

Study of the Effect of Chlorine Addition on Mercury Oxidation by SCR Catalyst under Simulated Subbituminous Coal Flue Gas

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ABSTRACT

An entrained flow reactor is used to study the effect of addition of chlorine-containing species on the oxidation of elemental mercury (Hg^0) by a selective catalytic reduction (SCR) catalyst in simulated subbituminous coal combustion flue gas. The reactor system consists of a natural gas burner for producing combustion flue gas and an electrically heated glass reactor with honeycomb vanadia-titania SCR catalyst operating under NO_x reduction conditions. The combustion flue gas is doped with sulfur dioxide (SO_2), nitrogen oxides (NO_x), hydrogen chloride (HCl), reductant ammonia (NH_3), and trace Hg^0 ($20 \mu\text{g}/\text{Nm}^3$) to simulate subbituminous flue gas. Concentrations of Hg^0 and oxidized mercury (Hg^{2+}) upstream and downstream of the SCR catalyst are measured using a continuous emission monitor (CEM). Doping with chlorine (Cl_2), hydrochloric acid (HCl), or chlorine dioxide (ClO_2) is done during the Hg^0 oxidation tests. Cl_2 and HCl are shown to be effective for improving Hg^0 oxidation. ClO_2 is not found to be effective for improving Hg^0 oxidation. The results imply that heterogeneous interaction of the gaseous chlorine molecules with the SCR catalyst is important for Hg^0 oxidation over SCR catalyst. The effects of fly ash and temperature on Hg^0 oxidation over SCR catalyst under simulated PRB flue gas are also discussed.

INTRODUCTION

The speciation amongst chemical forms of mercury (Hg) species has a strong influence on the control and environmental fate of Hg emissions from coal combustion.¹ The high-temperature coal combustion process releases Hg in the elemental form (Hg^0). A significant fraction of the Hg^0 can be subsequently oxidized in the low-temperature, post-combustion environment of a coal-fired boiler. Relative to Hg^0 , oxidized Hg (Hg^{2+}) is more effectively removed by air pollution control systems (APCS). For example, the water-soluble Hg^{2+} is much more easily captured than insoluble Hg^0 in flue gas desulfurization (FGD) units.²⁻⁴ Initial European field tests showed that the selective catalytic reduction (SCR) technology employing vanadia-titania SCR catalyst, which is widely applied for reducing power plant nitrogen oxides (NO_x) emissions, also affects the speciation of Hg.⁵ Recent full-scale field tests conducted in the U.S. also showed increases in Hg oxidation across the SCR catalysts for plants firing bituminous coals with sulfur (S) contents ranging from 1.0 to 3.9%.⁶ However, field tests at plants firing subbituminous Powder River Basin (PRB) coals that contain significantly lower chlorine (Cl) and S content and higher calcium (Ca) content than those of the bituminous coals, showed very little change in mercury speciation across the SCR reactors.⁶ Our recent pilot-scale study also found high Hg oxidation across SCR catalyst in tests firing Illinois coals with different S (1.1 – 3.5%) and Cl (0.13 – 0.29%) content, and no significant increase in oxidation across the catalyst in tests firing a PRB coal with low S (0.3%) and Cl (< 0.01%) content under similar coal combustion and SCR operating conditions.⁷

The observed coal-type dependence effect on Hg oxidation occurring in SCR systems can be explained by our bench-scale studies which suggest that hydrogen chloride (HCl) in the flue gas provides a source of chlorine required for mercury oxidation occurring under SCR reaction conditions.⁸ Our follow up bench-scale study showed that very little Hg^0 oxidation occurs across an SCR system under PRB coal firing conditions and may likely be attributed to the low HCl concentrations from combustion of the low-Cl PRB coals.⁹ The addition of Cl species upstream of SCR catalyst systems to improve Hg^0 oxidation was suggested for further evaluation of this hypothesis.

A study to investigate the effect of adding additional gaseous Cl species in simulated PRB coal combustion flue gases on Hg^0 oxidation across the SCR catalyst is reported in this paper. In this project, an entrained-flow reactor was used for evaluating the effectiveness of adding different gaseous Cl species on Hg^0 oxidation under simulated PRB coal combustion flue gas NO_x control conditions utilizing an SCR system. Two different gaseous Cl-containing agents, including molecular chlorine (Cl_2) and chlorine dioxide (ClO_2), were tested; in addition direct enhancement by HCl addition was also evaluated. Several parameters such as the presence of the entrained PRB fly ash, the concentration of the added Cl species, and SCR operating temperature were evaluated to

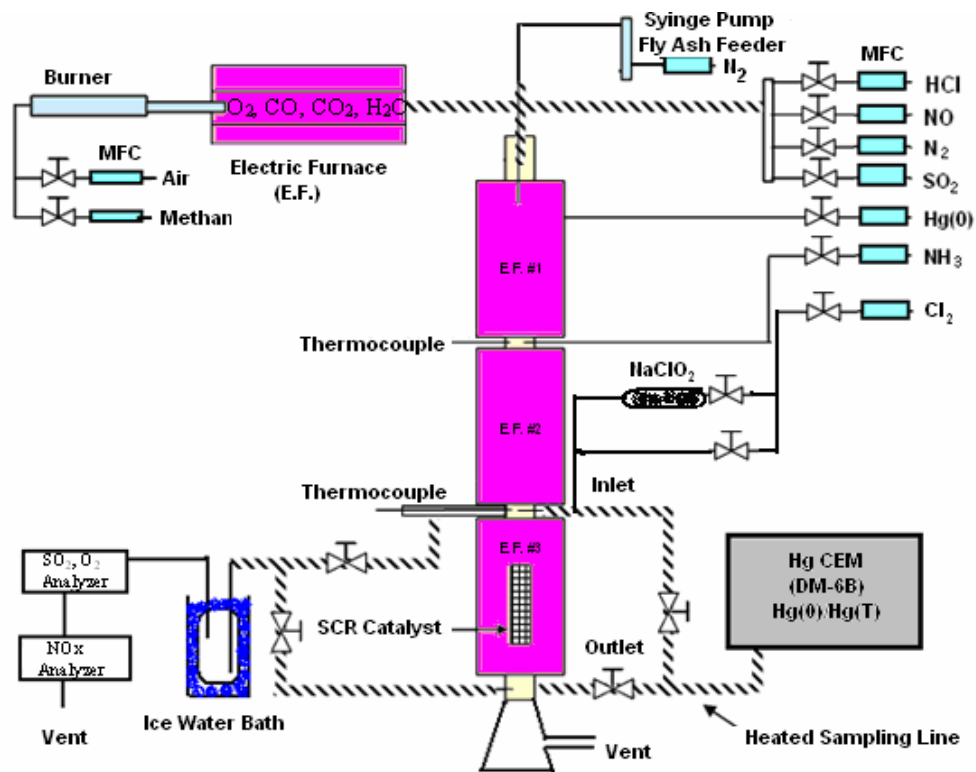
determine their effects on Hg^0 oxidation under simulated PRB conditions. The objectives of this project were to evaluate Hg oxidation and speciation under simulated PRB coal firing SCR system conditions and to gather insight into the effects of adding additional Cl species from an external source on the reaction mechanisms involved in mercury oxidation promoted by metal oxides in $\text{V}_2\text{O}_5 \cdot \text{WO}_3 / \text{TiO}_2$ (vanadium pentoxide and tungsten trioxide supported on titania) type SCR catalyst. The change of mercury speciation in the flue gas is of importance for applying the appropriate mercury control technology and predicting its mercury removal efficiency for PRB coal-fired boilers.

EXPERIMENTAL

Entrained-Flow SCR Reactor System

An entrained-flow reactor that had been used in our previous work for studying Hg^0 oxidation by an SCR catalyst under simulated PRB conditions⁹ was used in this project. Details of the reactor are available elsewhere.⁹ Fig. 1 shows a schematic drawing of the

Figure 1. Schematic diagram of the entrained-flow SCR reactor system



bench-scale reactor system. The reactor (310.5 cm in length and 4 cm inner diameter) was heated by three temperature-controlled electric furnaces to maintain SCR reaction temperature. Combustion flue gas including carbon monoxide (CO), carbon dioxide (CO₂), water vapor (H₂O), and oxygen (O₂) was produced by a methane gas burner. Other coal combustion flue gas components including SO₂, NO_x, and HCl were introduced into the top of the reactor to simulate PRB coal combustion flue gas. A constant quantity of Hg⁰ vapor generated by a permeation system (VICI Metronic Dynacalibrator model 190) was introduced near the top of the reactor. Ammonia (NH₃) gas was injected into the reactor upstream of the SCR catalyst, between the first and second heating zones of the reactor. Mass flow controllers (MFCs) were used for controlling all the gases which flowed into the reactor system.

A fly ash sample collected from a PRB coal-fired boiler was tested in this study. The alkaline PRB coal fly ash, which was also used in our previous studies,⁹ contains 31.5% calcium oxide (CaO), 25.2% silica, and 19.7% alumina as its major components. The fly ash was injected into the reactor at a controlled feed rate of 4.8 g/hr by using a syringe-type particle feeder and mixed with the flue gas at the top of the reactor. The fly ash loading (5.7 g/Nm³) is similar to the ash loading from full-scale PRB-fired boilers.

A fresh V₂O₅•WO₃/TiO₂ SCR catalyst sample, 322 mm in length with a square cross-section containing 9 channels (3x3 cell arrangement) with a pitch of 8.25 mm, was used in the tests. A total gas flow rate of 14 L/min with a calculated space velocity through the catalyst of 4000 hr⁻¹ was used, which is within the normal operating range of commercial SCR systems. The experimental conditions used for studying Hg oxidation under simulated PRB conditions are summarized in Table 1. The SO₂, NO_x and HCl

Table 1. Simulated PRB conditions used for studying Hg oxidation.

Parameters	Condition	Remark
Simulated Flue Gas		
HCl	10 ppm	Provided by gas cylinders.
SO ₂	500 ppm	
NO _x	200 ppm	
NH ₃	180 ppm	
CO ₂	3.5 %	Provided by methane burner.
CO	5 ppm	
O ₂	7.1 %	
H ₂ O	6.8 %	

Total Flow Rate	14 L/min	
NH ₃ /NO _x ratio	0.9	
Mercury Source	20 µg/Nm ³	Provided by Hg ⁰ permeation tube.
Fly Ash Feeding	4.8 g/hr	
Space Velocity	4000 hr ⁻¹	
Flue Gas Temperature	350 °C	

concentrations used in this project are those typical levels for a PRB coal flue gas. An online ultraviolet (UV) SO₂ and O₂ analyzer (Bovar Engineering, Inc. model 721AT2) was used for measuring SO₂ and O₂ near the inlet and outlet of the SCR catalyst zone. An online chemiluminescent NO_x analyzer (Advanced Pollution Instrumentation, model 200AH) downstream of the SO₂ and O₂ analyzer was used for measuring NO_x. No online analyzers were available for measuring NH₃ and HCl in the flue gas.

Gaseous Cl-containing agents, Cl₂ and ClO₂, were injected separately during the chlorine addition tests. The agent was injected near the inlet of the SCR catalyst, between the second and third heating zones of the reactor. Since ClO₂ is not sufficiently stable to be stored long term, it was generated in-situ during the ClO₂ injection tests. The ClO₂ was produced using a method¹⁰ based on the reaction of sodium chlorite (NaClO₂) with Cl₂: 2NaClO₂ + Cl₂ = 2ClO₂ + 2NaCl. To generate the desired amount of ClO₂ (20 ppm) for the ClO₂ addition tests, ~400 g of NaClO₂ flakes were placed inside Teflon tubing (15 ft long, 3/8 in inner diameter) through which 500 ppm of dry Cl₂ (in nitrogen) gas was passed continuously. More than 92% conversion of Cl₂ to ClO₂ was determined by the standard titration method for ClO₂ measurement.¹¹

An Hg continuous emission monitor (CEM) was used for measuring Hg speciation in the simulated coal combustion flue gas near the inlet and outlet of the SCR catalyst zone. The Hg CEM (Nippon Instrument Corporation, model DM-6B) measures both elemental and total gaseous mercury [Hg(T)] in combustion flue gas. It uses a dry reduction catalyst to reduce Hg²⁺ in the flue gas to Hg⁰. Details of the Hg CEM are described in our previous studies.⁹ The Hg²⁺ concentration is calculated by the difference between Hg(T) and Hg⁰ using the following simple formula:

$$Hg(T) - Hg^0 = Hg^{2+}$$

Where, Hg(T) is total Hg concentration in the flue gas (µg/m³), Hg⁰ is elemental Hg concentration in the flue gas (µg/m³).

$$Hg^{2+} \text{ percentage in flue gas (\%)} \text{ at location } y = \frac{Hg(T)_y - Hg^0_y}{Hg(T)_y} \times 100$$

$Hg(T)_y$ is total Hg concentration at location y (either the inlet or the outlet of the SCR zone). Hg^0_y is the elemental Hg concentration at the same location (inlet or the outlet). The CEM has a measurement range of 0 – 50 $\mu\text{g}/\text{m}^3$ with an accuracy of $\pm 0.5 \mu\text{g}/\text{m}^3$. In addition, this Hg CEM has a self-calibration function.

Test Procedures

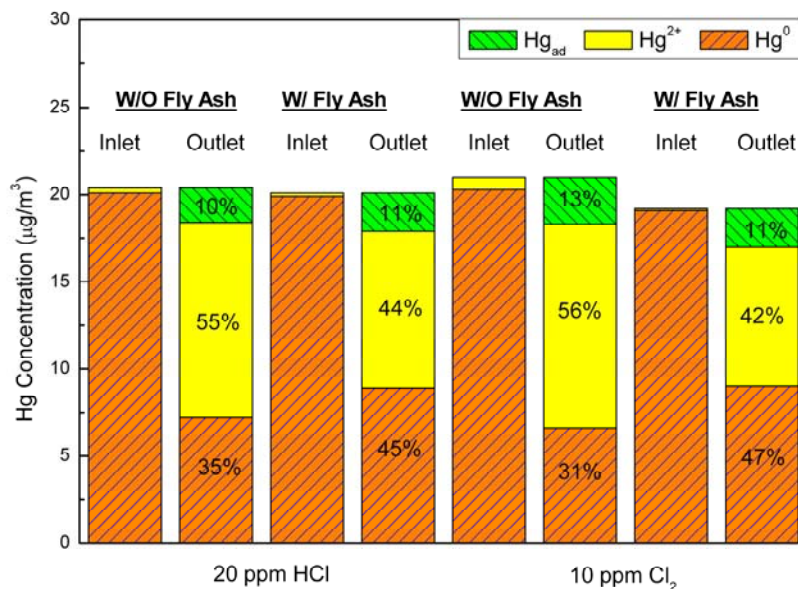
The SCR catalyst was heated overnight at the desired experimental temperature of 350 or 400 °C under air flow prior to the Hg speciation experiment. This cleanup procedure was followed before each test for removing the adsorbed species such as Hg to minimize any residual effect caused by the previous test. The methane burner was first turned on the next morning. Then, the flows of all other gas flue gas components including HCl, NO, NH₃, SO₂, Cl agent (Cl₂ or ClO₂), and Hg⁰ were simultaneously sent to the reactor. The N₂ flow was adjusted to get a total flow of 14 L/min through the reactor. The concentrations of all flue gas components were well controlled by the mass flow controllers (MFCs) to the predetermined values as shown in Table 2. The inlet Hg concentrations, Hg(T) and Hg⁰, were first measured with the Hg CEM. Then valves were diverted to the outlet sampling location in order to continuously monitor the changes of outlet mercury concentrations. After the outlet Hg concentrations reached steady state, the Hg speciation test was started. Fly ash was injected using a syringe-type feeder. The injection was continued for ~1 hour, and the concentrations of Hg(T) and Hg⁰ at the outlet of the SCR catalyst zone were measured and recorded continuously. The NO_x concentration was measured at the inlet at the beginning of the Hg speciation test, and the outlet NO_x concentration was measured continuously during the test.

RESULTS AND DISCUSSION

Importance of Chlorine Source for Promoting Hg Oxidation in SCR

Initial tests were conducted to investigate the effect of using Cl₂ and HCl as a chlorine source for promoting Hg oxidation by an SCR system. Two tests for injecting 20 ppm of HCl as the only chlorine source with and without fly ash injection were performed at a temperature of 350 °C. Another two tests for injecting 10 ppm Cl₂ as the only chlorine source (without HCl injection) were also performed. Results of the tests are shown in Fig 2. Generally less total mercury, Hg(T), was measured at the SCR catalyst outlet

Figure 2. Speciation of Hg at the Outlet of SCR with HCl or Cl₂ as the Chlorine Source



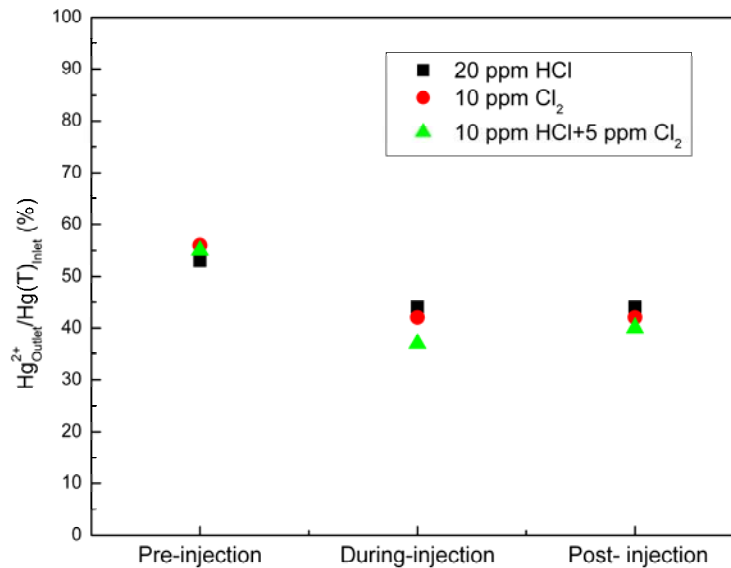
compared to that measured at the inlet (about 10 – 13%), which is likely caused by partial capture of Hg by the SCR catalyst. The difference of Hg(T) measured in the inlet compared to that measured in the outlet is considered mercury being retained or adsorbed by the catalyst (Hg_{ad}). The generally lower total Hg measured at the SCR outlet compared to those measured at the inlet were observed in full-scale field studies⁶ as well as in pilot-scale⁸, and bench-scale^{7,9} studies. Results shown in Fig. 2 indicate that Cl₂ and HCl are equally effective for promoting Hg⁰ oxidation by an SCR catalyst at the conditions of these tests. Both cases had the same amount of total Cl (20 ppm) in the simulated flue gases. For the case of 20 ppm HCl without fly ash injection, about 55% of the Hg injected into the SCR reactor system was converted into Hg²⁺. The conversion of Hg²⁺ decreased to 44% with fly ash injection. For the case of 10 ppm Cl₂, the conversions of Hg²⁺ (56% without fly ash injection, 42% with fly ash injection) were similar to those of the HCl cases. The approximately 10 percentage point reduction in Hg²⁺ conversion with fly ash injection compared to that without fly ash injection for both HCl and Cl₂ cases was also observed in our previous studies.⁹

Effect of Fly Ash Injection

The effect of fly ash injection on Hg oxidation promoted by an SCR catalyst was investigated by running the first set of tests at 350 °C, using a fresh catalyst without fly ash injection first, followed by the second set of tests with fly ash injection, then repeating the tests without fly ash injection. One of three different sources of chlorine

(either 20 ppm HCl, 10 ppm Cl₂, or 10 ppm HCl plus 5 ppm Cl₂), each with a total Cl content of 20 ppm in the flue gases, was used in each test. The test results are presented in Fig. 3. The results indicate that consistent levels of Hg oxidation of about 52 – 56%

Figure 3. Effect of Fly Ash on Hg Oxidation Promoted by an SCR Catalyst

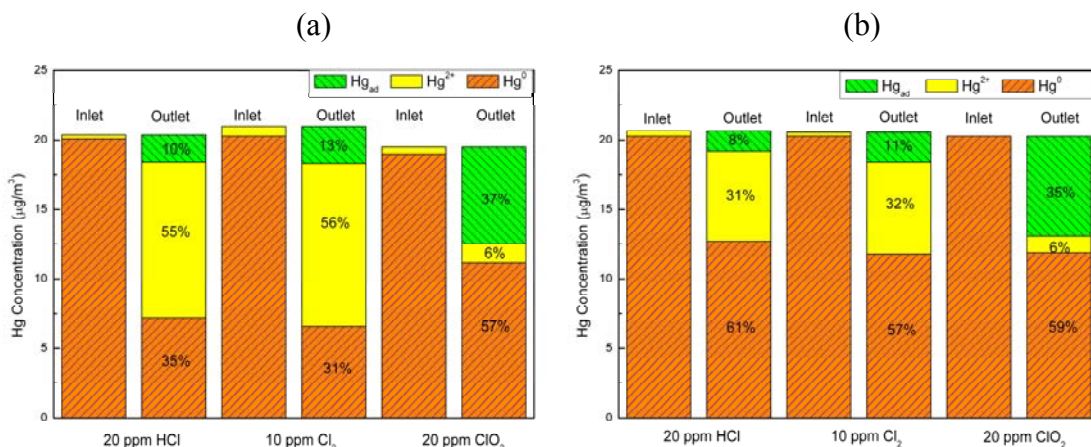


were achieved for the three tests before the catalyst was ever exposed to fly ash. Hg oxidation levels were reduced to 39 – 44% as fly ash was injected into the SCR reactor and the catalyst was exposed to the fly ash. After fly ash was first injected into the reactor system, subsequent tests, without fly ash injection, produced Hg oxidation levels (41 – 46%) similar to those of the previous tests with fly ash injection. It should be noted that repeated physical cleaning of the SCR catalyst by passing compressed air over the catalyst after fly ash injection tests were performed did not restore the Hg oxidation activity of the catalyst back to its original higher values prior to the first fly ash injection test. One potential explanation is that some of the fine fly ash particles were retained within the catalyst pores. This would reduce the diffusion rates of bulky Hg species within the porous catalyst for subsequent tests after the first test with fly ash injection. The potential retention of fly ash within the catalyst may explain the consistent decrease in outlet Hg²⁺ as a percentage of inlet Hg(T) that was observed in subsequent Hg speciation test runs. It should be noted that the outlet NO_x concentration remained at a nominal level of 30 – 35 ppm for all test runs and there was no effect on the extent of NO_x reduction. The concentration of NO_x (200 ppm) was much higher than that of Hg⁰ (< 1 ppb) used in the tests. Apparently the change in reactivity of the SCR catalyst resulting from fly ash retention was not significant to reduce NO_x reduction.

Effect of Temperature

Additional tests using HCl and Cl₂ as the chlorine source were performed at 400 °C for evaluating the effect of temperature on Hg oxidation promoted by an SCR catalyst. Tests were also conducted to evaluate ClO₂ as the chlorine source for Hg oxidation promoted by an SCR catalyst. The test results are summarized in Fig. 4. The results indicate a

Figure 4. Speciation of Mercury at the SCR Outlet using Three Different Chlorine Sources at (a) 350 °C and (b) 400 °C



significant temperature effect on Hg oxidation promoted by the SCR catalyst when using HCl and Cl₂ as the chlorine source. For the HCl case, the outlet Hg²⁺ level decreased from 55 to 31% as temperature increased from 350 to 400 °C. A similar decrease in outlet Hg²⁺ level as a result of increasing temperature was also observed for the Cl₂ tests. The decrease in net oxidation of Hg⁰ to Hg²⁺ with the increase in SCR operating temperature suggests that the oxidation occurs heterogeneously on the catalyst surfaces.

Results shown in Fig. 4 suggest that ClO₂ has little effect on promoting Hg oxidation. An insignificant outlet Hg²⁺ level of 6% was measured for the tests using ClO₂ as the chlorine source at both 350 and 400 °C. However, significant retention of Hg by the catalyst seems to be occurring for these two tests. The retained mercury (Hg_{ad}) was estimated to be 37 and 35% for the tests conducted at 350 and 400 °C, respectively. For the HCl and Cl₂ tests which show significant Hg⁰ oxidation, the Hg_{ad} levels are at much lower levels (8 – 13%) than those for the ClO₂ tests. It appears that the significant Hg capture is related to the lack of Hg oxidation observed for the two ClO₂ tests, and it is possible transient accumulation does not allow true steady-state oxidation to be observed during the two ClO₂ tests. Our previous studies show that significant Hg capture (45-50%) occurs without Hg oxidation at 0 ppm HCl, and the capture approaches zero with

significant Hg oxidation (>60%) as HCl concentration is increased to 20 ppm.⁹ The significant loss of Hg(T) at the outlet of an SCR catalyst in the absence of HCl for simulated PRB conditions was first noted in another study.⁸

The opposite trends of increased outlet Hg²⁺ level and decreased degree of Hg capture with respect to increased HCl concentration provide further evidence that HCl plays an important role by providing a source of reactive chlorine in the Hg chemistry under SCR NO_x reduction conditions. It appears that Hg⁰ oxidation does not proceed without a source of reactive chlorine, and both HCl and Cl₂ appear to provide this source. The chlorine, in the three chlorine-containing compounds tested, is in different oxidation states (1- for HCl, 0 for Cl₂, and 4+ for ClO₂). Although ClO₂ has the highest oxidation potential among the three compounds, it does not seem to provide a source of reactive chlorine for promoting Hg⁰ oxidation under SCR NO_x reduction conditions. The interaction between the SCR catalyst and the chlorine source through heterogeneous mechanisms may be important for promoting Hg oxidation. Both HCl and Cl₂ are diatomic, and they have more simple molecular structures and smaller molecular sizes than those of the tri-atomic ClO₂. Slightly slower diffusion of the larger ClO₂ molecules within the catalyst is not likely to be responsible for the observed effects. Perhaps ClO₂ is being more strongly bound to the catalyst than HCl and Cl₂, possibly as an oxychloride complex with Hg. Unfortunately the outlet Cl concentration was not monitored during the tests so the potential accumulation of Cl on the catalyst could not be determined. Another potential explanation is that the apparent absence of reactive chlorine in the ClO₂ tests may also make more active sites on the SCR catalyst surface available for capturing Hg⁰, and resulting in higher Hg_{ad}.

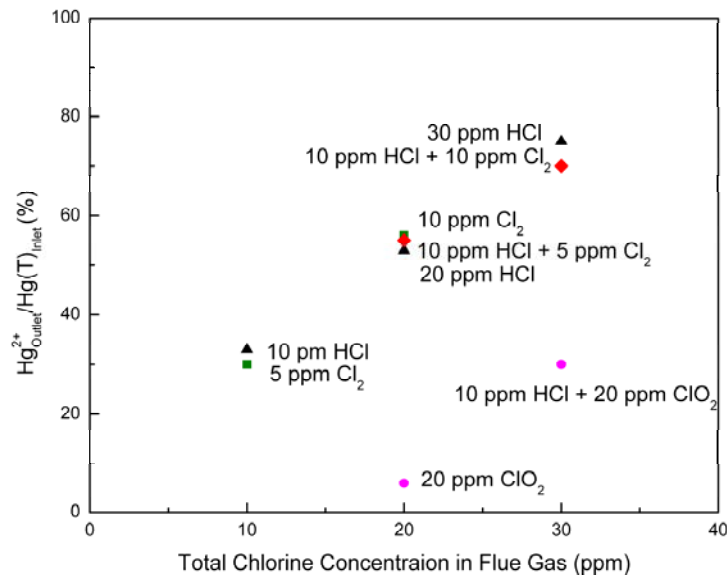
It has been suggested that V₂O₅, a major component of the SCR catalyst, is reactive for the adsorption of HCl to produce vanadium oxychloride complexes such as V₂O₃(OH)₂Cl₂ and VO₂Cl.¹² Further reaction of such complexes with Hg⁰ might produce the oxidized Hg²⁺ species. It should be noted that three tests for HCl, Cl₂, and ClO₂ as the chlorine sources (at a total Cl content of 20 ppm in the flue gas) were also performed without the SCR catalyst, and no Hg²⁺ was measured at the outlet of empty reactor for all three tests. Such observations provide another indication that Hg oxidation observed from the HCl and Cl₂ tests were occurring through heterogeneous mechanisms.

Effect of Adding Additional Chlorine on Hg Oxidation

A series of tests were conducted to evaluate the effect of adding additional chlorine to the simulated PRB flue gas on improving Hg oxidation in an SCR system. The simulated PRB flue gas which contains a baseline HCl concentration of 10 ppm was used in the tests. Higher chlorine levels, 20 and 30 ppm, than the baseline (10 ppm) were used in the tests by injecting Cl₂ or ClO₂. Tests were also performed by adding more HCl than the

baseline level for evaluating the injection of more HCl for improving Hg oxidation in PRB flue gas. The test results are presented in Fig. 5. A low level of 31 – 33% outlet Hg^{2+} was observed for the baseline case of 10 ppm HCl in the simulated PRB flue gas. A similar level of outlet Hg^{2+} was also observed in our previous tests of a PRB coal with an estimated HCl concentration of 8 ppm in the flue gas in a pilot-scale SCR system.⁷ Except for ClO_2 , the addition of more chlorine to the baseline level of chlorine has a strong effect for increasing Hg oxidation. It is interesting to note that Hg oxidation level seems to increase linearly with the total chlorine content in the flue gas. For example, adding 5 ppm of Cl_2 to the baseline level of 10 ppm HCl raises Hg oxidation from 33 to 55%. A similar level of Hg oxidation can also be achieved by either adding 10 ppm Cl_2 to the baseline case or by increasing the HCl concentration from the baseline case to 20 ppm. An increase in total chlorine content in the flue gas to 30 ppm by either adding

Figure 5. Effect of Chlorine Addition on Hg Oxidation



10 ppm Cl_2 to the baseline case or by increasing the HCl concentration to 30 ppm, raises the outlet Hg^{2+} level to 71 – 75%. However, after adding 20 ppm of ClO_2 to the baseline case, outlet Hg^{2+} still remained at the baseline level of 31%.

CONCLUSION

A bench-scale reactor system has been used to study the effect of chlorine addition on Hg oxidation promoted by a vanadia-titania SCR catalyst with simulated PRB combustion flue gas. Trace quantities (0 – 20 ppm) of gaseous chlorine (Cl_2), HCl, and chlorine dioxide (ClO_2) were added separately near the inlet of the SCR reactor during the Hg

oxidation tests. It was observed that Cl₂ and HCl were equally very effective for improving Hg⁰ oxidation. However, ClO₂ doesn't seem to provide a reactive chlorine source for improving Hg⁰ oxidation under SCR NO_x reduction conditions. It appears that the interaction between the chlorine source and the SCR catalyst is occurring through heterogeneous mechanisms to produce the reactive chlorine needed for Hg⁰ oxidation. Transient accumulation of Hg on the catalyst was observed during tests with ClO₂ addition. A significant reduction in Hg oxidation with increasing SCR operating temperature was observed for the HCl and Cl₂ addition tests. Such observations provide further evidence that complex interaction between the chlorine source and the catalyst through heterogeneous mechanisms occur during Hg oxidation over SCR catalyst.

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KEY WORDS

Mercury speciation, selective catalytic reduction, coal combustion, elemental mercury, oxidized mercury, hydrogen chloride, chlorine, chlorine dioxide