Hot/Warm Gas Cleanup

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Introduction

Using regenerable sorbents and transport or fluid-bed contacting, the Gas Process Development Unit (GPDU) at NETL-Morgantown will be used to demonstrate the process feasibility of removing sulfur from coal gasification or other fuel gas streams at temperatures above dew point of the gas. This technology, also known as hot or warm gas desulfurization, is expected to remove sulfur to concentrations lower than conventional systems at comparable cost. The project was constructed under the U.S. Department of Energy (DOE) Integrated Gasification Combined Cycle (IGCC) power system program and is an "enabling technology" in the Vision 21 program.

Objectives

The GPDU was designed to be the smallest scale research and development facility capable of providing viable scale-up design data for new integrated transport or fluid-bed desulfurization processes. With the capability to test at process conditions representative of anticipated commercial applications in terms of temperatures, pressures, major compositions, velocities, and sorbent cycling, the unit is expected to generate important information on process control, configuration, and sorbent suitability. In this way, the GPDU fills a strategic role between past/current small-scale testing and large-scale demonstrations.

A primary objective of the project is to gain insight into which reactor combination (i.e., both transport, both fluid bed, or mixed) is more suitable for desulfurization technology and why. Assuming process feasibility is demonstrated, this guides future development or commercial ventures by answering the question of what to build, and provides performance and scale-up data (e.g., required transport reactor densities). Another important objective, which naturally derives from the process development activities, is demonstration of sorbent suitability and readiness for commercial deployment (e.g., sorbent attrition and cycle life). In this sense, the GPDU can serve as a final testing ground to reduce the risks of large-scale sorbent failure.

Project Description

The GPDU is designed to conduct continuous desulfurization at temperatures up to 1200°F and pressures up 385 psig and can study four different process configurations or combinations of transport and fluid-bed reactors. Slide valves regulate sorbent circulation (up to 5,000 lb/hr) between the absorber (where fuel gas desulfurization occurs) and the regenerator (where sorbent is regenerated with air) and also control sorbent recirculation (up to 55,000 lb/hr) in transport configurations. Depending upon sorbent density and process considerations, generally about 2,000 to 3,000 pounds of sorbent are required for testing. Sulfur-laden fuel gas (also known as syngas) for GPDU operation is produced in an NETL-designed Syngas Generator (SGG). Natural gas is combusted with air in the presence of sulfuric acid (which converts to H_2S) to produce fuel gases with an expected composition similar to product gases produced by air-blown coal gasifiers. The SGG outputs up to 150,000 scfh (9,900 lb/hr) of fuel gas at temperatures up to 1,500°F and pressures up to about 600 psig.

Further details, parameter ranges, and pictures are available under the “Research Facilities and Equipment” link at www.netl.doe.gov/products/r&d/index.html.

Approach

The current approach to get the project fully operational is independent shakedown of the separate units (GPDU and SGG) followed by integrated shakedown. The general goal of independent shakedown is to end up with a sufficiently operable and characterized system and adequately trained personnel to proceed with integrated shakedown. This means demonstration of hot solids with steam in the GPDU, adequate sulfur-laden fuel gas production by the SGG, and training of roughly 14 operating personnel.

Both units progressed through various precommissioning and commissioning activities before the start of independent shakedown. Precommissioning activities involved non-operating adjustments and cold alignment checks, and verification that construction was in accordance with drawings, specifications, instructions, and applicable codes and regulations. Precommissioning included system mechanical checkouts, instrumentation/safety interlock system checkouts, and line clearing. Commissioning activities were those associated with the operation of items of equipment or facilities in preparation for actual startup/shakedown, and included leak testing, initial flow proving, and initial run-in of major equipment items (e.g., preheaters, incinerator, flare).

The ongoing sequence of shakedown activities is given below. Sub-bullets define some of the activity objectives.

GPDU Independent Shakedown Activity Sequence

1. Sorbent loading
    - Successfully load sorbent into feed hopper/receiver
    - Demonstrate feed hopper/receiver fluidization and inventory check
    - Demonstrate operability of feeder and feeder purge
    - Demonstrate sorbent convey into regenerator vessel

2. Cold circulation with air
    - Demonstrate sorbent fluidization and recirculation on each reactor side (i.e., absorber and regenerator)
    - Demonstrate sorbent circulation between absorber and regenerator
    - Validate procedures for getting the unit up and down with sorbent in it
    - Gain initial insight into operating characteristics and control
    - Identify problems that will need addressed mechanically and/or operationally

3. Sorbent removal

- Remove as much sorbent as possible from system
- Determine sorbent recovery factor
- Get samples for size analysis to determine attrition that occurred

4. System heatup with air (no solids)
   - Get the unit as hot as practical with preheated air
   - Dry out refractory of fired devices and reactors
   - Cure refractory field joints (i.e., flanges) for steam service
   - Confirm process piping thermal expansion design to temperature achieved
   - Confirm reactor refractory integrity and circulation piping thermal design to temperature achieved
   - Determine leak tightness at temperature

5. System heatup with steam (no solids)
   - Check for cold spots and condensation problems
   - Confirm heat tracing performance

6. Cold circulation with air
   - Address any problems or test modifications stemming from earlier (activity #2) circulation testing

7. Hot circulation with air
   - Demonstrate operation/control at elevated temperatures
   - Verify circulation temperature uniformity
   - Further assess sorbent durability

8. Hot circulation with steam
   - Demonstrate hot solids in system with steam
   - Demonstrate system startup to SGG gas phase-in
   - Demonstrate target mode for integrated shakedown
   - Commission other systems (e.g., scrubber, analytical)

SGG Independent Shakedown Activity Sequence

1. Initial light-off
   - Demonstrate light-off and stable operation of first-stage pilot and axial burner
   - Demonstrate governing operational interlocks

2. SGG startup
   - Achieve startup temperatures at low pressure
   - Prove interlocks governing startup sequence
   - Demonstrate operational mode transitions (e.g., combustion mode to syngas mode) at low pressure and safe firing system
   - Verify adequacy of GPDU protection interlock
   - Dry out SGG combustion vessel and transfer line (to GPDU) refractory
   - Confirm refractory integrity and unit thermal design to temperature achieved

3. Syngas production
   - Demonstrate sulfur-laden flow and gas composition needed for integrated shakedown
   - Demonstrate operational mode transitions at high pressure

Integrated Shakedown

This activity sequence will be defined towards the end of the above activities to take full advantage of knowledge gained during independent shakedown. In addition to demonstrating SGG-GPDU integration and desulfurization/regeneration, integrated shakedown will begin to establish operational bounds of the system.

Accomplishments and Results

Significant shakedown progress has been made during the last year and the unit is nearing full operational status. Here are some of the recent major accomplishments:

1. **Demonstration of sorbent circulation/recirculation at ambient temperature** - About 2,400 pounds of EX-SO3 sorbent (a zinc titanate sorbent developed by Research Triangle Institute and Intercat Development, Inc.) was loaded into the GPDU and fluidized/circulated/recirculated with air and nitrogen for about 42 hours. Continuous circulation was demonstrated between the absorber and regenerator, with simultaneous recirculation to both risers. Typical gas velocities were around 1 ft/s in the beds and around 15 ft/s in the risers. System pressure was varied from 35 psig to 85 psig. Smooth operations at densities up to about 5 lb/ft^3 in the absorber riser were demonstrated. Some reduction in mean particle size was observed. This initial testing validated the GPDU circulation system design and suggested that no mechanical design changes were needed for continued shakedown.

2. **GPDU heatup (without solids) to nominally 50% of design operating temperature** - The GPDU was heated at pressure using hot air from both fired preheaters in the system. All three primary objectives of the heatup were achieved: 1) dryout of the refractory in the fired devices and reactors, 2) confirmation of reactor refractory integrity and circulation piping thermal design, and 3) confirmation of process piping thermal expansion design. There were no discernable problems during heatup and mechanical integrity of the system looked good. Solids were not used in the system for this initial heatup because of refractory dryout considerations. Some approximate process conditions reached during the heatup are as follows:
   - Reactor pressure: 150 psig
   - Absorber refractory: 600-750°F
   - Absorber riser: 650-700°F
   - Regenerator refractory: 450-750°F
   - Regenerator riser: 550-700°F
   - Fuel gas filter inlet: 540°F
   - Regeneration gas filter inlet: 545°F
   - Inert gas preheater outlet: 1000°F
   - Air preheater outlet: 1000°F

3. **GPDU operation with steam (no solids)** - In a simulation of actual startup, the GPDU was initially heated with hot air from both fired preheaters, and then steam at 850°F was phased in. The primary purpose of this successful, short-duration test of steam in the unit was to determine if there are cold spots and condensation problems in the system. This was done without solids in the system to avoid the problems that solids and condensate (if any formed) would cause.

4. **Demonstration of SGG startup operations** - The first-stage burner was fired with natural gas and air to dry out refractory in the SGG combustion unit. Through controlled temperature ramps and holds, this culminated in first-stage temperatures of 2000°F, third-stage temperatures of 800°F, and combustion unit outlet temperatures up to about 1400°F. Various process parameters and procedures were investigated to achieve reliable ignition, good flame sustainability, and temperature control; and key aspects of startup were successfully demonstrated.

Future Activities

Shakedown will culminate in a demonstration test of continuous transport desulfurization and regeneration at design operating conditions with a zinc titanate sorbent. This approach follows recommendations from a group of industry representatives who participated in an external review of the project in April, 2001.

After shakedown and initial demonstration is successfully completed, the future test program (likely to begin summer, 2002) depends upon programmatic direction and could include the following kinds of desulfurization tests:

- Baselining continuous transport or fluid-bed performance at design temperatures (1,000°F – 1,200°F in the absorber).
- Investigation of transport or fluid-bed performance at "warm" temperatures (i.e., 500°F – 800°F in the absorber), initially in batch mode only.
- Demonstration of adequate safety and control needed for continuous "warm" testing.
- Baselining continuous transport or fluid-bed performance at "warm" temperatures.
- Investigation of conditions more representative of oxygen-blown gasification by adjusting SGG operation to achieve higher H_2S and moisture concentrations.

Testing will also begin to reveal the suitability of the equipment for alternate uses, such as desulfurization in other applications (e.g., refinery streams, pulp and paper processes) and advanced fuels and chemicals production.

The DOE is interested in industrial participation with the facility. In addition to desulfurization technology development and sorbent testing, the GPDU offers cooperative research and development opportunities in the general areas of reactor systems and process technology. Through cost-sharing arrangements, an industrial partner can gain testing access to the GPDU and secure intellectual property rights.