Investigation on Durability and Reactivity of Promising Metal Oxide Sorbents During Sulfidation and Regeneration

Quarterly Report

April 1 - June 30, 1996

Work Performed Under Contract No.: DE-FG21-94MC31206

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

By
Chemical Engineering Department
School of Engineering and Architecture
Tuskegee University
Tuskegee, Alabama 36088

MASTER

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ABSTRACT

Hot-gas desulfurization for the integrated gasification combined cycle (IGCC) process has been investigated by many researchers to remove effectively hydrogen sulfide with various metal oxide sorbents at high pressures and high temperatures. Metal oxides such as zinc titanate oxides, zinc ferrite oxide, copper oxide, manganese oxide and calcium oxide, were found to be promising sorbents in comparison with other removal methods such as membrane separations and reactive membrane separations. Some metal oxide sorbents exhibited the quite favorable performance in terms of attrition resistance and sulfur capacity. Removal reaction of H_2S from coal gas mixtures with promising sorbents of fine solid was carried on in a batch reactor.

The objectives of this research project are to formulate promising durable metal oxide sorbents for the removal of sulfur compounds from coal gas mixtures, to find intrinsic initial reaction kinetics for a sorbent-hydrogen sulfide heterogeneous reaction system, to obtain effects of concentrations of coal gas components such as hydrogen and moisture on equilibrium absorption as well as dynamic absorption of hydrogen sulfide into formulated sorbents for the reaction system at various reaction temperatures and pressures, and to obtain effects of concentrations of coal gas components such as hydrogen and moisture on reaction rates of hydrogen sulfide with formulated sorbents at various reaction temperatures and pressures.

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SCHEDULE AND MILESTONES

	FY 1994-1995												FY 1995-1996											
	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S
Procurements of Materials and Equipment																								
Fabrication of a Micro Batch Reactor																								
Development of Analytical Procedures for Concentrations of Sulfur compounds																								
Development of Experimental Procedures for Sulfidation Reaction of Fresh Metal Oxide Sorbents																								
Formulation of Durable Metal Oxide Sorbents with High-Sulfur Removal Capacity																								
Reaction Kinetics on Sulfidation of Metal Oxide Sorbents																								
Reaction Kinetics on Regeneration of Sulfur-loaded Metal Oxide Sorbents																								
Development of an Intraparticle Diffusivity Model for Sulfidation of Metal Oxide Sorbents																								
Delineation of Effects of Hydrogen Partial Pressures and Moisture Amounts on Sulfidation of Metal Oxide Sorbents																								
Equilibrium Absorption of Hydrogen Sulfide into Metal Oxide Sorbents																								
Development of a Micro Differential Reactor																								

EXECUTIVE SUMMARY

The main objectives of this research project during this quarter are to formulate metal oxide sorbents using various ingredients as well as formulation conditions, and test reactivity of formulated metal oxide sorbents with hydrogen sulfide for 120 seconds at 550°C, and develop a formula for the preparation of a sorbent suitable for the removal of hydrogen sulfide from coal gases.

INTRODUCTION

Metal oxide sorbents were formulated with zinc oxide as an active sorbent ingredient, and titanium oxide as a supporting metal oxide. Various additives such as Cu, Co, Ni, Mn, Cr and Ca were utilized to enhance sulfur-removal capacity of formulated metal oxide sorbents. This mixture was extruded 1-mm cylindrical rods. The formulated metal oxide sorbents were calcined for 2 hours at 850 - 920°C. The fresh formulated metal oxide sorbents in the form of 1-mm cylindrical rod were crushed to obtain 100- 200 mesh particles, and were reacted with simulated coal gases containing hydrogen sulfide in the 35 cm³ 316 stainless steel batch reactor for 120 seconds at 550°C. Concentrations of hydrogen sulfide were analyzed with a gas chromatograph to evaluate reactivity of formulated metal oxide sorbents with H₂S.

RESULTS AND DISCUSSION

Research activities and efforts of this research project were concentrated on formulating various metal oxide sorbents with various additives under various formulation conditions, and conducting experiments on initial reactivity of formulated sorbents with hydrogen sulfide.

Experiments on reactivity of formulated metal oxide sorbents with hydrogen sulfide contained in a simulated coal gas mixture were carried out for 120 seconds at 550°C (see Table 1) to evaluate reactivity of formulated sorbents with hydrogen sulfide. A typical simulated coal gas mixture consists of 9107-ppm hydrogen sulfide (0.005 g; 1 wt %), 0.085-g water (15.84 wt %), 0.0029-g hydrogen (0.58 wt %), and 0.4046-g nitrogen (81.34 wt%).

Table 1. Experimental conditions for the reaction of hydrogen sulfide with formulated metal oxide sorbents in the presence of moisture, nitrogen and hydrogen.

Reactor Volume, cm ³ :	35
Temperature, °C:	550
Reaction Time, s:	120
Particle Size, mesh	100 - 200
Amount of Sorbent, g	0.05
Initial Partial Pressure of Hydrogen at 25°C, psia:	14.7
Initial Amount of Water, g:	0.085
Initial Concentration of H ₂ S, ppm	8700
Initial Partial Pressure of Nitrogen at 25°C, psia:	150
Initial Total Pressure of Reaction Mixture at 25°C, psia:	164.7

Metal oxide sorbents were formulated with various additives to enhance their reactivity with hydrogen sulfide contained in simulated coal gas mixtures (see Tables 2 through 7). Reactivity of formulated metal oxide sorbents was compared by reacting sorbents with initial 8700-ppm hydrogen sulfide for 120 seconds at 550°C (see Figures 1 and 2).

The metal oxide sorbents shown in Tables 4 through 7 were formulated with the cuprous oxide additive and the chromous oxide additive under various formulation conditions such as calcination temperatures and concentrations of H₂O₂ in water.

Table 2. Formulation of various TU sorbents.

Sorbents	TU-2	TU-3	TU-4	TU-5	TU-6	TU-7	TU-8	TU-9	TU-10	TU-11	TU-12	TU-13	TU-14
Amounts of Ingredients (gram) and Formulation Conditions													
ZnO	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
TiO ₂	2	2	2	2	2	2	2	2	2	2	2	2	2
Corn Starch						0.1	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Flour	0.3	0.3	0.3	0.3	0.1								
Calcium Carbonate						0.055	0.102	0.05	0.05	0.05	0.05	0.05	0.05
Bentonite	0.3	0.3	0.4	0.5	0.1	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Cobalt Oxide								0.016	0.017				
Molybdenum Oxide										0.017			
Nickel Oxide											0.016		
Cuprous Oxide												0.016	
Ferrous Oxide													0.015
Manganese Oxide													
0.5 w% H ₂ O ₂	2.5								1.50				
1 w% H ₂ O ₂		2	3										
2 w% H ₂ O ₂				2.26	2	2	2	1.51		1.50	1.50	1.50	1.50
Calcination Temperature, °C	800	800	800	800	900	900	900	900	900	900	900	900	900
Calcination Duration, hr	2	2	2	2	2	2	2	2	2	2	2	2	2
H ₂ S, ppm at 2 min	4435		5140	4760	5443	4604	5293	5785	5179	6129	4659	4648	4836
Reaction Duration	4580		4605	4654	5706	4950	5146	5494	5432	5435	4848	5040	5231
										4554		4770	

Table 3. Formulation of various TU sorbents.

Sorbents	TU-15	TU-16	TU-17	TU-18	TU-19	TU-20	TU-21	TU-22	TU-23	TU-25	TU-26	TU-27	TU-28
Amounts of Ingredients (gram) and Formulation Conditions													
ZnO	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.8	1.8	0.5	0.5	0.5	1.7
TiO ₂	2	2	2	2	2	2	2	1.2	1.2	2.5	2.5	2.5	2
Corn Starch	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	
Flour													0.5
Calcium Carbonate	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Bentonite	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.1
Cobalt Oxide				0.015		0.017			0.018			0.015	
Molybdenum Oxide							0.016					0.015	0.016
Nickel Oxide						0.016	0.016						
Cuprous Oxide			0.015	0.015				0.016	0.015		0.015	0.015	
Ferrous Oxide													
Manganese Oxide	0.016											0.015	
0.5 w% H ₂ O ₂													
1 w% H ₂ O ₂													
2 w% H ₂ O ₂	1.50	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Calcination Temperature, °C	900	900	900	900	850	850	850	850	850	900	900	900	870
Calcination Duration, hr	2	2	2	2	2	2	2	2	2	2	2	2	2
H ₂ S, ppm at 2 min	4684	4971	4134	4334	4767	4564	4644	2543	3690	6074	5727	6445	5680
Reaction Duration	5030	5010	4009	4688	4892	4851	4440	3628	3997	6382	6012	6404	5952
								4100		6675	6236	5573	

Table 4. Formulation of various TU sorbents.

Sorbents	TU-32	TU-33	TU-34	TU-35	TU-36	TU-37	TU-38	TU-39	TU-40	TU-41	TU-42	TU-43	TU-44	TU-44C
Amounts of Ingredients (gram) and Formulation Conditions														
ZnO	2	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
TiO ₂	1	2	2	2	2	2	2	1.8	2.2	2	2	2	2	2
Corn Starch	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.03	0.07	0.05	0.05	0.05
Calcium Carbonate	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.07	0.03	0.03
Bentonite	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.10	0.15
Cuprous Oxide	0.03	0.015	0.015	0.015	0.015	0.015	0.030	0.015	0.015	0.015	0.015	0.015	0.015	0.015
Chromous Oxide				0.015	0.015	0.030	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015
0 w% H ₂ O ₂														
0.5 w% H ₂ O ₂														
1 w% H ₂ O ₂														
2 w% H ₂ O ₂		1.5		1.5		1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
5 w% H ₂ O ₂	1.5		1.5		1.5									
Calcination Temperature, °C	900	900	900	900	900	900	900	900	900	900	900	900	900	900
Calcination Duration, hr	2	2	2	2	2	2	2	2	2	2	2	2	2	2
H ₂ S, ppm at 2 min	3755	4692	4409	3503	5159	4393	5387	3170	3106	4213	3536	5132	5503	4325
Reaction Duration	3813	4647	4269	3673	5158	4217	5582	3490	3333	4398	3659	5009	5235	4229
	3486	4690	4396	3591	3768	4346	5498	3489			3533	5041	4710	4208

Table 5. Formulation of various TU sorbents.

Sorbents	TU-45	TU-46	TU-46C	TU-47	TU-47C	TU-48	TU-49	TU-50	TU-51A	TU-51B	TU-51C
Amounts of Ingredients (gram) and Formulation Conditions											
ZnO	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
TiO ₂	2	2	2	2	2	2	2	2	2	2	2
Corn Starch	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Calcium Carbonate	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Bentonite	0.10	0.020	0.20	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Cuprous Oxide	0.015	0.015	0.015	0.015	0.010	0.020	0.015	0.015	0.015	0.015	0.015
Chromous Oxide	0.015	0.015	0.015	0.015	0.015	0.015	0.010	0.020	0.015	0.015	0.015
0 w% H ₂ O ₂											
0.5 w% H ₂ O ₂											
1 w% H ₂ O ₂									1.5		
2 w% H ₂ O ₂	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5		1.5	
5 w% H ₂ O ₂											1.5
Calcination Temperature, °C	900	900	900	900	900	900	900	900	900	900	900
Calcination Duration, hr	2	2	2	2	2	2	2	2	2	2	2
H ₂ S, ppm at 2 min	5688	4575	5107	3976	4834	5476	4811	5101	4024	4774	5212
Reaction Duration	5359	4591	4678	4286	4617	5493	4879	5253	4156	4879	5447
	5077	4474		4117		5708	4918	5232	4107	4880	5317

Table 6. Formulation of various TU sorbents.

Sorbents	TU-52A	TU-52B	TU-52C	TU-52D	TU-53A	TU-53B	TU-53C	TU-53D	TU-54A	TU-54B	TU-54C	TU-54D
Amounts of Ingredients (gram) and Formulation Conditions												
ZnO	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
TiO ₂	2	2	2	2	2	2	2	2	2	2	2	2
Corn Starch	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Calcium Carbonate	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Bentonite	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Cuprous Oxide	0.015	0.015	0.015	0.015	0.01	0.01	0.01	0.01	0.02	0.02	0.02	0.02
Chromous Oxide	0.015	0.015	0.015	0.015	0.01	0.01	0.01	0.01	0.02	0.02	0.02	0.02
0 w% H ₂ O ₂	1.5	1.5	1.5	1.5								
0.5 w% H ₂ O ₂												
1 w% H ₂ O ₂					1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
2 w% H ₂ O ₂												
5 w% H ₂ O ₂												
Calcination Temperature, °C	860	880	900	920	860	880	900	920	860	880	900	920
Calcination Duration, hr	2	2	2	2	2	2	2	2	2	2	2	2
H ₂ S, ppm at 2 min	5151	6120	5890	5662	4874	4697	4884	5108	5073	5224	5204	5348
Reaction Duration	5473	6023	6086	5839	4566	4827	4927	4761	5239	5081	5059	5238
	5463	5903	5965	5779	4623	4827	4612	4933	5284	5014	5224	5266

Table 7. Formulation of various TU sorbents.

Sorbents	TU-55A	TU-55B	TU-55C	TU-56A	TU-56B	ZT-4
Amounts of Ingredients (gram) and Formulation Conditions						
ZnO	1.7	1.7	1.7	1.7	1.7	
TiO ₂	2	2	2	3	3	
Corn Starch	0.05	0.05	0.05	0.05	0.05	
Calcium Carbonate	0.05	0.05	0.05	0.05	0.05	
Bentonite	0.15	0.15	0.15	0.15	0.15	
Cuprous Oxide	0.015	0.015	0.015	0.015	0.015	
Chromous Oxide	0.015	0.015	0.015	0.015	0.015	
0 w% H ₂ O ₂						
0.5 w% H ₂ O ₂	1.5	1.5	1.5			
1 w% H ₂ O ₂				1.5	1.5	
2 w% H ₂ O ₂						
5 w% H ₂ O ₂						
Calcination Temperature, °C	900	860	920	860	900	
Calcination Duration, hr	2	2	2	2	2	
H ₂ S, ppm at 2 min	4342	5188	5808	4186	4600	4325
Reaction Duration	4362	5065	6142	4131	4501	4362
	4340	5427	5718	4187	4450	4340

FIGURE 1. Reactivity of various sorbents reacted with initial 8700-ppm dry H_2S for 120 s at 550°C.

FIGURE 2. Reactivity of various 0.05-g sorbents with initial 8700-ppm dry H_2S for 120 s at 550°C.

The metal oxide sorbents calcined for 2 hours at 900°C were formulated with various additives to identify their effects on reactivity of the sorbents (see Figure 3). The cuprous oxide additive, and the mixture consisting of the cuprous oxide additive and the chromous oxide additive appear to be most effective among other additives in enhancing reactivity of the sorbents.

A series of metal oxide sorbents such as TU-35, TU-39, and TU-40 (see Table 4) were formulated with various amounts of TiO_2 , holding the amounts of the other ingredients and the formulation conditions constant. Reactivity of fomulated sorbents appears to be independent of TiO_2 amounts (see Figure 4).

A series of metal oxide sorbents such as TU-35, TU-41, and TU-42 (see Table 4) were formulated with various amounts of corn starch, holding the amounts of the other ingredients and the formulation conditions constant. Reactivity of fomulated sorbents increases with increased amount of corn starch (see Figure 5).

A series of metal oxide sorbents such as TU-43, TU-44C, and TU-47 (see Tables 4 and 5) were formulated with various amounts of calcium carbonate, holding the amounts of the other ingredients and the formulation conditions constant. Reactivity of formulated sorbents decreases with increased amount of calcium carbonate (see Figure 6).

A series of metal oxide sorbents such as TU-45, TU-46, TU-46C and TU-47 (see Table 5) were formulated with various amounts of bentonite, holding the amounts of the other ingredients and the formulation conditions constant. Reactivity of formulated sorbents appears not to be affected by the amount of bentonite (see Figure 7).

A series of metal oxide sorbents such as TU-47, TU-47C, and TU-48 (see Table 5) were formulated with various amounts of cuprous oxide, holding the amounts of the other ingredients and the formulation conditions constant. Reactivity of formulated sorbents decreases with increased amount of the cuprous oxide additive (see Figure 8).

A series of metal oxide sorbents such as TU-37, TU-47, TU-49 and TU-50 (see Tables 4 and 5) were formulated with various amounts of chromous oxide, holding the amounts of the other ingredients and the formulation conditions constant. Reactivity of formulated sorbents appears not to be affected by the amount of the additive chromous oxide (see Figure 9).

A series of metal oxide sorbents such as TU-53A, TU-53B, TU-53C and TU-53D (see Table 6) were formulated in the presence of 1-w% H_2O_2 with 0.01-g cuprous oxide and 0.01-g chromous oxide, and calcined at various temperatures. Another series of metal oxide sorbents such as TU-54A, TU-54B, TU-54C and TU-55D (see Table 6) were formulated in the presence of 1-w% H_2O_2 with 0.02-g cuprous oxide and 0.02-g chromous oxide, and calcined at various temperatures. The reactivity of the sorbents formulated with 0.01-g cuprous oxide and 0.01-g chromous oxide is higher than that of those formulated with 0.02-g cuprous oxide and 0.02-g chromous oxide. The reactivity of the sorbents with H_2S decreases with increased calcination temperature (see Figure 12).

A series of metal oxide sorbents such as TU-52A, TU-52B, TU-52C and TU-52D (see Table 6) were formulated in the absence of H_2O_2 , and calcined at various temperatures. Another series of metal oxide sorbents such as TU-55A, TU-55B, and TU-55C (see Table 7) were formulated in the presence of 0.5-w% H_2O_2 , and calcined

at various temperatures. The reactivity of the sorbents formulated in the presence of 0.5-w% H_2O_2 is higher than that of those formulated in the absence of H_2O_2 . The reactivity of the sorbents with H_2S decreases with increased calcination temperature (see Figure 13).

The metal oxide sorbents such as TU-52A, TU-53A, and TU-55A were calcined at 860°C. The metal oxide sorbents such as TU-52C, TU-53C, and TU-55B were calcined at 900°C. The metal oxide sorbents such as TU-52D, TU-53D, and TU-55C were calcined at 920°C. The reactivity of the metal oxide sorbents increases with increased concentration of H_2O_2 as a formulation additive. The reactivity of the metal oxide sorbents increases with decreased calcination temperature (see Figure 14).

CONCLUSION

A promising formula for the preparation of a reactive sorbent was identified using various additives under various formulation conditions. A simple procedure was developed in evaluating reactivity of a sorbent formulated with various additives under a variety of formulation conditions

PRESENTATION

A research paper, entitled "Reactivity of Metal Oxide Sorbents", was presented at the Advanced Coal-Fired Power Systems '96 Review Meeting, Morgantown Energy Technology Center, West Virginia, July 16-18, 1996. A research paper, entitled "Reactivity of Metal Oxide Sorbents for Removal of Wet Hydrogen Sulfide", was accepted for presentation at the 1996 Annual AIChE Meeting, Chicago, November 10 - 15, 1996. A research paper, entitled "Formulation of Metal Oxide Sorbents for Removal of Wet Hydrogen Sulfide", was submitted for presentation at the 1997 Spring National AIChE Meeting, Houston, March 9 - 13, 1997.

PLANS ON FUTURE EXPERIMENTS

Various metal oxide sorbents will be formulated with various additives under various formulation conditions in order to search for a formula of a durable sorbent with high-sulfur-absorbing capacity.