ABSTRACT

Mercury and selenium are among the most volatile trace elements found in trace quantities in Illinois coal. Major fractions of mercury and selenium can pass through particulate control devices and therefore require additional control. Application of sorbent injection capable of trapping the volatile metallic species in the particulate (sorbent) phase would facilitate their removal. Research was conducted in three phases over a twelve-month period; in Phase I we studied mercury capture in a bench-scale reactor in a simulated combustion environment; in Phase II, bench-scale experiments were conducted to investigate selenium capture in a simulated combustion environment; and in Phase III, effect of sulfur dioxide on control of mercury emissions in a pilot-scale furnace was studied.

In Phase I, bench-scale experiments were conducted to evaluate capture of elemental mercury (Hg) by activated carbons, FGD and PC-100, at 100 and 140 °C. In addition, capture of mercuric chloride (HgCl₂) by FGD and PC-100 was studied at 140 °C. In Phase II, capture of selenium (Se) by Ca(OH)₂ particles at 800 and 900 °C was studied in the airtoxics reactor. Pilot-scale studies (Phase III) were conducted to evaluate effect of sulfur dioxide concentration on mercury (Hg) capture at 140 °C by FGD and PC-100.
EXECUTIVE SUMMARY

The Air and Energy Engineering Research Laboratory (AEERL) of the United States Environmental Protection Agency (EPA) has proposed co-funded research in conjunction with the Illinois Clean Coal Institute (ICCI) to evaluate the reduction of volatile trace elements, especially mercury and selenium, by dry sorbent injection. Title III of the Clean Air Act Amendments (CAAAnA) places limitations on emissions of various air toxics. Among these, mercury and selenium, both present in Illinois coals, present the most serious challenge to control technologies because of their high volatility. For coal-fired utilities, reduction of these metals is critical in complying with the emission standards set forth by the CAAA. Injection of dry sorbents under suitable conditions is a possible option for controlling mercury and selenium emissions. Since both these metals are present in Illinois coals, the proposed research will provide control strategies for utilities using Illinois coals in reducing air toxics emissions.

The objectives of the proposed research are to conduct bench-scale studies to identify sorbent type and optimum reaction conditions for removing mercury and selenium in a simulated combustion environment. For mercury, control technology experience gathered at our laboratories and from Municipal Waste Combustors (MWCs) will be used to identify sorbents and capture conditions. The parametric investigation to be conducted in our laboratories with these sorbents will allow optimization of the process for mercury capture in coal combustors using Illinois coal. Studies involving mercury capture in bench-scale reactors will be conducted in Phase I. For selenium removal, the proposed research will test sorbents already in use for acid rain control technology and identify optimum process and sorbent injection parameters. This will be performed in Phase II. In Phase III, control mercury will be studied in a coal-fired pilot-scale furnace.

In Phase I, bench-scale experiments were conducted to evaluate capture of elemental mercury (Hg^0) by activated carbons, FGD and PC-100, at 100 and 140 °C. In addition, capture of mercuric chloride (HgCl_2) by FGD and PC-100 was studied at 140 °C. In Phase II, capture of selenium (Se) by Ca(OH)_2 particles at 800 and 900 °C was studied in the air toxics reactor. Pilot-scale studies (Phase III) were conducted to evaluate effect of sulfur dioxide concentration on mercury (Hg) capture at 140 °C by FGD and PC-100.
OBJECTIVES

The specific goals of the research are to conduct bench-scale studies to identify sorbent type and optimum reaction conditions for removing mercury and selenium in a simulated combustion environment and to verify these findings in a pilot-scale coal combustor. The tasks performed to meet the objectives are as follows:

- Perform bench-scale tests to study capture of different species of mercury - elemental and mercuric chloride - by activated carbons and calcium-based sorbents. The effect of process variables like reaction temperature and gas-solid contact time will be examined. These experiments will be conducted in Phase I. As a result of these tests, information on process optimization to achieve cost-effective removal of mercury will be obtained.

- Sorbents already in use for acid gas control will be examined for selenium control at different process and injection parameters. This is to constitute Phase II of the research.

- The results from Phases I and II will be used to effectively remove selenium and mercury from a pilot-scale unit fired by Illinois coal.

INTRODUCTION AND BACKGROUND

Metallic air toxics in flue gases are found in elemental or various oxidized forms and exist in the solid, aerosol, or vapor state. These compounds originate from the raw coal and are typically enriched in the fine particles (Markowski and Filby, 1985). The form and size distribution of each metal is a function of metal- and system-specific properties. For example, various concentrations of chlorine or sulfur has been shown to significantly affect the expected equilibrium product distribution (Linak and Wendt, 1993). Trace metals in flue gas streams are generally classified into three groups, based on their volatility and partitioning in the flue gas (Benson et al. 1993). Such a classification is not rigorous, and some species are placed in different classes by different references. Class I metals are the least volatile and are distributed between bottom ash and fly ash; Class II metals are more volatile, and are enriched in the fly ash; and Class III metals are the most volatile, and tend to escape with the vapor phase.

While particulate collection devices such as electrostatic precipitators and baghouses collect most of the metallic emissions, highly volatile metals (Class III) may form submicrometer aerosol fumes which are difficult to effectively collect. These submicrometer particles are particularly susceptible to human inhalation, resulting in significant health concerns. Mercury and selenium, both present in coal, are considered Class III metals, and hence are of highest concern because these two metals can be found at unacceptable levels in the
emissions from coal-fired utilities.

Without additional modifications, conventional flue gas desulfurization systems in MWCs are capable of removing mercury emissions to some extent (White et al., 1993). Limited tests performed by White and co-workers without specific control for mercury emissions showed a reduction in mercury emissions ranging from 24-76%.

Andren et al. (1975) reported that nearly 70% of the selenium from a coal-fired plant was associated with the fly ash particles while the remaining 30% was discharged into the atmosphere. Shendrikar et al. (1983) also reported that Se penetration through a utility baghouse was an order of magnitude higher than that of other metals. Gladney et al. (1976) found that more than 50% of the in-stack Se was found on particles less than 1μm in size, confirming its tendency to escape into atmosphere.

If a sorbent can react with Hg or Se compounds, then the metal will shift from aerosol phase to the large sorbent particle phase which can be collected effectively by a particulate control device.

To reduce mercury emissions in MWCs, injection of activated carbon has been shown to be a promising technology (White et al., 1993; Clarke, 1992). Nevertheless, several problems still exist, both economic and technical, concerning removal of mercury from flue gases.

The control of mercury emissions has been found to be strongly dependent on the form of mercury. Pilot-scale data (Schager, 1991), where activated carbon injection technology was used, showed the oxidized form to be more easily captured than elemental mercury (Hg\(^0\)). Similar results were observed at the Electric Power Research Institute's High Sulfur Test Center's (HSTC) 4 MW pilot wet FGD system and at the MWC Sysav tests (Volland, 1991).

The ability to control mercury emissions varies with operating conditions. For instance, the presence of sulfur dioxide (SO\(_2\)) has been associated with decreased mercury capture (Schager, 1991). The type of coal has also been found to affect different levels of mercury capture (Felsvang, 1992; 1993). The chlorine content in coal, capable of forming the less volatile and more water soluble mercuric chloride (HgCl\(_2\)), is thought to be the factor for differences in mercury capture (Felsvang et al., 1993; DeVito et al., 1993).

Unlike mercury, control of selenium has received almost no attention. Studies on Se capture is timely because Se is bound to evoke the same concerns as Hg from emission control view point.

For capture of metals, past pilot-scale work in the Pt's laboratory (Gullett and Raghunathan, 1993) studied the effect of various dry sorbent injection processes in the emissions of coal-based metallic air toxics, including mercury and selenium. These tests involved furnace injection of four sorbents: hydrated lime, limestone, kaolinite and bauxite. Flue gas was
sampling was carried out using a mercury train and a size-classifying metals train. Results for mercury are shown in Figure 1. Without sorbent injection (baseline) almost all the mercury fed (as trace in coal) ended up in the vapor phase. However, both the calcium-based sorbents are effective in capturing mercury, reducing their vapor phase emissions substantially. Sampled selenium distributions from this work, shown in Figure 2, show that without sorbent, a significant part of the selenium is in the vapor (post-filter) phase. Again, with hydrated lime and limestone, vapor phase emissions are reduced. For both mercury and selenium, kaolinite and bauxite had little effect. These results also indicate that, for metal capture, simply providing additional surface, through sorbent injection, for metal condensation is not sufficient; a chemical affinity between the sorbent and the metal is necessary. However, little is known on the effect of other reaction parameters such as sorbent injection temperature, gas phase species and composition and other kinetic variables.

Fundamental understanding of Hg capture by sorbents is essential in optimizing processes for mercury reduction. Bench-scale experiments have been performed to this extent to study Hg\textsuperscript{o} capture by different activated carbons in the PI's laboratories (Gullett and Jozewicz, 1993; Jozewicz et al., 1999; Krishnan et al., 1994). The results obtained from these studies indicate the importance of physical, structural, and chemical properties of the individual activated carbon. The other parameters of significance were the capture temperature and inlet Hg\textsuperscript{o} concentration. Figure 3 shows the influence of sorbent type and reaction temperature on the capture of Hg\textsuperscript{o} by different activated carbons. Currently, work is ongoing in the PI's laboratories to evaluate several sorbents, including activated carbons and lime, with respect to capture of HgCl\textsubscript{2}. Preliminary results again indicate the importance of reaction temperature and sorbent type on HgCl\textsubscript{2} reduction.

Although limited information is available regarding capture of Hg\textsuperscript{o} by activated carbons (work performed at the PI's laboratories), fundamental understanding of selenium capture is unavailable.

**EXPERIMENTAL PROCEDURES**

Selenium tests are conducted in a bench-scale flow reactor termed the "Airtox" reactor. As shown in Figure 4, the apparatus consists of a reactor tube assembly surrounded by three vertical Lindberg furnaces mounted one on top of the other, and connected to controllers. Each furnace is equipped with three independently controlled zones. The apparatus also consists of a selenium feed system, sorbent powder feeder, a 10-stage MOUDI micro-orifice cascade impactor, two sets of four impingers each and a vacuum pump.

The reactor is made up of 10-ft long concentric tubes placed vertically in a Lindberg Furnace. The process gas is primarily air, and for selected runs will also include HCl/SO\textsubscript{2} in air. The process gas enters the reactor at the bottom of the outer concentric tube and is
heated to the furnace temperature (600 - 1000 °C) before entering the inner tube, which serves as the reaction chamber. Aqueous metal solution is metered into an injection probe where it is atomized and injected into a quartz wool bed located within the hot inner tube of the reactor. A U-tube fluidized bed feeder feeds the sorbent to the reactor. The sorbent feed tube from the feeder passes through the center of the injection probe, and its tip is located downstream of metal injection (below the quartz wool bed), allowing sufficient distance for the injected metal to vaporize before contacting the sorbent. Sorbent and vapor are entrained in the gas stream and passed through the inner reaction tube. At the reactor outlet, the sorbent-laden gas stream is split into an impactor stream and a bypass stream. The impactor stream is mixed with cool air to quench the reaction and is then passed through a heat exchanger and the impactor assembly where spent sorbent particles are collected. The flow is pulled through a series of impingers (two charged with 10 percent H₂O₂/10 percent HNO₃, one blank, and one with silica gel) into the pump and vented into the hood. The bypass flow is also drawn through an identical set of impingers into the pump and the hood.

The impactor is an inertial particle classification system, which classifies the particles into 11 different aerodynamic cut sizes, ranging from 0.05 to 18 μm. Each stage has a substrate where the particles are deposited and the cut sizes are known from impactor calibration. After a typical run, individual substrates are digested in 40% nitric acid to dissolve all the calcium and selenium. Then, the solution is analyzed in an in-house Inductively Coupled Plasma Spectrometer (ICP) for both calcium and selenium.

Figure 5 shows the schematic of the bench-scale apparatus to be used in our tests to study Hg⁰ capture by sorbents. A carrier stream of pure N₂ passes through a heater (constant temperature water bath) containing a Hg⁰ source. Downstream, the N₂/Hg⁰ stream is mixed with a flow of SO₂ and air. The sorbent to be evaluated is placed in the reactor (either glass or teflon). Before the sorbent is exposed to the Hg⁰ laden stream, a baseline (flow through by-pass). Once the baseline is established, the flow is diverted through the reactor. We have also added an on-line SO₂ analyzer (not shown in the figure) to measure SO₂ concentrations.

For high concentrations of Hg⁰ (i.e., above the detection limit of the mercury analyzer of 3-4 ppb), continuous on-line analysis of Hg⁰ concentration is possible. Therefore, a simple mass balance of the Hg⁰ signal recorded by the personal computer yields the fraction of Hg⁰ captured by the sorbent with time. However, for low concentrations of Hg⁰ (as is mostly the case for coal combustion), batch operations are necessary since the mercury analyzer cannot accurately detect concentrations below 3 ppb Hg⁰. In batch operations (i.e., exposing the sorbent to Hg⁰ for a specific time), the sorbent is analyzed for total mercury using X-ray fluorescence (XRF). Here 3 stages of sorbent are used. The sorbent to be evaluated is placed in stage 1. All 3 stages are analyzed for total mercury. The mercury fraction retained by stage 1, φ₁, is then obtained from

\[ φ₁ = φ₁/(φ₁ + φ₂ + φ₃) \]

where φᵢ is the measured mercury concentration in stage i.
Control of HgCl₂ is studied in a separate apparatus. The principle of generation and detection are exactly that for Hg⁰ and are not repeated here.

The pilot-scale studies to investigate Hg capture in a coal-fired situation and also to test the effect of SO₂ concentration were conducted in the innovative furnace reactor (IFR). A schematic of the apparatus is shown in Figure 6. The sorbents are placed in stages 1, 2, and 3 and are exposed to a slip stream. Each stage was exposed to the flue gas stream for a specific duration. Both FGD and PC-100 were tested and the effect of SO₂ concentration on total Hg capture was examined at 140 °C.

RESULTS AND DISCUSSION

Figure 7 shows the capture of Hg⁰ (approximately 2 ppb in 1000 ppm SO₂, 3% O₂, and rest N₂ at inlet) by 100 mg of FGD and PC-100 at 100 and 140 °C after 8, 12, and 24 hours of exposure. At the higher temperature, FGD is seen to capture lower fraction of incoming Hg⁰, whereas, temperature has only a marginal effect on the capture of Hg⁰ by PC-100. At the higher temperature, PC-100 captures a higher percentage of incoming Hg⁰ compared to FGD. However, at the lower temperature, both FGD and PC-100 capture similar amounts.

Table 1 shows the effect of flue gas on Hg⁰ capture by the two activated carbons. The values without paranthesis are the values from Figure 7. The values in paranthesis are the capture efficiencies for the activated carbons when exposed to a stream of only N₂ and Hg⁰ (i.e., no O₂ and SO₂). In the presence of SO₂ and N₂, activated carbon FGD is seen to capture more of incoming Hg⁰. In contrast, presence of SO₂ and O₂ causes a decrease in the capture of Hg⁰ by activated carbon PC-100. The trend is true at both 100 and 140 °C.

Experiments were also conducted to study HgCl₂ capture in the presence of SO₂ and O₂ by PC-100. Two runs were performed to obtain HgCl₂ capture at 140 °C by 100 mg of PC-100. The HgCl₂ capture efficiency obtained in this study for the two runs were both approximately equal to 60%. The results therefore are reproducible. HgCl₂ capture obtained in the absence of SO₂ and O₂ was found to be roughly 40%. Therefore, PC-100 captures higher amounts of HgCl₂ in the presence of SO₂ and O₂.

Figure 8 shows the effect of sorbent [Ca(OH)₂] injection on Se capture. The two solid lines show baseline particle size distribution in the particle size classifier (i.e., no sorbent injection). When Ca(OH)₂ is injected, Se is seen to be captured by the particles. A similar trend was observed at 800 °C.

Table 2 shows the results obtained from our IFR studies to investigate Hg capture. The table compares the capture of Hg by PC-100 and FGD at 140 oC. It also shows the effect of SO₂ concentration on Hg capture. Since the conditions of these tests do not replicate those of the bench-scale tests (amount of carbon/ face velocity in the bed) these results should not be
compared quantitatively with earlier results. As can be seen from Table 2, FGD captures a higher fraction of incoming Hg in the presence of higher SO$_2$ concentration. Whereas, SO$_2$ concentration has very little effect on Hg capture by PC-100. From Table 1 (results at 140 °C), a similar trend is seen for FGD and for PC-100 exposed for 8h.

CONCLUSIONS AND RECOMMENDATIONS

The data in Figure 7 suggest that injecting similar amounts of activated carbon FGD or PC-100 at 100 °C would result in similar amounts of Hg$^0$ capture. At the higher temperature, however, activated carbon PC-100 is expected to capture more Hg$^0$ than FGD. In the presence of SO$_2$ and O$_2$, activated carbon FGD's performance is enhanced. These conclusions are based on the few results shown in this report and more testing is required (with other components of a typical flue gas) to establish this conclusion. Higher HgCl$_2$ was observed in the presence of higher SO$_2$ concentration by PC-100 in our bench-scale experiments Injection of Ca(OH)$_2$ was found to be capable of capturing vapor phase Se. This was seen at two injection temperatures - 800 and 900 °C.

The IFR studies showed that Hg capture by FGD improves in the presence of higher SO$_2$ concentration. On the other hand, SO$_2$ concentration had very little effect on Hg capture by PC-100. These results were in agreement with our bench-scale tests.

DISCLAIMER STATEMENTS

This report was prepared by Dr. Brian Gullett of the U.S. EPA with a grant made possible by the Illinois Clean Coal Institute. Neither Dr. Brian Gullett nor any of its subcontractors, nor the Illinois Clean Coal Institute, nor any person acting on behalf of either:

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REFERENCES


Figure 1. Effect of sorbents on mercury distribution (Gullett and Raghunathan, 1993).
Figure 2. Effect of sorbent injection on Se emissions (Gullett and Raghunathan, 1993).
Figure 3. Capture efficiencies of different activated carbons (Krishnan et al., 1994).
AIRTOX REACTOR

Figure 4. Schematic of "airtox" reactor used for selenium capture studies.
Figure 5. Schematic of bench-scale reactor used for Hg° capture studies
IFR SCHEMATIC

Figure 6. Schematic of IFR (innovative furnace reactor) used to study Hg capture.
Figure 7. Capture of Hg⁰ (2 ppb in 1000 ppm SO₂, 3% O₂, and rest N₂) by 100 mg of FGD and PC-100 at 100 and 140°C.
Figure 8. Effect of Sorbent injection (Ca(OH)₂) on vapor phase Se capture at 900°C.
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<tr>
<th>t(hr)</th>
<th>FGD</th>
<th>PC-100</th>
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<tr>
<td>1</td>
<td>100 °C</td>
<td>140 °C</td>
</tr>
<tr>
<td>2</td>
<td>64(46)</td>
<td>44(30)</td>
</tr>
<tr>
<td>3</td>
<td>72(55)</td>
<td>28(16)</td>
</tr>
<tr>
<td>4</td>
<td>62(25)</td>
<td>35(12)</td>
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Table 1. Effect of flue gas components SO₂ and O₂ on Hg⁰ capture by FGD and PC-100. Values in parenthesis are captures without SO₂ and O₂. Values without parenthesis have been obtained from Figure 6.

<table>
<thead>
<tr>
<th>Run #</th>
<th>Sorbent/Amt.</th>
<th>Flow Rate (g)</th>
<th>Flow Rate (cfm)</th>
<th>Time (min)</th>
<th>Temp. (°C)</th>
<th>SO₂ (ppm)</th>
<th>Hg (in) (ppb)</th>
<th>Capture (%)</th>
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<tbody>
<tr>
<td>1</td>
<td>FGD/25</td>
<td>0.56</td>
<td>577</td>
<td>139</td>
<td>350</td>
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<tr>
<td>2</td>
<td>FGD/25</td>
<td>0.72</td>
<td>270</td>
<td>151</td>
<td>1050</td>
<td>0.06</td>
<td>100</td>
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<tr>
<td>3</td>
<td>PC-100/25</td>
<td>0.63</td>
<td>320</td>
<td>138</td>
<td>350</td>
<td>0.2</td>
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<tr>
<td>4</td>
<td>PC-100/25</td>
<td>0.54</td>
<td>300</td>
<td>159</td>
<td>1050</td>
<td>0.92</td>
<td>41</td>
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Table 2. Capture of Hg by PC-100 and FGD obtained from IFR study.
PROJECT MANAGEMENT REPORT
June 1, 1995 through August 31, 1995

Project Title: **REMOVAL OF COAL-BASED VOLATILE TRACE ELEMENTS: MERCURY AND SELENIUM**

ICCI Project Number: 94-1/2.4A-1
Principal Investigator: Dr. Brian K. Gullett,
U.S. EPA
Other Investigators: Dr. S.V. Krishnan and Dr. K. Raghunathan
Acurex Environmental Corporation
Project Manager: Dr. Franklin I. Honea
Illinois Clean Coal Institute

COMMENTS

Tests for all Phases (I, II, and III) were completed as planned.
## Projected and Estimated Expenditures by Quarter

<table>
<thead>
<tr>
<th>Quarter*</th>
<th>Types of Cost</th>
<th>Direct Labor</th>
<th>Fringe Benefits</th>
<th>Materials &amp; Supplies</th>
<th>Travel</th>
<th>Major Equipment</th>
<th>Other Direct Costs</th>
<th>Indirect Costs</th>
<th>Total</th>
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<td>Estimated</td>
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<td>$0</td>
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<td>$0</td>
<td>$12,289</td>
<td>$3,557</td>
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* Cumulative by quarter
Cumulative Costs by Quarters

Removal of coal-based volatile trace elements: mercury and selenium

Projected Expenditures: $48,185
Estimated Actual Expenditures: $48,185
Total ICCI Award: $48,185
Schedule of Project Milestones

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<td>A</td>
<td>Complete Phase I of testing (Hg - Bench Scale)</td>
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<tr>
<td>B</td>
<td>Complete Phase II of testing (Se - Bench Scale)</td>
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<td>C</td>
<td>Complete Phase III of testing (IFR - Pilot Scale)</td>
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<td>D</td>
<td>Technical reports prepared and submitted</td>
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<td>E</td>
<td>Project management reports prepared and submitted</td>
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Comments:

All milestones completed