

TECHNICAL REPORT

December 1, 1994 through February 28, 1995

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Project Title: **COMPOSITION MODIFICATION OF ZINC TITANATE
SORBENTS FOR HOT GAS DESULFURIZATION**

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 3)
ICCI Project Number: 94-1/2.2A-2M
Principal Investigator: James H. Swisher, Southern Illinois University
at Carbondale
Co-Principal Investigator: Ranajit K. Datta, Virginia Polytechnic Institute
and State University
Project Manager: Franklin I. Honea, Illinois Clean Coal Institute

ABSTRACT

For new coal gasification systems, zinc titanate sorbents are being developed to remove sulfur from the hot product gas prior to its use in combined cycle turbines and high temperature fuel cells. Although most of the properties of these sorbents are very attractive, there are still concerns about durability over many sulfidation-regeneration cycles and zinc losses due to vaporization. Doping the zinc titanate with other metal ions could alleviate both concerns, which are the objectives of this project. A screening study was completed during the second quarter in which Ni, Cr, Cu, Mg, and Al were evaluated as dopants in zinc titanate. Measurements that were made include solubility, crush strength, and sulfidation-regeneration behavior in a thermogravimetric analyzer. A formulation containing Cr showed the most promise. It and other formulations containing Cr will be emphasized during the remainder of the year. Fixed bed experiments will start during the third quarter.

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EXECUTIVE SUMMARY

Sulfur sorbents which contain zinc as the primary desulfurizing agent continue to show promise for coal gasification applications. Scale-up tests are now in progress for Integrated Gasification Combined Cycle (IGCC) systems. There are still concerns, however with sorbent durability over many sulfidation-regeneration cycles and with vaporization losses of zinc. Therefore research and development directed at these potential problems is continuing. Zinc titanate sorbents are among the few that have reached the stage of large scale testing.

An approach that has received little attention to date for improving the properties of zinc titanate is to dope the material with other metal ions. In principle, it should be possible to both strengthen the material and reduce zinc vaporization losses in this way. Based on ceramic crystal chemistry principles, it was decided to study the possible beneficial effects of Ni, Cr, Cu, Mg, and Al as dopants in zinc titanate. A set of screening experiments was completed during the second quarter. At Virginia Tech (VPI) the solubilities of the dopants were measured at 1000° and 1100° C, using x-ray techniques. All of the dopants were found to have a high solubility, with the possible exception of Cu. (An experimental problem occurred with some of the Cu experiments.)

At Southern Illinois University at Carbondale (SIUC), the same systems were studied through measurements of crush strength, bulk density, porosity, and reactivity in a thermogravimetric analyzer (TGA). Formulations containing Ni and Cu had the highest crush strengths, but they were chemically unstable in reducing gases. The best combination of properties was obtained with Cr as the dopant. It has a high solubility, and pellets made with a Zn/Cr ratio of 4 had a high sulfur capacity, were chemically stable in reducing gases, and had good reactivity in TGA experiments. Their crush strengths were not among the highest, but this property can probably be improved by varying the preparation procedures.

It is recommended that emphasis during the last half of the contract period be placed on Cr-containing sorbents. Three or more formulations will be prepared and evaluated. The variables in sorbent preparation will be chemical composition, sintering temperature, and initial oxide particle size. Fixed bed experiments on large batches of these formulations will start during the third quarter.

OBJECTIVES

The general objective of the project is to improve the engineering properties of zinc titanate sorbents for the removal of H_2S from coal gasification product gases. These improvements will be made by systematically adding dopants, such as Ni, Cu, Cr, Mg, and Al, to zinc titanate formulations. The main tasks designed to meet the objectives are sorbent preparation, solubility measurements, and bench-scale testing of sorbent pellets. Expected results include both engineering property characterization and an understanding of the crystal chemistry of the doped sorbents.

INTRODUCTION AND BACKGROUND

All modern coal combustion and coal conversion processes must have provisions to meet strict specifications on emissions of H_2S and SO_2 . For many systems, removal of sulfur-bearing species from hot gases is also needed to prevent corrosive damage to materials and components. Both requirements apply to Integrated Gasification Combined Cycle (IGCC) and Molten Carbonate Fuel Cell (MCFC) systems. An important milestone in the development of IGCC systems was successful completion of the Cool Water Coal Gasification Program (Douglas, 1990). In this program, the fuel gas was cooled down for H_2S removal, then reheated before injection into the turbine combustor. To avoid the associated efficiency loss with cooling of the fuel gas, most of the active development projects now include hot gas desulfurization with regenerable sorbents.

Many metals, oxides, and other compounds have been evaluated for their ability to remove H_2S from hot, coal-derived gases. In recent years, one of the classes of compounds that has been studied extensively and continues to show promise is zinc titanate (Swisher, O'Brien, and Gupta, 1994; Gupta, Gangwal and Jain, 1993; Gupta and Gangwal, 1993; Datta, 1994; Mei, et al., 1993; Ayala, et al., 1993; and Grindley, 1990). The status of the technology has matured to the point where large scale testing in process development units and pilot plants is in progress (Bevan, 1994; Kontinen, et al., 1994).

There are still a few concerns with zinc titanate sorbents which are being investigated in current projects. The most important of these is durability over many sulfidation cycles in fixed and fluidized bed reactors. There is a lesser concern with vaporization losses of zinc. In this regard, current sorbent formulations are adequate unless the sulfidation reaction is carried out above a temperature of approximately $600^\circ C$.

In the project described here, the research is directed toward both problem areas. By the incorporation of a third metal ion into the zinc titanate crystal structure, the thermodynamic activity and vapor pressure of zinc can be reduced. Also, the dopant may improve the strength and durability of the sorbents. Scanty information has been found in the literature on this subject, so the present work is pioneering in nature.

During the past quarter, screening experiments were completed on five dopants added to zinc titanate. Different but complementary approaches were taken at SIUC and VPI in this phase of the project. At SIUC, a standard dopant concentration was selected, and measurements were made on crush strength, bulk density, and reactivity in a thermogravimetric analyzer (TGA). At VPI, x-ray methods were used to study the solubility of the dopants in zinc titanate.

EXPERIMENTAL PROCEDURES

In the experiments done at SIUC, cylindrical pellets were prepared using the same procedures as in prior ICCI projects. Reagent-grade oxide powders were blended in a dilute solution of starch in water. Excess water was removed until the material had a consistency like toothpaste, then it was extruded through a 1 cm dia. metal tube and cut to lengths of approximately 1.5 cm. After drying further, the green pellets were sintered for three hours at 900° C.

The weights of the oxide powders were calculated to be consistent with final compositions corresponding to $Zn_{1-x}M_xTiO_3$, (atomic basis), where M denotes the dopants, Ni, Cr, Cu, Al, and Mg. It was not known in advance whether or not the dopants dissolved completely. Also the composition is described under the assumption that all dopants substitute for divalent zinc in the structure, which may not be correct for Cr and Al. Characterization of the as-sintered pellets consisted of compressive strength measurements, and determinations of the bulk densities, from which the porosities were calculated. The remainder of the effort at SIUC consisted of experiments carried out in a thermogravimetric analyzer (TGA) and a controlled-atmosphere tube furnace to study the sulfidation-regeneration behavior and the composition of the sulfide phases formed. For these experiments, pellets were crushed to a particle size of 2 to 4 mm.

In the solubility experiments carried out at VPI, reagent-grade oxide powders were mixed in appropriate proportions under acetone in a mortar and pestle and air dried. The process of mixing and drying was repeated 3 to 4 times. One gram from each of the 15 gram formulations was fired in an open alumina crucible. The sintering time was 24 hours, and the sintering temperatures were 900°, 1000°, and 1100° C. A Philips x-ray diffractometer (PW1729) employing $CuK\alpha$ radiation, operating at ~40kV and ~25mA and equipped with a goniometer scanning at a rate of 1° - 2 θ /minute/inch of the record chart was used for identification of the phases present in all samples. For lattice parameter measurements, the speed was reduced to 0.25° - 2 θ /minute/inch of the record chart. All x-ray measurements were made at room temperature. No internal standard was used but the instrument is calibrated weekly. The (511) reflection, ~56.6° 2 θ , and either the (422) reflection, ~53.1° 2 θ , or the (440) reflection, ~62.2° 2 θ , were used for lattice parameter, a_o , measurements. Since these are not very high angle reflections, a_o is not precise; however, comparisons of relative values are valid.

RESULTS AND DISCUSSION

At SIUC, the data base on the effects of additions of Ni, Cr, and Cu to zinc titanate obtained last quarter was extended to include Mg and Al. Summary data on bulk density, porosity, and crush strength for all five formulations are listed in Table 1. For comparison purposes, data are also given for a formulation with no dopant but excess TiO_2 added for strengthening. The porosity values were calculated after estimating the theoretical density two ways. In one method, a weighted average of the starting oxide densities was used. In the second method, the ionic radii of the dopants were used to estimate changes in the crystal lattice constant of zinc titanate. The two methods agreed within 4%, so the average was used in preparing Table 1.

TABLE 1. Porosity and Crush Strength Results for Doped Zinc Titanate Sorbents

NOMINAL SORBENT COMPOSITION	BULK DENSITY (g/cm^3)	ESTIMATED POROSITY (%)	CRUSH STRENGTH (N/mm)
$\text{Zn}_2\text{TiO}_4 + 50\% \text{TiO}_2$	2.38	46.6	55.8
$\text{Zn}_{1.6}\text{Ni}_{0.4}\text{TiO}_4$	2.70	49.5	113.7
$\text{Zn}_{1.6}\text{Cr}_{0.4}\text{TiO}_4$	2.25	54.6	65.3
$\text{Zn}_{1.6}\text{Cu}_{0.4}\text{TiO}_4$	2.79	47.4	137.5
$\text{Zn}_{1.6}\text{Mg}_{0.4}\text{TiO}_4$	2.11	56.0	47.3
$\text{Zn}_{1.6}\text{Al}_{0.4}\text{TiO}_4$	1.92	61.8	51.8

It can be seen that the bulk densities and crush strengths of the formulations containing Ni and Cu are higher than for the others. The oxides of these metals are less refractory, leading to more rapid sintering by either diffusion or viscous flow mass transport mechanisms. All of the properties listed in the Table are adjustable up or down by adjusting the sintering time and temperature, so it is not appropriate to make final judgements on dopant benefits unless sintering parameters are studied as variables.

The results of TGA experiments on the formulations containing Mg and Al are given in Figures 1 and 2. Similar results for Ni, Cr, and Cu were presented last quarter. In Figure 1, the weight gain during the first sulfidation was 11.6 %, which is slightly more than one would expect for sulfidation of all the Zn but none of the Mg (10.7%). The specimen regenerated without difficulty, but a significant loss in sulfur capacity was observed during the second sulfidation.

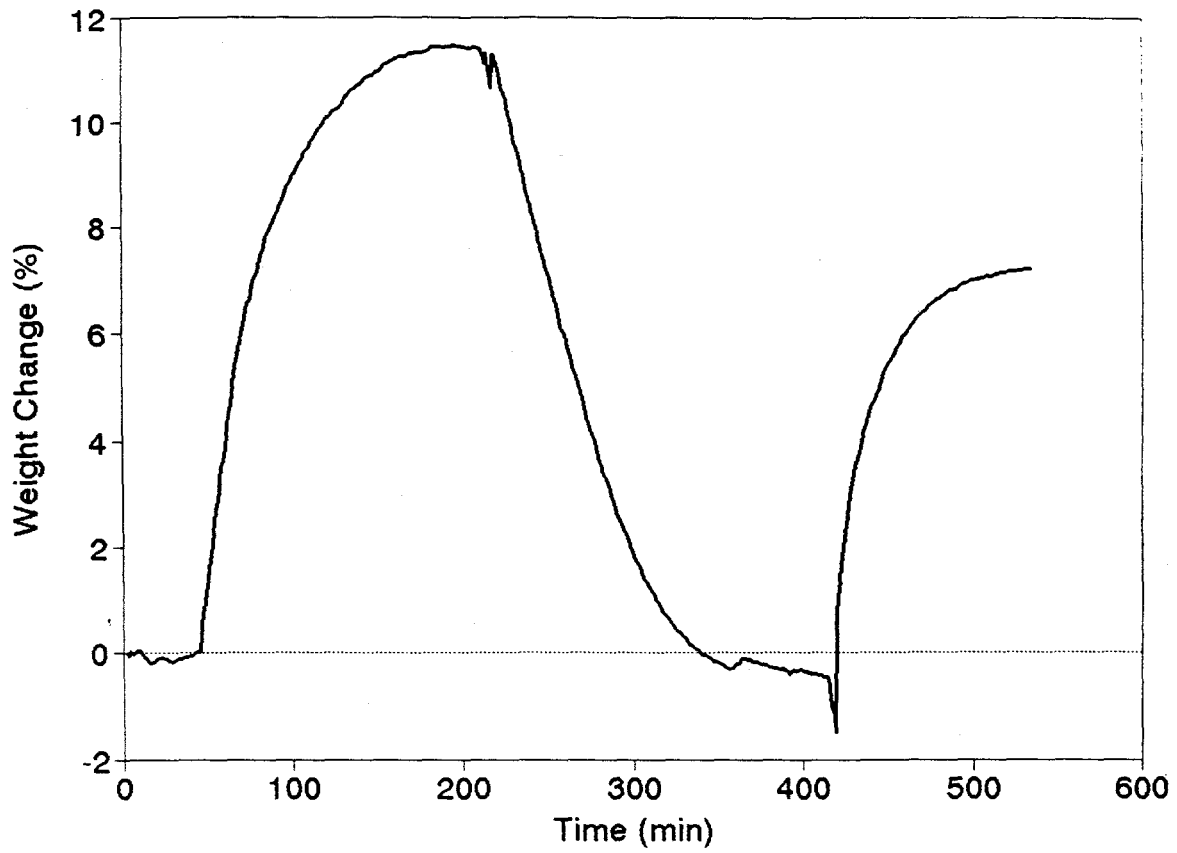


Figure 1. Sulfidation of $Zn_{1.6}Mg_{0.4}TiO_4$ in 0.9% H_2S , 92.4% H_2 and 6.7% N_2 , and Regeneration in 5% O_2 and 95% N_2 at 650° C.

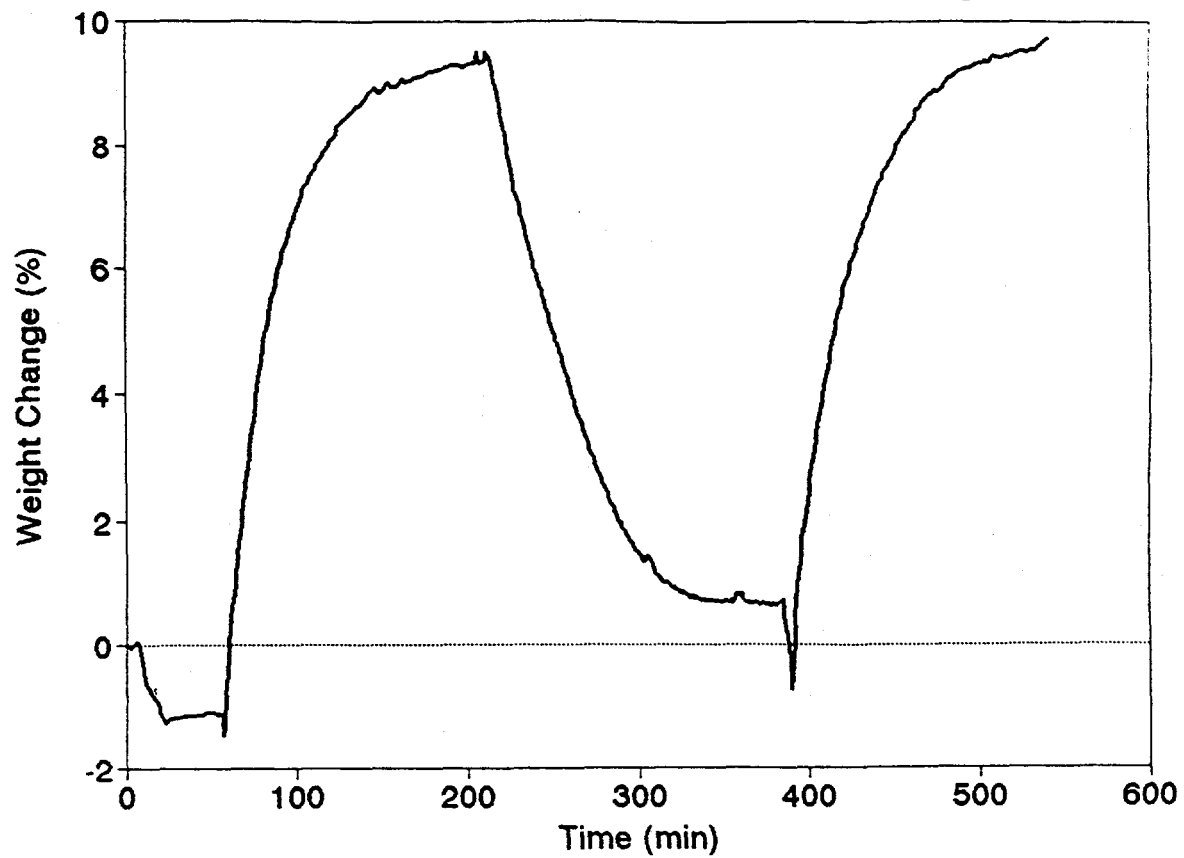


Figure 2. Sulfidation of $Zn_{1.6}Al_{0.4}TiO_4$ in 0.9% H_2S , 92.4% H_2 and 6.7% N_2 , and Regeneration in 5% O_2 and 95% N_2 at 650° C.

Figure 2 shows similar results for an Al-doped specimen. The 1.1 % weight loss at the beginning was probably due to vaporization of moisture. When this factor is taken into consideration, the weight gain on sulfidation is 10.6%, almost exactly the value expected for conversion of all the Zn in the titanate to ZnS. The weight loss on regeneration was less than the initial gain, indicating that some sulfate may have formed. The second sulfidation shows the same sulfur capacity as the first. A discussion of how these results for Mg and Al compare to the other dopants will be given at the back of the report.

A summary of the dopant solubility results obtained at VPI will now be presented. For all systems, reactions at 900° C were incomplete as determined by the amount of oxides that remained unreacted. At 1000° and 1100° C, incorporation of the cations was achieved as seen in Figures 3-6. The lattice parameter decreased with increasing amounts of the cations. Each cation has a smaller atomic radius than Zn^{+2} , and several have a smaller radius than Ti^{+4} , i.e. Cr^{+3} and Al^{+3} .

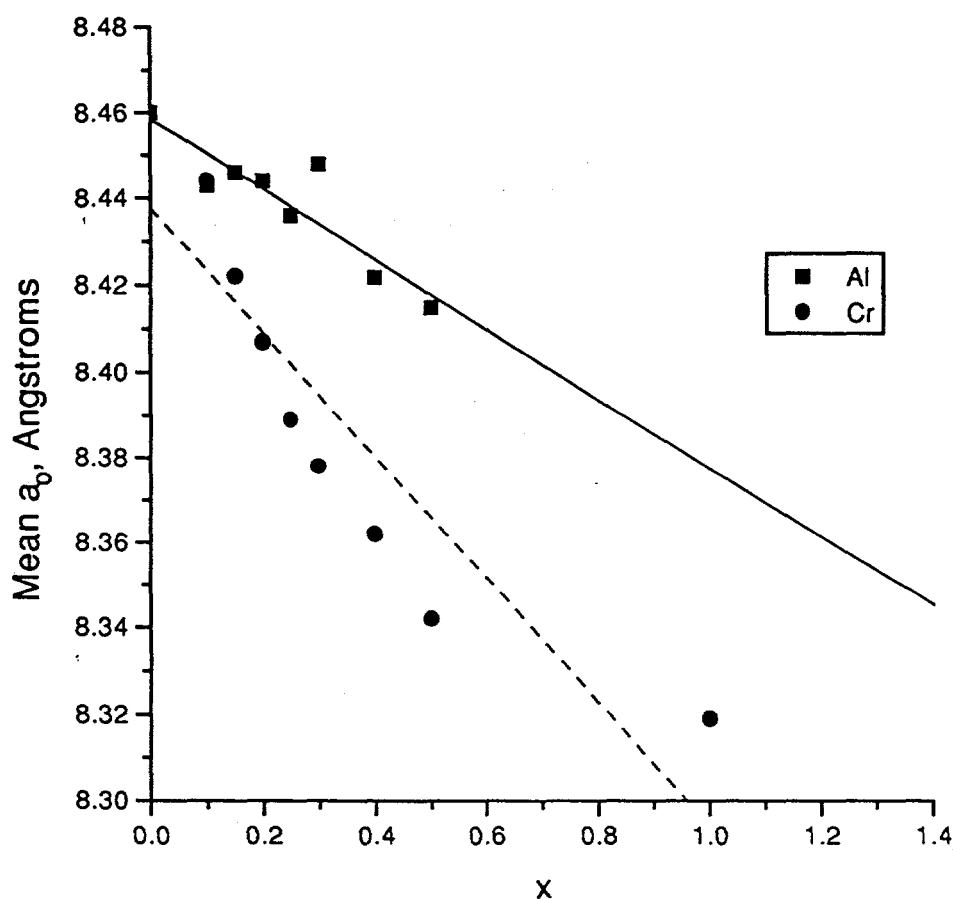


Figure 3. Comparison of Mean a_0 for $Zn_2TiO_4-MO_x$, $M = Al, Cr$ at 1000° C.

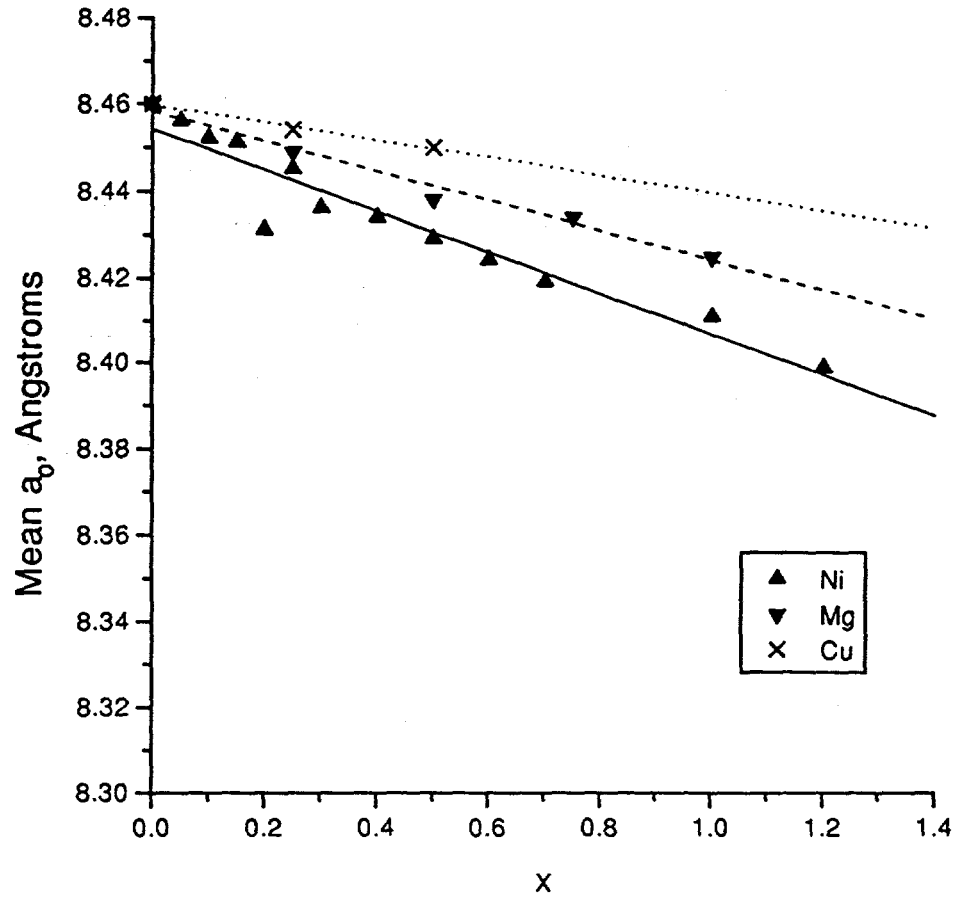


Figure 4. Comparison of Mean a_0 for $Zn_2TiO_4-MO_x$, $M = Ni, Mg$ and Cu at $1000^\circ C$.

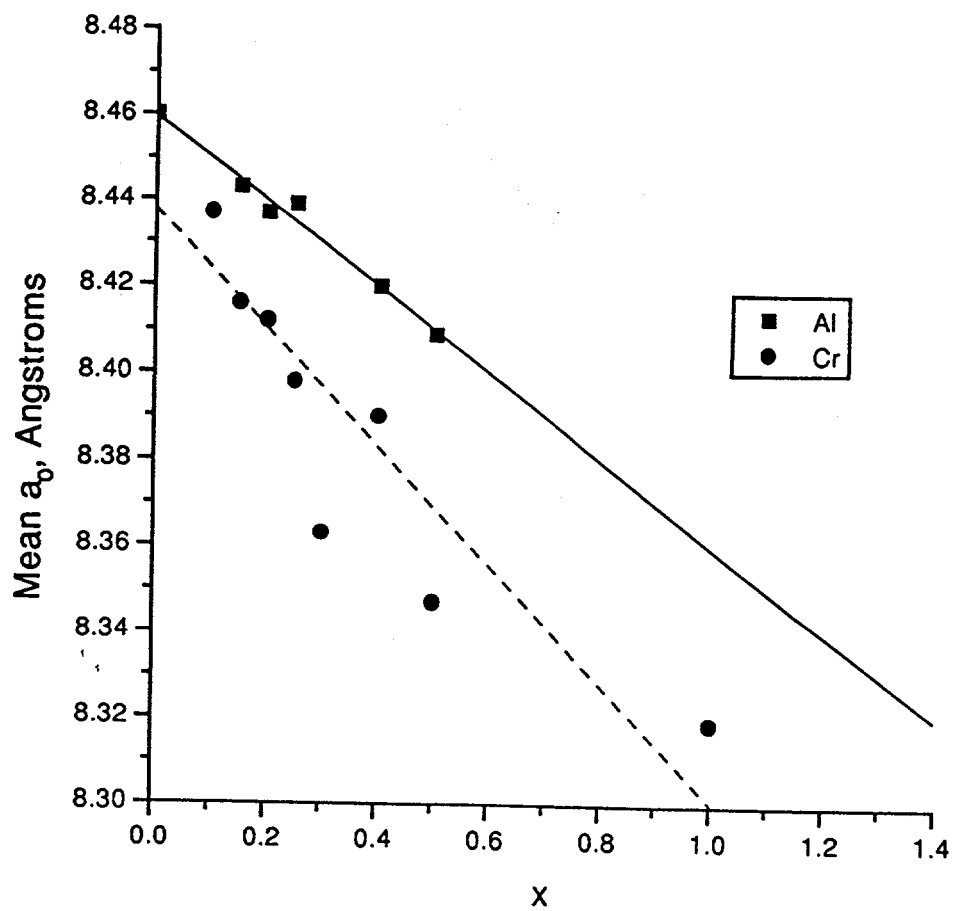


Figure 5. Comparison of Mean a_0 for $Zn_2TiO_4-MO_x$, $M = Al, Cr$ at $1100^\circ C$.

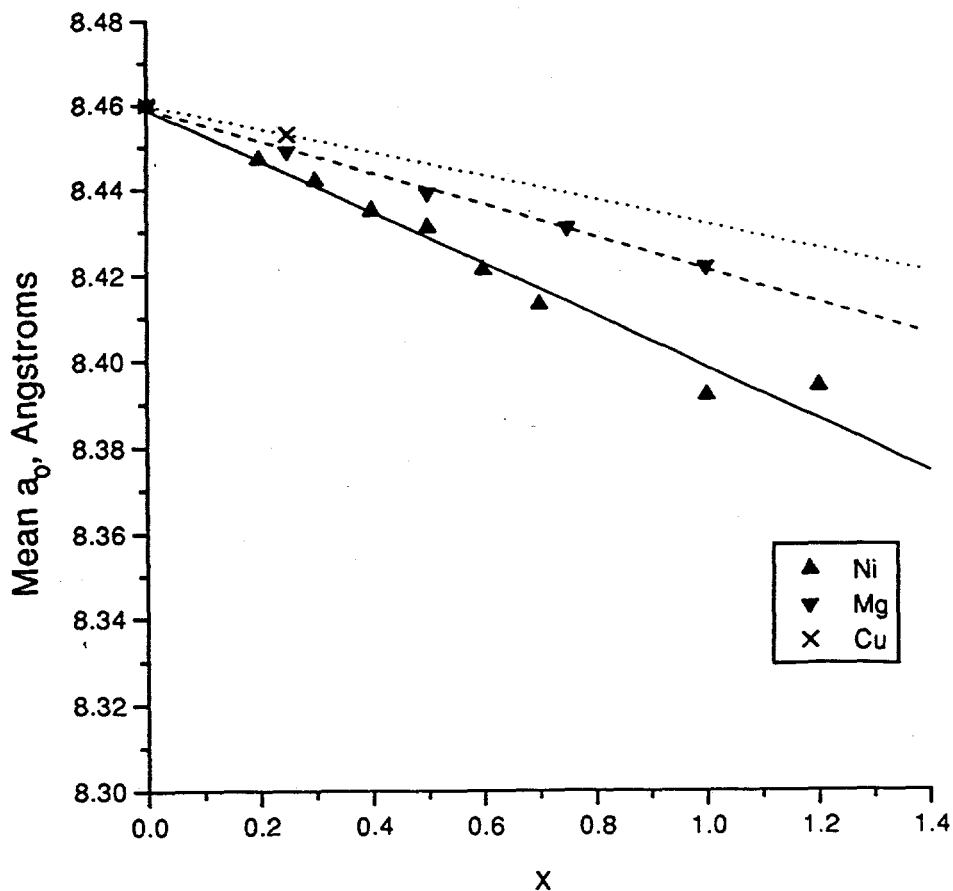


Figure 6. Comparison of Mean a_0 for $Zn_2TiO_4-MO_x$, $M = Ni, Mg$ and Cu at $1100^\circ C$.

For the purpose of this discussion, it is assumed that $Cu, Ni,$ and Mg substitute directly for Zn in the zinc titanate structure. Cr and Al normally have $+3$ valences. To maintain overall charge neutrality, two Cr or two Al ions are assumed to substitute for one Zn^{2+} and one Ti^{4+} ions.

For all systems except $Zn_{2-x}Cu_xTiO_4$, an upper limit on the concentration is reached, above which a larger percentage of oxides remain unreacted, and an essentially single-phase spinel solid solution is obtained below that composition. Above this composition, the lattice parameter is still decreasing indicating cation incorporation; however, the slope becomes more gradual and, in some systems, seems to level off. The results for each system will be discussed separately.

System $Zn_{2-x}Ni_xTiO_4$: Twelve compositions with x ranging from 0 to 1.2 were prepared and studied in this system. Ni^{+2} was able to be incorporated into the Zn_2TiO_4 lattice for compositions up to 35 mole% ($x = 0.7$). A predominantly single-phase spinel solid solution was obtained for each of these compositions at the higher temperatures.

System $Zn_{2-x}Ti_{1-x}Cr_{2x}O_4$: Cr^{+3} was also well incorporated into the Zn_2TiO_4 lattice. At 1000° and 1100° C, a single-phase spinel solid-solution with trace amounts (< 5 weight%) of oxides was obtained for all eight compositions prepared (x ranging from 0 to 0.5). Cr^{+3} is able to replace both Zn and Ti in the lattice and there is a sharper decrease in a_0 than was observed for Ni. Between $x = 0.5$ (33 mole% Cr) and $x = 1.0$ (66 mole% Cr), very little change in a_0 is observed and the slope is more gradual than for the compositions below $x = 0.5$.

System $Zn_{2-x}Ti_{1-x}Al_{2x}O_4$: At 1000° and 1100° C, a predominantly single-phase spinel solid solution was obtained for all eight compositions prepared with x ranging from 0 to 0.5 (0 to 33 mole% Al). Again, Al^{+3} can replace both Zn and Ti in the lattice and there is a sharp decrease in a_0 . This decrease in a_0 is not as sharp as the slope for Cr although the radius of Al^{+3} is smaller than Cr^{+3} , see Figures 3 and 5. Due to the larger size difference between Al^{+3} , Zn^{+2} , and Ti^{+4} , Al can be incorporated with less distortion to the lattice.

System $Zn_{2-x}Mg_xTiO_4$: Four compositions with x ranging from 0 to 1.0 were prepared for study in this system. At 1000° and 1100° C, compositions with x less than 0.50 (25 mole%), were essentially a single-phase spinel solid solution. The decrease in a_0 was less than Ni although they both have the same atomic radius.

System $Zn_{2-x}Cu_xTiO_4$: Between 900° - 1100° C, it was observed that the samples, five compositions with x ranging from 0 to 2.0, reacted with the alumina crucible. Because of the reaction, the exact composition of the spinel phase cannot be deciphered. XRD of the samples which did not react completely with the crucible shows a spinel phase along with $CuTiO_x$ and oxide phases. Lattice parameters could be calculated for only two compositions at 1000° C and one composition at 1100° C. A decrease in a_0 is observed for these compositions indicating incorporation of Cu^{+2} into the lattice. The upper limit of Cu incorporation could not be determined; however, at least 12.5 mole% ($x=0.25$ in the formulated composition) of Zn can be replaced by Cu at 1100° C and an essentially single-phase spinel solid solution obtained.

The only one of these systems that has been studied before is the Zn-Ni-Ti-O system (Datta and Roy, 1965). The experiments were similar, except that a larger temperature range was studied, and the experiments were done at high pressure to assist in attaining equilibrium. The results are consistent with those reported here; the solubility of Ni_2TiO_4 in Zn_2TiO_4 was found to be 62 mole percent at 1000° C.

CONCLUSIONS AND RECOMMENDATIONS

The screening experiments on the five dopants were completed on schedule during the first half of the contract period. With the time remaining, it is recommended that most of the effort be directed toward one system that appears to offer the most promise. The choice is not difficult; the formulation containing chromium was the only one that had a high sulfur capacity, chemical stability in reducing gases, and good sulfidation-regeneration

characteristics in TGA tests. Its crush strength was not among the highest, but that property is relatively easy to improve by varying preparation parameters. Some of the other dopants may still have promise, but they will only be studied further, other than tying up a few details, if time is available later in the year.

DISCLAIMER STATEMENTS

This report was prepared by James H. Swisher of Southern Illinois University at Carbondale with support, in part by grants made possible by the U.S. Department of Energy Cooperative Agreement Number DE-FC22-92PC92521 and the Illinois Department of Energy through the Illinois Coal Development Board and the Illinois Clean Coal Institute. Neither James Swisher of Southern Illinois University at Carbondale nor any of its subcontractors nor the U.S. Department of Energy, Illinois Department of Energy and Natural Resources, Illinois Coal Development Board, Illinois Clean Coal Institute, nor any person acting on behalf of either:

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PROJECT MANAGEMENT REPORT
December 1, 1994 through February 28, 1995

Project Title: **COMPOSITION MODIFICATION OF ZINC TITANATE SORBENTS
FOR HOT GAS DESULFURIZATION**

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 3)
ICCI Project Number: 94-1/2.2A-2M
Principal Investigator: James H. Swisher, Southern Illinois University at
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Co-Principal Investigator: Ranajit K. Datta, Virginia Polytechnic
Institute and State University
Project Manager: Franklin I. Hoena, Illinois Clean Coal
Institute

COMMENTS

Two milestones were scheduled for completion this quarter. One, which involved a literature search and equipment procurement, had been postponed from the first quarter, partly because of the timing on the award of a subcontract to VPI. This milestone was completed in January. The second milestone included completion of a set of solubility experiments. It was also completed on time in January. To date, spending is less than projected at the beginning of the project. At SIUC, the labor charges are lower than projected because the principal investigator shifted part of his salary to a different account in order to permit the hiring of another student next summer. At VPI, there are two main reasons for undercosting. One is that VPI's indirect cost rate was changed from 53% to 46% between the time when the proposal was written and the project started. The second reason is that an undergraduate student was not hired for the project as originally planned.

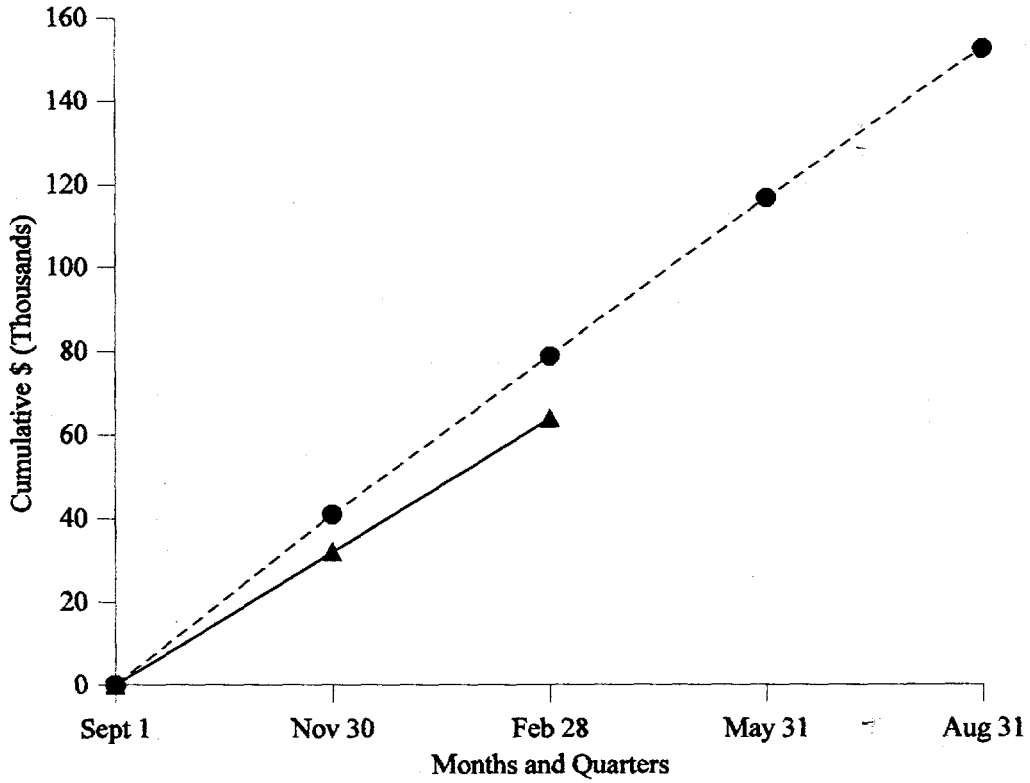
PROJECTED AND ESTIMATED EXPENDITURES BY QUARTER

Quarter*	Types of Cost	Direct Labor	Fringe Benefits	Materials and Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Cost	Total
Sept. 1, 1994 to Nov. 30, 1994	Projected	8,064	63	775	2,350	300	25,858	3,711	41,121
	Estimated	4,307	25	1,000	1,100	0	22,924	2,936	32,292
Sept. 1, 1994 to Feb. 28, 1995	Projected	16,128	126	1,550	2,350	300	51,715	7,187	79,356
	Estimated	10,708	87	1,778	1,481	0	43,551	5,823	64,048
Sept. 1, 1994 to May 31, 1995	Projected	24,192	189	2,325	2,350	300	77,573	10,663	117,592
	Estimated								
Sept. 1, 1994 to Aug. 31, 1995	Projected	32,256	252	3,100	2,700	300	100,820	13,943	153,371
	Estimated								

*Cumulative by Quarter

COSTS BY QUARTER

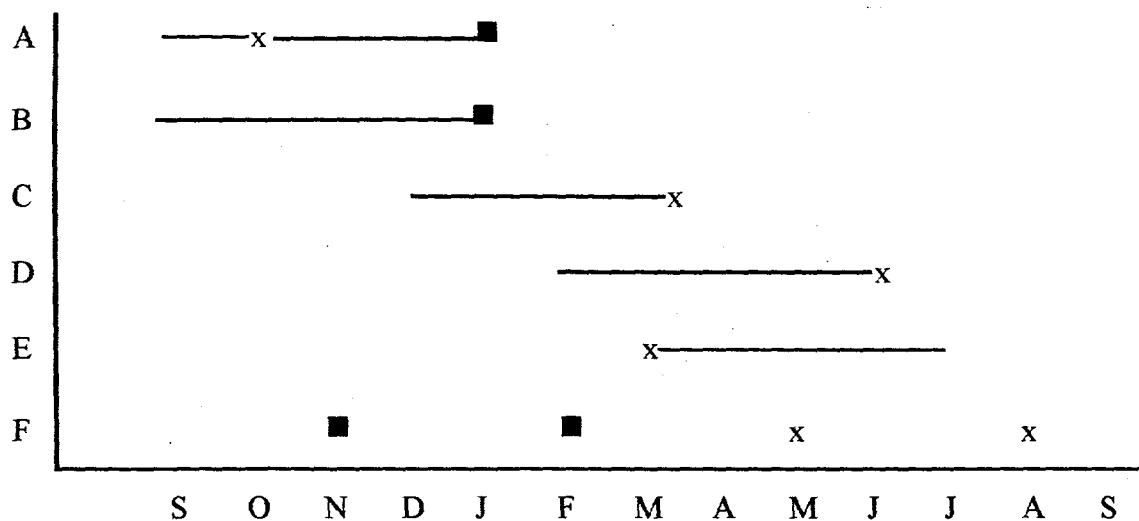
Composition Modification of Zinc Titanate Sorbents for Hot Gas Desulfurization



● = Projected Expenditures -----
▲ = Actual Expenditures _____

Total Illinois Clean Coal Institute Award \$153,371

SCHEDULE OF PROJECT MILESTONES



Begin
Sept. 1
1994

Hypothetical Milestones:

- A: Literature search; materials and equipment procurement (SIUC and VPI)
- B: $\text{ZnTiO}_4\text{-MO}_x$ solubility experiments (VPI)
- C: Sorbent preparation (VPI)
- D: Bench-scale testing (SIUC)
- E: Characterization of bench-scale samples (SIUC and VPI)
- F: Reporting (SIUC)