1995-1996 ANNUAL RESEARCH REPORT
(The Annual Reporting Period: 10/01/95 - 9/30/96)

ON

Characterization and Optimization of Sorbents Utilized

For

Emission Control During Coal Gasification

Program Identification Number: DE-FG21-94MC31426—11

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Date: October 25, 1996   **MASTER**

The research activities performed at PV A&M University in the year of 1995-1996, reporting period from 10/01/95 to 9/30/96 are summarized below.

- The operating procedures for the Low Pressure Bench Scale Hot Gas Desulfurization unit started in Q4 1995. These procedures include Safety Precautions, Pre-Run Procedures and Startup Procedures.

- The Operating Procedures, Shutdown Procedures and Emergency Procedures was also under planing in Q4, 1995.

- An automatic data acquisition system has been acquired from National Instrumentation in December, 1995. The automatic data acquisition system will help calculate gas flow rate, pressure and temperature monitoring, data storage and presentation. The students were trained to use this advanced software first and Dr. Mei helped the student perform programming. Dr. Mei also led the interfacing of electronic devices, RS 232, IEEE 488 interfacing boards and instruments with the computer and data acquisition system for test data analysis and results presentation.

- The automatic data acquisition system is an advanced system, it will took eight weeks for the students to understand the programming and how it does control work. The faculty work together with the students.

- Adjusted parts and supplies for the Bench Scale Hot Gas Desulfurization Unit is in the procurement process. Quotations for parts required for the sorbent testing chamber were sent out.

- Tubings and fittings for the system were in the pricing bidding procurement process.

- The design of the laboratory scale process unit for the hot gas desulfurization had been layout. It is a fixed-bed setup. This design has the advantages of high sulfur absorption at low sulfur breakthrough and low sorbent attrition.

- The major operational procedures were briefly described below.

    1) Simulated coal-derived gas will bve provided for the tests by mixing constituents from an array of rotameters through a gas mixer.

    2) Reactor exit gases during sulfidation and regenerarion pass seperately through a hot gas filter and condenser.

    3) Particle elutriation can be measured by weighing the filter element before and after a test.

    4) A typical test run consists of sulfidation period and regeneration period.

    5) Zinc based sorbent formulations will be evaluated in this test plan.

- The operating procedures for the Low Pressure Bench Scale Hot Gas Desulfurization unit had been complete in Q1 1996.

- The hardware of the automatic data acquisition system had been put together. The automatic data acquisition system would help calculate gas flow rate, pressure and temperature monitoring, data storage and presentation. Dr. Huque, Dr. Mei and the students started workingon the interfacing the instruments with the computer and data acquisition system for test data analysis and results presentation. This task required a lot of work to complete due to the lack of adequate drivers and interfacing programming available.

- With very much limited budget, about a 50% reduction, the design had been modified to adjusted parts and supplies for the Bench Scale Hot Gas Desulfurization Unit build.

- The number of tubings and fittings for the system would be reduced to put together even a smaller unit.

- The modified design of the laboratory scale process unit for the hot gas desulfurization had been complete.

- The major hardware components for the Low Pressure Bench Scale Hot Gas Desulfurization unit had been acquired in Q2 1996.

- The automatic data acquisition system successfully passed testing for automatic measurement on electrical signals and pressure sensor outputs. More efforts were put in the process of interfacing different instruments with the computer via RS 232 and IEEE 488 communications on data acquisition system. We were also learning to prepare programming for test data analysis and results presentation..

- The major research output of this quarter is to learn that there are several disposable type metal oxides could be utilized for desulfurization purpose to replace the expensive sorbents being developed lately. This concept was discussed between Daniel Mei and Hot Gas Cleanup industry in July 1995. This idea was also briefly discussed with METC HGCU research team members.

- The most promising features of using these one time disposable oxides are listed below.

  - The metal oxides can be brought to farther upstream during gasification or combustion process to provide metal oxides with more time in reacting with SO_2, H_2S and other chemicals to maximize desulfurization operation.

  - The cost of these disposable metal oxides can be economical enough to make IGCC and PFBC feasible economically in the future.

  - The size distribution of candidate metal oxides can be controlled within certain range to optimize the dust cake removal during back pulse dust cake cleaning.

- The discharged ash and metal oxides would cause unexpected environmental safety issues.

- The size distribution controls of metal oxides will play a very important role for using the disposable type of metal oxides in the Hot Gas Cleanup program.

- Based on the permeability characterization of ceramic candle filters, conducted at Prairie View A&M University, Daniel Mei and Ziaul Huque found out that the permeability of the used filter varies axially and circumferentially after operation in particulate filtration vessel.

- According to the permeability variation distributions on filter, samples of the external filter surface of these sections with low permeability were analyzed in the material laboratory. The low permeability sections displayed with deposits of foreign materials and chemical residuals. These deposition of fine size particles sintered at high temperature with time might be the cause that decreased the original highpermeabilityy distribution of the unused filter.

- If the size of particulate distribution can be adjusted within certain range to facilitate the forming of proper dust cake withoutsacrificingg gas filtering and dust cake removal, then the metal oxides can be easily controlled within the specified band range to optimize Hot Gas Cleanup in both dust cake removal andsorbett pricing reduction.

- Prairie View A&M University sorbent research team conducted the following studies in Q3 1996 besides the continuing pursuit to complete small scale laboratory desulfurization system.

   - Started the study of the availability of metal oxides that can be utilized for dusulfurization purpose.

   - Started the study of the pricing of the metal oxides that can contribute to proper desulfurization purpose.

   - Proposed the study of particulate size distribution effects to METC and Westinghouse particulate research group to "determine" a particulate size distribution "band range" to optimize the particulate removal during backpulsee cleaning process.

   - Westinghouse had sent four used filters to us for evaluation. We will analyze the filtered particulates and try to correlate its size and concentration of chemicals and possible residual sorbent particles left in the external surface of the filters. These study will be based on the history of the individual filters.

   - Started the study of the cost issues of controlling the metal oxides size within the proposed size distribution.

   - Started the study of the safety issues of using metal oxides in desulfurization process.

   - Started the build of the gases and sorbent reaction chamber. The fittings and valves required for testing has been installed on the chamber.

- Started the study of the gas chromatograph operation manual for gases analysis.

- Procured sorbent samples from IF&P company.

Future Plan Of Action

- To continue the cost study of the disposable type of sorbent

- To continue the study of the influence of particle sizes to the removal of the particulates during back pulse cleaning.

- To plan the study of Westinghouse filters and its history effects to the desulfurization effect.

- To continue the build of the test system.

- To complete the instrumentation driver interfacing with different instrumentation.

- To perform sorbent desulfurization testing and optimization evaluation.

APPENDIX 1

PRELIMINARY OPERATING PROCEDUREWS

FOR

THE LOW PRESSURE BENCH SCALE HOT GAS DESULFURIZATION

UNIT

1.0 SAFETY PRECAUTIONS

This procedure lists general safety precautions that shall be observed by the research operator for any operations of this low pressure bench-scale hot gas desulfurization unit.

1.1   Safety glasses and protective clothing shall be worn at all times while working in the laboratory.

1.2   The low pressure bench-scale HGD unit is designed for safe operaions up to 1800 degree F and up to 30 psig. The system shall not be operated under conditions which exceed either limits of the pressure or the temperature.

1.3   The high temperature lines shall be insulated prior to the beginning of the heat-up procedure.

1.4   Pressurized gas cylinders shall be secured against a wall or in a cylinder racks.

1.5   Explosive gases (hydrogen, carbon monoxide, methane and hydrogen sulfide) and toxic gases (hydrogen sulfide, hydrogen chloride, sulfur dioxide, and carbon monooxide) shall be leak checked prior to the starting of the testing.

1.6   Portable gas monitors shall be used to detect toxic gases during the testing period.

1.7   During the sampling of the process gas stream, samples shall be taken in the chamber only.

1.8   All gas cylinder valves shall be closed and all process lines between the cylinders and the instrument panel shall be bled off for overnight and weekend hot hold conditions and at the completion of any test.

1.9   The reactor shall be visually inspected for cracks or flaws before installation and at the end of each test run. If a crack or flaw is observed, the reactor shall not be installed.

1.10   The system pressure shall be reduced to 0 psig during heat-up, cool-down or during the hot hold conditions.

1.11   Only authorized personnel shall be permitted in the laboratory.

2.0 PRE-RUN PROCEDURES

In preparation for testing, several pre-run checks will be performed. Each activity shall be recorded and dated for reference. The required activities are summaried below.

2.1 Make sure there is sufficient gas available for each gas required.

2.2 Make sure there are sufficient operating supplies prior to each test.

2.3 Check that all thermocouples are operational.

2.4 Check all the pressure gages and pressure indicators are operational.

2.5 Verify all the mass flow controllers are functioning and readout are on.

2.6 Refill the water reservoir with distilled water.

2.7 Check that the water outlet and drain valves are closed.

2.8 Load required amount of sorbent into the reactor, record sorbent mass.

2.9 Make sure the reactor top is securely sealed.

2.10 Fasten safety shield around the top and bottom of the reactor.

2.11 Leak test the sulfidation flow path and hydrogen sulfide lines.

2.12 Purge the lines with Nitrogen at 40 psig.

2.13 Mark the location of any leaks. Tighten the leaking joint or fix the leaks prior to the test.

2.14 Test the panel mounted emergency shut-down switch.

2.15 Leak test the regeneration flow path and fix any leaks prior to the test.

3.0 START-UP PROCEDURES

After the completion of Pre-Run procedures, proceed to Start-Up procedures.

Heat-Up section

3.1   Prepare for nitrogen flow through the sulfidation flow path.

3.2   Verify the nitrogen flow rate is the same as the test operations plan.

3.3   Adjust the mass flow controller set points to have the accurate mass flow per test plan.

3.4   Turn on appropriate circuit breakers for heat up sources.

3.5   Turn on pump controller.

3.6   Turn on furnace controller.

3.7   Start the air flow and check the pressure readings.

3.8   Set the temperature for the preheater.

3.9   Set the furnace temperature to 200 degree C.

3.10   Establish the water flow to the sulfidation condensor.

3.11   Check reactor temperature. If they are not within 25 degree F of the desired temperature, adjust the furnace controller as required.

Sulfidation section

3.12   Turn on the automatic data acquisition system.

3.13   Connect thermocouples to the sulfidation condenser lines.

3.14   Establish water flow to the sufidation condenser.

3.15   Turn on water chiller.

3.16   Establish the system pressure per the operation test plan.

3.17   Turn on water to the preheater.

3.18   Turn on methane (CH_4) bottle valve.

3.19 Turn on CO_2 valve.

3.20 Turn on CO valve.

3.21 Turn on H_2 valve.

3.22 Turn on $H_2S + N_2$ valve.

3.23 Check that the system and reactor differential pressure is in the right position for sulfidation.

3.24 Start the gas flow through the reactor in the following order: N_2, H_2O, CH_4, CO_2, CO, H_2 $H_2S + N_2$.

3.25 Record the time and this is the start of the test.

Regeneration section

3.26 Turn on the automatic data acquisition system 30 minutes minimum prior to the start of the testing.

3.27 Fill the regeneration condensor with ice.

3.28 Connect the thermocouple to the ice bath lines.

3.29 Close air flow and stop the water pump.

3.30 Establish the line pressure per the testing plan.

3.31 Turn on water to the preheater.

3.32 Start the flow of air.

3.33 Prepare the air flow path to the preheater.

3.34 Start gas flow through the reactor in the following order: N_2, H_2O, air.

3.35 Record the time, this is the start of the test.