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Hot Coal Gas Desulfurization with Manganese Based Sorbents

**Quarterly Report
August 1, 1993 - September 30, 1993**

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ABSTRACT

The focus of work being performed on Hot Coal Gas Desulfurization at the Morgantown Energy Technology Center is primarily in the use of zinc ferrite and zinc titanate sorbents; however, prior studies at the U. S. Steel Fundamental Research Laboratories in Monroeville, PA, by E. T. Turkdogan¹ indicated that an alternate sorbent, manganese dioxide-containing ore in mixture with alumina (75 wt% ore + 25 wt% Al₂O₃) may be a viable alternative to zinc-based sorbents. Manganese, for example, has a lower vapor pressure in the elemental state than zinc hence it is not as likely to undergo depletion from the sorbent surface upon loading and regeneration cycles. Also manganese oxide is less readily reduced to the elemental state than iron hence the range of reduction potentials for oxygen is somewhat greater than for zinc ferrite. In addition, thermodynamic analysis of the manganese-oxygen-sulfur system shows it to be less amenable to sulfation than zinc ferrite. Potential also exists for utilization of manganese at higher temperatures than zinc ferrite or zinc titanate.

This report documents progress in pelletizing and testing via thermogravimetric analysis of individual pellet formulations of manganese ore/alumina combinations and also manganese carbonate/ alumina with two binders, dextrin and bentonite. Preliminary results indicate that the manganese carbonate material, being of higher purity than the manganese ore, has a higher degree of sulfur capacity² and more rapid absorption kinetics.

A 2-inch fixed-bed reactor has been fabricated and is now ready for subjecting pellets to cyclic loading and regeneration.

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¹Turkdogan, E. T. and Olsson, R. G.: "Desulfurization of Hot Reducing Gases with Manganese Oxide Pellets", *Proceedings Third International Iron and Steel Congress* ASM Metals Park, Ohio, 1979, pp.277-288.

²Capacity is defined as grams of sulfur absorbed per 100 grams of indurated pellets.

INTRODUCTION

Zinc ferrite and zinc titanate are being developed as regenerable sorbents capable of removing hydrogen sulfide from hot coal gases. Utilization is only about 10% sulfur loading, however, and zinc ferrite tends to spall and be limited in temperature to effective utilization below about 600°C.

Manganese-based sorbents combined with alumina to form indurated pellets hold promise of being highly-effective, inexpensive, regenerable sorbents for hot fuel gases. These sorbents are effective in making very strong (>50 pounds crush strength for a 4-mm pellet), which can be loaded and regenerated at higher temperatures than zinc ferrite (which is limited to about 1100 to 1200°F or 593° to 649°C), and which appear to be capable of improved loading with each cycle and also load sulfur to a higher capacity. The thermodynamics for sulfur removal by manganese predicts sulfur removal to terminal levels (in terms of the over-pressure of hydrogen sulfide) somewhat less favorable than can be accomplished with zinc oxide; however, zinc can be reduced under coal gasification conditions and tends to volatilize, limiting the temperatures for which desulfurization can be effectively accomplished to values less than 700°C.^{3,4} Also the capabilities for regeneration of manganese pellets (with an alumina filler @ 75 wt% Moanda ore, 25 wt% Al₂O₃) under oxidizing conditions may be superior to those of zinc ferrite in that they can be loaded from a reducing gas and regenerated at higher temperatures (regeneration up to 1300°C). Upon regeneration of the loaded sorbent, sulfur can be obtained from the off-gases by combination of the products of the regeneration: hydrogen sulfide and sulfur dioxide or alternatively, a high strength sulfur dioxide gas mixture suitable for sulfuric acid manufacture can be produced.

A significant domestic source of manganese in Minnesota is being explored for an *in situ* leach process which has potential for producing large tonnages of solutions which may be ideal for precipitation and recovery of

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³Woods, M. C., Gangwal, S. K., Jothimurugesan, K., and Harrison, D. P.: "Reaction between H₂S and Zinc Oxide-Titanium Oxide Sorbents. 1 Single-Pellet Kinetic Studies", *I & EC Research*, 1990, 29, p. 1160-1167.

⁴Hepworth, M.T. and Jha, M.C., "Enhanced Durability and Reactivity for Zinc Ferrite Desulfurization Sorbent," *Proceedings of the Eighth Annual Gasification and Gas Stream Cleanup Systems Contractors Review Meeting*, Vol. 1, DOE/METC-88/6092.

pure manganese as a carbonate in a reactive form.⁵

In this current program the following studies are being addressed:

- preparation and characterization of manganese-based sorbents;
- thermodynamics and kinetics of sulfur removal from hot fuel gases by this sorbent;
- cyclic loading and regeneration of the sorbent by oxidation and recovery of the sulfur.

This study will extend earlier studies made at the laboratories of U. S. Steel undertaken by Dr. E. T. Turkdogan. Those studies, which utilized a manganese ore blended with alumina, were successful in achieving desulfurization from reducing gases. This earlier work was not undertaken with the gas mixtures which are proposed for this study, nor were the manganese formulations the same as are planned; however, the initial success of the work of Turkdogan indicates that significant progress can be made in moving forward toward developing a high-temperature regenerable sorbent.

The developed information will be of value to METC in determining whether or not a manganese-based regenerable sorbent holds real promise for sulfur cleanup of hot fuel gases. This information is necessary before pilot-scale testing leading to commercial development is undertaken.

Current Status of Test Work:

The following tasks have been completed (★), are being undertaken (◆), or will be undertaken (■) during the next 12 months of the contract:

- 1) (★)Providing Information Required for the National Environmental Policy Act;
- 2) (★)Sorbent Preparation and Characterization Tests on Individual Pellets
- 3) (★)Provision for Bench-Scale Test Unit
- 4) (■)Bench-Scale Testing
- 5) (■)Data Analysis

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⁵Hepworth, M. T.: *Selective Mining Methods for Minnesota, MN Initiative Project: Manganese Recovery from In-Situ Leach Liquors*, Final Report to U. S. Bureau of Mines from the Mineral Resources Research Center 1992.

The program consists of the following studies:

- Establish the thermodynamic feasibility for the use of manganese-based sorbents for the removal of sulfur in comparison with other oxide systems.
- Prepare and characterize manganese sorbent pellets. Initial characterization is based upon strength and surface area.
- Determine the comparative kinetics of loading and regeneration of individual sorbent pellets via thermogravimetric analysis (TGA). Preliminary TGA tests are showing that sorbents prepared with the organic binder, dextrin, on Moanda ore appear to offer the best combination of strength, reactivity, and capacity. Sorbents prepared with bentonite on manganese carbonate appear to offer the best combination of capacity and reactivity. Further testing will utilize these two leading sorbents.
- Build a two-inch reactor fixed-bed test unit. This unit is currently built and being tested.
- Perform bench-scale tests on the optimum sorbent pellets in a two-inch diameter reactor.

THERMODYNAMIC CONSIDERATIONS

The prior quarterly report detailed thermodynamic considerations for the use of manganese as a sorbent and will not be repeated here.

EXPERIMENTAL PROCEDURES AND RESULTS

Since the prior studies by Turkdogan and Olsson were conducted on a manganese ore (Moanda ore), the starting point for the present investigation is using similar materials for comparison. Then manganese oxide derived from pure carbonate, similar to that which would be recovered from in-situ mining is studied. Accordingly, the following materials are being utilized: (Chemical analyses were reported in the Second Technical Report [12/1/1992 to 2/28/93])

- MnO_2 Moanda ore (Africa);
- Ultrafine MnCO_3 (93-95%) (Chemetals, Inc.);
- Manganese carbonate recovered from *in situ* leach liquors produced by the Salt Lake City Research Center of the U. S. Bureau of Mines
- Alundum (96.6% Al_2O_3) (Norton Materials, Inc.);
- Bentonite, inorganic binder (Aldrich Chemical Co.); and
- Dextrin, an organic binder.

Pelletization:

The procedures followed for pelletization were reported in the prior quarterly report.

Following the above procedure, six formulations were prepared using combinations of Moanda ore, manganese carbonate from Chemetals and manganese carbonate from the USBM, alundum, bentonite, and dextrin. The composition of the initial make up of samples is reported in Table 1.

Table 1

Formulation No.	Raw Material Content in initial make-up, wt.%					
	Moanda Ore	Alundum	MnCO ₃ (Chemetals) ⁶	MnCO ₃ (USBM) ⁶	Bentonite	Dextrin
1	68.18	22.73	-	-	-	9.09
2	73.53	24.51	-	-	1.96	-
3	-	15.51	75.4	-	-	9.09
4	-	16.73	81.81	-	1.96	-
5	-	15.51	-	75.4	-	9.09
6	-	16.73	-	81.31	1.96	-

Induration:

Twenty-four induration campaigns were conducted. As established by previous tests, Moanda-ore base pellets required a temperature of 1200°C and manganese-carbonate-based pellets required an induration temperature of 1250°C, to produce required strength minima (> 5 lbs per mm of pellet diameter). In addition two time periods for induration were explored, namely, 1 hour and 2 hours.

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⁶Calcination of manganese carbonate effectively reduces its mass upon loss of carbon dioxide. Precalcination mass was chosen to produce 75% by wt. MnO.

Two induration procedures were followed:

In both procedures, the pellets were dried to a constant weight at 110°C prior to induration.

- In the first procedure which was applied to the first 12 tests summarized in Table 2 below, the samples were pre-weighed and slowly introduced into a furnace already at the required temperature. After residence at temperature for the required time, they were slowly extracted and allowed to air-cool and then re-weighed. These weights are reported as W_o and W_z in the Table. Unfortunately this procedure produced thermal stresses resulting in cracking of the pellets.
- In the second procedure, the pellets were introduced into the furnace at 500°C and then furnace-heated to the desired induration temperature over a heating cycle of 150 minutes. (The manganese carbonate-based pellets, however, were allowed to dissociate to the oxide in a prior step at 350°C. For these pellets the weight of the decomposed pellets for some of the tests is reported as W_n in Table 2 to serve as a check to determine whether the decomposition was complete.)

In interpreting Table 2, the recorded weight loss for the ore-based pellets is associated with the loss of structurally-bound water and also the loss of oxygen as MnO_2 is transformed to Mn_3O_4 , the stable form of manganese under induration conditions.

Table 2: Induration Conditions

Test I.D.	Formulation No.	Temp.,(°C)	Duration (hrs)	W _o	W _{f1}	W _{f2}
IN93G29A.R33	1	1200	2	25.69	-	21.01
IN93G29B.R33	2	1200	2	27.04	-	23.77
IN93G29C.R33	1	1200	1	21.16	-	17.31
IN93G29D.R33	2	1200	1	26.21	-	23.08
IN93G29E.R34	3	1250	2	43.59	-	28.01
IN93G29F.R34	4	1250	2	41.40	-	29.31
IN93G29G.R34	3	1250	1	33.80	-	21.72
IN93G29H.R34	4	1250	1	32.76	-	23.20
IN93G29I.R35	5	1250	2	14.10	-	9.01
IN93G29J.R35	6	1250	2	16.17	-	11.41
IN93G29K.R35	5	1250	1	15.46	-	9.85
IN93G29L.R35	6	1250	1	12.90	-	9.15
IN93G30A.R36	1	1200	2	13.58	-	11.10
IN93G30B.R36	2	1200	2	16.83	-	14.72
IN93G30C.R36	1	1200	1	12.68	-	10.37
IN93G30D.R36	2	1200	1	20.37	-	17.88
IN93G30E.R37	3	1250	2	21.55	14.94	13.81
IN93G30F.R37	4	1250	2	24.17	19.20	16.90
IN93G30G.R37	3	1250	1	26.85	18.75	17.22
IN93G30H.R37	4	1250	1	26.19	20.93	18.55
IN93G30I.R38	5	1250	2	10.66	7.79	6.03
IN93G30J.R38	6	1250	2	4.00	3.32	2.84
IN93G30K.R38	5	1250	1	10.50	8.25	6.26
IN93G30L.R38	6	1250	1	2.09	1.79	1.50

Figure 1 displays the strength of representative pellets from the prior twenty-four induration tests each assigned an identification letter (I.D.) ranging from A₁ through L₁ for the first half of Table 2 and A₂ through L₂ for the second half of the table. The pellets were subjected to a crush test using an anvil and hydraulically loaded plate equipped with a pressure sensor. Tests were made on pellets which were approximately 4 mm in diameter. The loads are expressed in pounds per mm of diameter. Since the equipment is only capable of sensing loads of less than 9 pounds per mm of pellet diameter, identifications: B₁, D₁, B₂, and D₂, are shown as off-scale and represent values significantly higher than indicated.

From this table the strongest formulations are bentonite-bonded as expected. At the conclusion of these tests, four samples were selected for TGA analysis. They were given the following designations: FORM1-A, FORM2-A, FORM2-B, and FORM4-A. Letter "A" corresponds to induration for 2 hours at 1200°C for the ore-based pellets, and 1250°C for carbonate-based pellets; whereas, letter "B" corresponds to an induration time of 1 hour.

Chemical Analyses:

Table 3 summarizes the manganese assays and strengths of the formulations which were subjected to TGA analysis.

Table 3

Sample Designation	Weight %Mn	Strength, lbs/mm
FORM1-A	41.14	5.8
FORM2-A	37.48	14.7
FORM2-B	39.83	11.9
FORM4-A	44.22	5.4

Pellet Porosity and Surface Area:

BET surface area tests on indurated pellets have shown them to have a surface area less than 0.2 m²/gm, which is below the sensitivity of our equipment and therefore required krypton as a carrier gas. We are attempting to increase pellet surface area; however, TGA testing at this time does show them to be about as reactive as the zinc titanate pellets which have been studied by other investigators. Typical specific surface area values are as follows:

FORM2-A: 0.055 m²/gm
 FORM1-A: 0.127 m²/gm

Porosity tests made by combination of aqueous pycnometry on crushed pellets from formulation 2 in combination with mercury pycnometry showed the pellets to have a porosity of about 36%. In addition, several characterization measurements are being made with the help of outside laboratories, namely, the Twin Cities Research Center of the U. s. Bureau of Mines and also through METC.

Tests via TGA on Pellet Reactivity:

The TGA testing is divided into two campaigns the first being only comparative loading for one cycle, and the second for regeneration studies after repeated cyclic loading and regeneration. The second campaign activity is described in the section entitled Future Plans and will be executed in the next quarter.

During the first campaign, emphasis was placed upon studying the kinetics of sulfidation using the above four sorbents. The reaction variables included temperature (700° to 1000°C), H₂S content (0.5%, 1%, 2%, and 3% in H₂/H₂S gas mixtures), and pellet diameter of approximately 3, 5, and 8 mm. As was established in prior reports, the reduction power of the reducing gas is not sufficient in the sulfiding atmospheres chosen to allow reduction of MnO to Mn°. For these same reasons, it was not considered important in these scoping tests to consider the kinetics of reduction since there are no losses to be expected by evaporation as would be the situation with zinc-based sorbents.

Figure 2 is a schematic diagram of the experimental equipment employed for TGA testing. The lower right-hand portion of the diagram shows the pan assembly which suspends up to twelve pellets in a platinum basket hung via nichrome extension wire from a Cahn 1000-Series microbalance. The sample and hangdown assembly are contained in an alumina reaction tube, which is positioned in a high-temperature molybdenum-wound furnace. The gas delivery systems and data acquisition systems are as depicted. Vent gases are scrubbed in a sodium hypochlorite solutions prior to flaring and venting.

The results of these TGA tests are shown in Figures 3 through 8, studies on loading of pellets described below:

Figure 3 shows results of TGA analysis on the four selected sorbents which were described above. In this plot and each of the plots (Figs 4-8) which follow, the weight of the pellets divided by the initial weight is expressed on the ordinate as a weight ratio: W/W_0 . Initially, this ratio for all pellets is unity; however, there is a decline in this value (reduction of Mn₂O₄ to

MnO) when the pellets undergo reduction with hydrogen during the initial 20 minutes. The actual reduction data are not shown on this plot but rather a minimum asymptotic value is reached within about ten minutes and is seen in these plots as a horizontal line. After the passage of 45 minutes of prior reduction, all pellets remained constant in weight for at least 20 minutes.

At this time, hydrogen sulfide is added at 3% by volume to the gas which is flowing at a rate of 500 cc per minute. In the lower right-hand corner of the diagram are the theoretical maximum weight ratios which the pellets could exhibit based upon total conversion of the manganese in the pellet, as determined by chemical analysis, to the stoichiometry: MnS.

Upon examining these calculated values, one should note that the formulation which resulted from manganese originating from purified carbonate (i. e., FORM4-A) has a greater capacity for sulfur than the formulations which originate from the ore. Furthermore, dextrin-based formulations also yield higher sulfur capacities based upon the fact that dextrin volatilizes upon induration of the pellet; whereas, bentonite is a non-volatile impurity which effectively lowers the manganese content of the pellet.

The asymptote for time periods exceeding 90 minutes for formulation 4-A actually exceeds the calculated value for sulfur capacity, which may indicate that the original sulfur assay is slightly on the low-side. The asymptotic value for formulation 1-A is very close to the calculated value; whereas, the asymptote for 2-A (Moanda ore plus bentonite) falls below the calculated value indicating that some of the manganese may be chemically combined with silicate compounds in the ore and therefore not available to react with hydrogen sulfide.

In continuing the examination of Figure 3, one should note the rapid kinetics upon switching to the loading gas, which is most pronounced in formulation 4-A, i. e. manganese carbonate plus bentonite. Formulation 1-A (ore plus dextrin) has somewhat slower kinetics followed by formulation 2-A (ore plus bentonite).

Figure 4 shows similar results for tests conducted at 900°C. In this Figure, formulation 2-B (ore plus bentonite indurated for 1 hour at 1200°C) has been included in the analysis. Again formulation 4-A shows the highest sulfur capacity and the most rapid kinetics of the four pellet groups.

From Figure 5 it is seen that tests conducted at 800°C show somewhat slower kinetics again with the pellet groups showing similar trends in

kinetics. Pellet groups 1-A; 2-B and 2-A do not reach their asymptotic values for complete loading within the time frame shown (i. e. three hours).

In Figure 6 for loading studies at 700°C the kinetics are significantly slower so that only a fraction of the pellet capacity is utilized for all four formulations within the time-frame of the three-hour test regime.

Figure 7 is a composite of the data for formulation 4-A, the best of all the formulations studied, for the temperatures 700, 800, 900, 950, and 1000°C. The theoretical capacity for this formulation is shown as a dashed line on the plot with reactivity rates for all temperatures (except 700°C) being rapid. It should be noted that earlier pellet strength tests on manganese pellets originating from the carbonate and indurated at 1200°C were even superior in reactivity to similar pellets indurated at 1250°C; however, the strengths of these pellets indurated at the lower temperature were not sufficient to maintain the strength requirements which were imposed.

Figure 8 is a similar plot to Figure 7 except that it is for formulation 1-A.

Regeneration tests:

Regeneration tests were limited to gas atmospheres containing air plus nitrogen. They were not conducted in the TGA system shown in Figure 2 but rather they were conducted on twenty pellets contained in a ceramic "boat" placed within a horizontal tube furnace subjected to loading in hydrogen plus hydrogen sulfide (3% H₂S) for exposure times of four hours at 800°C. In Figure 9 the time requirement to reach 90% regeneration is plotted against the number of cycles (expressed as "Run #") on the abscissa. For this plot formulation 1 was used. The regeneration was conducted at a temperature of 1000°C. The sulfur emerging from the system was determined by titration of the exit gas after it was sparged through an alkaline solution of peroxide and the amount of titer measured to calculate the quantity of sulfur emitted. The percent regenerated is actually determined as a function of the asymptotic value associated with no further demand for titer in the sparged solution. Typically, regeneration was complete in about 20 minutes; however, the regeneration was allowed to proceed for one hour to assure no further reaction.

In this figure (Figure 9), the time to reach 90% regeneration is seen to be initially high for fresh pellets and decline as the number of cycles increased. This increased rate of regeneration may be a result of cracking of pellets, which was observed under an optical microscope. This

improvement in reaction kinetics with repeated cycling is consistent with earlier observations by Turkdogan. Figure 9 shows that the per cent of the theoretical sulfur released is relatively constant at 85% over the ten cycles studied.

Finally in Figure 10, the kinetics of the regeneration tests are exhibited by examining the rate of titration of the acid gas released. It is seen that there is actually an increase in rate of regeneration with increasing cycles of loading with cycle 10 exhibiting significantly more rapid regeneration kinetics than cycle 2. Again this effect may be a result of micro-cracks in the pellet. Further study on the kinetics of cyclic loading and regeneration via TGA analysis is required along with microscopic examination of the pellets in both loaded and regenerated condition is required before final conclusions can be reached regarding the mechanism of this enhanced effect.

BENCH-SCALE TEST UNIT

A reactor system, pictured in Figure 11 has been constructed and tested. This Figure, which is a schematic of the reactor detail, shows the two-inch (I.D.) closed-end alumina reactor furnace tube equipped with an alumina perforated-disc plate suspended over alumina chips. A simulated coal gas is passed through a small central tube (without contacting the reaction zone of manganese pellets) through the perforated disc and into a preheat zone of alumina chips which supports the disc. The gas is then discharged from the small central tube into the larger tube and moves upward, after reaching temperature, into gas preheat zone of a two-zone furnace. The gas, now at temperature, ranging from 800 to 1100°C then contacts the sorbent bed consisting of pellets ranging in size from 4 to 5 mm in diameter. The off gas then is flared and discharged to a hood. A sample is taken via gas chromatograph (labeled in Figure 11 as GC).

The overall Schematic Diagram of the Experimental Arrangement for the Fixed-Bed Tests is shown in Figure 12. The split or two-zone furnace is positioned with respect to the bed to accomplish gas preheating and careful control of the bed temperature. Gas combinations for flows can be produced via control of bottled gases through the valves and flow-meters shown in the diagram. The water content of the system is controlled by a metering pump acting on liquid water which is discharged into a heated stainless tube to vaporize the water and provide the gas composition required by the Tampella-U Gas composition as mentioned above.

Initial testing will, however, be conducted using only hydrogen in

combination with hydrogen sulfide in a manner similar to that conducted by E. T. Turkdogan in his studies as discussed previously. After corroboration of his test results, then the gas mixture referred to above will be used as a loading gas.

Loading will continue at a rate of 2.5 liters per minute until break-through is observed. Breakthrough is defined as a sudden rise in the sulfur content of the gas as determined by gas chromatography. The bed will then be flushed with nitrogen in order to expel remaining hydrogen and a regeneration gas consisting of air plus nitrogen will be initiated with the sulfur content of the gas being continuously analyzed for sulfur dioxide. The regeneration step will be discontinued when the bed is 95% regenerated as determined by an approximate mass balance of the system based upon the calculated sulfur content of the off gas. The loading and regeneration cycles will then be repeated until at least ten cycles have been completed.

SUMMARY AND CONCLUSIONS TO DATE

Four sorbent formulations have been prepared using Moanda ore and manganese carbonate as starting materials in combination with alumina. Both dextrin and bentonite have been employed as bonding materials for pellet formation; however, dextrin, which appears to produce favorable porosity and is volatilized upon induration is a preferred binder. All pellets made met strength requirements. The carbonate required an induration temperature of 1250°C; whereas, the oxide (ore) requires an induration temperature of 1200°C.

Single loading tests via TGA analysis at temperatures ranging from 700°C to 1000°C show that the manganese carbonate source material bonded with bentonite exhibits the best combination of loading capacity and kinetics. Higher temperatures for loading (i. e. 800° to 1000°C) show much more favorable kinetics than at 700°C; however, the thermodynamics of sulfur removal in terms of the terminal sulfur levels achievable become less favorable (as described in the prior Quarterly Report).

A limited series of regeneration tests show that the kinetics of regeneration appear to improve with cyclic loading possibly as a result of micro-crack formation as seen by optical microscopy. These radial microcracks appear not to significantly impair pellet strength.

FUTURE WORK PLANS

Future work will continue characterization tests on individual pellets followed by bench scale testing in a two-inch reactor of the optimum pellet formulation as determined by on-going TGA testing.

- Prepare and characterize manganese sorbent pellets. Initial characterization is based upon strength and surface area.
- Determine the comparative kinetics of loading and regeneration of individual sorbent pellets via thermogravimetric analysis (TGA).
- Perform bench-scale tests on the optimum sorbent pellets in a two-inch diameter reactor. There now appear to be two formulations: manganese carbonate-based bonded with bentonite and indurated for 2 hours at 1250°C, and manganese ore-based bonded with dextrin and indurated at 1200°C for 2-hours.

Figure 1. Strength of Pellets from Several Induration Campaigns

FIGURE 2. SCHEMATIC DIAGRAM OF ASTRO-TGA APPARATUS

Figure 3. Normalized Weight vs. Time for Sulfidation of Three Formulations at 1000°C in a 3% H_2S - H_2 Gas Mixture.

Figure 4. Normalized Weight vs. Time for Sulfidation of Three Formulations at 900° C in a 3% H₂S-H₂ Gas Mixture.

Figure 5. Normalized Weight vs. Time for Sulfidation of Three Formulations at 800°C in a 3% $\text{H}_2\text{S-H}_2$ Gas Mixture.

Figure 6. Normalized Weight vs. Time for Sulfidation of Three Formulations at 700°C in a 3% $\text{H}_2\text{S-H}_2$ Gas Mixture.

Figure 7. Effect of Temperature on Sulfidation Kinetics of Sorbent FORM4-A

Figure 8. Effect of Temperature on Sulfidation Kinetics of Sorbent FORM1-A

Figure 9. Time Required to Reach 90% Regeneration for Several Sulfidation and Regeneration Cycles

Figure 10. Comparison of Regeneration Kinetics of Recycled Pellets

Figure 11. Schematic of Fixed-Bed Reactor Details

Figure 12 Schematic of Overall Experimental Arrangement for Fixed-Bed Tests

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