THE EFFECT OF WATER (VAPOR-PHASE AND CARBON) ON ELEMENTAL MERCURY REMOVAL IN A FLOW REACTOR

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ABSTRACT

The effect of vapor-phase moisture on elemental mercury (Hg^0) removal by activated carbon was studied in a flow reactor. Tests were conducted in which activated carbon was injected into both a dry and 4% moisture nitrogen (N_2)/Hg^0 gas stream. A bituminous-coal-based activated carbon (Calgon WPL) was injected into a Hg^0-laden gas stream (124 ppbv) at 150 °C at carbon to mercury ratios (C:Hg) between 1300 and 4400:1. The addition of 4% (vol.) water (H_2O) into the gas resulted in a ~10% drop in removal (21-80%) compared to tests in dry N_2 (26-89%). It is hypothesized that the H_2O molecules can form hydrogen bonds with the carbon thus reducing the number of active sites available for Hg^0.

The effect of activated carbon moisture content on Hg^0 removal was also studied. Darco FGD (lignite-based) activated carbon was injected at 100 °C and a Hg^0 concentration of 86 ppbv. At a C:Hg of 11000:1, Hg^0 removal for as-received Darco FGD carbon (3% moisture) was 30%. Increasing the moisture content of the carbon to 16% resulted in a Hg^0 removal of 60%. Similar tests in which the surface moisture in the carbon was reduced to 0% resulted in a Hg^0 removal of less than 5%. Similar results were obtained with WPL. The effect of carbon moisture content on Hg^0 removal was also tested in a simulated flue gas. Results in the flue gas from a methane flame (doped with sulfur dioxide) showed that carbon surface moisture had a small impact (positive) on Hg^0 removal by FGD carbon and no impact on removal by the WPL carbon.

The injection of dry activated carbon into a wet (4% moisture) N_2 stream did not result in increased Hg^0 capture. It is believed that the moisture did not have sufficient time to diffuse into the carbon and modify the carbon-oxygen functional groups that may be responsible for adsorption.

INTRODUCTION

Mercury is a naturally occurring element that is contained in coal. Mercury that is emitted from coal-fired power plants can deposit in our rivers and watersheds and ultimately end up in the food chain causing adverse health effects in humans. The EPA has determined that the emission of mercury from coal-fired power plants poses a risk to human health and the environment. The EPA has announced its finding that regulation of mercury emissions from electric utility steam generating units is necessary. Emission standards are to be proposed on or before December 15, 2003, and the standards are to be promulgated by December 15, 2004 [1].

The concentration of mercury in coal is highly variable with average concentrations ranging from 40 to 330 ppbw [2]. When coal is burned, mercury is vaporized. As the flue gas is cooled, the mercury is present as one of several forms including elemental (Hg^0) and oxidized species such as mercuric chloride ($HgCl_2$) and mercuric oxide (HgO). Mercury partitioning depends on several factors including the type of coal burned; for example, Hg^0 is the dominant form when burning North Dakota lignite [3]. The control strategy may depend on the form in which the mercury is present. Oxidized species are water-soluble and can be removed in units that have a wet scrubber, whereas Hg^0 is insoluble in water [4,5,6]. One of the methods that have been demonstrated to remove Hg^0 as well as oxidized forms is adsorption using activated carbon. Activated carbon is injected upstream of the particulate control device such as an electrostatic precipitator (ESP) or fabric-filter baghouse where the carbon is removed with the fly ash. Higher Hg^0 removals have been obtained in units that have a baghouse as compared to those that have an ESP for particulate control.

Most bench-scale research on Hg^0 adsorption has been done in fixed-bed reactors which simulate capture by a baghouse filter cake.[7-10] Most coal-fired power plants use ESPs, in which there is a short contact time between carbon and the gas to remove any Hg^0. In this work a bench-scale flow reactor was used to study in-flight capture of Hg^0 in the presence of carbon sorbents, thus simulating capture through an ESP. The results obtained in this research will be compared to those obtained in fixed-bed tests.

In previous work with the flow reactor, the effects of particle size, temperature, carbon to mercury ratio (C:Hg), and carbon type were examined[11,12] In that work it was found that two carbons with similar physical properties had different Hg^0 adsorption results in the flow reactor. The main difference between the two carbons was the carbon moisture content. In other work, Li et al.[13] found that the surface moisture of activated carbon has a significant effect on Hg^0 adsorption in fixed-bed reactors. The present study examines the effects of carbon moisture content and vapor-phase moisture on Hg^0 removal in a flow reactor.

Activated carbons contain a variety of chemically reactive surface sites, such as hydroxyl, carboxyl, quinone, peroxide, and aldehyde groups[14]. According to Bansal et al.[15], these carbon-oxygen surface complexes are by far the most important structures influencing the surface characteristics and adsorption behaviors of activated carbons. Heating activated carbon and reducing the amount of moisture in the carbon may reduce the number of surface carbon-oxygen functional groups thus creating a less-reactive carbon. Carbon will also adsorb water from the surrounding gas. Water molecules are first adsorbed on primary adsorption centers, such as

oxygen groups, with secondary adsorption occurring on the adsorbed water molecules [16]. H_2O adsorption at lower relative pressures (e.g., $P/P_0 < 0.4$) has been correlated directly to the amounts of chemisorbed oxygen on the carbon surface [15]. It has also been shown that interactions between H_2O and carbon-oxygen complexes influence the reactivities of activated carbons for adsorption of organic vapors [17]. In tests with a sulfur-impregnated carbon at 140 °C, Liu et al.[18] found that the adsorptive capacity of the carbon decreased by as much as 25% when 10% moisture was added to the gas stream compared to tests in dry N_2.

EXPERIMENTAL

A schematic of the flow reactor is shown in Figure 1, and a more detailed description of the experimental setup can be found in Serre et al.[11,12]. The quartz reactor is 310.5 cm long with an inner diameter of 4 cm. Three gas sampling ports are located along the length of the reactor and are labeled SP1, SP2, and SP3. The reactor is heated with three Lindberg, 3-zone electric furnaces in series. The baseline Hg^0 concentration is measured in the absence of activated carbon using an ultraviolet (UV) analyzer (Buck Scientific, model 400A). Once the baseline is established, activated carbon is fed into the top of the reactor using a fluidized-bed feeder (0.2-0.5 std. L/min). The gas-phase Hg^0 concentration is then measured at one of the sample ports by pulling a gas sample (0.5 std. L/min) through a 1 μm filter to remove any particles, then through a reducing furnace to convert any oxidized Hg to Hg^0 (method described in Ghorishi and Gullett [8]). After the reducing furnace, the gas is dried using a Nafion® gas sample dryer (Perma Pure, Inc.) and is finally sent to a Buck analyzer.

Initial tests were conducted using nitrogen (N_2) as the carrier gas with later tests performed in a flue gas from a methane flame. In the N_2 carrier gas tests, industrial grade N_2 [1 std. L/min (0 °C, 1 atm)] flows over a Hg^0 permeation tube that is housed in a permeation oven (VICI Metronics, model 190) to generate a Hg^0-laden gas stream. The N_2/Hg^0 stream is diluted with a second N_2 stream (12 std. L/min) to the desired concentration before entering the top of the reactor. Other gases (SO_2, NO, O_2, water vapor) can be blended into the N_2 carrier gas in the mixing manifold.

In tests with the methane flame flue gas, methane was burned in a water-cooled burner and NO and SO_2 were added (post-flame). Table 1 shows the typical flue gas composition when operating the flow reactor with flue gas.

A fluidized-bed feeder is used to inject sorbent into the reactor. An inlet line of N_2 is used to fluidize and carry the activated carbon to the reactor. The carbon feed rate is adjusted by varying the amount of N_2 (0.2 to 0.5 std. L/min) entering the feeder.

Because the UV analyzer used to detect Hg^0 is sensitive to particles, a filter was used to remove any carbon that may have been carried with the gas. Tests were conducted to determine if carbon particles were accumulating on the filter, as this would act like a packed bed and the reactor's removal of Hg^0 would be a combination of in-flight + filter (packed-bed) capture. Activated carbon was injected in the absence of Hg^0, and a gas sample was pulled through the

Table 1
Methane Flame Flue Gas Composition

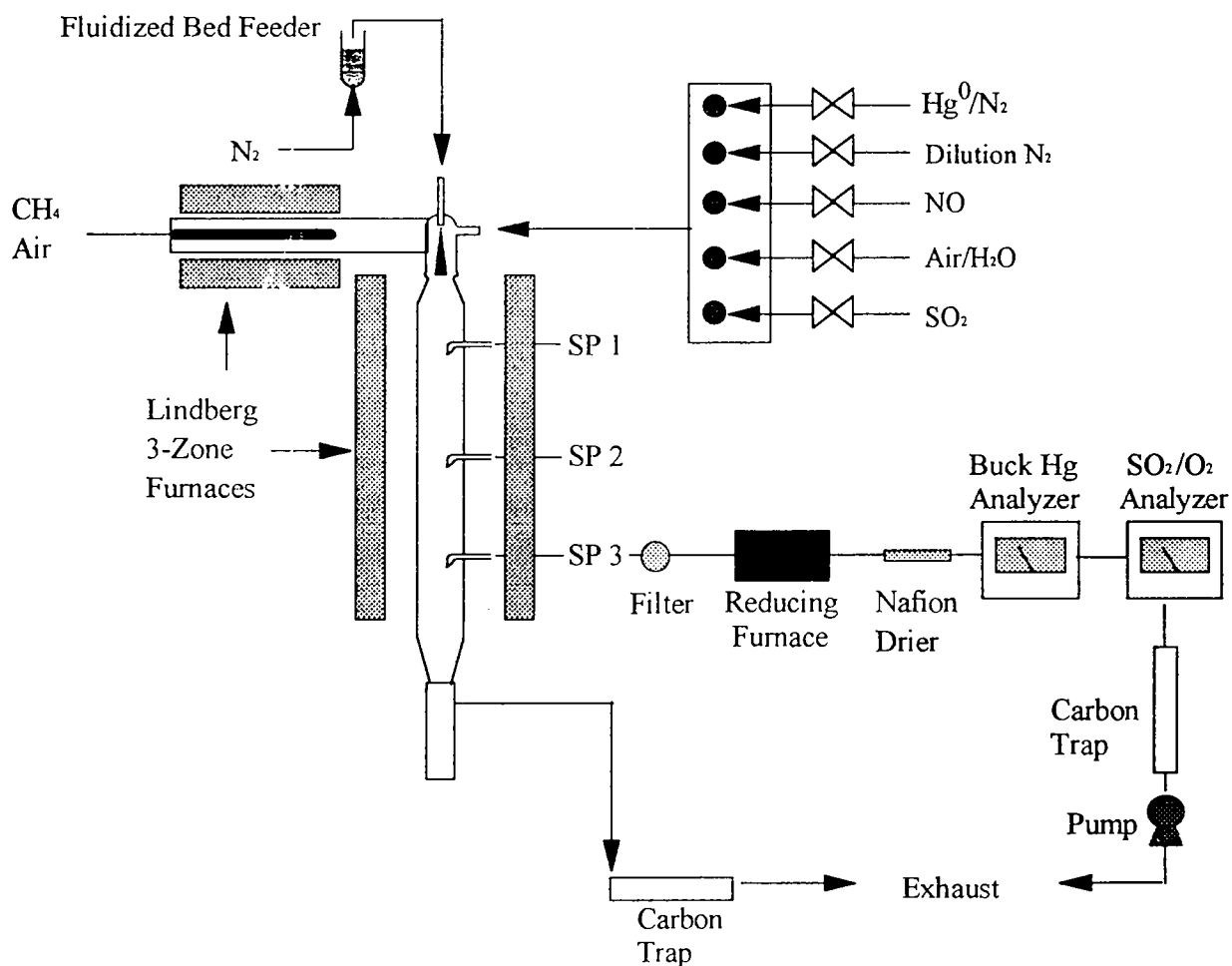| Flue Gas Component	Concentration
SO_2	500 ppm
NOx	200 ppm
CO_2	3.5%
CO	5 ppm
O_2	7.1%
H_2O	6.8%

Figure 1. Schematic of the flow reactor with methane burner.

filter. After 1 minute, Hg^0 was added to the gas stream to see if there was a lag in the time it takes for the baseline to return. The results were the same as for a blank filter, suggesting that the filter did not have an effect on the results.

The Hg^0 UV analyzer also responds to any SO_2 that is present by increasing the signal on the analyzer. The net increase in the signal is dependent on the SO_2 concentration and was compensated for by measuring the net increase and subtracting this number from the measured Hg^0 concentration.

The total flow through the reactor was 13 std. L/min (0 °C, 1 atm) which gave residence times of 5.2, 11.5, and 17.7 s at ports SP1, SP2, and SP3, respectively. The velocity of the particles through the reactor is assumed to be the same as that of the gas flow since the terminal velocity of the particles is smaller than the velocity of the gas through the reactor, by a factor of 3.

Two activated carbons were tested, a lignite-based activated carbon (FGD, Norit Americas, Inc.), and a bituminous-based carbon (WPL, Calgon Carbon Corp.). The carbons were size classified to 4-8 μm for the FGD and 5-25 μm for the WPL. The total surface areas, measured using an adsorption apparatus (ASAP 2400, Micromeritics Inc.) based on the Brunauer-Emmett-Teller (BET) method, were 515 for the FGD and 1080 m^2/g for the WPL.

Activated carbon contains a small amount of moisture that remains from the activation process. According to the literature from the carbon manufacturers, the amount of moisture in both the WPL and FGD carbon should be less than 8% [19,20]. The amount of moisture contained in the WPL and FGD carbons was checked using an infrared moisture determination balance (AND® model AD-4714) at 120 °C for 20 minutes. The amount of moisture in the test samples was 3% for the FGD carbon and 13% for the WPL carbon.

RESULTS AND DISCUSSION

Vapor-phase water

Since water vapor is one of the main products of combustion, its effect on Hg^0 removal must be evaluated. The effect of vapor-phase water on Hg^0 removal, at a given C:Hg ratio, is shown in Figure 2 for WPL. WPL was fed at a C:Hg ratio of 1250 to 4500:1 into dry nitrogen at 150 °C and 124 ppbv Hg^0. Hg^0 removal at ports SP1 (3.4 s) and SP3 (11.5 s) is shown. A Hg^0 removal of approximately 90% was achieved at a C:Hg ratio of 4500:1 at SP3. Water was then added to the system at a concentration of 4% (vol.). A 10% drop in removal occurred with the addition of moisture to the N_2/Hg^0 stream, compared to dry conditions. It is hypothesized that the water molecules are competing for active sites by forming hydrogen bonds with the carbon thus reducing the number of active sites available. At high enough concentrations water can condense in the microporous regions and block access to pores in the carbon, though this is not likely at a temperature this high. Liu et al.[18] found that the total Hg^0 uptake capacity by a sulfur-impregnated carbon did not change when 5% moisture was added to the gas stream in fixed-bed tests. However, the same researchers found that when the vapor-phase moisture reached 10%,
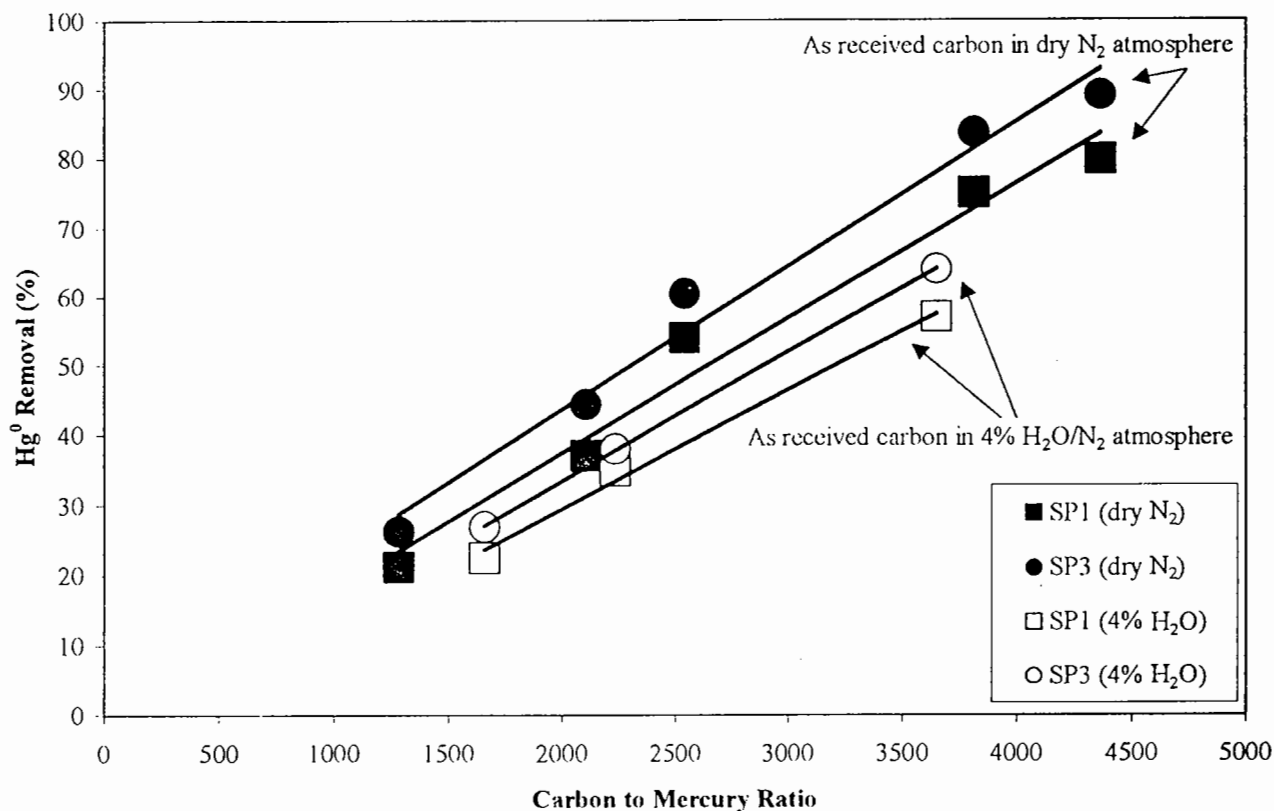
Figure 2 Effect of water in gas stream (4% vol.) on Hg^0 removal at 150 °C and a $Hg°$ concentration of 124 ppbv for WPL carbon.

the capacity dropped by 25%. Carey et al.[21] examined the effect of water-vapor on Hg^0 capacity in a fixed-bed reactor using simulated flue gas. They found that capacity was not affected when water was present in concentrations of 1 to 10% at a temperature of 135 °C. Our experiments resulted in a higher removal at longer residence times for both dry and moist carrier-gas conditions (Figure 2).

Carbon water content

The effect of carbon moisture content on Hg^0 removal at 200 °C and 44 ppbv Hg^0 in a dry N_2 atmosphere for the WPL carbon is shown in Figure 3. Hg^0 removal is shown for port SP3 (10.2 s). The diagonal band of points is Hg^0 removal for the as-received WPL (13% moisture) as a function of C:Hg ratio. Removal approaches 80% for the as-received carbon at a C:Hg of 16000:1. The amount of moisture in the carbon was then adjusted by heating the carbon at 110 °C under vacuum, resulting in carbon moisture contents of 0, 2.5, and 4%. These carbons were then tested at a single C:Hg ratio.

The results of these experiments show direct correlation between Hg^0 removal and the moisture in the carbon. As the carbon moisture content is reduced to 4%, Hg^0 removal decreases slightly compared to the as-received carbon. Hg^0 removal approaches zero as the moisture in the
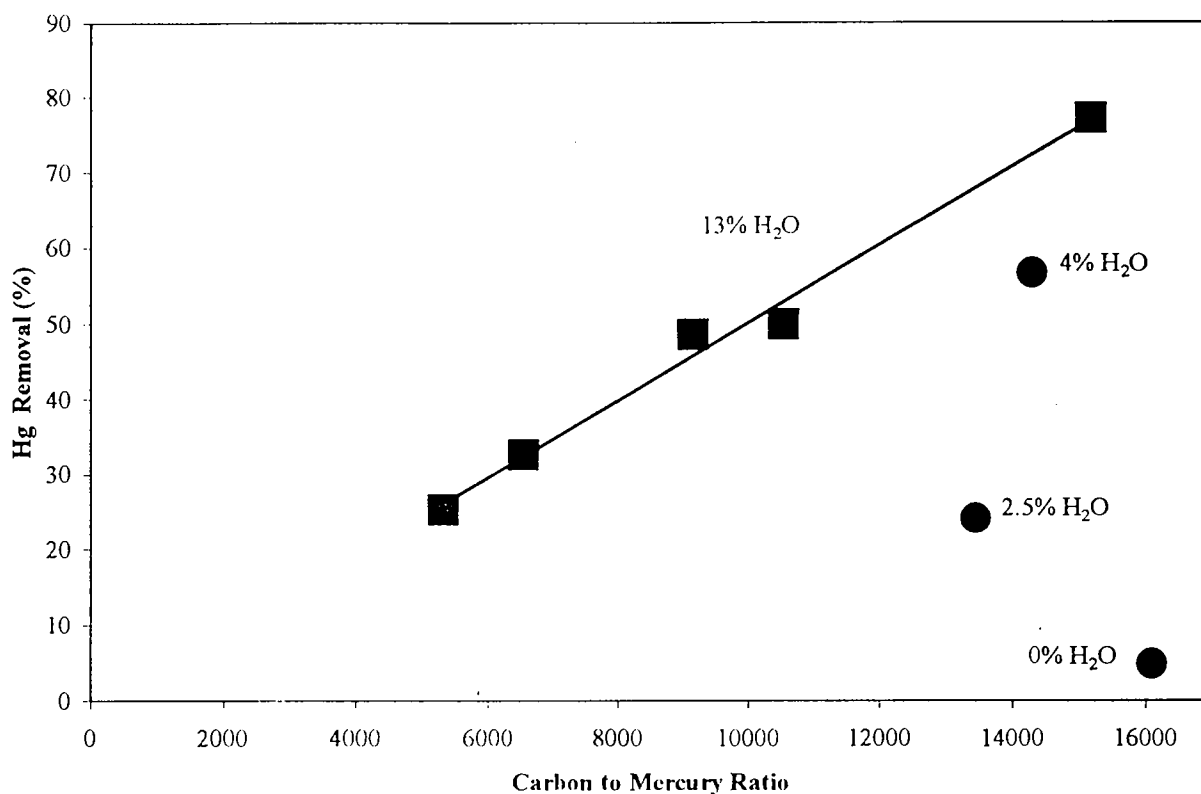
Figure 3   Effect of carbon water content on removal for the WPL carbon at 200 °C and a Hg° concentration of 44 ppbv in a dry N_2 atmosphere at SP3 (10.2 s).

carbon approaches zero. Hg^0 removal for the 0% moisture sample is nearly zero compared to 80% for the as-received moisture content of 13% at a similar feed rate. In a N_2 atmosphere the presence of moisture in the carbon appears to be necessary for Hg^0 adsorption.

Evaporation or desorption of moisture from the carbon surface will occur when injecting a moisture-containing sample into a dry atmosphere. The evaporation of moisture could create a cooling effect of the carbon particle surface which could increase Hg^0 capture since removal has been shown to increase with a decrease in temperature [7,8,22,23]. In tests by Delage et al.[24], a dry gas (nitrogen and oxygen) was blown over wet granular activated carbon bed to measure the temperature drop of a carbon bed. A maximum temperature drop of 8 °C was obtained over several minutes. The drop in temperature was attributed to the desorption of water from the carbon surface. If the increase in Hg^0 removal was a sole result of the water desorption from the carbon surface, the difference of Hg^0 removal between the 13 and 4% moisture samples would be expected to be much larger than those between 4 and 2.5%, as well as between the 2.5 and 0% moisture samples. A 10% difference in Hg^0 removal was obtained when the moisture content decreased from 13 to 4%, while there was a difference of more than 25% between the 4 and 2.5% moisture carbon. This suggests that the increase in Hg^0 removal cannot be entirely attributed to the cooling effect.

To further confirm the effect of surface moisture on Hg^0 removal, moisture was added to the as-received FGD carbon and tested at 100 °C and 86 ppbv Hg^0 in a dry N_2 atmosphere. The moisture content was increased by placing the as-received FGD sample in a sealed container with a pan of deionized water at 23 °C for 2.5 hr. After this period of exposure the moisture increased to 16%. Results with the as-received FGD (3% moisture) are shown in Figure 4 by the solid squares. At a C:Hg ratio of 11000:1, a removal of 30% was obtained. Tests were then repeated using the moisture-enhanced carbon, shown by the circles in Figure 4. Increasing the water content of the carbon increased Hg^0 removal. At a C:Hg of 11000:1, the removal doubled from 30% for the 3% moisture carbon to 60% for the 16% water carbon. A sample was also dried under vacuum at a temperature of 110 °C. The dried sample (0% moisture) is also shown in Figure 4. The 0% moisture sample performed very poorly under these conditions, removal was below 10% at a C:Hg of 16000:1. These results with the moisture-enhanced carbon in Figure 4 are similar to that of the decreased moisture samples in Figure 3. Thus, it can be concluded that carbon surface moisture plays an important role in Hg^0 adsorption in N_2. Eliminating the moisture in the carbon deactivated or reduced some of the active sites in the carbons.

As described previously, adsorbed surface moisture plays an important role in Hg^0 adsorption, and the presence of surface moisture appears to be necessary for Hg^0 adsorption in N_2. However, it should be understood that, with microporous material such as activated carbon, adsorption is a dynamic process and consists of several steps: bulk phase transportation, pore diffusion and
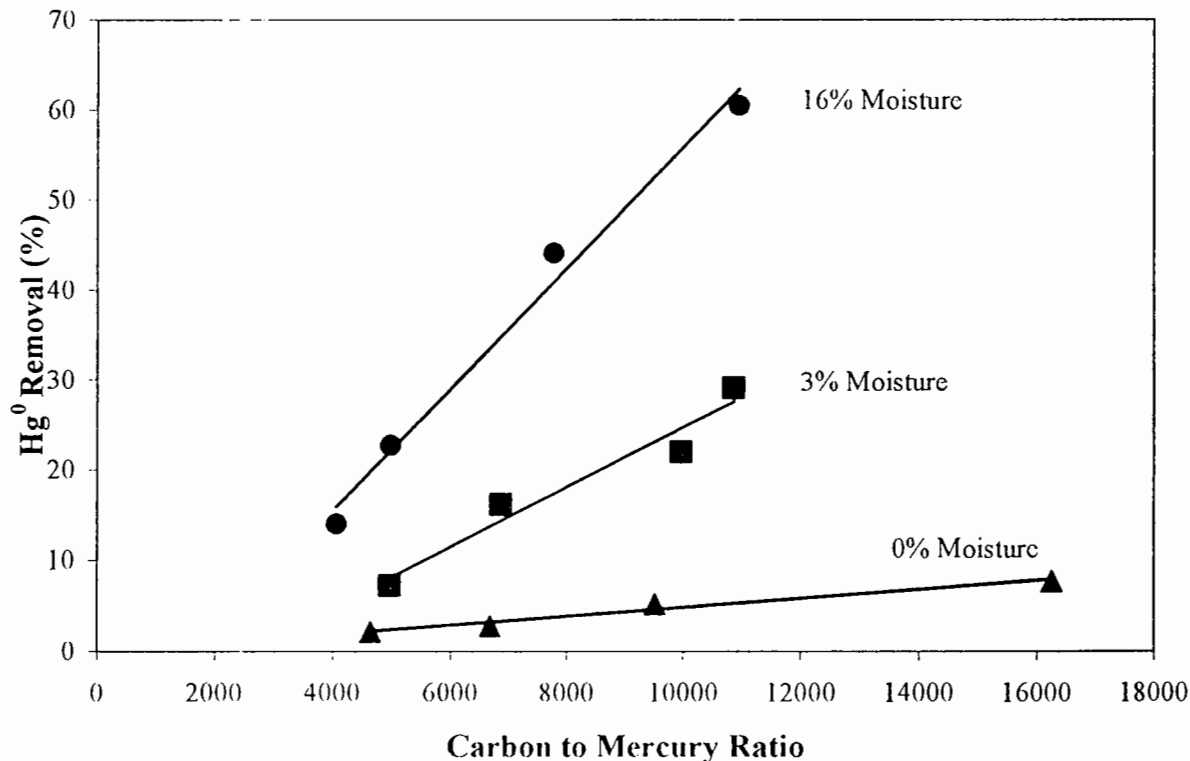

Figure 4 Hg° removal as a function of carbon water content for FGD carbon at 100 °C and 86 ppbv in a dry N_2 atmosphere. The size of the carbon is 16-24 μm.

adsorption. The dynamic effect of adsorption by activated carbon would be more pronounced in a flow reactor system with a short residence time, than in fixed-bed reactor studies.

It was shown that Hg^0 removal correlates with carbon moisture content, but the addition of moisture to the N_2 gas stream resulted in a slightly lower Hg^0 removal (Fig. 2). What happens when dry activated carbon is injected into a gas stream that contains water? Will the carbon be able to absorb moisture from the vapor phase thus increasing the carbon moisture content? These questions are addressed in this section. Hg^0 removal for WPL at 150 °C and 124 ppbv at a C:Hg ratio of 3750:1 is shown in Figure 5 for 0 and 4% moisture contents. The first set of bars for the 0% moisture carbon show that Hg^0 removal is below 5%. The addition of 4% water to the nitrogen/Hg^0 stream did not improve removal. The next data set for the 4% moisture carbon shows a similar trend. It appears that the water did not have time to diffuse into the carbon and modify the carbon-oxygen functional groups that may be the active sites for adsorption.
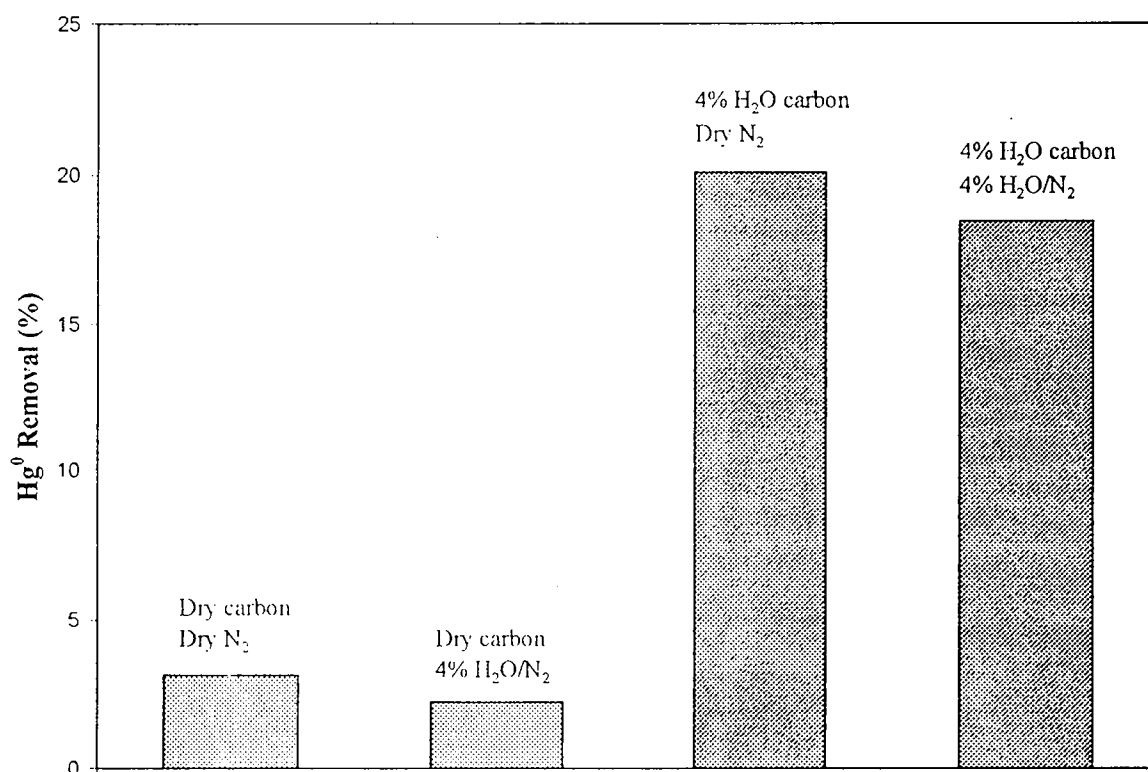

Figure 5 Hg^0 removal for WPL carbon with varying moisture contents in the carbon and gas stream at 150 °C and a Hg^0 concentration of 124 ppbv at a C:Hg = 3750:1.

Effect of carbon water content in a methane flame flue gas atmosphere

All the data presented to this point have been obtained in experiments with N_2 as the carrier gas. The effect of carbon moisture content on Hg^0 removal in a methane flame flue gas (doped with SO_2 and NO) is shown in Figure 6 with a gas composition shown in Table 1. The 0 and 13% moisture samples of WPL were fed into the reactor at an average C:Hg ratio of 3100 ($\pm$ 200):1. Hg^0 removal for the 0% moisture sample was 10% and 17% for the 13% moisture sample. A slight increase in removal was observed for the higher moisture containing carbon, though not as great as was seen in the tests in dry N_2. The main difference between the tests is the presence of acid gases, which have been shown to have a negative effect on Hg^0 adsorption [25,26].

The two carbons (0 and 13% moisture) were also injected in the absence of SO_2. Hg^0 removal increased to 21% for the 0% moisture WPL carbon and to 43% for the 13% moisture content WPL carbon. Since SO_2 is present at a much higher concentration than the Hg^0, the SO_2 may be competing for the active sites in the carbon. The SO_2 may also act as a poison for the active sites that are responsible for Hg^0 adsorption. In fixed-bed tests by Miller et al.[26], the addition of SO_2, in the presence of NO_2, resulted in desorption of any Hg^0 that had been captured by the carbon sorbent.
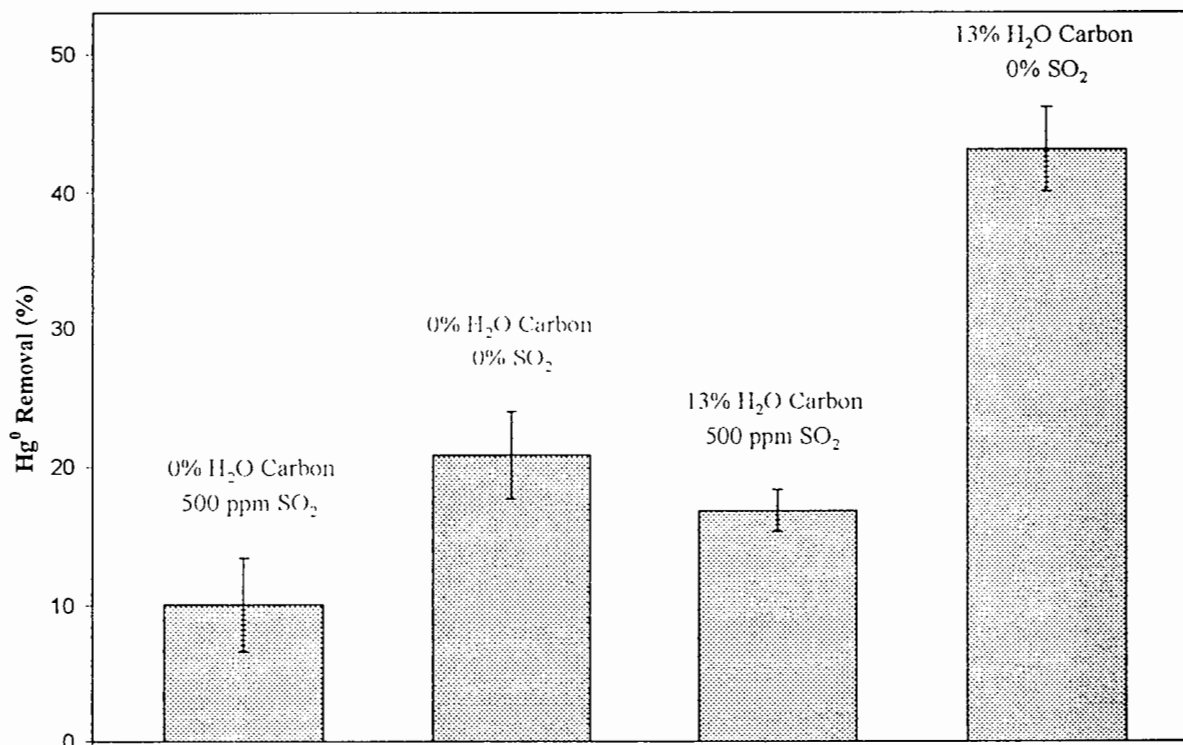

Figure 6 Effect of carbon moisture content on Hg^0 removal for WPL carbon (C:Hg=3100:1) in methane flame flue gas at 100 °C and 86 ppbv Hg^0. Error bars represent 1 standard deviation (n=3).

Hg^0 removal for FGD carbon in a methane flue gas is shown in Figure 7 for a C:Hg of 10000 ($\pm$ 1000):1. Increasing the moisture from 3% for the as-received carbon to 16% for the moisture-enhanced carbon resulted in approximately a 2-fold increase in Hg^0 removal. Increasing the carbon moisture did not produce the same results as seen in the N_2 tests, similar to the WPL tests. The effect of SO_2 was also examined. The presence of SO_2 in the flue gas did not have an impact on Hg^0 removal for both the 3 and 16% moisture samples. Other interactions may have been occurring in the tests with flue gas. Future tests are planned to further evaluate the effect of SO_2 and NO_x on Hg^0 control.

CONCLUSION

The effect of carbon moisture content on Hg^0 removal was tested for lignite- and bituminous-coal-based carbons. The moisture content of the carbon had a significant effect on Hg^0 removal in a N_2 gas stream, simply increasing the moisture content resulted in an increase in removal. Conversely, removing moisture from the carbon resulted in a decrease in removal. This suggests that certain hydrated functional groups may act as active sites for binding Hg^0. Similar tests in a methane flue gas showed a small improvement in Hg^0 removal for the FGD carbon with an increase in carbon moisture. However, tests with the WPL carbon showed that carbon moisture did not have an effect on removal in the flue gas. The main difference between the two sets of tests (N_2 vs. flue gas) is the presence of acid gases.
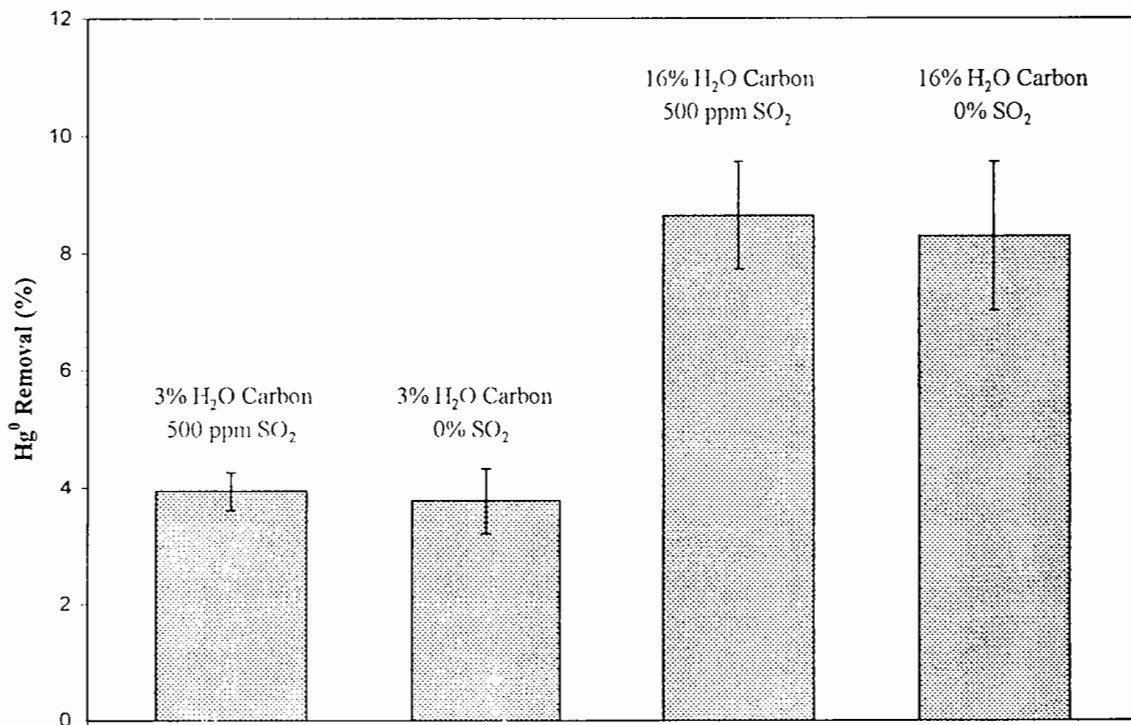

Figure 7 Effect of carbon moisture content on Hg^0 removal for FGD carbon (C:Hg=10000:1) in methane flame flue gas at 100 °C and 86 ppbv Hg^0. Error bars represent 1 standard deviation (n=3).

The effect of vapor-phase moisture was also studied in N_2. The addition of moisture to the gas stream resulted in a slight decrease in Hg^0 removal. Water molecules may be forming hydrogen bonds with the carbon, thus reducing the number of active sites available for Hg^0. Injecting a dry carbon into a wet gas stream did not improve Hg^0 removal. The vapor-phase moisture may not have had sufficient time to diffuse into the carbon pore structure and modify the carbon-oxygen functional groups that may be the active sites for adsorption.

The presence of SO_2 in the flue gas had an effect on Hg^0 removal using the WPL (C:Hg=3100:1) carbon, but not the FGD (C:Hg=10000:1) carbon. Further tests are planned to examine the effect of SO_2 and other acid gases on Hg^0 removal using activated carbon.

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16. ABSTRACT The paper gives results of studying the effect of vapor-phase moisture on elemental mercury (Hgo) removal by activated carbon (AC) in a flow reactor. Tests involved injecting AC into both a dry and a 4% moisture nitrogen (N2)/Hgo gas stream. A bituminous-coal-based AC (Calgon WPL) was injected into an Hgo-laden gas stream (124 ppbv) at 150 C at carbon-to-mercury ratios (C:Hg) between 1300 and 4400 :1. Adding 4% (vol.) water (H2O) into the gas resulted in about a 10% drop in removal (21-80%), compared to tests in dry N2 (26-89%). The H2O molecules probably form hydrogen bonds with the AC, reducing the number of active sites available for Hgo. The effect of AC moisture content on Hgo removal was also studied. Darco FGD (lignite-based) AC was injected at 100 C and 86 ppbv Hgo. At a C:Hg of 11000:1, Hgo removal for as-received Darco FGD AC (3% moisture) was 30%. Increasing the moisture content of the AC to 16% resulted in an Hgo removal of 60%. Similar tests, in which the surface moisture of the AC was reduced to 0%, resulted in an Hgo removal of < 5%. Similar results were obtained with WPL. The effect of AC moisture content on Hgo removal was also tested in a simulated flue gas (small positive impact with FGD; no impact with WPL). Injecting dry AC into a wet (4% moisture) N2 stream did not increase Hgo capture. The moisture probably had insufficient time to diffuse into the AC and modify the carbon-oxygen functional groups that may be responsible for adsorption.

17.	KEY WORDS AND DOCUMENT ANALYSIS	
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