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Advanced Low-Temperature Sorbents

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CONTRACT INFORMATION

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Period of Performance September 30, 1994 to February 27, 1997

Schedule and Milestones

FY95 Program Schedule

	O	N	D	J	F	M	A	M	J	J	A	S
Task 1 – NEPA Report	—											
Task 2 – Sorbent Preparation												
Task 3 – Provision of Bench Unit												
Task 4 – Bench Testing												
Task 5 – Sorbent Cost Assessment												→FY'96
Task 6 – Topical Report												→FY'96
Task 7 – Market Plan												→FY'96
Task 8 – Long-Term Testing (option)												→FY'96

OBJECTIVES

The objective of the proposed program is to develop regenerable sorbents to remove hydrogen sulfide from coal-derived fuel gases in the temperature range 343 to 538 °C (650 to

1000 °F) using the fixed-, moving-, and fluidized-bed reactor modes. Sorbent materials should be prepared in bench-scale quantities for screening and characterization of physical properties, mechanical durability, and chemical reactivity. A cost assessment and a market plan for large-scale fabrication of the sorbents is to be

developed. Under a Contract Option, a long-term bench-scale test (at least 100 cycles) will be conducted to demonstrate durability and reactivity of the sorbents.

BACKGROUND INFORMATION

A number of promising technologies are currently being optimized for coal-based power generation, including the Integrated-Gasification Combined Cycle (IGCC) system. If IGCC is to be used successfully for power generation, an economic and efficient way must be found to remove the contaminants, particularly sulfur species, found in coal gas. Except for the hot gas desulfurization system, all major components of IGCC are commercially available or have been shown to meet system requirements [Corman, 1986]. Over the last two decades, the U.S. Department of Energy/Morgantown Energy Technology Center (DOE/METC) has sponsored development of various configurations of high-temperature desulfurization systems [Bossart et al., 1990] including fixed-bed, moving-bed, transport-bed, and fluidized-bed systems. Because of their mode of operation and requirements for sorbent manufacturing, the fixed-bed systems can generally use the same materials as moving-bed configurations, i.e., pelletized or extruded sorbents, while fluidized-bed (circulating or bubbling configurations) and transport reactor configurations use materials generally described as agglomerated or granulated.

Moving-bed systems

Moving-bed systems are under development at GE [Bevan et al., 1994]. Although the sorbent materials move slowly between the absorber and regenerator vessels, the level of deterioration at process conditions requires the material to be mechanically durable.

Sorbent formulations prepared for moving-bed systems must have mechanical durability and exhibit satisfactory desulfurization kinetics at 538 °C (1000 °F) [Ayala et al., 1994]; however, larger-scale demonstration systems such as the Tampa Electric Co.'s Polk Station will operate at lower temperatures, close to 482 °C (800-900 °F). Hence, development of sorbents with reactivity in this middle temperature range of 343 to 538 °C (650-1000 °F) is important for moving-bed systems.

Fluidized-Bed Systems

Although the fixed-bed approach using metal oxides has been shown to reduce the H₂S content of the cleaned gas to very low levels, the operation is not continuous, it suffers from operational problems, and requires large-scale high temperature valves. To overcome these problems, US DOE has also sponsored fluidized-bed hot gas desulfurization research in recent years. The fluidized-bed approach offers advantages because of its ability to control the reactor temperature during the highly exothermic regeneration step.

The key characteristic of concern in the fluidized-bed application is maintenance of the physical integrity of the sorbent during the chemical transformations associated with absorption-regeneration. Testing appears to verify that excessive attrition results primarily from the changes in composition during transformation and not from mechanical forces. The extreme temperature and severe chemical conditions of air-steam regeneration is deleterious to many inorganic support materials and sorbent compounds. A sorbent suitable for fluidized-bed application must, therefore, have both good thermodynamic equilibrium and initial chemical reactivity in the range of 343-538 °C, as well as acceptable long-term physical and

chemical durability with a reasonable cost of production.

PROJECT DESCRIPTION

The technical work is directed at developing regenerable desulfurization sorbents in the temperature range 343 to 538 °C (650 to 1000 °F). The proposed program is divided into eight tasks:

In Task 1, the information required for preparation of a NEPA report by DOE will be provided. In Task 2, several sorbents suitable for fixed-, moving-, and fluidized-bed applications will be fabricated. Three classes of materials will be used: zinc-based materials (e.g., zinc titanates), advanced metal oxide (non-zinc) materials, and proprietary materials. Selection will be based on thermodynamic and laboratory evaluation of powder materials. GE-CRD, IGT, and Prof. M. Flytzani-Stephanopoulos (consultant, Tufts University) will be responsible for identifying and screening the chemical compositions of the sorbents. Pelletization (for moving/fixed beds) and granulation (for fluidized beds) will then be conducted on the promising materials using suitable binders and pore formers. Standard techniques (extrusion and calcination) as well as novel techniques (solution precipitation or sol/gel formation) will be used to modify the morphology of the formed materials. GE-CRD will focus on moving/fixed-bed applications, while IGT will focus on fluidized-bed applications. Commercial sorbent and catalyst developers will pelletize or agglomerate the final formulations for bench testing according to recommendations by GE-CRD and IGT. In Task 3, the moving/fixed-bed bench reactor at GE-CRD and the fluidized-bed reactor at IGT will be refurbished for use. In Task 4, the eight most promising sorbents that passed screening under Task 2 will be tested for ten cycles of absorption and regeneration each in the GE-

CRD moving/fixed-bed bench reactor and IGT fluidized-bed reactor, and the properties of the used materials will be compared against those of the fresh sorbents. In Task 5, a sorbent cost assessment will be performed to determine costs and sensitivity of costs for raw materials, fabrication, disposal, or recycle. In Task 6, a Topical Report will be prepared summarizing the results obtained in the program. In Task 7, a Market Plan will be developed jointly with the commercial sorbent manufacturers and users to develop a strategy for commercialization, teaming/licensing approaches, and benefits to IGCC technologies.

Finally as an optional task, Task 8 will test a minimum of two formulations, one for moving/fixed-bed systems and the other for fluidized-bed systems for long-term (at least 100 cycles) durability and chemical reactivity in the corresponding bench-scale reactor configurations.

RESULTS

Literature review results

The research and development for high-temperature desulfurization of fuel gases has been sponsored primarily by the Morgantown Energy Technology Center (METC) of the United States Department of Energy (U.S. DOE). Over the last decade a number of studies have been reported on high-temperature H₂S removal, primarily using various transition metal oxides as regenerable sorbents [Grindley and Steinfeld, 1981; Jalan and Wu, 1980].

The sorbent most intensively studied is iron oxide, which yields equilibrium H₂S concentrations in the few hundred ppm range for a composition representative of low-Btu coal-derived gas and temperatures of above 500 °C. While the sulfidation kinetics of iron oxide are very good [Tamhankar et al., 1981], this sorbent

cannot be used for single-stage coal gas desulfurization to reduce the H₂S content of the fuel gas down to a few ppm of sulfur.

Zinc oxide was originally used as a non-regenerable sorbent in "guard beds" protecting catalyst beds from trace sulfur impurities. More recently, zinc oxide has also been investigated as a regenerable sorbent [Lew and Flytzani Stephanopoulos, 1989; Westmoreland et al., 1977]. The thermodynamic equilibrium for sulfidation of ZnO is quite favorable, yielding desulfurization down to a few ppm H₂S. The sulfidation kinetics of ZnO, however, are much slower [Westmoreland et al., 1977] compared to those of iron oxide, and the regenerability of ZnO is restricted by the loss of surface area at high temperatures and the formation of zinc sulfate at low regeneration temperatures. Reduction to metal and sulfidation take place simultaneously when the hot fuel gas contacts the sorbent.

Until the early 1980's most of the elements in the periodic table were being considered on an empirical basis for use as sorbents for sulfur removal. Westmoreland and Harrison [Westmoreland and Harrison, 1976] did a systematic thermodynamic analysis of 28 elements and arrived at 11 useful elements (i.e., Fe, Zn, Mo, Mn, V, Ca, Sr, Ba, Cu, and W) to be considered in the temperature range of 400 to 1200 °C. Jalan and coworkers [Jalan, 1983] studied some of these metals for desulfurization capabilities at 650 °F. Even under adverse conditions of interference from coke formation and water gas shift reactions, these candidates were attractive for desulfurization to low levels. Other researchers have studied many other pure metal oxides, and their review will be postponed because of space limitations.

Work on zinc titanates such as ZnTiO₃, Zn₂TiO₄, and Zn₂Ti₃O₈ [Lew et al., 1989;

Flytzani-Stephanopoulos, 1987] has shown that titanium oxide is a better alternative to iron oxide additives in terms of the higher stability of the titanates over the ferrite compounds of zinc, and their similar sulfidation equilibria. However, resistance to attrition is still an issue. With zinc titanates, the sulfidation temperature has been shown to extend to 700 °C and sintering of the ZnO was greatly reduced, depending on the Zn:Ti ratio chosen. Although zinc titanate has also shown better attrition resistance than zinc ferrite in pilot tests, this sorbent also suffers gradual loss of reactivity in long-term cyclic operation, resulting in high fresh sorbent makeup rate to maintain the desired level of desulfurization. Other mixed-metal oxides such as copper-based and cobalt-based sorbents have also been investigated [Abbasian et al, 1992]; however, the research has been limited to laboratory-scale equipment.

Thus far, the temperatures of interest have been mostly above 500 °C (932 °F) with emphasis in the range 538–700 °C (1000–1302 °F). This temperature range was chosen because higher coal gas temperatures to the turbine inlet resulted in higher overall efficiency for the IGCC system. However, recent economic studies indicate that very high coal gas temperatures also carry significant penalties in terms of costs of the materials of construction and choice of alloys needed to withstand temperature, pressure, and corrosion requirements of the coal gas. A reasonable compromise between cycle efficiency and capital and operating costs appears to be now in the range of 343–538 °C (650–1000 °F) [Droog et al., 1992]. In addition, the stringent requirement for sulfur removal efficiency at temperatures above 538 °C (1000 °F) limits the choice of the sorbents to a few metal oxides (based on thermodynamic equilibrium) that have been shown to have other limitations as described earlier. The thermodynamic equilibria of many

metal oxides significantly improve as the temperature decreases, making many metal oxide sorbents suitable for hot gas cleanup application in the temperature range of 343 to 538 °C (650 to 1000 °F). On the negative side, the intrinsic rate of reaction decreases exponentially with temperature, so much slower desulfurization kinetics will be anticipated below 538 °C (1000 °F).

Although the initial chemical reactivities of the sorbents generally decrease with decreasing temperature, the lower thermal stresses incurred can lead to better sorbent reactivity after a large number of cycles, reducing the sorbent replacement cost. In addition, issues such as zinc evaporation are no longer a problem when going to absorption temperatures below 1000 °F. In general, the benefit to be gained by lower temperature application to 343 to 538 °C (650–1000 °F) may outweigh the slight loss of efficiency due to lower temperature application, resulting in lower overall cost of electricity. The investigation to be undertaken in this program must include selection of the appropriate material that can offer good thermodynamic equilibrium and initial chemical reactivity, as well as acceptable long-term physical and chemical durability with reasonable cost of production.

Copper-based sorbents were recently evaluated at IGT for their application at temperatures of 650–850 °C [Abbasian et al, 1992]. Two “promising” copper-based sorbents were identified in this program for further development. H₂S breakthrough curves for these sorbents typically indicated very low H₂S pre-breakthrough levels, ranging from less than less than 1 to about 10 ppmv, which are significantly lower than those predicted for the elemental Cu-H₂S equilibrium (implying that the copper in the compound oxide was not completely reduced to elemental copper by the fuel gas during sulfidation reaction). These

results and the fact that the thermodynamic equilibria of many metal oxides improve as the temperature decreases, suggest that copper-, or other metal oxide-based sorbents may also have a potential for hot gas cleanup application at the lower temperatures of interest in this program. Although the initial chemical reactivities of metal oxide sorbents generally decrease with decreasing temperature, the lower thermal stress incurred at lower temperatures can lead to better sorbent reactivity after a large number of cycles, reducing the sorbent replacement cost.

Thermodynamic calculations

The thermodynamic H₂S equilibrium concentration in simulated fuel gases (U-GAS and Texaco) for a number of potential metal oxides were determined at elevated temperatures (i.e. 350–550 °C) and pressure (20 bar). The metal oxides considered for this analysis included copper, zinc, iron, nickel, tin, cobalt, molybdenum, manganese, and tungsten. The number of moles of the fuel gas and the solid oxides were selected to correspond to 50% sorbent conversion during sulfidation. Based on the available data, certain restrictions were imposed on formation of a number of species, to better simulate the actual environment. The results of these analyses are presented in Figures 1 and 2 for U-GAS and Texaco fuel gases respectively, indicating that the calculated H₂S equilibrium is not significantly affected by the fuel gas composition, if one allows for water-gas-shift reaction equilibration. Furthermore, because of the lower temperature range (i.e. 350–550 °C), a large number of metal oxides are capable of reducing the H₂S level of the fuel gases to below 20 ppmv for IGCC application. It should be noted that the H₂S equilibrium for copper oxide is the lowest (e.g. below 0.1ppmv) if reduction to metallic copper is prevented,

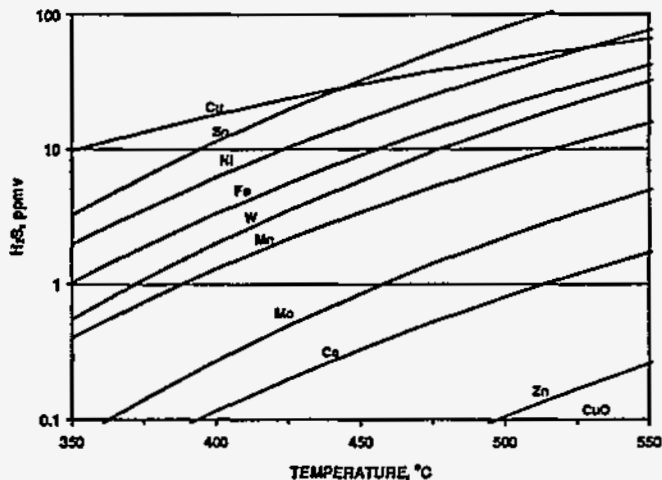


Figure 1.- H₂S equilibrium concentration in fuel gas with various oxides (U-GAS)

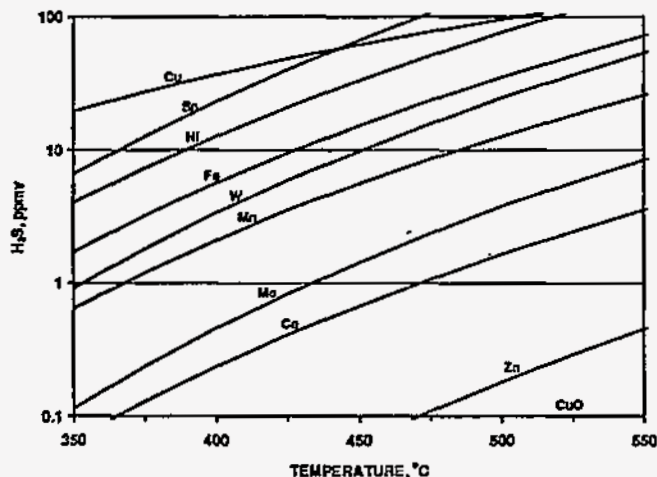


Figure 2.- Equilibrium Concentration In Fuel Gas With Various Oxides (Texaco)

while in the reduced form, metallic copper is still acceptable candidate.

Phase stability diagrams for reducing gas atmospheres (i.e., coal gases) and oxidizing gas atmospheres (i.e., regeneration gases) have been constructed using algorithms that predict the stability of chemical species based on minimization of the Gibbs free energy of the

system. The phase stability of metals under regeneration gases containing SO₂ and O₂ is important because spalling and pellet deterioration occurs more often as a result of metal sulfate formation. Figure 3 shows the stability of copper and molybdenum as a function of the log partial pressure of O₂ and SO₂. For gas compositions containing approximately 0.02 atm of O₂ and 0.5 to 0.1 atm SO₂, (typical of atmospheric regeneration gases in the moving-bed system) the thermodynamically stable phase of copper is copper sulfate, while the thermodynamically stable form of molybdenum is the oxide. For most pure oxides, including zinc oxide, the metal sulfate is the stable form under oxidizing gas conditions at temperatures around 500-700 °C, depending on SO₂ and O₂ concentration. Of course, thermodynamic predictions do not take into account the rate of approach to equilibrium (i.e., kinetic rates) and hence experimental data is still needed to confirm the extent of formation of the predicted species. Nevertheless, thermodynamics allows the investigator to determine if formation of certain compounds is favored.

Selection of metal oxides process

Early thermodynamic analysis by Westmoreland and Harrison in 1976 set the stage for desulfurization candidates in the range of 400 to 1200 °C (752 to 2182 °F). With the new requirements of lower temperatures (343 to 538 °C), an update of the thermodynamic analysis was made to redefine the applicability of the various metal oxides to desulfurization in temperature regime of interest. Furthermore, the sorbent evaluation considered process-specific requirements for fixed/moving-bed and fluidized-bed configurations.

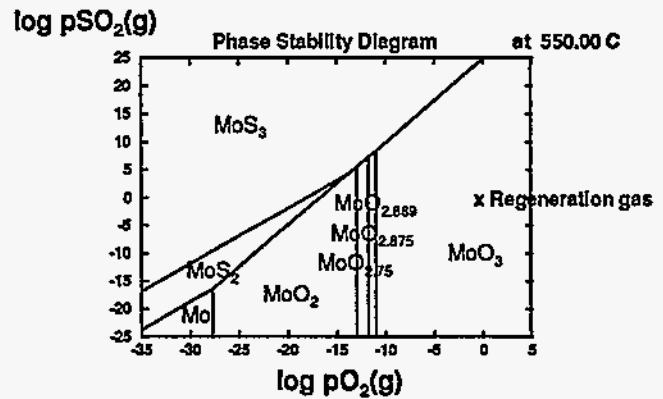
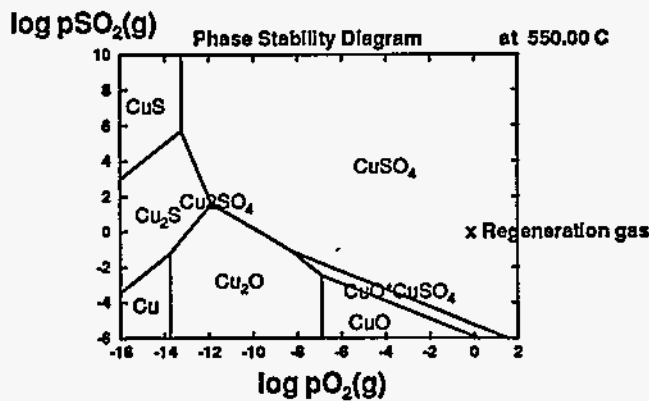


Figure 3.- Phase stability diagrams for Cu-S-O (left) and Mo-S-O (right) systems

Process requirements were taken as a basis for determining metal oxides suitability for high-temperature desulfurization.

1. Rate of desulfurization and stability of the sulfide under reducing gas conditions
2. Potential for detrimental secondary reactions on the solid under reducing coal gases (e.g., metal carbides, reduction to zero-oxidation state, formation of chlorides from HCl)
3. Rate of regeneration and production of sulfur dioxide or elemental sulfur under oxidizing gas conditions.
4. Potential for detrimental secondary reactions on the solid under oxidizing gases (e.g., sulfates) and hydrothermal stability during regeneration.

Taking previous experimental data and thermodynamic predictions into consideration, four metals (Zn, Fe, Ce, Cu) were selected as primary desulfurization species, four other metals (Mo, Co, Cu, Fe, as secondary species (i.e., additives) for desulfurization, and three metal species (Ti, Zr, Ce) were selected as

support and structure stabilizers. Combinations of these metal species will be tested in the laboratory and pelletization/agglomeration feasibility will be evaluated at commercial vendors.

Preliminary Sorbent Evaluation

The work on the preliminary sorbent evaluation focused on the determination of the sulfidation reactivity and effective capacity of metal oxide compounds in the temperature range of 350-500 °C to identify potential candidate sorbents for evaluation of other important characteristics such as sorbent regenerability and mechanical durability in multicycle operation.

The work performed included fixed-bed desulfurization testing of two copper-chromite sorbents and one zinc titanate sorbent. As mentioned above, these sorbents were shown to be highly efficient desulfurization sorbents at higher temperatures (550-850°C). The copper-chromite sorbents used in these tests contain about 10% (CuCr-1) to 25 % (CuCr-2) copper and have an average particle size of about 550 micron. The zinc titanate sorbent tested (UCI-4) has an average particle size of about 260 micron.

The sulfidation tests were carried out in a quartz fixed-bed reactor system at about one atmosphere pressure using a simulated coal derived-fuel gas mixture containing 0.5 vol% H₂S at a space velocity of 2000 hr⁻¹. Sulfided sorbents were regenerated by using nitrogen-air mixtures at temperatures of 650-750 °C. The product gas was analyzed for H₂S and SO₂ by a dedicated gas chromatograph.

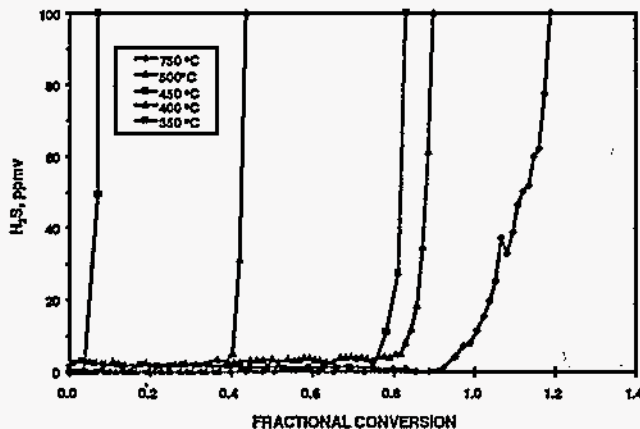


Figure 4.- H₂S Breakthrough Curves For Copper Chromite Sorbent (CuCr-1)

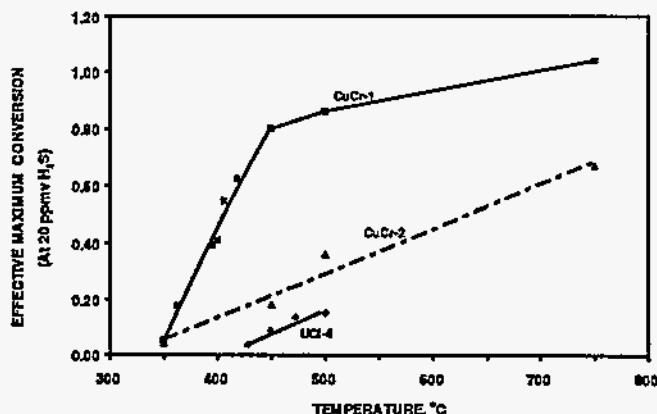


Figure 5.- Effect of temperature on effective maximum conversion

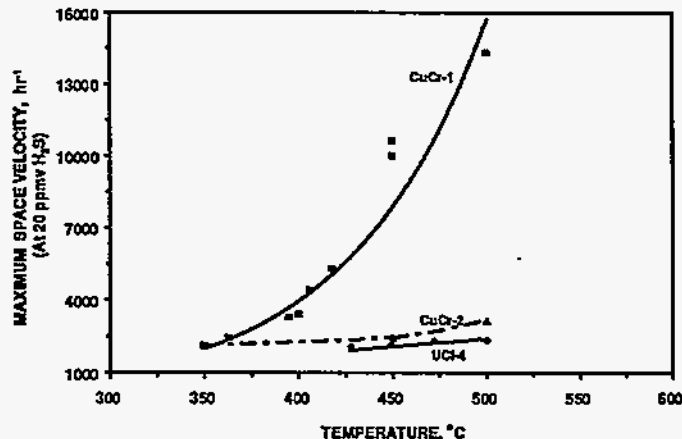


Figure 6.- Effect of temperature on maximum space velocity

The results of the sulfidation reactivity testing indicate that:

- All three mixed metal oxide compounds can reduce the H₂S content of the coal-derived fuel gas to below 20 ppmv in the desired temperature range; specifically, copper chromite can reduce the H₂S exit gas concentration to less than 5 ppmv (Figure 4), corresponding generally to the CuO-H₂S equilibrium.
- The reactivity of the copper chromite sorbents is generally higher than that of the zinc titanate sorbent tested.
- High levels of copper dispersion significantly improve copper chromite sulfidation reactivity up to very high effective maximum conversion levels of 85% (Figure 5), and allow the use of increased space velocities above 15,000 hr⁻¹ (Figure 6).

FUTURE WORK

The matrix of powder formulations selected was falls generally into three classes:

1. Modified zinc titanate and zinc-based formulations
2. Other novel (i.e. non-zinc) compositions
3. Optimization of proprietary existing formulations.

The composite and compound approaches to exploit the product and synergistic properties of the constituting components will also be focused upon. For example, the beneficial effects of small amounts of additives such as MoO₃ or CuO on the ZnO-TiO₂ system should be further explored by proper thermodynamic study of the CuO and MoO₃ doped compounds of ZnO-TiO₂. Spalling due to sulfate formation is a problem because the sorbent pore morphology is not fully compatible with (1) the thermodynamics of desulfurization and (2) the preparation technique used to fabricate the sorbent, both in moving-bed pellets and in fluidized-bed sorbent granules.

Chemical composition screening of powders is independent of the final reactor configuration. Further screening of the compositions will be based on the material cost and ceramic fabricability into useful sorbent morphologies at reasonable costs for the desired reactor applications. In the next step, pelletization of powders for moving beds and granulation for fluidized beds will be conducted jointly with commercial sorbent vendors. This step is extremely important to identify potential problems in the production of sorbents in commercial quantities at reasonable costs.

Moving-bed systems

Characterization techniques for pelletized formulations will be performed using the following techniques:

- Chemical reactivity on single pellets by thermogravimetric analysis (TGA)
- Attrition resistance before and after desulfurization testing by the modified ASTM D-4058-81 test.
- Crush strength by dead-weight load testing
- Long-term mechanical and chemical durability by the 10-cycle bench-scale tests

Characterization of moving-bed sorbents will be performed before and after bench testing to compare sorbent performance and/or degradation as a function of time.

Fluidized-bed systems

Based on the superior sulfidation performance of the chromium-rich copper chromite in the sulfidation reactivity tests, this sorbent will be evaluated for other important characteristics, such as sorbent regenerability and mechanical durability, in multicycle sulfidation-regeneration tests. Secondary components, such as Fe, Mo, or Co, will be added to enhance desulfurization reactivity, chemical stability, porosity and/or maintain mechanical durability as needed.

The kinetic and thermodynamic benefits of using pure copper oxide, as well as iron oxide, for desulfurization will also be examined.

Copper and iron oxide sorbents, stabilized against reduction and sintering by using inert support/dispersant materials, are expected to be more reactive and possibly lead to even lower thermodynamic equilibrium H₂S levels than the respective mixed metal oxide compounds.

Two materials, titania and zirconia, have been identified as potential candidates to have the proper chemical and physical properties needed for a regenerable metal oxide sorbent. Pure metal oxide supported sorbents containing different amounts of uncombined copper and iron oxide with zirconia and titania will be prepared in a granular form using a similar technique used for producing the highly dispersed, porous copper-chromite sorbents. The sulfidation reactivity and capacity of these supported sorbents will also be determined.

The most reactive and mechanically durable sorbent/additive combination will then be produced in a form and quantity suitable for fluidized-bed testing in the bench-scale desulfurization test unit.

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