Attrition Evaluation of Oxygen-Carriers in Chemical Looping Systems

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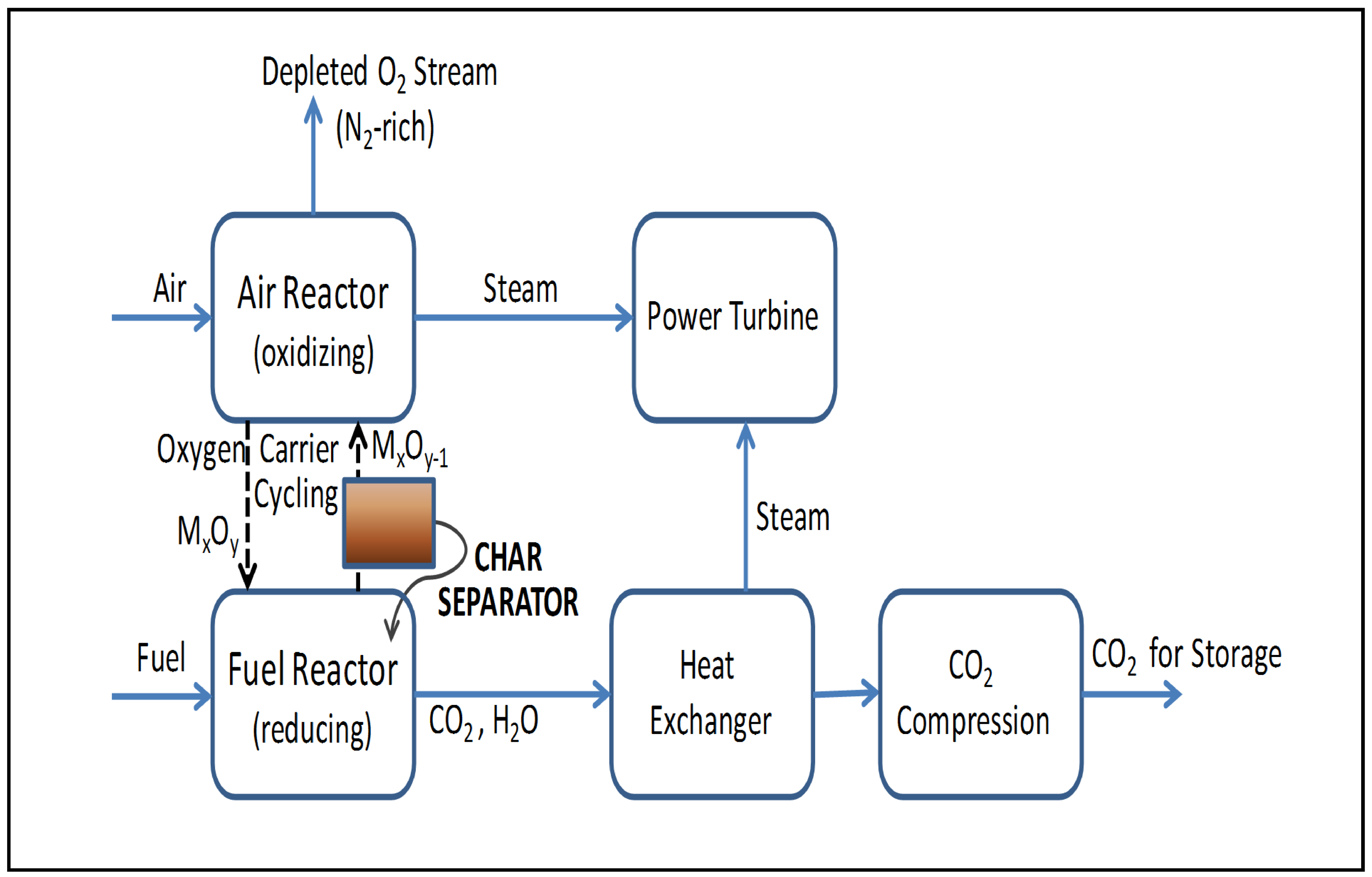
Background - CO_2 Capture

- Chemical-Looping-Combustion (CLC) is an innovative power generation technology for carbon capture at a lower cost and higher efficiency than state-of-the-art
  - Near-pure CO_2 stream produced without using oxygen from air separation
  - Solid oxygen-carrier (OC) used to provide oxygen to fuel
  - Oxygen-depleted solids regenerated separately in air
  - Solid oxygen carriers undergo attrition and loss of reactivity over time
  - Loss due to attrition and loss of reactivity creates significant operating cost burden



Project Objectives and Approach Methodology

- **Objectives**
  - Address critical element of CLC - loss of OC due to attrition/reactivity degradation
  - Evaluate attrition characteristics of oxygen carrier materials under high temperature, reacting conditions to establish correlations between process parameters
- **Methodology**
  - Basis - existing standard, (ASTM D5757), used for determining attrition characteristics of powdered catalysts by air jets
  - Incorporate modifications to attain test protocol more representative of chemical looping process conditions

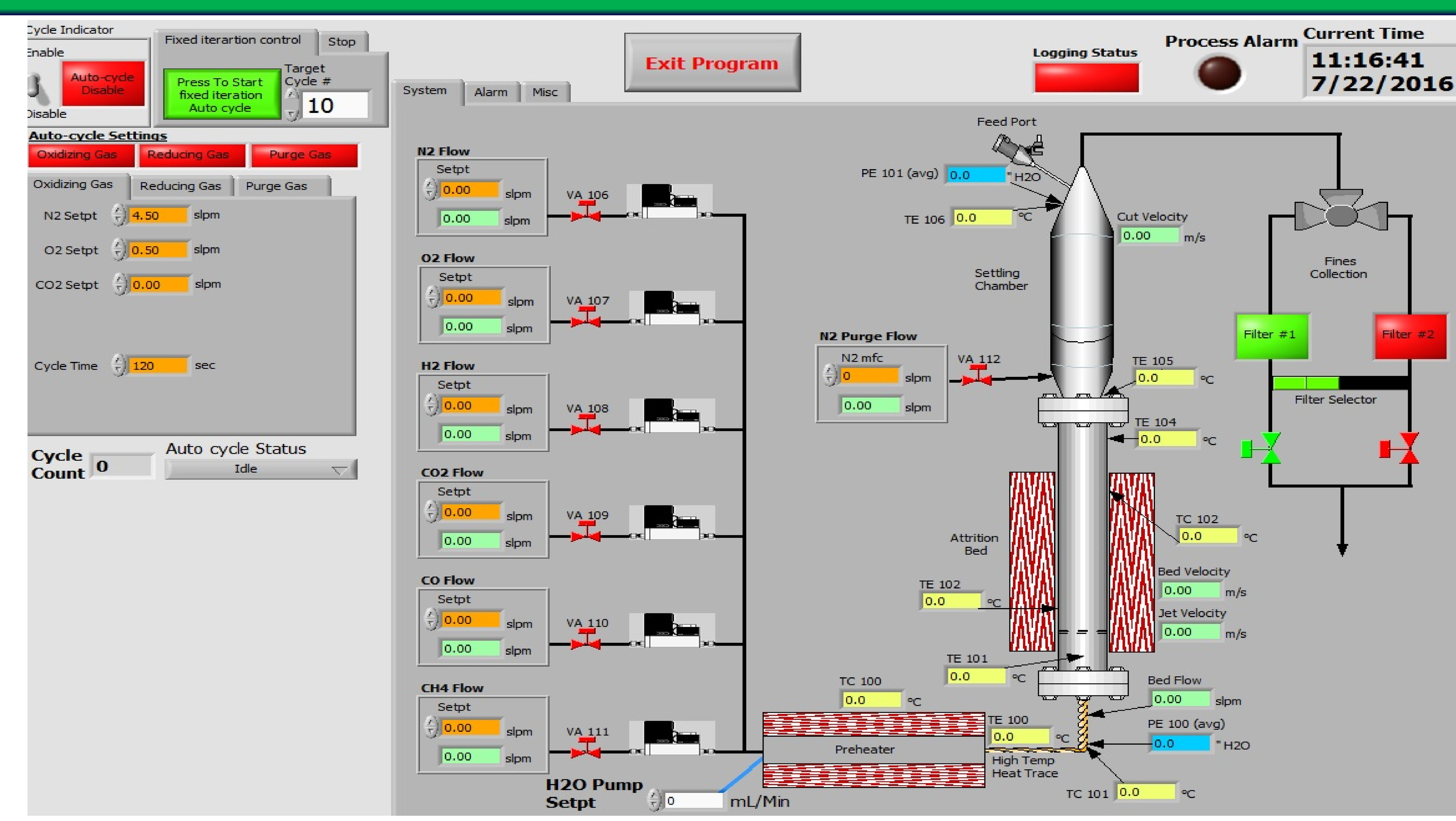
Evaluation Strategy

- Evaluated multiple oxygen carriers at baseline conditions
- Ilmenite (2), Hematite, Red Mud, $CaSO_4$-based, MnO_x-ore, Engineered iron oxide
- Cyclic operation between reduction and oxidation conditions
- Measured reduction/oxidation gas concentrations at reactor outlet

Temperature	800-1000°C
Oxidation Composition	90% N_2 , 10% O_2
Oxidation Cycle Time	4 minutes
Purge Composition	100% N_2
Purge Cycle Time	2 minutes
Reduction Composition	95% N_2 , 5% H_2/CO/CO_2
Reduction Cycle Time	4 minutes
Test Duration (No. Cycles)	500 minutes (~50 cycles)

- Measure attrition rate by collecting and weighing attrited material
- Examine the effects of varying jet velocities on the attrition of the oxygen-carriers
- Evaluate use of coal as fuel for reduction step on performance of selected oxygen carriers

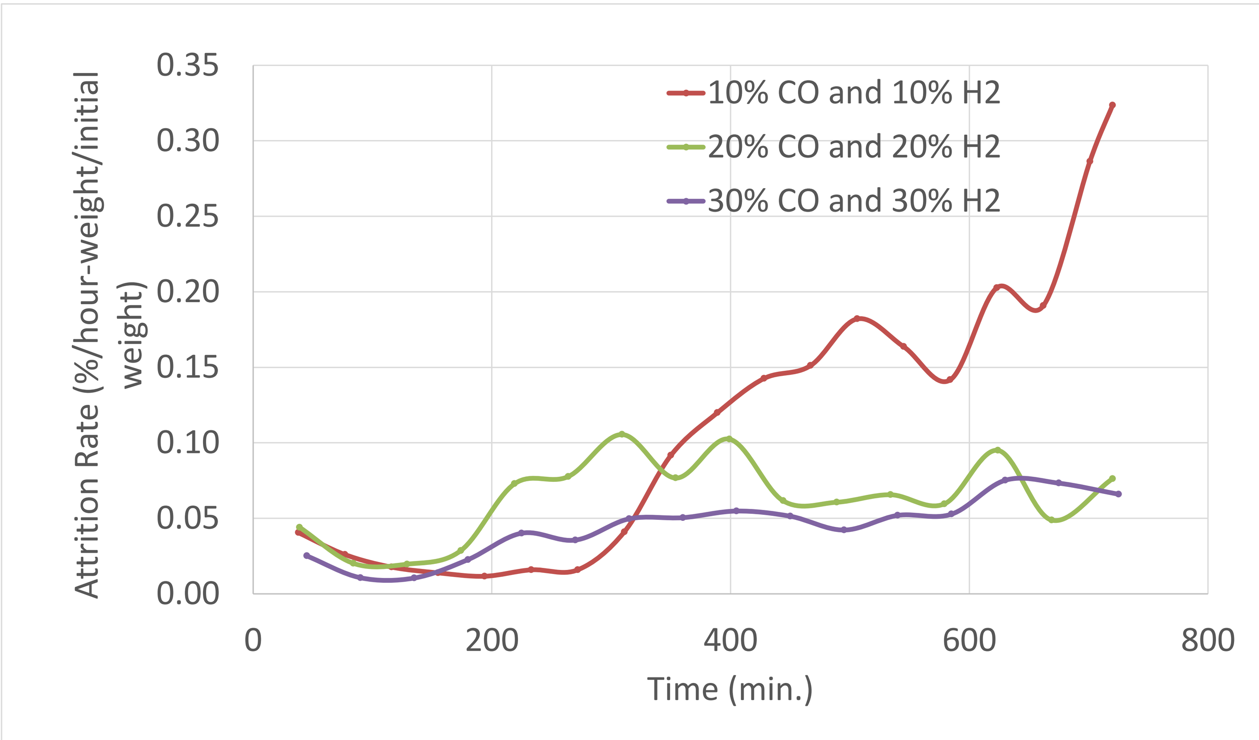
Experimental Setup

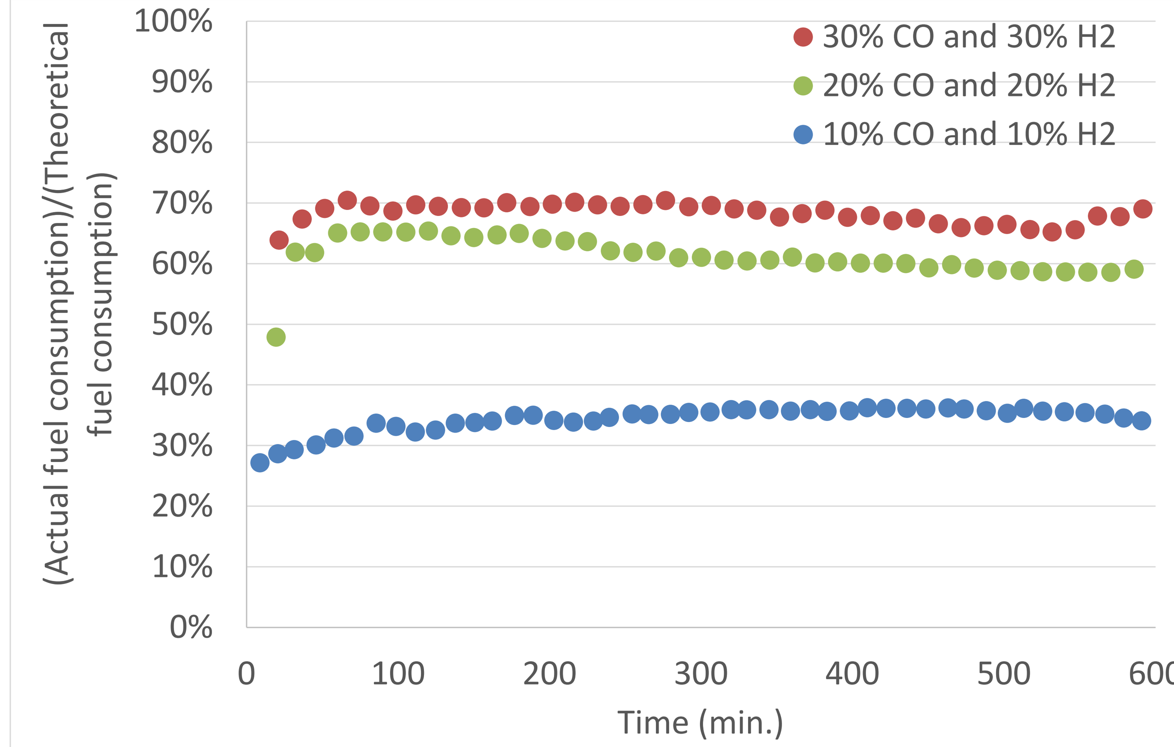


Results - Evaluation Tests

- *Ilmenite* and *Calcium-Based* oxygen carriers had lowest attrition rates.
- *Iron-Based-Engineered* oxygen carrier had highest rate of attrition.
- *Hematite* displayed a significant breakage event during testing; thereafter attrition rate decreased.
- *Red Mud* and *Mn-Oxide-Based* materials exhibited attrition rates that were slightly increasing or relatively stable over entire test period.

Results – Effect of Gas Composition on OC Performance

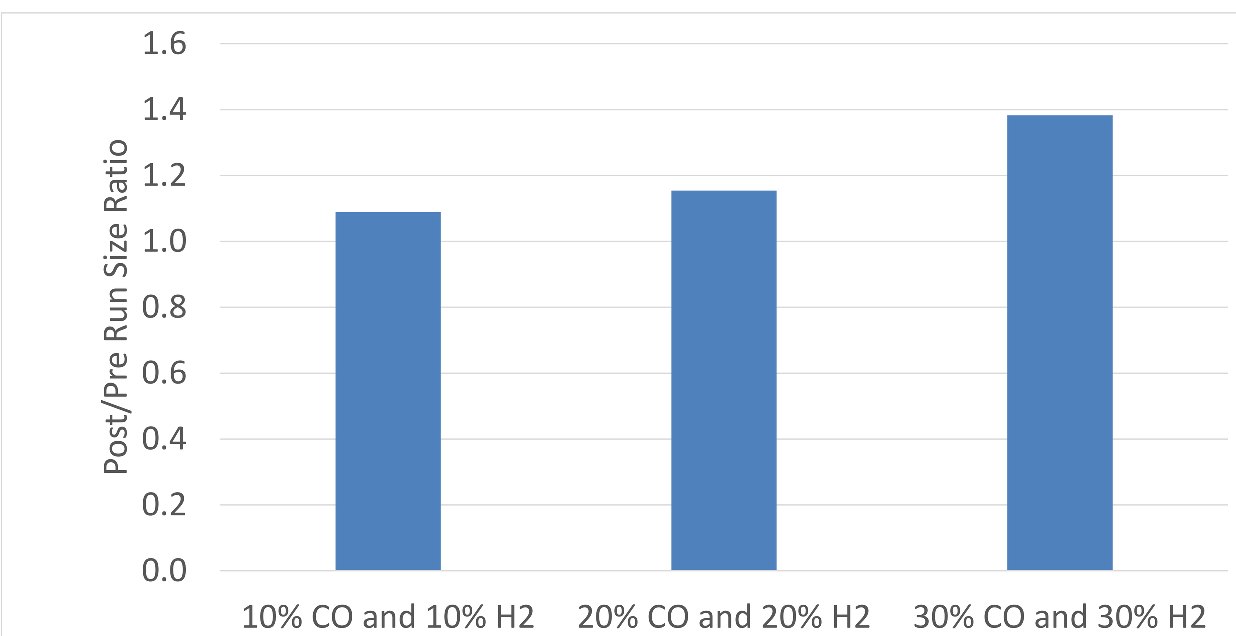




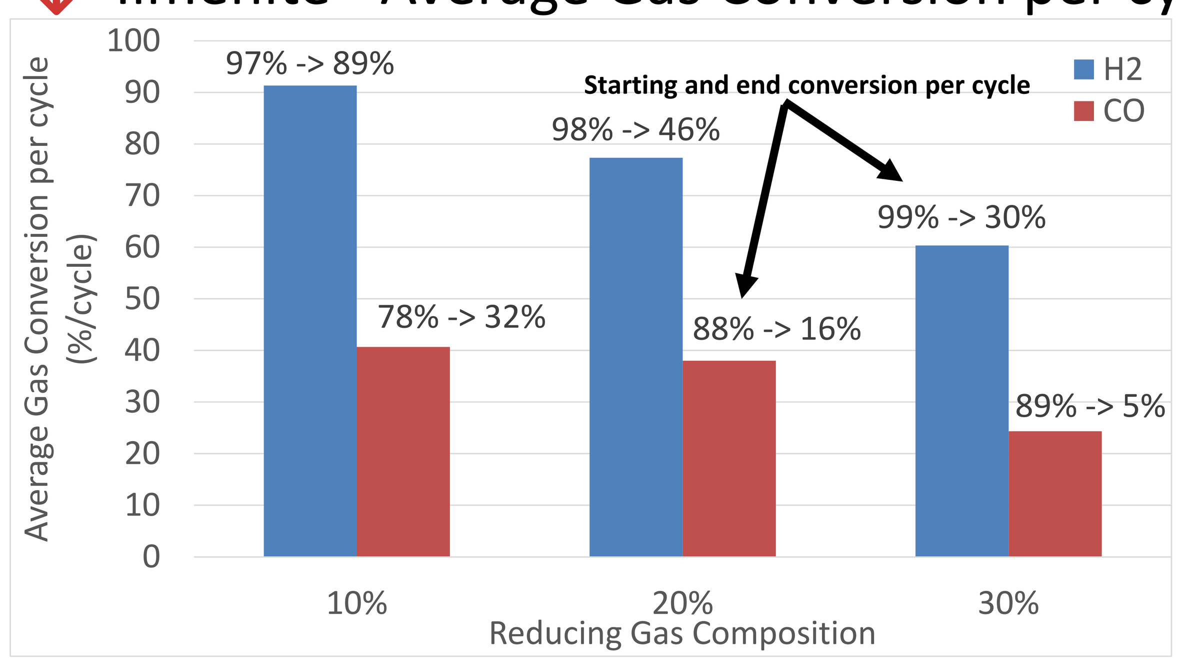
↑ Ilmenite- attrition, under differing reducing gas compositions

↑ Ilmenite- reactivity performance

↓ Ilmenite - PSD of pre- and post-run samples

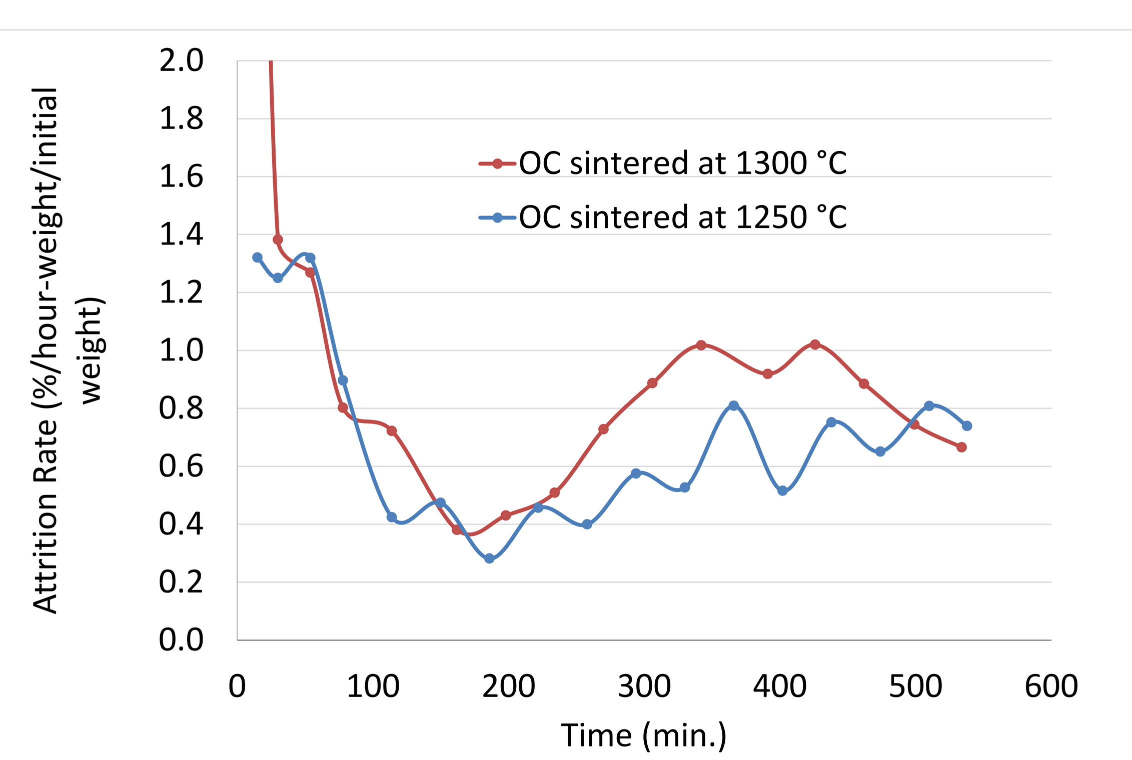


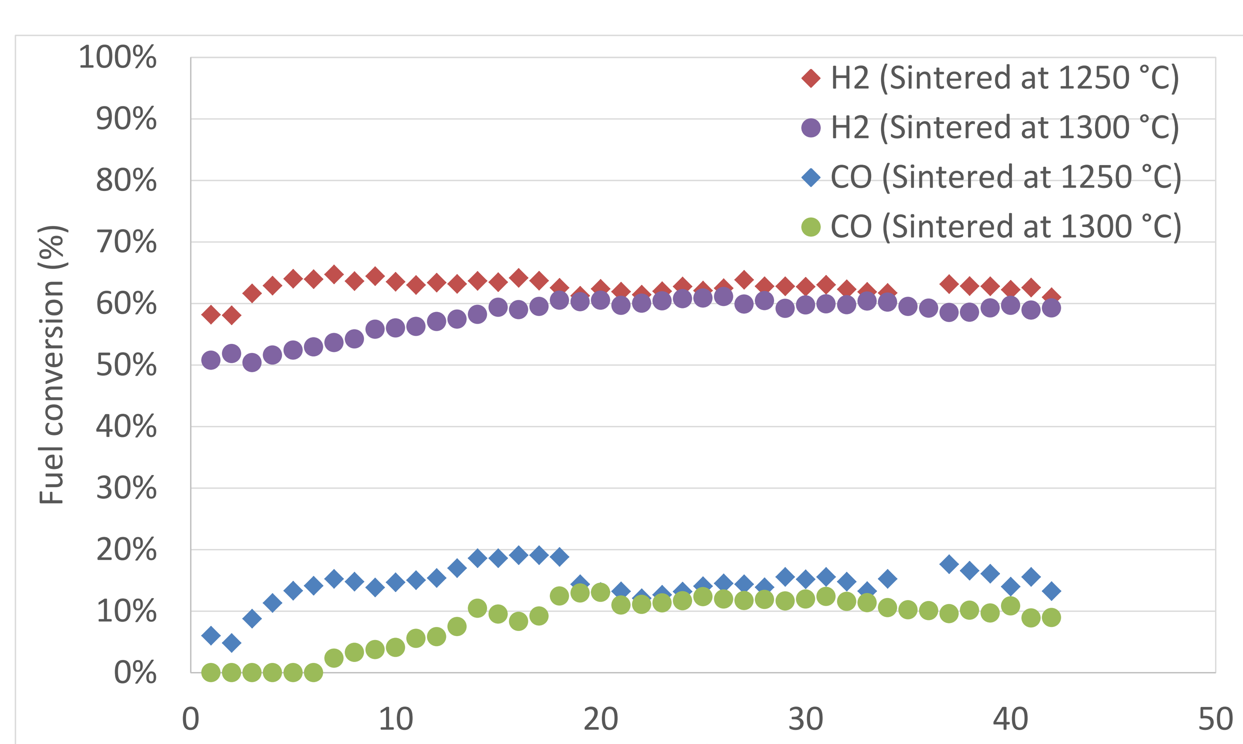
↓ Ilmenite - Average Gas Conversion per cycle



- Degree of reduction affected attrition and reactivity performance
  - Increasing CO/H_2 concentration resulted in better fuel utilization indicating higher reduced state of OC
  - Increasing CO/H_2 concentration caused agglomeration (20% - 30% concentration)
  - Degree of reduction critically important

Results – Effect of Manufacturing Conditions



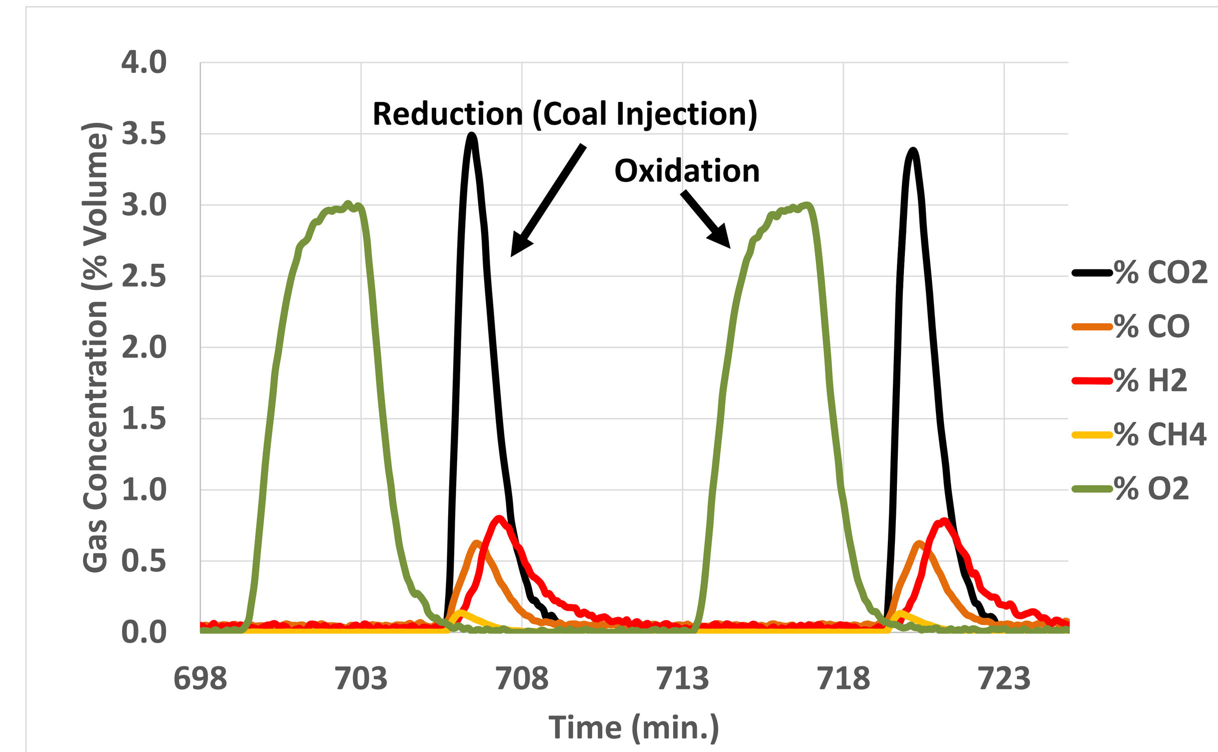


↑ Sintered Samples-attrition

↑ Sintered Samples-reactivity

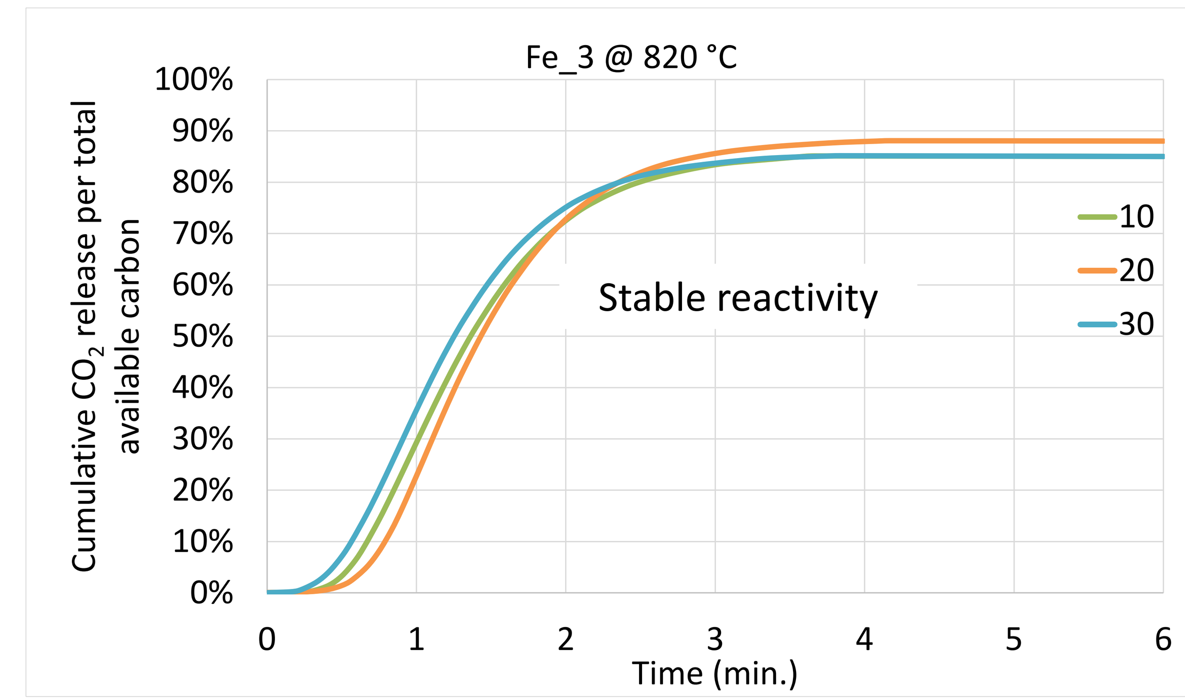
- Effect of sintering on OC performance
  - The attrition rate between the two samples exhibited similar trends
  - Manufacturing (Sintering) at higher temperature caused a decrease in reactivity

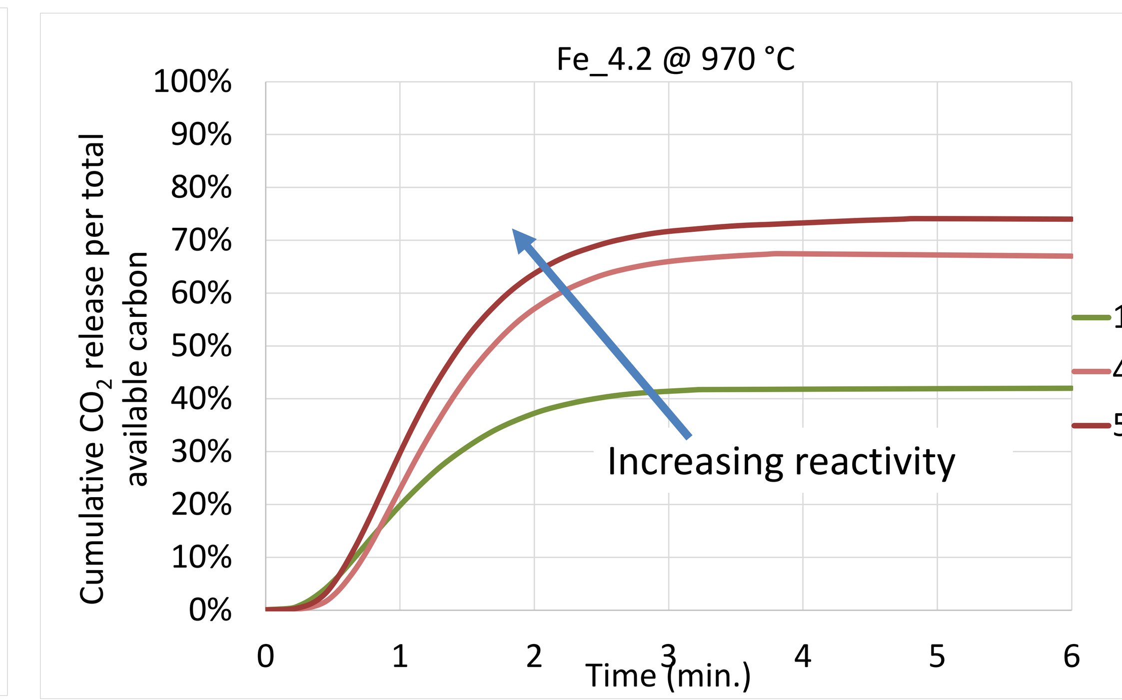
Results – OC Performance with Solid Fuel Combustion



- CLC reaction commenced quickly as CO and CO_2 is formed immediately upon injection of lignite coal. Formation of CO_2 confirms CLC reaction, as no oxygen was fed to the reactor.
- The oxygen carrier and steam were sources of oxygen for reaction

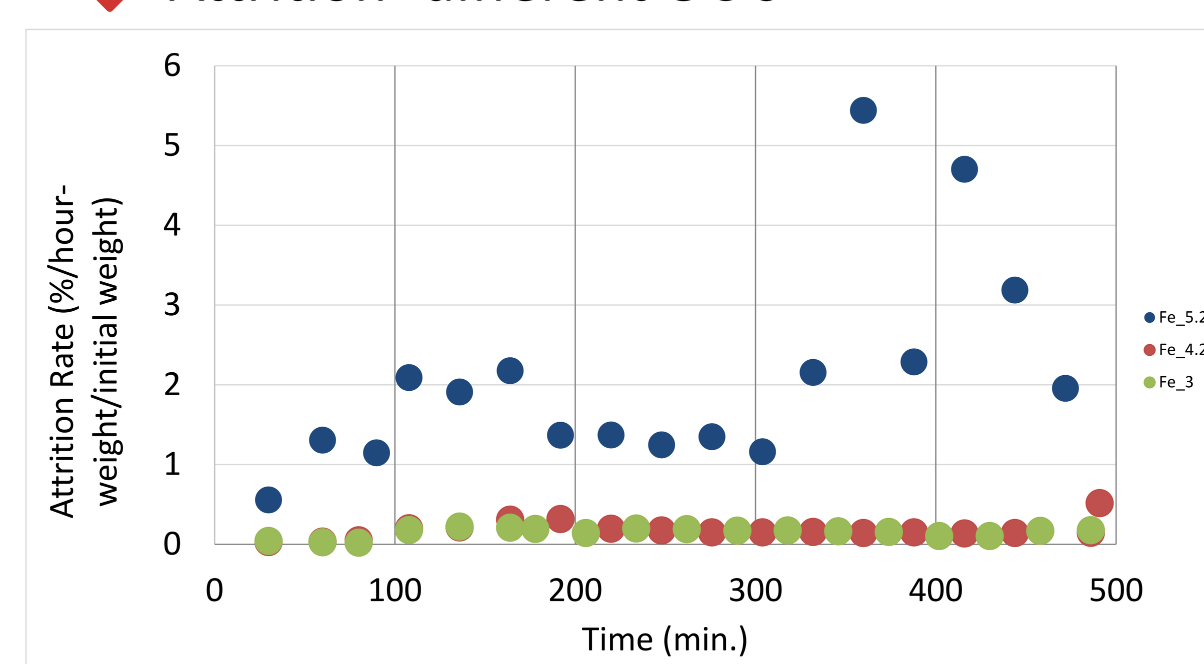
- Reactivity of different OC's at same test conditions



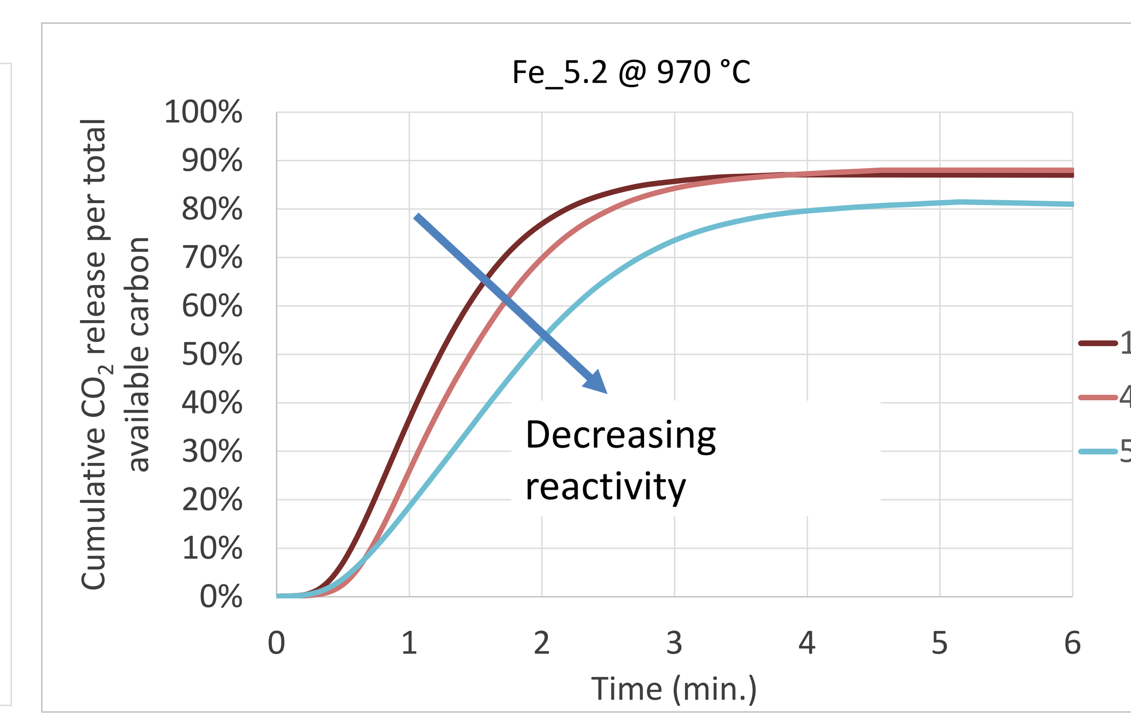


↑ Fe-based carrier under coal injection

↓ Attrition- different OC's



↕ Fe-based carrier under coal injection



Conclusions

- Methodology: effective tool to down-select oxygen carriers based on attrition propensity and reactivity
- Successful coal injection tests illustrate application of attrition setup as a valuable tool for future CLC research.

Future Work

- Effect of operating parameters on attrition and reactivity will be characterized to develop knowledge database and formulate strategies for commercial test service offerings.
- Expansion of work to study effect of cyclonic/impaction conditions on attrition and reactivity characteristics of Oxygen Carriers.

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