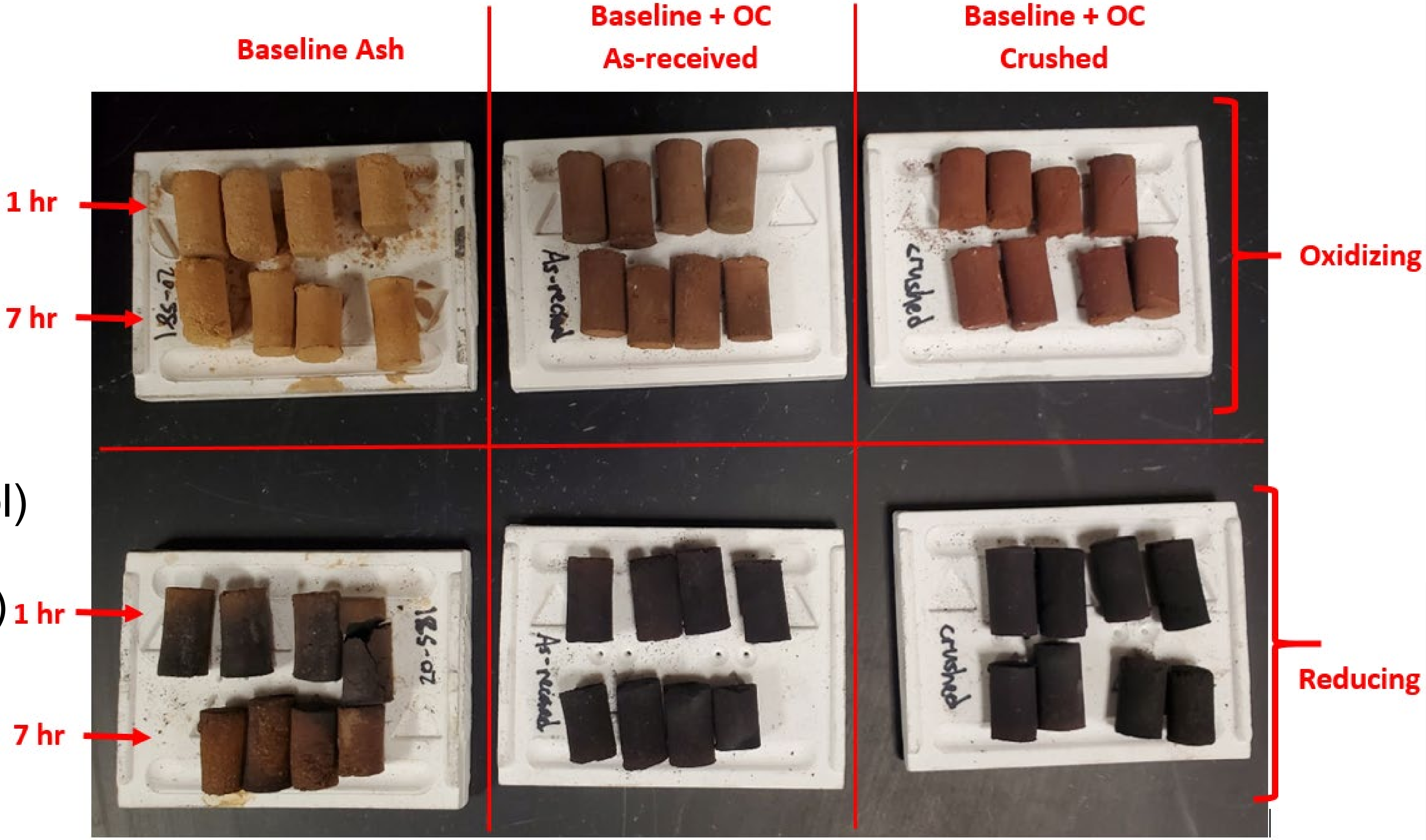
OC-Ash Interactions

- **Preparation**
- 48 Samples to date
- Step 1 – Dry Mix Powders
- Step 2 – Add Dextrin binder solution
- Step 3 – Wet Mix
- Step 4 – Place mix into die
- Step 5 – Press with block (same pressure)
- Step 6 – Remove pellet



OC-Ash Interactions

- **Preparation**
- Oxidizing (900 – 950˚C) vs. reducing (850 – 900˚C)
- Time effect: 1 hr vs. 7 hrs
- Baseline (100 wt% ash – control)
- Other 50/50 wt% (OC/Coal-ash)
- Material size:
  - As-received
  - Crushed



Post-sinter reducing and oxidizing runs. Baseline composition is 100% coal ash, and Baseline + OC (as-received and crushed) are 50/50 wt.%.

OC-Ash Interactions

Future work

- Investigate interactions using bituminous, sub-bituminous, and lignite
- XRD & SEM analyses
- XRD data for FactSage comparison
- SEM to examine morphological changes to OC particles
- Crush strength comparison before and after heat treatment

PROJECT PROGRESS

4. OC MANUFACTURING – COMMERCIAL (INITIAL TEA)

OC Manufacturing TEA

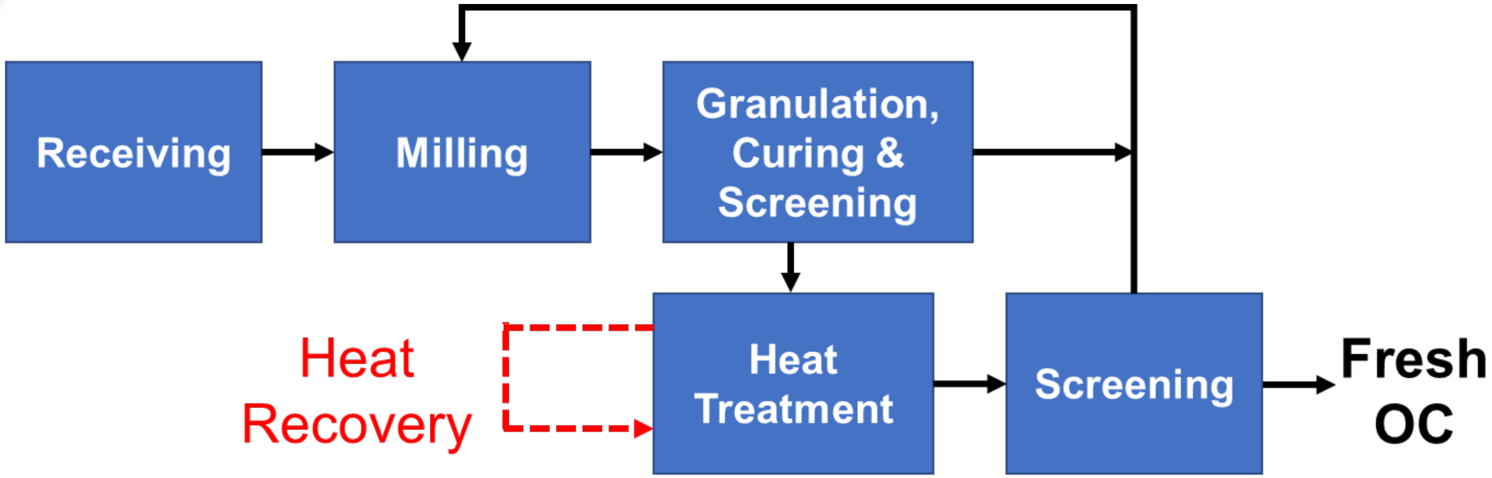
- **UND Approach significance**
  - OC composition designed to enable simplistic recovery
  - Cost of reformulating kept low
    - Recycle OC
    - Use existing infrastructure at CLC-plant level
    - Improved attrition resistance
  - Cost target: ~ $0.20/lb

OC Manufacturing TEA

- **Approach and Main Challenge:**
  - Dedicated manufacturing plant of 1,000,000 mt per year
    - Heat integration (exothermic process) vs process complexity
  - Localized manufacturing at CLC power plant – Recycled OC
    - Fresh OC replenishment rate = f(separation efficiency, segregation efficiency, attrition rate, …x_n)

OC Manufacturing TEA

- **Dedicated OC Manufacturing Setup:**
  - Conventional unit operations similar to ore processing
  - Heat integration possible – currently investigation options

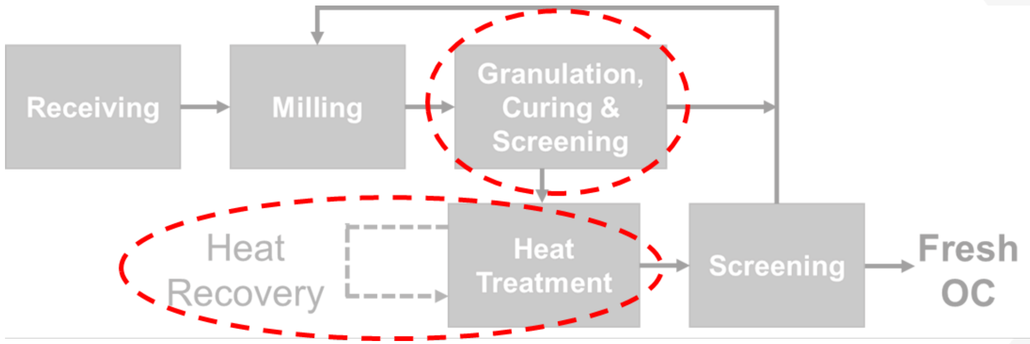


Heat Recovery

- Opportunity: Hot solids (~900°C) heat recovery improves thermal efficiency
- Challenge: Construction materials / operating conditions a concern

OC Manufacturing TEA

- **Mechanical Mixing Considerations:**
  - Fluid-Bed Granulator: Simple operation
  - Mixer Granulator: Particle size control – need to establish
  - Spray drying: Well proven, adequate size range
- **Heat Treatment Considerations:**
  - Rotary Kiln – Packages available from vendors
  - Multiple hearth furnace – Investigating viability
  - Fluid-Bed – Investigating viability



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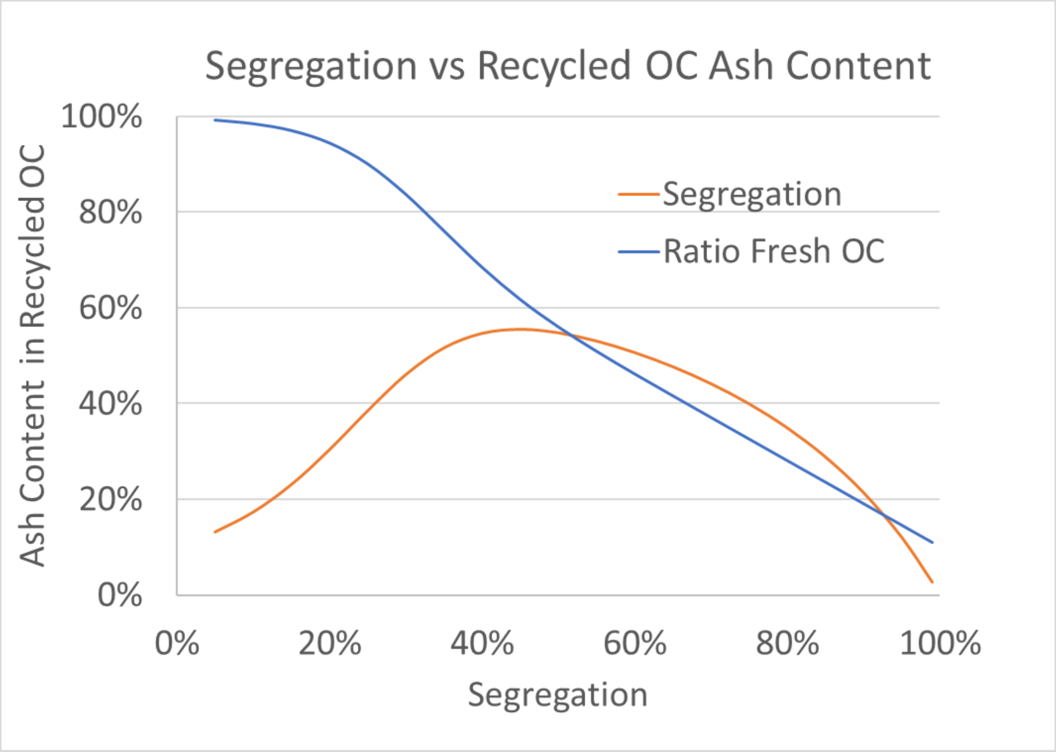
OC Manufacturing TEA

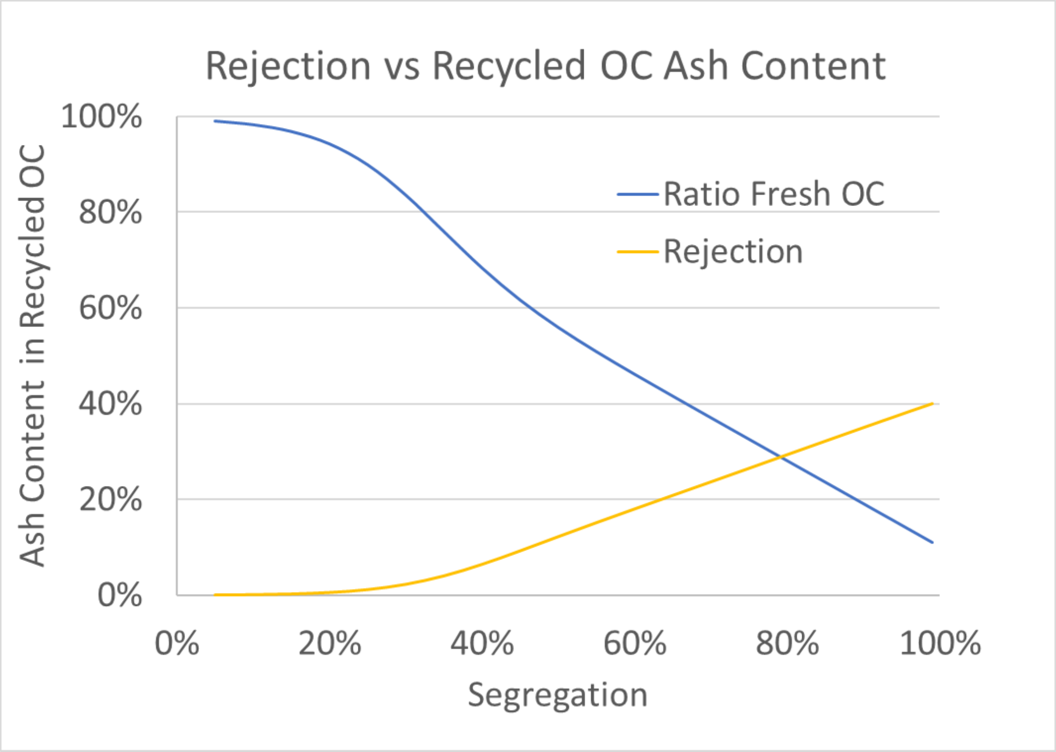
- **Localized OC Manufacturing Setup:**
  - OC recycling process integrated with CLC facility
    - Ash loading high / separation from attrited OC critical
    - Process still requires fresh OC makeup
  - Use CLC reactor (oxidizer) for heat treatment
  - Equipment smaller / lower cost

OC Manufacturing TEA

Segregation: separation of attrited OC from coal ash (rejection fixed)



Rejection: Fraction of ash + attrited OC rejected and disposed (segregation fixed)



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Future Work and Concluding Remarks

Future Work:

- Bench Testing
  - Extended testing at > 800˚C
  - Chemical looping combustion test with ilmenite and coal as baseline
- OC/Ash Interactions
  - Finish XRD/SEM analyses for OC/Ash with first of 3 coal samples
  - Evaluate, compare and discuss modeling and experimental results

Future Work and Concluding Remarks

Future Work:

- Bench Unit OC Manufacturing
  - Obtain desired main looping ingredient and finish 1000 lb delivery
  - Coal testing of FEL3 material
- TEA
  - Select heating configuration for dedicated OC manufacturing plant
  - Assess heat integration options and benefits
  - Finalize Aspen Plus Models (Dedicated and CLC-Located Manufacturing Plants)

Acknowledgements

- Acknowledgment: "This material is based upon work supported by the Department of Energy Award Number DE-FE0031534."
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