1 of 1

DOE/PC/90309-11

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**ANION-EXCHANGE RESIN-BASED
DESULFURIZATION PROCESS****Quarterly Technical Progress Report****April 1, 1993 - June 30, 1993****Prepared by:****Atul C. Sheth
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EXECUTIVE SUMMARY

Under DOE Grant No. DE-FG22-90PC90309, the University of Tennessee Space Institute (UTSI) is contracted to further develop its anion-exchange, resin-based desulfurization concept to desulfurize alkali metal sulfates. From environmental as well as economic viewpoints, it is necessary to remove soluble sulfates from the wastes created by flue gas desulfurization systems. In order to do this economically, a low-cost desulfurization process for spent sorbents is necessary. UTSI's anion-exchange resin-based desulfurization concept is believed to satisfy these requirements.

During the reporting period, April 1, 1993 - June 30, 1993, using a slightly revised procedure, UTSI completed the fixed-bed mode experiments to evaluate the cycle efficiency of IRA-68, -35 and 93 resins. The results from experiments with revised procedure for IRA-35 resin are still being analyzed. However, the similar results for IRA-68 and -93 resins show no significant degradation in the performance after ten consecutive cycles of exhaustion/carbonation and regeneration. The process schematics for the spent sorbent/seed regeneration/desulfurization have been put together and the mass balance calculations will be initiated shortly.

As discussed in our previous progress reports, the program fell 6-8 weeks behind the schedule because of certain circumstances. As a result, we requested a 4-month no-cost extension to the project which was granted by DOE/PETC (letter from Mr. Keith Miles dated July 2, 1993). Hence, the new completion date for the subject grant will be December 31, 1993. The project is, however, still within the budget.

1.0 INTRODUCTION

Under the current grant (# DE-FG22-90PC90309), the University of Tennessee Space Institute (UTSI) will perform the bench scale evaluation and further development of the anion-exchange resin-based desulfurization concept to desulfurize alkali metal sulfates. This concept has been developed and patented by UTSI under U.S. Patent No. 4,917,874. The technical feasibility of this concept has been demonstrated and reported in the M.S. theses prepared by Butler⁽¹⁾ and Modeste⁽²⁾. The developmental program proposed under this DOE grant includes screening of commercially available resins to select three candidate resins for further study. These three resins will undergo a series of experiments designed to test the resins' performance under different process conditions (including the use of spent MHD seed material). The best of these resins will be used in optimizing the regeneration step and in testing the effects of performance enhancers. The process schematic developed from the results will be used to estimate the related economics.

During this reporting period, April 1, 1993 to June 30, 1993, the procedure to evaluate the cycle efficiency of candidate resins in the fixed-bed mode was slightly modified to ensure complete regeneration of the exhausted resin. Using this revised procedure, ten consecutive cycles for all the three resins have been completed and the results are being analyzed.

2.0 OBJECTIVES AND PROJECT DESCRIPTION

Objectives to be satisfied as well as the detailed description of the project tasks and subtasks were described in the Quarterly Technical Progress Report for the period August 9, 1990 - December 31, 1990 (report no. DOE/PC/90309-1).

3.0 PROJECT STATUS/PROGRESS TO DATE

The progress made during the period April 1, 1993 - June 30, 1993 is indicated in Figures 1 and 2. Figure 1 shows the proposed schedule and the progress to date for each task. Figure 2 lists the major milestones to be accomplished under each task and their planned and actual completion dates. Activities performed for each task are as follows:

3.1 Task 1.1

This task was completed in September 1991 and the results were reported in prior quarterly reports.

3.2 Task 1.2

This task was completed in August 1992 and the results were reported in the Annual Technical Progress Report for the period October 1, 1991 - September 30, 1992 (report no. DOE/PC/90309-8). The cycle efficiency of the selected three resins (IRA-68, -35, and -93) is being evaluated under Task 1.3.

Milestone Schedule and Status Report

Contract Identification		ANION - EXCHANGE RESIN - BASED DESULFURIZATION PROCESS		Reporting Period April 1, 1993 - June 30, 1993		Grant Number DE-FG22-90PC90309		
Contractor: Name, Address		THE UNIVERSITY OF TENNESSEE SPACE INSTITUTE TULLAHOMA, TENNESSEE 37388		Contract Start Date September 1, 1990		Contract Completion Date December 1993		
ID	Reporting Category - Contract Line Item or Work Breakdown Structure Element	CY 90	I	CY 91	I	CY 92	I	CY 93
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MILESTONE LOG

I.D. NO.	DESCRIPTION	ACTUAL COMPLETION DATE	PLANNED COMPLETION DATE	EXPECTED COMPLETION DATE	COMMENTS
1.1 A	Complete literature survey, vendor contacts and procure samples of candidate resins	Oct 90	Oct 90		
1.1 B	Complete bench scale screening tests and select three resins	Sep 91	Aug 91		
1.2 A	Complete bench scale process variables study and select one resin	Aug 92	Apr 92		
1.3 A	Complete optimization of resin regeneration step using selected resins	Nov 92	Aug 92		
1.3 B	Complete evaluation of performance enhancers and cycle efficiency for selected resins	Jun 93	Dec 92		
1.4 A	Validate math model using bench scale process variables study data	Sep 92	Apr 92		
1.4 B	Validate math model using data from optimized resin regeneration step and evaluation of performance enhancers		Jan 93	Jul 93	Delay due to delays in 2.0A and 3.0B
1.4 C	Complete process economics for the Best Process Schematic		Jun 93	Oct 93	Delay due to delays in 2.0A, 3.0B, and 4.0B
1.5 A	Submit quarterly technical reports		(As Indicated)		Eleventh quarterly to be submitted in Jul '93
1.5 B	Submit annual technical reports		(As Indicated)		
1.5 C	Submit final project report		Aug 93	Dec 93	Because of no-cost extension

Figure 2

3.3 Task 1.3

In the last quarter, the poor performance of the IRA-35 resin was reported based on the cycle efficiency experiments. However, it was suspected at that time that the observed poor performance could be due to incomplete regeneration of the exhausted resin. Hence, the experimental procedure was slightly modified by increasing the NH_4OH solution concentration as well as its flow rate and treatment time. In the future, however, this step needs to be studied in detail to identify optimum regeneration conditions for the fixed-bed operations.

Using this revised procedure, the fixed-bed experiments were carried out to determine cycle efficiency of candidate resins. The resin under study was subjected to ten consecutive cycles of exhaustion/carbonation and regeneration. The exhaustion/carbonation step was carried out using a 5 wt % K_2SO_4 solution, a 10 psig CO_2 static pressure, 11 cm^3/min of sulfate solution flow rate and 100 cm^3 of candidate resin charge. The regeneration of the exhausted resin was carried out in-situ by using 13.6 wt % NH_4OH solution at a 25 cm^3/min flow rate for 90 minutes duration. During exhaustion/carbonation step, the samples of the column effluent were taken at regular time intervals to determine an experimental breakthrough curve and the resin's capacity for that particular cycle. However, similar samples during the resin regeneration step were not taken, as analytical technique to measure SO_4^{2-} ion in highly alkaline ammonia-containing solution is not available and the regeneration conditions for the fixed-bed operations need to be further optimized in a separate study.

The experimentally determined breakthrough curves for IRA-68 and -93 resins are shown in Figures 3 and 4. The results for IRA-35 using this revised procedure are still being analyzed. From these two figures, within the experimental errors, both the resins (IRA-68 and -93) show no degradation in performance. This conclusion is reached based on the closeness of the breakthrough curves for various exhaustion/carbonation cycles.

To further confirm the above conclusion, using numerical integration, the resin's capacity for each cycle was calculated following the approach used in the past UTSI studies. The calculated resin's capacities as a function of corresponding cycle number for IRA-68 and -93 resins are shown in Figures 5 and 6. The similar plot for the IRA-35 resin will be prepared later on as the experimental data are still being analyzed. From Figures 5 and 6, it is evident that the variations in the calculated resin's capacities are within $\pm 10\%$. Also, there is no trend showing either a continuous decline or improvement in the resin's capacity. Therefore, the observed variations can be attributed to possible experimental errors. Thus, based on Figures 3-6, one can say that the performance of IRA-68 and -93 resins remained the same for up to ten consecutive cycles.

To confirm that the resin has very high life expectancy and is suitable for repeated use in industrial applications, one needs to carry out hundreds of consecutive cycles. However, this can only be demonstrated at a bigger pilot scale (prototypical units) since the small size of a bench scale unit and the limited degree of automation would make it very difficult. This is an important parameter to be addressed in a future

Figure 3. Cycle Efficiency Experiments For IRA-68 Resin

- CYCLE #1
- CYCLE #2
- ◻ CYCLE #3
- × CYCLE #4
- + CYCLE #5
- △ CYCLE #6
- ▣ CYCLE #7
- CYCLE #8
- ◆ CYCLE #9
- ▲ CYCLE #10

- Flow Rate of K_2SO_4 Solution = 11 cc/min
- Resin volume = 100 cc
- 5 wt% K_2SO_4 solution
- CO_2 Pressure = 10 psig.

Figure 4. Cycle Efficiency Experiments For IRA-93 Resin

CG-3013

**Figure 5. Calculated Equilibrium Capacity of IRA-68 Resin
As a Function of Cycle Number**

CG-3014

**Figure 6. Calculated Equilibrium Capacity of IRA-93 Resin
As a Function of Cycle Number**

CG-3015

study, as high resin life expectancy means low replacement (operating) cost, and, therefore, improved process economics.

As discussed in a previous quarterly progress report (i.e. report no. DOE/PC/90309-10), the batch mode experiments already carried out at that time to locate the position of the CO_3^{2-} ion with respect to SO_4^{2-} ion in the affinity chart could only indicate that both are very close to each other. Hence, additional experiments were carried out in this quarter to shed further light on this matter. In these additional experiments, the candidate resin (available in $\text{R}\cdot\text{OH}$ form) was as usual first converted to $\text{R}\cdot\text{HCO}_3$ form. This $\text{R}\cdot\text{HCO}_3$ form of resin was then treated with a 5 wt % K_2CO_3 solution for sufficient time (45 minutes) to convert HCO_3^- ions on the resin into CO_3^{2-} ions form. The resulting $\text{R}_2\cdot\text{CO}_3$ form of resin was then filtered and washed with de-ionized water and treated with a 5 wt % K_2SO_4 solution. By measuring the SO_4^{2-} ion concentration in the initial and final solution, the percent SO_4^{2-} removal by the candidate resin was calculated. The results from these additional experiments are given in Table 1. From this table, it can be seen that the SO_4^{2-} removal efficiencies for candidate resins varied from 31 to 33%. In comparison, the $\text{R}\cdot\text{HCO}_3$ form of resin when exhausted with a 5 wt % K_2SO_4 solution, the SO_4^{2-} removal efficiencies under similar conditions were found to vary from 42 to 60% (see report no. DOE/PC/90309-4). Hence, the removal efficiency of the candidate resins seems to decrease by about 46% for IRA-68 resin to about 73% for IRA-93 resin. These observed decreases in the SO_4^{2-} removal efficiencies of candidate resins in $\text{R}_2\cdot\text{CO}_3$ form can be attributed to the inability of the CO_3^{2-} ions on the resin from being exchanged by the SO_4^{2-} ions in the bulk solution.

Table 1. Removal of SO₄²⁻ Ion by CO₃²⁻ Form of Resin

- Basis:
- R·HCO₃ converted to R₂·CO₃ using 5 wt % K₂CO₃ solution
 - R₂·CO₃ form of resin exhausted using 5 wt % K₂SO₄ solution
 - solution-to-resin ratio = 2

Type of Resin	Initial SO ₄ ²⁻ Concentration (ppm)	Final SO ₄ ²⁻ Concentration (ppm)	% SO ₄ ²⁻ Removed
IRA-68	26,574	17,900	32.6
IRA-93	26,574	18,320	31.1
IRA-35	26,574	17,840	32.9

In the previous quarterly progress report (i.e. report no. DOE/PC/90309-10), we showed that the exchange of the SO₄²⁻ ions on the resin by the CO₃²⁻ ions in the bulk solution depended on the concentration of the CO₃²⁻ ions in the bulk solution and the extent of it increased with the solution concentration. Hence, by combining the results of that series of experiments with the present series of experiments, one can say that most likely the CO₃²⁻ ion is present to the left of the SO₄²⁻ ion in the affinity chart as shown below:

This conclusion would also explain the observed increase in the resin's capacity for the SO₄²⁻ ions during the exhaustion/carbonation cycle, as the CO₂-pressure is increased. Because at higher CO₂ pressure, the R₂·CO₃ form is significantly reduced in comparison to R·HCO₃ form as per the following reaction:

3.4 Task 1.4

Under this task, the efforts are now underway to evaluate the possible effects of the process parameters such as sulfate solution concentration, regeneration characteristics, type of resin, mode of effluent concentration/evaporation and possible integrated versus stand-alone configuration. For regeneration/desulfurization of the MHD spent seed, preliminary schematics are now developed to process the streams coming from a wet electrostatic precipitator (WESP). These schematics for a low K_2/S ratio (that means high sulfur coal) and a high K_2/S ratio (that means low sulfur coal) are shown in Figures 7 and 8 respectively. A similar schematic for the desulfurization of the spent sodium-based flue gas desulfurization (FGD) sorbent obtained from an in-duct sorbent injection type of SO_2 -removal option is shown in Figure 9. These three schemes will be further analyzed in our future efforts to compare the major similarities and differences. Based on the findings, a unified "Best Process Schematic" will be developed to include all possible situations and applications. This Best Process Schematic will then be analyzed using the EPRI-TAG document to develop the related process economics.

3.5 Task 1.5

The following activities were completed under this task.

- The tenth quarterly technical progress report (No. DOE/PC/90309-10) covering the performance period of January 1, 1993 - March 31, 1993 was issued on April 26, 1993.
- The final draft of the accepted technical paper entitled, "Spent Seed Regeneration in Coal-Fired MHD System Using Weak-Base, Anion-Exchange Resins," was submitted to the Elsevier Science Publishers for

Figure 7. Process Schematic For Ion - Exchange Resin - Based Seed Regeneration Process (Low K_2/S Ratio)

Figure 8. Process Schematic For Ion - Exchange Resin - Based Seed Regeneration Process (High K₂/S Ratio)

Resin Exhaustion/Carbonation Step

Resin Regeneration Step

The diagram illustrates a chemical process for producing sodium-based sorbent and ammonium sulfate fertilizer. The process is divided into two main sections by a dashed line.

Left Section (Sorbent Production):

- Flue Gases From Boiler** enter a **Baghouse/Esp** unit.
- Sodium-based Sorbent** is added to the **Baghouse/Esp** unit.
- The output of the **Baghouse/Esp** unit goes to a **Dissolver** unit, where **Water** is added.
- The output of the **Dissolver** unit goes to a **Solid-Liquid Separator**.
- The **Solid-Liquid Separator** produces **Insoluble Fly-ash to Disposal** and a **> 5 wt% Na₂SO₄ Solution**.
- The **> 5 wt% Na₂SO₄ Solution** is sent to the **Active Resin** unit.
- The **Active Resin** unit produces **NaHCO₃ Solution**.
- The **NaHCO₃ Solution** is sent to a **Multi-Effect Evaporator**, which also receives **Steam**.
- The output of the **Multi-Effect Evaporator** goes to a **Dryer**, which also receives **Heat Source**.
- The output of the **Dryer** is **NaHCO₃/Na₂CO₃ Recycle**, which is sent back to the **Baghouse/Esp** unit.
- The output of the **Dryer** also goes to a **Cooling Water Cooler/Condenser**, which also receives **Cooling Water**.
- The output of the **Cooling Water Cooler/Condenser** is **CO₂ Make-up**, which is sent to the **Active Resin** unit.
- The output of the **Cooling Water Cooler/Condenser** also goes to a **CO₂ Recycle** unit.
- The output of the **CO₂ Recycle** unit is **CO₂**, which is sent to the **Active Resin** unit.
- The output of the **CO₂ Recycle** unit also goes to a **Condenser/Cooler**, which also receives **Cooling Water**.
- The output of the **Condenser/Cooler** is **NH₃ Recycle**, which is sent to the **Decomposer (200 - 250°C)** unit.
- The output of the **Decomposer (200 - 250°C)** unit is **H₂SO₄ for Sell**.
- The output of the **Decomposer (200 - 250°C)** unit also goes to a **Condenser/Cooler**, which also receives **Cooling Water**.
- The output of the **Condenser/Cooler** is **NH₃ Make-up**, which is sent to the **Decomposer (200 - 250°C)** unit.
- The output of the **Condenser/Cooler** also goes to a **Decomposer (200 - 250°C)** unit.
- The output of the **Decomposer (200 - 250°C)** unit is **(NH₄)₂SO₄ Solution**, which is sent to the **Decomposer (200 - 250°C)** unit.
- The output of the **Decomposer (200 - 250°C)** unit is **Sell as Fertilizer**.

Resin Regeneration Step

inclusion in the Proceedings of the 5th International Conference on Processing and Utilization of High Sulfur Coals, to be held from October 24-28, 1993 in Lexington, Kentucky.

- A technical paper entitled, "Recent Advances in the UTSL's Anion-Exchange Resin-Based Seed Regeneration Process," was presented (by Atul Sheth) in the poster session and published in the Proceedings of the 31st Symposium on Engineering Aspects of Magnetohydrodynamics, held from June 29 - July 1, 1993 in Whitefish, Montana.
- A two-page write-up describing the abstract and highlighting the accomplishments of our research project along with a completed technical subject index were submitted to the DOE/PETC office on May 3, 1993, for inclusion in the report assembled by DOE on the various University Coal Research (UCR) projects. This report was distributed at the 1993 UCR Contractors' Review meeting scheduled by DOE in Pittsburgh.
- Rajeev Dharmapurikar (a graduate student) and Atul Sheth (a principal investigator) attended the DOE/PETC sponsored UCR Contractors' Review meeting in Pittsburgh from June 23 - 25, 1993. They also made a poster presentation of the work done so far at this meeting.
- A no-cost extension was requested from DOE via a letter dated June 17, 1993. In this letter, a request was made to DOE to extend the completion date of our project to December 31, 1993 so that the proposed program can be successfully completed and documented in a final project report.

We received DOE approval on July 2, 1993 to our no-cost extension request.

At the present time, the project is still about 6 - 8 weeks behind the original schedule (due to earlier equipment related problems (now resolved), increase in work scope due to additional batch mode screening, holidays and UTSI's Chemistry Laboratory moving to a new location). However, it is well within the budget.

4.0 REFERENCES

1. Butler, W.A., "Bench Scale Evaluation of Anion-Exchange Resin-Based Seed Regeneration Concept for Coal-Fired Magnetohydrodynamics Power System," a thesis presented for M.S. degree, The University of Tennessee Space Institute, December 1986.
2. Modeste, D.C., "Evaluation of Improved Anion-Exchange Resin-Based Seed Regeneration Concept for Coal-Fired MHD Power System," a thesis presented for M.S. degree, The University of Tennessee Space Institute, December 1988.

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