

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

Technical Progress Report January 1 - March 31, 1997

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

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

By
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K. C. Kwon

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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 durable metal oxide sorbents with high-sulfur absorbing capacities, 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 of a sorbent suitable for the removal of hydrogen sulfide from hot coal gases.

INTRODUCTION

Metal oxide sorbents were formulated with zinc oxide as an active sorbent ingredient, and titanium dioxide as a supporting metal oxide. Various metal oxide additives such as Ce, Zr, Cu, Co, Ca and Cr 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 1 hour at 860°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. Hardness of formulated sorbents was tested with a pellet hardness tester. 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, conducting experiments on initial reactivity of formulated sorbents with hydrogen sulfide, and testing hardness of formulated sorbents.

Experiments on reactivity of formulated metal oxide sorbents with wet 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. Hardness of formulated sorbents was evaluated in addition to testing their reactivity 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 5). 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 through 8). Hardness of formulated metal oxide sorbents was compared by testing their hardness with a hardness tester (see Figures 1 through 8). Hardness of a fresh formulated sorbent is tested 7 times. The average value of seven hardness data is reported in the Tables. The hardness range of fresh formulated sorbents at room temperature is 3 -16 kg.

The metal oxide sorbents TU-97 through TU-105, as shown in Tables 2, were formulated without zirconium oxide, whereas the metal oxide sorbents TU-106 through TU-144, as shown in Tables 2 through 5, were formulated with zirconium oxide. Hardness of fresh formulated sorbents increases significantly in the presence of zirconium oxide.

Table 2. Formulation of various TU sorbents.

Sorbents	TU-97	TU-98	TU-99	TU-100	TU-101	TU-102	TU-103	TU-104	TU-105	TU-106	TU-107	TU-108
Ingredient Amounts (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
Zirconium Oxide (Zr)										0.05	0.05	0.05
Gypsum	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Cobalt Oxide (Co)	0.005	.01	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.005	0.010	0.015
Cuprous Oxide (Cu)	0.015	0.015	0.005	0.010	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.005
Cerium Oxide (Ce)	0.015	0.015	0.015	0.015	0.015	0.015	0.005	0.010	0.015	0.015	0.015	0.015
Chromous Oxide (Cr)	0.015	0.015	0.015	0.015	0.005	0.010	0.015	0.015	0.015	0.015	0.015	0.015
1 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	1.5
Calculation Temperature, °C	860	860	860	860	860	860	860	860	860	860	860	860
Calcination Duration, hr	1	1	1	1	1	1	1	1	1	1	1	1
H ₂ S, ppm at 2-min reaction	3998	4092	4475	4728	4163	4730	4536	5016	4410	4739	4314	4946
	3934	4208	4421	4652	4048	4372	4648	5050	4503	4676	4417	5109
	4112	3998	4405	3217	5216	4288	4519	4868	4338	4418	4401	5194
	3801	4471		3908	4616	4340	4501	4758	4311	4855	4254	5123
		4540		4679		4413	4319	4982	4435	4775	4514	4827
Hardness, kg	4.87	4.03	4.85	5.70	4.67	7.93	6.90	3.27	3.75	5.47	4	5.67

Table 3. Formulation of various TU sorbents.

Sorbents	TU-109	TU-110	TU-111	TU-112	TU-113	TU-114	TU-115	TU-116	TU-117	TU-118	TU-119	TU-120
Ingredient Amounts (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
Zirconium Oxide (Zr)	0.05	0.10	0.15	0.150	0.150	0.150	0.200	0.200	0.200	0.200	0.200	0.200
Gypsum	0.15	0.150	0.150	0.050	0.10	0.0	0.0	0.050	0.100	0.15	0.200	0.250
Cobalt Oxide (Co)	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015
Cuprous Oxide (Cu)	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015
Cerium Oxide (Ce)	0.015	0.015	0.015	0.015	0.015	0.010	0.015	0.015	0.015	0.015	0.015	0.015
Chromous Oxide (Cr)	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015
1 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	1.5
Calcination Temperature, °C	860	860	860	860	860	860	860	860	860	860	860	860
Calcination Duration, hr	1	1	1	1	1	1	1	1	1	1	1	1
H ₂ S, ppm at 2-min reaction	4210	3488	5224	5423	4548	4427	4694	5232	4820	4623	4378	4210
	4257	3613	5003	5431	5075	4466	4469	5064	4485	4767	4267	4231
	4172	3565	5167	5515	4771	4285	4724	5051	4843	4940	4517	4094
	4141	3543	4753	5388	4661	4122	4699	4890	4757	4159	4290	4162
	4276	3600	4830	5432		4705		5093	4570	4996	4307	4307
Hardness, kg	3.5	4.67	10.75	11.2	11.83	9.83	5.75	9.50	11.5	10.88	8.75	8.88

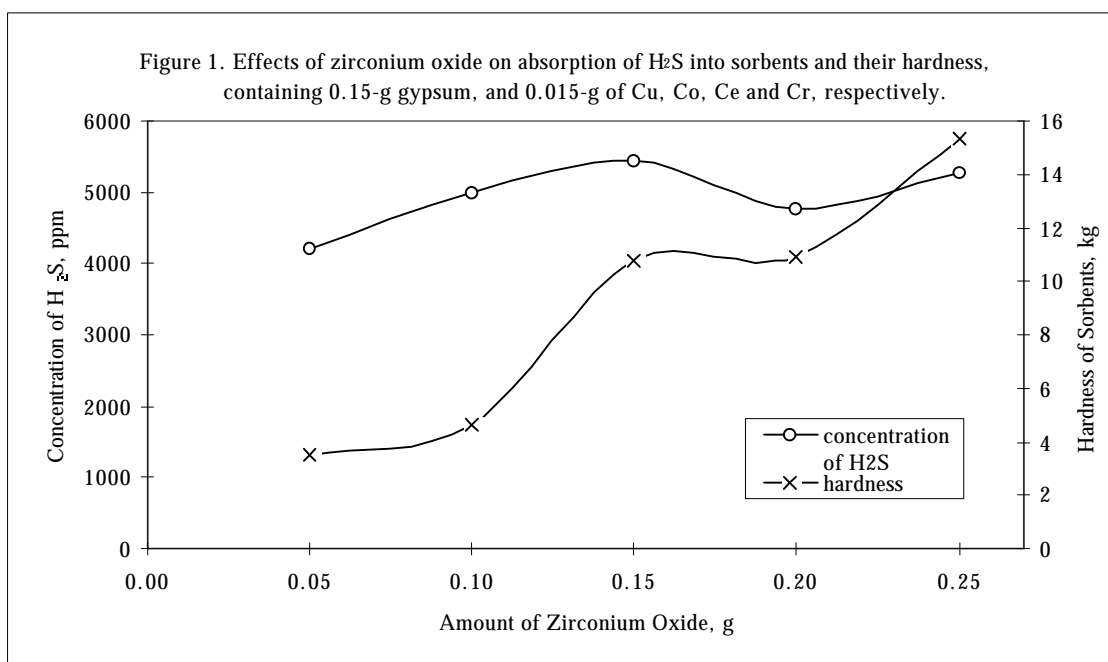
Table 4. Formulation of various TU sorbents.

Sorbents	TU-121	TU-122	TU-123	TU-124	TU-125	TU-126	TU-127	TU-128	TU-129	TU-130	TU-131	TU-132
Ingredient Amounts (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	2
TiO2	2	2	2	2	2	2	2	2	2	2	2	1.7
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
Zirconium Oxide (Zr)	0.250	0.250	0.250	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.200
Gypsum	0.05	0.100	0.150	0.10	0.10	0.10	0.10	0.10	0.100	0.100	0.100	0.100
Cobalt Oxide (Co)	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.005	0.01	0.015	0.015	0.015
Cuprous Oxide (Cu)	0.015	0.015	0.015	0.015	0.015	0.005	0.01	0.015	0.015	0.005	0.01	0.015
Cerium Oxide (Ce)	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015
Chromous Oxide (Cr)	0.015	0.015	0.015	0.005	0.01	0.015	0.015	0.015	0.015	0.015	0.015	0.005
1 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	1.5
Calculation Temperature, °C	860	860	860	860	860	860	860	860	860	860	860	860
Calcination Duration, hr	1	1	1	1	1	1	1	1	1	1	1	1
H ₂ S, ppm at 2-min reaction												
	4864	5144	5101	4334	4741	4905	5649	4783	4615	5318	5035	5064
	4866	4942	5083	4446	5042	4834	5927	4800	5032	5183	4775	4834
	4816	4991	5325	4604	4949	4733	5906	4916	4926	5177	4759	4601
	4753	5024	5417	4440	4556	5011	5607	4830	5030	5090	4953	5181
	4830	6143	5419	4513	4882	4859	5274	4712	4748	5164	4770	4953
Hardness, kg	11.7	11.13	10.38	11.82	12.36	12.04	11.30	12.38	11.70	11.67	10.38	11.17
	10.83	13.57	15.31									

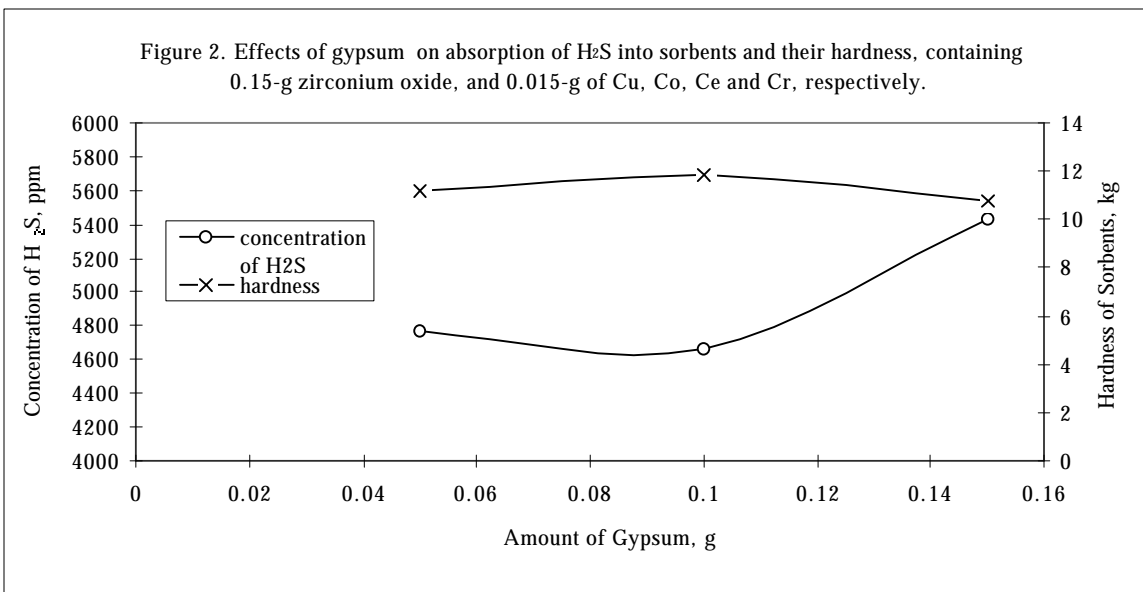
Table 5. Formulation of various TU sorbents.

Sorbents	TU-133	TU-134	TU-135	TU-136	TU-137	TU-138	TU-139	TU-140	TU-141	TU-142	TU-143	TU-144
Ingredient Amounts (gram) and Formulation Conditions												
ZnO	2	2	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
TiO2	1.7	1.7	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
Zirconium Oxide (Zr)	0.200	0.200	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.25
Gypsum	0.100	0.100	0.100	0.100	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.20
Cobalt Oxide (Co)	0.015	0.015	0.015	0.015	0.020	0.015	0.015	0.015	0.015	0.015	0.015	0.015
Cuprous Oxide (Cu)	0.015	0.015	0.015	0.015	0.015	0.020	0.015	0.015	0.025	0.030	0.035	0.015
Cerium Oxide (Ce)	0.015	0.005	0.01	0.02	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015
Chromous Oxide (Cr)	0.01	0.015	0.015	0.015	0.015	0.015	0.020	0.015	0.015	0.015	0.015	0.015
1 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	1.5
Calculation Temperature, °C	860	860	860	860	860	860	860	860	860	860	860	860
Calcination Duration, hr	1	1	1	1	1	1	1	1	1	1	1	1
H ₂ S, ppm at 2-min reaction												
	5035	4860	4926	4912	4597	4209	4966	4322	5087	4920	4933	5152
	5068	5355	4976	5021	4776	4333	5008	3458	4802	4936	4874	5143
	5278	4581	4768	4993	5054	3972	4767	4443	5065	4996	4805	4848
	4986	5241	4806	4606	5034	4216	4848	4105	4850	4790	4713	5038
	4856	4294	4747	4851	4561	4045	4560	4032	5020	4979	4951	4881
Hardness, kg	11.00	13.14	11.92	12.29	11.42	12.25	10.25	12.20	10.57	11.00	12.07	10.36

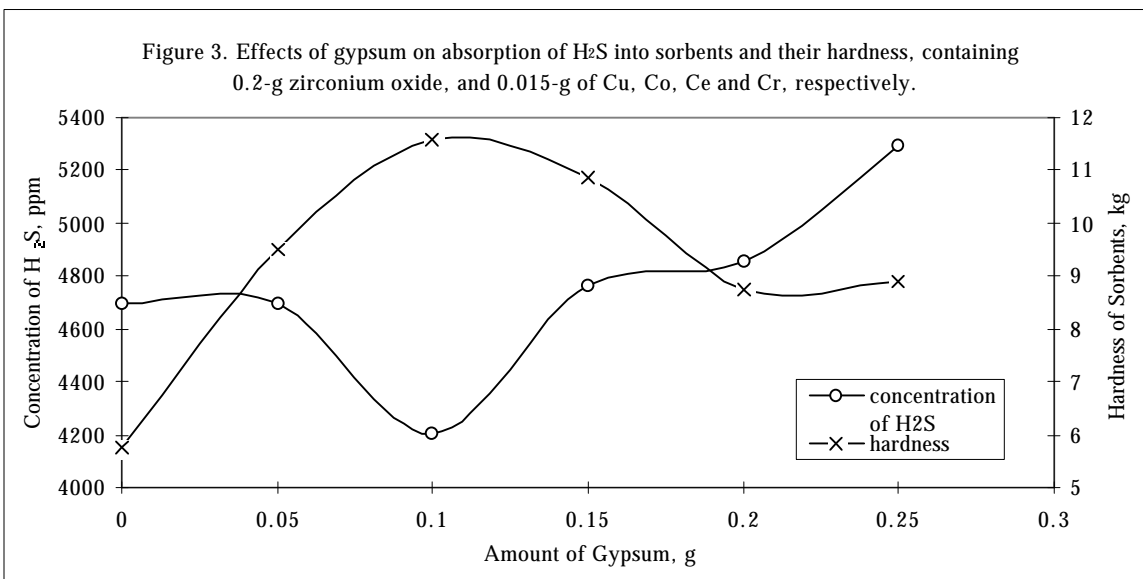
The sorbents TU-109, TU-110, TU-111, TU-118 and TU-123 were formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.15-g gypsum, and 0.015 g of cuprous oxide, cobalt oxide, cerium oxide, and chromous oxide, respectively, varying amounts of zirconium oxide. The sorbents TU-109, TU-110, TU-111, TU-118 and TU-123 were formulated with zirconium oxide of 0.05 g, 0.1 g, 0.15 g, 0.2 g and 0.25 g, respectively. The TU-109, TU-110, TU-111, TU-118 and TU-123 were reacted with the initial 8700-ppm H₂S for 120 seconds at 550°C. Reactivity of the sorbents with H₂S decreases with increased amounts of zirconium oxide, while hardness of the sorbents increases with amounts of zirconium oxide (see Figure 1). Hardness of the sorbents increases from 3.5 kg to 15.31 kg by varying the amounts of zirconium oxide in the formulation of the sorbents from 0.05 g to 0.25 g. These observations indicate that zirconium oxide in the formulation of the sorbents affect significantly their hardness.



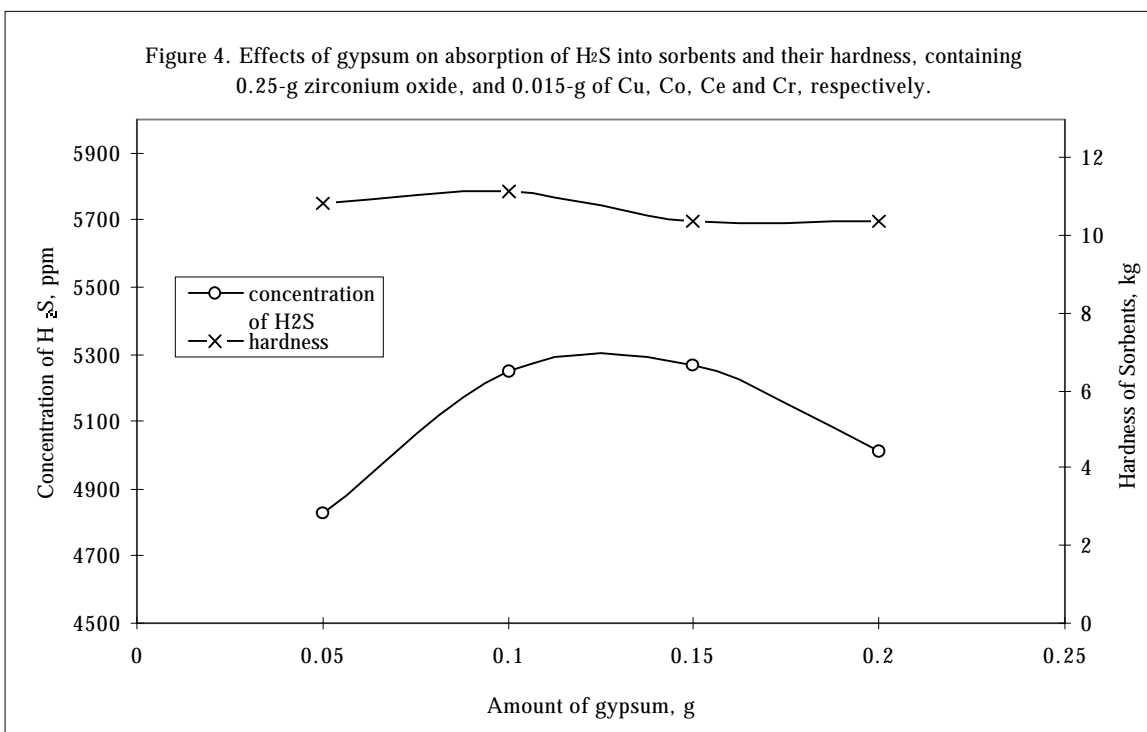
The sorbents TU-112, TU-113, and TU-111 were formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.15-g zirconium oxide, and 0.015 g of cuprous oxide, cobalt oxide, cerium oxide, and chromous oxide, respectively, varying amounts of gypsum. The sorbents TU-112, TU-113, and TU-111 were formulated with gypsum of 0.05 g, 0.1 g, and 0.15 g, respectively. The sorbents TU-112, TU-113, and TU-111 were reacted with the initial 8700-ppm H₂S for 120 seconds at 550°C. Reactivity of the sorbents with H₂S decreases with increased amounts of gypsum, while hardness of the sorbents is independent of amounts of gypsum (see Figure 2). These data show that amounts of gypsum do not affect hardness of the formulated sorbents in the presence of 0.15-g zirconium oxide, but affect their reactivity with hydrogen sulfide.



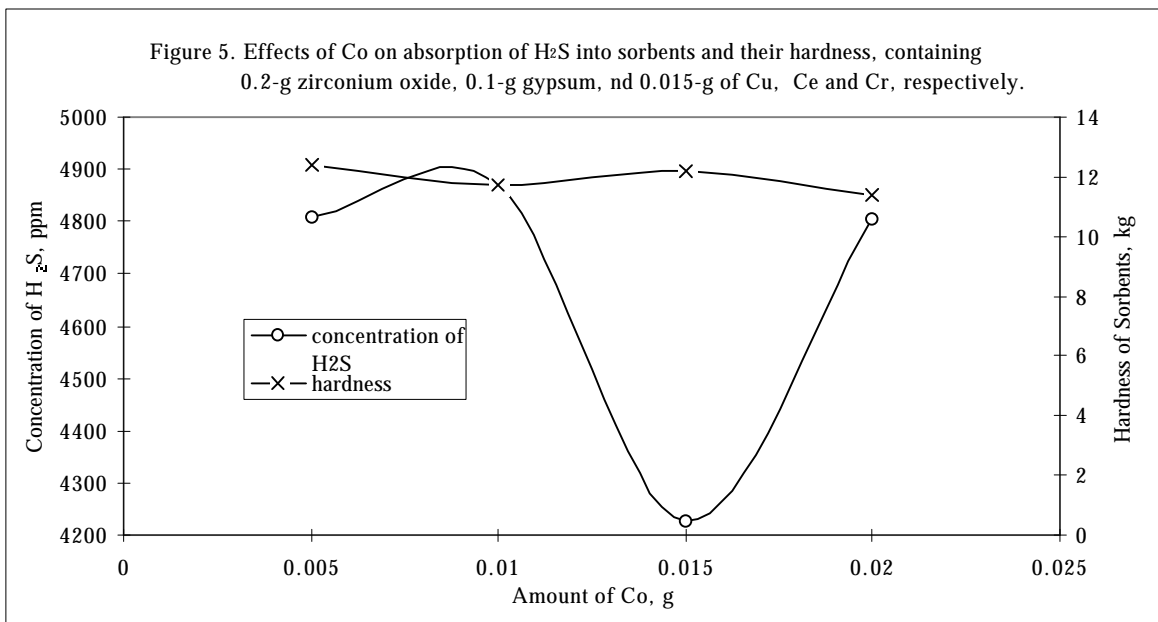
The sorbents TU-115, TU-116, TU-117, TU-118, TU-119 and TU-120 were formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.2-g zirconium oxide, and 0.015 g of cuprous oxide, cobalt oxide, cerium oxide, and chromous oxide, respectively, varying amounts of gypsum. The sorbents TU-115, TU-116, TU-117, TU-118, TU-119 and TU-120 were formulated with gypsum of 0.05 g, 0.1 g, 0.15 g, 0.2 g, and 0.25 g, respectively. The sorbents TU-115, TU-116, TU-117, TU-118, TU-119 and TU-120 were reacted with the initial 8700-ppm H₂S for 120 seconds at 550°C. Reactivity of the sorbent, containing 0.1-g gypsum, with H₂S is highest among others, while hardness of the sorbent, containing 0.1-g gypsum, is highest among others (see Figure 3). These data show that amounts of gypsum affect both hardness of the formulated sorbents and their reactivity with hydrogen sulfide in the presence of 0.2-g zirconium oxide.



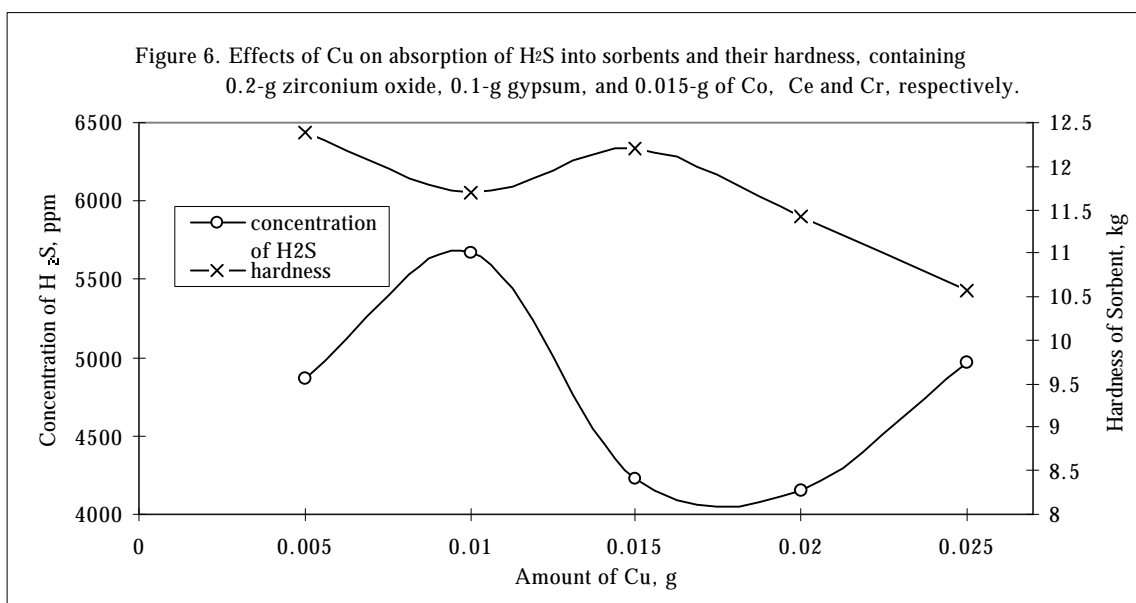
The sorbents TU-121, TU-122, TU-123, and TU-144 were formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.25-g zirconium oxide, and 0.015 g of cuprous oxide, cobalt oxide, cerium oxide, and chromous oxide, respectively, varying amounts of gypsum. The sorbents TU-121, TU-122, TU-123, and TU-144 were formulated with gypsum of 0.05 g, 0.1 g, 0.15 g, and 0.2 g, respectively. The sorbents TU-121, TU-122, TU-123, and TU-144 were reacted with the initial 8700-ppm H₂S for 120 seconds at 550°C. Reactivity of the sorbent, containing 0.1-g gypsum, with H₂S is lowest among others, while hardness of the sorbents decreases slightly with amounts of gypsum (see Figure 4). These data show that amounts of gypsum do not affect significantly hardness of the formulated sorbents in the presence of 0.25-g zirconium oxide, but affect their reactivity with hydrogen sulfide.



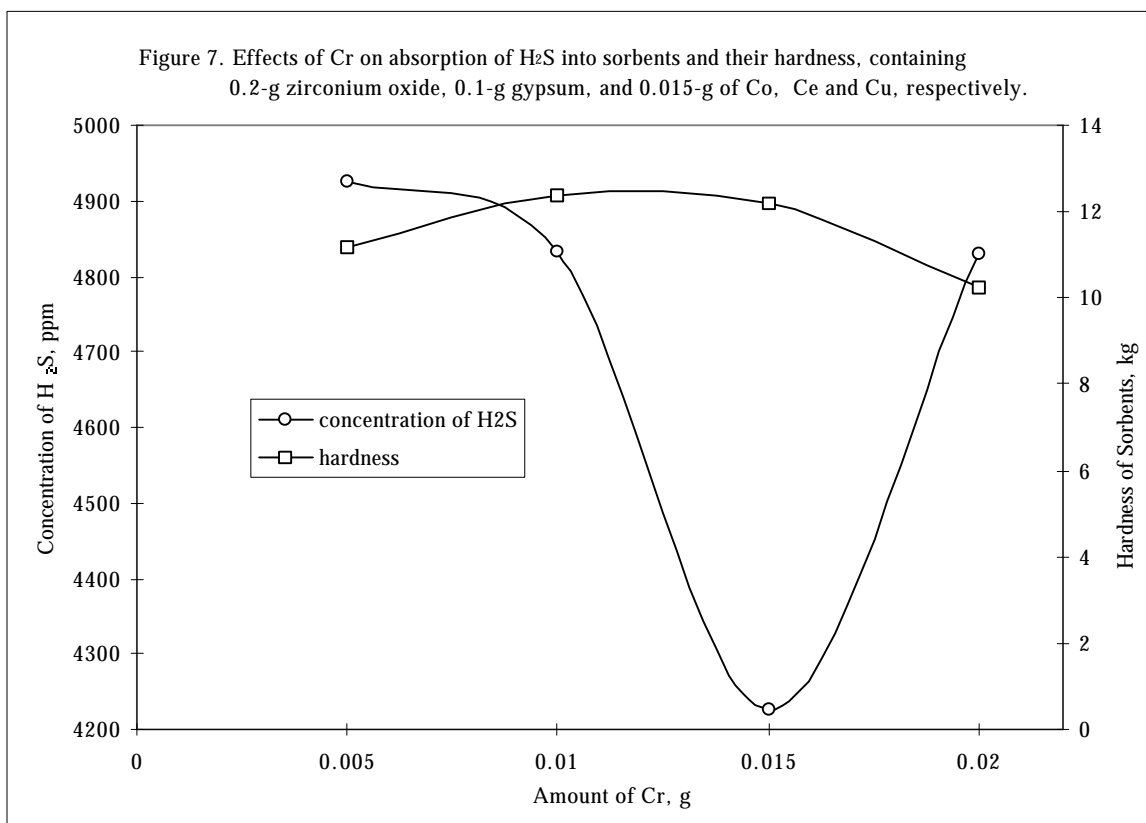
The sorbents TU-128, TU-129, TU-140, and TU-137 were formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.2-g zirconium oxide, 0.1-g gypsum, and 0.015 g of cuprous oxide, cerium oxide, and chromous oxide, respectively, varying amounts of cobalt oxide. The sorbents TU-128, TU-129, TU-140, and TU-137 were formulated with cobalt oxide of 0.05 g, 0.1 g, 0.15 g, and 0.2 g, respectively. The sorbents TU-128, TU-129, TU-140, and TU-137 were reacted with the initial 8700-ppm H₂S for 120 seconds at 550°C. Reactivity of the sorbent, containing 0.015-g cobalt oxide, with H₂S is highest among others, while hardness of the sorbents decreases slightly with amounts of cobalt oxide (see Figure 5). These data show that amounts of cobalt oxide do not affect significantly hardness of the formulated sorbents, but affect their reactivity with hydrogen sulfide.



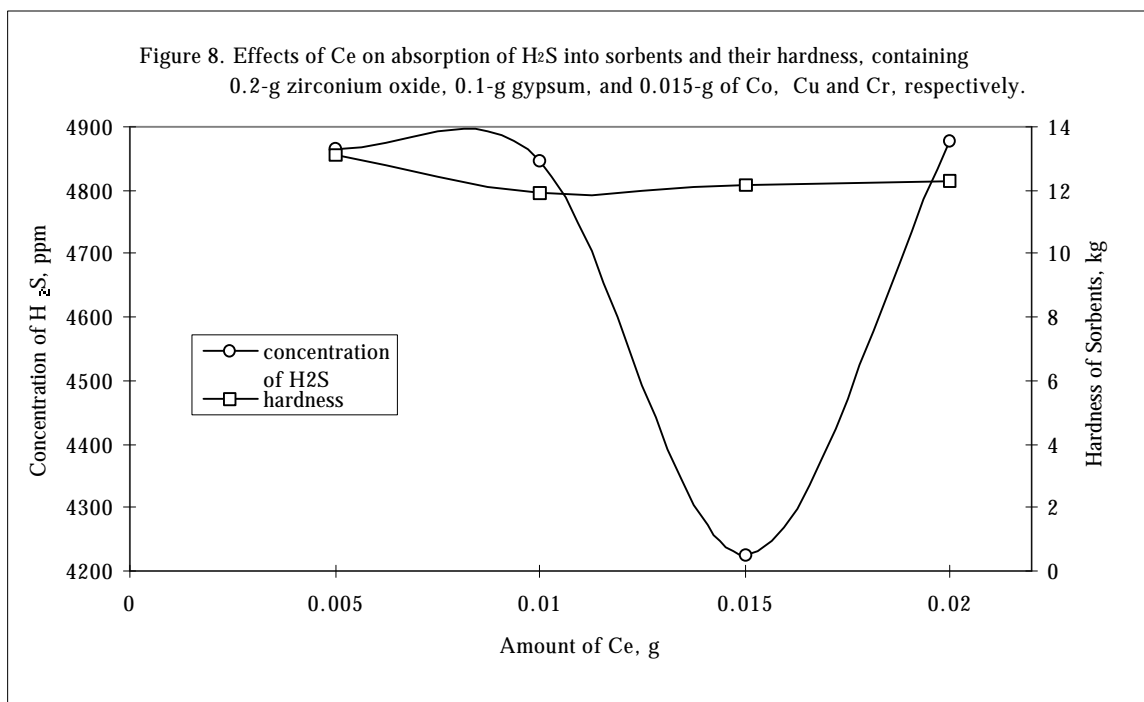
The sorbents TU-126, TU-127, TU-140, TU-139 and TU-141 were formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.2-g zirconium oxide, 0.1-g gypsum, and 0.015 g of cobalt oxide, cerium oxide, and chromous oxide, respectively, varying amounts of cuprous oxide. The sorbents TU-126, TU-127, TU-140, TU-139 and TU-141 were formulated with cuprous oxide of 0.005 g, 0.01 g, 0.015 g, 0.02 g and 0.25 g, respectively. The sorbents TU-126, TU-127, TU-140, TU-139 and TU-141 were reacted with the initial 8700-ppm H₂S for 120 seconds at 550°C. Reactivity of the sorbent, containing 0.02-g cuprous oxide, with H₂S is highest among others, while hardness of the sorbents decreases with amounts of cuprous oxide (see Figure 6). These data show that amounts of cuprous oxide affect significantly both hardness of the formulated sorbents and their reactivity with hydrogen sulfide.



The sorbents TU-132, TU-125, TU-140, and TU-139 were formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.2-g zirconium oxide, 0.1-g gypsum, and 0.015 g of cobalt oxide, cerium oxide, and cuprous oxide, respectively, varying amounts of chromous oxide. The sorbents TU-132, TU-125, TU-140, and TU-139 were formulated with chromous oxide of 0.005 g, 0.01 g, 0.015 g, and 0.02 g, respectively. The sorbents TU-132, TU-125, TU-140, and TU-139 were reacted with the initial 8700-ppm H₂S for 120 seconds at 550°C. Reactivity of the sorbent, containing 0.015-g chromous oxide, with H₂S is highest among others, while hardness of the sorbents is independent of amounts of chromous oxide (see Figure 7). These data show that amounts of chromous oxide do not affect significantly hardness of the formulated sorbents, but affect their reactivity with hydrogen sulfide.



The sorbents TU-134, TU-135, TU-140, and TU-136 were formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.2-g zirconium oxide, 0.1-g gypsum, and 0.015 g of cobalt oxide, cuprous oxide, and chromous oxide, respectively, varying amounts of cerium oxide. The sorbents TU-134, TU-135, TU-140, and TU-136 were formulated with cerium oxide of 0.005 g, 0.01 g, 0.015 g, and 0.02 g, respectively. The sorbents TU-134, TU-135, TU-140, and TU-136 were reacted with the initial 8700-ppm H₂S for 120 seconds at 550°C. Reactivity of the sorbent, containing 0.015-g cerium oxide, with H₂S is highest among others, while hardness of the sorbents is independent of amounts of cerium oxide (see Figure 8). These data show that amounts of cerium oxide do not affect significantly hardness of the formulated sorbents, but affect their reactivity with hydrogen sulfide.



CONCLUSION

The sorbent TU-138 and the sorbent TU-140 appear to be most promising sorbents among others, considering their reactivity as well as hardness (see Table 5). The sorbent TU-138 was formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.2-g zirconium oxide, 0.1-g gypsum, 0.02-g cuprous oxide, and 0.015 g of cobalt oxide, cerium oxide and chromous oxide, respectively. The sorbent TU-140 was formulated with 1.7-g zinc oxide, 2-g titanium dioxide, 0.05-g corn starch, 0.2-g zirconium oxide, 0.1-g gypsum, and 0.015 g of cobalt oxide, cuprous oxide, cerium oxide, and chromous oxide, respectively.

PRESENTATION

A research paper, entitled "Formulation of Metal Oxide Sorbents for Removal of Wet Hydrogen Sulfide", was presented at the 1997 Spring National AIChE Meeting, Houston, March 9 - 13, 1997.

A research paper, entitled "Reactivity of Metal Oxide Sorbents for Removal of Wet Hydrogen Sulfide at High Temperatures", was published in the Proceedings of the Fifth Annual HBCU/Private Sector-Energy Research and Development Technology Transfer Symposium, Baton Rouge, Louisiana, March 4-5, 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.