Effects of HCl and SO$_2$ Concentration on Mercury Removal by Activated Carbon Sorbents in Coal-Derived Flue Gas[†]

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The effect of the presence of HCl and SO$_2$ in the simulated coal combustion flue gas on the Hg^0 removal by a commercial activated carbon (coconut shell AC) was investigated in a laboratory-scale fixed-bed reactor in a temperature range of 80−200 °C. The characteristics (thermal stability) of the mercury species formed on the sorbents under various adsorption conditions were investigated by the temperature-programmed decomposition desorption (TPDD) technique. It was found that the presence of HCl and SO$_2$ in the flue gas affected the mercury removal efficiency of the sorbents as well as the characteristics of the mercury adsorption species. The mercury removal rate of AC increased with the HCl concentration in the flue gas. In the presence of HCl and the absence of SO$_2$ during Hg^0 adsorption by AC, a single Hg^0 desorption peak at around 300 °C was observed in the TPDD spectra and intensity of this peak increased with the HCl concentration during mercury adsorption. The peak at around 300 °C may be derived from the decomposition and desorption of mercury chloride species. The presence of SO$_2$ during mercury adsorption had an adverse effect on the mercury removal by AC in the presence of HCl. In the presence of both HCl and SO$_2$ during Hg^0 adsorption by AC, the major TPDD peak temperatures changed drastically depending upon the concentration of HCl and SO$_2$ in flue gas during Hg^0 adsorption.

1. Introduction

Coal produces a wide variety of pollutants when it is burnt. A range of technologies are being developed to reduce the environmental impact of coal-fired power stations. Atmospheric emission of elemental mercury from the flue gas of coal-fired power plants remains a major environmental issue that has drawn considerable attention in recent years. Thus, the development of an efficient method for the removal of Hg^0 from the flue gas of coal-fired power plants is of great importance. It has been reported that solid sorbents, particularly activated carbons, can remove elemental mercury from flue gases.[1,2] However, activated carbons also adsorb other gases, which may reduce the adsorption capacity of the activated carbon for mercury. Generally, activated carbons are modified chemically with various chemicals, such as sulfur, iodine, and chlorine, to improve the adsorption capacity of the sorbent.[3,4] Unfortunately, this increases the cost of activated carbon significantly. Therefore, development of

mercury removal processes that use cheaper sorbents is a necessity for practical application.

Flue gas composition has significant effects on the adsorption of Hg^0 on carbon-based sorbents.[5−9] Millers et al. have conducted the mercury breakthrough test with simulated flue gas on lignite-based activated carbon (LAC) in a laboratory-scale reactor at about 100 °C. When the sorbent was exposed to SO$_2$ (mixture of O$_2$, CO$_2$, N$_2$, and H$_2$O) in addition to baseline gas, LAC sorbent capture improved slightly. Upon exposure of the sorbent to the HCl-, NO-, or NO$_2$-added one at a time to the baseline gases, the mercury capture improved to 90−100%. However, the interaction between SO$_2$ and NO$_2$ caused a rapid decrease in mercury capture capacity of the sorbent.[5] Recently, Presto et al. have reported that mercury capture with activated carbon injection was suppressed in flue gases containing high concentrations of sulfur oxides (SO$_x$). The final mercury content of the activated carbons was independent of the SO$_2$ concentration in the SFG, but the presence of SO$_3$ inhibits mercury capture even at the lowest concentration tested (20 ppm).[6,7] Bench-scale testing of Hg^0

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sorption on selected activated carbon sorbents was conducted by Olson et al.[8] to develop a better understanding of the interaction among the sorbents, flue gas constituents, and Hg[0]. They explained the role of acid gases in simulated flue gas on mercury capture with X-ray photoelectron spectroscopy (XPS) of the sorbents: a competition between the bound hydrogen chloride (HCl) and increasing sulfur [S^{VI}] for a basic carbon-binding site affected mercury removal performance of the sorbents. Huggins et al. studied the mercury-adsorbed species on various carbonaceous sorbent materials. Their data from S and Cl X-ray absorption near edge structure (XANES) spectra, as well as from the Hg X-ray absorption fine structure (XAFS) data, strongly support the hypothesis that the interaction of acidic species (HCl, HNO_3, H_2SO_4, etc.) in the flue gas with the sorbent surface is an important mechanistic process that is responsible for creation of active sites for mercury capture by chemisorption.[9]

We have been developing an effective mercury-removal process in our laboratory for both coal combustion flue gas and coal gasification fuel gas using a flow-type fixed-bed reactor. This procedure does not simulate/predict mercury adsorption/capture when the AC is injected upstream of an electrostatic precipitator (ESP) because there is no or limited build up of a filter cake. However, in our study, we mainly consider that the process where mercury could be captured before desulfurization equipment in a packed-bed flow system. To remove before ESP, a honeycomb-type adsorption device could be used. Previously, we have reported that iron oxides are very effective in removing elemental mercury from coal gasification fuel gas in the presence of H_2S.[10−12] In this process, elemental mercury is removed as HgS on iron oxide sorbent. It has also been reported that, under certain conditions, elemental mercury can be removed from coal combustion flue gases effectively in the presence of H_2S and SO_2 over activated carbon.[13,14] The partial oxidation of H_2S with O_2 to elemental sulfur and the Clause reaction may contribute to Hg[0] removal over activated carbon. We have also suggested that Hg[0] is adsorbed on the activated carbon as $HgSO_4$ in the presence of SO_2, H_2O, and O_2 in the flue gas.[15]

Although the effects of acid gases in simulated flue gas on mercury capture with the sorbents have been studied extensively, the complex interactions of these gases on Hg capture on the sorbents are not well-understood yet. In this work, we studied the effect of acid gases, such as HCl and SO_2, on the Hg[0] removal performance of activated carbons obtained from coconut shell AC in a coal combustion flue gas system. The Hg[0] removal process in simulated coal combustion flue gases containing HCl, SO_2, CO_2, H_2O, and O_2 was studied using a laboratory-scale reactor system. The characteristics (thermal stability) of the mercury species formed on the sorbents under various adsorption conditions were investigated by the temperature-programmed decomposition desorption (TPDD) technique.

2. Experimental Section

2.1. Activated Carbon Sample. Activated carbon produced from coconut shell (AC) was purchased from Wako Pure Chemical Co. Ltd. The granular AC particles were crushed and sieved into an average diameter of 1 mm. The surfaced area of the sample was measured by a conventional N_2 adsorption method (Micromeritics Gemini 2375). The coconut shell AC had a specific surface area of 1250 m^2/g and a surface area per unit packed volume of 472 m^2/cm^3.

2.2. Apparatus and Procedure. The mercury vapor (Hg[0]) removal performance of the activated carbon adsorbent was carried out in a fixed-bed flow-type reactor. A detailed description of the experimental setup and procedure is given elsewhere.[12] The test rig consisted of a mercury permeation device, a simulated coal combustion flue gas feed system, a tubular reactor (size of 6.0 mm) with an electric furnace, and a mercury analysis system. The mercury permeation device, designed to deliver a controlled amount of Hg[0], is an array of liquid mercury containing u-tubes connected in series immersed in an ice−water bath. A controlled amount of nitrogen gas is introduced into the inlet of the horizontally laid mercury containing u-tubes, which carry trace amounts of elemental vapor, and the mercury concentration can be varied by varying the nitrogen flow rate. The model coal combustion flue gases were simulated by mixing the stream of N_2 carrying Hg[0] and other gas streams before entering the reactor. All lines of feed gases after H_2O injection were heated to 100 °C with a tape heater. Prior to the Hg[0] removal test run, about 0.125 cm^3 of the activated carbon sample (size of 1 mm) was packed into the quartz tube reactor. The Hg[0] removal efficiency of the sample was evaluated at atmospheric pressure in a temperature range of 80−200 °C using a simulated gas mixture containing Hg[0] (4.9 ppb), SO_2 (0−900 ppm), HCl (0−200 ppm), CO_2 (10%), H_2O (16%), O_2 (5%), and N_2 (balance gas) at a gas flow rate of 500 cm^3 STP/min (SV of 2.4×10^5 h^{-1}) in a quartz tube reactor. Measurement of the inlet and outlet concentration of mercury was carried out using a cold vapor mercury analyzer (atomic absorption spectrophotometer, Shimadzu). The mercury removal efficiency of the sorbents was quantified by comparing the Hg[0] contents of the gas before and after adsorption.

When the effect of HCl in the flue gas was investigated, the reactor effluent was passed through either a trap containing an aqueous solution of $SnCl_2$ or a trap containing deionized H_2O only. $SnCl_2$ can reduce Hg^{2+} to Hg[0]; therefore, when the reactor effluent passes through the aqueous solution of $SnCl_2$, the total concentration of Hg^{2+} and Hg[0] can be measured. On the other hand, when it passes through the H_2O trap, only the Hg[0] concentration can be measured. We measured the mercury removal efficiency at 0.5, 1, 2, and 3 h time on stream by passing the reactor effluent through a trap containing an aqueous solution of $SnCl_2$ and at 2.5 h time on stream by passing through a trap containing deionized H_2O to check the presence of oxidized mercury in the reactor outlet gas after Hg removal. However, we confirmed that unsorbed oxidized mercury species were not formed downstream of the bed in this system.

2.3. TPDD of Mercury-Adsorbed Sorbents. After Hg[0] removal experiments, the TPDD experiments were carried out in the same fixed-bed flow-type reactor as the Hg[0] removal experiments and the desorbed mercury species were monitored with an atomic absorption spectrophotometer. In the TPDD experiments, about 0.125 cm^3 of the mercury-adsorbed

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activated carbon sample was packed in a quartz tube reactor and N_2 was flowed in the reactor at a rate of 250 cm^3 STP/min. Then, the sample was heated from room temperature to 550 °C at a heating rate of 10 °C/min, and the reactor effluent was monitored continuously during TPDD for Hg^0 with the atomic absorption spectrophotometer after passing the reactor effluent through an aqueous $SnCl_2$ and H_2O_2 solution scrubber to convert the oxidized mercury (if any formed) to elemental mercury and to remove SO_2 in the effluent gas, respectively. Also, in these cases, we did not observe the unsorbed oxidized mercury species downstream of the bed.

3. Results and Discussion

3.1. Effect of the HCl Concentration on the Hg^0 Removal by AC.

Figure 1 shows the effect of the HCl concentration on the Hg^0 removal by AC in the presence of 0−200 ppm HCl and the absence of SO_2 at 80 °C. In the absence of both HCl and SO_2, Hg^0 removal efficiency of AC was about 0%. However, the presence of 1 ppm HCl enhanced the Hg^0 removal performance of AC drastically to 90%. A further increase in the HCl concentration resulted in almost 100% removal of Hg^0 in all cases.
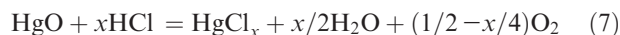
Figure 2 shows the TPDD spectra of the spent sorbents used in Hg^0 removal in the experiments of Figure 1. As can be observed in Figure 2, the Hg^0 desorption peaks at around 300 °C increased with the increase of the HCl concentration. In this case, no other peaks were observed. Therefore, these results indicate that the TPDD peak at around 300 °C is related to desorption of Hg^0 from mercury chloride species.

Therefore, it is evident that the presence of HCl in the flue gas system played an important role in the Hg^0 removal by AC. In the presence of HCl but in the absence of SO_2, elemental mercury may be captured on the AC surface through two major routes: a direct reaction to HgCl$_x$ and a reaction via HgO. Reactions 1−234567 are heterogeneous reactions that could occur on the AC surface.

Direct reaction to HgCl$_x$:

$$Hg + xHCl = HgCl_x + x/2H_2 \tag{1}$$

$$H_2 + 1/2O_2 = H_2O \tag{2}$$

$$2HCl + 1/2O_2 = Cl_2 + H_2O \tag{3}$$

$$Hg + x/2Cl_2 = HgCl_x \tag{4}$$

Reaction via HgO:

$$Hg + 1/2O_2 = HgO \tag{5}$$

$$2HgO + xCl_2 = 2HgCl_x + O_2 \tag{6}$$

$$HgO + xHCl = HgCl_x + x/2H_2O + (1/2 - x/4)O_2 \tag{7}$$
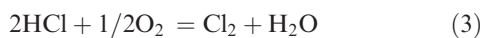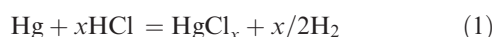
It was confirmed by mercury extraction experiments with various solvents that $HgCl_2$ was not formed in the free state on AC in the presence of HCl and the absence of SO_2. The extraction experiments were carried out using deionized water, hydrochloric acid, and aqua regia as solvents. If $HgCl_2$ was formed on AC in the free state, it should be dissolved in water. However, mercury was not observed in the extracted water. Therefore, we found that at least free $HgCl_2$ did not exist in the spent AC in this case, which is in the presence of HCl and the absence of SO_2. At present, we
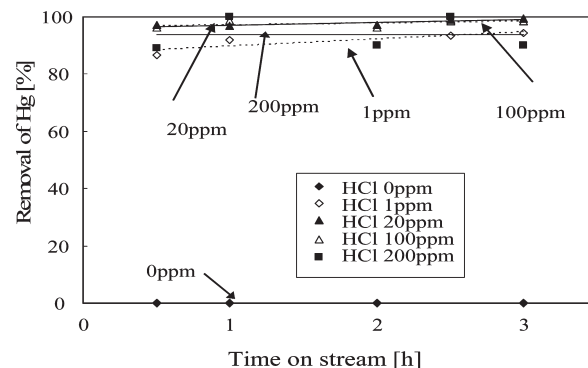

Figure 1. Effect of the HCl concentration on Hg^0 removal in the absence of SO_2 at 80 °C.
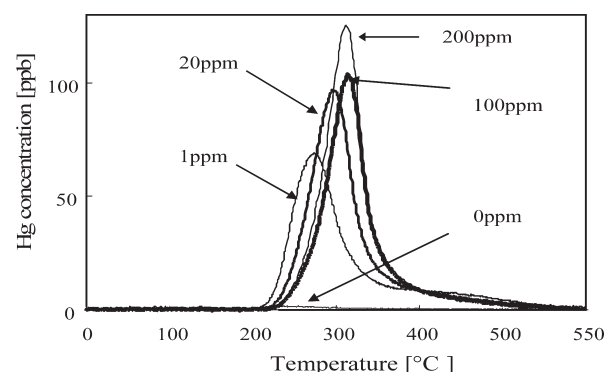

Figure 2. Effect of the HCl concentration during Hg^0 removal in the absence of SO_2 at 80 °C on TPDD spectra.
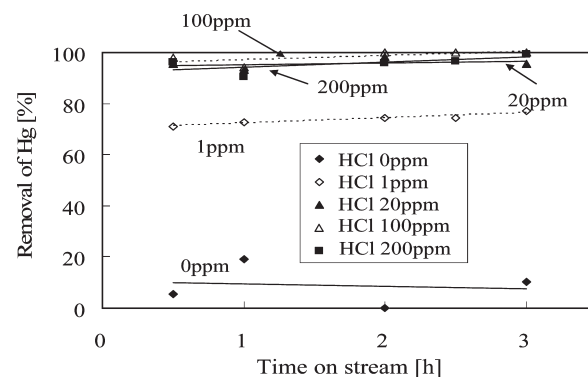

Figure 3. Effect of the HCl concentration on Hg^0 removal in the presence of 500 ppm SO_2 at 80 °C.

think that mercury chloride species HgCl$_x$, including $HgCl_2$, might be formed on AC.

We also suggest that the reactions via HgO (reactions 5−67) could be preferred over the direct reactions (reactions 1−234) for the formation of HgCl$_x$ on AC, because from our previous study, it was observed that the mercury removal rate became higher in the presence of O_2 than in the absence of O_2 in the HCl-absence system (but in the SO_2-presence system).[15] It was suggested that the reaction via HgO might occur for the formation of $HgSO_4$. However, it was not direct evidence of the reaction via HgO for the present system; therefore, we need more studies to clarify the reaction mechanism.

Figure 3 shows the effect of the HCl concentration on the Hg^0 removal by AC in the presence of SO_2 at 80 °C. In
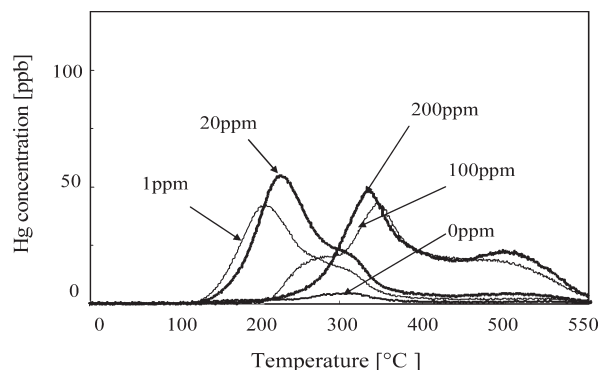
Figure 4. Effect of the HCl concentration during Hg^0 removal in the presence of 500 ppm SO_2 at 80 °C on TPDD spectra.

comparison to the case above of the SO_2-absence system (in Figure 1), the Hg^0 removal rate decreased in the presence of 1 ppm HCl and 500 ppm SO_2. However, the Hg^0 removal rate of AC was also enhanced in the presence of HCl, especially at HCl concentrations over 20 ppm.

Figure 4 shows the effect of the HCl concentration on the TPDD spectra of the spent AC used in Hg^0 adsorption in the presence of 500 ppm SO_2 at 80 °C. In the above case of the SO_2-absence system (in Figure 2), only a single Hg^0 desorption peak at around 300 °C was observed. However, the Hg^0 desorption peaks in the TPDD spectra were observed in a wide range of temperatures for the SO_2-presence system, as shown in Figure 4. It was also observed that the position (temperature) of the main peaks in TPDD spectra changed drastically between lower temperatures for the spent samples of the 1 and 20 ppm HCl-presence system to higher temperatures for the spent samples of the 100 and 200 ppm HCl-presence system. These indicate that the composition of acid gases (concentration of HCl and SO_2) in simulated flue gas is decisive to the type of adsorbed mercury species formed on the sorbent. Therefore, for the co-existence of HCl and SO_2, we suggested that mercury species containing both SO_2^{2-} and Cl^- might be formed on activated carbon. Moreover, at higher HCl concentrations (100−200 ppm), some new peaks appeared at around 500 °C. We have suggested in our previous study that the TPDD peak at around 500 °C resembled the decomposition peak of $HgSO_4$ (reagent). At present, we do not know the exact nature of this species.[16] However, it is certain that some different kinds of mercury species are formed in the presence of SO_2.

3.2. Effect of the Temperature and HCl Concentration on the Hg^0 Removal of AC. Figure 5 shows the effect of the Hg^0 adsorption temperature on the mercury removal efficiency in the presence of 20 ppm HCl and 500 ppm SO_2. As the Hg^0 adsorption temperature increased from 80 to 200 °C, the mercury removal efficiency declined from about 96 to about 45%. Figure 6 shows the effect of the Hg removal temperature (80, 120, 150, and 200 °C) in the presence of 20 ppm HCl and 500 ppm SO_2 on the TPDD spectra of the spent AC, as shown in Figure 5. In all cases, the main mercury desorption peak temperature appeared at around 240 °C in the TPDD spectra. We mentioned earlier (in Figure 4) that the position (temperature) of the main peaks in TPDD spectra changed
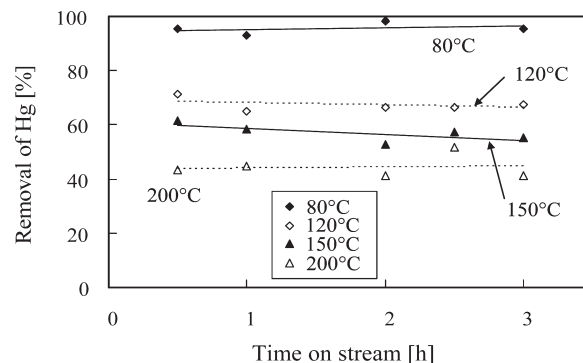
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Figure 5. Effect of the temperature on Hg^0 removal in the presence of 20 ppm HCl and 500 ppm SO_2.
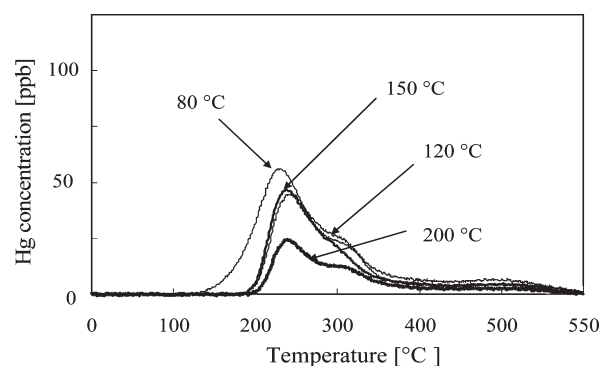

Figure 6. Effect of the adsorption temperature during Hg^0 removal in the presence of 20 ppm HCl and 500 ppm SO_2 on TPDD spectra.
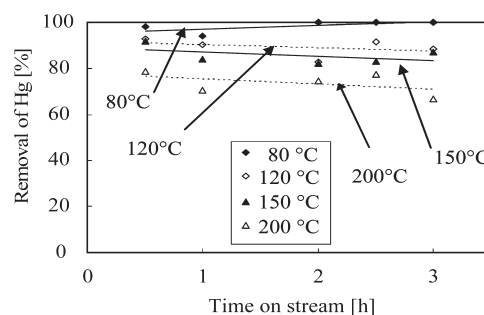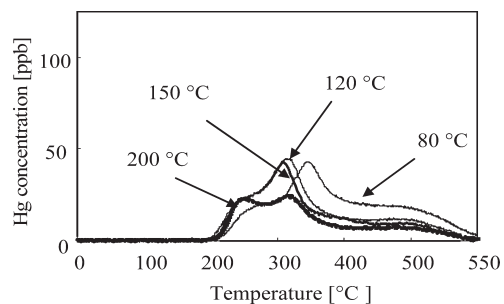

Figure 7. Effect of the temperature on Hg^0 removal in the presence of 100 ppm HCl and 500 ppm SO_2.

drastically between lower temperatures for the spent samples of the 1 and 20 ppm HCl-presence system to higher temperatures for the spent samples of the 100 and 200 ppm HCl-presence system. Therefore, we performed some experiments to confirm the peak temperature position of the TPDD spectra of spent AC used in the presence of 100 ppm HCl and 500 ppm SO_2 at different adsorption temperatures (80, 120, 150, and 200 °C). Figure 7 shows the effect of the mercury removal temperature (80, 120, 150, and 200 °C) in the presence of 100 ppm HCl and 500 ppm SO_2 on the Hg^0 removal efficiency, and Figure 8 shows the TPDD spectra of spent AC. As the Hg^0 adsorption temperature increased from 80 to 200 °C, the mercury removal rate decreased from about 100% at 80 °C to about 70% at 200 °C. The main Hg^0 desorption peak temperature in the TPDD spectra appeared at above 300 °C for the 100 ppm HCl-presence system (in Figure 8). These results imply that there are some reaction

Figure 8. Effect of the temperature during Hg^0 removal in the presence of 100 ppm HCl and 500 ppm SO_2 on TPDD spectra.
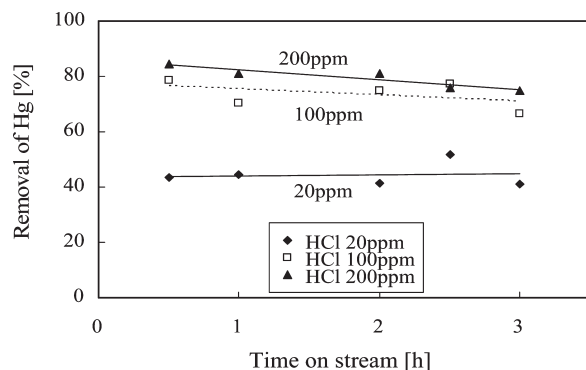

Figure 9. Effect of the HCl concentration on Hg^0 removal in the presence of 500 ppm SO_2 at 200 °C.
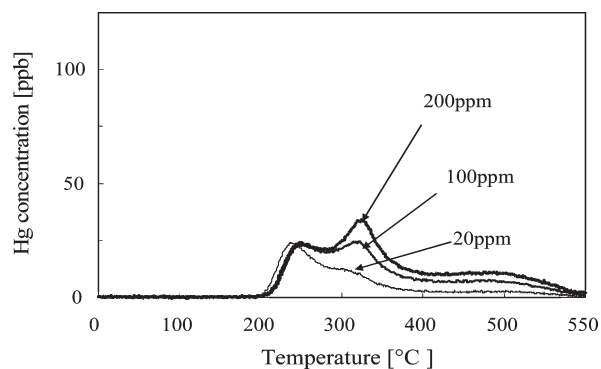

Figure 10. Effect of the HCl concentration during Hg^0 removal in the presence of 500 ppm SO_2 at 200 °C on TPDD spectra.

routes (or different mechanisms) for Hg^0 removal in the presence of HCl and SO_2 and that the reaction pathways are determined by the simulated flue gas composition, especially the concentration of HCl and SO_2.

To enhance the Hg^0 removal rate of the AC at 200 °C, the HCl concentration was raised from 20 to 200 ppm. Figure 9 shows the effect of the HCl concentration on Hg^0 removal by AC at 200 °C. It is evident in this figure that the Hg^0 removal rate increased with the increase of the HCl concentration during Hg^0 removal. About 80% Hg^0 removal was achieved in the presence of 200 ppm HCl. Therefore, it can be concluded that a high level Hg^0 removal efficiency could be achieved at a high HCl concentration in the flue gas, even at higher temperatures (~200 °C).

Figure 10 shows the effect of the HCl concentration during the Hg^0 removal in the presence of 500 ppm SO_2 at 200 °C by AC (in Figure 9) on the TPDD spectra of spent AC. The
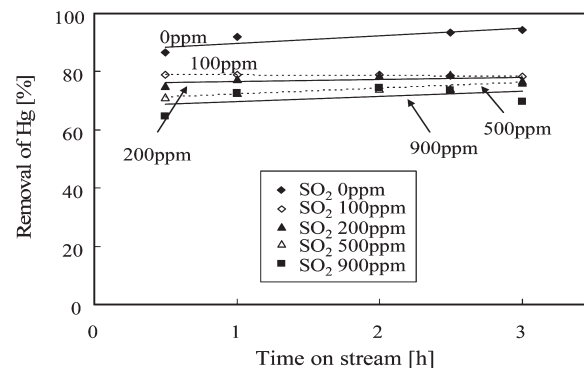

Figure 11. Effect of the SO_2 concentration on Hg^0 removal in the presence of 1 ppm HCl at 80 °C.
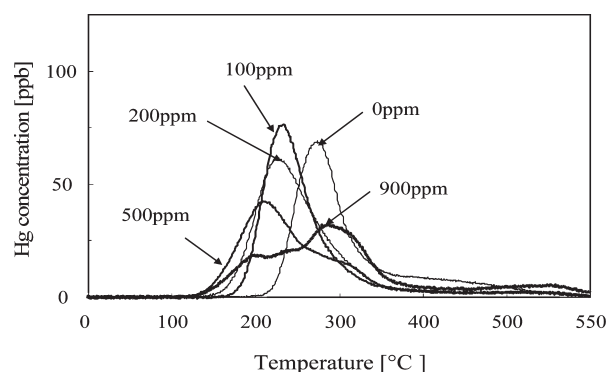

Figure 12. Effect of the SO_2 concentration during Hg^0 removal in the presence of 1 ppm HCl at 80 °C on TPDD spectra.

mercury desorption peak intensity at around 320 °C increased with the increase of the HCl concentration during mercury removal. In particular, the main desorption peaks shifted from around 240 °C for the spent samples of the 20 ppm HCl-presence system to around 320 °C for the spent samples of the 100 and 200 ppm HCl-presence system.

3.3. Effect of the SO_2 Concentration on the Hg^0 Removal by AC. Figure 11 shows the effect of the SO_2 concentration on the Hg^0 removal of AC at 80 °C in the presence of 0−900 ppm SO_2 and 1 ppm HCl. In the presence of 1 ppm HCl, the presence of SO_2 suppressed the Hg^0 removal rate of AC. In the presence of 100−900 ppm SO_2, the mercury removal remained almost constant at 70−80%, indicating that the concentration of SO_2 in the range of 100−900 ppm has very little effect on Hg removal.

Figure 12 shows the effect of the SO_2 concentration during Hg^0 adsorption in the presence of 1 ppm HCl on the TPDD spectra. A similar tendency, as shown in Figure 4, that is a shift in the main peak temperature between the 500 and 900 ppm SO_2-presence system, was also observed. From these results, we suggest that the adsorbed mercury species could be changed by the flue gas compositions, even at the same Hg removal rate.

The effect of the SO_2 concentration on Hg^0 removal in the absence of HCl was also investigated, and the results are shown in Figure 13. It was found that the Hg^0 removal rate of AC increased slightly in the presence of SO_2. However, in comparison to the effect of the HCl concentration, the effect of the SO_2 concentration on the Hg^0 removal was considerably low in the range of 100−900 ppm. There was a shift in the Hg desorption peak temperature in the TPDD spectra for
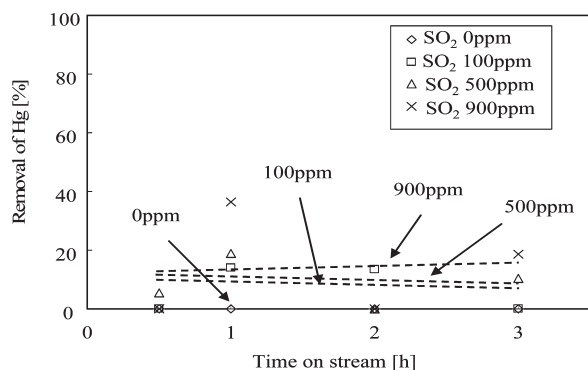
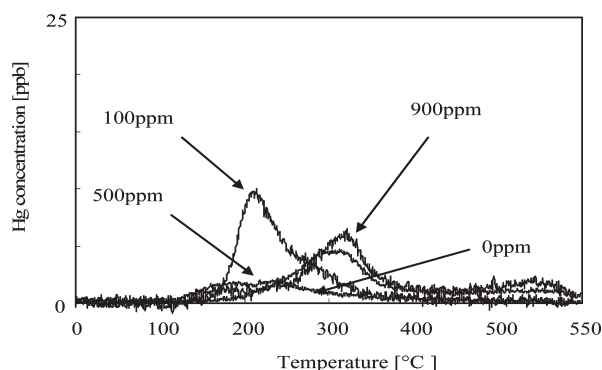**Figure 13.** Effect of the SO_2 concentration on Hg^0 removal in the absence of HCl at 80 °C.

Figure 14. Effect of the SO_2 concentration during Hg^0 removal in the absence of HCl at 80 °C on TPDD spectra.

the 500 and 900 ppm SO_2-presence system compared to the 0 and 100 ppm SO_2-presence system (Figure 14).

4. Conclusions

In this study, the effect of the presence of HCl and SO_2 in the simulated coal combustion flue gas on the Hg^0 removal by a commercial activated carbon (coconut shell AC) was investigated in a laboratory-scale fixed-bed reactor. The characteristics (thermal stability) of the mercury species formed on the sorbents under various adsorption conditions were investigated by the TPDD technique. It was found that the presence of HCl and SO_2 in the flue gas during Hg^0 removal by AC played important roles on the Hg^0 removal efficiency of the sorbents. The characteristics of the Hg^0 adsorption species also depended upon the concentration of HCl and SO_2 in the flue gas. The Hg^0 removal efficiency increased with the increase of the HCl concentration during Hg^0 removal. In the presence of HCl and absence of SO_2 during Hg^0 adsorption by AC, a single Hg^0 desorption peak at around 300 °C was observed in the TPDD spectra, and it is suggested that this peak may be derived from the decomposition and desorption of mercury chloride species.

The presence of SO_2 during Hg^0 removal suppressed the Hg^0 removal capacity of AC in the presence of HCl. In the presence of both SO_2 and HCl during Hg^0 removal by AC, several Hg^0 desorption peaks were observed over a wide range of temperatures in the TPDD spectra, indicating that the composition of acid gases (concentration of HCl and SO_2) in simulated flue gas is decisive to the type of adsorbed mercury species formed on the sorbent. However, additional experimental data are needed to further understand the mechanism of Hg^0 removal, particularly in the presence of HCl and SO_2.