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Modeling of Mercury Oxidation and Adsorption by Cupric Chloride-Impregnated Carbon Sorbents

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Cupric chloride-impregnated activated carbon sorbents (CuCl₂-ACs) showed good performance in Hg⁰ oxidation and adsorption in previously published entrained-flow test results. In addition, our previous studies indicated that Hg⁰ is oxidized by CuCl₂-ACs and the resultant oxidized mercury is readsorbed onto the sorbents. This study conducted mathematical modeling to interpret these previous results and better understand the Hg⁰ oxidation and adsorption mechanism of CuCl₂-ACs. As a result, the mathematical modeling results confirmed that Hg⁰ oxidation by CuCl₂-ACs is determined by the injection level of CuCl₂ impregnated onto the sorbents. An increase in CuCl₂ loading in CuCl₂-ACs was found to increase Hg⁰ oxidation but very slightly increased Hg adsorption because an increase in CuCl₂ loading decreased the active CuCl₂-free carbon sites available for the readsorption of resultant oxidized mercury on the surfaces and inside the pores.

1. Introduction

The performance of mercury sorbents has been tested at bench and pilot levels to reduce mercury emissions from coal-fired power plants. Among the sorbents, raw activated carbon has been extensively studied and has shown the capability to adsorb elemental mercury (Hg⁰) in the presence of other gaseous compounds such as hydrogen chloride (HCl).^{1–4} Preimpregnation of halogen compounds onto raw activated carbon has been reported to significantly enhance Hg⁰ adsorption capability of activated carbon.^{3,5–8} Hence chemically treated carbon has also demonstrated good performance in mercury emissions control from subbituminous coal and lignite-burning units which typically generate flue gases with low HCl concentration.⁵ However, while the performance of chemically treated activated carbons in Hg adsorption has been examined in these laboratory and field studies and the kinetic models of Hg adsorption onto raw activated carbon have been reported in several studies,^{9–11} very few publications are found to investigate the kinetics and mechanisms for Hg⁰ oxidation and adsorption by chemically treated carbon sorbents.

Cupric chloride-impregnated activated carbon sorbents (CuCl₂-ACs) demonstrated very similar performance in elemental mercury (Hg⁰) removal to commercial brominated activated carbon (DARCO Hg-LH) in our previous entrained-flow tests.¹² In addition, Hg⁰ oxidation and adsorption characteristics of CuCl₂-ACs were investigated through our previous experimental studies using fixed-bed and entrained-flow reactor systems.^{12,13} These results indicated that CuCl₂-ACs have different sites for Hg⁰ oxidation and adsorption. In addition, significant amounts of effluent oxidized mercury (Hg²⁺) were found from the entrained-flow tests of CuCl₂-ACs. While the amount of effluent oxidized mercury was not dependent on the injection rate of

CuCl₂ impregnated onto CuCl₂-ACs, the sum of Hg adsorption and effluent oxidized mercury was found to be dependent on the CuCl₂ injection rate and increased with an increase in the CuCl₂ injection rate. Almost all of the effluent oxidized mercury was also found to be readsorbed onto CuCl₂-ACs in the filter connected at the end of the entrained-flow portion due to efficient contact with sorbent particles. Therefore, these previous results indicated that elemental mercury (Hg⁰) is oxidized by CuCl₂ impregnated onto CuCl₂-ACs and then resultant oxidized mercury seeks different sites of CuCl₂-ACs for readsorption.^{12,13} On the basis of these results, this study conducted further experimental tests and mathematical modeling to understand and ascertain Hg⁰ oxidation and adsorption mechanism of CuCl₂-ACs.

2. Thermochemical Equilibrium

Thermochemical equilibrium calculations were conducted to find applicable reactions for Hg⁰ oxidation in our entrained-flow tests conducted with a mercury doped air flow. In these calculations, the following reaction equations were assumed to be applicable for Hg⁰ oxidation in our entrained-flow tests:



The enthalpy and Gibbs free energy values for each compound at 300, 400, and 500 K were obtained from the NIST-JANAF thermochemical tables¹⁴ and were determined for each reaction. On the basis of the Gibbs free energy values, the equilibrium constant values were determined for each reaction at different temperatures as summarized in Table 1. On the basis of the equilibrium constant values, Hg⁰ oxidation percentages at equilibrium were also determined with respect to temperature. As shown in the table, the formations of mercuric chloride (HgCl₂) from reactions 1 and 2 between Hg⁰ and CuCl₂ were

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Table 1. Summary of Thermochemical Values for the Reactions Considered in the Entrained-Flow Tests with Conditions of 6.5 ppbv Inlet Hg⁰ and 21% Inlet O₂

reaction	temperature (K)	ΔH^0 (kJ/mol)	ΔG^0 (kJ/mol)	equilibrium constant	Hg ⁰ oxidation percent (%)
1	300	-72.2	-95.4	4.1×10^{16}	100.0
	400	-72.1	-103.1	2.9×10^{13}	100.0
	500	-71.9	-110.9	3.8×10^{11}	100.0
2	300	-1.9	-15.3	460	99.8
	400	-2.9	-19.6	365	99.7
	500	-3.9	-23.7	298	99.7
3	300	-209.5	211.4	1.5×10^{-37}	0.0
	400	-210.2	202.2	3.9×10^{-27}	0.0
	500	-210.8	193.4	6.3×10^{-21}	0.0

found to be most probable in our entrained-flow tests, while the formation of mercurous chloride (HgCl) shown in reaction 3 is not.

3. Experimental Section

A 1.6% cupric chloride-impregnated clay sorbent and 3.8%, 6.5%, and 10% cupric chloride-impregnated carbon sorbents synthesized in our laboratory were used in this study. The detailed preparation procedures and physical properties of these sorbents can be found in our previous publication.^{12,15} Figure 1 also presents a schematic of the fixed-bed system and a summary of the experimental conditions. The detailed descriptions of the fixed-bed system and the experimental procedures can be found in our previous publication.¹⁶

4. Results and Discussion

4.1. Fixed-Bed Test Results. The thermochemical calculations in the previous section showed that HgCl₂ is the most probable reaction product as a result of the reaction between Hg⁰ and CuCl₂. To find its reaction molar ratio, 1.6% cupric chloride-impregnated clay sorbent (1.6% CuCl₂-clay), which was found in our previous studies^{15,16} to mostly oxidize Hg⁰ rather than adsorb, was tested in the fixed-bed system. During this test, the outlet Hg⁰ and Hg²⁺ concentrations for each 12-h segment (total of 96 h) were determined from the measurements by the Ontario Hydro Method.¹⁷ Figure 2 shows that 1.6% CuCl₂-clay has the capability to oxidize Hg⁰, and its oxidation capability is almost complete at 96 h of the test. On the basis of this result, the Hg⁰ oxidation capacity of 1.6% CuCl₂-clay sorbent was determined to be 11 μg Hg⁰/mg sorbent. It is approximately equivalent to the reaction molar ratio of 1:2 between Hg⁰ and CuCl₂. Therefore, reaction 1 was assumed in this study to be a main reaction pathway for Hg⁰ oxidation by cupric chloride-impregnated sorbents.

A further fixed-bed test was conducted with 6.5% CuCl₂-AC at 7 ppbv inlet Hg⁰ concentration. Figure 3 shows the outlet Hg⁰ and Hg²⁺ concentrations determined for each 12-h segment (total 72 h) from this test. It shows that almost the entire inlet Hg⁰ was directly adsorbed onto 6.5% CuCl₂-AC or converted into oxidized mercury during the first 36 h of the test while most of the total Hg adsorption onto the sorbent during the entire test was achieved during the first 36 h. Since our previous studies showed that Hg⁰ may not be directly adsorbed onto CuCl₂-impregnated sites of CuCl₂-ACs as mentioned earlier in the Introduction, this test result suggests that almost the entire inlet Hg⁰ is oxidized by CuCl₂-AC while Hg adsorption onto CuCl₂-AC occurs.

Hg adsorption capacities of CuCl₂-ACs were also determined with different CuCl₂ loadings and different inlet Hg⁰ concentrations using the fixed-bed system. During these tests, the outlet

Hg⁰ and Hg²⁺ concentrations for a certain time segment were determined from the measurements by the Ontario Hydro Method.¹⁷ As found in the results of using 6.5% CuCl₂-AC at a 7 ppbv inlet Hg⁰ concentration in Figure 3, Hg adsorption onto 6.5% CuCl₂-AC was essentially complete after around 60 h of the test, although Hg⁰ is continuously oxidized. Therefore, each fixed-bed test was conducted until the sum of the outlet concentrations of Hg⁰ and Hg²⁺ reached the inlet Hg⁰ concentration. The amount of Hg adsorption onto each sorbent was determined after the completion of each test by the digestion procedure described in the Ontario Hydro Method. Figure 4 illustrates the Hg adsorption capacities of 3.8%, 6.5%, and 10% (w) CuCl₂-ACs with respect to the inlet Hg⁰ concentration. As shown in Figure 4, CuCl₂-AC with lower CuCl₂ loading showed higher Hg adsorption capacity. This indicates that an increased CuCl₂ loading in CuCl₂-AC may decrease the active sites available for the adsorption of the resultant oxidized mercury. The adsorption capacities of the resultant oxidized mercury determined for each CuCl₂ loading with different inlet Hg⁰ concentrations were expressed by the following Freundlich isotherm model:

$$q = \frac{1}{K} C_e^{1/n} \quad (5)$$

where C_e is the equilibrium concentration of the resultant oxidized mercury in the gas phase (μmol Hg/m³); q is the equilibrium mercury capacity per unit mass of a sorbent (μmol Hg/g sorbent); K and n are parameters. The test results of CuCl₂-ACs shown in Figure 3 indicated that almost the entire inlet Hg⁰ is converted into an oxidized form of mercury (assumed as HgCl₂) while resultant oxidized mercury adsorption onto CuCl₂-AC occurs. Therefore, those isotherm parameters determined from the tests with inlet Hg⁰ for each CuCl₂ loading will be used in the following mathematical modeling to provide equilibrium data for the readsorption of the resultant oxidized mercury in an entrained-flow reactor system.

4.2. Mathematical Models of Hg⁰ Oxidation and Hg Adsorption in the Entrained-Flow Reactor. On the basis of Hg⁰ oxidation and adsorption mechanisms suggested from our previous studies^{12,13} as discussed in the Introduction, Table 2 summarizes the reaction and mass balance equations used to model Hg⁰ oxidation and subsequent Hg adsorption by CuCl₂-ACs. In this mathematical modeling, CuCl₂-AC sorbent particles were assumed to be spherical and uniformly distributed in the entrained-flow reactor. In addition, our previous study¹² reported that additional mercury capture by the sorbent deposited on the entrained-flow reactor wall was minimized through several experimental modifications such as coinjection of montmorillonite clay, relocation of sorbent injection, and vibration of reactor. Therefore, the gas flow in the entrained-flow reactor was also assumed to be plug flow.

The Hg⁰ oxidation column of Table 2 shows mass balance equations for the chemical reaction between Hg⁰ and CuCl₂ impregnated onto CuCl₂-ACs. Since the sorbent mass was found to be negligibly changed by the reaction with Hg⁰ based on mass balance calculations, the sorbent loading (m_r/Q) was assumed to be constant with the reaction time in those equations. The equations were solved to determine the extent of Hg⁰ oxidation with respect to sorbent loading at a residence time of 0.75 s. Two observed kinetic parameters of the reaction rate constant (k_1) and the reaction order (N) were determined for all CuCl₂ loadings. As shown in Figure 5, the model predictions correspond well to the experimental results for Hg⁰-removal total of CuCl₂-ACs in the entrained-flow reactor. Since Hg⁰-removal

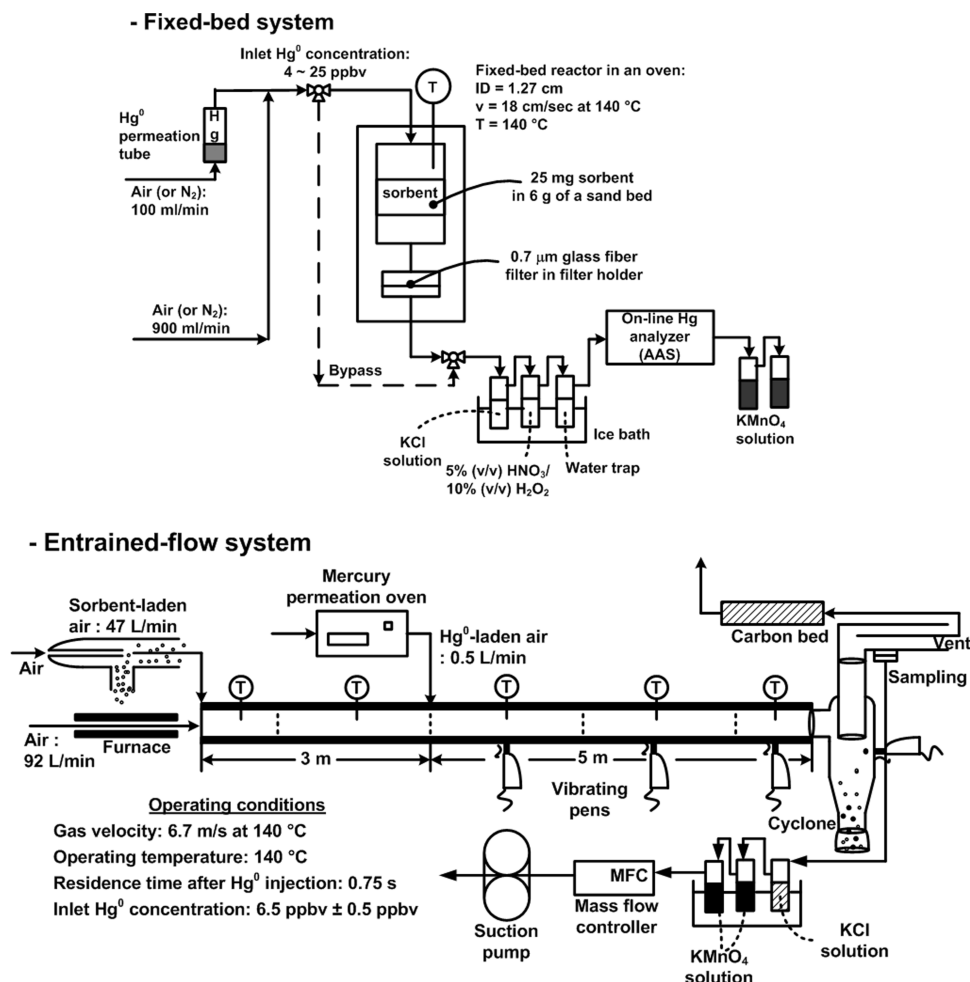


Figure 1. Schematics of the fixed-bed system (top) and the entrained-flow system (bottom).

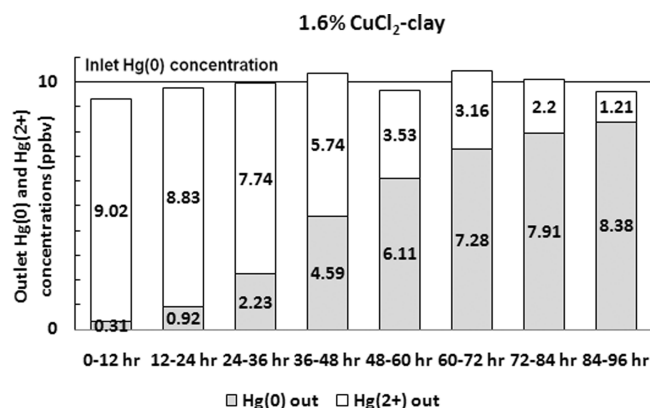


Figure 2. Outlet elemental mercury (Hg⁰) and oxidized mercury (Hg²⁺) concentrations measured for each 12 h of fixed-bed tests of 1.6% CuCl₂-clay at 10 ppbv inlet Hg⁰ concentration.

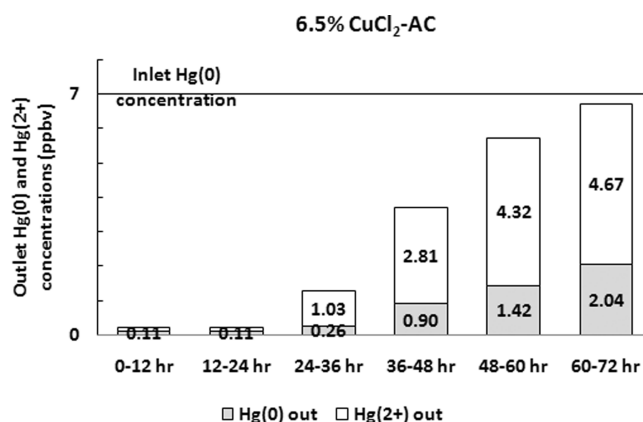


Figure 3. Outlet elemental mercury (Hg⁰) and oxidized mercury (Hg²⁺) concentrations measured for each 12 h of fixed-bed tests of 6.5% CuCl₂-AC at 7 ppbv inlet Hg⁰ concentration.

total is determined from the sum of Hg adsorption and effluent oxidized mercury, these modeling results show that the Hg⁰-removal total reflects the total Hg⁰ oxidation (including adsorbed mercury onto the sorbent (Hg_{ads}) and oxidized mercury entrained to the gas phase (Hg_{out}²⁺)) achieved by the reaction between Hg⁰ and CuCl₂ impregnated onto CuCl₂-ACs. Therefore, Hg⁰-removal total was found to increase with an increase in the injection rate of CuCl₂ which depends on the sorbent injection rate and the CuCl₂ loading in CuCl₂-ACs in our entrained-flow tests.

The Hg adsorption column of Table 2 summarizes mass balance equations for readsorption of the resultant oxidized mercury (assumed as HgCl₂). The HgCl₂ concentration in the sorbent (C_c) was determined by the surface area (S) of the sorbent and mass-transfer coefficient (k_c). The equilibrium concentration of HgCl₂ (C_A^*) was obtained from the Freundlich isotherm model shown in eq 5. In addition, the mass-transfer coefficient (k_c) was determined from the correlation with the Sherwood number (Sh) which is generally applied for the flow past single spheres in Reynolds numbers up to 1000.¹⁸

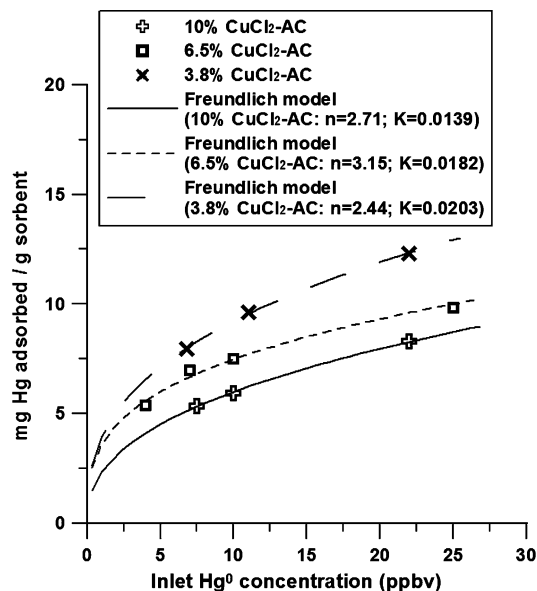


Figure 4. Adsorption isotherms for 3.8%, 6.5%, and 10% CuCl₂-ACs obtained from the fixed-bed tests at 140 °C under air flow.

Table 2. Summary of Reaction and Mass Balance Equations Considered for Mathematical Modeling

	Hg ⁰ oxidation	Hg adsorption
reaction	$\text{Hg}^0(\text{g}) + 2\text{CuCl}_2(\text{s}) \rightarrow \text{HgCl}_2(\text{g}) + 2\text{CuCl}(\text{s})$	$\text{Hg}^0(\text{g}) + 2\text{CuCl}_2(\text{s}) \rightarrow \text{HgCl}_2(\text{g}) + 2\text{CuCl}(\text{s})$ $\text{HgCl}_2(\text{g}) + \text{C}(\text{s}) \rightarrow \text{HgCl}_2 \cdot \text{C}(\text{s})$
mass balance equations	(1) Gas-phase Hg ⁰ concentration (C_H) $-(dC_H/d\tau) = k_1(C_H)^N(1/\alpha)C_B(m_t/Q)$ when $\tau = 0$, $C_H = 0.192$ $\mu\text{mol}/\text{m}^3$ $C_B = C_{B0}$ (2) Mass fraction of CuCl ₂ in sorbent (C_B) $C_B = C_{B0} - A(Q/m_t)(C_{H0} - C_H)\alpha$	(1) Gas-phase Hg ⁰ concentration (C_H) $-(dC_H/d\tau) = k_1(C_H)^N(1/\alpha)C_B(m_t/Q)$ when $\tau = 0$, $C_H = 0.192$ $\mu\text{mol}/\text{m}^3$ $C_B = C_{B0}$ (2) Mass fraction of CuCl ₂ in sorbent (C_B) $C_B = C_{B0} - A(Q/m_t)(C_{H0} - C_H)\alpha$ (3) HgCl ₂ concentration in sorbent (C_C) $(dC_C/d\tau) = S\beta k_c(C_A - C_C^*)$ when $\tau = 0$, $C_H = 0.192$ $\mu\text{mol}/\text{m}^3$ $C_B = C_{B0}$ $C_A = 0$ $C_C = 0$ (4) Surface area of sorbent (S) $S = S_0 - (S_0/q)C_C$ (5) Gas-phase HgCl ₂ concentration (C_A) $C_A = C_{H0} - C_H - C_C(m_t/Q)$

$$Sh = 2 + 0.6Re^{0.5}Sc^{1/3} = \frac{d}{D}k_c \quad (6)$$

where Re is the Reynolds number of a single sphere; Sc is the Schmidt number; d is the particle diameter of sorbent (m); D is the gas-phase molecular diffusivity of HgCl₂ (m²/s). These values were obtained from the entrained-flow experimental conditions and the physical properties of CuCl₂-ACs which can be found in our previous publication.¹² These five balance equations were solved to have Hg adsorption onto CuCl₂-ACs (Hg_{ads}) with respect to sorbent loading (sorbent injection rate) in the entrained-flow reactor for each CuCl₂ loading in sorbent.

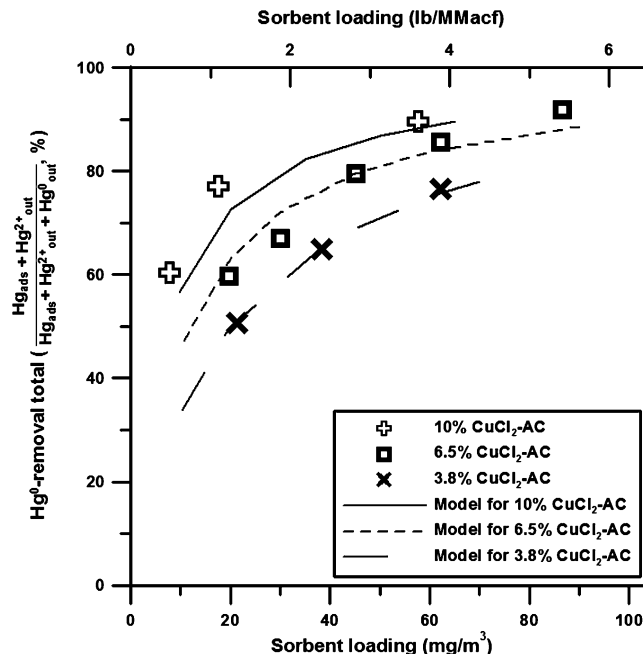


Figure 5. Comparison between entrained-flow experimental results¹² and modeling results for Hg⁰ oxidation by CuCl₂-ACs ($k_1 = 18615$ (m³)²/μmol·g·s, $N = 2$).

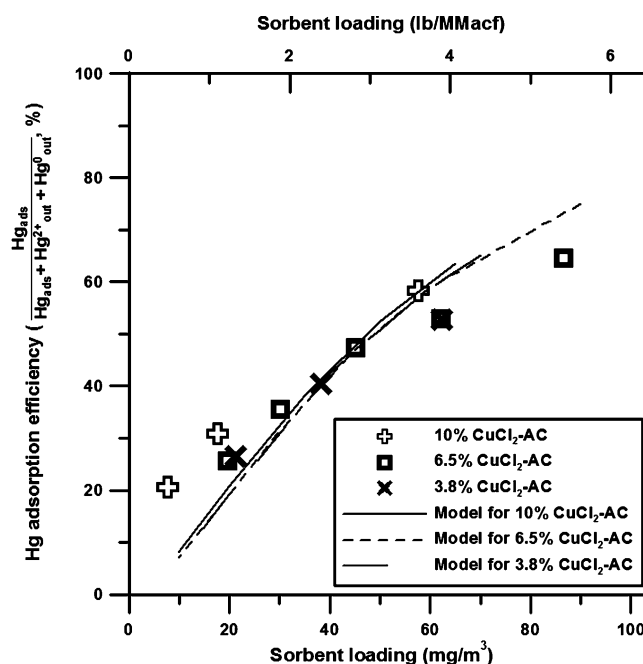


Figure 6. Comparison between entrained-flow experimental results¹² and modeling results for Hg adsorption onto CuCl₂-ACs ($\beta = 0.026$ for 10% CuCl₂-AC, $\beta = 0.030$ for 6.5% CuCl₂-AC, and $\beta = 0.044$ for 3.8% CuCl₂-AC).

One parameter, β , was used to apply the surface area of CuCl₂-AC available for adsorption. The parameter, β , was determined for each CuCl₂ loading because CuCl₂-ACs with different CuCl₂ loadings may have different surface areas available for Hg adsorption as found in the adsorption isotherm results in Figure 4. Figure 6 shows that the model predictions represent the experimental Hg adsorption results with respect to sorbent loading and CuCl₂ loading in the sorbent. The parameter of β decreased from 4.4 to 2.6% with an increase in CuCl₂ loading from 3.8 to 10% in the sorbent. Since 3.8%, 6.5%, and 10% (w) CuCl₂-ACs were found to have similar total surface areas

each other from the previous measurements,¹² this result shows that CuCl₂-AC with higher CuCl₂ loading has less active sites available for the readsorption of the resultant oxidized mercury. Therefore, although Hg⁰ oxidation by CuCl₂-AC increased with an increase in CuCl₂ loading, Hg adsorption onto CuCl₂-AC did not significantly increase with an increase in CuCl₂ loading in its range from 3.8% to 10%.

5. Conclusions

On the basis of the mechanisms suggested from entrained-flow and fixed-bed test results, Hg⁰ oxidation by CuCl₂-ACs and the readsorption of the resultant oxidized mercury onto these sorbents were sequentially modeled in this study. As found in our previous entrained-flow test results, the mathematical modeling demonstrated that the sum of Hg adsorption and effluent oxidized mercury reflects the total amount of Hg⁰ oxidation by CuCl₂-ACs and is determined by the injection level of CuCl₂ impregnated onto CuCl₂-ACs. The modeling results also showed that while an increase in CuCl₂ loading of the sorbent increases Hg⁰ oxidation capability of CuCl₂-ACs, it very slightly increases their Hg adsorption capability because an increase in CuCl₂ loading decreases the active CuCl₂-free carbon sites available for the readsorption of the resultant oxidized mercury on the surfaces and inside the pores. This study assumed that HgCl₂ is produced from the reaction between Hg⁰ and CuCl₂ in a 1:2 molar ratio based on the thermochemical calculations and fixed-bed test results. Further experiments with HgCl₂ would clarify the Hg⁰ oxidation mechanism by CuCl₂-ACs. In addition, reaction mechanism is to be studied by X-ray absorption fine-structure spectroscopy in order to identify the mercury species generated as a result of the reaction and a change in the oxidation state of copper.

Nomenclature

A = conversion factor = 0.000 134 5 g CuCl₂/μmol CuCl₂
 C_A = gas-phase HgCl₂ concentration, μmol Hg/m³
 C_A^* = gas-phase HgCl₂ concentration in equilibrium with sorbent, μmol Hg/m³
 C_B = mass fraction of CuCl₂ in sorbent
 C_{B0} = initial mass fraction of CuCl₂ in sorbent
 C_C = HgCl₂ concentration in sorbent, μmol Hg/g sorbent
 C_H = gas-phase Hg⁰ concentration, μmol Hg/m³
 C_{H0} = initial gas-phase Hg⁰ concentration, μmol Hg/m³
 C_e = equilibrium HgCl₂ concentration in the gas phase, μmol Hg/m³
 d = particle diameter of sorbent = (23 μm for 3.8%, 6.5%, and 10% CuCl₂-ACs)
 D = gas-phase diffusivity of HgCl₂, m²/s
 K = parameter of Freundlich equation, g/m³
 k_1 = reaction rate constant, m^{3N}/μmol^{N-1}·g·s
 k_c = mass-transfer coefficient, m/s
 m_t = sorbent injection rate, g sorbent/s
 m_t/Q = sorbent loading, g sorbent/m³
 N = reaction order
 n = parameter of Freundlich equation
 Q = gas flow rate, m³/s
 q = mercury concentration in sorbent in equilibrium with gas-phase mercury, μmol Hg/g sorbent
 Re = Reynolds number of a single sphere

S = surface area of sorbent, m²/g sorbent

Sc = Schmidt number

S_0 = initial surface area of sorbent, m²/g sorbent

α = moles of CuCl₂ used to oxidize 1 mol of Hg⁰ = 2

β = fraction of active surface area for adsorption in the total surface area of sorbent

τ = residence time of sorbent in the entrained-flow reactor, s

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