

Long Life ZnO-TiO₂ and Novel Sorbents

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Introduction

Combined cycles (combinations of a gas turbine and a steam bottoming cycle) are the most efficient power generation technology, while coal is the lowest cost fuel. Therefore, the combination of Coal Gasifiers and Combined Cycles is predicted to be the lowest cost source of baseload electric power in the next decade. In a GCC, the sulfur and particulates are removed from the gasifier gases before they enter the turbine combustor. While H₂S (and COS/CS₂) can be removed effectively by cooling hot gases down to near room temperature and scrubbing them with an aqueous amine solution, removing the H₂S without cooling the gases (*i.e.*, hot gas cleanup) is more advantageous.

The leading hot gas sulfur absorbent uses a regenerable zinc oxide (ZnO) based sorbent, zinc titanate (Zn₂TiO₄ and/or ZnTiO₃), to remove the H₂S and other sulfur compounds from the hot coal gases. The zinc absorbs H₂S, forming zinc sulfide (ZnS); ZnS is then regenerated with oxygen (air), releasing the sulfur as a concentrated stream of SO₂. The SO₂ can be converted into sulfuric acid, sulfur, or reacted with calcium carbonate to form calcium sulfate (gypsum). The sorbent may be operated in a fluidized bed reactor, transport reactor, or moving bed reactor.

Both the fluidized-bed and the transport reactor use two separate reactors; one absorbs H₂S COS and CS₂ and converts the ZnO to ZnS; the second bed regenerates the sorbent with air converting the ZnS back to ZnO and producing SO₂ (Figure 1); the sorbent moves between the two reactors to carry sulfur out of the absorber and return regenerated sorbent. Fluidized bed and transport reactors circulate very small particles at high gas velocity. The high gas-solid contact area of very small particles rapidly transfers both heat and mass within the reactor.

The fluidized bed and transport reactor hot gas cleanup desulfurization systems are very similar and the sorbent particles are also of the same size (*i.e.*, 50 to 400 micron). While there are several differences between the two reactors, the one which most affects the sorbent is the operation of the regenerator: fluidized bed reactors operate with diluted air while transport reactors operate with undiluted air.

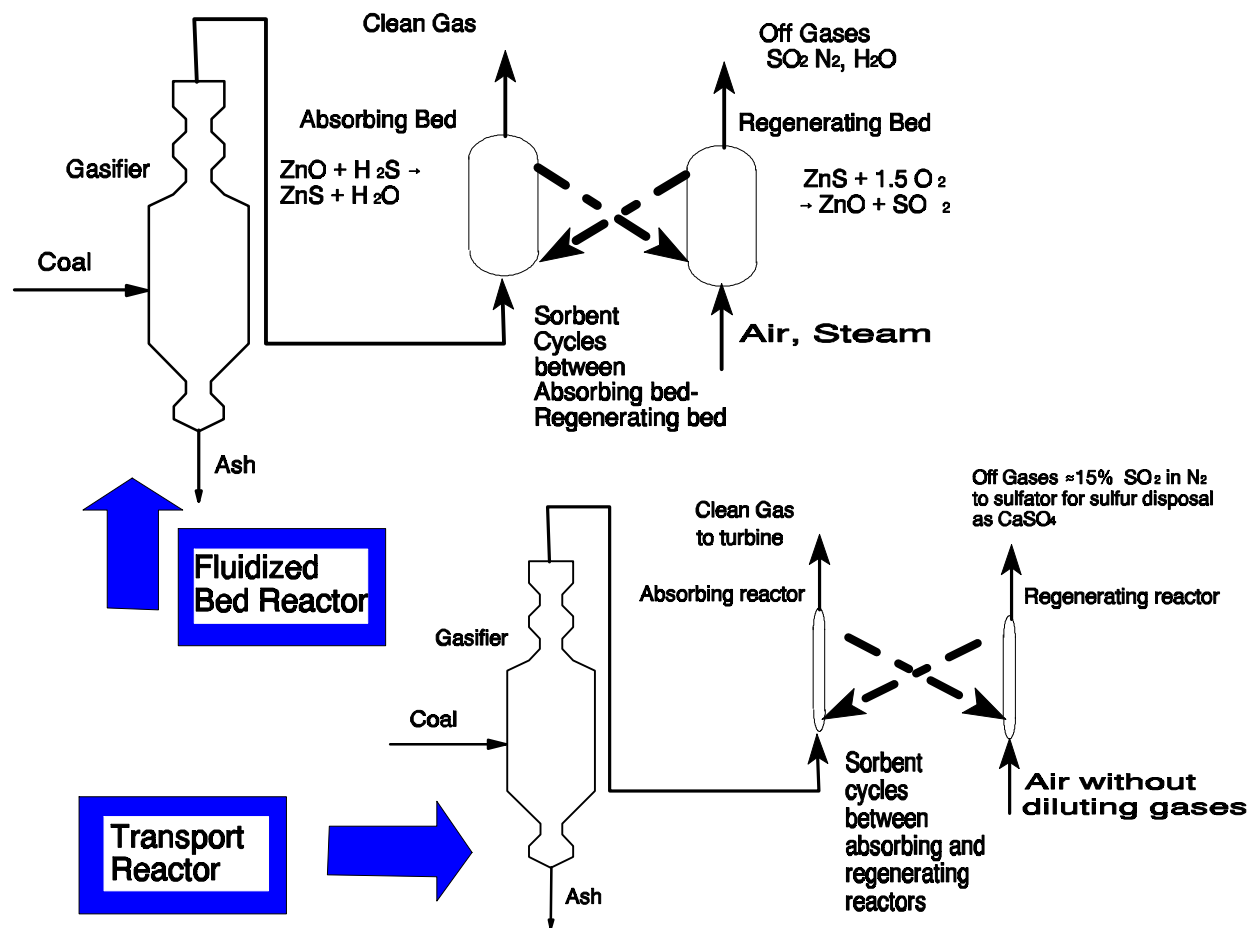


Figure 1 Hot gas desulfurization with fluidized bed and transport reactors.

A schematic of a moving bed system with the production of sulfuric acid from the SO_2 is shown in Figure 2. Again, two reactors are utilized with the sorbent moving between the two. The particle size is large (*i.e.*, 3 to 5 mm = 3000 to 5000 micron). Due to the particle size difference and other factors the reactions occur in a relatively short time in a fluidized or transport reactor, the dwell time in a moving reactor typically being on the order of 6 hours resulting in a different sorbent formulation for the different types of reactors.

ZnO has a high affinity for H_2S and produces a very clean gas stream. Unfortunately, the life of many ZnO based sorbents is very limited, especially in fixed and moving bed reactors. The molar volume of the ZnS is 50% greater than that of ZnO . During regeneration some zinc sulfate can form, which can occupy more than 250% of the original ZnO volume of the original ZnO . Prior to returning to the absorber, the zinc sulfate is decomposed and returns the sorbent to its original oxide form. Thus, the zinc atoms move substantially during absorption (to ZnS), to regeneration (to ZnO and some $ZnO \cdot 2ZnSO_4$), and finally back to the oxide (ZnO). This repeated expansion and contraction of the sorbent as it is cycled causes many sorbents to spall (*i.e.*, break into small pieces), which eventually destroys the sorbent (Jung *et al.* 1992)(Mei *et al.* 1993). Because of the high sorbent loss rate, fresh sorbent must be continually added to the process which in turn increases the cost of sulfur removal; substantial cost reductions could be achieved by making a more durable sorbent.

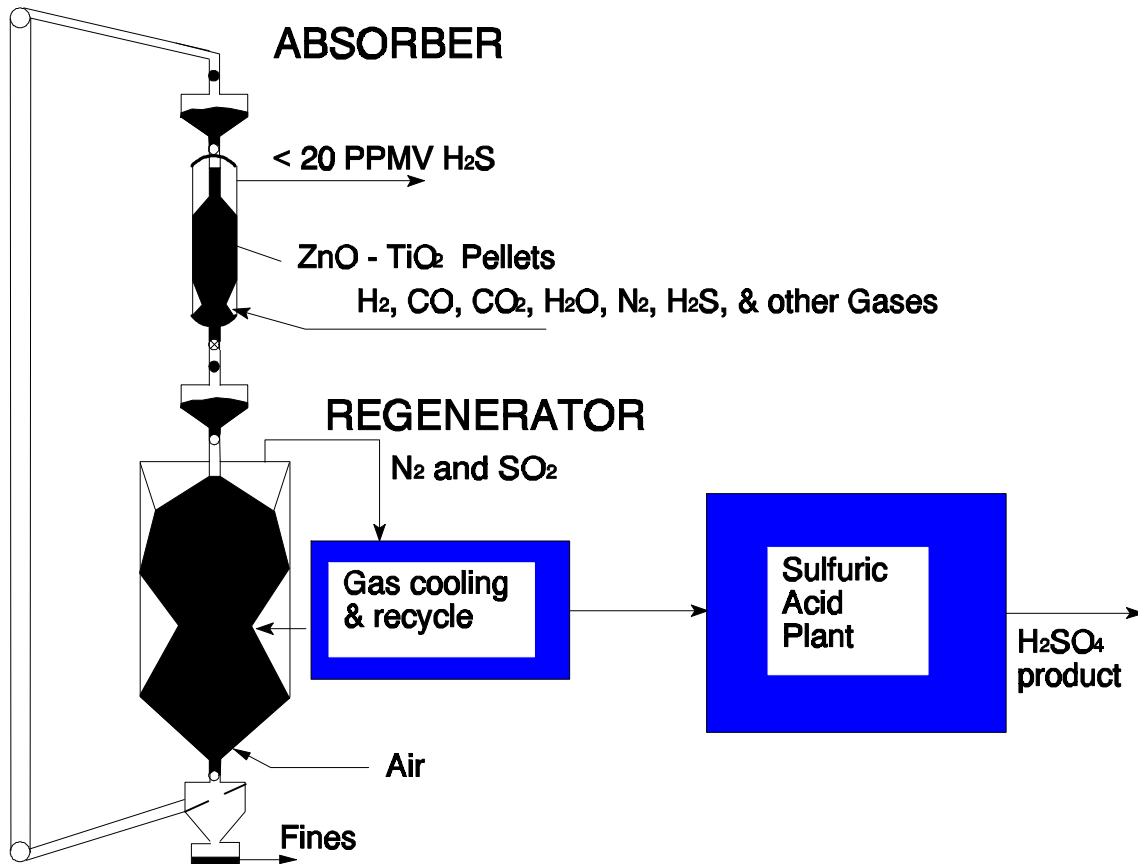


Figure 2. Moving Bed Reactor System

Recent developments in hot gas cleanup sorbents have reduced the spalling of the sorbent. Ayala *et al.* (1994) present data on both Z-Sorb and an improved zinc titanate. Z-Sorb, a product of Phillips Petroleum Company, performs well but is currently expensive. Similarly the zinc titanates are also expensive. These high costs are in part due to the high costs associated with small scale production but there may be factors in their production that require high costs. For example, several of Phillips' patents (*e.g.*, Khare 1994, Khare 1993, and Kidd *et al.* 1993) require the use of an aqueous acid solution (*e.g.*, nitric acid or acetic acid), which is later decomposed. Since the acids are lost in manufacture and the production equipment to handle acidic mixtures may be inherently expensive, Z-Sorb may be unavoidably expensive. A low cost sorbent that contains a large amount of active and accessible chemical sorbent and maintains a long life is necessary.

TDA Research, Inc. (TDA) is developing sorbents suitable for all three types of reactors. Under one contract, TDA is evaluating 3 to 5 mm sized sorbents for moving bed reactors ("A Long Life ZnO-TiO₂ Sorbent"); under a second contract ("A Novel Desulfurization Sorbent") we are evaluating 50 to 400 micron sized sorbents suitable for fluidized bed or transport reactors.

Objectives

The objective of this work was to develop and test long life sorbents for hot gas cleanup. Specifically, we measured the sulfur loading at space velocities typically used for absorption of H₂S and regenerated the sorbent with diluted air for multiple cycles. Based on the experimental results, we prepare a conceptual design of the sorbent fabrication system, and estimated the cost of sorbent production and of sulfur removal.

Technology

In order to make a sorbent pellet capable of holding large amounts of sorbent without being destroyed by the absorption-regeneration process, TDA has developed a new sorbent structure with the following characteristics: 1) is very strong, 2) allows the sorbent to expand and contract freely without disrupting the pellet structure, 3) allows the H₂S to diffuse quickly into the interior of the pellet, and 4) can contain very large amounts of sorbent without spalling.

TDA's process to produce the sorbent is also inherently low in cost and can achieve high loadings of the chemically active material. The sorbent is formed by mixed metal oxide techniques (*i.e.*, all of the ingredients are mixed at ambient temperature and fired only once), yet the sorbent has the high strength and long life typical of a catalyst support. In addition, the sorbent fabrication process does not require the use of an expensive decomposable salt.

Approach

TDA assembled a team to evaluate, produce and test sorbents for moving bed and fluidized bed reactors. The team members are TDA, Norton Chemical Process Products Corporation (Norton), and the Institute of Gas Technology (IGT). TDA defines the sorbents to be evaluated and conducts multiple cycle tests in our fixed bed reactors. Norton produces the sorbent by techniques representative of commercial production. IGT tested our sorbents for fluidized bed and/or transport reactor systems. General Electric will also be a subcontractor to evaluate our sorbents for moving bed applications.

TDA prepared small quantities of sorbents with many different compositions in order to identify the preferred firing conditions and starting materials. First we screened our raw materials by producing small batches of either chemical sorbent only or inert support only. Several sources of ZnO and titania were evaluated and tested to determine their crush strength, porosity, and surface area. From this matrix, we selected one source of zinc oxide and one of titania. To minimize the loss of surface area in the sorbent, we required that the sorbent be fired at a temperatures of 900°C (1652°F) or less.

We then produced sorbents with varying ZnO contents. We exposed these samples to 1% H₂S using simulated gasifier gases. We measured the sulfur content by chemical analysis after the first absorption and eliminated those compositions that had low sulfur loadings. We prepared a larger quantity of different sorbents and cycled them through several absorption and regeneration

cycles. We measured the sulfur content, pore volume, and crush strength of the initial material, and then remeasured these properties after absorptions after regenerations.

Norton Chemical Process Products Corporation (Norton) Based upon the test results we selected formulations for further evaluation. We then described the sorbent preparation methods to Norton. Norton is one of the world's leading producers of ceramic products and catalyst supports. Once under contract with TDA, Norton prepared larger batches of sorbents. Norton prepared >1 kg samples of the most promising formulations using techniques that are representative of commercial practice. After analysis of the Norton materials, we then tested formulations for multiple cycles measuring the breakthrough with the sorbent.

IGT Measurement of Attrition Resistance for Fluidized Bed Reactors A total of three zinc-oxide based sorbents were provided by TDA for testing in the attrition test unit to determine the relative attrition resistance of the sorbents. The attrition test unit at IGT was used to measure the relative resistance of the sorbents to attrition. In a unit of this type, the sorbent is exposed to a high velocity air jet for a specified period of time and the change in particle size distribution is measured. The unit consists of a 2.54-cm ID by 152-cm tall glass tube with a single 0.38-mm orifice gas inlet. During an attrition test the air flow is set to generate sonic velocity through the orifice, thus creating turbulence and a strong shearing effect to provide measurable attrition between the particles. These effects simulate attrition in commercial fluid-bed reactors over a much longer period of time.

To compare the attrition resistance of TDA's sorbents with that of other known material, IGT also conducted attrition tests with a sample of UCI 4169 zinc titanate sorbent as well as a limestone and a dolomite. This UCI sorbent was tested at Enviropower's pilot unit and was shown to have acceptable resistance to attrition during the pilot test. Therefore, it was decided to use this sorbent as the benchmark to evaluate the attrition resistance of other sorbents.

The results of the attrition tests are shown in Figure 3, indicating that all three TDA sorbents are more attrition resistant than the UCI's 4169 zinc titanate sorbent, which was tested by the Enviropower in the pilot scale unit. Furthermore, the attrition resistance of the sorbent 40ZN appears to be better than the limestone and the dolomite samples tested at the same operating condition. Based on the results of the attrition tests, the sorbent 40ZN was selected by TDA for sulfur reactivity test in a fluidized bed reactor and the same sorbent was tested at TDA in their fixed bed reactor.

Results

Moving Bed Reactor

TDA prepared sorbents and evaluated them for moving bed applications. Last year (Copeland *et al.* 1995) we reported on a spalling resistant sorbent tested for 20 cycles. This year we have improved our sorbent formulation. Over 60 formulations have been screened, non-promising sorbents (defined as having low reactivity, strength or attrition resistance) being eliminated. We have improved our attrition resistance to better than 98.5% while retaining high chemical activity

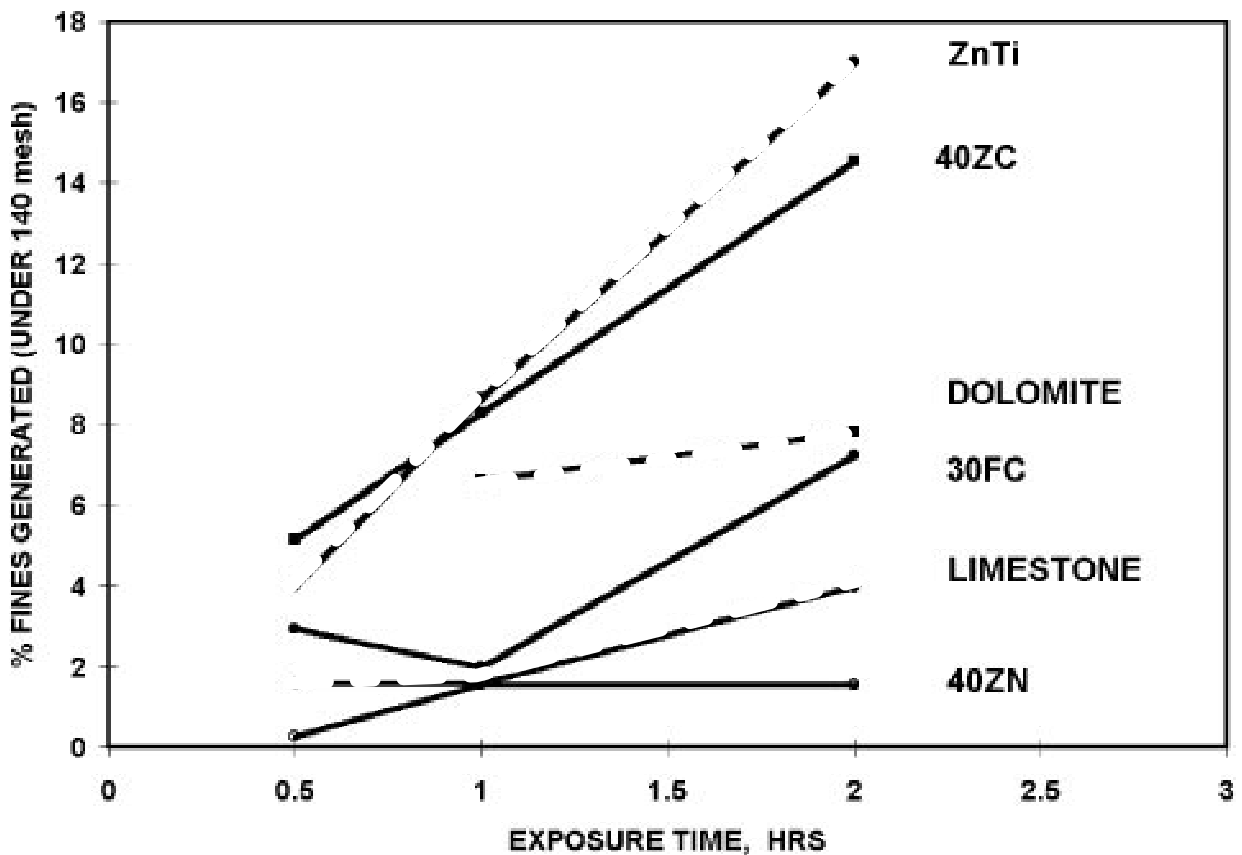


Figure 3 Attrition resistance of various sorbents.

and sulfur loadings.

Figure 4 presents the breakthrough of one of our latest sorbents with the addition of an improved (quartz liner) reactor. In this case, the sorbent removes the H_2S to less than 10 ppmv for several hours before breakthrough occurs. Since we are in the early stages of testing, additional cycles are currently being conducted.

The breakthrough in Figure 4 was conducted at 490°C and 11 atm in 1% H_2S and simulated Texaco gasifier gases. This sorbent, identified as ZVT12LN, has an initial crush strength of 14.8 lb/mm, porosity of 38% void, surface area of 1.3 m^2/g , an attrition resistance of >98.8% and a bulk density of 1.75 g/cc (109 lb/ft³).

Table I presents the crush strength and sulfur capacity of several other sorbents currently being evaluated by TDA. Several different formulations were prepared and fired at different temperatures. Some of the series (*e.g.*, the ZZVTs) had high strength but relatively low sulfur capacities. Others high sulfur but relatively low strength. The ZZVS2 and ZAF11 had both high sulfur and high strengths. TDA is conducting additional cycling tests on these materials and will select one sorbent for 50 complete absorption/regeneration cycles.

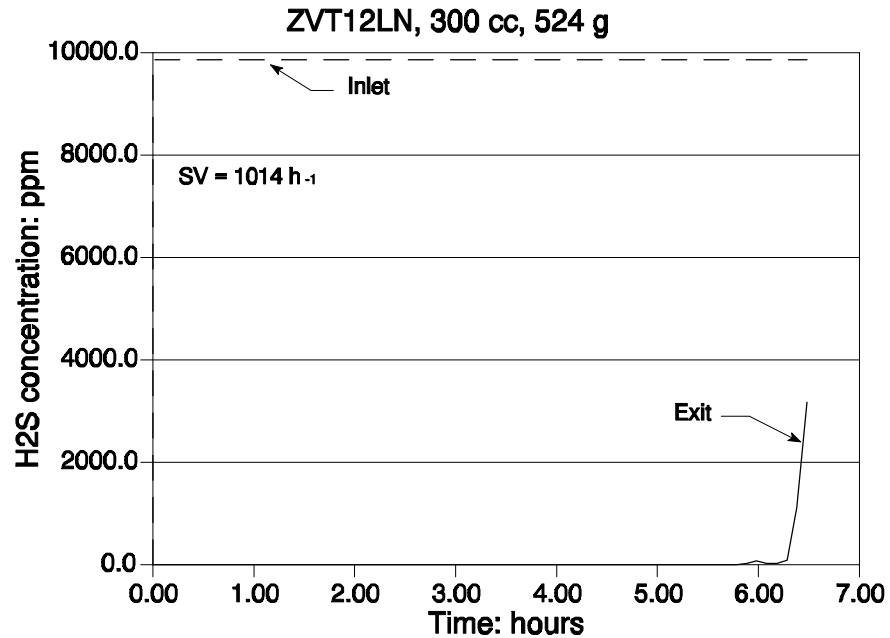


Figure 4. Cycling Of high strength/high capacity sorbent.

Table I Sulfur capacity sorbents for moving bed reactors at 482°C (900°F).

Sorbent	Crush strength fresh	Crush strength after regeneration	Sulfur loading lb/100 lb sorbent
ZZVT5	8.3	8.0	6.3
ZZVT6	10.6	8.4	4.5
ZZVS2	6.5	5.4	10.8
ZZVS3	6.2	5.5	8.5
ZZVS5	6.8	5.9	8.8
ZAF9	3.5	3.5	11.7
ZAF10	3.5	3.3	12.4
ZAF11	6.1	6.3	10.7

Fluidized Bed and Transport Reactors

Breakthrough Test Results Each of the absorption half cycles was conducted with simulated fluidized bed gasifier gases containing 1% H₂S entering the reactor at 538°C (1,000°F). The breakthrough data were measured until the exit gas stream exceeded 500 ppmv H₂S. Breakthrough cycles were carried out as follows:

- 40ZN 20 cycles in a fixed bed at TDA
- 40ZN 5 cycles in a fluidized bed at IGT

During the regenerations we raised the sorbent bed to 600°C and flowed diluted air for about 8 hours to oxidize the ZnS to ZnO. We then increased the O₂ concentration in stages, finally flowing only air through the reactor at 600°C to 650°C (all 40ZN tests). SO₂ was never added to the inlet air throughout any of these regenerations, which simulates the operating conditions of fluidized bed and transport reactors.

Table II presents the measured properties of the 40ZN sorbent before and after a 20 cycle test in a fixed bed reactor. There is very little change in the characteristics of the sorbent over the 20 cycles. The crush strength increased slightly but the porosity and surface area decreased. The bulk density was increased and some sulfur remained at the end of the 20th regeneration, probably in the form of zinc sulfate. Perhaps most importantly, we measured the mass of the sorbent added to and removed from the reactor after the tests.

Table II Properties of fresh and cycled 40ZN.

Property	Fresh	20 cycles *
Crush Strength	9.6 lb/mm	10.4 lb/mm
Pore Volume	0.22 ml/g	0.20 ml/g
Bulk Density	1.35 g/cc 84.2 lb/ft ³	1.47 g/cc 91.8 lb/ft ³
Surface Area	2.0 m ² /g	1.26 m ² /g
Sulfur (wt.)	0.00%	1.69% **

* After 20th Regeneration

** Equivalent to a 3.9% weight gain, if all sulfur is sulfate, indicating a net 2.2% loss in 20 cycles or 0.11%/cycle.

Figure 5 presents the breakthrough characteristics for 40ZN through 20 cycles. The sorbent removes H₂S effectively, a level less than 10 ppm(v) H₂S measured in our tests prior to breakthrough. The breakthrough data also show that the sorbent remained active throughout the 20 cycles. Unfortunately, the data for the first

five cycles were lost which was due to experimental difficulties including problems with the mass flow controllers, which showed some times registered high and at others low. IGT also measured the breakthrough for the first five cycles on the same sorbent (see Figure 7) and showed no significant change in reactivity for the first cycles.

Figure 6 shows the sulfur capacity of the 40ZN, cycles 6-20. The data show some scatter in the breakthrough time. Another problem was found after we received data on the sulfur content of the regenerated sorbent after cycle 10. Those data showed that the sorbent was not being completely regenerated. Thus, some of the loss in the sulfur capacity may be due to incomplete regeneration in the fixed bed reactor (flowing gases for a fluidized bed reactor).

Figure 6 also presents data on the sulfur capacity of Z-SORB as a function of cycling. These data were measured by Gangwal *et al.* (1994) in a fluidized bed reactor and show a continuing loss of capacity for more than 20 cycles. Gangwal *et al.* measured the data for 50 cycles and the

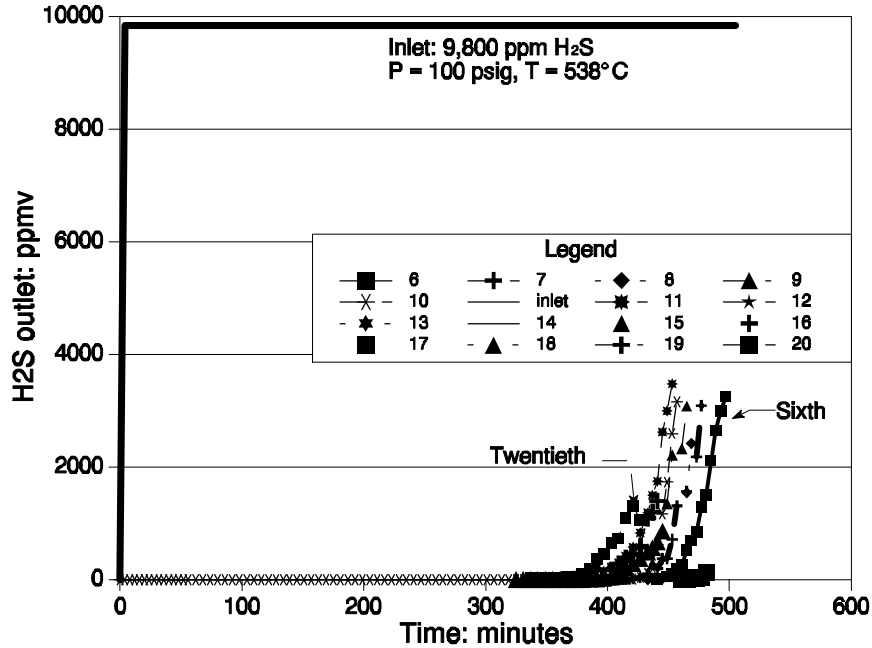


Figure 5 Cycling of 40ZN.

Z-SORB sorbent had been reduced to about half of its initial capacity at the end of their testing.

Sulfur Reactivity Tests at IGT The cyclic sulfidation/regeneration tests were conducted with the 40ZN sorbent over five cycles in the high-pressure/high-temperature reactor (HPTR) unit in the fluidized-bed mode of operation. This unit includes simulated hot coal-derived gas feed systems, an absorption/regeneration reactor, and associated process instrumentation and control devices. This bench-scale unit at IGT has been used for over hundreds of tests with excellent reproducibility and reliability. The gas feed system is capable of delivering simulated gas mixtures with different compositions representing various gasifiers containing CO, CO₂, H₂, H₂O, H₂S, N₂ and CH₄.

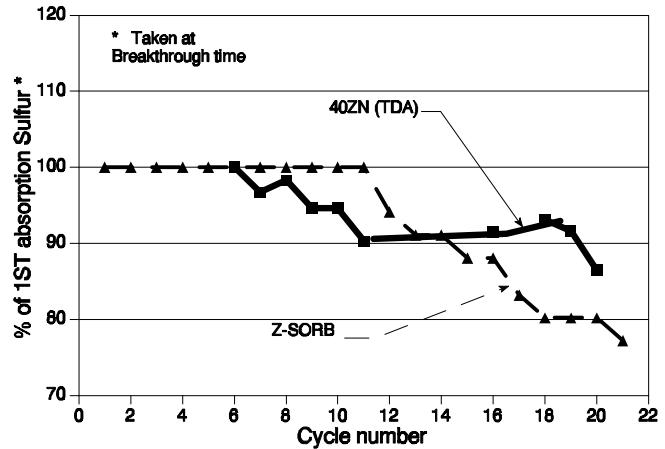


Figure 6 Sulfur capacity of 40ZN and Z-Sorb with cycling.

The H₂S breakthrough concentrations as a function of sulfur loading for the five sulfidation cycles are shown in Figure 7. The results indicate that the reactivity of the 40ZN sorbent did not decrease during the course of the five-cycle test. It should be emphasized that in order to test the sorbent and determine the effect of cycling on the sorbent reactivity, the experiments were conducted in a very short fluidized bed (and gas residence time), making the H₂S breakthrough concentration very sensitive to the reactivity of the sorbent. Therefore, in actual process, where

the gas residence time is expected to be several seconds (compared to about 0.5 seconds in these experiments), the H₂S breakthrough concentration will be very low.

In an earlier work performed by IGT for Enviropower Inc., the zinc titanate sorbent (ZnTi) selected for the pilot-scale testing was evaluated under similar operating conditions. Comparison of the results indicate that the initial reactivity of TDA's 40ZN is slightly lower than UCI's 4169. Although the reactivity of the latter has been shown to decrease gradually by about 50% in 30 cycles, mathematical simulation at IGT and pilot plant testing at Enviropower have shown that the reactivity of the UCI's 4169, after 30 cycles, is sufficient to reduce the H₂S content of the coal gas to below 20 ppmv.

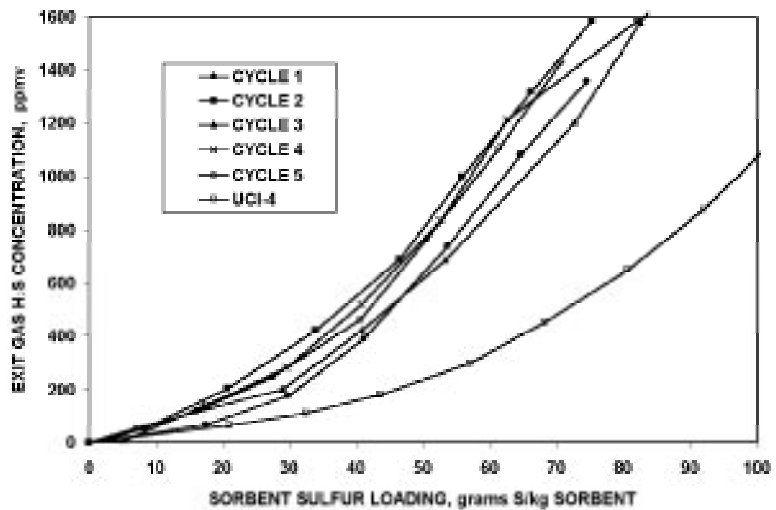


Figure 7 Reactivity of the 40ZN sorbent in successive cycles.

Given the excellent attrition resistance of the 40ZN, it is logical to assume that the rate of deterioration of this sorbent (if any), should be lower than that of other zinc titanate sorbents tested. Therefore, after a large number of cycles, the reactivity of the 40ZN is expected to be higher than the other zinc titanate sorbents, making it suitable for hot gas desulfurization in the IGCC process.

Application

We compared our sorbent to other sorbents currently under development for hot gas cleanup. The results for the moving bed application were presented last year (Copeland *et. al.* 1995) and showed significant advantages for our sorbents.

We compared our fluidized bed/transport reactor sorbents to other sorbents currently under development for hot gas cleanup. Table III presents a comparison between the 40ZN and Z-Sorb B, produced by the Phillips Petroleum Corporation. Our sorbents are more dense and have a similar, though lower, sulfur capacity.

The attrition resistance of 40ZN is very good. The data from IGT show loss rates of only 1% per hour under test conditions where zinc titanate lost 8% per hour. Z-Sorb, when tested under fluidized bed conditions (Gangwall *et al.* 1994), lost only 0.5% in 50 cycles, which is similar to zinc titanate (less than 2% in 100 cycles with a measured attrition resistance of 8%/h) (Gangwal *et al.* 1995). Thus, we can anticipate that our higher attrition resistance will lead to a sorbent with a longer life. Although an improvement factor of 8 is indicated by the available attrition

data, we conservatively estimate an **Table III** Comparison of ZnO based improvement factor of 3 in the life of the sorbents. sorbent.

Although the sulfur loadings of both TDA's sorbent and Z-Sorb are similar, only a fraction of the sulfur capacity is utilized in a transport reactor. The transport reactor at Piñon Pine is designed to never fully regenerate the sorbent and a significant fraction of the sorbent remains in the sulfided form when it leaves the regenerator. This design eliminates any formation of sulfate, due to the very low equilibrium oxygen pressure. The reactor design also limits the quantity of sulfur that is removed per cycle to about 3 lbs of sulfur per 100 lbs of fresh sorbent, regardless of the total sulfur loading in the sorbent. Although TDA's sorbents have lower sulfur loading, the greatly higher attrition resistance improves our economic potential substantially, since Sierra Pacific reports that attrition loss is one of the major concerns with Z-Sorb in their plant.

	40ZN TDA	Z-SORB B Phillips
Sulfur Capacity (lb S/100 lb fresh)	12	10.6-16.2
lb sorbent/ft ³ of Bed	84	60

The improvement in sorbent replacement costs, calculated from the available data, is shown in Table IV. The data on Z-Sorb was taken from the letter from Sierra Pacific; *i.e.*, a cost of \$8/lb for Z-Sorb and 10 lbs of sulfur removed per lb of sulfur loss (although we have other information from Kellogg that indicates that Z-Sorb will not meet this goal). As can be seen by our estimates, the cost/performance for 40ZN is significantly less than other systems developed to date, and can meet or exceed the cost goals set for hot gas cleanup systems. As we are still in the early stages of our development, we anticipate the cost/performance of our sorbents to further improved with future work.

Future Activities

TDA produced several different sorbent formulations with different zinc oxide contents, and different strengths and pore volumes. One sorbent formulation was selected for multiple cycle tests, and the measured properties of the sorbent improved slightly over the 20 cycle test. The initial selection was a sorbent that has a high sulfur loading capacity. The sorbent is resistant to spalling and retained its reactivity, porosity, and crush strength with cycling.

TDA compared the performance of our sorbents to that of other sorbents. We found that our sorbents have a higher sulfur content per unit volume and similar costs to other hot gas cleanup sorbents. However, additional work will be needed to develop and demonstrate that these improvements can be achieved on a larger scale and for more cycles.

We will be improving the properties of our sorbent. A number of multiple cycle breakthrough tests will be conducted to determine the impact of cycling on different methods of forming the sorbent. Finally, after an improved sorbent formulation has been developed and proven, a revised

Table IV Sorbent replacement costs for zinc oxide based sorbents.

Cost Element	40ZN	Z-Sorb
Sulfur removal rate	3 lbs sulfur removed per 100 lbs of fresh sorbent per cycle	3 lbs sulfur removed per 100 lbs of fresh sorbent per cycle
Loss Rate:	1% per hour of fluidization (about 30 lbs sulfur/lb sorbent consumed)	10 lbs sulfur/lb sorbent (goal, currently less than this value)
Cost: \$/ton sorbent	≈ 16,000*	16,000**
Cost of Sorbent Replacement	≈ \$600 per ton sulfur	> \$1,800 per ton sulfur

* at \$8/lb, estimated by Norton for small production lots.

** Cost data from Sierra Pacific.

production cost analysis will be conducted and the economics of hot gas cleanup calculated and compared to that of other sorbents.

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Contract Information

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