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Simultaneous Hot Desulfurization and Improved Filtration in Coal Utilization Processes

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PB.8 Simultaneous Hot Desulfurization and Improved Filtration in Coal Utilization Processes

CONTRACT INFORMATION

Contract Number DE-FG02-94ER81786

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Period of Performance August 15, 1994 to March 10, 1995

Schedule and Milestones

Program Schedule

	1994 J F M A M J J A S O N D	1995 J F M A M J J A S O N D
Procurement	***	
Equipment Shakedown	***	
SO₂ Testing	****	
H₂S Testing	****	
Phase 2 Submittal / Final Report Preparation	*****	

OBJECTIVES

The primary objective of this Phase I SBIR effort was to investigate the relative effectiveness which various waste metal oxides might have in the removal of sulfur from sulfur

dioxide (SO₂) and hydrogen sulfide (H₂S) gas streams, under controlled laboratory conditions. A number of waste metal oxides were tested both individually and in combination, to assess their capacity for sulfur capture in sulfur laden oxidizing and reducing

atmospheres. Additionally, inert materials such as silica sand, as well as more traditional sorbents, such as dolomite and limestone, were tested to serve as reference data. The Phase I effort also explored the overall domestic availability of the best performing waste metal oxide sorbents, taking into account their geographic distributions, intrinsic value, etc. to provide the groundwork for a low cost, highly effective sulfur sorbent which can be quickly implemented on a commercial scale for such processes as IGCC and PFBC. Finally, in that all of the sorbent samples tested were of an identical, carefully selected particle size, the data collected serve as the basis for a proposed Phase II effort, which will further utilize the waste metal sorbents as a means of increasing the permeability of the dust collected on downstream particulate control devices, such as ceramic filters.

BACKGROUND INFORMATION

Much of America's vast coal reserves will remain unusable until technology capable of the effective, economical removal of particulate and sulfur emissions becomes more highly developed. In recent years, ceramic filtration devices have shown that particulate removal levels well within the limits of New Source Performance Standards (NSPS) can be achieved at high process temperatures, thereby enhancing the overall performance and efficiency of processes such as Integrated Gasification Combined Cycle (IGCC) and Pressurized Fluidized Bed Combustion (PFBC). Unfortunately, removal of the high levels of sulfur indigenous to much of our Nation's coal reserves has been problematic, requiring exotic (and expensive) sorbents, substantial coal feed pretreatment, or a reduction in off-gas temperature to achieve acceptable sulfur emissions.

Many researchers are aware that metal oxide sorbents can be used to effectively reduce sulfur emissions in such processes. However, because of the high cost of most metal oxides, especially such materials as zinc titanate and zinc ferrite, economic studies have shown that for such materials to be cost effective they must be used and regenerated as much as several hundred times¹ before a return on investment is realized. Unfortunately, preliminary testing of such sorbents in a pelletized bed configuration, which lends itself to such a regenerative process, have shown that the strength of the pelletized metal sorbent significantly decreases in as few as five or six regenerative cycles. In view of this, it is apparent that a "once through", non-regenerable metal oxide sorbent can be a much more attractive solution from the standpoints of capacity, overall sulfur removal efficiency, and process economics, particularly if a low cost (or no cost) source of sorbent is readily available. Moreover, in combination with ceramic filtration technology, the use of such an external sorbent becomes even more practical in that the sorbent can be introduced well upstream of the candle filter, have sufficient residence time for sulfur removal to take place, and then be easily collected on the surface of the ceramic filter, along with the ash and other particulate. By properly classifying and sizing the waste metal oxide sorbent particles, the permeability of the dust accumulating on the filter candles can be readily increased, resulting in lower pressure differentials, better pulse cleaning efficiency, and less susceptibility to candle pore "blinding".

The Phase I SBIR effort reported herein showed that significant sulfur removal capacities can be obtained using classified, spent metal oxides. Such materials are generated in tremendous volume as a result of

metal processing, smelting, and refining operations, and are generally available for little if any cost. Among the spent metal oxides tested in Phase I which exhibited a strong affinity for sulfur capture in oxidizing and reducing atmospheres were the waste oxides of tin, zinc, and iron, some of which had significantly higher capacity than a mixture of dolomite and limestone.

Phase II testing will concentrate on these three promising candidate waste materials, to verify their sorbent capacity using thermogravimetric analysis (TGA) and to determine their additional capability as a high temperature filter aid, after classifying to a specific particle size distribution. Ultimately, when utilized as a "once-through" sorbent in combination with ceramic filtration technology, these spent metal oxides will provide sulfur removal and enhanced filter performance in a cost-effective manner.

PROJECT DESCRIPTION

The Phase I research program involved the testing and assessing of the sulfur removal capacity of waste metal oxides, under controlled laboratory conditions, by exposing a prepared bed of each waste metal oxide to sulfur dioxide (SO₂) and hydrogen sulfide (H₂S), in separate trials. The testing was performed at the Research & Development Department of Industrial Filter & Pump Mfg. Co., Inc. The waste metal oxides tested in Phase I included:

- A) Iron oxide dust from a Basic Oxygen Furnace (B.O.F.) application;
- B) Zinc oxide dust from a Chicago area zinc smelting operation;
- C) Zinc oxide dust from a 2nd Chicago

area zinc smelting operation;

- D) Tin oxide dust from a Chicago area tin refiner;
- E) Zinc/iron/lead oxide mixture from another local smelter.

In addition to the testing of the above waste metal oxides, test trials were performed using:

- F) Nitrogen carrier gas only, without any SO₂ (or H₂S) gas or test sample;
- G) Nitrogen carrier gas and SO₂ (or H₂S) only, without a test sample;
- H) Nitrogen carrier gas and SO₂ (or H₂S) with an inert (silica sand) test sample;
- I) Nitrogen carrier gas and SO₂ (or H₂S) with a moderately inert (diatomaceous earth) test sample;
- J) Nitrogen carrier gas and SO₂ (or H₂S) with a calcium laden waste material from a cement kiln process as a test sample;
- K) Nitrogen carrier gas and SO₂ (or H₂S) with a calcium laden lime waste from a sugar refining operation as a test sample;
- L) Nitrogen carrier gas and SO₂ (or H₂S) with a production grade dolomite/limestone combination as a test sample.

In each of the above test scenarios, the flowrate of nitrogen carrier gas used as a diluent to the SO₂ (or H₂S) was held constant at 83 ml./min. Likewise, the SO₂ or H₂S used

in the testing was maintained at a constant flowrate of 38 ml./min. Test sample volumes were also held constant at 50 cm³, and the test temperature was maintained at 70°F in all cases. By controlling the rate of gas flow through the closed system across the 50 cm³ test sample bed in a downflow fashion, then bubbling the effluent through a 500 ml. volume of analyzed collection water, a depression in the pH of the collection water would occur when the test sample bed no longer removed sulfur from the sulfur laden gas stream. By maintaining a constant system pressure from test to test, and monitoring the elapsed time required to depress the pH of the collection water to a predetermined end point, the relative sulfur removing capacity of each material tested could be observed for the test conditions cited above.

The initial test apparatus, shown in Figure 1, was used but modified to eliminate the inert dust bed and its corresponding collection water beaker, since it was felt that equivalent flow through two material beds simultaneously at the low flowrate envisioned would be virtually impossible.

Initial test trials involved only nitrogen carrier gas, without a test sample bed, and were conducted to determine the effect which pure nitrogen might have on the pH of the collection water, if any. It should be pointed out that carefully analyzed tap water was selected in favor of either demineralized or distilled water for use in the collection beaker, as the pH of these latter materials was found to be altogether too sensitive to external influences (such as CO₂ and other gases in the atmosphere surrounding the collection beaker, which form carbonic acid that causes pH depression).

Following the initial tests, trials involving nitrogen carrier gas and either SO₂ or H₂S, again without a test sample bed, were conducted to determine the time delay (due to piping lengths and volumes) from the onset of the test until pH depression.

Both the effect on pH of the nitrogen carrier gas as well as the "time delay" associated with the internal volume of the test apparatus were used in the final data analysis.

All tests involving sample test beds were conducted in an identical fashion. Test samples were obtained from local metal processors, and were prepared by drying at 250°F for 4 hours in an air-circulated oven, after which time the weight of the sample stabilized, indicating the removal of any moisture present. The material then underwent particle size classification using a CE Tyler Sieve Products, Inc. portable sieve shaker. Each sample material was classified and the particle fraction passing through a 140 mesh (106 micron opening) screen which was retained on a 200 mesh (75 micron opening) screen was utilized for each test specimen. This particle size is representative of that which would be used to enhance filter cake porosity in the eventual and effective use of U. S. Patent #4,865,629, which is the focus of Phase II of this research effort. By using an identical particle size fraction for each sample tested, it was hoped that the bed permeability in each sample trial would be nearly equal at constant gas flowrate conditions; also, the total available surface area exposed to the sulfur laden gas would be similar from one test to the next. Just prior to each test, the sample material was again dried in an air circulated oven to insure no moisture was retained, after which time 50 cm³ of the classified sample was extracted and weighed for test purposes.

A total of five test trials involving the aforementioned test samples were performed. In evaluating the results of each of the first three sets of trials, a great deal of inconsistency was observed and it was concluded that intermittent leakage within the test apparatus was the cause of the inconsistencies in the results. The test apparatus was modified with high pressure tubing and connectors, and a "leak check" procedure, involving the pressurizing and monitoring of the apparatus static pressure, was performed prior to the actual testing of each sample in the last two test series, to insure that the results were highly accurate.

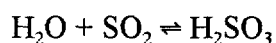
Because of the above, only the results corresponding to trials #4 and #5 were deemed accurate and are discussed in this report, to eliminate confusion with earlier inconsistent results.

RESULTS

SO₂ Trials

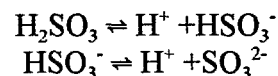
In the tests which involved the capture of sulfur from an SO₂ laden gas stream at ambient temperature, the following findings were observed:

- A) Sulfur dioxide is known to dissolve in water to create a weakly acidic solution of sulfurous acid, according to the reaction below:



Sulfurous acid is unstable and cannot be isolated in the anhydrous condition; all of the sulfur dioxide formed as the acid decomposes is expelled from solution by boiling. Like other diprotic acids, sulfurous acid ionizes in two

steps:



The amount of ionization is slight in both stages, but it is much less in the secondary stage than in the primary. Specifically, the ionization constants at ambient conditions for each of the above equilibrium reactions are:

$$\begin{aligned} K_1 (\text{H}_2\text{SO}_3) &= 1.2 \times 10^{-2} \text{ moles/liter} \\ K_2 (\text{HSO}_3^-) &= 6.2 \times 10^{-8} \text{ moles/liter} \end{aligned}$$

Hence, although sulfurous acid is by definition a weak acid, it will readily depress the pH of tap water. For this reason, in the tests involving SO₂, only pH was monitored as a means of evaluating the total sulfur removal capacity of each material tested. Based on preliminary tests, an end-point pH of 5.00 was chosen for the SO₂ test series.

- B) Of the various materials tested using SO₂ gas, tin oxide appeared to have a significant potential for sulfur removal. The parameters and breakthrough curve for the tin oxide/SO₂ test are shown in Figure 2; for comparison, the dolomite/SO₂ test results are shown in Figure 3.

H₂S Trials

In the tests which involved the capture of sulfur from a gas stream containing H₂S at ambient temperature, the following findings were observed:

- A) Hydrogen sulfide, a diprotic acid, is known to ionize in water through two

steps, the first of which yields hydrogen and hydrosulfide ions, as shown below:



The expression for the ionization constant for this primary ionization of H_2S is:

$$\frac{[\text{H}^+][\text{HS}^-]}{[\text{H}_2\text{S}]} = K_{\text{H}_2\text{S}} = 1.0 \times 10^{-7} \text{ moles/liter}$$

The hydrosulfide ion in turn ionizes and forms hydrogen and sulfide ions, according to the following reaction:



The expression for the ionization constant for the hydrosulfide ion is:

$$\frac{[\text{H}^+][\text{S}^{2-}]}{[\text{HS}^-]} = K_{\text{HS}^-} = 1.3 \times 10^{-13} \text{ moles/liter}$$

It was noted that K_{HS^-} is smaller than $K_{\text{H}_2\text{S}}$ by almost 10^6 times, which means that very little of the HS^- formed by the ionization of H_2S actually ionizes to give hydrogen ions and sulfide ions; thus the concentration of H^+ and HS^- are practically equal in a pure aqueous solution of H_2S .

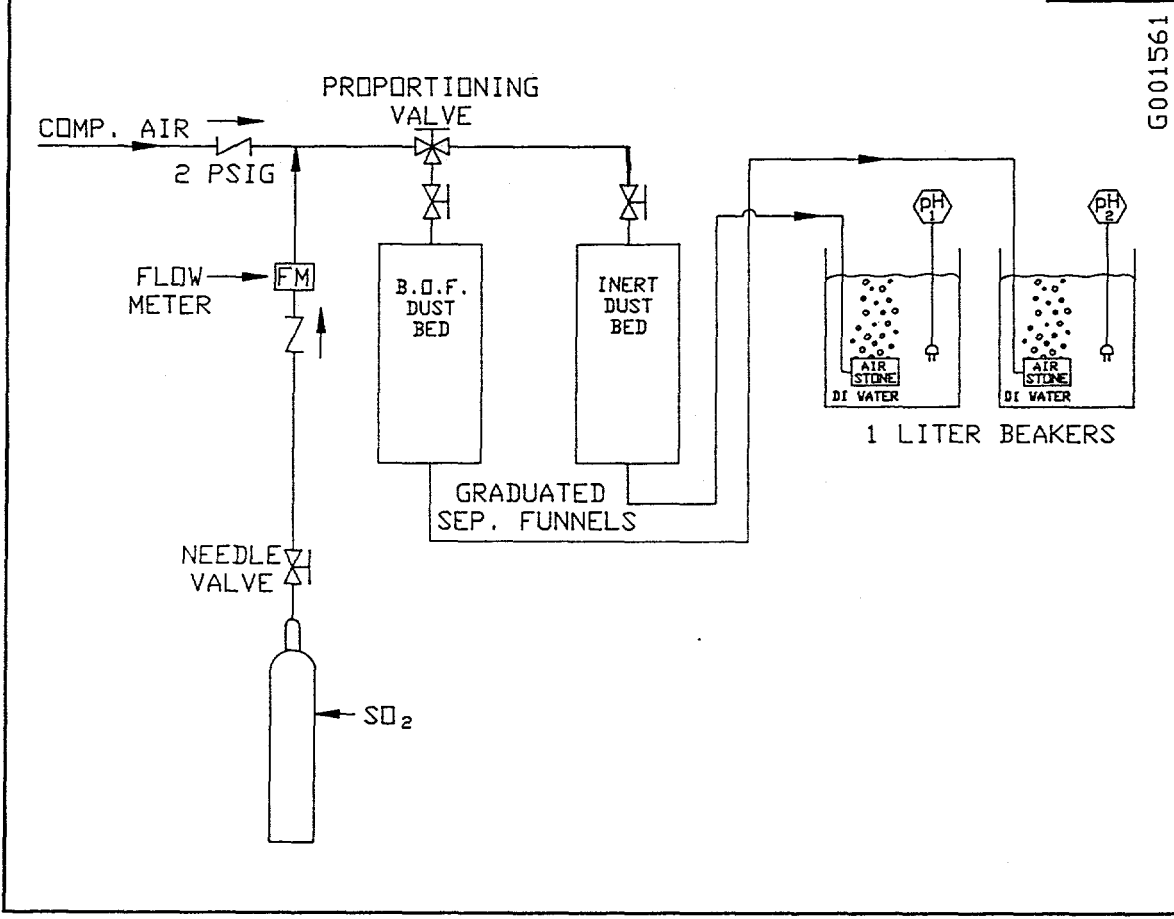
Because H_2S is a weaker acid than SO_2 when dissolved in water, and because tap water (which was purposely selected in favor of distilled or demineralized water) was used in the collection beaker, the pH depression observed upon exhaustion of the sample bed in the H_2S test series would not be nearly as notable as that observed in the SO_2 test series.

Due to the above observation, it was decided that a pH end point value of 6.50 (rather than 5.00) would be used in the H_2S test series. Additionally, total suspended solids (TSS) and total sulfites would be monitored and periodically recorded in the H_2S test series to serve as two other means of validating the data.

- B) Of the materials tested in an H_2S laden atmosphere, zinc oxide and zinc oxide/iron oxide mixtures appear to exhibit the highest affinity for sulfur. Figures 4 and 5 show the parameters and breakthrough curves for these tests; for comparison, the dolomite/ H_2S test results are shown in Figure 6.
- C) It should be pointed out that while copper oxide has been used as a sorbent in flue gas and fuel gas applications with a great deal of success, waste copper oxide dust from copper smelting operations often contains significant amounts of arsenic. For the safety of the research personnel involved and because of the unlikelihood that such a hazardous waste material would ever be utilized for sulfur removal, waste copper oxide materials were not evaluated in the scope of this research.

Waste Metal Oxides: Availability

An additional task in the Phase I undertaking involved the compilation of information² related to the availability, geographical location, and processes which generate the various waste materials tested in this research effort. While such information for each material tested is covered in detail in the Phase I final report, only the three best



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Figure 1. Test Apparatus

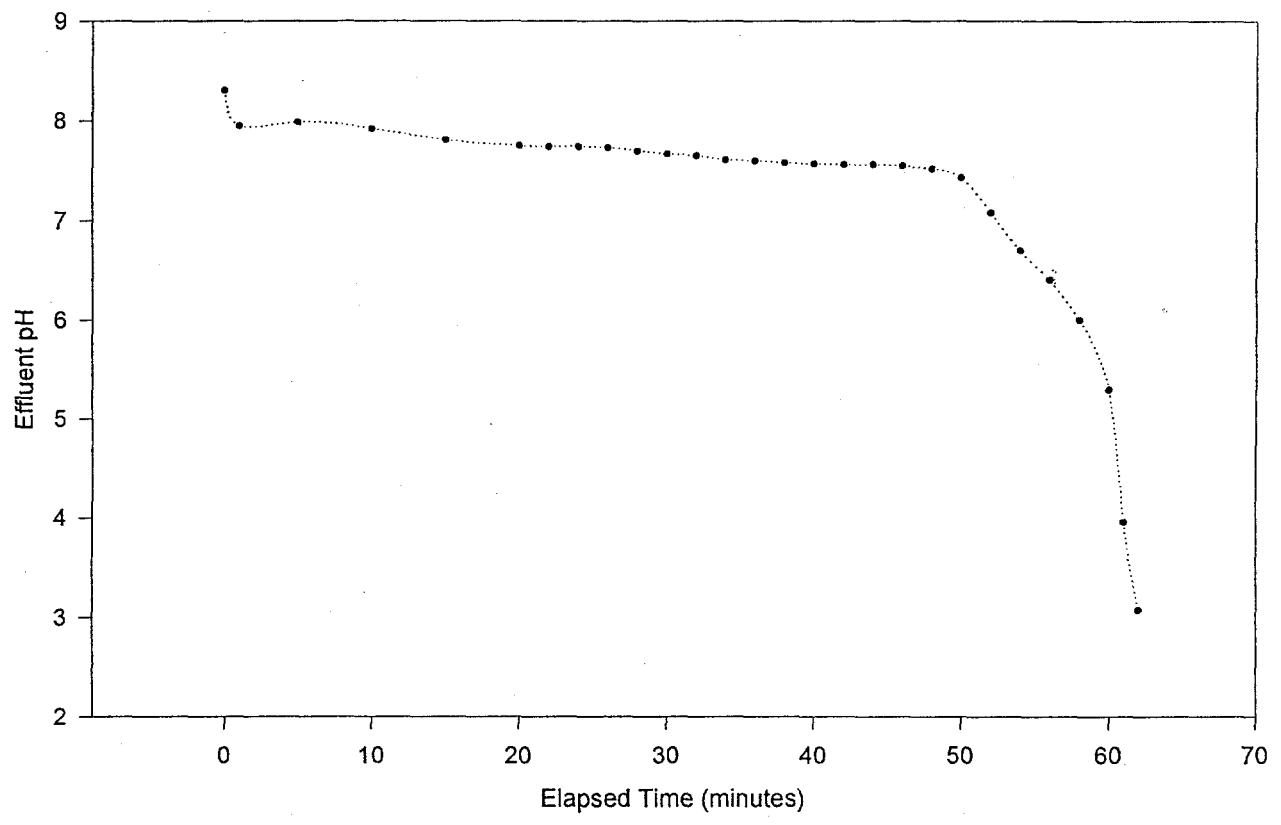


Figure 2. SO₂ Adsorption Using Waste Tin Oxide Sorbent

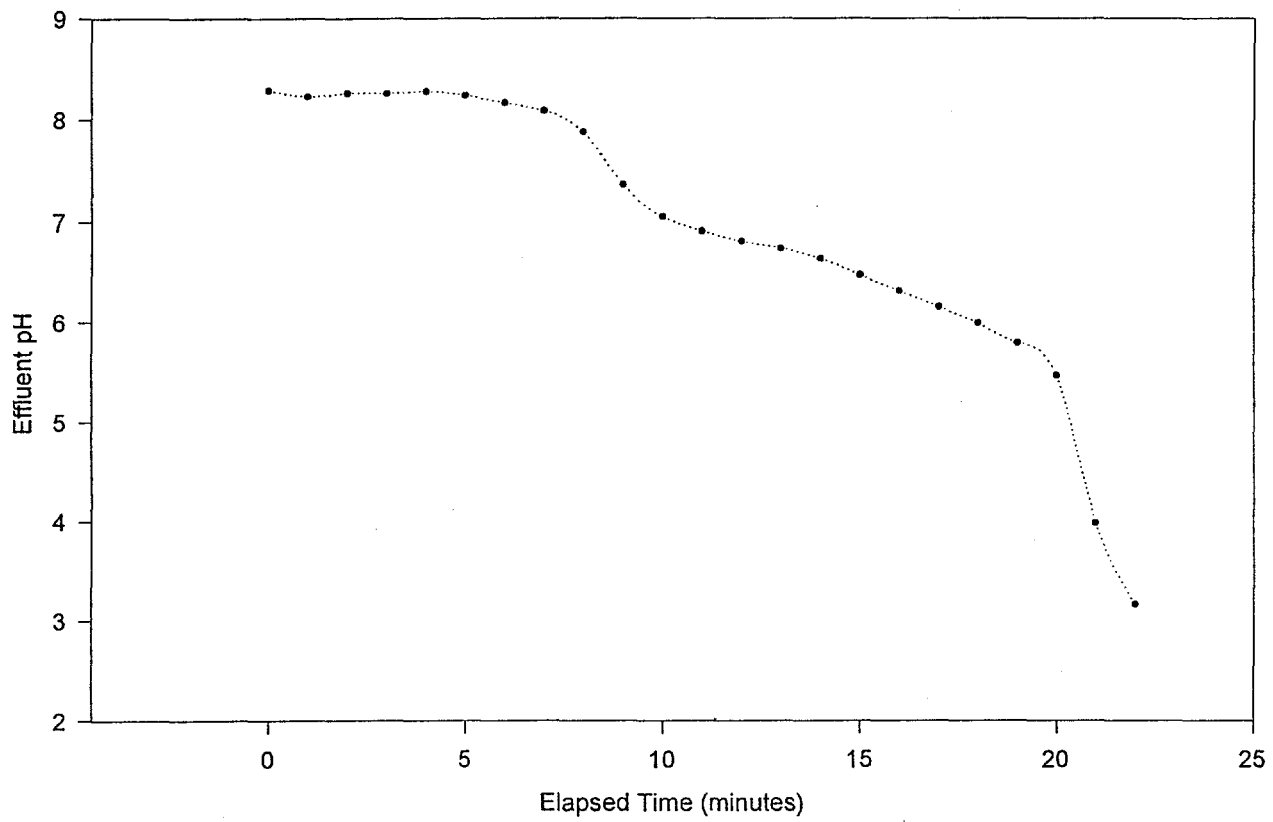


Figure 3. SO₂ Adsorption Using Dolomite/Limestone Sorbent

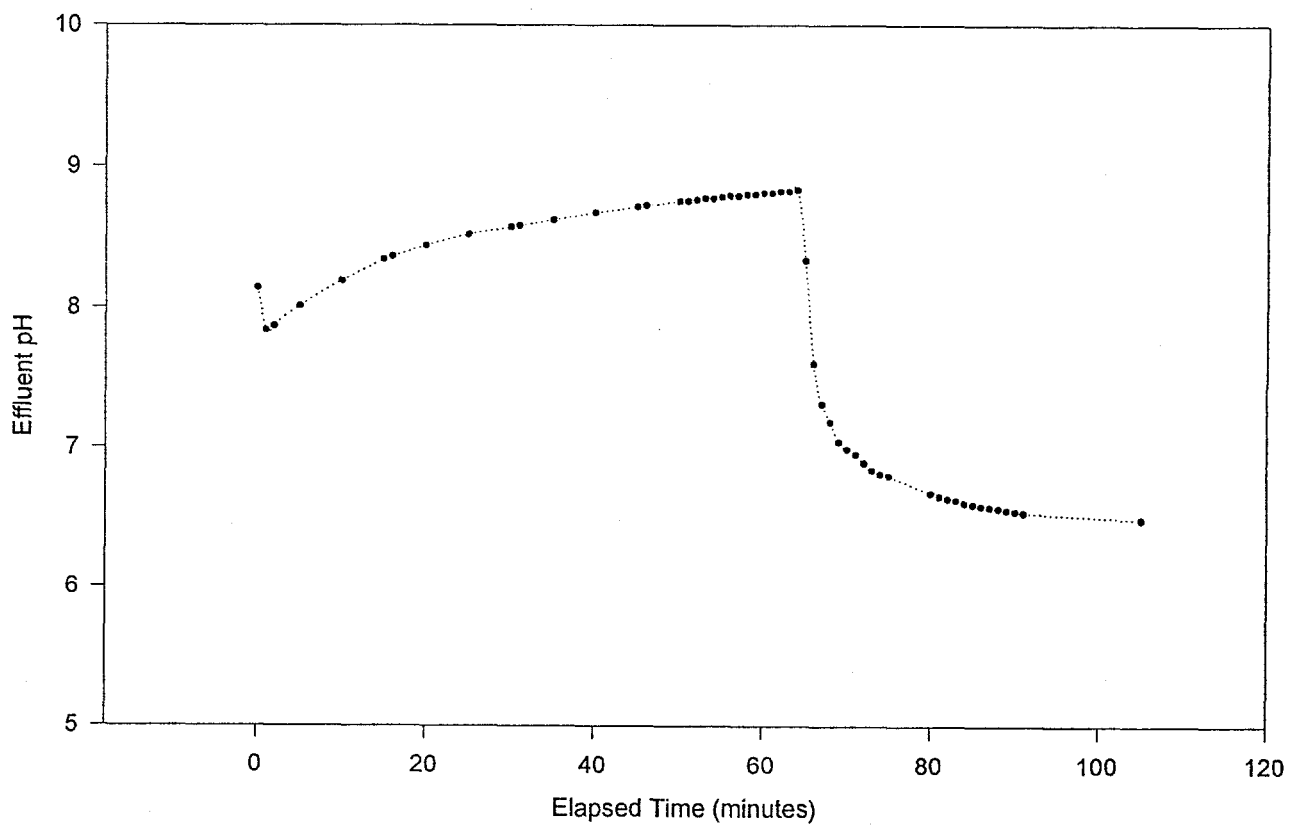


Figure 4. H₂S Adsorption Using Waste Zinc Oxide

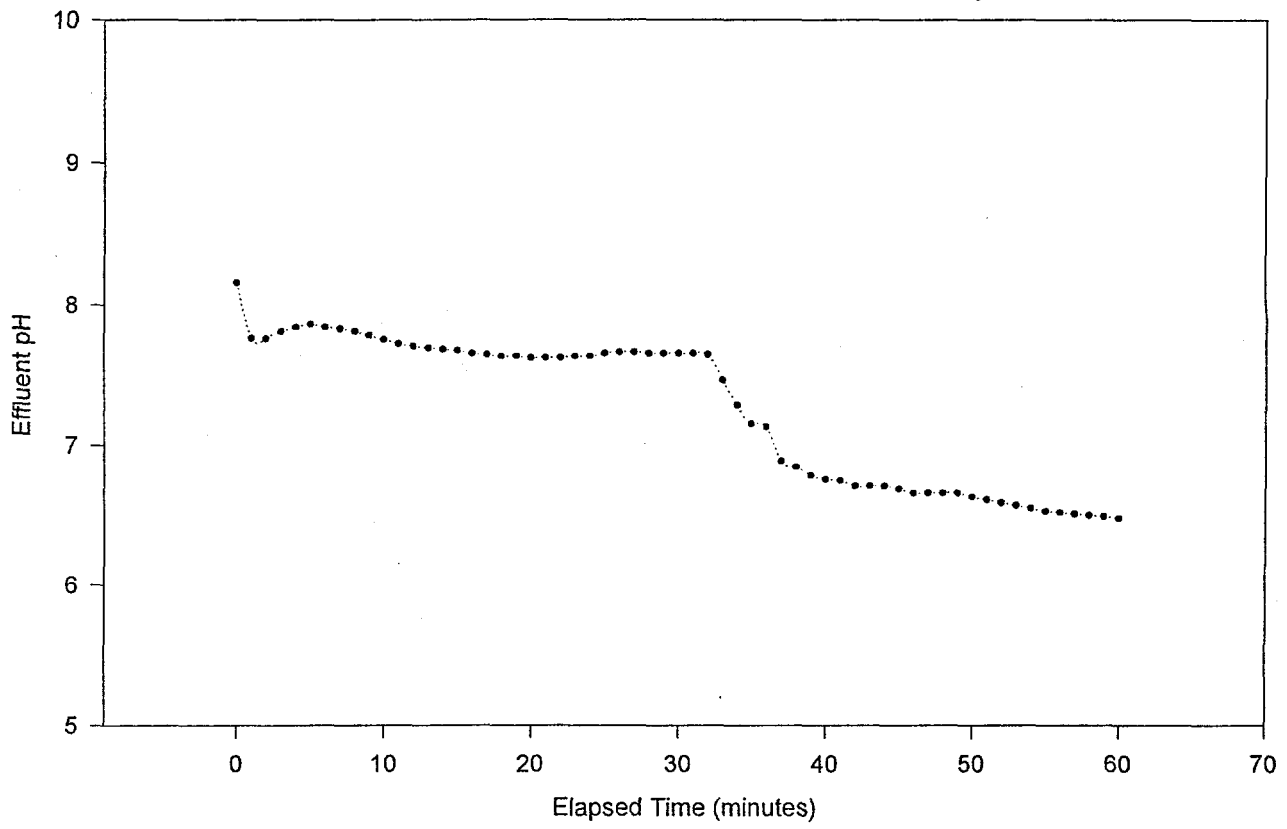


Figure 5. H₂S Adsorption Using Waste Iron Oxide/Zinc Oxide Sorbent Mixture

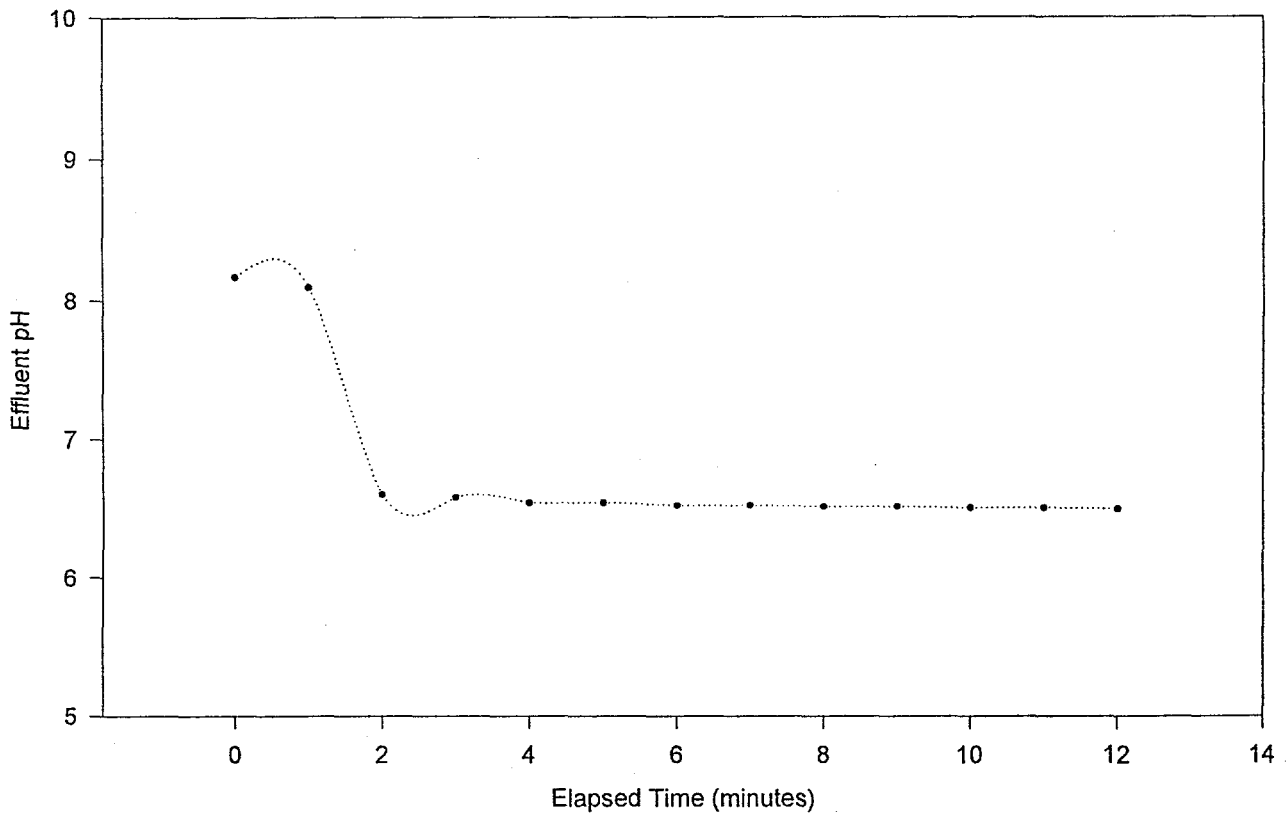


Figure 6. H₂S Adsorption Using Dolomite/Limestone Sorbent

performing materials mentioned in the preceding section, namely the oxides of tin, zinc, and iron, appear to be the most promising; highlights of the availability and intrinsic values for these materials are given below.

Tin

Tin is one of the earliest metals known to humankind, and is commonly used as a protective coating or as an alloying metal with other metals. The major uses for tin are: cans and containers, 32%; electrical, 22%; construction, 10%; transportation, 11%; and other, 25%.

Waste tin oxides are readily available, since most new and old tin scrap is recycled and recovered. In 1991, an estimated 98% of the new tin scrap generated was recovered and an estimated 96% of the old tin scrap generated was recovered.

Detinning facilities are unique in the tin scrap industry, and nine plants are located across the country. Most tin scrap processing facilities are located close to the tin-using industries and to areas of high-density populations. Most are in the Midwest and Northeast. Table 1 gives statistical information on tin.

Zinc

Zinc is the fourth most widely used metal after iron, aluminum, and copper. More than 90% of the metal is used for galvanizing steel and for alloys; the remainder is used to produce dust, oxide, and various chemicals. In 1991, about 354,000 mt of secondary zinc valued at about \$412 million, was recovered in basic forms--refined metal, alloys, dusts, and chemicals. Scrap, containing about 96,000 mt

of zinc and valued at \$62 million, was exported in 1991, whereas 38,000 mt of zinc in scrap, valued at \$19 million, was imported.

Old zinc and brass scrap is collected at hundreds of domestic scrap yards, at more than 200 U. S. automobile and appliance shredding operations, and at numerous municipal collection centers. Tables 2 and 3 cite source and recovery data for zinc.

Iron

Iron is the most widely used of all the metals. Consumption of steel mill products and steel and iron castings in the United States amounts to about 100 Mmt/yr. Each year, over 50 Mmt of scrap iron and steel are recovered and recycled into new products. A major industry exists to collect such a vast amount of scrap and prepare it for recycling. Over 5,000 individual establishments, located throughout the country, purchase steel from individuals and businesses and prepare it to specification for consuming mills and foundries.

Steel production generates a number of wastes that contain ferrous or nonferrous metal residues. The USBM has developed a process to recover nickel and chromium from acid solutions used to pickle (clean) stainless steel, and currently is investigating means to remove and recycle zinc and lead from BOF dust, allowing the remaining iron to be recovered from a waste material that is normally disposed on land. A process to produce high-quality graphite from kish, a common steel plant waste, was recently developed by the USBM and is being introduced at a major steel plant. Also, investigations are underway to recover waste oils from steel plant sludge, allowing the contained iron to be recovered also.

Table 1. Salient Tin Statistics, United States
(Metric tons, tin content)

	1950	1960	1970	1980	1990
Reported consumption	104,464	80,560	73,829	53,362	44,363
Secondary production	33,273	29,030	20,802	12,020	7,747
Secondary production (as percent of consumption)	32	36	28	21	17

Table 2. Salient Zinc Statistics, United States
(Thousand metric tons, zinc content)

	1950	1960	1970	1980	1990
Estimated industrial consumption	1,184	1,051	1,426	1,142	1,240
Secondary production	295	241	308	304	341
Secondary production as percent of consumption	25	23	22	27	28
U. S. scrap trade					
Imports	3	29	2	8	38
Exports	6	11	2	30	109

Table 3. Principal Scrap Source Materials and Form of Recovery
(Metric tons, zinc content)

	1950	1960	1970	1980	1990	1991
Slab zinc	60,166	61,698	68,450	29,396	95,708	124,078
Brass and bronze	146,413	97,452	148,438	172,040	148,247	127,009
Oxide	12,723	16,038	18,312	31,306	34,726	34,394
Dust	24,459	27,346	26,482	35,557	24,105	22,750
Sulfate	4,243	(¹)	8,320	13,195	12,769	23,385
Chloride	11,431	10,881	9,622	10,944	7,436	5,311
Other	36,335	27,733	28,390	11,925	18,409	16,772
TOTAL	295,770	241,148	308,014	304,363	341,400	353,699

¹Included in other.

Waste Metals Oxides: Costs

As a result of the preceding discussions on tin, zinc, and iron oxide, it becomes apparent that extremely large quantities of all three materials are and will continue to be readily available in our Nation. In many large metal processing plants, particle size fractions similar to the range tested in the Phase I effort are immediately available. In instances where such classified particles are not available, the use of "off-the-shelf" bulk handling, particle size reduction, and classification equipment can be easily used to achieve the desired particle size fraction to optimize sorbent capacity and filtration performance.

In view of the availability of all three materials, one further remaining aspect from an economic standpoint (assuming handling costs, permits, and particle size reduction and classification are similar amongst all three materials) is the intrinsic value of each material.

As of April, 1995, the intrinsic value of each of the three base metal materials is as follows:

Tin (composite)	\$3.94/lb.
Zinc (high grade)	\$.54/lb.
Iron (steel)	\$.07/lb.

The above prices, it should be emphasized, are commodity prices for the base metals of each of the candidate waste oxides tested, and do not represent the cost of the corresponding waste metal oxides, which are much less. (In the case of iron oxide dust from BOF processing, the material is essentially free of charge, since on-site storage costs will often exceed the value of the dust). The prices do, however, provide an idea of the value of each material after it has been recycled and

recovered for sale as pure metal, and are indicative of the relative value of each of the three metal oxides tested which showed significant affinity for sulfur.

By contrast, pelletized zinc ferrite or zinc titanate is significantly higher in cost. Even the most novel, fluidized zinc titanate formulations, such as "ZT-4", were reported to be \$7.91/lb., as of June, 1994.³

Given their high affinity for sulfur compared to calcium based sorbents, overall availability, and low cost in relation to other metal oxide sorbents, the select use of waste metal oxides appears to be a most effective means of reducing sulfur in coal utilization applications.

FUTURE WORK

Phase I results showed that waste tin oxide appears to have a very high affinity for sulfur removal in a gas stream containing SO₂ gas at ambient temperature. Similarly, waste zinc oxide, as well as a mixture of zinc oxide and iron oxide wastes, have a high affinity for the removal of sulfur in a gas stream containing H₂S gas at ambient temperature.

The technical objectives of future work in this area have been proposed as a Phase II SBIR effort and include:

- 1) A focus on the development, to commercialization, of several waste metal oxide materials which, with minimal preparation, can be used as effective sorbents for sulfur emissions control in oxidizing and reducing atmospheres, such as those encountered in PFBC and IGCC processes;

- 2) Further evaluation of the effect by which the use of classified waste metal oxide sorbent particles, when introduced as entrained particles into simulated ambient and high temperature gas streams containing sulfur and particulate, will serve to act as filter aids by enhancing dust cake permeability, as described in U. S. Patent No. 4,865,629 awarded to Industrial Filter & Pump Mfg. Co.;
- 3) Following the Phase II test efforts, recommendations will be made for the preparation of and prescribed use of these waste metal oxides for immediate commercial deployment in PFBC and IGCC processes.

As mentioned above, the goal of this project is to combine sorbent utilization with ceramic filter technology. U. S. Patent No. 4,865,629, "Control of Particle Size Distribution in Gas Filtration", assigned to Industrial Filter & Pump Mfg. Co., makes specific use of this concept. The invention relates in general to the filtration of hot gases, but more specifically to a method and apparatus for the efficient filtering of hot gases which are contaminated with a high fraction of fine particulates (such as a combustor or gasifier stream) after several stages of cyclones.

Since a waste source is used for the metal oxide sorbent, as outlined earlier, the cost effectiveness of the sorbent/filter aid concept is very attractive. By utilizing the metal oxide materials directly from the waste source, processing of the material will only include particle size classification (sieve-screening) for application of the metal oxide sorbent as a filter aid. Packaging and transportation of the metal oxide sorbent from its source (e.g., steel

mill) to the utility site would be minimal, if local sources were sought.

In both the private and governmental sectors, waste metal oxide usage, as both a sorbent and high temperature filter aid, has immediate commercial application in any facility where sulfur emission is a concern. Utilities, for example, will benefit from compliance with regulations on emissions, and process performance and efficiency will increase due to the filter aid capability of the sorbent, when injected into the gas stream ahead of a ceramic filter.

Both the private sector and Federal Government will benefit from this project, in that our Nation's prevailing high sulfur content coal reserves will be utilized more fully, in a cost effective manner. Less reliance on other types and sources of our fossil fuels, more effective use of our own resources, and potential for exporting of these resources and technology all translate into increased jobs, productivity, and a cleaner environment for our country.

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