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Advanced Sulfur Control Concepts for Hot Gas Desulfurization Technology

Quarterly Technical Progress Report

Submitted to

U.S. Department of Energy
Federal Energy Technology Center
3610 Collins Ferry Road
P.O. Box 880
Morgantown, WV 26507-0880

Submitted by

Research Triangle Institute
P.O. Box 12194
Research Triangle Park, NC 27709

DOE COR: Thomas P. Dorchak
RTI Project Manager: Santosh K. Gangwal
RTI Project Engineer: Jeffrey W. Portzer

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1. CONTRACT OBJECTIVE:

The objective of this project is to develop a hot-gas desulfurization process scheme for control of H₂S in HTHP coal gas that can be more simply and economically integrated with known regenerable sorbents in DOE/METC-sponsored work than current leading hot-gas desulfurization technologies. In addition to being more economical, the process scheme to be developed must yield an elemental sulfur byproduct.

2. TECHNICAL APPROACH:

The Direct Sulfur Recovery Process (DSRP), a leading process for producing an elemental sulfur byproduct in hot-gas desulfurization systems, incurs a coal gas use penalty, because coal gas is required to reduce the SO₂ in regeneration off-gas to elemental sulfur. Alternative regeneration schemes, which avoid coal gas use and produce elemental sulfur, will be evaluated. These include (i) regeneration of sulfided sorbent using SO₂; (ii) partial oxidation of sulfided sorbent in an O₂ starved environment; and (iii) regeneration of sulfided sorbent using steam to produce H₂S followed by direct oxidation of H₂S to elemental sulfur. Known regenerable sorbents will be modified to improve the feasibility of the above alternative regeneration approaches. Performance characteristics of the modified sorbents and processes will be obtained through lab- and bench-scale testing. Technical and economic evaluation of the most promising processes concept(s) will be carried out.

3. CONTRACT TASKS:

Phase I - Concept Assessment:

Completed.

Phase II:

Bench-Scale Sorbent Testing

Two candidate sorbent formulations – AHI-1 and AHI-2 – that had previously been tested by micro-scale thermogravimetric analysis were tested at laboratory-scale with multiple cycles of sulfidation and oxygen regeneration. Promising reductions of H₂S concentration in the outlet gas were obtained, with AHI-2 performing slightly better and achieving approximately 10 ppm. AHI-1 generally achieved better than 20 ppm H₂S outlet concentration, and always less than 40 ppm. Figures 1 - 4, below, summarize the results of these initial trials.

AHI-1 Sorbent

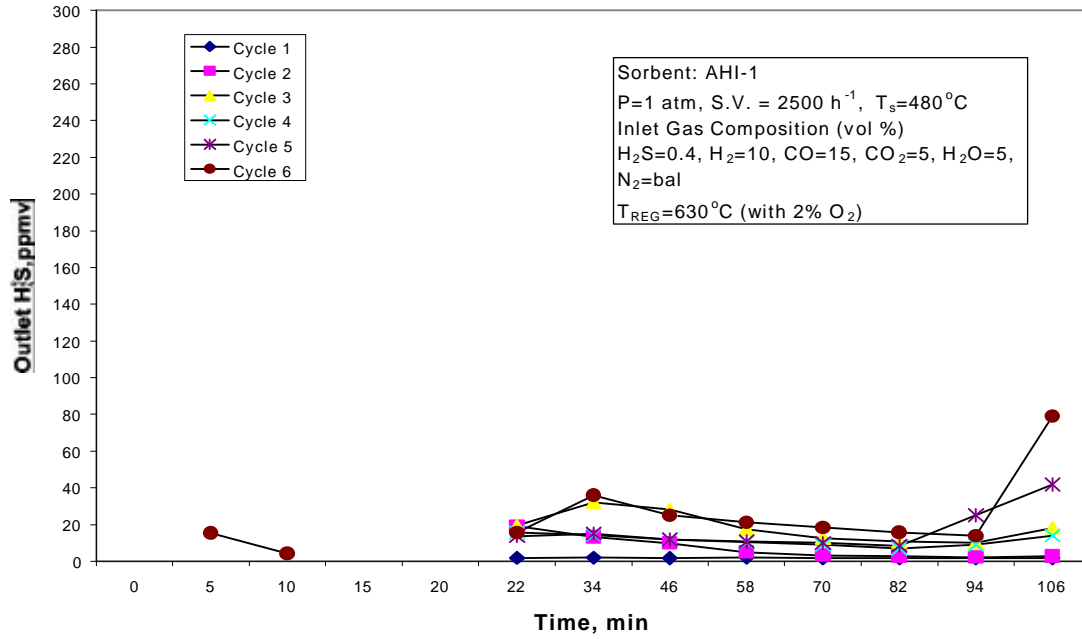


Figure 1. Multi-cycle lab-scale sulfidation of AHI-1 Sorbent showing H₂S leakage of approximately 20 ppm.

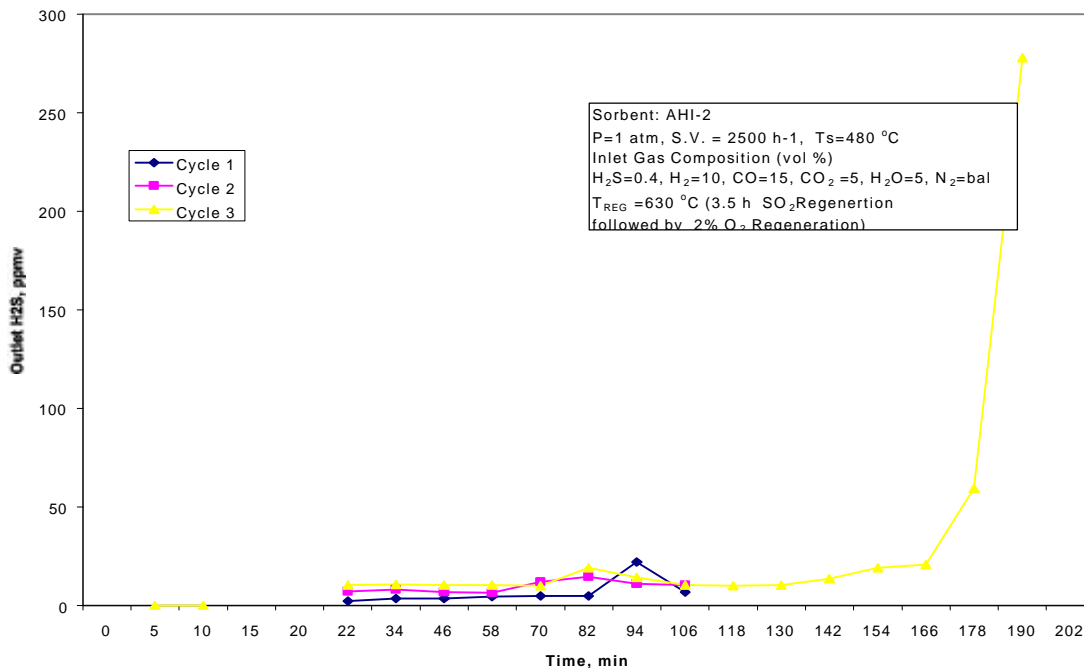


Figure 3. Laboratory-scale sulfidation testing of AHI-2 sorbent including SO₂ regeneration and dilute air regeneration; desired H₂S concentration of <20 ppm is achieved.

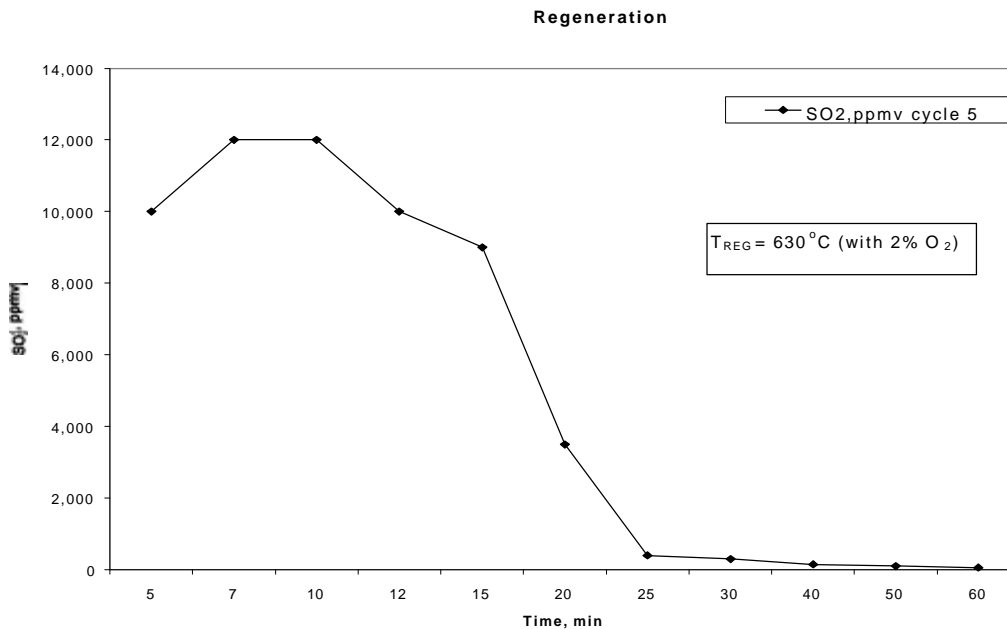


Figure 2. Lab-scale regeneration of AHI-1 sorbent following sulfidation cycle #5 using 2% oxygen in nitrogen.

Laboratory-scale testing of the more promising sorbent formulation – AHI-2 – continued with the addition of an SO₂ regeneration step to the multi-cycle test protocol of sulfidation and oxygen regeneration. The target level of H₂S concentration in the outlet gas continued to be obtained. Two and one-half cycles of testing were completed using the protocol of sulfidation, followed by SO₂ regeneration, followed by dilute air regeneration. Preliminary data analysis suggests that approximately 50% of the H₂S absorbed in the sulfidation step was removed during the SO₂ regeneration step. The remainder was removed by the dilute air regeneration step.

Figure 5, below, shows the H₂S outlet concentration during the sulfidation step. The desired level of less than 20 ppm was achieved in all the cycles. Figure 6 shows the SO₂ evolution during the dilute air regeneration steps of cycles 1 and 2.

Multicycle testing, including SO₂ regeneration, of the AHI-2 material is continuing with 20 cycles planned. The goal is to determine if sorbent efficiency (ability to remove H₂S to less than 20 ppm level in the outlet) and capacity will be maintained following multiple cycles of SO₂ regeneration.

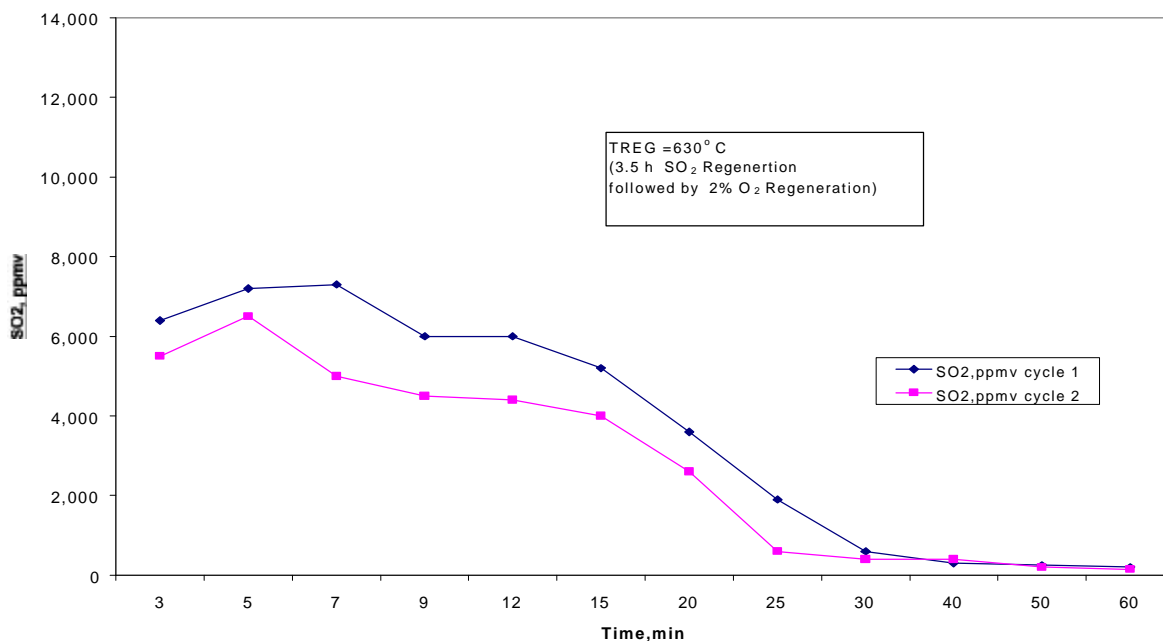


Figure 4. Lab-scale dilute-air regeneration of AHI-2 sorbent following SO₂ regeneration.

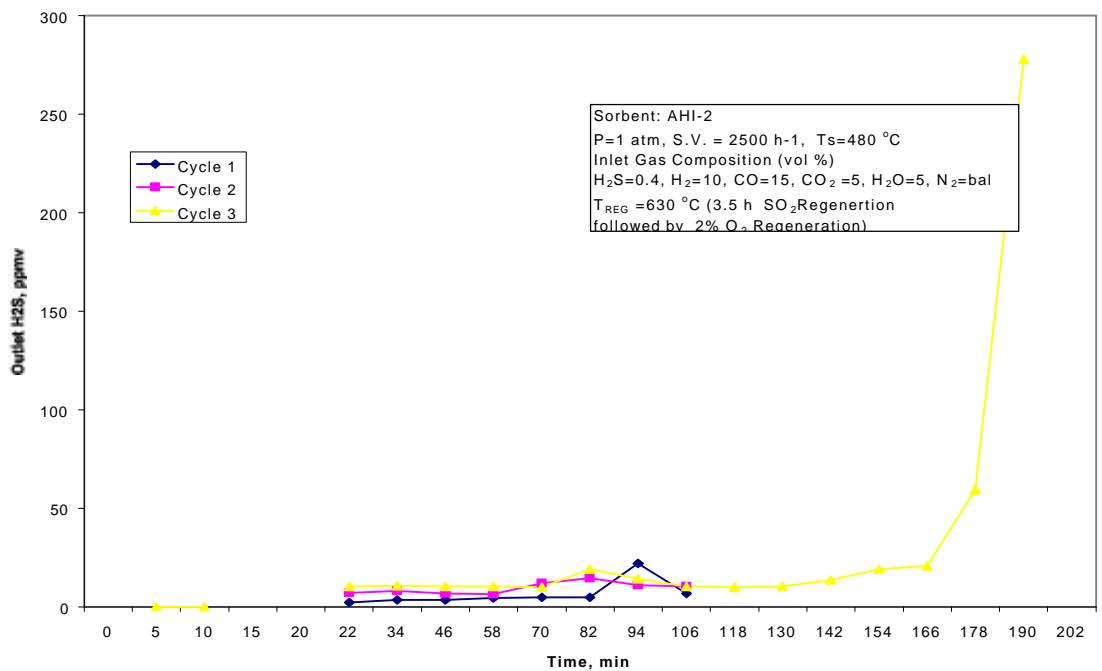


Figure 5. Laboratory-scale sulfidation testing of AHI-2 sorbent including SO₂ regeneration and dilute air regeneration; desired H₂S concentration of <20 ppm is achieved.

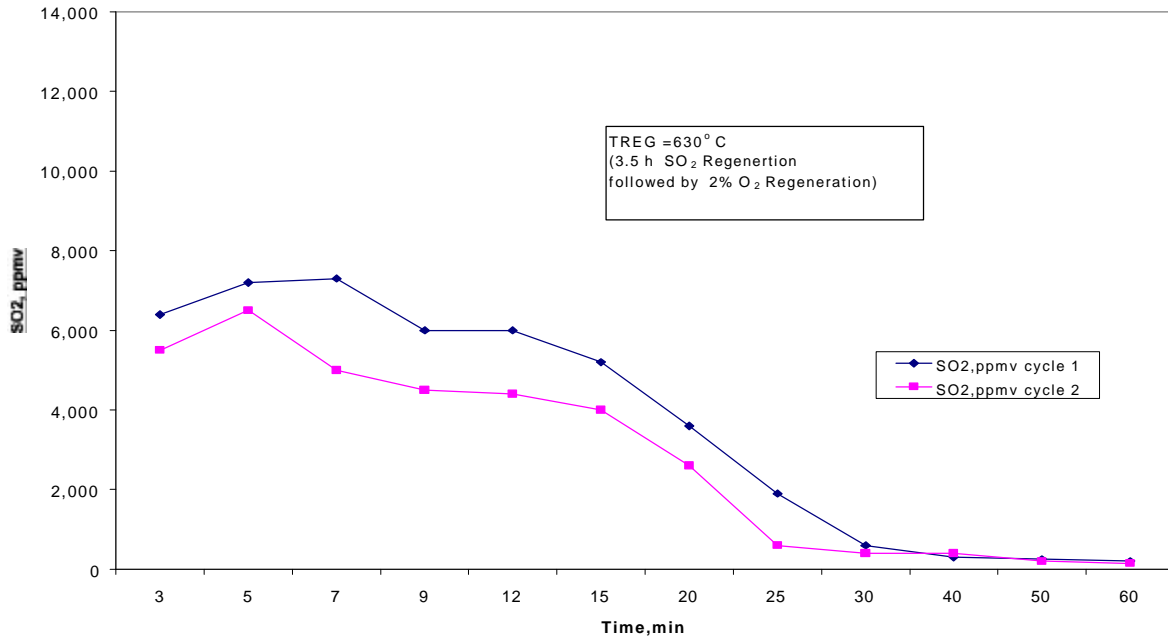


Figure 6. Lab-scale dilute-air regeneration of AHI-2 sorbent following SO₂ regeneration.

PSDF Field Test

Design, engineering, and construction are continuing on the renovation of the Mobil Laboratory for the Advanced Hot Gas Process (AHGP) field test. The fluid-bed reactor vessel was temporarily removed from the trailer, the flange joint ring was replaced, the vessel was hydrostatically tested to verify proper flange sealing, and the reactor was reinstalled with an improved support stand. Process tubing, valves, thermocouples, and instrument air lines that were used with the previous setup were removed. Most of the new process tubing has been installed.

In order to compress the space in the trailer that will be occupied by the AHGP instrumentation and control equipment, a new thermocouple read-out panel was fabricated and installed. All the thermocouples on the process equipment skids were rewired to this new panel, and to the new data logging system. The software for the data acquisition and process control system (LabTech ControlPro) is being installed and tested.

4. OPEN ITEMS

None.

5. PLANS FOR NEXT QUARTER:

- ! Continue the engineering design effort for refurbishing the Mobile Laboratory.
- ! Continue the construction activities in the Mobile Laboratory.
- ! Continue the lab-scale testing of a promising sorbent formulation with multiple cycles of sulfidation, SO₂ regeneration, and air regeneration, prior to bench-scale testing.