



# The role of iodine monochloride for the oxidation of elemental mercury

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## ABSTRACT

The removal of  $\text{Hg}^0$  by the homogenous gas-phase reaction and particle-induced reaction was investigated under various conditions. Iodine monochloride was found to be efficient for  $\text{Hg}^0$  oxidation, with the apparent 2nd-order rate constant of about  $10.5(\pm 0.3) \times 10^{-17} \text{ cm}^3 \text{ molecules}^{-1} \text{ s}^{-1}$  and  $5.7(\pm 0.3) \times 10^{-17} \text{ cm}^3 \text{ molecules}^{-1} \text{ s}^{-1}$  at 273 K and 373 K, respectively. The pilot-scale tests showed that the removal of  $\text{Hg}^0$  by ICl increased significantly in presence of flyash. It was predicted that over 90% of  $\text{Hg}^0$  removal efficiency can be obtained with 0.2 ppmv ICl and 20 g/m<sup>3</sup> flyash in flue gas. Though the reaction between  $\text{Hg}^0$  and ICl was by far faster than that of  $\text{Hg}^0/\text{Cl}_2$ , the major product was found to be  $\text{HgCl}_2$  rather than  $\text{HgI}_2$ , which implicated that iodine might partly act as the accelerant in  $\text{Hg}^0$  oxidation by facilitating the formation of certain intermediates. The results indicated that using ICl to oxidize elemental mercury in coal-fired flue gas can save the consumption of iodine, and it appeared to be a promising oxidant to enhance the removal of  $\text{Hg}^0$ .

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## 1. Introduction

Iodine monochloride (ICl) has been reported to be a typical interhalogen of iodine and chlorine, which can be formed from the atmospheric chemical reaction or synthesized artificially. Iodine was found at parts per trillion (ppt) levels in the coastal marine boundary layer (MBL), and it can be further converted to other iodine compounds by a series of photochemical reactions [1–3]. Among these iodine compounds, hypoiodous acid (HOI) has been proposed to enhance chlorine activation and release iodine monochloride (ICl) which could accelerate ozone depletion [4–6]. The reactions between ozone and halogen species are known to be responsible not only for the ozone depletion events, but also for the depletions of tropospheric mercury [7–9]. Meanwhile, the thermochemistry research of mercury and ICl showed that it is possible for ICl to oxidize  $\text{Hg}^0$  to a form which has a much higher deposition rate [10]. Therefore, ICl might play an important role in the atmospheric mercury transformation and the global cycling of mercury.

In addition, ICl might be a potential oxidant to be used in coal-fired flue gas to enhance the mercury capture. As we have known, mercury emission from coal-fired power plants has become the worldwide predominant anthropogenic source, and some effective methods are demanded to reduce the emission. There are three

basic forms of mercury in flue gas: particulate-bound mercury, oxidized mercury and elemental mercury [11]. The particulate-bound mercury and oxidized mercury could be captured by existing air pollution control devices (APCDs), such as electrostatic precipitator (ESP), fabric filters (FF), and flue gas desulfurization (FGD) [11–13]. However, it is still difficult to directly remove  $\text{Hg}^0$  from flue gas with these APCDs because of its high volatility and insolubility in water [14]. Therefore, the additional methods to convert  $\text{Hg}^0$  to its oxidized form in or ahead of APCDs are necessary for the effective capture of  $\text{Hg}^0$ . According to our previous studies, the removal efficiency of mercury could be enhanced by using halogens, such as bromine, iodine [15,16], and iodine was found to be the most efficient oxidant. However, iodine is not the usual component of flue gas as chlorine, and it have to be added to coals or flue gas when employed as the oxidant, which may bring unexpected pollution problems if too much iodine was used in flue gas. Furthermore, iodine is more expensive than chlorine. Thus, it will be helpful to find a substitute which could reduce the consumption of iodine. As mentioned above,  $\text{Hg}^0$  could be oxidized by ICl, and it may be a compromising candidate to replace iodine. Nevertheless, the exact mechanism and the kinetics of the reaction between ICl and  $\text{Hg}^0$ , and the role of ICl in the mercury removal from flue gas and the atmospheric mercury transformation have not been well understood. According to the above considerations, a series of experiments were performed to investigate the performance of ICl on the removal of elemental mercury. The mechanism of the reaction was also discussed.

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## 2. Experimental methods and materials

### 2.1. Materials

The chemicals employed were as follows: mercury (99.99%), chlorine (99.9%), iodine (99.9%) and iodine monochloride (99%) from Sigma–Aldrich Co.; halocarbon wax (HW, Series-1500) from Halocarbon Product Co. NO (10.0%), SO<sub>2</sub> (99.9%), CO<sub>2</sub> (10.0%), and N<sub>2</sub> (99.9%) stored in the cylinders were from Dalian Date Gas Co. Ltd. Flyash was provided by Wujing coal-fired power plant. Darco-Hg-LH (task-specific activated carbon) and Darco-KB (all-purpose activated carbon) were from Norit America Company.

The loss of ignition (LOI) and BET surface of flyash was about 1.6(±0.2)% and 2.0(±0.2) m<sup>2</sup>/g, respectively. In order to test the effect of LOI, flyash was calcined at 1073 K for 3 h and employed in this research (which was marked as flyash\*, and the LOI of flyash\* near zero after be calcined). The main components of flyash were 12(±2)% CaO, 22(±2)% Al<sub>2</sub>O<sub>3</sub>, 8(±1)% Fe<sub>2</sub>O<sub>3</sub>, 1.2(±0.5)% Na<sub>2</sub>O, which was analyzed by inductively coupled plasma (ICP) spectrometer (Iris Advantage 1000, Thermo). Ionic chromatography (MIC, Metrohm) was employed to analyze the product from the reaction between Hg<sup>0</sup> and ICl.

### 2.2. Experimental methods

#### 2.2.1. The UV/vis test method

Iodine monochloride (99%) was ordered from Sigma–Aldrich Co. It can also be produced from the reaction of iodine and chlorine readily (Eq. (1)). Though the reaction is reversible, the equilibrium constant for the positive reaction is very large, about 2 × 10<sup>5</sup> at 298 K, which indicates that ICl is a very stable compound at room temperature.



However, there still lacks the necessary information about the equilibrium constant at higher temperature, which is an important parameter if ICl is employed in industry. Therefore, we performed an experiment to test the equilibrium constant at 373 K. The detail process was as follows: (1) Taking out 5 mL ICl and keep it in a 50 mL brown bottle. (2) Maintaining the brown bottle at 298 K. (3) Taking out 5 mL saturation vapor of ICl from the headspace of brown bottle one hour later. (4) Injecting the 5 mL ICl vapor into 250 mL cell which was maintained at 373 K. Then, the decomposition process of iodine monochloride in the cell was in situ monitored and recorded by the UV/vis spectrometer (Bwtek, BRC641E, USA) equipped with the optical fiber for UV-beam transmission, CCD detector (2048 × 14) and recorder in the range of 200–800 nm, with 0.4 nm for the resolving wavelength and 200 ms for the minimum data acquisition intervals. In order to identify the mechanism of the reaction between Hg<sup>0</sup> and iodine monochloride, the reaction process were also recorded by the UV/vis spectrometer. The maximum adsorption of UV for iodine, iodine monochloride and chlorine was around at the wavelength of 530 nm, 467 nm and 330 nm, respectively [17–19]. The concentration of I<sub>2</sub>, ICl and Cl<sub>2</sub> could be calculated according to the following formula:

$$I = \varepsilon LC \quad (2)$$

where  $I$  is the intensity of the absorption peak;  $\varepsilon$  is the absorption coefficient (cm<sup>2</sup> molecules<sup>-1</sup>);  $L$  is the optical path length in the cell (cm); and  $C$  is the concentration of I<sub>2</sub>, ICl or Cl<sub>2</sub> (molecules cm<sup>-3</sup>).

#### 2.2.2. The gas-phase reaction test method

The reaction kinetics between Hg<sup>0</sup> and ICl were investigated with in situ monitoring the change of the concentration of Hg<sup>0</sup> with the process of reaction by a mercury cold vapor atomic absorption spectrophotometer (CVAAS), which was similar to the method used in our previous studies [16,20]. In order to improve the sensitivity of the system, a reference beam was added to minimize the noise in the monitoring signal. The tests were mainly performed in a stainless steel cylinder with the volume of 1100 mL. The signal was collected with a data transition and acquisition device (N2000, Zhejiang Zhida) and recorded by a computer, and the minimum data acquisition interval was 20 ms [20]. The Hg<sup>0</sup> concentration employed in this paper was about 160 ppbv (1 ppbv = 10<sup>-9</sup> by volume). The wall of reactor was coated with halocarbon wax (HW) to minimize Hg<sup>0</sup> adsorption and the surface-induced reaction by the wall. The contribution of surface-induced reaction to Hg<sup>0</sup> oxidation was evaluated [20]. The Hg<sup>0</sup> adsorbed on the reactor wall was reduced remarkably after it was coated with HW.

#### 2.2.3. The pilot-scale tests

A pilot-scale setup was built to test the performance of ICl on Hg<sup>0</sup> capture from the simulated flue gas. As shown in Fig. 1, an electric heater was used to provide 150–200 m<sup>3</sup>/h hot gas. Meanwhile, SO<sub>2</sub> (1000 ppmv), NO (100 ppmv), water vapor (5%) and CO<sub>2</sub> (5%) were injected into the reactor with the hot gas as the simulated flue gas. The slipstream reactor was a stainless steel cylinder with the inner diameter of 0.1 m and valid length of 5.0 m. 10–40 g/m<sup>3</sup> flyash was injected into the simulated flue gas by a controllable screwing thruster, which could adjust the concentration of flyash or other sorbents (e.g. activated carbon) in flue gas. The average temperature of the flue gas was from 353 K to 393 K, and the gas residence time was about 0.9 s. The initial concentration of Hg<sup>0</sup> in inlet gas of the reactor was maintained at about 20–40 μg/m<sup>3</sup>. The concentration of Hg<sup>0</sup> in flue gas could be detected by Lumex mercury analyzer (Lumex RA915, Russia). The flue gas was sampled from the reactor and passed through a vessel containing saturated NaOH solution and a buffer vessel before detection. The saturated NaOH solution was used to minimize the effect of oxidant gas and flyash on Hg<sup>0</sup> detection. The Hg<sup>0</sup> removal efficiency based on the data detected from the 2# outlet sampling point was 10–15% higher than that from the 1# outlet sampling point. It means Hg<sup>0</sup> could be constantly removed in cyclone because 2# outlet sampling point was in the downstream of cyclone. In order to minimize the effect of cyclone, 1# outlet was employed as outlet sampling point in this research.

The measurement errors of Hg<sup>0</sup> were ±2 ppbv and ±0.05 ppbv for in situ CVAAS and Lumex analyzer, respectively. The accuracy of the data reported here was estimated to be within 10% for the tests.

## 3. Results and discussion

### 3.1. Gas-phase oxidation of Hg<sup>0</sup> by ICl

Because ICl will decompose at high temperature, Cl<sub>2</sub> and I<sub>2</sub>, the decomposition products of ICl, may affect the reaction of Hg<sup>0</sup> and ICl. In order to estimate the exact rate constant of the reaction between Hg<sup>0</sup> and ICl, the contribution of I<sub>2</sub> and Cl<sub>2</sub> to the oxidation of Hg<sup>0</sup> should be deducted by Eq. (3):

$$\frac{d[\text{Hg}^0]}{dt} = -\{k_{\text{ICl}}[\text{ICl}] + k_{\text{I}_2}[\text{I}_2] + k_{\text{Cl}_2}[\text{Cl}_2]\} \cdot [\text{Hg}^0] \quad (3)$$

where  $k_{\text{ICl}}$ ,  $k_{\text{I}_2}$  and  $k_{\text{Cl}_2}$  are the reaction rate constant of Hg<sup>0</sup>/ICl, Hg<sup>0</sup>/I<sub>2</sub> and Hg<sup>0</sup>/Cl<sub>2</sub>, respectively. In order to acquire the concentrations of I<sub>2</sub> and Cl<sub>2</sub> which were produced because of the

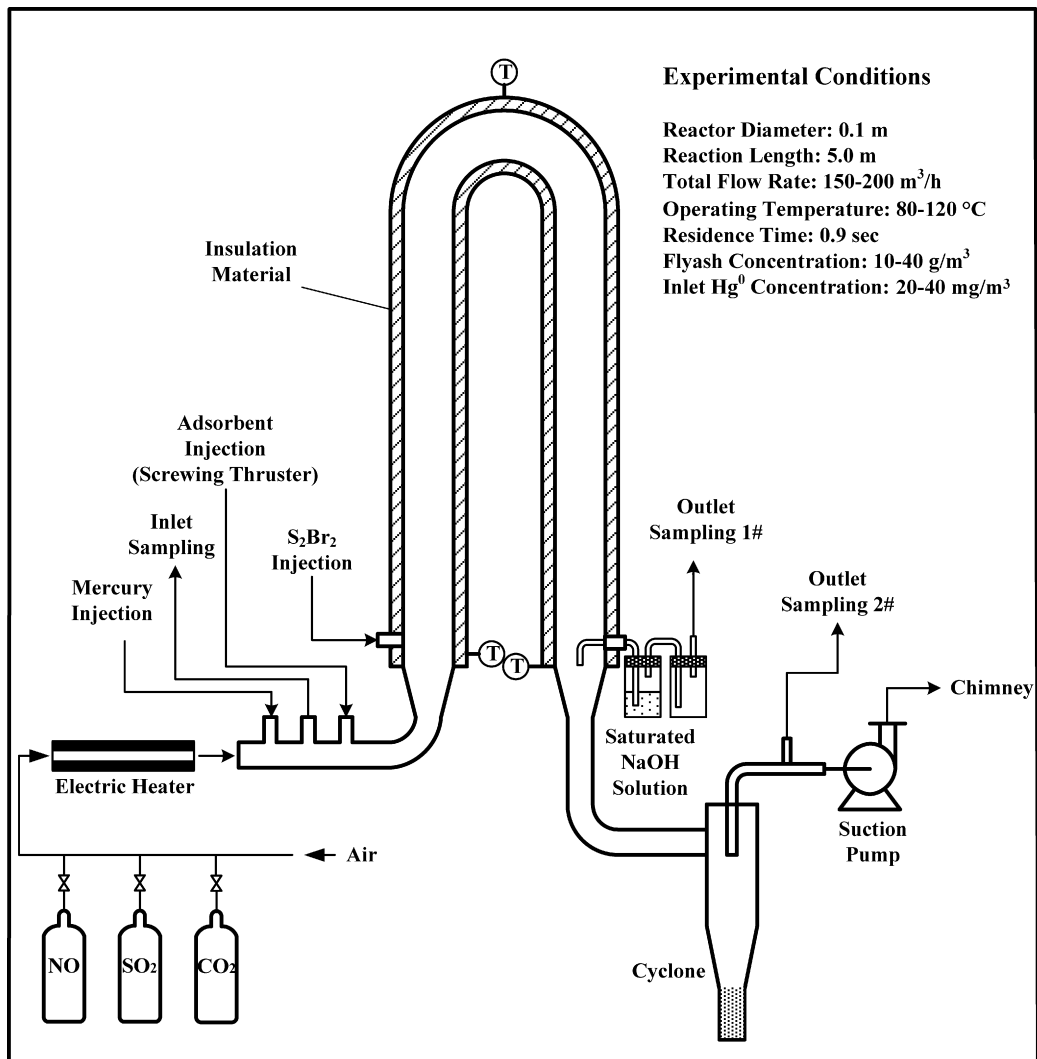


Fig. 1. The schematic of pilot-scale setup for the removal of Hg<sup>0</sup> by ICl and particulates.

decomposition of ICl, the decomposition process of ICl at 373 K was investigated in this paper. According to the experiment data at the beginning time of 3 min, the apparent decomposition rate constant was evaluated to be around  $3.5 \times 10^{-20} \text{ cm}^3 \text{ molecules}^{-1} \text{ s}^{-1}$ . It is very slow compared with the reaction rate of Hg<sup>0</sup> and ICl. Therefore, the decomposition of ICl could be ignored. Accordingly, Eq. (3) could be simplified to Eq. (4):

$$\frac{d[\text{Hg}^0]}{dt} = -k_{\text{ICl}}[\text{ICl}] \cdot [\text{Hg}^0] \quad (4)$$

The removal efficiencies of Hg<sup>0</sup> in the presence of ICl, Cl<sub>2</sub> and I<sub>2</sub> at various concentrations were investigated, and the results were illustrated in Fig. 2. Since the concentration of Cl<sub>2</sub>, I<sub>2</sub> or ICl was much higher than that of Hg<sup>0</sup> in the gas phase, they could be considered as a constant during the reaction. The 2nd rate constant of Hg<sup>0</sup>/ICl,  $k_{\text{ICl}}$ , can be calculated by Eq. (4), and it was  $5.7(\pm 0.3) \times 10^{-17} \text{ cm}^3 \text{ molecules}^{-1} \text{ s}^{-1}$  at 373 K. Apparently, I<sub>2</sub> was the most efficient oxidant in the Hg<sup>0</sup> oxidation, followed by ICl, and Cl<sub>2</sub> was the last one among the three oxidants. However, the apparent 2nd-order rate constant of Hg<sup>0</sup> oxidation increased about 20% with ICl comparing to the sum of that with iodine and chlorine separately. To get the same removal efficiency of Hg<sup>0</sup>, the used amount of iodine atom could be saved 20% if I<sub>2</sub> was substituted by ICl as the oxidant.

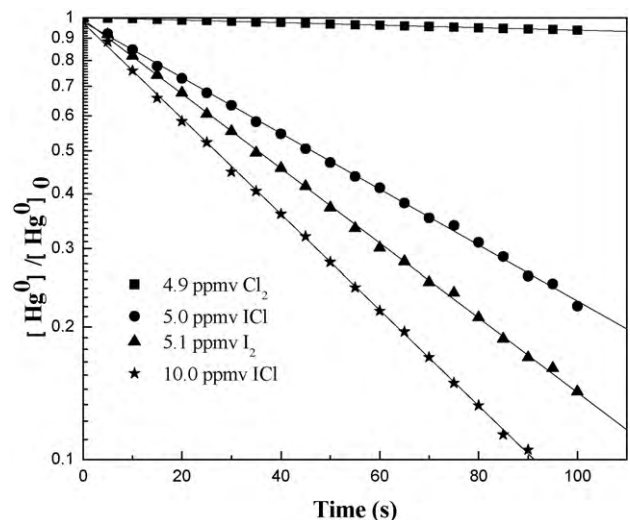
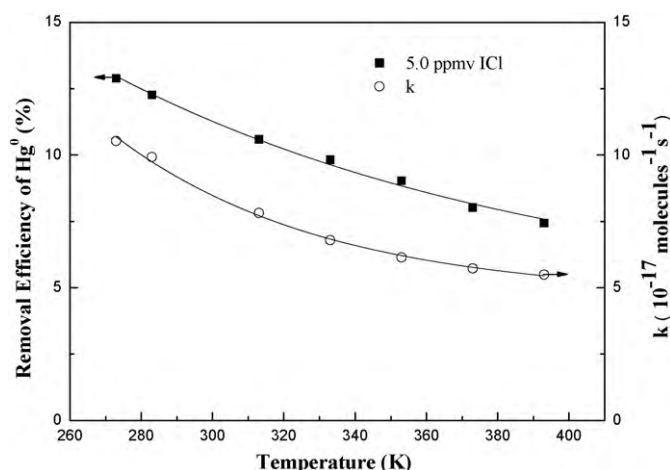


Fig. 2. The concentration depletion curves of Hg<sup>0</sup> by the oxidation of Cl<sub>2</sub>, ICl and I<sub>2</sub>. The initial concentration of Hg<sup>0</sup>, [Hg<sup>0</sup>]<sub>0</sub>, was about 160 ppbv, and the tests were conducted at 373 K and 760 Torr with nitrogen balance.



**Fig. 3.** The temperature dependence of  $\text{Hg}^0$  oxidation efficiencies and the 2nd rate constants with ICl. The initial concentration of  $\text{Hg}^0$ ,  $[\text{Hg}^0]_0$ , was about 160 ppbv, and the reaction time was 5 s at 760 Torr with nitrogen balance.

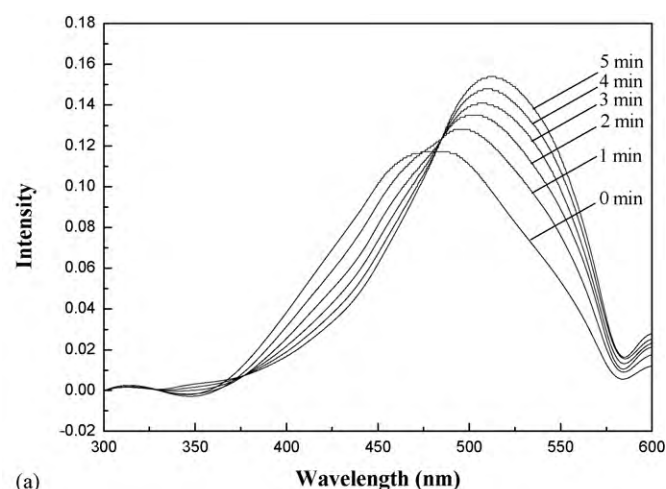
### 3.2. The temperature effect of $\text{Hg}^0$ oxidation by iodine monochloride

In order to investigate the effect of temperature on the  $\text{Hg}^0$  removal, a series of experiments were conducted under different temperatures from 273 K to 393 K. As can be seen in Fig. 3, the removal efficiency of  $\text{Hg}^0$  dropped from 13% to 7% as the temperature increased from 273 K to 393 K with 5.0 ppmv of ICl concentration at 5 s. Meanwhile, the 2nd-order rate constant between  $\text{Hg}^0$  and ICl was estimated to be around  $5.4(\pm 0.3) \times 10^{-17} \text{ cm}^3 \text{ molecules}^{-1} \text{ s}^{-1}$  at 393 K. It was only about 51% of that at 273 K. Although increasing temperature decreased the  $\text{Hg}^0$  oxidation rate and the rate constant, the decreasing tendency slowed down remarkably when the reaction temperature beyond 373 K. Therefore,  $\text{Hg}^0$  could be removed effectively in presence of ICl even under higher temperature in flue gas. According to the reaction rate constant, it could be estimated that the half life of  $\text{Hg}^0$  which means the time required for the concentration of  $\text{Hg}^0$  to fall to one-half of its initial value, will be less than 50 hours with 1 ppbv ICl at 273 K. While, the atmospheric life time of  $\text{Hg}^0$  was believed to be in the range of six months to one year [21]. Therefore, the deposition rate of atmospheric mercury will be improved greatly in the presence of ICl.

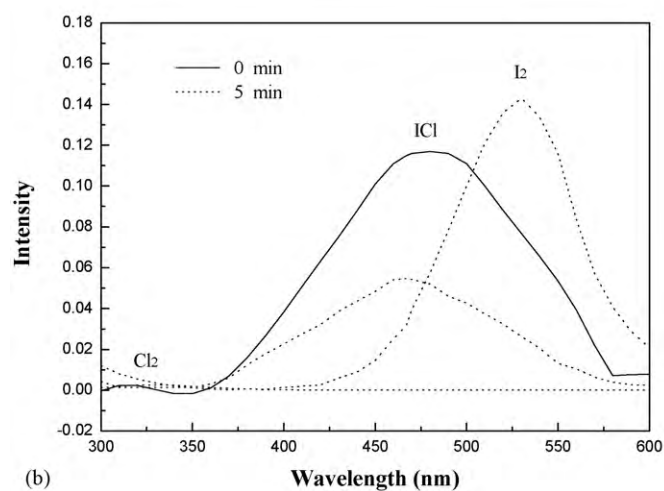
### 3.3. Product analysis and the discussion on the reaction mechanism

In order to probe on the mechanism of the reaction between  $\text{Hg}^0$  and iodine monochloride, the reaction was also recorded by the UV/vis spectrometer, and the results were shown in Fig. 4. As shown in Fig. 4, the absorption peak of 530 nm ( $\text{I}_2$ ) increased with the decrease of the absorption peak of ICl. This result suggested that the iodine was formed with the reaction between  $\text{Hg}^0$  and ICl. Ion chromatography analysis was employed to analyze the oxidized mercury products which were dissolved into the ultrapure water. Since the ratio of iodine and chlorine in the gas was 1:1 and iodine was found more reactive than chlorine (two magnitudes) to oxidize  $\text{Hg}^0$ , iodide ion ( $\text{I}^-$ ) was supposed to be the main anion in the products. However, it was unexpectedly observed that chloride ion ( $\text{Cl}^-$ ) was the dominant anion in the products, and the iodide ion was less than 10% among the total anions.

Based on the above results, the possible reaction paths for  $\text{Hg}^0$  removal can be speculated as follows. Both  $\text{Hg}^0$  and ICl can attract each other, resulting in the formation of  $\text{Hg-ICl}^*$  which maybe an



(a)



(b)

**Fig. 4.** (a) and (b) were the spectra evolution of ICl during the reaction between the mercury and ICl and the spectra after peak separation, respectively. The concentration of  $\text{Hg}^0$  and ICl was 300 ppmv and 1000 ppmv, respectively.  $\text{Cl}_2$ : 330 nm; ICl: 467 nm;  $\text{I}_2$ : 530 nm; temperature: 373 K.

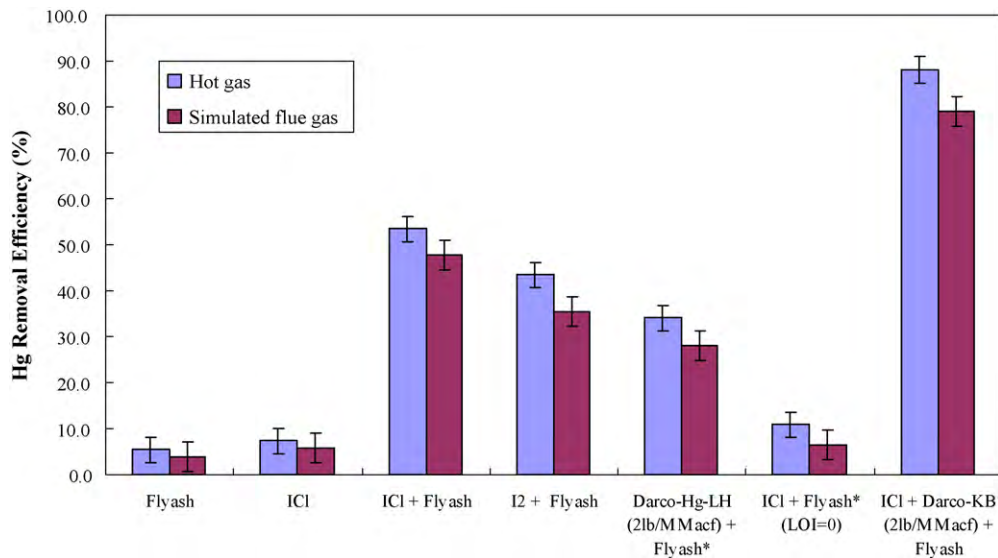
intermediate complex (Eq. (5)). Then,  $\text{Hg-ICl}^*$  will react with ICl to form stable product,  $\text{HgCl}_2$  or  $\text{HgI}_2$ . Eqs. (5)–(8) are some of the possible reactions during  $\text{Hg}^0$  conversion.



Because  $\text{Hg-ICl}^*$  is not a stable product, the London dispersion between Hg and ICl become stronger with the increase of temperature. That makes  $\text{Hg-ICl}^*$  become more unstable, and it will affect the progress of next reactions. Accordingly, the  $\text{Hg}^0$  oxidation rate decreased with the increase of temperature. Meanwhile, the intermediate complex could react with ICl continuously to form  $\text{HgCl}_2$  and  $\text{I}_2$ . That might be the reason why  $\text{I}_2$  increased during the reaction which could be observed from Fig. 4.

### 3.4. Pilot-scale tests of ICl

The performance of ICl was tested in pilot-scale reactor. As shown in Fig. 5, the  $\text{Hg}^0$  removal efficiency was only about 5% when 0.2 ppmv ICl or 20 g/m<sup>3</sup> flyash was injected into the flue gas,



**Fig. 5.**  $\text{Hg}^0$  removal efficiencies under various cases at 393 K. The concentration of ICl,  $\text{I}_2$  and flyash was 0.2 ppmv, 0.1 ppmv and  $20 \text{ g/m}^3$ . Darco-KB or Darco-Hg-LH was injected into the flue gas blended with  $20 \text{ g/m}^3$  flyash. Flyash\* was the flyash calcined at 1073 K for 3 h to get rid of LOI.

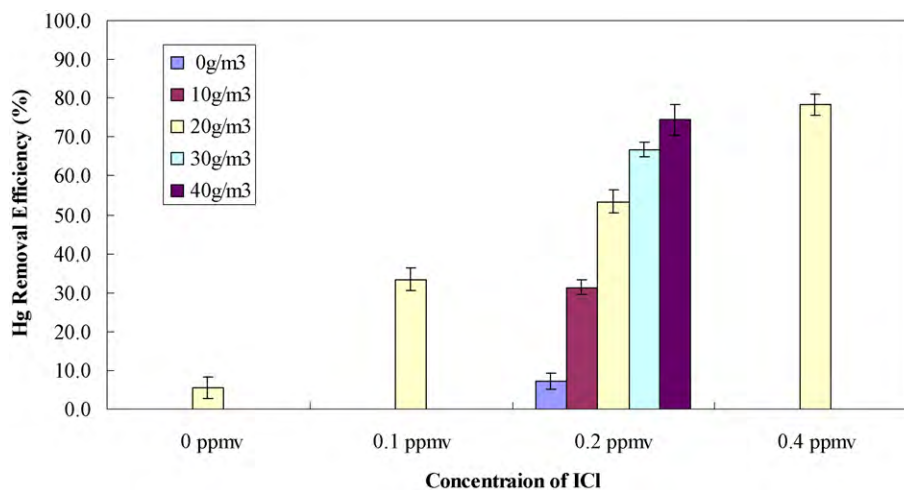
respectively. That means it was not effective enough for the mercury capture by the merely gas-phase reaction of  $\text{Hg}^0$  and ICl or just by the adsorption of flyash. However, 53%  $\text{Hg}^0$  could be removed in presence of 0.2 ppmv ICl and  $20 \text{ g/m}^3$  flyash in the hot gas. Meanwhile, the  $\text{Hg}^0$  removal efficiency dropped to 43% when 0.2 ppmv ICl was substituted by 0.1 ppmv  $\text{I}_2$ .

As we know that ACI (activated carbon injection) technique was widely tested in the mercury removal from coal-fired power plants and a series of activated carbons were studied. Among these activated carbons, Darco-Hg-LH was reported to have a good performance in mercury removal [22]. Therefore, Darco-Hg-LH was tested as a reference in this study. The removal efficiency of  $\text{Hg}^0$  was only about 34% when 2 lb/MMacf ( $32 \text{ mg/m}^3$ ) Darco-Hg-LH was injected into the slipstream reactor. Obviously, ICl cooperated with flyash showed more efficiency than commercial activated carbon, Darco-Hg-LH. From Fig. 5, it can be seen that flyash\* (without LOI) did not show good performance on  $\text{Hg}^0$  removal even in presence of ICl, which meant that LOI played an important role in  $\text{Hg}^0$  removal. Thus, this research tried to enhance the  $\text{Hg}^0$  removal by increasing the LOI of flyash, adding extra activated carbon into the

flyash. The result showed that the  $\text{Hg}^0$  removal efficiency increased remarkably to 88% when 2 lb/MMacf Darco-KB was injected into the reactor with 0.2 ppmv ICl and  $20 \text{ g/m}^3$  flyash. It can also be observed from Fig. 5,  $\text{Hg}^0$  removal efficiency in the simulated flue gas was slightly lower than that in the clean hot air.

The influences of the concentration of ICl and flyash were also studied. As shown in Fig. 6 the removal efficiency of  $\text{Hg}^0$  increased significantly with the increase of concentration of ICl or flyash. When the concentration of flyash increased from  $10 \text{ g/m}^3$  to  $40 \text{ g/m}^3$ , the mercury efficiency increased from 33% to 78%. Likewise, the mercury removal efficiency increased from 33% to 78% when ICl concentration increased from 0.1 ppmv to 0.4 ppmv. Therefore, increasing the flyash or ICl concentration in flue gas was helpful for the  $\text{Hg}^0$  removal.

According to the above results and the prediction model introduced previously [23], the  $\text{Hg}^0$  removal efficiency could be estimated. Taking the case of 0.2 ppmv ICl,  $20 \text{ g/m}^3$  of flyash and 500 ppmv  $\text{SO}_2$  in the flue gas as the example, it was predicted that the  $\text{Hg}^0$  removal efficiency was about 94% when the reaction time was 4 s.



**Fig. 6.** The relationship of  $\text{Hg}^0$  removal efficiency and ICl or flyash concentration at 393 K. The concentration of flyash was kept at  $20 \text{ g/m}^3$  when the concentration of ICl was changed from 0 ppmv to 0.4 ppmv. The concentration of ICl was maintained at 0.2 ppmv when the concentration of flyash was changed from  $0 \text{ g/m}^3$  to  $40 \text{ g/m}^3$ . The flue gas was hot gas without NO or  $\text{SO}_2$ .

#### 4. Conclusion

The removal of  $\text{Hg}^0$  by the homogenous gas-phase reaction was investigated under various conditions. Iodine monochloride was found to be a very efficient oxidant for  $\text{Hg}^0$  oxidation, having a 2nd-order reaction rate constant of about  $10.5(\pm 0.3) \times 10^{-17} \text{ cm}^3 \text{ molecules}^{-1} \text{ s}^{-1}$  and  $5.7(\pm 0.3) \times 10^{-17} \text{ cm}^3 \text{ molecules}^{-1} \text{ s}^{-1}$  at 273 K and 373 K, respectively. In addition, it could be estimated that the half life of  $\text{Hg}^0$  will be less than 50 hours with 1 ppbv of ICl at 273 K. The predicted  $\text{Hg}^0$  removal efficiency was about 94% with the pilot-scale tested result when 0.2 ppmv ICl was injected into the flue gas with  $20 \text{ g/m}^3$  flyash. It will save the use amount of iodine remarkably to get the same mercury removal efficiency when ICl was used as the substitute oxidant of iodine. The results suggested that the potential of iodine monochloride to oxidize elemental mercury in coal-fired flue gases was clearly promising for further study on a larger scale.

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