

# *Low-cost Oxygen (LCO) for Small-scale Modular Gasification Systems*

**Project DE-FE0028002**

U. S. Department of Energy  
National Energy Technology Laboratory  
PO: Steven Markovich

Prime Contractor:

**Thermosolv LLC**

Partners:

**Western Research Institute**

2019 Project Review Meeting for Crosscutting, Rare Earth Elements, Gasification  
and Transformative Power Generation  
April 9, 2019

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# Who is Thermosolv...

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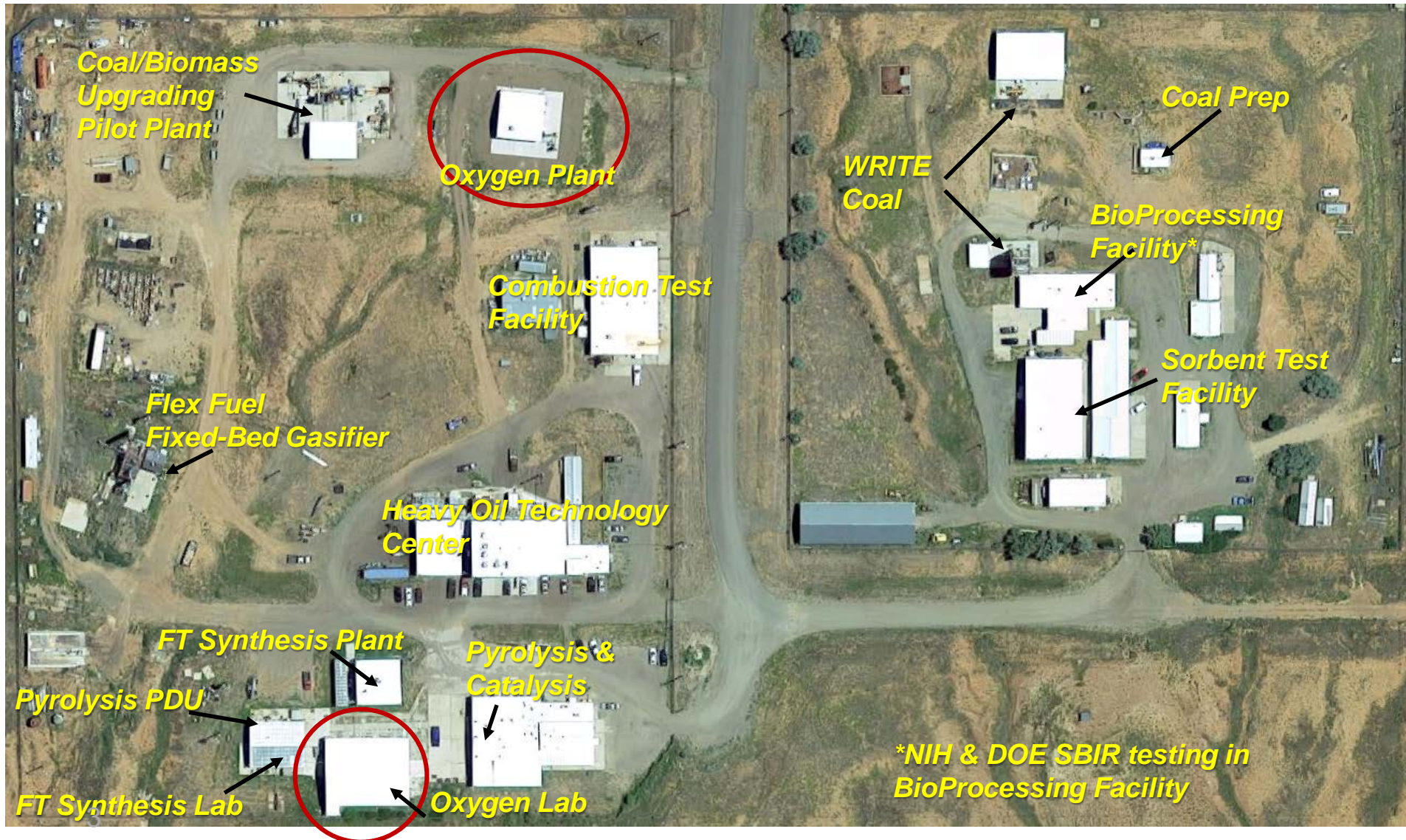
## Western Research Institute (WRI)

- WRI is a 501 (c) 3 research, technology development and contract services organization serving the energy, environment and highway materials industries.
- WRI is a former U.S. DOE Energy Technology Center (LETC). In 1983, LETC was privatized and WRI came into existence.
- Science to Technology to Commercialization

## Thermosolv LLC

- For profit spin-off of a business unit from Western Research Institute
- Established in 2011 to commercialize technologies
- Ten employees with full access to laboratory and pilot facilities
- Analytical Support from Wyoming Analytical Laboratory
- Access to Advanced Instrumentation at University of Wyoming

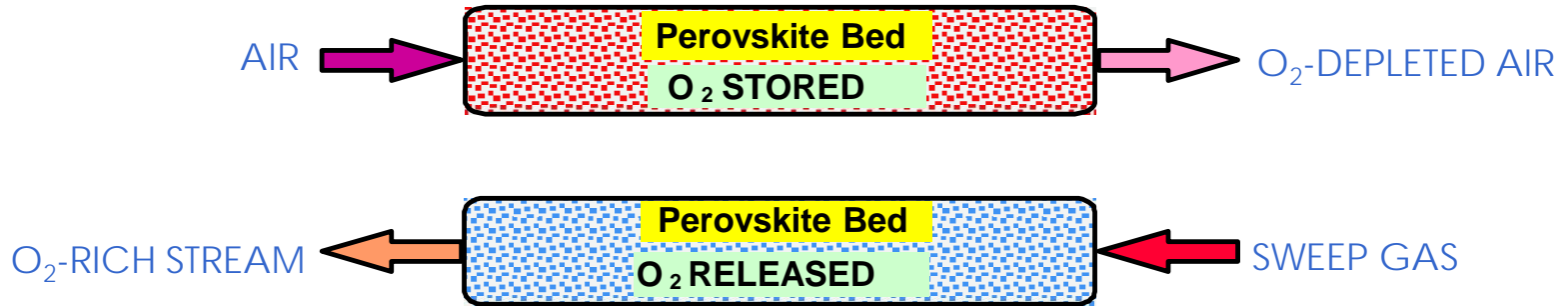
# Advanced Technology Center



*Low-Cost Oxygen...*

# Background

## LCO Process (Perovskite Sorbent-based Oxygen)

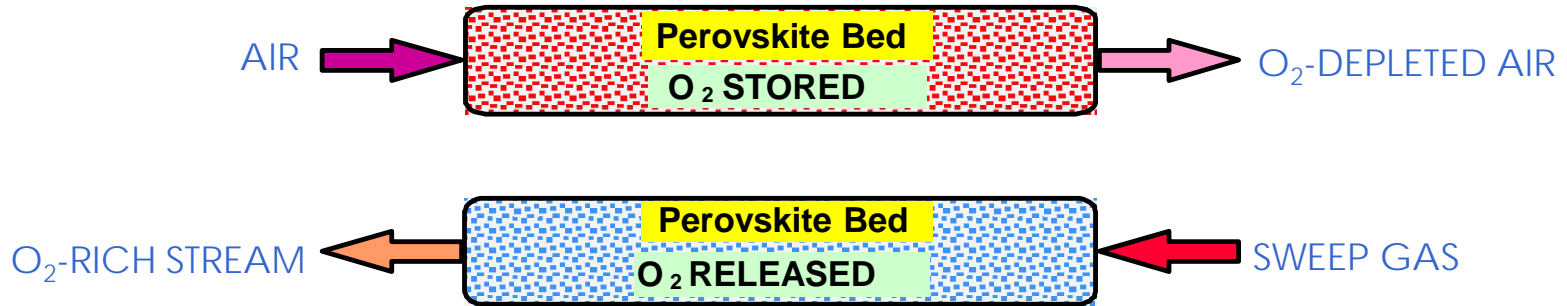


- Adsorb O<sub>2</sub> from air in a solid sorbent
- Use of CO<sub>2</sub>-rich flue gas as sweep gas allows optimization of the O<sub>2</sub> concentration for oxy-combustion
- Use of vacuum or condensing steam sweep to produce oxygen
- Elevated-temperature process driven by partial pressure of oxygen

# Background

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## LCO Process (Perovskite Sorbent-based Oxygen)



Between 2005 and 2008, under two separate Cooperative Agreements, a two-bed, 60-pph unit was developed by BOC/Linde and tested at EP&G/WRI (Thermosolv LLC). The unit was integrated with an existing 250,000 Btu/h Combustion Test Facility to demonstrate oxy-fuel combustion concepts.

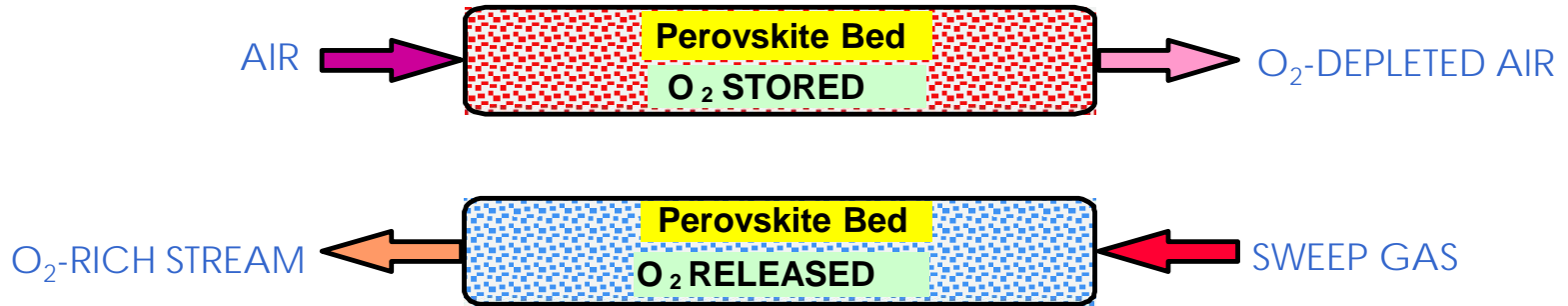
### Conclusions:

- Improve sorbent oxygen uptake capacity
- Lower operating temperature from 850° C
- Improve desorption kinetics



# Background

## LCO Process (Perovskite Sorbent-based Oxygen)



### Project DE-FE0024075 (Completed in Late 2016)

Perovskite(s) with order-disorder transition ( $\text{La}_{0.1}\text{Sr}_{0.9}\text{Co}_{0.9}\text{Fe}_{0.1}\text{O}_{3-\delta}$ , LSCF1991)

- Lower heat of oxygen sorption
- Improved oxygen uptake capacity
- Lower operating temperature (about 500° C)
- Improved desorption kinetics
- CO<sub>2</sub> sweep can provide oxygen for oxy-fuel combustion
- Using air sweep enriched air can be provided for commercial applications
- VPSA cycle optimized to demonstrate 95% pure oxygen

# Project FE28002

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## Goal

Develop and demonstrate an advanced oxygen production technology for use in coal-fed gasification plants. The specific technical objectives are to scale-up the low-cost oxygen production based on vacuum pressure swing with high-temperature perovskite sorbent, evaluate performance as a function of operational parameters, and perform cyclic adsorption/rinse/desorption experiments to demonstrate oxygen production rate and purity.

## Scope of Work

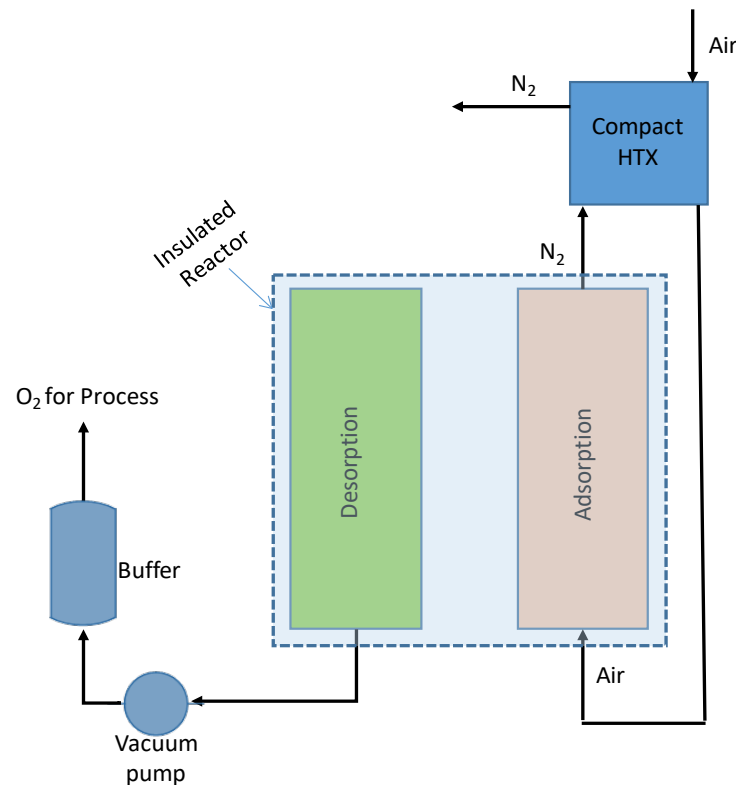
- Upgrade an existing bench-scale test setup to include provisions for modified rinse and desorption steps.
- Optimize the adsorption, rinse, desorption cycles as a function of operating temperature and pressure.
- Based on the results from bench-scale testing, develop a simulation model
- Using the model design a reactor and oxygen process of nominal 1-ton/day capacity
- Construct, debug and operate the 1-ton/day oxygen production facility to perform parametric tests
- Perform long-term performance tests to establish sorbent durability and service life
- Develop credible process economics for small-scale modular coal gasification power plants in the less than 5MW size range.



# Project DE-FE0028002

## Low-cost Oxygen for Small-scale Modular Gasification

- Stand-alone >95% purity oxygen process for small-scale modular < 5MW coal gasification plants
- Design, build and operate a 1-tpd Oxygen Plant

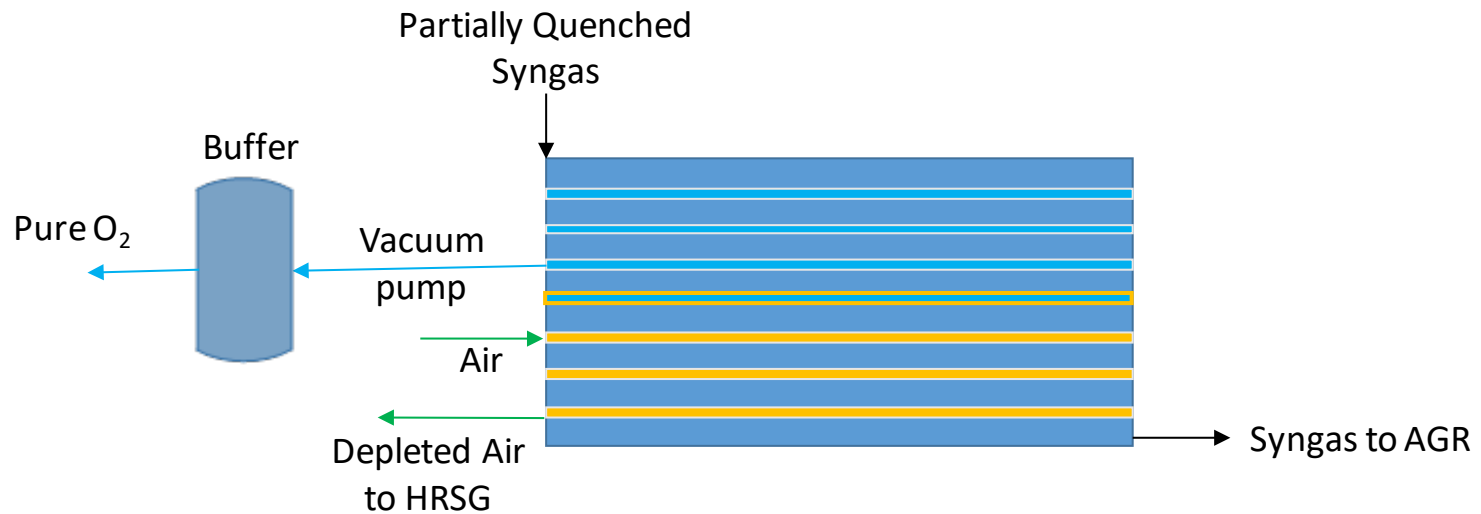


# Project DE-FE0028002

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Full integration within a plant is possible

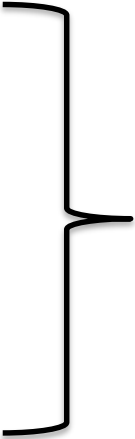
- Nearly adiabatic cycle
- Low pressure process reduces CAPEX and OPEX



# Process Design

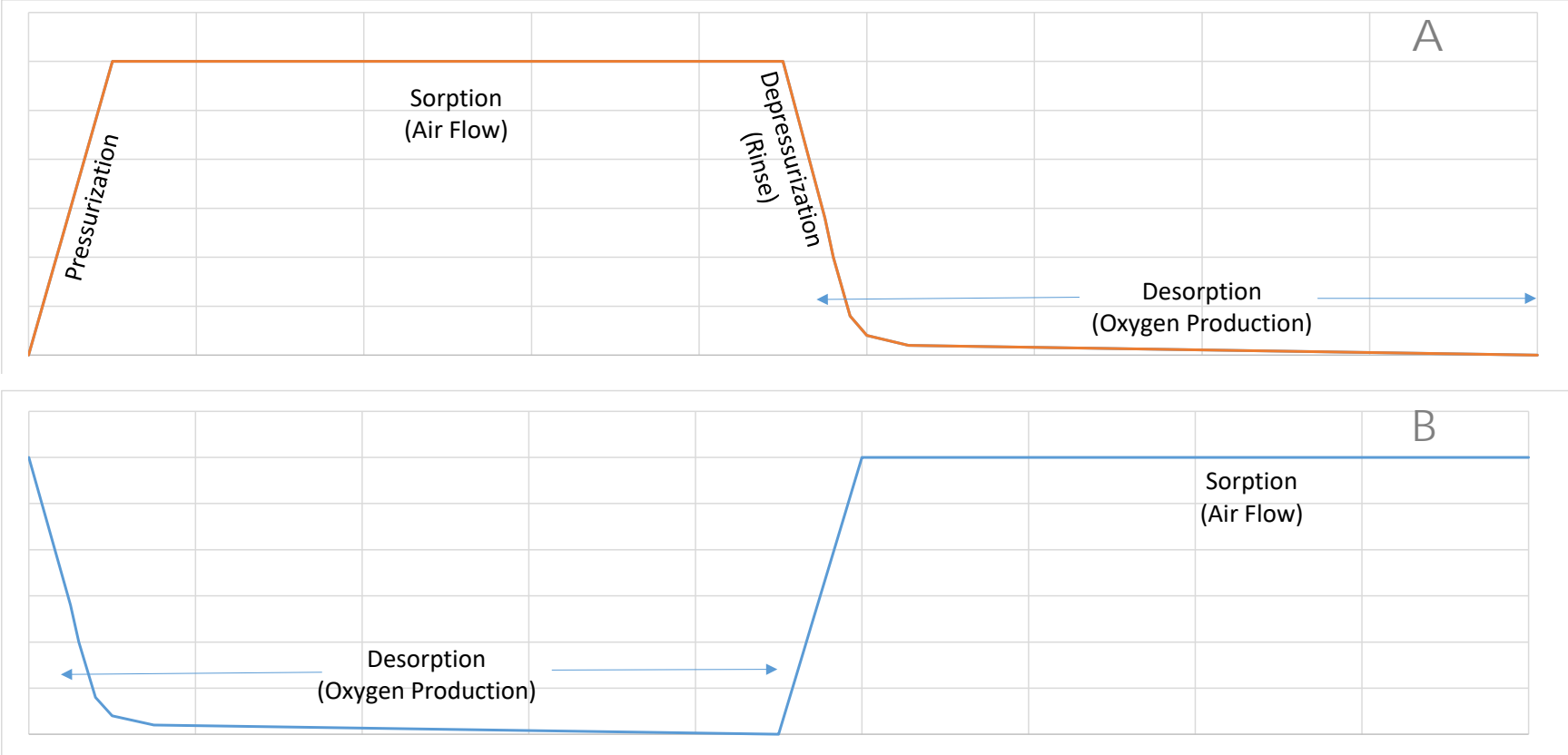
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## Important Properties/Variables...

- Sorbent Capacity, Kinetics, Density
  - Sorption Pressure
  - Pressure Swing
  - Cycle Time
    - Sorption
    - Desorption
    - Rinse
  - Packing Length
  - Packing density
  - Temperature
- Material Properties
- Plant/Equipment Design
- Integration
- 




# Project FE28002

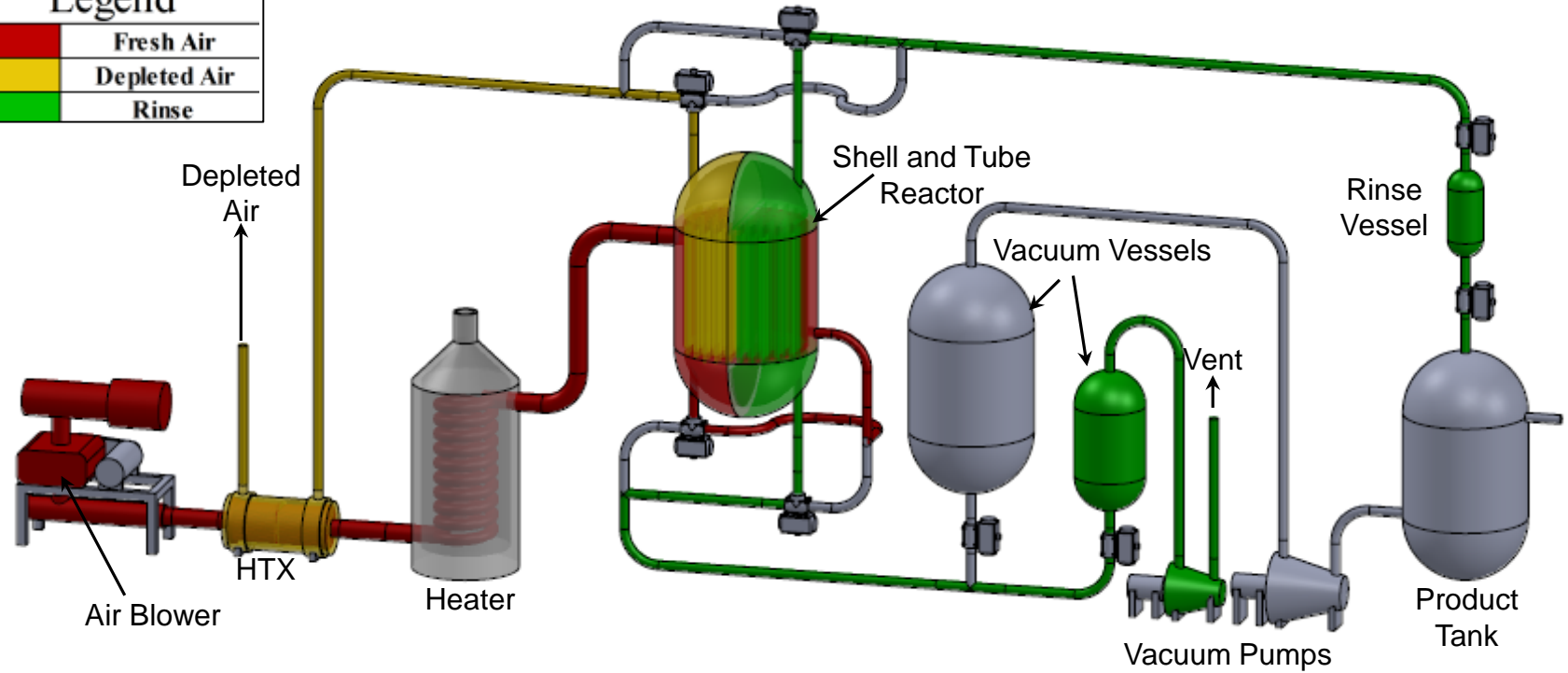
## Two-Bed VPSA Cycle



# Project FE28002

## Two-Bed VPSA Cycle




Legend	
	Fresh Air
	Depleted Air
	Rinse

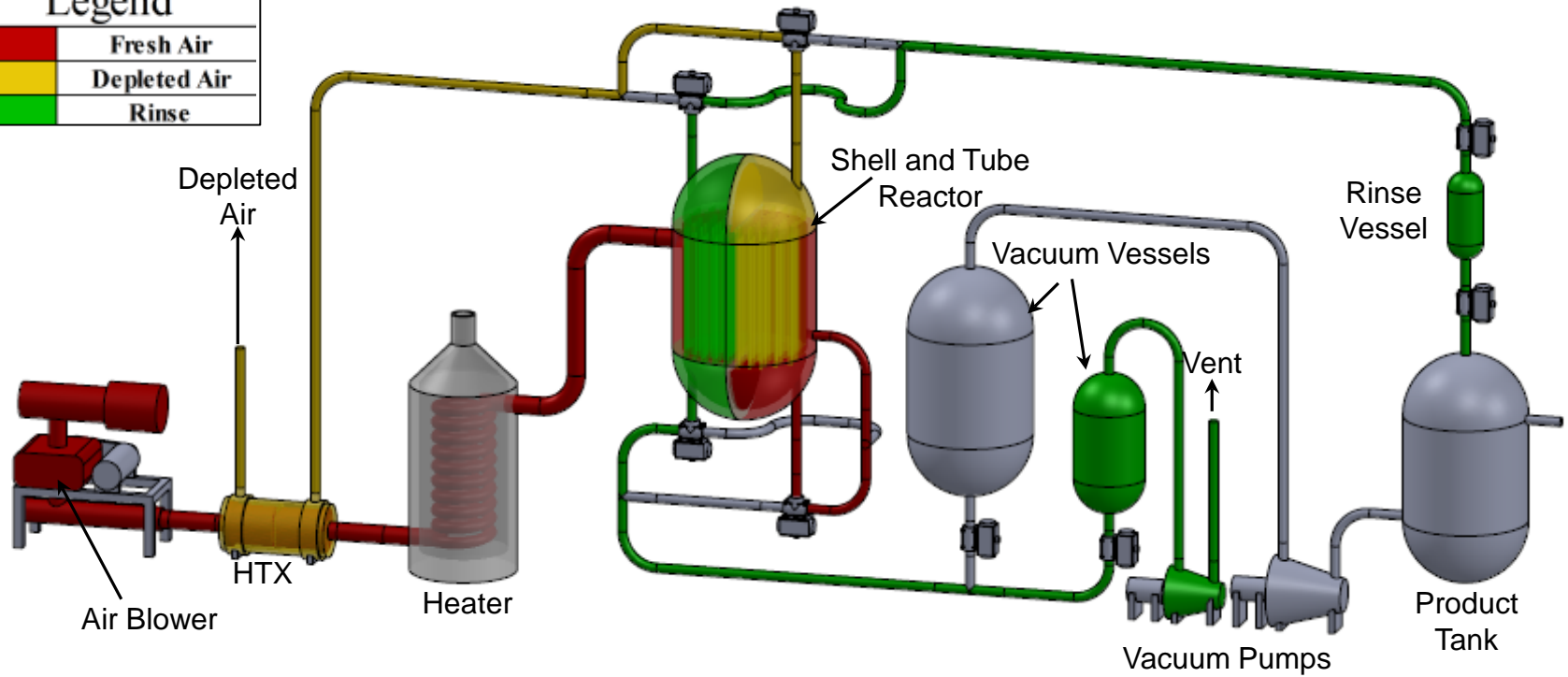




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## Two-Bed VPSA Cycle

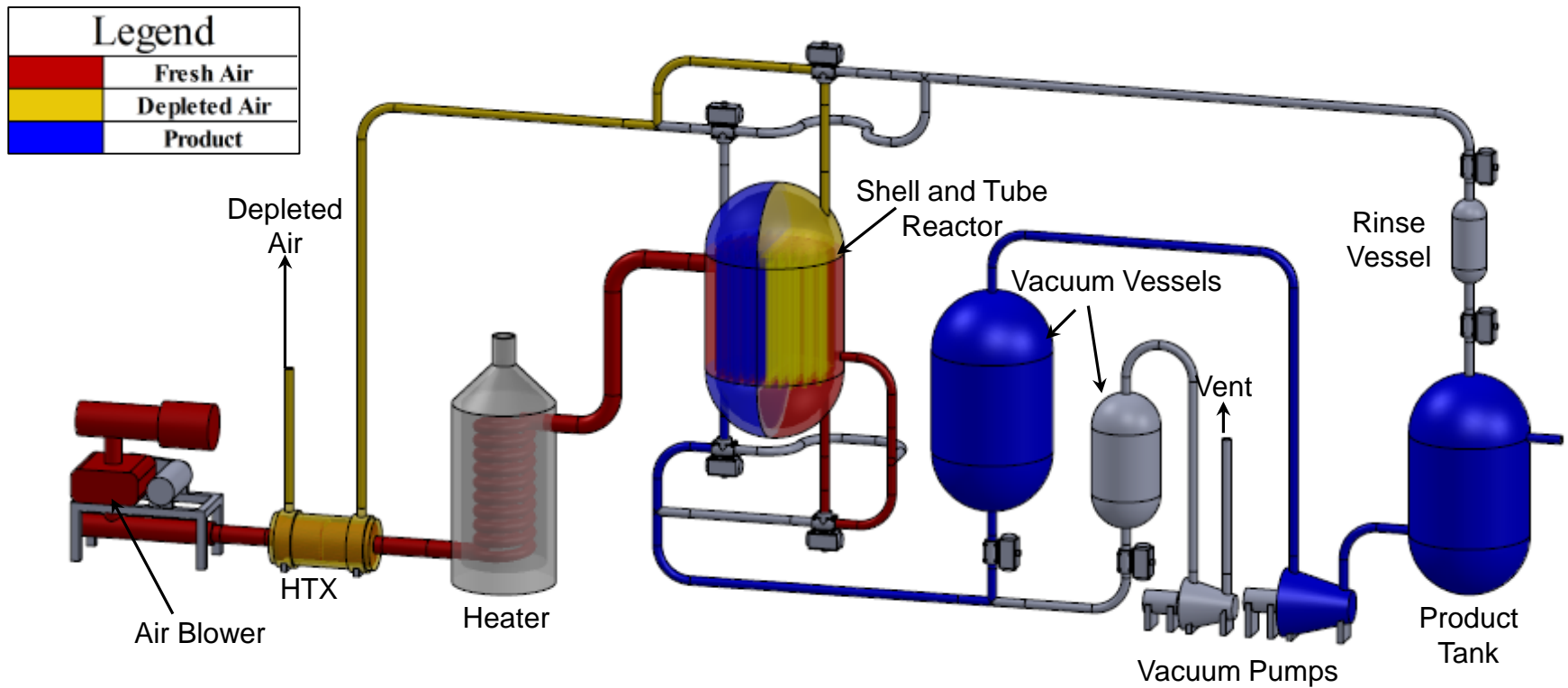
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# Project FE28002

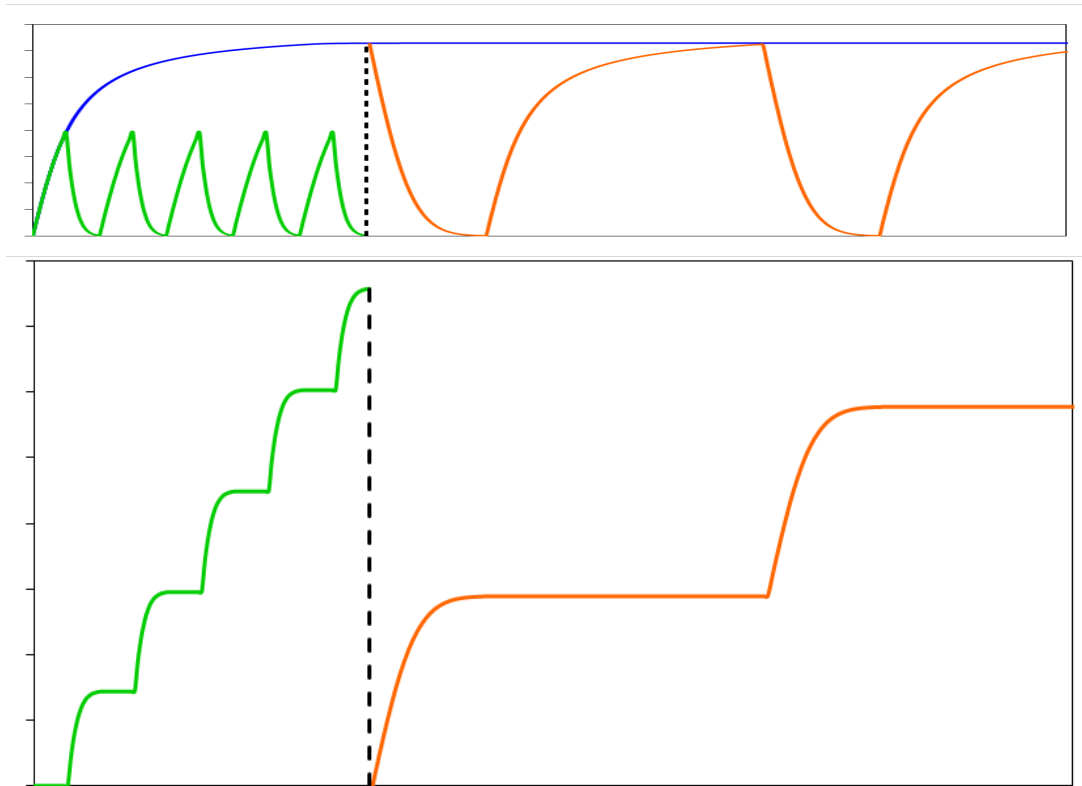
## Two-Bed VPSA Cycle



# Process Design

## Cycle Optimization

- Faster cycle times increase production  
*(equipment limitations and sorbent pack ( $\Delta P$ ) limit how fast)*

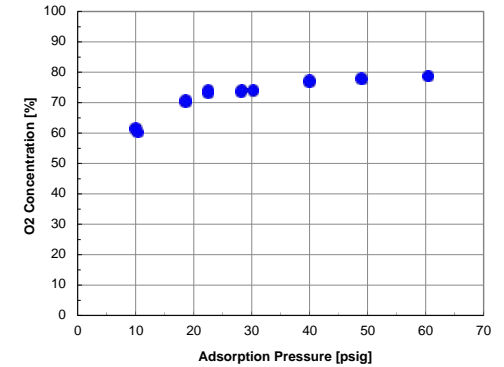
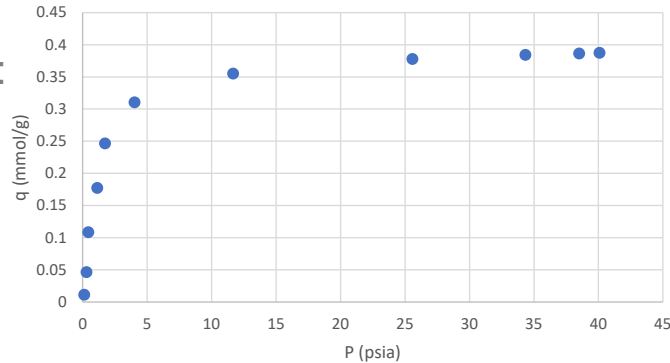


# One Ton/Day Plant

## Design Basis

- Sorption Pressure:

- Use PD blowers
- 20 psig



- Blower Capacity:

- 300 SCFM

3X Theoretical

- Reactor :

- 0.8 m<sup>3</sup> (~1,500 kg Sorbent)
- Shell and Tube
- 2" dia. 40" long tubes
- Refractory-lined

Bench Data

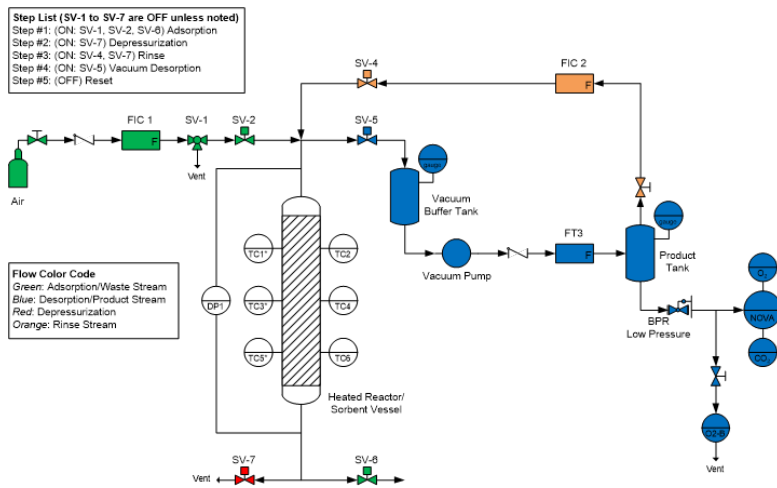
- Cycle Time:

- 2:1 sorption to desorption time (three sorbent beds)
- 180 to 270 s/cycle

Bench Data

# Bench-Scale Testing

- Bench-scale unit operated to determine optimal cycle structure, temperature, pressure, and required sorbent quantity
- Utilized to determine efficacy and stability of multiple sorbent samples
- Generated isotherm data for modelling purposes
- Bench testing completed successfully
- Through testing, determined that required sorption time is longer than desorption time
- Determined that pelletized sorbent is stable for several thousand hours
- Guided the design of the pilot to a 3-bed system which should give the highest chance of project success



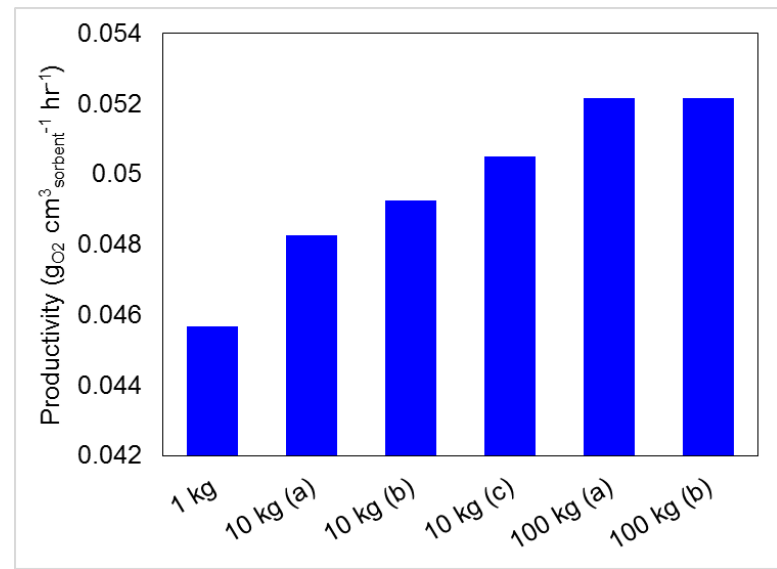
# Sorbent Manufacturing

- Sorbent manufacturing was scaled from gram quantities to kilogram quantities
- Methods and procedures were transferred to a commercial partner for scaling to hundreds of kilograms
- Sorbent manufactured at scale has similar productivity to lab-synthesized sorbent
- Final pelletized sorbent has required mechanical properties and sorption kinetics
- Final manufacture of the sorbent is complete



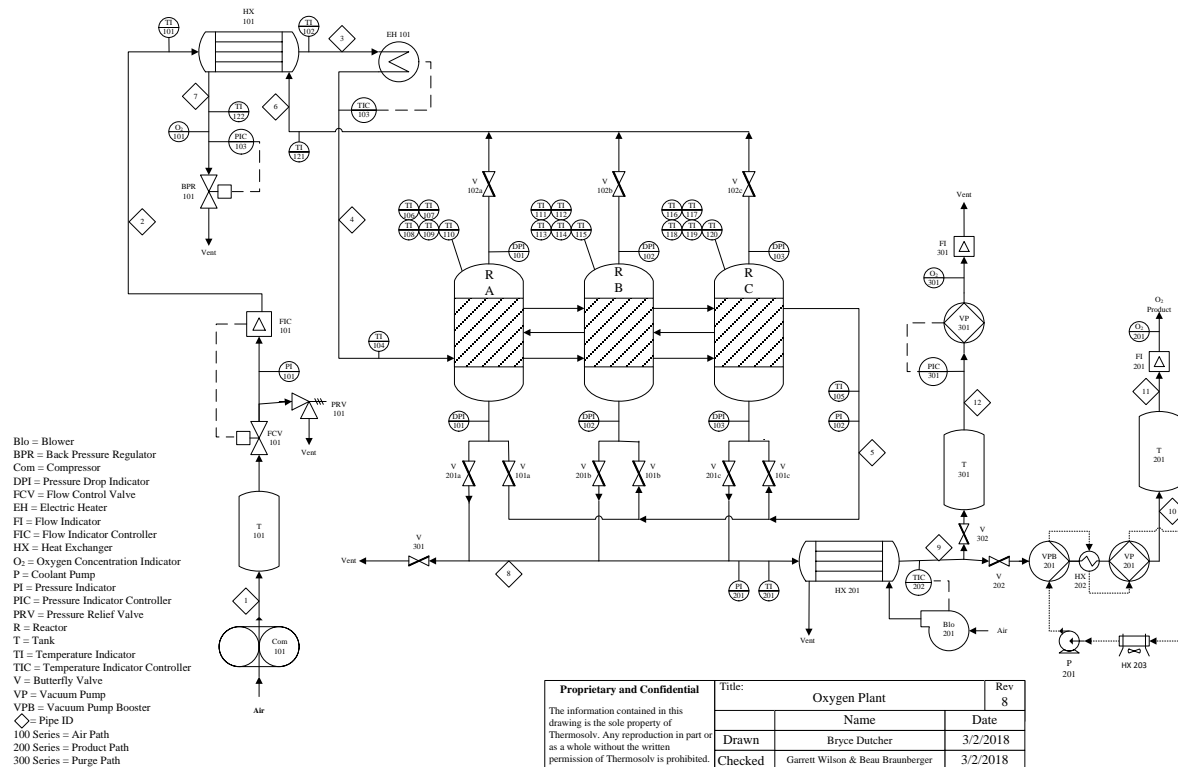
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# Equipment and Process Design

- Required instrumentation located and specified
- Cycle structure mapped and simulated
- Safety review conducted to determine safe operational envelope
- Emergency mode operations designed and programmed into process logic

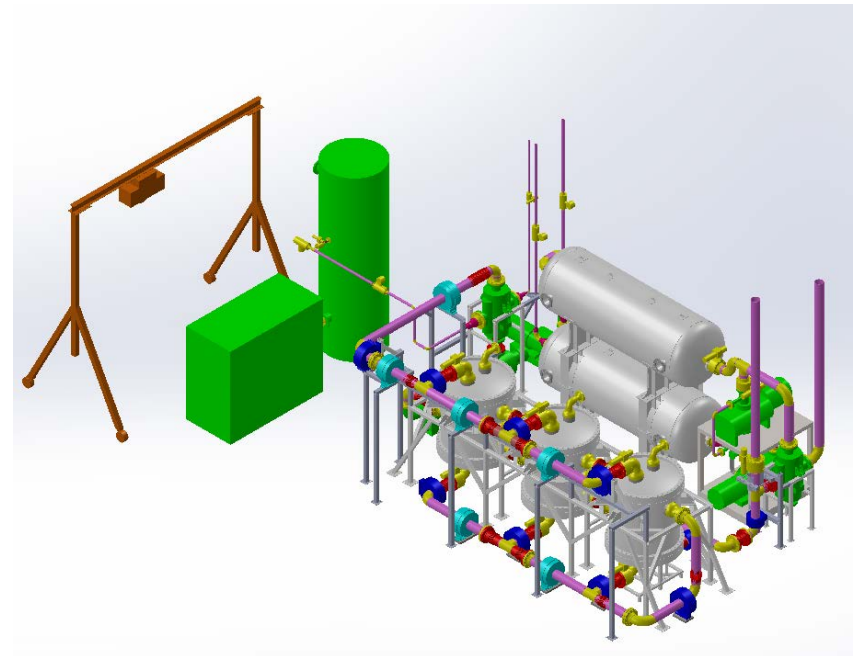




# Equipment and Process Design

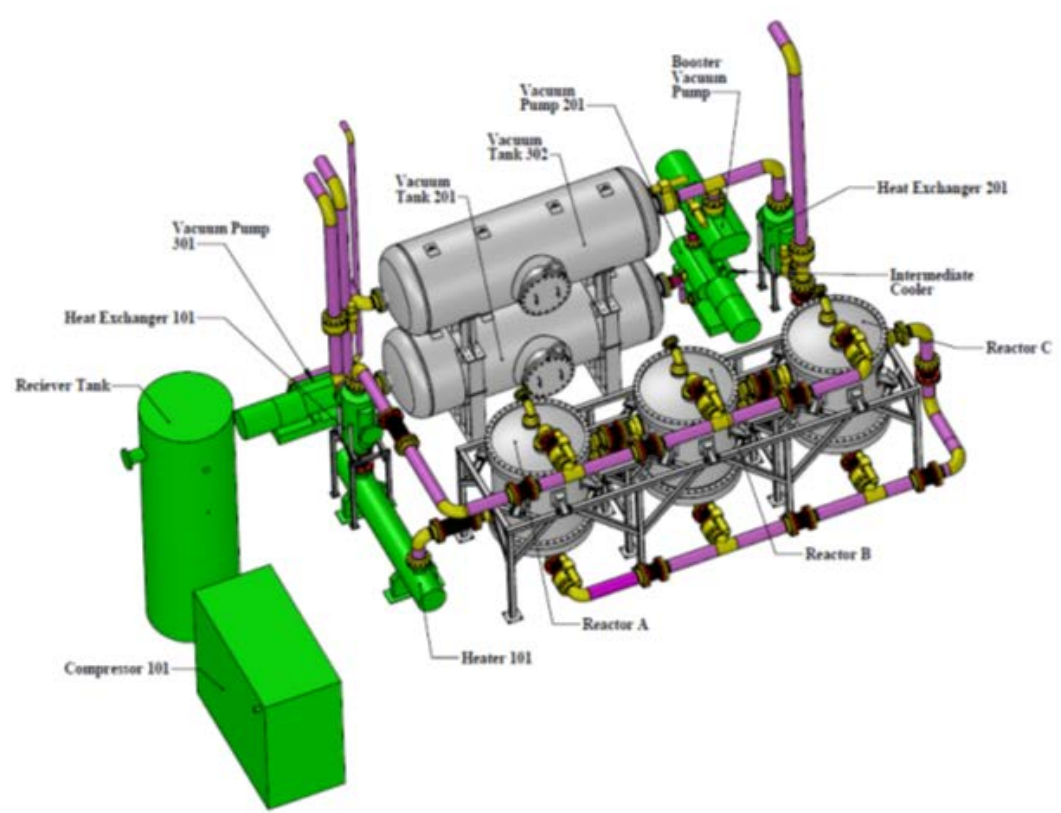
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- Entire plant designed
- Sorption vessels require 0.8 m<sup>3</sup> pelletized sorbent to achieve project goals
- Controls architecture and programming designed
- Metallurgy examined for temperature and oxygen stability
- Major and minor equipment items specified



# Equipment and Process Design

- Simulations of final design operations show capable of successfully meeting project goals



# Plant Fabrication

## Current Status

- Plant is undergoing final shakedown
- All equipment has been tested and programmed
- Refractory has been cured up to reaction conditions
- Reactor loading and operation scheduled



## Key Challenges

- Fabricated sorption reactors did not meet specified tolerances and required repair
- Commercial sorbent did not meet initial performance requirements



# Risk Management

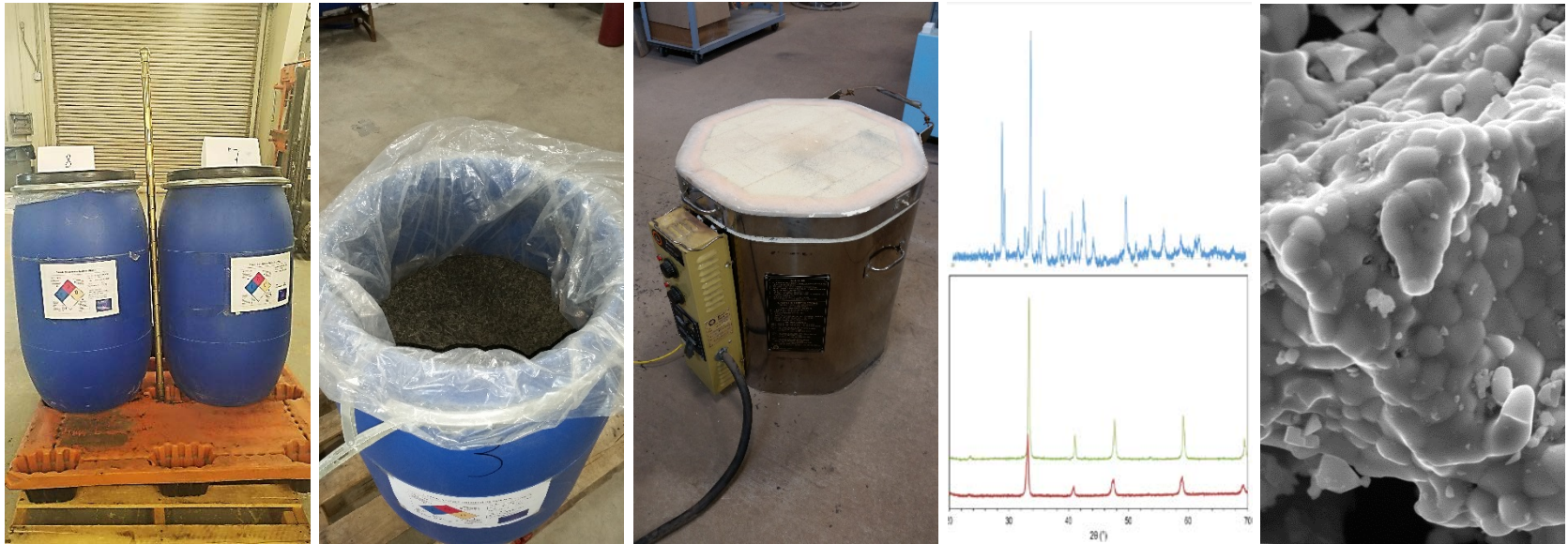
- Remaining major risk is to the schedule due to equipment unsuitability
- Tongue and groove seal between reactor shell and tube sheet was too long. This would result in excessive sealing pressure to the sealing faces when thermally cycled
- Refractory lining of shells was out of specified range and not concentric within the vessel
- All affected parts have been returned to the fabricator, and repairs have been completed





# Risk Management

- Major risk to project due to low initial performance of sorbent
- After characterization, it was found that the phase purity was inadequate
- Testing showed that additional calcination would restore the material to satisfactory performance
- $\frac{1}{4}$  of the material was sent back to the manufacturer for repair
- Remaining material is being processed at Thermosolv



# Risk Mitigation - Reactors

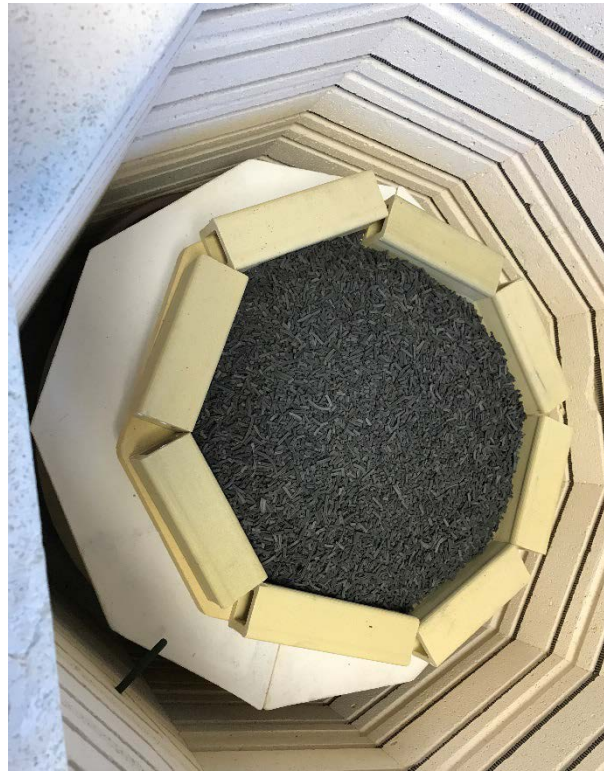
- Sent bottom reactor heads back to the fabrication shop for alterations
- Removed some of the refractory lining to make concentric for the tube bundles
- Shell/tube seal face modified for higher compression seal material
- Thermal imaging being utilized to ensure even temperature distribution throughout the shell





# Risk Mitigation - Sorbent

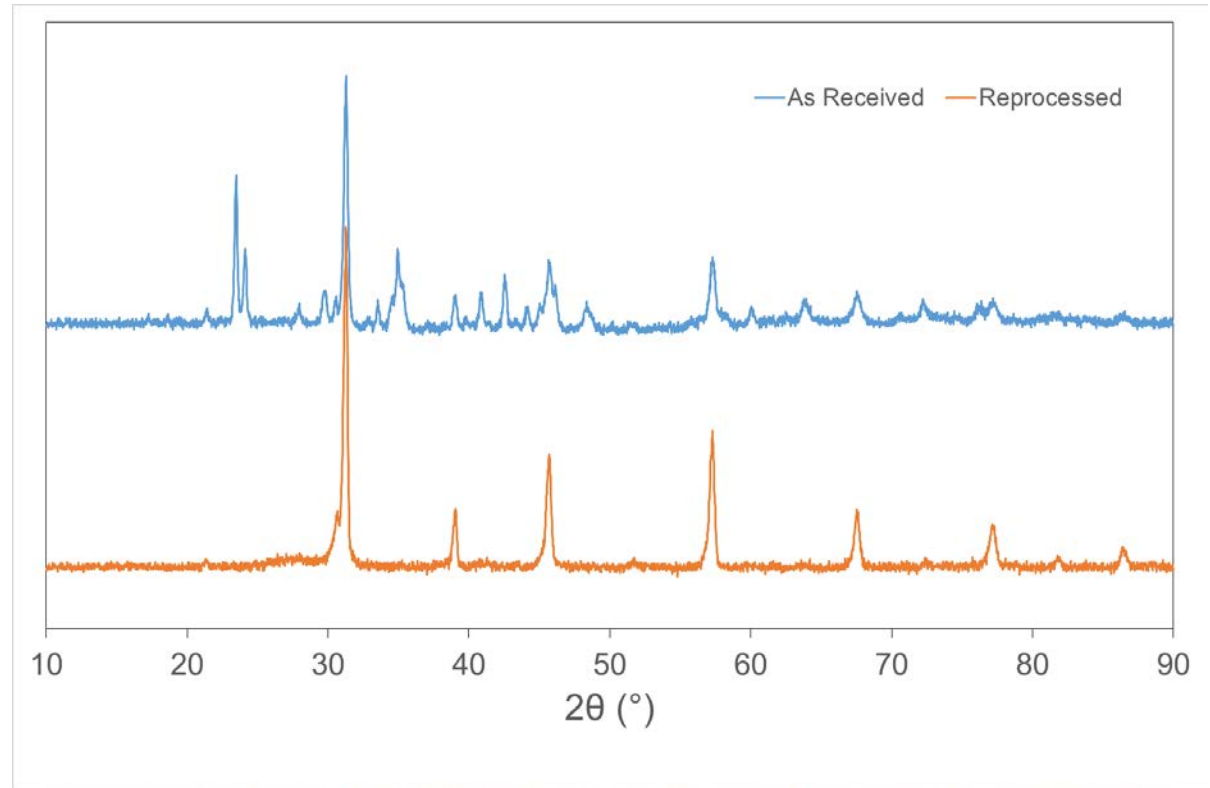
- Testing showed that 8 hours at 1100C would improve material performance by 35%-50%
- Two kilns are being operated around the clock to reprocess the material
- Manufacturer took back  $\frac{1}{4}$  of the material for processing
- Full thermal treatment of the necessary fill volume will be completed next week





# Analytical Results - XRD

- XRD reveals phase purity

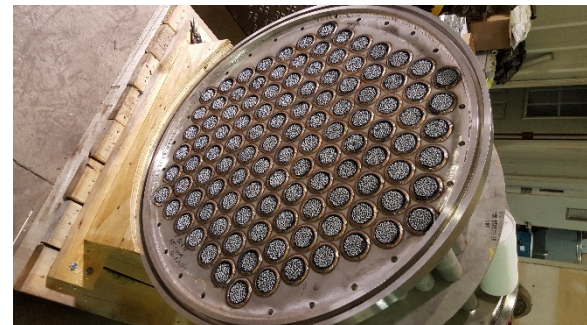


# Plant Construction and Design Features



# Plant Construction and Design Features

- Tubesheet constructed of 304H SS for thermal stability
- Support plate utilized to maintain tube integrity
- SiC filter material utilized to keep sorbent in place
- Baffles allow for even thermal distribution throughout system
- Support struts to maintain bed integrity on top tubesheet





# Plant Construction and Design Features

- Reactors hung from reactor shell to allow for vertical thermal expansion
- Roller-pad supports for horizontal thermal expansion
- Strain relief at all major joints to protect rigid plumbing
- Shells of all reactors linked to allow for even heating across all reactors



# Plant Construction and Design Features

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- Fully integrated support system for reactors and plumbing
- Access for floor crane for reactor and tubesheet maintenance



# Plant Construction and Design Features

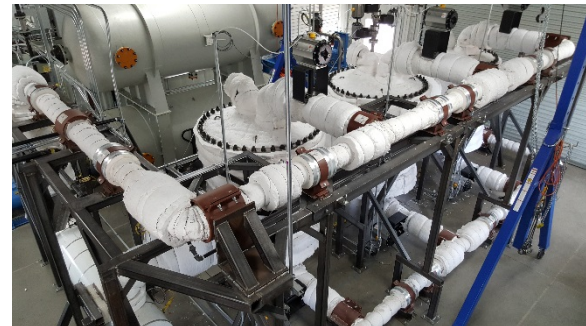
- Insulated piping guides and strain relief for safety and longevity
- Flanged construction for ease of maintenance and alterations





# Plant Construction and Design Features

- Electrical heat for main process heating
- Heat exchange for recovery from depleted air
- Fully insulated system for process efficiency and safety
- All subsystems have been commissioned



## *Path Forward*

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- Final thermal testing – This week
- Intermediate reactor seal testing – Next week
- Begin reactor fill and tuning – Anticipated April 22nd
- Final sintering of sorbent material – Anticipated April 22nd
- First full shakedown run – Anticipated April 29th



# Acknowledgements

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- Department of Energy – Advanced Energy Systems Team
- Steven Markovich – Project Manager
- LP Amina – Modelling and Economic Analysis
- Western Research Institute

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*Questions?*