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Simultaneous Removal of H₂S and HN₃ From Coal Gas

Final Report
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ABSTRACT

Hydrogen sulfide (H_2S) and ammonia (NH_3) are the primary sulfur and nitrogen contaminants released when coal is gasified. Before coal gas can be utilized in an integrated gasification combined cycle (IGCC) plant to produce electricity, these contaminants need to be removed. The objective of this research was to develop sorbent-catalysts with the ability to simultaneously remove H_2S and NH_3 from coal gas. Microreactor tests with HART-49, a zinc-based sorbent-catalyst with Ni, Co, and Mo as catalyst additives, showed that this material had the potential to remove 90 percent NH_3 and reduce H_2S to <20 ppmv at 1 atm and 550 to 700 °C. HART-49 was prepared in attrition-resistant fluidizable form (HART-56) using up to 75 wt% binder. Bench-scale fluidized-bed multicycle tests were conducted with the attrition-resistant sorbent-catalyst, HART-56, at 20 atm and 550 °C. The H_2S and NH_3 removal performance over the first two cycles was good in the presence of 5 percent steam but deteriorated thereafter when steam level was increased to 15 percent. The results point to a complex mechanism for simultaneous H_2S and NH_3 removal, potentially involving both chemisorption and catalytic decomposition of NH_3. Further research and development is needed to develop a sorbent-catalyst for simultaneous H_2S and NH_3 removal at IGCC hot-gas cleanup conditions.

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EXECUTIVE SUMMARY

Advanced integrated gasification combined cycle (IGCC) power plants under development employ hot particulate removal and hot-gas desulfurization (HGD) to achieve higher thermal efficiency than conventional IGCC plants employing cold gas cleanup. HGD involves the removal of hydrogen sulfide (H_2S) using metal regenerable metal oxide sorbents. These sorbents, typically zinc-based, remove the H_2S by sulfidation and are regenerated by air-oxidation of the metal sulfide in a cyclic manner.

Coal gas also contains ammonia (NH_3) that needs to be removed to reduce NO_x formation in the gas turbine. Catalytic decomposition of NH_3 to nitrogen (N_2) and hydrogen (H_2) is an attractive method for NH_3 removal. By combining the NH_3 decomposition catalyst with the H_2S metal oxide sorbent to produce a simultaneous H_2S and NH_3 removal sorbent-catalyst, cost savings can be potentially achieved.

The objective of this study is to develop and demonstrate catalytic approaches for decomposing a significant percentage (up to 90 percent) of the NH_3 present in coal gas to N_2 and H_2 at elevated temperatures (550 to 900 °C).

Both high-temperature catalytic decomposition upstream of HGD and simultaneous H_2S and NH_3 removal approaches were investigated. A Topical Report on high-temperature catalytic decomposition submitted earlier details the work until the end of FY97 and is attached as Appendix 1. In this work, catalytic decomposition of up to 90 percent NH_3 in the presence of H_2S at temperatures >850 °C was demonstrated in both bench-scale and coal gasifier slipstream tests. Preliminary attempts at developing a combined sorbent-catalyst were only moderately successful. Hampton University and Research Triangle Institute (RTI) in a parallel study (DOE Contract No. DE-FG22-93MT93005) developed a promising zinc-based HART-49 sorbent-catalyst in laboratory research. Based on this research, the present work was undertaken at RTI with Hampton University as a subcontractor.

The HART-49 sorbent-catalyst powder showed >90 percent NH_3 removal and H_2S removal down to <20 ppmv in microreactor tests at 1 atm and 500 to 700 °C. Using the HART-49 sorbent composition, a series of attrition-resistant fluidizable sorbent-catalysts (HART-50 to HART-55) were prepared with up to 75 percent binder. HART-55 gave the best combination of attrition resistance and performance in microreactor testing. HART-55 was scaled up to produce a 200-g batch (designated HART-56) which was selected for fluidized-bed bench-scale testing. A 20-cycle bench-scale test was conducted on this material at 20 atm and 550 °C.

Microreactor tests with HART-55 showed that pressure promoted the removal of NH_3, contrary to thermodynamic predictions. Some NH_3 release occurred from the sorbent-catalyst during regeneration, which indicated chemisorption during sulfidation as a potential explanation of the pressure effect. At 10 atm, >90 percent NH_3 removal was maintained up to 6 wt% sulfur loading on the sorbent-catalyst, consistently over seven cycles.

The 20-cycle bench-scale test showed a high degree of NH_3 removal with 5 percent steam in coal gas over the initial cycles. This result agreed with the microreactor results. However, when the

steam was increased to 15 percent in subsequent cycles, the NH_3 removal performance deteriorated significantly. The results indicate that HART-56 was not successful in achieving significant NH_3 removal at 550 °C and 20 atm except when low steam (around 5 percent) was present in the coal gas. Also, reversible adsorption of NH_3 appeared to occur on the sorbent, which could be removed by purging.

The NH_3 removal mechanism on the HART-49 based sorbent-catalysts is complex with reversible adsorption as well as decomposition occurring at higher pressure. Further work is needed to optimize the sorbent-catalyst preparation to get a balanced material with acceptable attrition resistance, NH_3 removal performance, and cost. With high levels of binder used here, an attrition resistance better than needed was achieved but at the expense of reactivity. The NH_3 removal mechanism needs to be evaluated with fundamental studies, particularly with respect to adsorption behavior on the sorbent catalyst at higher pressure and the dramatic effect of steam. The attrition-resistant HART-56 sorbent appears to have potential for removing 90 percent NH_3 and reduce H_2S to <20 ppmv, when 5 percent steam is present in the coal gas. However, further development of the material is needed for coal gas with higher steam.

1.0 INTRODUCTION AND OBJECTIVES

Integrated coal gasification combined cycle (IGCC) electric power plants are being developed worldwide because of their potential for higher thermal efficiency and superior environmental performance compared to conventional pulverized-coal combustion-based plants (1-4). Coal gasification results in a number of contaminants that are released into the coal gas. These include particulates, sulfur gases (principally hydrogen sulfide [H_2S]), nitrogen compounds (principally ammonia [NH_3]), and trace contaminants such as alkali, chloride, and heavy metals. These contaminants need to be removed or significantly reduced before the coal gas can be burned in an IGCC gas turbine to make electricity. The coal gas is typically quenched using a water spray down to low temperatures (below 50 °C), and conventional contaminant removal operations are conducted. The cooling, however, results in a significant thermal efficiency penalty and a waste-water stream that must be disposed of. These plants also end up being capital intensive.

1.1   Hot-Gas Desulfurization

To reduce the cost of IGCC and to increase its overall thermal efficiency, advanced IGCC plants employing hot-gas cleanup methods, rather than quench/low-temperature cleanup, are being developed (3,5-7). The sponsorship and leadership in this development have been provided by the U.S. Department of Energy/Federal Energy Technology Center (DOE/FETC). Hot-gas cleanup methods employ hot particulate removal (using ceramic filters) and hot-gas desulfurization (HGD) subsystems operating at high-temperature, high-pressure (HTHP) conditions downstream of the coal gasifier and upstream of the gas turbine.

Research on HGD methods for coal gas in IGCC systems has concentrated on the use of regenerable metal oxide sorbents (8-16). Oxides of copper, cerium, manganese, cobalt, tin, iron, and zinc, both individually and in combinations, have been evaluated as regenerable sorbents. These metal oxide or mixed-metal oxide sorbents have been investigated without, as well as combined with, a secondary oxide, typically silica, alumina, titania, and chromium oxide. The roles of these secondary oxides include support for strengthening mechanical structure and stabilizer against reduction of the metal.

Zinc titanate, formed by combining zinc oxide and titanium oxide at high temperature, is currently one of the sorbent candidates for HGD (8,9). It can remove H_2S down to low parts-per-million levels over a wide temperature range and it can be regenerated for multicycle operation. Depending on the Zn to Ti ratio in zinc titanate, a number of phases such as $ZnTiO_3$, $Zn_2Ti_3O_8$, and Zn_2TiO_4 can form.

At demonstration-scale, the HGD process is carried out in a two-reactor system, consisting of a sulfider and an air-regenerator (8,11). An example of such a system is the M.W. Kellogg transport-reactor HGD unit (Figure 1) at the Sierra-Pacific Clean Coal Demonstration Plant (11). Using $ZnTiO_3$ as an example, the HGD reactions are shown below:
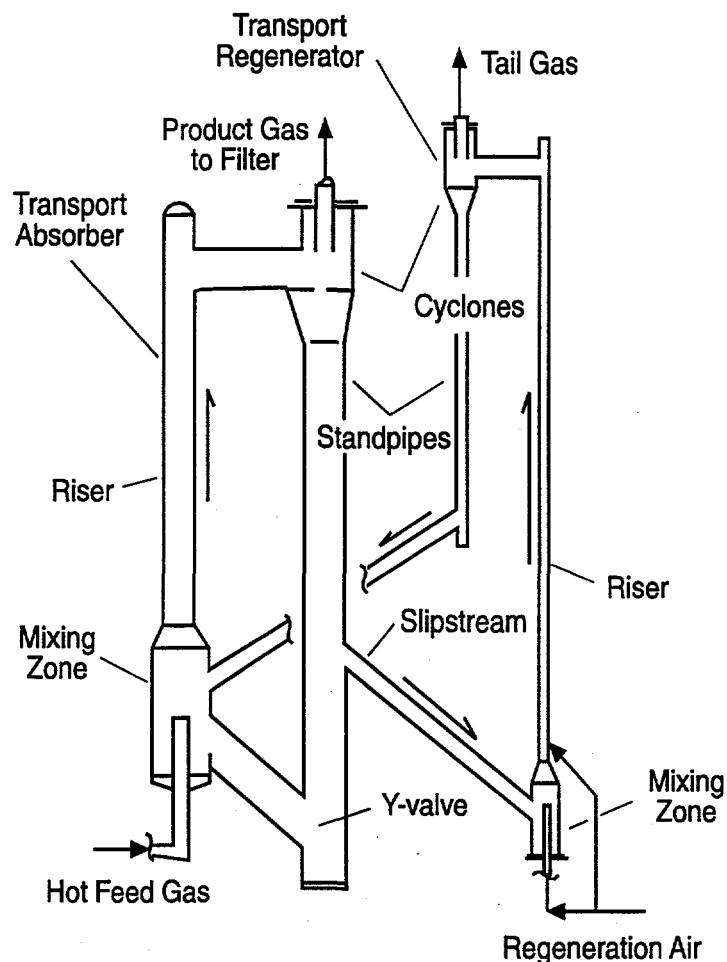
Figure 1. Schematic of Sierra-Pacific transport reactor system.

$$ZnTiO_3 + H_2S \rightarrow ZnS + TiO_2 + H_2O \qquad \text{(sulfider)}$$

$$ZnS + TiO_2 + \frac{3}{2} O_2 \rightarrow ZnTiO_3 + SO_2 \qquad \text{(regenerator).}$$
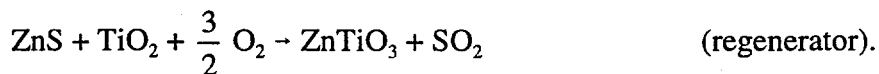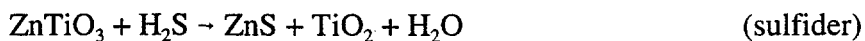
The zinc titanate sorbent continuously circulates between the two reactors. The SO_2 containing tail gas from the regenerator can be converted to salable products such as sulfuric acid or elemental sulfur or disposed of as calcium sulfate in downstream operations (17).

1.2   Ammonia Removal

The principal nitrogen contaminant released during coal gasification is NH_3. When coal gas is combusted in a gas turbine, NH_3 has the propensity to form oxides of nitrogen (NO_x) which are difficult-to-remove pollutants and precursors to acid rain and ozone. There are three potential methods of controlling NO_x emissions in an advanced IGCC plant (18,19). These include

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postcombustion selective catalytic reduction (SCR), two-stage combustion in the turbine and catalytic NH_3 decomposition ($NH_3 \rightleftharpoons [1/2] N_2 + [3/2] H_2$) upstream of the gas turbine. SCR, while a proven technology, would be very expensive downstream of the turbine due to a large volume, dilute tail gas. Scaleup will be difficult for the two-stage combustion approach. A somewhat analogous approach of selective catalytic oxidation of NH_3 using NO has also been investigated (20,21). The most attractive option is believed to be catalytic decomposition of the NH_3 in the HTHP coal gas since the high temperature is already available.

Studies of catalytic NH_3 decomposition under hot coal gas conditions are relatively few. A ZnO catalyst, a bulk iron catalyst, a $Pt-Al_2O_3$ catalyst, and an $Ag-Al_2O_3$ catalyst were tested under simulated coal gas conditions at 425 to 750 °C (21). Although these catalysts moderately reduced the NH_3 concentration, they were quickly poisoned in the presence of H_2S. A nickel-based catalyst from Haldor-Topsoe was shown to be effective for NH_3 decomposition under simulated coal gas conditions above 800 °C (18,19) in the presence of moderate levels of H_2S and at 550 to 725 °C in the presence of low parts-per-million levels of H_2S. However, moderate levels of H_2S quickly poisoned the catalyst in the 550 to 725 °C range. Molybdenum in its sulfided form (MoS_2) has been shown to have moderate activity for NH_3 decomposition (18). The addition of molybdenum to zinc titanate has also been shown to be somewhat effective for NH_3 decomposition (22). The use of very high temperatures (>800 °C) is one option for catalytic NH_3 decomposition. However, this temperature is available only upstream of the high-temperature particulate filter in an IGCC system. Thus, the catalyst would have to be active in the presence of particulates. The high temperature would also tend to sinter the catalyst (23,24).

1.3   Simultaneous H_2S and NH_3 Removal

An attractive approach appears to be to add a NH_3 decomposition catalyst to the zinc titanate sorbent and use the resulting sorbent-catalyst to simultaneously remove the H_2S and the NH_3 from hot coal gas. If successful, this approach would result in reducing the cost of the IGCC plant (22,25). However, the catalyst must be stable in both reducing and oxidizing atmosphere and in the presence of H_2S over multiple cycles.

1.4   Objective

The objective of this study is to develop and demonstrate catalytic approaches for decomposing a significant percentage (up to 90 percent) of the NH_3 present in coal gas to N_2 and H_2 at elevated temperatures (550 to 900 °C).

2.0 PREVIOUS WORK

Previous work until the end of FY97 under this contract was submitted as a Topical Report to DOE and is attached as Appendix 1. In this work, catalytic decomposition of up to 90 percent NH_3 in the presence of H_2S at temperatures >850 °C was demonstrated in both bench-scale and coal gasifier slipstream tests. Preliminary attempts at developing a combined sorbent-catalyst using zinc titanate for simultaneous H_2S and NH_3 removal were only moderately successful. Hampton University and the Research Triangle Institute (RTI) in a parallel study (DOE Contract No. DE-FG22-93MT93005) developed a zinc-based HART-49 sorbent-catalyst in laboratory research. The HART-49 sorbent-catalyst was a zinc-based sorbent-catalyst containing 5 wt% each of Ni, Co, and Mo (the preparation procedure is proprietary). The BET surface area of this material was 4.7 m^2/g. In a 1-g fixed-bed microreactor, at 1 MPa and 500 to 700 °C, this material showed decomposition of >90 percent NH_3 and removal of H_2S to <20 ppmv over a substantial portion of the desulfurization cycle. Also, the material, tested over 30 cycles, showed excellent regenerability. Thus the HART-49 sorbent-catalyst was chosen in this study for further development and scaleup in attrition-resistant form. Based on the research at Hampton University, the present work was undertaken at RTI with Hampton University as a subcontractor.

3.0 HART-49 SORBENT-CATALYST DEVELOPMENT

Using the HART-49 sorbent composition, a series of attrition-resistant fluidizable sorbent-catalysts (HART 50-55) were prepared with up to 75 percent binder. HART-55 gave the best combination of attrition resistance and performance in microreactor testing. HART-55 was scaled up to produced a 200-g batch (designated HART-56) which was selected for fluidized-bed bench-scale testing.

3.1   Experimental Apparatus and Conditions

The fixed-bed microreactor and bench-scale fluidized-bed reactor systems are shown in Figures 2 and 3, respectively.

The microreactor system consisted of a simulated coal gas delivery subsystem, a reactor with 1 g of sorbent-catalyst surrounded by a furnace, and a downstream sample collection, vent and gas analysis setup. The simulated coal gas and conditions used for the tests are shown in Table 1. This gas simulated a typical gas from an air-blown KRW coal gasifier. Steam was added using a high-pressure syringe pump feeding deionized water into a vaporizer. NH_3 and H_2S were added downstream of the vaporizer to prevent undesirable low-temperature reactions in the tubing.
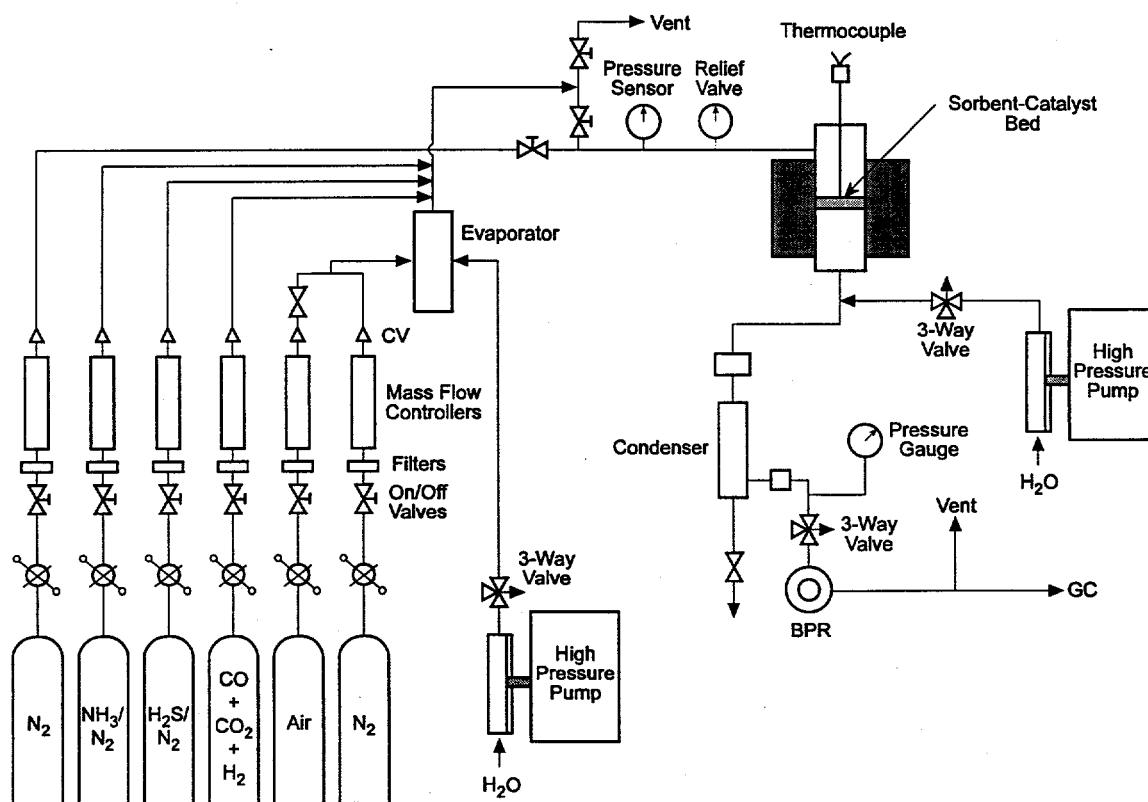

Figure 2. Fixed-bed microreactor sorbent-catalyst test unit.
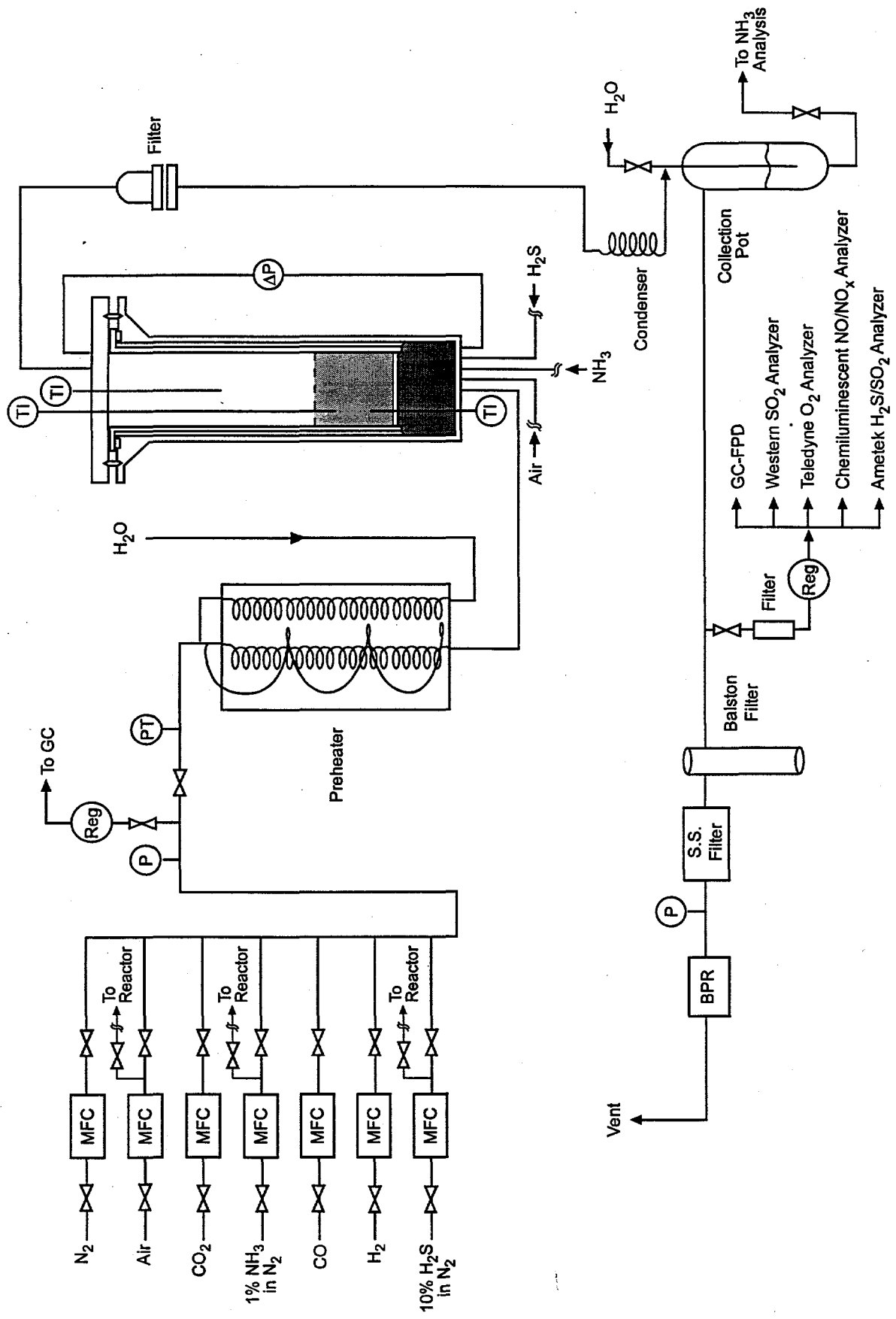
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Figure 3. Bench-scale fluidized sorbent-catalyst test unit.

The reactor was constructed using a ½-in. 316 stainless steel (SS) tube, aluminized on the inside (by Alon Processing, Tarentum, Pennsylvania) to prevent corrosion due to H_2S. A porous alumina frit in the middle of the tube was used to support 1 g of the material. A thermocouple was positioned to measure the sorbent-catalyst bed temperature.

The gas leaving the reactor was mixed with deionized water supplied by a pump at 0.5 cm^3/min to scrub out the NH_3. The liquid sample was collected in a catchpot-condenser and analyzed every 15 min for NH_3 breakthrough and to measure percent NH_3 conversion (removal). Thus, the NH_3 conversion was based only on the disappearance of NH_3 and products associated with NH_3 removal or decomposition were not analyzed. The NH_3 measurement in the liquid sample was conducted using an Orion Model 95-12 ammonia ion selective electrode (ISA). Independent measurements of the liquid samples for ammonium ion using a Dionex ion chromatograph gave about 10 to 15 percent higher values of NH_3 in the sample than the ISA.

The gas leaving the condenser was verified to be free of NH_3 by bubbling it through an impinger containing water over a long period of time and analyzing the water using the ISA. The gas was vented through a back-pressure regulator and sent to a gas chromatograph (GC) with flame-photometric detector (FPD) for measurement of H_2S during sulfidation and SO_2 during regeneration.

A typical cycle in a run consisted of flowing coal gas at desired temperature through the sorbent-catalyst bed. H_2S and NH_3 were periodically measured in the effluent. When H_2S broke through to a level of 100 to 500 ppm, the sulfidation was stopped, and regeneration was begun at 550 to 700 °C using 2 vol% O_2 in N_2. SO_2 was measured in the effluent gas and when SO_2 dropped below 50 ppm, the regeneration was stopped. Multicycle tests were conducted by repeating cycles in this manner.

The bench-scale fluidized-bed reactor system (Figure 3) was essentially a scaled-up version of the microreactor system, with minor differences. The reactor housed a 2.0 in. Alon-processed 316 SS sorbent-catalyst cage capable of testing up to 300 g of material. The NH_3 was collected in the water effluent and analyzed using ion chromatography. The effluent gas was analyzed for H_2S during sulfidation and SO_2, O_2, and NO/NO_x during regeneration. The simulated coal gas composition and sulfidation test conditions used for the microreactor and bench-scale reactor are shown in Tables 1 and 2.

3.2   Results and Discussion

The results of microreactor testing of HART-49 sorbent-catalyst powder are shown in Figures 4-6. The performance of HART-49 at 1 atm and 700 °C is shown in Figure 4. The H_2S removal below 20 ppm was maintained for 400 min prior to breakthrough in both the presence and absence of steam. NH_3 removal of >90 percent was maintained throughout the cycle in the absence of steam. Even with 15 percent steam, >90 percent NH_3 removal was maintained for 190 min. Results of the 10th, 20th, and 30th cycle for HART-49 at 700 °C and 1 atm (Figure 5) show consistent NH_3 and H_2S removal over 30 cycles by this sorbent-catalyst. The effect of tem-

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Table 1. Simulated KRW Gas Composition (vol%)

	Bench-scale fluidized bed	Fixed-bed microreactor
H_2	10.6	10.0
CO	17.0	15.0
CO_2	9.0	5.0
H_2O	5.0 to 15.0	0 to 15.0
H_2S	0.3 to 0.6	1.0
NH_3	0.16	0.18
N_2	Balance	Balance

Table 2. Reaction Conditions

	Bench-scale fluidized bed	Fixed-bed microreactor
Pressure (atm)	20	1 to 10
Temperature (°C)	550	550 to 700
Space velocity $\left(\dfrac{\text{std cm}^3}{\text{cm}^3 \cdot \text{h}} \right)$	7,500	2,500

perature on performance of the HART-49 sorbent is shown in Figure 6. Even at a temperature as low as 500 °C, NH_3 removal remained high for 70 min, up to 6 wt% sulfur loading on the sorbent.

At this point, modifications were made to the HART-49 powder to develop it in attrition resistant fluidizable form. The preparation is proprietary. After a number of trials, HART-55 sorbent was prepared. Its attrition resistance was better than commercial fluidized catalytic cracking (FCC) catalysts used extensively in refineries. A high level of binder was incorporated in the sorbent to impart it a high degree of attrition resistance.

The effect of pressure on the window of high NH_3 removal by HART-55 is shown in Figure 7. As seen, pressure promotes the removal of NH_3. This is contradictory to thermodynamic predictions and indicates the possibility of NH_3 chemisorption on the sorbent, in addition to decomposition. Some NH_3 release from the sorbent was observed during regeneration, which lends credence to the possibility of NH_3 chemisorption during sulfidation. Figure 8 shows the
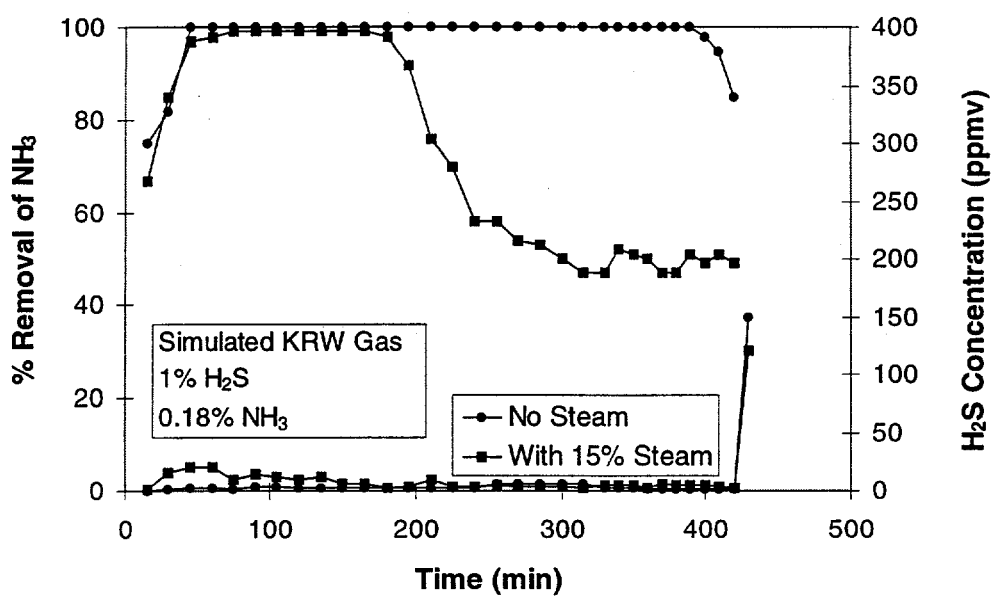
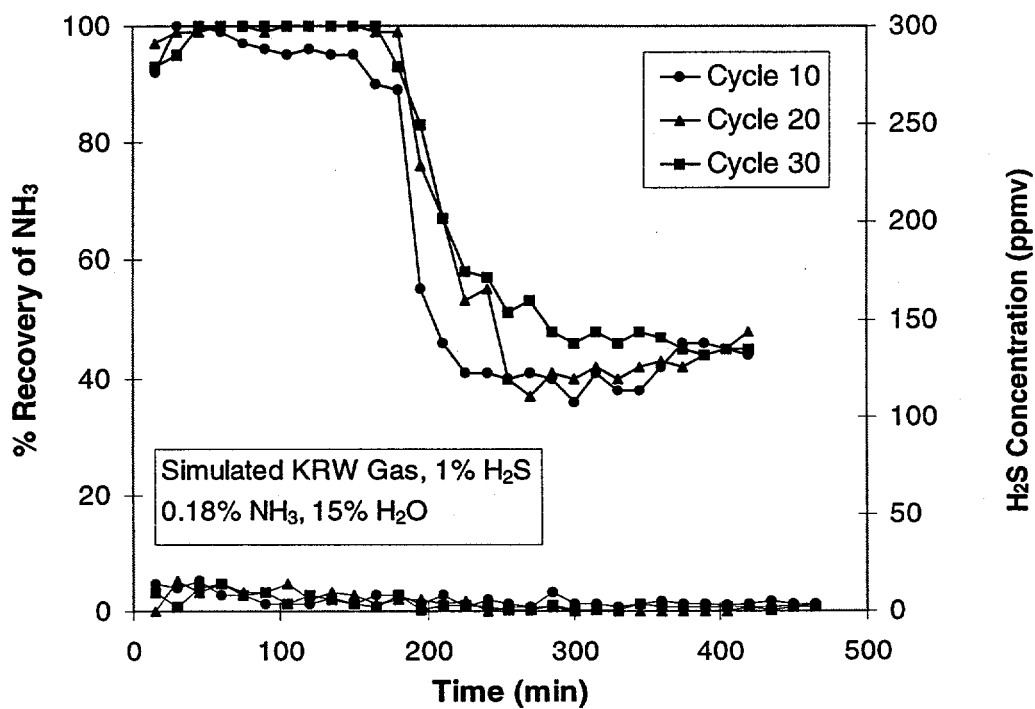**Figure 4. Simultaneous H₂S and NH₃ removal using HART-49 at 700 °C and 1.0 atm.**

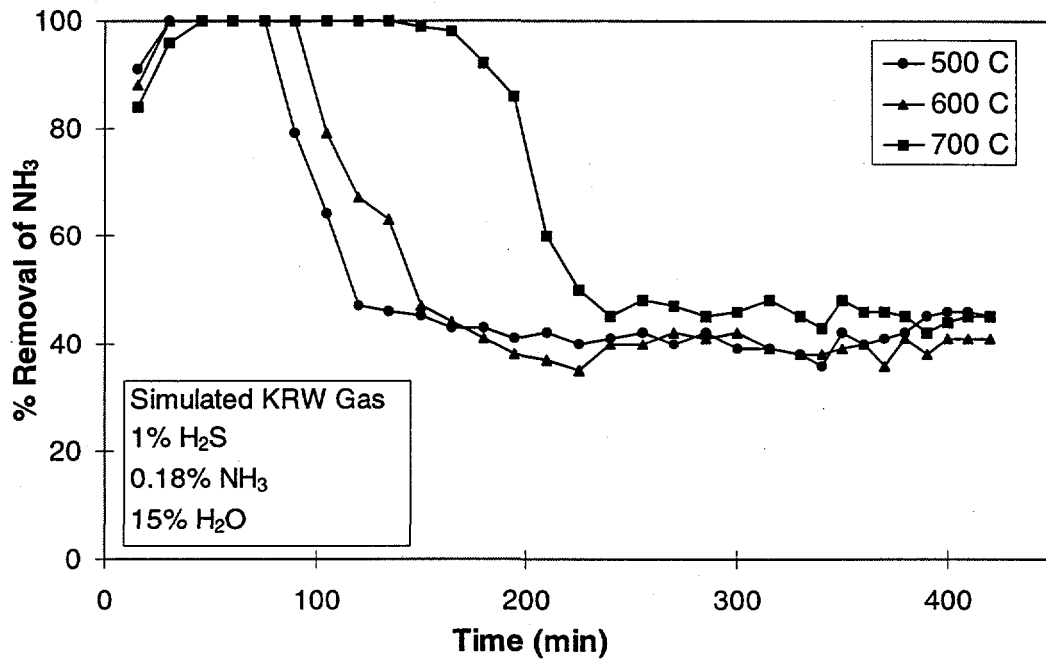**Figure 5. Multicycle NH₃ removal performance of HART-49 at 700 °C and 1.0 atm.**

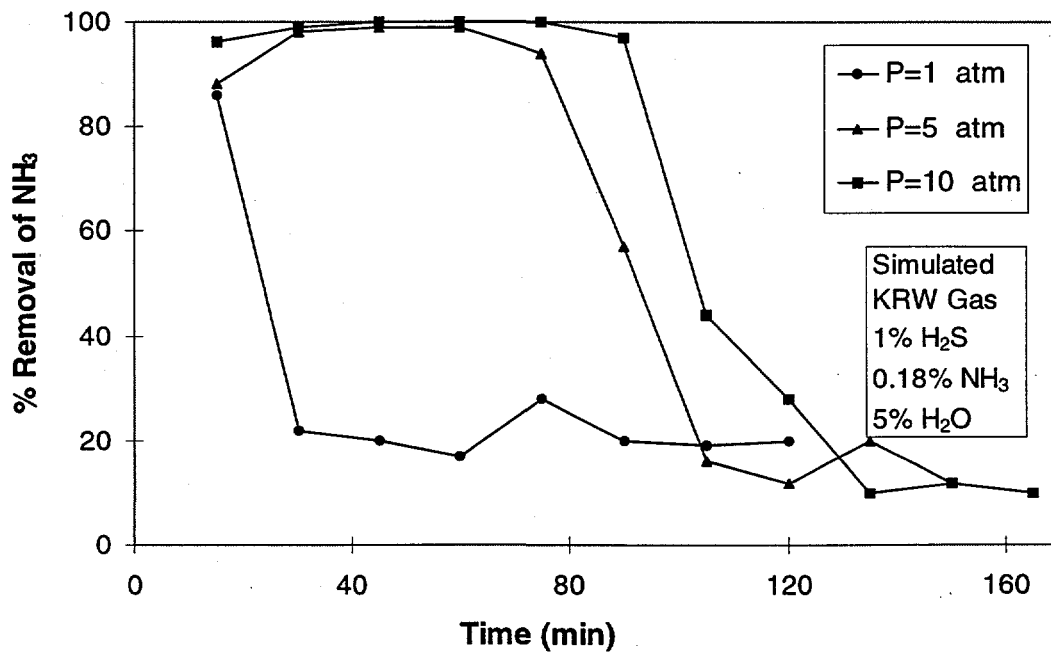**Figure 6. Effect of temperature on NH$_3$ removal by HART-49 at 1.0 atm.**

Figure 7. Effect of pressure on NH$_3$ removal by HART-55 at 550 °C.
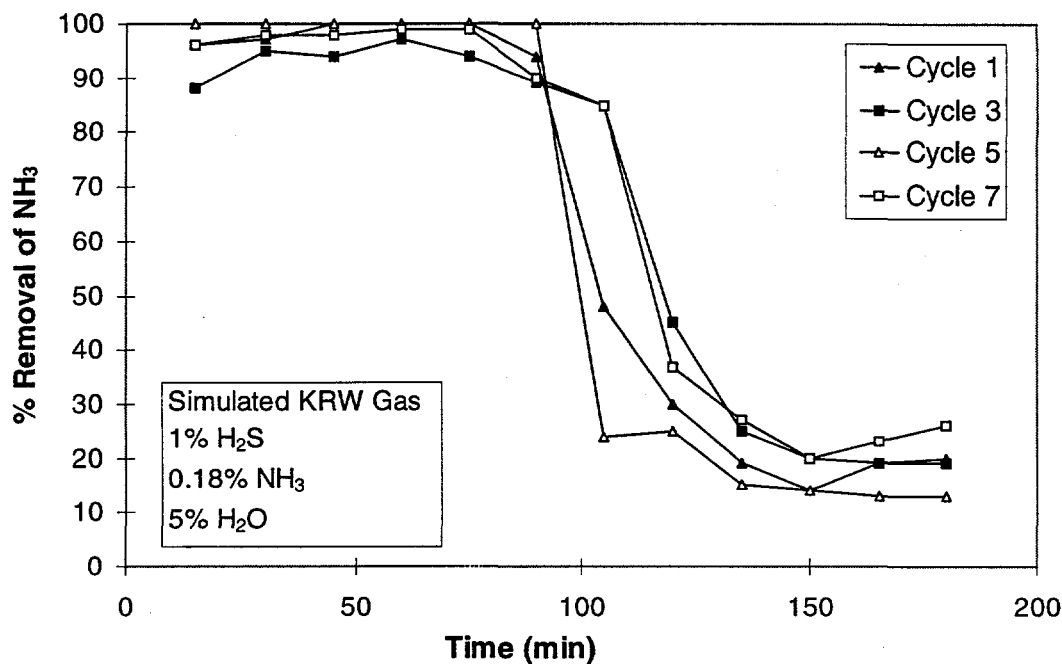
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Figure 8. Removal of NH₃ on HART-55 at 550 °C and 10 atm.

results of a seven-cycle test at 10 atm on HART-55. The consistent results over seven cycles show that the sorbent is behaving reproducibly after regeneration.

At this point the HART-55 sorbent-catalyst preparation was scaled up to prepare 200 g for the 20 atm fluidized-bed tests. The scaled-up material, designated HART-56, was tested over 20 cycles in the 2.0-in. bench-scale fluidized-bed reactor system. The average NH_3 and H_2S removal results for the first two cycles (with 5 percent steam in the KRW gas) are shown in Figure 9. A high degree of NH_3 and H_2S removal was achieved over the first two cycles, confirming the micro-reactor results of Figure 8. However, NH_3 was seen to elute from the material during purges and regeneration, again indicating chemisorption during sulfidation. Performance after the first two cycles, when steam in the coal gas was increased to 15 percent, deteriorated. The NH_3 balance over the 20 cycles is shown in Figure 10.

The reactor was purged with N_2 after both sulfidation (purge #1) and regeneration (purge #2) during the 20-cycle test. During regeneration, no NO or NO_2 was found in the regeneration tail gas. As seen, the average NH_3 accounted for during cycles 1 and 2 was about 59 percent, indicating only about 41 percent average removal during sulfidation. The average removal deteriorated significantly during cycles 3 to 20, carried out with 15 percent steam. The results indicate that HART-56 was not successful in achieving significant NH_3 removal at 550 °C and 20 atm except when 5 percent steam was present in the simulated coal gas. Also, reversible adsorption of NH_3 appeared to occur on the sorbent, which could be removed by purging.
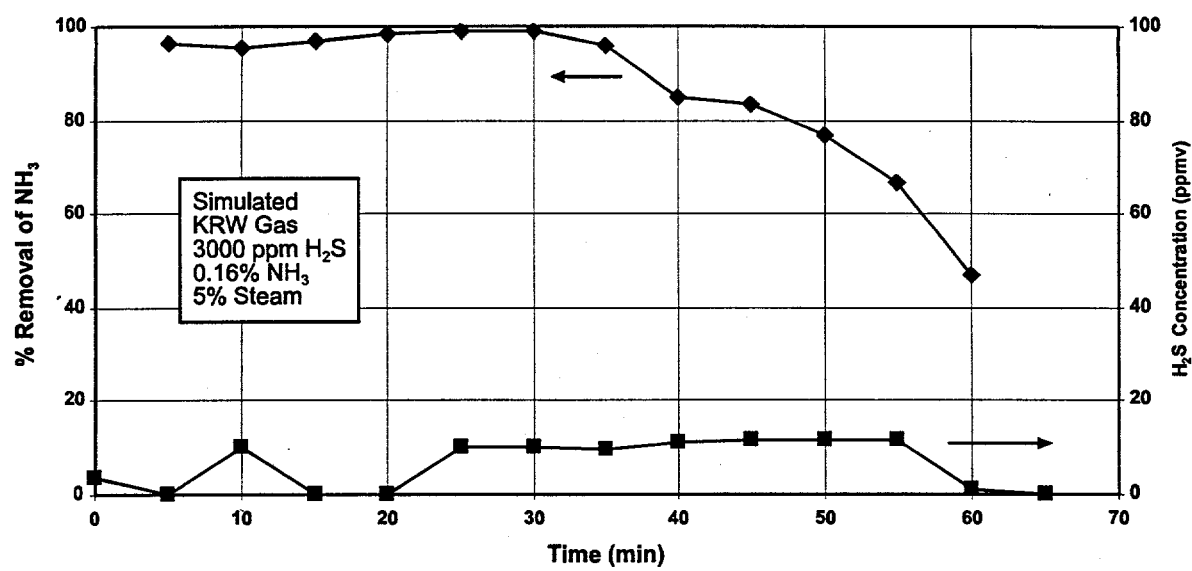
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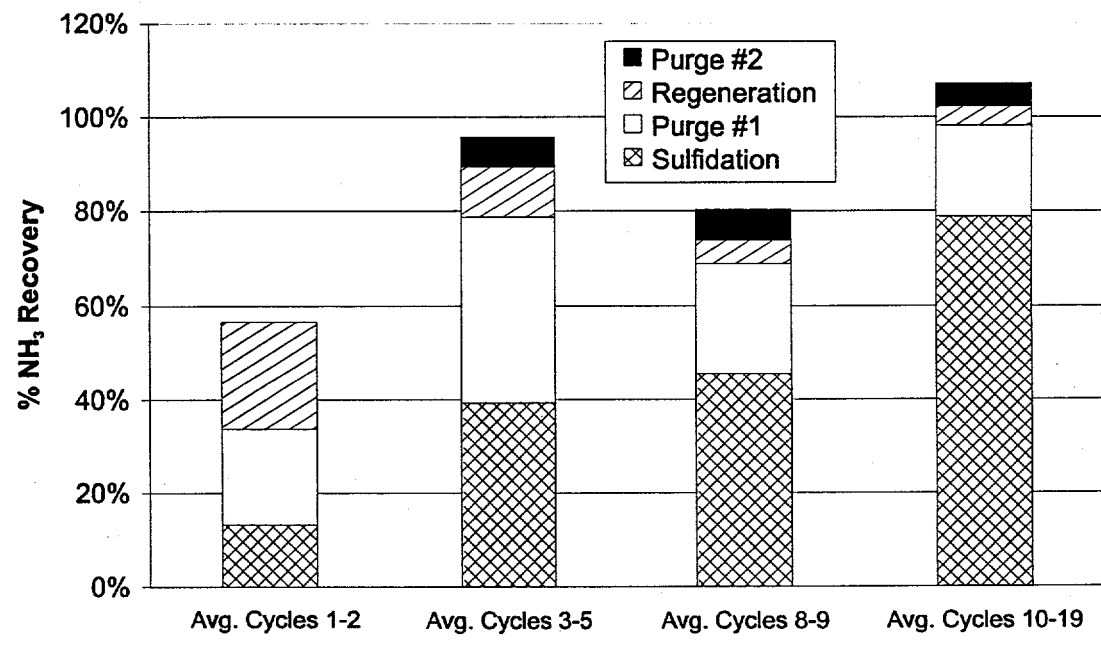**Figure 9. NH₃ and H₂S removal by HART-56 at 20 atm and 550 °C, bench-scale cycles 1 and 2.**

Figure 10. Summary of NH₃ removal and recovery in bench-scale tests.

4.0 CONCLUSIONS AND RECOMMENDATIONS

Successful development of a sorbent-catalyst for simultaneous removal of H_2S and NH_3 will result in reducing the overall cost of IGCC systems. HART-49, a lab-scale powder preparation (with high levels of active materials and no binder), showed promising results for simultaneous H_2S and NH_3 removal at 1 atm and 500 to 700 °C. The removal efficiency decreased as temperature decreased but increased significantly as steam concentration in simulated coal gas decreased. Addition of significant level of binder (up to 75 wt%), with corresponding reduction in the amount of active ingredients, resulted in a hard attrition-resistant sorbent-catalyst with better attrition resistance than FCC catalyst. However, its H_2S and NH_3 removal performance deteriorated. The NH_3 removal mechanism on the HART-49-based sorbent-catalysts is complex with reversible adsorption as well as decomposition occurring at higher pressure.

Further work is needed to optimize the sorbent-catalyst preparation to get a balanced material with acceptable attrition resistance, NH_3 removal performance, and cost. With high levels of binder used here, an attrition resistance better than needed was achieved but at the expense of reactivity. The NH_3 removal mechanism needs to be evaluated with fundamental studies, particularly with respect to adsorption behavior on the sorbent-catalyst at higher pressure and the dramatic effect of steam. The attrition-resistant HART-56 sorbent appears to have potential for removing 90 percent NH_3 and reduce H_2S to <20 ppmv, when 5 percent steam is present in the coal gas. However, further development of the material is needed for coal gas with higher steam.

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