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Oxidation of Elemental Mercury by Active Species Generated From a Surface Dielectric Barrier Discharge Plasma Reactor

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Abstract A surface dielectric barrier discharge plasma reactor was employed to study Hg^0 oxidation in coal-fired flue gas. The experimental results showed that 98 % of Hg^0 oxidation efficiency and 13.7 µg kJ⁻¹ of energy yield were obtained under a specific energy density (SED) of 7.9 J L⁻¹. Increasing SED was beneficial for Hg^0 oxidation due to higher production of active species. Higher initial concentration resulted in lower Hg^0 oxidation efficiency, but higher amount of Hg^0 oxidation. Water vapor inhibited Hg^0 oxidation because the generation of O₃ was suppressed. The presence of NO remarkably restrained Hg^0 oxidation, while SO₂ showed little effect on Hg^0 oxidation. Roles of active species in Hg^0 oxidation were examined under different gas atmospheres (O₂ and air), indicating that O₃ played an important role in Hg^0 oxidation. Deposits on the internal surface of the reactor were analyzed by energy dispersive spectroscopy and the product was identified as HgO.

Keywords Surface discharge plasma reactor · Active species · Elemental mercury · Mercury oxidation

Introduction

Mercury has been listed as a hazardous and toxic pollutant under Title III of the 1990 Clean Air Act Amendments (CAAA) in the United States because of its volatility, persistence and bioaccumulation as methylmercury in the environment and its neurological health impacts

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[1]. Coal-fired power plants are the major anthropogenic mercury emission sources in China and the U.S. because of the huge coal consumption for power generation [2]. Mercury is presented in coal-fired flue gas as elemental mercury (Hg^0), oxidized mercury (Hg^{2+}) and particle-bound mercury (Hg^p) [3]. As reported, different species of mercury have different physical and chemical properties [4]. Hg^p can be captured by particulate matter (PM) control devices such as electrostatic precipitators (ESPs) and fabric filters (FF). Hg^{2+} is soluble in water and has the tendency to associate with PM. Hence, Hg^{2+} is relatively easy to be removed from flue gas using typical air pollution control devices (APCDs). Hg^0 , however, is hardly captured by APCDs due to high equilibrium vapor pressure and water insoluble [5]. Therefore, with consideration of the properties of Hg^{2+} , Hg^p and Hg^0 , studies for the Hg^0 oxidation methods should be first considered. Injection of powder activated carbon (PAC) impregnated with certain chemicals (i.e., sulfur species) has been successfully applied for the control of mercury adsorption technology in coal-fired flue gas purification, which includes high cost and high carbon-to-mercury ratio (3,000:1–100000:1).

As effective oxidation technologies, the preoxidation of Hg^0 to Hg^{2+} is necessary for the removal of Hg^0 by combining the present typical APCDs. Among preoxidation technologies under development, advanced oxidation processes (AOPs) are receiving great emphasis on pollution control because of their ability to rapidly oxidize hazardous air pollutants [7, 8]. Among the AOPs, non-thermal discharge plasmas (NTPs), which generate a large number of chemical active species such as $\cdot O$ and O_3 , have been developed as a new method applicable for processing waste gas [9, 10], the removal of volatile organic compounds (VOCs) [11, 12] and oxidizing Hg^0 [13–17]. NTP processes are highly effective in producing free radicals to enhance the removal of hazardous air pollutants.

Dielectric barrier discharge (DBD), one of NTPs, has received the most extensive investigation due to its high removal efficiency and environmental compatibility. DBD can be divided into four types based on the reactor geometry: typical DBD, surface discharge, coplanar discharge, and packed-bed discharge [12]. Surface DBD (SDBD) plasma reactors (SDBDs) have aroused considerable interest because of its unique features and characteristics, for example, SDBDs are easily manufactured than other DBD reactor, and SDBDs result in a decrease in the breakdown voltages, and thus better energy efficiency is obtained [18]. Moreover, the flue gas resistance in SDBDs is much less than that in other DBD reactors due to its wider electrode gap in SDBDs when flue gas passes through DBD reactors. Up to now, some researchers mainly focused on the removal of VOCs at low concentration levels using SDBDs [19-21]. For example, Oda et al. studied decomposition of gaseous organic contaminants by surface discharge induced plasma chemical processing (SPCP) and removal rates of 95 % for acetone and 100 % for 2-propanol were obtained using a ceramic tube reactor with strip-like electrodes [19]. Seung-Min Oh et al. used a surface discharge plasma reactor (SDBD) packed with various zeolites and the toluene adsorbed in micropores was decomposed by active oxygen species generated in the plasma zone on the zeolite surface [21]. However, only Masuda et al. [22] studied the oxidation of Hg⁰ in simulated flue gas using SPCP, and the study demonstrated that SPCP was a feasible technology to oxidize Hg⁰ in the flue gas, but the important parameters in Hg⁰ oxidation should be further evaluated in detail.

The objective of the study is to evaluate Hg^0 oxidation performance of SDBD, and the important parameters including energy input, Hg^0 initial concentration, gas atmospheres, and flue gas components (H₂O, NO, SO₂) were examined. In addition, the evolution of Hg^0 , Hg^{2+} and total Hg concentrations was investigated, and the Hg^0 oxidation mechanisms were also identified.

Experimental Section

Experimental Setup

The experimental setup designed (Fig. 1) for study of the Hg⁰ oxidation was composed of a continuous gas flow sample generation system, a SDBD and a gas sampling analysis and detection system. The SDBD was comprised of a quartz tube (o.d. of 15 mm, i.d. of 12 mm and length of 260 mm), the outside of which was closely wrapped with the silver paper to act as a grounded electrode (length 200 mm). The high voltage electrode was a stainless steel wire coiled (diameter of 1 mm) on the inside of the tube. An AC voltage (50 Hz) was applied to produce the discharge plasma, and the peak voltage was varied from 4 to 12 kV. The peak voltage and current were measured with an oscilloscope (Tektronix TDS2014) equipped with a voltage probe (Tektronix P6015A) and a current probe (Tektronix P6021). The typical voltage and current waveform obtained in the experiment was shown in Fig. 2. The voltage-charge (V-Q) Lissajous method (C = 1 μ F) was used to determine the discharge power (W).

Experimental Methods

All the experiments were run at a constant gas flow rate of 4.5 L min⁻¹ at room temperature. A Dynacal mercury permeation device (VICI Metronic, Inc. USA) was used to provide trace mercury vapor of desired Hg^0 concentration. The permeation device was located at the bottom of a glass U-tube that was immersed in a temperature-controlled water bath. Water vapor was generated by an evaporator and introduced into the flue gas with the carrier of air. The humidity of flue gas was measured with a humidity tester (Rotronic HP22-A + HC2-HK40). A RA-915 + mercury analyzer (Lumex, RU) coupled with RP91 was employed to measure Hg^0 concentration. The balance of mercury was conducted in accordance with the ASTM method of Ontario Hydro sampling (ASTM D6784-02) [23]. The ozone (O₃) concentrations in flue gas were measured by the iodometry method [24]. The deposited mercury species elements were determined by energy-dispersive X-ray spectroscopy (EDX, NORAN system).

The definition of Hg⁰ oxidation efficiency was as follows:

$$Hg^{0} \text{ oxidation effciency } (\%) = \frac{Hg^{0}_{in} - Hg^{0}_{out}}{Hg^{0}_{in}} \times 100$$
(1)

where Hg_{in}^0 and Hg_{out}^0 are the concentrations of Hg^0 (µg m⁻³) measured at the outlet of plasma reactor without or with high voltage discharge, respectively.

The specific energy density (SED) and energy yield were calculated:

Specific energy density
$$(J L^{-1}) = \frac{\text{Consumption power } (J s^{-1})}{\text{Gas flow rate } (L min^{-1})} \times 60(s min^{-1})$$
 (2)

Energy yield (µg kJ⁻¹) =
$$\frac{\text{Gas flow rate } (\text{L s}^{-1}) \times (\text{Hg}_{\text{in}}^0 - \text{Hg}_{\text{out}}^0)}{\text{Consumption power } (\text{J s}^{-1})}$$
. (3)



Fig. 1 Schematic diagram of the experimental system





Results and Discussion

Effect of Energy input on Hg⁰ Oxidation Efficiency

The effect of energy input on Hg^0 oxidation efficiency was illustrated in Fig. 3. Obviously, increasing SED enhanced Hg^0 oxidation efficiency due to a higher energy input introduced in the discharge. With an increase of SED from 0.8 to 7.9 J L⁻¹, Hg⁰ oxidation efficiency increased from 80 to 98 %. Increasing SED leads to the production of more plasma channels and chemically active species, therefore, Hg^0 oxidation efficiency is enhanced. In addition, Hg^0 oxidation efficiency of 80 % achieved at SED of 0.8 J L⁻¹, indicated that SDBD showed excellent performance of Hg^0 oxidation.

$$\mathbf{e} + \mathbf{O}_2 \to \mathbf{\cdot}\mathbf{O} + \mathbf{\cdot}\mathbf{O} + \mathbf{e} \tag{4}$$

$$\cdot \mathbf{O} + \mathbf{O}_2 + \mathbf{M} \to \mathbf{O}_3 + \mathbf{M} \tag{5}$$

$$\cdot \mathbf{O} + \mathbf{Hg}^0 \to \mathbf{HgO} \tag{6}$$

$$O_3 + Hg^0 \to HgO + O_2. \tag{7}$$

Fig. 3 Effect of energy input on Hg^0 oxidation efficiency (conditions: Hg^0 initial concentration 100 µg m⁻³; dry air)



Table 1 Statement of experimental results obtained with different plasma reactors

Reactor	Q (L min ⁻¹)	$Hg_{in}^0 ~(\mu g ~m^{-3})$	SED (J L ⁻¹)	Oxidation efficiency (%)	$\begin{array}{c} EY \\ (\mu g \ k J^{-1}) \end{array}$	Ref.
CCDR	2.5	300	23.7	59	7.5	[13]
Wire-plate type	2	50	18	80	2.2	[25]
Wire-cylinder reactor	6	110	894	98	0.1	[26]
			80	98	1.3	
O ₃ injection	2.5	300	23.7	93	12.3	[13]
SDBD	4.5	110	7.9	98	13.7	This study

In order to show performances of the SDBD compared to other types of plasma reactor, a brief comparison of the present results with other similar works was summarized in Table 1. Concentric cylinder discharge reactor (CCDR) and wire-plate type reactor exhibited lower Hg⁰ oxidation efficiency and energy yield compared with the present study. For example, 59 % of Hg⁰ oxidation efficiency and 7.5 μ g kJ⁻¹ of energy yield was obtained for the CCDR [13], and 80 % of Hg⁰ oxidation efficiency and 2.2 μ g kJ⁻¹ of energy yield was observed for the wire-plate type reactor [25]. Hg⁰ oxidation efficiencies obtained by the wire-cylinder reactor [26] were comparable with that of the present research, but the energy yields were much lower than that of the present study. For ozone injection technology [13], Hg⁰ oxidation efficiency and energy yield were comparable with that of the present research. For example, 93 % of Hg⁰ oxidation efficiency and 12.3 μ g kJ⁻¹ of energy yield was obtained for the ozone injection technology [13]. Hence, it is believed that it is an alternative and efficient method for SDBD to oxidize Hg⁰ in the coal-fired flue gas. However, energy yield is strongly dependent on multiple factor, including air flow rate (Q), initial concentration and SED, for example, higher initial concentration and lower SED results in higher energy yield. Therefore, Table 1 gives only a qualitative and approximate comparison.

Effect of Hg⁰ Initial Concentration on Hg⁰ Oxidation Efficiency

Generally, Hg^0 concentration of actual flue gas strongly varies. The effect of Hg^0 initial concentration on its oxidation was studied in the range of 50–170 µg m⁻³, and Hg^0



oxidation efficiency and energy yield were presented in Fig. 4. The results showed that Hg^0 oxidation efficiency and energy yield were directly related to the initial concentration. Hg^0 oxidation efficiency had a slight drop with the increase of Hg^0 initial concentration from 50 to 170 µg m⁻³ at about 7.9 J L⁻¹. However, energy yield remarkably enhanced from 6.3 to 21.1 µg kJ⁻¹ under the same condition of SED. This phenomenon can be explained by the fact that the DBD energy dissipation levels remained comparable, the average energy availability per molecule decreased with the increase of the initial concentration. Hence, Hg^0 oxidation efficiency decreased with an increase of the initial concentration. However, the absolute removal of Hg^0 molecules increases because of higher initial concentration and higher probability of interaction with short-lived active species.

Effect of Gas Atmospheres on Hg⁰ Oxidation Efficiency

In order to investigate the roles of active species in Hg^0 oxidation, experiments were conducted separately under oxygen and air atmospheres. The experimental results were shown in Fig. 5. As observed, Hg^0 oxidation efficiencies under oxygen atmosphere were higher than that under air atmosphere. Under the condition of oxygen atmosphere, the density of oxygen-based active species produced in the discharge was larger than those under air atmosphere, resulting in higher oxidation efficiency in the case of oxygen atmosphere.

In order to study the contribution of O_3 to Hg^0 oxidation, the experiment of Hg^0 oxidation by O_3 treatment was conducted and the result was shown in Fig. 5. Herein, the O_3 concentration was equal to that obtained by the discharge treatment under air atmosphere. O_3 was generated by another of the same SDBD and injected into the reactor to react with Hg^0 . Under the condition of 0.8 J L⁻¹, 75 % of Hg^0 was oxidized, which was slightly lower than that under air atmosphere. These results indicated that (1) in the case of air atmosphere, there were other active species involved with the Hg^0 oxidation in addition to O_3 . (2) O_3 was the main active species for Hg^0 oxidation.

Effect of H₂O on Hg⁰ Oxidation Efficiency

The effect of H_2O on Hg^0 oxidation efficiency was presented in Fig. 6. At SED of 7.9 J L⁻¹, compared with the dry gas, the addition of 3 and 5 % water vapor dropped Hg^0



oxidation efficiency from 98 to 96 and 91 %, respectively. Therefore, the presence of H₂O exhibited a slightly suppressed effect on Hg⁰ oxidation, and the similar result was reported by the literature [26]. H₂O may react with O₃ to form ·OH, resulting in the decreasing of Hg⁰ oxidation efficiency due to the fact that the O₃/·O radicals are more effective than ·OH to oxidize Hg⁰. Simultaneously, ·OH can act as a promoter in the reactions (10) and (11) decreasing O₃ and ·O [27–29]. As shown in Table 2, O₃ concentration dropped with an increase of H₂O contents, and higher O₃ concentration was obtained for dry gas as compared to that for 3 and 5 % H₂O. Only 10.8 µg L⁻¹ of O₃ was observed for 5 % H₂O, whereas O₃ concentration of 18 µg L⁻¹ was obtained for dry gas at the same SED 7.9 J L⁻¹. The reactions can be described as follows:

$$O_3 + H_2O \rightarrow H_2O_2 + O_2 \tag{8}$$

$$H_2O_2 \rightarrow \cdot OH + \cdot OH$$
 (9)

$$O_3 + 2 \cdot OH \rightarrow 2O_2 + H_2O \tag{10}$$

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SED (J L ⁻¹)	O_3 concentration (µg L ⁻¹)					
	Without H ₂ O	3 % H ₂ O	5 % H ₂ O			
0.8	3.6	2.4	1.2			
2.0	6.7	5.8	3.3			
3.9	10.0	9.0	5.8			
5.9	14.2	11.8	7.4			
7.9	18.0	14.2	10.8			

Table 2 O3 concentrations under different SED and H2O contents





$$\cdot \mathbf{OH} + \cdot \mathbf{O} \to \mathbf{O}_2 + \cdot \mathbf{H} \tag{11}$$

$$\cdot \mathbf{H} + \mathbf{O}_3 \to \mathbf{O}_2 + \cdot \mathbf{OH}. \tag{12}$$

Effect of NO on Hg⁰ Oxidation Efficiency

NO is ubiquitous in flue gas environment and has significant impacts on Hg^0 oxidation efficiency. As shown in Fig. 7, the presence of NO showed obvious inhibition on Hg^0 oxidation. In the absence of NO, Hg^0 oxidation efficiency was 98 % at around 7.9 J L⁻¹, when NO concentration increased to 300 ppm, Hg^0 oxidation efficiency had a dramatically decrease to 69 %, and with NO concentration increased to 600 ppm, Hg^0 oxidation efficiency further dropped to 58 % at the same SED. The competitive reactions of Hg^0 and NO gases with the chemical active radicals generated in the reactor. The reaction rate coefficient of NO with O₃ (1.8×10^{-14} cm³ molecules⁻¹ s⁻¹) (Reaction 13) [29] is much faster than that of (10.5 ± 0.65) × 10^{-19} cm³ molecules⁻¹ s⁻¹ [30] for Hg⁰ reacting with O₃, implying that the O₃ can rapidly react with NO. Therefore, O₃ will be preferentially exhausted, Hg⁰ oxidation efficiency markedly reduced, being in agreement with the results obtained in previous studies [31–33].

$$NO + O_3 \rightarrow NO_2 + O_2 \tag{13}$$



Effect of SO₂ on Hg⁰ Oxidation Efficiency

The concentration of SO₂ was varied to study effect of SO₂ on the Hg⁰ oxidation (Fig. 8). As observed, no obvious decrease of Hg⁰ oxidation efficiency was detected when 500 ppm SO₂ was introduced to the gas flow or even when SO₂ concentration further increased to 1100 ppm. The reason can be explained from the difference of the possible reaction mechanisms between SO₂ and active species (·OH, ·O and O₃). As indicated in the previous research [28, 33], most SO₂ is oxidized to HSO₃ and SO₃ by ·OH and ·O radicals which are the main radical species for oxidation due to the large rate constant compared with the other radical species such as O₃. Therefore, the consumption of O₃ by the reaction with SO₂ is very minor. Additional, Hg⁰ and O₃ ((10.5 ± 0.65) × 10⁻¹⁹ cm³ molecules⁻¹ s⁻¹) [30] reveal a relatively higher reaction rate constant than that of SO₂ and O₃ (2.7 × 10⁻²³ cm³ molecules⁻¹ s⁻¹) [34]. Therefore, it has been clearly demonstrated that SO₂ shows little impact on Hg⁰ oxidation.

$$SO_2 + O_3 \to SO_3 + O_2 \tag{14}$$

The Mercury Mass Balance and Analysis of the Mercury Species Deposited on the Reactor Surface

The evolution of Hg^0 , Hg^{2+} , and total Hg concentrations was shown in Fig. 9. With an increase of the SED, Hg^0 concentration exhibited a dramatic decrease from 100 to 1.6 µg m⁻³, while the concentration of Hg^{2+} increased from 0 to 96.1 µg m⁻³, and then the total Hg concentration slightly decreased from 100 to 97.7 µg m⁻³ due to the reason that some mercury species in form of yellow deposits were accumulated on the internal surface of the reactor, presumably from the deposition of HgO formed through the oxidation of Hg⁰.

Energy dispersive spectroscopy (EDS) was used to determine the deposited mercury species elements and the measurement result was shown in Fig. 10. No Hg peak was detected at the blank sample and an Hg peak was detected for the inner surface of the reactor. Surface elemental composition of deposited mercury species in wt% and at.% on the inner surface of the reactor were illustrated in Table 3. For the deposited mercury species, after excluding the composition of SiO₂, the at.% of O and Hg on the surface was



Fig. 10 EDS analysis of the reactor (quartz) surface a blank sample; b deposited mercury species

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Samples	Blank samp	Blank sample		Deposited mercury species		
	Si	0	Si	0	Hg	
wt (%)	46.85	53.15	4.23	14.48	81.29	
at (%)	32.78	67.22	9.55	55.81	34.64	

Table 3 Surface elemental composition of deposited mercury species in wt % and at. % on the reactor surface derived from EDS analyses

observed to be approximately 36.71 and 34.64 %, implying that Hg/O atomic ratio (1.05) of the sample was obtained. The results indicated that the deposited mercury species predominantly existed in the form of HgO.

Conclusions

The application of SDBD for the oxidation of Hg^0 in coal-fired flue gas was investigated in this study. High oxidation efficiency and energy yield of SDBD was obtained, which indicated that SDBD could be an alternative to other conventional technologies for efficient oxidation of Hg^0 . The investigation of contributions of active species to Hg^0 oxidation indicated that O₃ played a more important role during the treatment process. The deposited mercury species was monitored by EDS. Identified products were HgO.

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