

**FINAL TECHNICAL REPORT**  
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**Project Title: STABILIZATION OF SPENT SORBENTS FROM COAL-BASED  
POWER GENERATION PROCESSES**

DOE Cooperative Agreement Number: DE-FC22-92PC92521(Year 4)  
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**ABSTRACT**

The overall objective of this study was to determine the effect of implementation of the new and more stringent EPA Protocol Test Method involving sulfide containing waste, on the suitability of the oxidized spent sorbents from gasification of high sulfur coals for disposal in landfills, and to determine the optimum operating conditions in a "final" hydrolysis stage for conversion of the residual calcium sulfide in these wastes to materials that are suitable for disposal in landfills. An additional objective was to study the effect of ash on the regeneration and ash-sorbent separation steps in the Spent Sorbent Regeneration Process (SSRP).

To achieve these objectives, a large set of oxidized samples of sulfided calcium-based sorbents (produced in earlier ICCI-funded programs) as well as oxidized samples of gasifier discharge (containing ash and spent sorbent) were tested according to the new EPA test protocol. Samples of the oxidized spent sorbents that did not pass the EPA procedure were reacted with water and carbon dioxide to convert the residual calcium sulfide to calcium carbonate.

The result of EPA strong acid tests conducted with the oxidized samples of sulfided calcium-based sorbents indicate that when the calcium sulfide content of these solid waste materials is below 4.5%, they will meet the EPA requirement for sulfur release, making them suitable for landfill disposal. When the calcium sulfide content of such solid waste materials are above 4.5%, they can be reacted with water and carbon dioxide to remove the residual sulfide to below 0.5%, making them suitable for landfill disposal. The presence of ash in the LASH (a mixture of limestone-derived and ash-derived) material did not appear to affect the sulfur leachability of such waste materials.

## EXECUTIVE SUMMARY

Advanced or second-generation power generation systems such as Integrated Gasification Combined Cycle (IGCC) and two-stage Pressurized Fluidized-Bed Combustion (PFBC) plants can achieve higher thermal efficiencies and a lower cost of electricity than a conventional pulverized coal-fired (PC fired) plant equipped with flue gas desulfurization. Because these advanced processes can use high sulfur coals in an environmentally acceptable manner, they have the potential to expand the marketability of Illinois coals. Projected costs for IGCC and PFBC plants are substantially lower than comparable pulverized-coal (PC) combustion plants, especially if air emission regulations were to require much lower SO<sub>2</sub> emissions.

These advanced coal conversion processes generally involve gasification of coal followed by combustion of the fuel gas in a gas turbine. In these processes, the sulfur compounds present in coal are converted to hydrogen sulfide when the coal is gasified. To comply with the New Source Performance Standards (NSPS) and lessen corrosion of the gas turbine, a high fraction of the sulfur must be removed from the fuel gas stream.

One approach for removing sulfur compounds from the fuel gas is the use of calcium-based sorbents, such as dolomite and limestone in the gasifier. In such processes, dolomite and limestone react with the sulfur compounds (mainly H<sub>2</sub>S) to significantly reduce (i.e., more than 90%) the sulfur content of the fuel gas.

The solid wastes, produced from the reactions of calcium-based sorbents with hydrogen sulfide in the fuel gas, contain calcium sulfide which is unstable and has the tendency to decompose when it contacts moisture in the air, releasing hydrogen sulfide to the atmosphere. To dispose of the solid waste products in an environmentally acceptable manner, calcium sulfide can be converted either to stable calcium sulfate through reaction with oxygen, or calcium carbonate through reaction with water and carbon dioxide.

Stabilization of partially sulfided dolomite with oxygen has been studied by the principal investigator of this project in earlier ICCI-funded programs. The results of these studies indicate that limestone can only be partially sulfated (about 30%) while dolomite can be sulfated above 95%. The extent of sulfation depends not only on the sorbent type but also on the extent of sulfidation in the gasifier.

Surprisingly, essentially all the oxidized sorbents (especially those with high calcium sulfide content) produced in the earlier studies successfully passed the less stringent EPA test protocol, making them suitable for disposal. However, because the test procedure was believed to be inadequate, further studies were limited to dolomite where a high level of conversion of calcium sulfide could be achieved.

A new protocol for testing sulfide containing waste, which is much more stringent than the old method, has been proposed by EPA.

It was believed that because of stringent disposal requirements, single-stage oxidative stabilization process involving limestone or even dolomite, would prove to be insufficient for production of acceptable material for disposal. Under such a scenario, a second stage would be necessary to remove the residual calcium sulfide from the sample. Earlier studies at IGT under sponsorship of ICCI have shown that over 99% of calcium sulfide in the spent sorbent can be removed through the reaction of calcium sulfide with water and carbon dioxide (hydrolysis). Such a process is believed to be suitable for conversion of the residual calcium sulfide to calcium carbonate.

The overall objective of this study was to determine the effect of implementation of the new and more stringent EPA Protocol Test Method involving sulfide containing waste, on the suitability of the oxidized spent sorbents from gasification of high sulfur coals, and to determine the optimum operating conditions in a "final" hydrolysis stage for conversion of the residual calcium sulfide in these wastes to materials that are suitable for disposal in landfills. An additional objective of this program was to study the effect of ash on the regeneration and ash-sorbent separation steps in the Spent Sorbent Regeneration Process (SSRP).

In this study, oxidized samples of the spent sorbents (produced in earlier ICCI-funded projects) covering a broad range of operating variables, including sorbent type, particle size, level of sulfidation, and level of sulfation, were evaluated in terms of their suitability for disposal under the new and more stringent EPA test procedure. The results of these tests were used to establish a guideline for selection of the relevant operating conditions in the one-step oxidative stabilization process.

Samples that did not pass the EPA test procedure were further reacted with water and carbon dioxide in the "hydrolysis" process to convert the residual calcium sulfide to calcium carbonate at the optimum operating conditions for this second stage determined in this project. The hydrolyzed sample were also tested under the new EPA procedure.

The result of EPA strong acid tests conducted with the oxidized samples of sulfided calcium-based sorbents indicate that when the calcium sulfide content of these solid waste materials is below 4.5%, they will meet the EPA requirement for sulfur release, making them suitable for landfill disposal.

When the calcium sulfide content of such solid waste materials are above 4.5%, they can be reacted with water and carbon dioxide in the temperature range of 60° to 120°C at 5 bar to remove the residual sulfide to below 0.5%, making them suitable for landfill disposal.

The presence of ash in the LASH (a mixture of limestone-derived and ash-derived) material did not appear to affect either the sulfur leachability of such waste materials, or the removal of the residual sulfide through reaction with water and carbon dioxide. A modest separation of LASH material into sorbent-rich and ash-rich materials (suitable for recycle and disposal, respectively) in the context of SSRP process could be achieved with

gravity-assisted separation technique. The sorbent-rich fraction had about 65% weight of the LASH material and contained 45% of the ash as well as 80% of the sorbent in the LASH. It should be noted that in order to achieve even such a modest level of separation, one has to deal with two separate streams in the context of SSRP process. While the sorbent-rich stream is hydrolyzed and recycled, the ash-rich stream should also be hydrolyzed separately to produce suitable material for landfill disposal.

## OBJECTIVES

The overall objective of this study was to determine the effect of implementation of the new and more stringent EPA Protocol Test Method involving sulfide containing waste, on the suitability of the oxidized spent sorbents from gasification of high sulfur coals, and to determine the optimum operating conditions in a "final" hydrolysis stage for conversion of the residual calcium sulfide in these wastes to materials that are suitable for disposal in landfills. An additional objective of this program was to study the effect of ash on regeneration and ash-sorbent separation in Spent Sorbent Regeneration Process (SSRP).

The specific objectives of this project were to:

- Determine the suitability of a broad set of oxidized spent sorbents for disposal under the new and more stringent EPA testing procedure.
- Establish guidelines relating process variables to the acceptability of the product for disposal.
- Determine the optimum operating conditions for removal of residual sulfide from oxidized spent sorbents.
- Determine the effect of ash on spent sorbent regeneration and ash-sorbent separation.
- Provide a preliminary assessment of the economic issues involved in spent sorbent stabilization.

## INTRODUCTION AND BACKGROUND

Advanced or second-generation power generation systems such as Integrated Gasification Combined Cycle (IGCC), and two-stage Pressurized Fluidized Bed Combustion (PFBC) plants can achieve higher thermal efficiencies and a lower cost of electricity than a conventional pulverized coal-fired (PC fired) plant equipped with flue gas desulfurization. Because these advanced processes can use high sulfur coals in an environmentally acceptable manner, they have the potential to expand the marketability of Illinois coals. Projected costs for IGCC and PFBC plants are substantially lower than comparable pulverized-coal (PC) combustion plants, especially if air emission regulations were to require much lower SO<sub>2</sub> emissions.

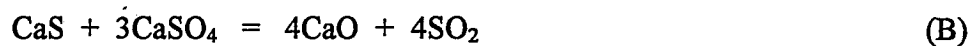
These advanced coal conversion processes generally involve gasification of coal followed by combustion of the fuel gas in a gas turbine. In these processes, the sulfur compounds present in coal are converted to hydrogen sulfide when the coal is gasified. To comply with the New Source Performance Standards (NSPS), a high fraction of the sulfur must be removed from the gas stream.

One approach for removing sulfur compounds from the fuel gas is the use of calcium-based sorbents, such as dolomite and limestone in the gasifier. In such processes, dolomite and limestone react with the sulfur compounds (mainly H<sub>2</sub>S) to significantly reduce (i.e., more than 90%) the sulfur content of the fuel gas.

The solid wastes produced from the reactions of calcium-based sorbents with hydrogen sulfide in the fuel gas, contain calcium sulfide which is unstable and has the tendency to decompose when it contacts moisture in the air, releasing hydrogen sulfide to the atmosphere. To dispose the solid waste products in an environmentally acceptable manner, calcium sulfide can be converted to stable calcium sulfate through reaction with oxygen (air) --



Stabilization of partially sulfided dolomite with oxygen has been studied by Abbasian<sup>(1-4)</sup> in earlier ICCI-funded projects. The results of these investigations indicate that limestone can only be partially sulfated while dolomite can be nearly completely sulfated at about 800°C.<sup>(1-10)</sup> The results (shown in Figures 1 and 2) also indicate that the extent of sulfation depends not only on the sorbent type but also on the extent of sulfidation in the gasifier. The calcium sulfate produced by Reaction (A), at temperatures of interest to the overall process (800°-1000°C), can also react with the calcium sulfide in an apparent solid-solid reaction to release SO<sub>2</sub> --



Reaction (B) is generally undesirable because of SO<sub>2</sub> evolution. Evolution of SO<sub>2</sub> during sulfation of sulfided calcium-based sorbents has also been reported by many investigators and reported in the literature<sup>(11-14)</sup>, indicating that the fraction of sulfur released as SO<sub>2</sub> during oxidation of CaS appeared to be very sensitive to temperature. The effect of reaction temperature on the distribution of the products of CaS oxidation (i.e. CaSO<sub>4</sub> and SO<sub>2</sub>) was investigated by Abbasian<sup>(3-5)</sup> in earlier ICCI-funded programs, and the results indicated that the fraction of sulfur released as SO<sub>2</sub> increases exponentially with temperature. The overall reaction for the release of SO<sub>2</sub> is



The release of sulfur as SO<sub>2</sub> during stabilization is undesirable because it reduces sorbent utilization and requires a further cleanup procedure for the sulfur dioxide.

Of immediate interest to this study is the fact that at 1500°F (815°C) a significant fraction of the calcium sulfide remains in the oxidized sorbent (see Figure 3). An earlier study<sup>(1)</sup> involving reactor modeling has shown that in terms of the overall process, 800-850°C is the optimum temperature range for oxidation of the spent sorbents.

Earlier studies regarding the suitability of the oxidized material for disposal under proposed 1987 testing protocol indicated that essentially all the oxidized samples (especially those with high calcium sulfide content) were acceptable for disposal in the landfill. However, because of a strong belief in the inadequacy of the EPA testing procedure, the research focused on studies resulting in high conversion conditions (i.e., more than 90%) to maximize the probability of producing acceptable solid waste material under (then unknown) future EPA regulations. In this regard, no further work was performed on the stabilization of limestone derived material, as the final sulfide conversion was proven to be limited.

A new protocol for testing sulfide containing waste has been proposed by EPA, which is much more stringent than the old method. This new method involves exposing the sulfide containing waste to an acidic solution while maintaining the pH level of the solution at about 2. Under this method, a high fraction of the residual calcium sulfide is expected to react with the acid, releasing hydrogen sulfide. Under such circumstances, in order to satisfy the regulation the level of conversion of calcium sulfide to calcium sulfate is expected to be very high (i.e., >99%). However, because no systematic study has been conducted, there are no guidelines for selection of process variables that would lead to acceptable material after the oxidation step.

A systematic evaluation of oxidized samples of the spent sorbents covering a broad range of operating variables, including sorbent type, particle size, level of sulfidation, and the level of sulfation was deemed necessary to establish a guideline for the selection of the relevant variable in the process. The availability of the large number of oxidized spent sorbent samples at IGT facilitated the achievement of the project objective and resulted in significant savings in the cost of such work.

It was believed that because of stringent requirements, the single-stage oxidative stabilization process involving limestone, or even dolomite, would prove to be insufficient for production of acceptable material for disposal. Under such a scenario, a second stage would be necessary to remove the residual calcium sulfide from the sample.

In an earlier ICCI-funded study<sup>(15)</sup> on the development of Spent Sorbent Regeneration Process (SSRP), it has been shown that over 99% of the sulfide in the spent sorbent (both limestone and dolomite) can be converted to calcium oxide or calcium carbonate by reacting the spent sorbent with water and CO<sub>2</sub>, producing a final waste product that is suitable for disposal under the most stringent environmental regulations.

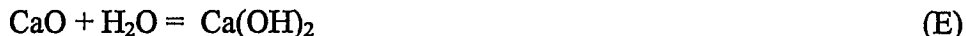
In the SSRP process, the spent sorbent is sent to a reactor containing water at temperatures of 60-120°C and pressures of 1-5 bar. Process derived CO<sub>2</sub>, such as that from the flue gas, is bubbled through the mixture as needed to reduce the pH of the solution to less than 8. Under these reaction conditions the CaS will react with the water to produce H<sub>2</sub>S, which may be scrubbed or converted to elemental sulfur in a Claus Unit. The residual CaS is converted to Ca(OH)<sub>2</sub> and/or CaCO<sub>3</sub>, depending on the solution pH

and the amount of CO<sub>2</sub> bubbled through the system. The main chemical reactions involved in the SSRP are-

Reactions Involving Sulfided Sorbent and Water, pH > 8:



Reactions Involving Sulfided Sorbents and Water, pH < 8:



Reactions occurring when CO<sub>2</sub> is added to the system are Reactions (D), (E), and (F) as well as the following reactions:



The KEL-S process<sup>(16)</sup> utilized spent CaSO<sub>4</sub> from limestone flue gas desulfurization scrubber waste to recover CaCO<sub>3</sub> and sulfur. This process involved the reduction of CaSO<sub>4</sub> to CaS using coal as the reducing agent, followed by a slurring of the CaS in an aqueous solution to form CaCO<sub>3</sub> and H<sub>2</sub>S. In a similar process, Gasowski and coworkers<sup>(17)</sup> used carbon monoxide to reduce CaSO<sub>4</sub> to CaS. The calcium sulfide was further hydrolyzed to produce calcium oxide and hydrogen sulfide.

Brothers and Crandlemire<sup>(18)</sup> investigated the desulfurization of CaS formed during coal-lime pyrolysis of high-sulfur coals. The CaS derived from a char/CaS slurry was first dissolved in an aqueous H<sub>2</sub>S solution, where the CaS is 1000 times more soluble than in pure water. The sulfur was recovered by passing carbon dioxide through the solution.

A number of researchers have investigated high-temperature methods of CaS regeneration. Gruncharov et al.<sup>(19)</sup> obtained complete conversion of CaS to CaO using a gas mixture with 10% steam at 1050°C, and by using a mixture of argon, 1% hydrogen, and 20% steam at 1000 °C.

In two related patents, Bauer<sup>(20,21)</sup> investigated methods for the safe disposal of CaO and CaS containing wastes generated in the iron ore reduction process. One method involved the high-temperature oxidation of CaS to CaSO<sub>4</sub>, followed by a low-temperature (24-38°C) aqueous carbonation with CO<sub>2</sub> to convert remaining CaO and CaS to CaCO<sub>3</sub>. In the second process, CaO, CaS, and iron ore wastes were slurried and sparged with CO<sub>2</sub> to produce CaCO<sub>3</sub>. Residual iron ore was magnetically removed, but there was no attempt to recover the sulfur.



Although it has shown that shown that more than 99% of calcium sulfide can be converted to calcium hydroxide and/or calcium carbonate through the hydrolysis reactions, the residual calcium sulfide in the oxidized spent sorbents may be trapped by layers of calcium sulfate and calcium oxides, making penetration of water into the sample difficult, resulting in limited conversion of calcium sulfide to calcium hydroxide or calcium carbonate.

A different approach for treatment of sulfide-containing spent sorbent is to regenerate and recycle the sorbent to the gasifier. The regeneration of calcium sulfide within the context of IGT's Spent Sorbent Regeneration Process (SSRP) has been studied. Successful application of the SSRP system requires separation of the regenerated sorbent and coal ash mixture into sorbent- and ash-rich streams for recycle and disposal, respectively. A practicable level of separation of the ash from the regenerated calcium carbonate could yield a stream that is 89% ash for disposal in a landfill. This means that about 63% of the total ash in the incoming stream from the regenerator is removed, resulting in a recycle stream of ash plus calcium carbonate from the regeneration/separation process that is about 34% ash and 66% calcium carbonate. With these levels of separation, approximately 90% of the calcium required in the gasifier is supplied by regeneration of the spent sorbent.

This study focused on establishing a guideline regarding the process requirements that would lead to a suitable single-stage oxidative stabilization process. Furthermore, this study geared toward determination of the "optimum" conditions for the final "hydrolysis" stage that is definitely required for stabilization of limestone, or, should such an extra stage become necessary for stabilization of dolomite-based solid wastes to environmentally acceptable material, under current or future, more stringent regulations. Furthermore, the effect of ash on spent sorbent regeneration as well as ash-sorbent separation in the context of Spent Sorbent Regeneration Process (SSRP), wee addressed in this program.

## EXPERIMENTAL PROCEDURE

This project was divided into the following six tasks:

- Task 1. Oxidation of Spent Sorbents
- Task 2. Sulfur Leaching Tests on Oxidized Sample
- Task 3. Development of Guideline for Spent Sorbents Stabilization
- Task 4. Removal of Residual Sulfur by Hydrolysis
- Task 5. Process Integration and Economic Assessment
- Task 6. Preliminary Spent Sorbent Regeneration and Recycle Studies

### Material

The calcium sulfide containing solid waste materials used in this study included oxidized samples of partially sulfided calcium-based sorbents from earlier projects, as well as a samples of actual ash-limestone (LASH) from IGT's U-Gas pilot-scale gasifier that utilized calcium-based sorbent for *in-situ* desulfurization. Samples of the oxidized spent

sorbents were selected to cover a wide range of operating variables such as sorbent type, sorbent particle size, extent of sulfidation, the extent of sulfation, as well as the oxidization temperature.

### Task 1. Oxidation of Spent Sorbents

The objective of this task was to prepare an oxidized sample of actual LASH (a mixture of calcium-derived and ash-derived materials ) from a gasifier for further testing in Tasks 2 and 4.

In this task, a sample of LASH from IGT pilot-scale gasifier was reacted with oxygen in the quartz fluidized-bed reactor. The schematic diagram of this unit is shown in Figure 4. The unit essentially consists of a quartz fluidized-bed reactor which is externally heated by two electric furnaces, equipment for feeding and measuring the flow rates of the gases, measuring and controlling the bed temperature, and monitoring the fluidized-bed pressure. The fluidized-bed distributor is made of a quartz fritted disk.

In a typical test, about 50 to 100 grams of the partially sulfided sorbent are loaded into the fluidized-bed reactor. The bed of solids is heated by the external furnace, while a stream of nitrogen flows through the reactor until the reactor reaches the desired temperature. The gas flow rate is then increased to fluidize the bed. At this point, the gas is switched to a reactant gas containing the desired concentration of oxygen. The desired composition of the reactant gas is achieved by mixing a stream of air with nitrogen at predetermined ratios. A slip stream of the reactor off-gas is analyzed to determine the concentration of the sulfur dioxide that may evolve during the reaction. The reaction continues until the desired solids residence time is reached. During the test, the temperature is controlled by the temperature controller. The reactor temperature and pressure are monitored by a computer data acquisition system. The reactor off-gas passes through a filter for collection of elutriated fines and then through a liquid scrubber before being vented. After the test, the reactor solids are analyzed to determine the extent of sulfation and the fraction of sulfur released during the stabilization stage.

The oxidation test was conducted at 815°C (1500°F ) which has been shown to be the optimum temperature for oxidization of calcium sulfide containing solids. The reactant gas contained about 0.5% oxygen to prevent temperature excursions which could lead to undesirable release of sulfur as SO<sub>2</sub> during oxidation.

The feed and the oxidized samples were analyzed to determine the extent of oxidation of the calcium sulfide in the sample. The oxidized LASH sample was also analyzed for sulfur release under the "new" more stringent strong acid test. The results of the chemical analyses and sulfur leaching tests performed on the LASH sample are given under "RESULTS AND DISCUSSION".

## Task 2. Sulfur Leaching Tests on Oxidized Samples

The objective of this task was to determine the suitability of a wide range of oxidized spent sorbent samples for disposal under the revised EPA sulfur leaching test procedure.

The new EPA sulfur leaching test procedure was applied to oxidized samples of sulfided calcium based sorbents (from earlier ICCI-funded programs) as well as the oxidized LASH and DASH samples produced in Task 1. The revised test procedure involves reacting the sample with an acidic solution where a pH level of about 2 is maintained for the duration of the test (30 min). As indicated earlier, the revised method is much more stringent than the earlier version, resulting in much higher amount of H<sub>2</sub>S release from the sample.

A total of 17 samples (collected in the earlier projects) were selected to cover a wide range of operating variables including sorbent type, extent of sulfidation, extent of sulfation, and oxidation temperature. These samples were analyzed for sulfur release under both "old" weak acid test as well as "new" more stringent strong acid test. The results of sulfur leaching tests are presented in "RESULTS AND DISCUSSION".

## Task 3. Development of Guidelines for Spent Sorbent Stabilization

The objective of this task was to determine operating conditions needed for an oxidative stabilization under the new EPA procedure.

The results of sulfur leaching tests (under new EPA procedures) with the broad range of samples (obtained in Task 2) were analyzed to develop guidelines for selection of the operating conditions that can lead to production of acceptable solid product for landfill disposal in a single-stage oxidative stabilization. The results of the analyses of sulfur leaching tests conducted in this task are presented in "RESULTS AND DISCUSSION".

## Task 4. Removal of Residual Sulfur by Hydrolysis

The objective of this task was to investigate the feasibility of removing the residual calcium sulfide in oxidized spent sorbent through hydrolysis reactions.

In this task, samples of the oxidized spent sorbent that did not pass the new EPA sulfur leaching procedures were reacted with water and carbon dioxide at elevated temperature and pressure in the pressurized bench-scale liquid bed reactor (LBR) unit. The temperature was in the range of 60° to 120°C, while the pressure was maintained at 5 bar.

The schematic diagram of the LBR unit is shown in Figure 5. This bench-scale reactor is capable of operation with up to 500 grams of solid reactant. This unit was constructed under an earlier ICCI-funded project and required minor upgrading and shakedown for this project. The stainless steel reactor vessel is heated with an external electrical furnace. A mechanical mixer system is used to provide good solid-liquid contacting. The water

level inside the reactor is maintained by a pump. Carbon dioxide is introduced to the base of the reactor through a specially designed bubble-cap distributor. The reactor temperature is controlled by temperature controllers while the reactor pressure is maintained by a back pressure regulator.

In a typical test, a required amount of water and the solid sample are added to the reactor and the reactor is heated under a desired flow of nitrogen and pressurized. After the reactor reaches the desired temperature and pressure, carbon dioxide is introduced into the reactor. Periodic samples of the slurry are withdrawn into the pH sample pot (before and after CO<sub>2</sub> introduction) and analyzed. The pH values of the slurry before carbon-dioxide is introduced is about 11 to 12, indicating that the slurry contains "basic" Ca(OH)<sub>2</sub>. The pH level of the slurry gradually decreases after CO<sub>2</sub> introduction to about 6 to 7 indicating that the slurry content is mostly calcium carbonate. The hydrogen sulfide released from the sample during the test is absorbed by the zinc acetate scrubber. After achieving the predetermined solid/liquid residence time at the desired temperature, pressure, and pH values, the test is terminated.

After the test, the slurry is removed from the reactor and its calcium as well as sulfide contents are determined. The acetate is also analyzed to determine the amount of sulfur released during the test. The hydrolyzed samples are subjected to the new EPA leaching test procedure (if residual sulfide is still present in the sample) to determine their suitability for landfill disposal. The applicability of the guideline developed in Task 3 (for oxidized samples) to the hydrolyzed samples will also be determined.

In this task, samples of oxidized sulfided calcium based sorbents and LASH material, as well as sulfided samples (i.e., prior to oxidation) which had failed the EPA strong acid test were reacted with water and carbon dioxide at elevated temperature and pressure in the pressurized bench-scale liquid bed reactor (LBR) unit. The temperature was in the range of 60° to 120°C, while the pressure was maintained at 5 bar. The hydrolyzed samples were analyzed for chemical composition and were subjected to strong acid sulfur leaching tests. The results of the chemical analyses and sulfur leaching tests performed on the hydrolyzed samples are given under "RESULTS AND DISCUSSION".

#### Task 5. Process Integration and Economic Assessment

Using the data obtained in Tasks 2 through 4, the viability of single-stage and two-stage processes for stabilization of calcium-based sorbents from coal gasification were evaluated. This information was used for a preliminary assessment and comparison of processes involving a typical gasifier using dolomite or limestone for sulfur capture of sorbents with single-stage as well as two-stage oxidative stabilization of the calcium sulfide. The results of this task are given under "RESULTS AND DISCUSSION".

### Task 6. Preliminary Spent Sorbent Regeneration and Recycle Studies

The objective of this task was to study the effects of ash on sorbent regeneration and sorbent-ash separation in the Spent Sorbent Regeneration Process (SSRP).

The calcium sulfide-containing waste material were hydrolyzed in the liquid-bed reactor as described previously in Task 4 at conditions based on the results obtained in previous studies which resulted in over 95% sulfur removal from the sorbent (converting CaS to CaCO<sub>3</sub> and Ca(OH)<sub>2</sub>). The extent of sorbent regeneration was determined by a material balance on the system and analyses of feed and product sorbent for calcium and sulfur.

In this task, samples of the LASH material were hydrolyzed in the LBR unit at 71°C (160°F) at 5 bar. The regenerated sample was analyzed for sulfur release using the strong acid test. The results are presented under "RESULTS AND DISCUSSION".

The LASH material is converted to ultrafine particles during the hydrolysis process. Different methods were used to separate the products into an ash-rich and a sorbent rich streams. The goal was to retain about 90% of the sorbent and about 1/3 of the ash in the sorbent rich stream that can be recycled in the SSRP process, while about 10% of the sorbent-derived materials and 2/3 of the ash can be disposed as the system waste. In this task, gravity-assisted separation as well as particle size classification were used with the LASH sample before and after the hydrolysis stage. The results are presented in the next section of this report.

## RESULTS AND DISCUSSION

The result of the chemical analysis of the oxidized LASH sample (i.e., from U-Gas pilot-scale gasifier) is presented in Table 1. The sorbent used in the gasifier was a dolomitic limestone with a magnesium to calcium molar ratio of about 0.5. The results indicate that the maximum level of conversion of calcium sulfide to calcium sulfate is about 50%, which is consistent with the results obtained in earlier ICC-funded projects.<sup>(4)</sup>

**Table 1. CHEMICAL ANALYSIS OF OXIDIZED LASH SAMPLE**

<u>Analyses</u>	<u>wt%</u>
Calcium	24.3
Magnesium	8.08
Sulfur ( as Sulfide )	2.37
Sulfur ( as Sulfate )	2.36

Samples of sulfided (i.e., feed) and oxidized (i.e., spent) calcium based sorbents as well as the LASH materials, were analyzed using both weak acid and more stringent strong acid tests. The results indicate that all samples (including those that were not oxidized) pass

the EPA requirement of 500 mg H<sub>2</sub>S per kg of solid waste. However, the results of the strong acid sulfur leachability test, shown in Figure 6, indicate that, under the more stringent test procedure, only samples containing less than about 2% sulfur as sulfide, (i.e., less than 4.5% calcium sulfide ) pass the EPA requirement. This level of sulfide content is about fifty times higher than 500 mg/Kg allowable under the EPA test, indicating that about 98% of the sulfide is retained in the oxidized sample under the strong acid test. It is important to note that the oxidation of the LASH sample did not results in suitable waste product for landfill disposal.

Samples of oxidized sulfided calcium based sorbents, including feed materials, which had failed the EPA strong acid test were reacted with water and carbon dioxide at elevated temperature and pressure in the pressurized bench-scale liquid bed reactor (LBR) unit. Additionally, the LASH sample from a U-Gas gasifier was also hydrolyzed in the LBR unit. The hydrolyzed samples were analyzed for chemical composition and were subjected to strong acid sulfur leaching tests. The sulfide contents of the feed and reacted sorbent as well as the level of sulfur released during the EPA strong acid tests are given in Table 2.

**Table 2. SULFIDE CONTENT AND SULFUR LEACHED IN FEED AND PRODUCTS OF REGENERATION REACTION**

Sample No.	Feed Sulfide %	Feed Sulfur Leached mg/Kg	Product Sulfide %	Product Sulfur Leached mg/Kg
1	4.36	N/A	.5	<60
2	8.96	N/A-	.12	<60
3	2.68	1700	.25	<60
4	10.3	1700	.22	<60
5	2.99	1200	.08	<50
6	19.8	6300	.21	<50
7*	6.83	46000	.37	61
8*	2.36	1000	.2	<50

\*LASH samples

The results shown in Table 2 indicate that the residual sulfide in the treated samples is below 0.5% and that all hydrolyzed samples meet EPA requirement, which is consistent with the guideline established and presented in this report.

Based on the results obtained in this study, it appears that single-stage oxidative stabilization is a viable process for conversion of the gasifier spent sorbent to environmentally acceptable material for landfill disposal, as long as the unreacted calcium sulfide content of the material is less than 4.5%. This implies that for a Ca/S ratio 2 during desulfurization of the coal gas (i.e., 50% sulfidation of the sorbent ), the minimum extent of conversion of calcium sulfide to calcium sulfate for limestone and dolomite are

88.5% and 83.5% respectively, if desulfurization is carried out outside the gasifier (i.e., with no coal ash present). Furthermore, for *in-situ* desulfurization process, because approximately 50% of the waste material is ash, the minimum extent of conversion of calcium sulfide to calcium sulfate for limestone and dolomite are 77% and 67% respectively. In an earlier ICCI-funded project, <sup>(4)</sup> it has been shown that these level of calcium sulfide to calcium sulfate can only be achieved when either dolomite is used, or when very fine (i.e.,  $d_p < 0.02$  cm ) limestone with a Ca/S ratio of higher than 2.5. is used during *in-situ* desulfurization, (see figures 1 and 2).

Given the restrictions on the Ca/S and particle size imposed by the gasification process, it can be concluded that dolomite will be the only sorbent suitable for a single-stage oxidative stabilization process. In another ICCI-funded project, <sup>(1)</sup> it was also shown that the reaction of calcium sulfide (in the partially sulfided dolomite) with oxygen is very fast, requiring a short residence time, and consequently a small reactor to achieve high conversions.

The results of the hydrolysis of the calcium sulfide containing waste material (shown in Table 2 ) suggest that limestone can be used for *in-situ* desulfurization only in the context of two-stage stabilization process. Given that by using fine limestone particle (i.e.,  $d_p=0.02$  cm) and a Ca/S ratio of 2, The maximum conversion of calcium sulfide to calcium sulfate achievable is only about 60% (see figure 2), A high fraction (i.e., about 30%) of the sulfide must be removed in the second stage, making it economically unattractive.

Although hydrolysis of the spent sorbent material proved to be very effective for removal of the residual calcium sulfide to meet the EPA limits for release of  $H_2S$ , separation of the products into an ash-rich and a sorbent-rich streams could not be accomplished by physical methods such as gravity-assisted separation or particle size classification. It was believe that for SSRP to be a viable process, it will be necessary to retain about 90% of the sorbent and 1/3 of the ash in the sorbent-rich stream. This level of separation results sufficient reduction in the quantity of sorbent needed by and the wastes generated by the process without requiring significantly larger gasifier, to accommodate the extra ash recycled in the process.

As expected, the LASH material was converted to ultrafine particles during the hydrolysis process. However, the ash-derived and sorbent-derived materials produced were very similar in terms of particle densities and diameter. Therefore, all efforts for separation of the ultrafine material were unsuccessful. The results of separation of the sorbent-derived and ash-derived materials in the oxidized samples (prior to hydrolysis) by particle size and gravity methods, indicate that the best separation result ( with the LASH sample used) may be achieved using a liquid media with a density of 2.6 g/cc, where about 35% of material can be collected as "float" to represent the ash-rich stream containing 55% of the ash and 20% of the sorbent in the LASH material. The "sink" fraction which represent the sorbent-rich fraction had about 65% weight of the LASH material and contained 45% of the ash as well as 80% of the sorbent in the LASH.

It should be noted that in order to achieve even such a modest level of separation, one has to deal with two separate streams in the context of SSRP process. While the sorbent-rich stream is hydrolyzed and recycled, the ash-rich stream should also be hydrolyzed separately to produce suitable material for landfill disposal.

Given the results obtained in this investigation, it is believed that the cost savings associated with the reduction in the quantities of sorbent utilized will not be sufficient to make the SSRP an economically attractive process.

### CONCLUSIONS AND RECOMMENDATIONS

Based on the results obtained in this project, it can be concluded that:

- The oxidized sulfided sorbents containing less than 2% sulfur (as calcium sulfide) can meet EPA requirement.
- All samples of the spent sorbents will meet the EPA limit after undergoing the hydrolysis stage.
- Single-stage oxidative stabilization process is a viable approach when dolomite is used.
- Conversion of sulfided limestone to environmentally acceptable waste products does not appear to be economically attractive.
- Effective separation of the hydrolyzed LASH materials into sorbent-rich and ash-rich streams does not appear to be economically achievable to make SSRP a viable process.



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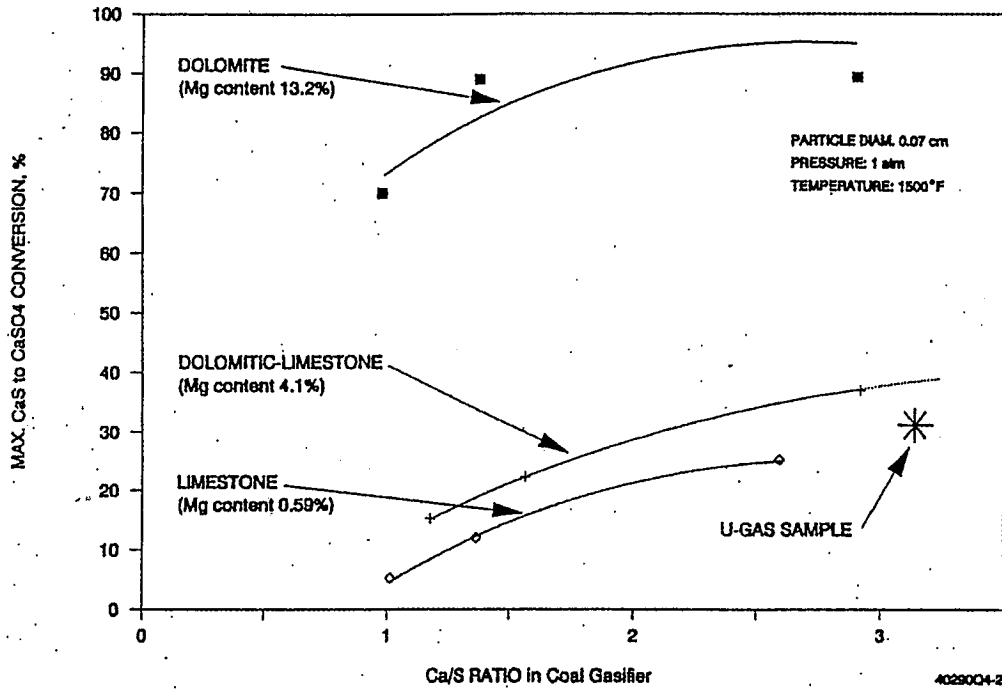


Figure 1. THE EXTENT OF SULFATION OF DIFFERENT Ca-BASED SORBENTS (COARSE PARTICLES)

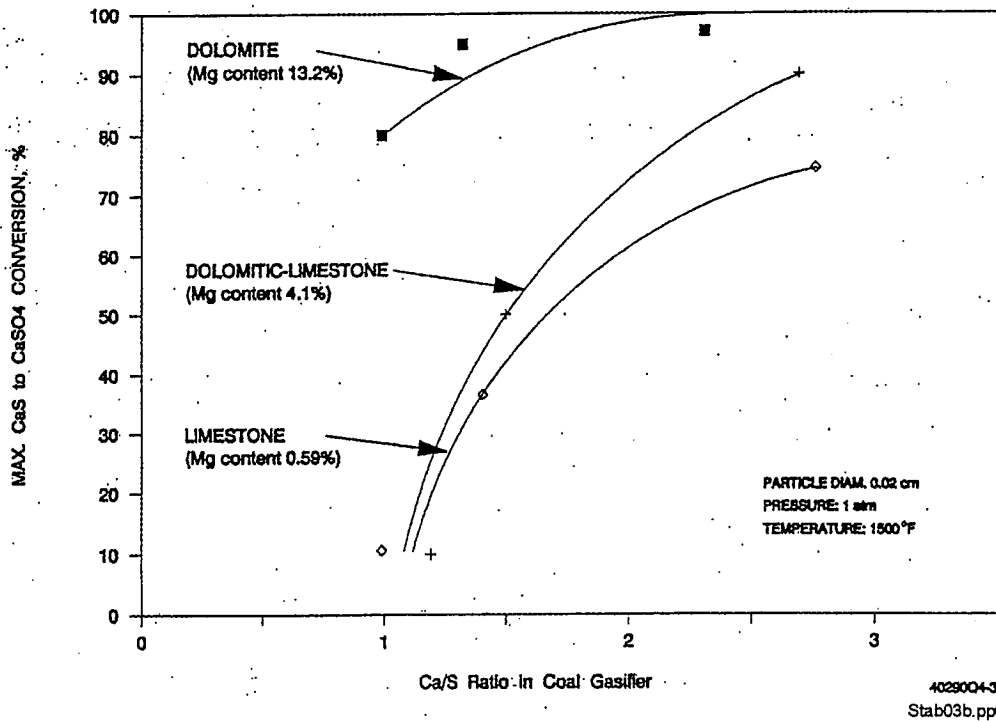
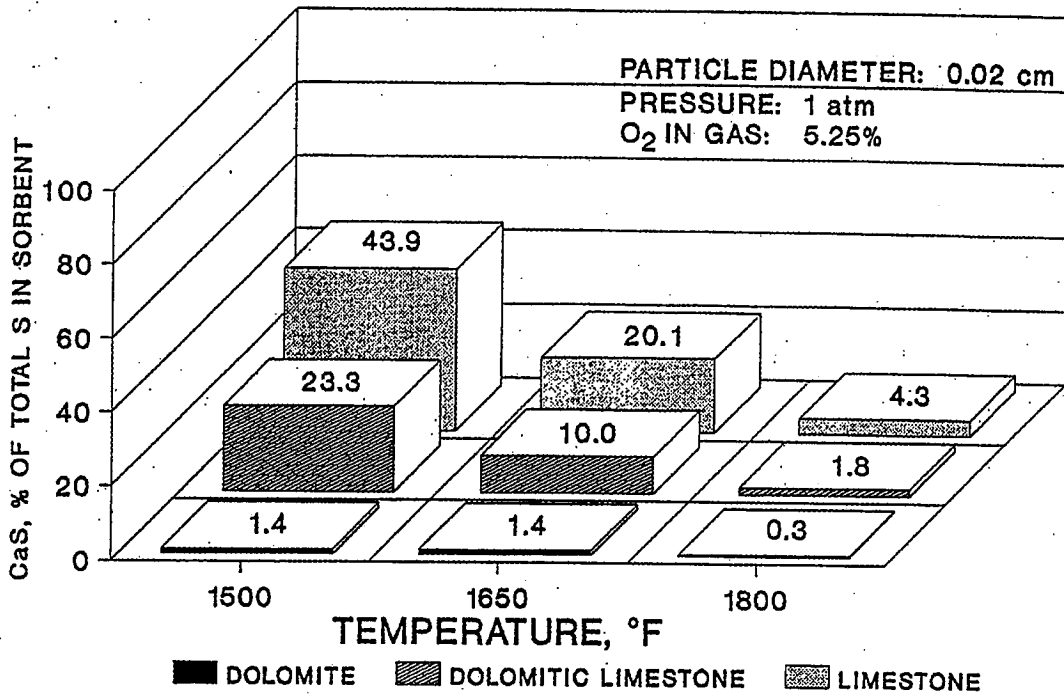
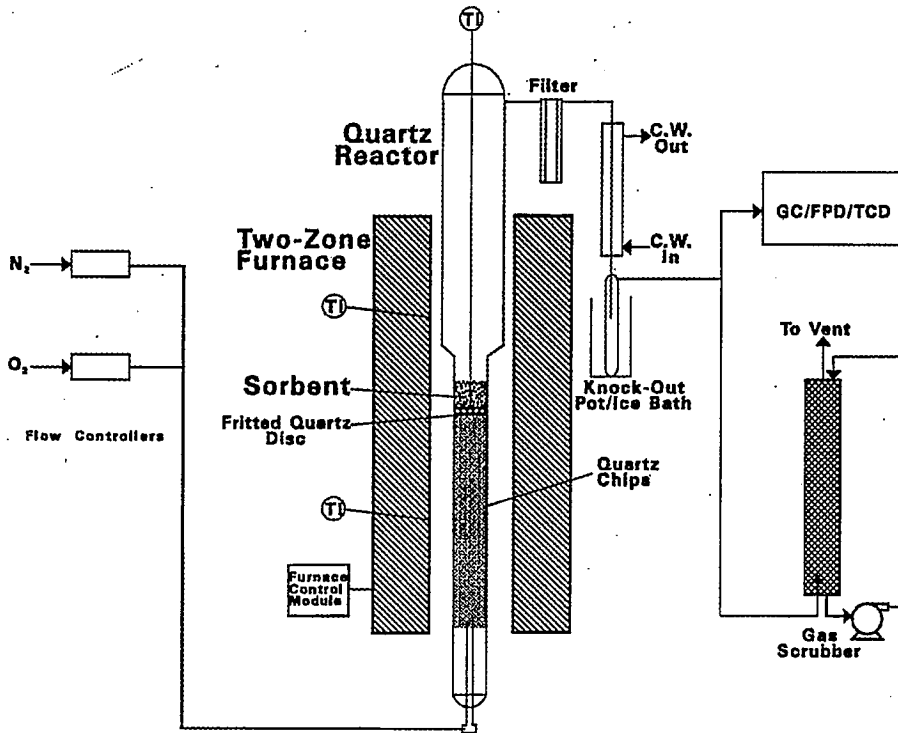


Figure 2. THE EXTENT OF SULFATION OF DIFFERENT Ca-BASED SORBENTS (FINE PARTICLES)



**Figure 3. UNREACTED CaS REMAINING AFTER OXIDATION (Fine Partially Sulfided Sorbents)**



**Figure 4. SCHEMATIC DIAGRAM OF THE PACKED/FLUIDIZED-BED REACTOR SYSTEM**

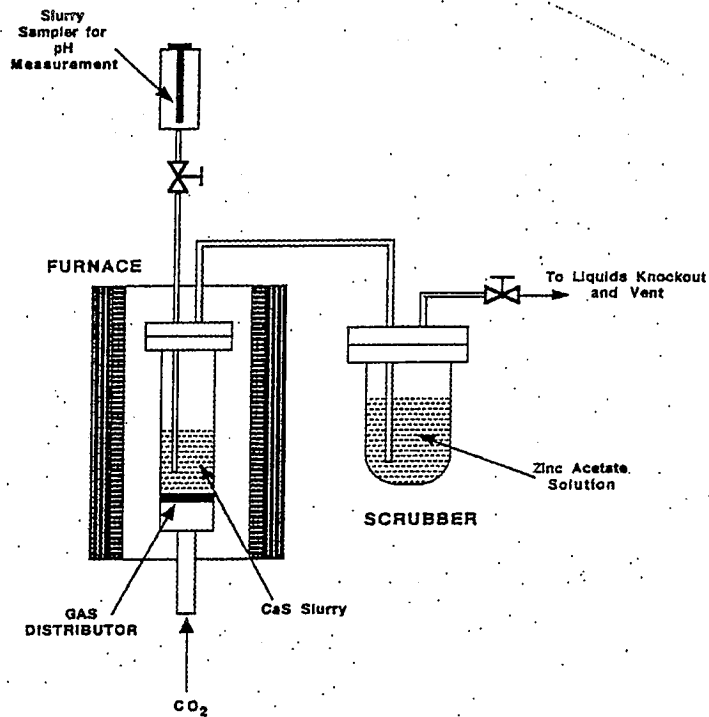


Figure 5. SCHEMATIC DIAGRAM OF THE LIQUID-BED REACTOR UNIT

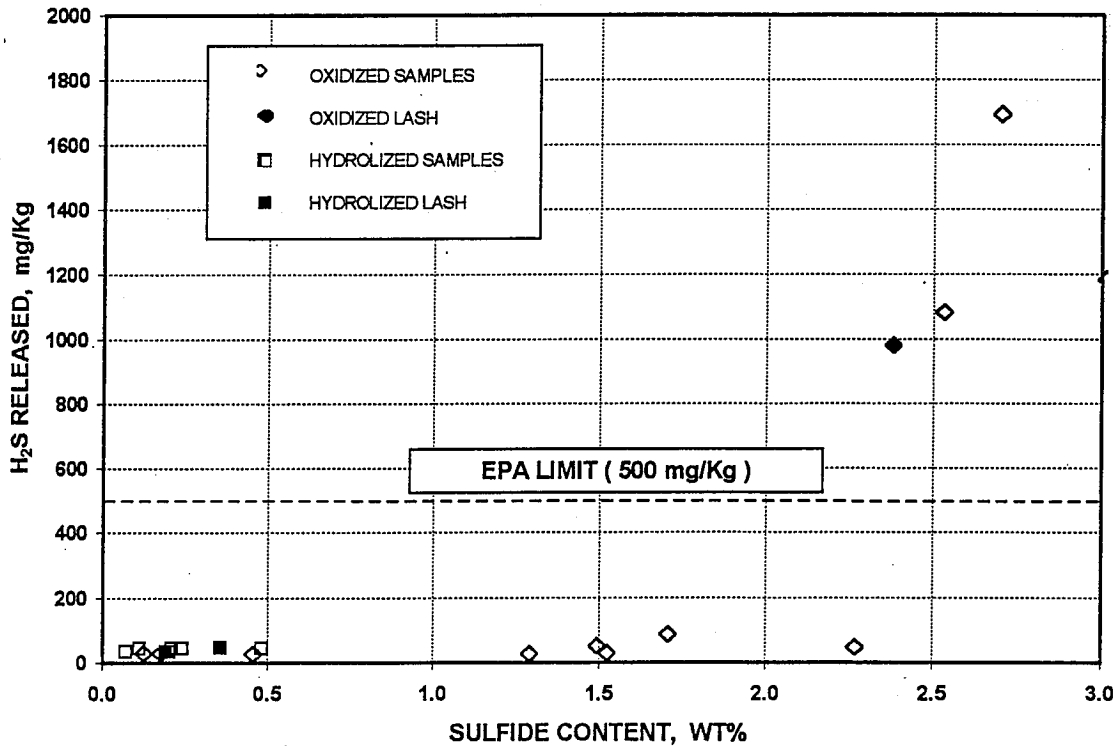


Figure 6. EPA STRONG ACID LEACHING TEST RESULTS FOR OXIDIZED SULFIDED Ca-BASED SORBENTS

## DISCLAIMER STATEMENT

“This report was prepared by Javad Abbasian, Institute of Gas Technology, 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 Commerce and Community Affairs through the Illinois Coal Development Board and the Illinois Clean Coal Institute. Neither Javad Abbasian, Institute of Gas Technology, nor any of its subcontractors nor the U.S. Department of Energy, Illinois Department of Commerce and Community Affairs, Illinois Coal Development Board, Illinois Clean Coal Institute, nor any person acting on behalf of either:

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PROJECT MANAGEMENT REPORT  
June 1, 1996, through August 31, 1996

Project Title: **STABILIZATION OF SPENT SORBENTS FROM COAL-BASED  
POWER GENERATION PROCESSES**

DOE Cooperative Agreement Number: DE-FC22-92PC92521(Year 4)  
ICCI Project Number: 95-1/3.3A-2M  
Principal Investigator: Javad Abbasian, Institute of Gas Technology  
Other Investigators: James R. Wangerow,  
Institute of Gas Technology  
Project Manager: Daniel D. Banerjee, ICCI

COMMENTS

The project was completed on schedule and all project objectives were achieved.

**EXPENDITURE - EXHIBIT B**

**CUMULATIVE PROJECTED AND ESTIMATED EXPENDITURES BY QUARTER**

Stabilization of Spent Sorbents from Coal-Based Power Generation Processes

Quarter*	Types of Cost	Direct Labor	Fringe Benefits	Materials & Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sept. 1, 1995 to Nov. 29, 1995	Projected	10,501		2,500				19,006	32,007
	Estimated	14,000		1,000				24,821	39,821
Sept. 1, 1995 to Feb. 29, 1996	Projected	22,003		5,500	1,310			40,161	68,974
	Estimated	20,800		2,500	1,300			37,386	61,986
Sept. 1, 1995 to 31-May-96	Projected	32,461		6,500	1,310			58,759	99,030
	Estimated	30,000		1,500	1,300			53,332	86,132
Sept. 1, 1995 to Aug. 31, 1996	Projected	41,458		6,500	1,898			74,697	124,553
	Estimated	44,000		2,500	1,500			78,197	126,197

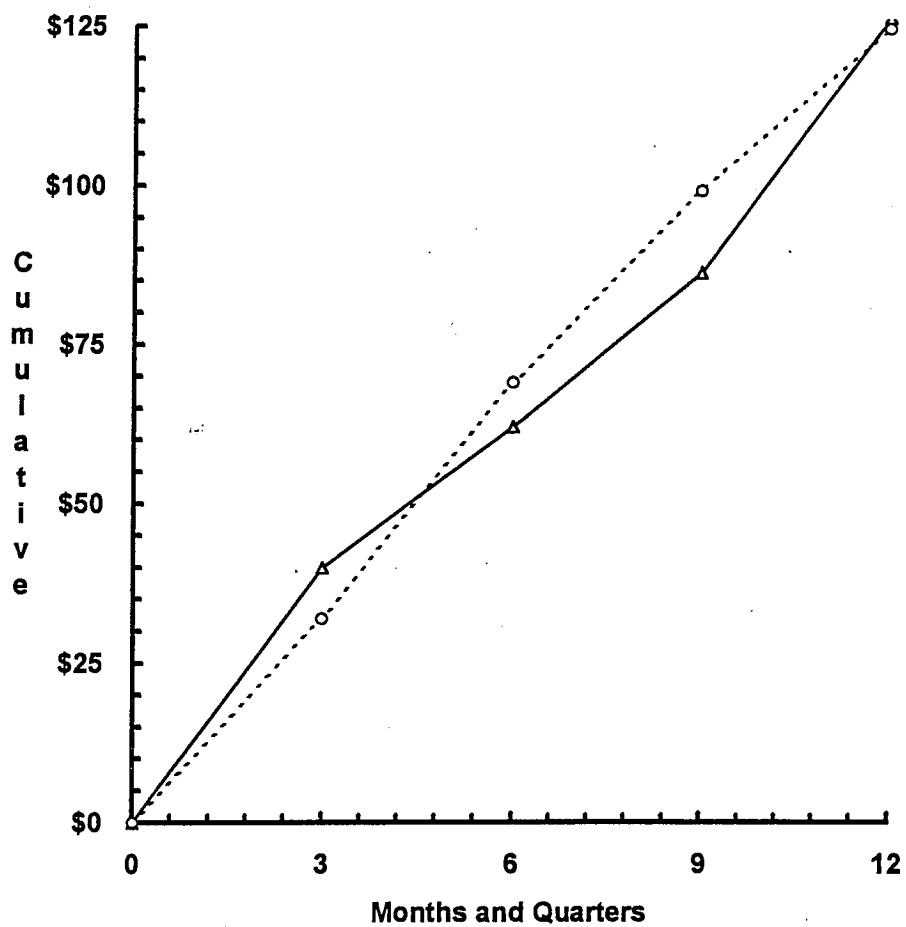
\* Cumulative by Quarter



## CUMULATIVE COSTS BY QUARTER - EXHIBIT C

Stabilization of Spent Sorbents from Coal-Based Power Generation Processes

(In Thousands)

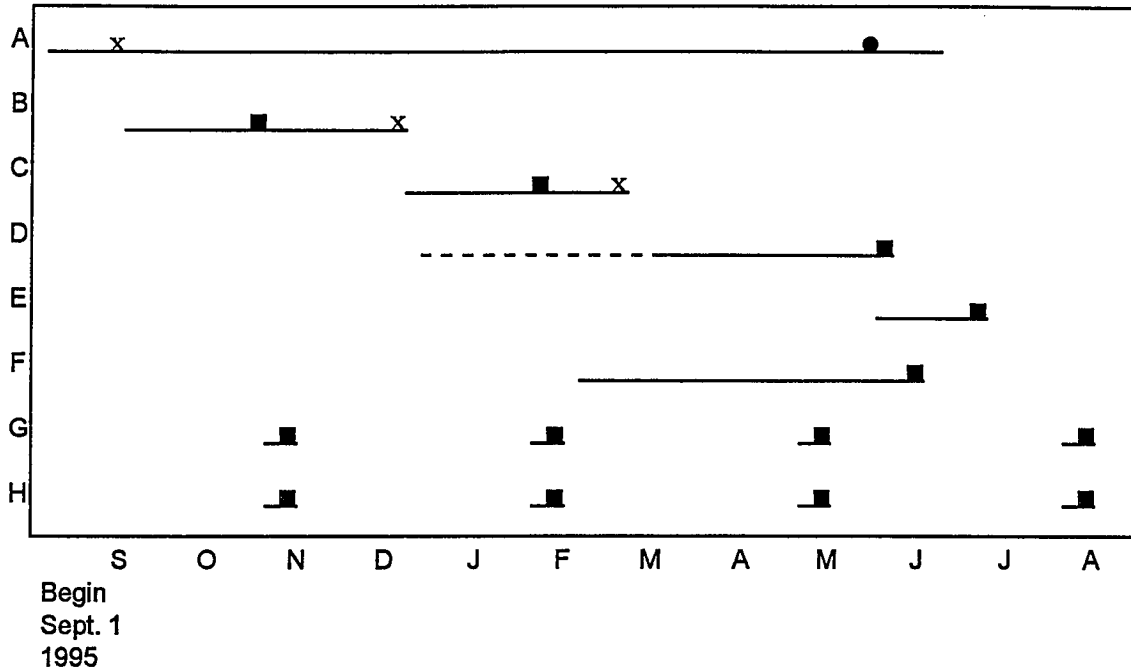


O = Projected Expenditures \$124,553

<> = Actual Expenditure \$126,197

Total ICCI Award \$124,553

## PROJECT SCHEDULE AND MILESTONE CHART



- A. Oxidation of Spent Sorbents
- B. Sulfur Leaching Tests on Oxidized Samples
- C. Development of Guidelines for Spent Sorbent Stabilization
- D. Removal of Residual Sulfur by Hydrolysis
- E. Process Integration and Economic Assessment
- F. Preliminary Spent Sorbent Regeneration and Recycle Studies
- G. Quarterly and annual technical reports prepared and submitted
- H. Quarterly project management reports prepared and submitted

**EQUIPMENT INVENTORY LIST**  
September 1, 1995, through August 31, 1996

**Project Title: STABILIZATION OF SPENT SORBENTS FROM COAL-BASED  
POWER GENERATION PROCESSES**

DOE Cooperative Agreement Number: DE-FC22-92PC92521(Year 4)  
ICCI Project Number: 95-1/3.3A-2M  
Principal Investigator: Javad Abbasian, Institute of Gas Technology  
Other Investigators: James R. Wangerow,  
Institute of Gas Technology  
Project Manager: Daniel D. Banerjee, ICCI

**COMMENTS**

No equipment with life expectancy of one year or more was purchased in this project.

**HAZARDOUS WASTE REPORT**  
September 1, 1995, through August 31, 1996

**Project Title: STABILIZATION OF SPENT SORBENTS FROM COAL-BASED  
POWER GENERATION PROCESSES**

DOE Cooperative Agreement Number: DE-FC22-92PC92521(Year 4)  
ICCI Project Number: 95-1/3.3A-2M  
Principal Investigator: Javad Abbasian, Institute of Gas Technology  
Other Investigators: James R. Wangerow,  
Institute of Gas Technology  
Project Manager: Daniel D. Banerjee, ICCI

**COMMENTS**

None of the hazardous substances defined under 40CFR261, Subpart D, entitled "List of Hazardous Wastes," were purchased, utilized, or generated in this project.

**LIST OF PUBLICATIONS AND PRESENTATIONS**

September 1, 1995, through August 31, 1996

**Project Title: STABILIZATION OF SPENT SORBENTS FROM COAL-BASED  
POWER GENERATION PROCESSES**

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 4)  
ICCI Project Number: 95-1/3.3A-2M  
Principal Investigator: Javad Abbasian, Institute of Gas Technology  
Other Investigators: James R. Wangerow,  
Institute of Gas Technology  
Project Manager: Daniel D. Banerjee, ICCI

**COMMENTS**

Abbasian, J., Hill, A. H., Wangerow, J. R., and Banerjee, D. D., "Development of Spent Sorbent Stabilization Process (SSSP)," Paper presented at the 1996 AIChE Spring National Meeting, February 25-29, New Orleans, Louisiana.

Abbasian, J. and Wangerow, J. R., "Stabilization of Spent Sorbents from Coal-Based Power Generation Processes," Paper presented at the Illinois Clean Coal Institute's 14<sup>th</sup> Annual Contractors' Technical Meeting, July 30-31, 1996, Champaign, Illinois.

# STABILIZATION OF SPENT SORBENTS FROM COAL-BASED POWER GENERATION PROCESSES

Javad Abbasian and James R. Wangerow  
Institute of Gas Technology

ICCI Project Manager: Daniel D. Banerjee

## ABSTRACT

The overall objective of this study is to determine the effect of implementation of the new and more stringent EPA Protocol Test Method involving sulfide containing waste, on the suitability of the oxidized spent sorbents from gasification of high sulfur coals for disposal in landfills, and to determine the optimum operating conditions in a "final" hydrolysis stage for conversion of the residual calcium sulfide in these wastes to materials that are suitable for disposal in landfills. An additional objective is to study the effect of ash on the regeneration and ash-sorbent separation steps in the Spent Sorbent Regeneration Process (SSRP).

To achieve these objectives, a large set of oxidized samples of sulfided calcium-based sorbents (produced in earlier ICCI-funded programs) as well as oxidized samples of gasifier discharge (containing ash and spent sorbent) are tested according to the new EPA test protocol. Samples of the oxidized spent sorbents that do not pass the EPA procedure are reacted with water and carbon dioxide to convert the residual calcium sulfide to calcium carbonate.

The results obtained during this project indicate that under the strong acid test procedure, all samples containing less than about 2% sulfur as calcium sulfide, pass the EPA requirement. Furthermore, the results also indicate that all samples of oxidized sulfided calcium based sorbents, which fail the EPA strong acid test, can be reacted with water and carbon dioxide to produce waste materials that will pass the EPA strong acid test.

## DEVELOPMENT OF SPENT SORBENT STABILIZATION PROCESS (SSSP)

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### ABSTRACT

Advanced power generation systems such as Integrated Gasification Combined Cycle (IGCC), and two-stage Pressurized Fluidized-Bed Combustion (PFBC) plants can achieve higher thermal efficiencies and a lower cost of electricity than a conventional pulverized coal-fired (PC fired) plant equipped with flue gas desulfurization. Projected costs for IGCC and PFBC plants are substantially lower than comparable pulverized-coal (PC) combustion plants, especially if air emission regulations were to require much lower SO<sub>2</sub> emissions. These advanced coal conversion processes generally involve gasification of coal followed by combustion of the fuel gas in a gas turbine. The sulfur compounds present in coal are converted to hydrogen sulfide when the coal is gasified. To comply with the New Source Performance Standards (NSPS) and lessen corrosion of the gas turbine, a high fraction of the sulfur must be removed from the gas stream.

One approach for removing sulfur compounds from the fuel gas is the use of calcium-based sorbents, such as dolomite and limestone in the gasifier. In such processes, dolomite and limestone react with the sulfur compounds (mainly H<sub>2</sub>S) to significantly reduce (i.e., more than 90%) sulfur content of the fuel gas. The solid wastes, produced from the reactions of calcium-based sorbents with hydrogen sulfide in the fuel gas, contain calcium sulfide which is unstable and has the tendency to decompose when it contacts moisture in the air, releasing hydrogen sulfide to the atmosphere. To dispose of the solid waste products in an environmentally acceptable manner, calcium sulfide can be converted to stable calcium sulfate by reaction with oxygen. The results of oxidation studies involving partially sulfided dolomite and oxygen indicate that limestone can only be partially sulfated (about 30%) while conversions levels of over 95% can be achieved with dolomite. The extent of sulfation depends not only on the sorbent type but also on the extent of sulfidation in the gasifier. Because of stringent requirements, one step oxidative stabilization involving limestone will definitely be ruled out, even with dolomite, one step oxidative stabilization process will prove to be insufficient for production of acceptable material for disposal. Under this scenario, the residual calcium sulfide can be removed from the solid wastes through the reaction with water and carbon dioxide.

This paper will address the results of a study directed toward determination of the optimum operating condition in the development of the Spent Sorbent Stabilization Process (SSSP) for conversion of calcium-sulfide containing solid wastes to environmentally acceptable material for disposal.