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# Hg<sup>2+</sup> reduction and re-emission from simulated wet flue gas desulfurization liquors

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# ABSTRACT

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*Keywords:* Desulfurization solutions Hg<sup>2+</sup> reduction Re-emission Removal efficiency In this study, considering that Hg<sup>2+</sup> in wet flue gas desulfurization (FGD) systems can easily be reduced and then released into atmosphere, causing secondary pollution, the researches about Hg<sup>2+</sup> reduction and Hg<sup>0</sup> re-emission mechanism were carried out. The effects of several experimental parameters on the reduction were studied, including initial pH, temperature, and concentrations of Cl<sup>-</sup> and S(IV). Our experimental results indicated that Cl<sup>-</sup> had a restraining effect on the Hg<sup>2+</sup> reduction and Hg<sup>0</sup> re-emission, after 24 h reaction, only 20.5% of Hg<sup>2+</sup> was reduced with 100 mM Cl<sup>-</sup> in simulated desulfurization solution. Cl<sup>-</sup> can slow Hg<sup>2+</sup> reduction and Hg<sup>0</sup> re-emissions dramatically through changing reaction mechanism, with formation of new intermediate: ClHgSO<sub>3</sub><sup>-</sup>, which can decompose to Hg<sup>0</sup>, but much more slowly than Hg(SO<sub>3</sub>)<sub>2</sub><sup>2-</sup> or HgSO<sub>3</sub>. Simulating the conditions of the practical application (initial pH 5,  $T = 50 \,^{\circ}$ C, S(IV) = 5 mM, Cl<sup>-</sup> = 100 mM), we also found that Ca<sup>2+</sup>, NO<sub>3</sub><sup>-</sup>, F<sup>-</sup>, etc. all had obvious effects on reduction rates. Based on the material balance and characteristic of the reactants, the reduction emission mechanism of Hg<sup>2+</sup> has been established, providing theoretical basis for industrial application of mercury control in wet FGD systems.

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# 1. Introduction

For the past several decades, mercury (Hg) has received increasing attention as a pollutant of serious concern [1]. The amount of mercury in earth's biosphere is increasing gradually due to both natural and anthropogenic emissions [2]. The major manmade sources of mercury emission into the atmosphere are the flue gases from coal combustors and municipal waste incinerators [3]. The large quantity and scale of boilers make coal burning the largest anthropogenic source of mercury emissions, emitting approximately 50t of mercury into the atmosphere annually, in other words, about one-third of the total anthropogenic emission [1,2]. In China, coal now accounts for about 67% of the primary energy consumption and will continue to play an important role in energy uses for a long time to come [4]. Mercury contamination is a serious problem in China, particularly concentrated in southwestern China (particularly Guizhou Province) [5]. Pacyna and Pacyna estimated that China's mercury emissions from coal combustion contributed more than 25% to total global mercury emissions [6]. Wu et al. reported that Hg emissions from coal combustion in China increased from 202.4t in 1995 to 256.7t in 2003 at an average annual rate of 3.0%. Among all of the coal consumption sectors, the power sector is the leading one in Hg emission growth, up by 5.9% annually and reaching 100.1 t in 2003 [7].

The emission of mercury into the atmosphere and its transport away from emission sources impose serious health concerns. Mercury buried deep in the ground or beneath lake and ocean floors does not appear to present a threat to either the environment or human health [2]. However, mercury released to the environment can be converted through biological process into an organic form, methyl mercury (MeHg), a neurotoxin that can bio-accumulate in the food chain [3]. After many years of coal burning and other industrial processes, the level of organic mercury in many water bodies rises to an extent that consumption of fish is unsafe [2]. Reports estimated that methyl mercury level in edible fish would double near a 1000 MW power plant [4]. Based on available evidences of the mercury effects on health effects, the US Environmental Protection Agency (EPA) issued the Clean Air Mercury Rule (CAMR) to regulate mercury release into the environment [4,8].

There are three forms of mercury which may be present in the coal combustion flue gas: elemental mercury (Hg<sup>0</sup>), oxidized mercury (Hg<sup>2+</sup>), and particle-bound mercury (Hg<sup>P</sup>) [9]. Generally, oxidized mercury is water-soluble and can be efficiently removed in the wet flue gas desulfurization (FGD) processes of coal combustion, which exhibits up to 90% removal of Hg<sup>2+</sup>. Particle-bound mercury comprises only ca. 10% of the total, most can be captured by the typical air pollution control devices (APCD) such as electrostatic precipitators and baghouses. However, the elemental mercury is difficult to capture with typical APCD due to its volatility

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and chemical stability [3,4,9]. Mercury in atmosphere is mostly in the form of elemental mercury vapor, it can circulate in the atmosphere for 1-2y, allowing its wide dispersion and long-distance transportation [10]. Of total coal combustion emission, the main mercury forms in China are Hg<sup>2+</sup> and Hg<sup>P</sup> that about 61.2% of the Hg is released as  $Hg^{2+}$ , 22.5% as  $Hg^{P}$ , and 16.3% as  $Hg^{0}$  in 2000 [5,11].

Nowadays wet FGD systems have already obtained wide application in developed countries because of their high removal efficiency, reasonable cost, and wide applicability. If a high removal efficiency of mercury is also achieved, wet FGD systems can be used for cost-effective multi-pollutant control (operation cost is only 30-50% of activated carbon injection (ACI)), hence research of controlling the mercury emission also has received special attention. However, field data have shown that, in general, less than 70% mercury removal efficiency is achieved by wet FGD. There presences two problems as follows: firstly, Hg<sup>0</sup> can hardly be dissolved in desulfurization solutions, leading to poor removal efficiency of Hg<sup>0</sup>; secondly, it is speculated that some of the absorbed mercury (e.g., ionic form of mercury (Hg<sup>2+</sup>) such as mercuric chloride  $(HgCl_2)$ ) can be converted back to elemental mercury  $(Hg^0)$  and re-emitted [12]. Therefore, it is necessary to control secondary pollution of mercury in desulfurization solutions or desulfurization byproducts.

In this study, a sequence of experiments was carried out in order to evaluate the influence of different operational parameters on Hg<sup>2+</sup> reduction efficiency in a 1-L three-necked flask. In the end, the mechanism of the reactions between Hg<sup>2+</sup>, SO<sub>3</sub><sup>2-</sup> and Cl<sup>-</sup> in the process was also hypothesized.

#### 2. Experiments and methods

#### 2.1. Chemicals

All of the chemicals, such as anhydrous sodium sulfite ( $\geq$ 97.0%, AR), sodium chloride ( $\geq$ 99.8%, GR), nitric acid (65–68%, GR), hydrochloric acid (36–38%, GR), sodium hydroxide (≥96.0%, AR), stannous chloride (≥98.0%, AR), stannum (≥99.9%, AR), potassium dichromate (99.95-100.05%, GR), mercury chloride (>99.0%, AR), etc. were all used as received without further purification. The gases, such as Ar ( $\geq$ 99.99%) and N<sub>2</sub> ( $\geq$ 99.9%), stored in the steel cylinders were provided by Jingong Gas Co. Ltd.

#### 2.2. Experimental procedure

The batch experiments for Hg<sup>2+</sup> reduction in simulated desulfurization solutions proceeded in a 1-L three-necked flask inside a water bath maintained at the desired temperature, and HgCl<sub>2</sub> aqueous solution were added into the flask as the source of Hg<sup>2+</sup>. Other chemicals, such as Na<sub>2</sub>SO<sub>3</sub>, NaCl, CaCl<sub>2</sub>, MgCl<sub>2</sub>, NaNO<sub>3</sub> and NaF were selectively added to simulate the source of  $SO_3^{2-}$ , Cl<sup>-</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, NO<sub>3</sub><sup>-</sup> and F<sup>-</sup>. The reaction solution was stirred under nitrogen atmosphere to remove production Hg<sup>0</sup>. Samples of 5 mL were periodically collected at selected time intervals, then it was diluted to 50 mL in the volumetric flask with 5 mL stationary liquid already in, ready for analysis.

#### 2.3. Analytical method

Concentrations of Hg<sup>2+</sup> in aqueous solution were analyzed by QM201C fluorescence arsenic/mercury analyzer (Qing'an Instrument Co., Suzhou, China). Samples and 7% (w/w) SnCl<sub>2</sub> solution were transferred by a peristaltic pump into mercury analyzer at a constant rate, superfluous  $SnCl_2$  was used to reduce  $Hg^{2+}$  to  $Hg^0$ . Mercury vapor produced was supplied with Ar as carrier gas and



Fig. 1. Effect of initial pH on the Hg<sup>2+</sup> reduction efficiency ( $T = 50 \circ C$ , S(IV) = 5.0 mM).

flushed out and was measured by cold vapor generation atomic absorption spectrometry.

The ultraviolet (UV) spectra characterization was analyzed by TU-1900 UV-VIS spectrophotometer (Beijing Purkinje General Instrument Co. Ltd.), and pH value was measured with a digital pH meter (Mettler-Toledo Instruments (Shanghai) Co. Ltd.).

#### 3. Results and discussion

#### 3.1. Effect of initial pH value

The initial pH value of solution is important in reduction of Hg<sup>2+</sup>. Prior to starting reaction all reactant solutions were adjusted to different pH values by diluted H<sub>2</sub>SO<sub>4</sub> and NaOH, and during the reaction pH values were not adjusted. As Fig. 1 shows, the Hg<sup>2+</sup> concentration continued to decrease as the experiment proceeded, but the rate of decrease gradually slowed, especially after an hour. With the decrease of initial pH, mercury removal efficiency increased gradually. At higher initial pH value (pH 6), concentration of Hg<sup>2+</sup> in simulated desulfurization solutions decreased more slowly, keeping relatively stable. When the initial pH values increased from 3.0, 4.0, 5.0 to 6.0, after 2 h, the Hg<sup>2+</sup> reduction efficiencies were 84.4%, 63.1%, 51.1% and 23.3%, respectively. It is speculated that the presence of OH<sup>-</sup> at higher pH, greatly reduced the possibility of reduction by transforming  $Hg^{2+}$  to  $Hg(OH)^+$  or  $Hg(OH)_2$ . On the other hand, decreasing the liquor pH can decrease the sulfite-tobisulfite ratio, leading to the reduction of Hg<sup>2+</sup>. However, caution should be exercised for lower pH may decrease the SO<sub>2</sub> removal efficiency and encourage corrosion in wet flue gas desulfurization systems [13].

#### 3.2. Effect of reaction temperature

The performances under different reaction temperatures (40–60 °C) were analyzed to study the temperature effect on Hg<sup>2+</sup> reduction. Results were shown in Fig. 2 that when initial pH value was 5.0 and S(IV) concentration reached 5.0 mM, Hg<sup>2+</sup> reduction efficiency greatly improved with the increase of reaction temperature. When the temperature was raised from 40 to 60 °C, reduction efficiency increased from 37.1% to 76.3% after 2 h reaction, which could be explained by the fact that reduction of the mercuric ion appears to proceed uniquely via the HgSO<sub>3</sub> complex whose decomposition rate is strongly temperature dependent [13]. Loon et al. has found that the rate constant  $k_0$  roughly quadruples with each 10 °C increase in temperature [14].





Fig. 2. Effect of temperature on the  $Hg^{2+}$  reduction efficiency (initial pH 5,  $S(IV)\!=\!5.0\,\text{mM}).$ 

#### 3.3. Effect of S(IV) concentration

The mercury removal curves with different initial  $SO_3^{2-}$  concentrations are shown in Fig. 3. Evidently, increase in S(IV) concentration led to a slower reduction of mercury, when S(IV) concentration was at low level,  $Hg^{2+}$  concentration sharply decreased, for  $Hg^{2+}$  was reduced to  $Hg^0$  as the reaction continued. The removal percentage of  $Hg^{2+}$  reached 43.7% in 20 min with S(IV) concentration of 1 mM, and 91.8%  $Hg^{2+}$  was removed in 2 h. In contrast, only less than 35% of removal efficiency was obtained for 8 mM S(IV) after 2 h.

Equimolar amounts of  $Hg^{2+}$  and  $HSO_3^{-}$  in the solutions decompose to  $Hg^0$  and  $SO_4^{2-}$  within minutes at room temperature (E1) [14]. As is found, the reactions for  $Hg^0$  emission are as follows, the main pathway is through mercuric-sulfite complexes (E2, 3):

$$Hg^{2+} + HSO_3^{-} + H_2O \rightarrow Hg^0 \uparrow + SO_4^{2-} + 3H^+,$$
 (1)

 $Hg^{2+} + SO_3^{2-} \leftrightarrow HgSO_3, \tag{2}$ 

$$HgSO_3 + SO_3^{2-} \leftrightarrow Hg(SO_3)_2^{2-}.$$
 (3)

Only HgSO<sub>3</sub> decomposes to give reduction of Hg<sup>2+</sup> with a half-life of approximately 1 min at 25 °C:

$$HgSO_3 + H_2O \rightarrow Hg^0 \uparrow + SO_4^{2-} + 2H^+$$
(4)



**Fig. 3.** Effect of S(IV) concentration on the Hg<sup>2+</sup> reduction efficiency (initial pH 5,  $T = 50 \degree$ C).



**Fig. 4.** Effect of Cl<sup>-</sup> concentration on the Hg<sup>2+</sup> reduction efficiency (initial pH 5,  $T = 50 \degree C$ , S(IV) = 5.0 mM).

However, HgSO<sub>3</sub> is not generally assumed to be the dominant mercuric species with excess sulfite, the major species is thought to be  $Hg(SO_3)_2^{2-}$  which is compared to be more stable than HgSO<sub>3</sub>, and this can explain why rates generally decrease at higher S(IV) concentration.

#### 3.4. Effect of Cl<sup>-</sup> concentration

Fig. 4(a) and (b) presents the reduction of  $Hg^{2+}$  in simulated desulfurization solutions with chloride in it. Concentration of Cl<sup>-</sup> had an evident effect on the reduction of  $Hg^{2+}$  that the simulated desulfurization solution without chloride showed fastest reduction rate, and higher Cl<sup>-</sup> typically lowered re-emission. From Fig. 4(a), it can be seen that about 55.1%  $Hg^{2+}$  was reduced in solution without Cl<sup>-</sup> while only 39.4% with 200 mM Cl<sup>-</sup>. Considering  $Hg^{2+}$  reduction rates were slow at practical conditions, runs were carried out for 24 h open without stirring. Oxygen in the atmosphere greatly depressed the reduction, the trend after 24 h successive reaction is presented in Fig. 4(b). With the increase of Cl<sup>-</sup> concentration, the removal rate decreased correspondingly, and only 20.5% of  $Hg^{2+}$  was reduced with 100 mM Cl<sup>-</sup> in simulated desulfurization solution even after 24 h while almost 100% reduction efficiency was obtained without Cl<sup>-</sup> in it.

Chloride slows re-emissions dramatically through changing reaction mechanism, with formation of new intermediate:  $ClHgSO_3^-$ , which can decompose to  $Hg^0$ , but much more slowly than  $Hg(SO_3)_2^{2-}$  or  $HgSO_3$ . In addition,  $Cl_2HgSO_3^{2-}$  is formed reversibly at higher chloride concentration, which does not decompose to  $Hg^0$ . The reactions are below:

$$HgSO_3 + Cl^- \rightarrow ClHgSO_3^-, \tag{5}$$



**Fig. 5.** Effect of  $Ca^{2+}$  concentration on the Hg<sup>2+</sup> reduction efficiency ( $T = 50 \degree C$ , initial pH 5, S(IV) = 5.0 mM, Cl<sup>-</sup> = 100.0 mM).

$$ClHgSO_3^- + Cl^- \leftrightarrow Cl_2HgSO_3^{2-}.$$
 (6)

# 3.5. Effect of $Ca^{2+}$ and $Mg^{2+}$ concentration

Based on the references, we estimated the effect of other ions upon the baseline conditions of the practical application (initial pH 5, T=50 °C, S(IV)=5 mM,  $Cl^{-}=100$  mM). As is shown in Figs. 5 and 6, the addition of Ca<sup>2+</sup> and Mg<sup>2+</sup> accelerated the reaction and increased the mercury removal efficiency dramatically. When the initial  $SO_3^{2-}$  and  $Cl^-$  concentrations were 5.0 and 100.0 mM, the removal efficiency of mercury reached 26.5%, 45.8% and 55.1% with an initial Ca<sup>2+</sup> concentration of 5, 10 and 20 mM, respectively. Similarly, the mercury removal efficiency reached 12.7%, 21.0% and 46.0% with an initial Mg<sup>2+</sup> concentration of 10, 30 and 40 mM. However, Ca<sup>2+</sup> provided better accelerated effect than Mg<sup>2+</sup>. The results may be explained by considering that Ca<sup>2+</sup> and Mg<sup>2+</sup> would form ion pairs with sulfite, which can lower S(IV) concentration and affect mercury reduction rates. It also indicates that the concentration of the aqueous species S(IV) is of major importance. On the other hand, the effect of Ca<sup>2+</sup> can be interfered by the low solubility product of CaSO<sub>3</sub>.

### 3.6. Effect of $NO_3^-$ concentration

Our experimental results indicate that  $NO_3^-$  had an adverse effect on the Hg<sup>0</sup> re-emission, as increasing the  $NO_3^-$  concentration



**Fig. 6.** Effect of Mg<sup>2+</sup> concentration on the Hg<sup>2+</sup> reduction efficiency (T = 50 °C, initial pH 5, S(IV) = 5.0 mM, Cl<sup>-</sup> = 100.0 mM).



**Fig. 7.** Effect of  $NO_3^-$  concentration on the  $Hg^{2+}$  reduction efficiency ( $T = 50 \degree C$ , initial pH 5, S(IV) = 5.0 mM, Cl<sup>-</sup> = 100.0 mM).

from 1 to  $20 \text{ mg L}^{-1}$  led to the decrease of the removal efficiency from 41.9% to 6.20% in 2 h (Fig. 7). The negative effect was serious especially at lower NO<sub>3</sub><sup>-</sup> concentration. Based on the analysis above, we have guessed that the additive of NO<sub>3</sub><sup>-</sup> can inhibit the formation of HgSO<sub>3</sub> complexes in Hg<sup>2+</sup> emission.

## 3.7. Effect of $F^-$ concentration

Fig. 8 illustrates the concentration depletion curves of  $Hg^{2+}$  in the presence of  $F^-$ . It is shown that the higher the  $F^-$  concentration was, the lower the mercury removal rate was. The removal percentage reached 57.2% in 2 h in solution with 0.5 mM  $F^-$ , but only less than 10.1% of removal efficiency was obtained with 4 mM  $F^-$  in it. Reasons have been presumed that stable chelate compound may be formed with  $Hg^{2+}$  and  $F^-$  in solution.

#### 3.8. Reaction mechanism and model

UV spectrum was further investigated to make sure the change of the intermediate product in the  $Hg^{2+}$  reduction. In this study, the UV spectrum and rate curve was recorded by analyzing a solution containing 1 mg L<sup>-1</sup> Hg<sup>2+</sup> and 1.0 mM Na<sub>2</sub>SO<sub>3</sub> in 1 cm square quartz cuvettes at pH 3 and room temperature, as is shown in Fig. 9. A peak was observed with  $\lambda_{max}$  230 nm, the absorbance decreased from 0.073 to 0.023 in 19 min, and the rate curve at 230 nm varied curvi-



Fig. 8. Effect of  $F^-$  concentration on the  $Hg^{2+}$  reduction efficiency ( $T=50\,^{\circ}C$ , initial pH 5, S(IV)= 5.0 mM, Cl<sup>-</sup> = 100.0 mM).



Fig. 9. UV spectra and rate curve of  $1 \text{ mg L}^{-1} \text{ Hg}^{2+}$  at  $1.0 \text{ mM Na}_2\text{SO}_3$  solution.



**Fig. 10.** Diagram of main reaction pathways of Hg<sup>2+</sup> reduction and Hg<sup>0</sup> re-emission in desulfurization solutions.

linear with reaction time. The complex, two sulfite anions bound to the aqueous mercuric cation, forming in the reaction of the aqueous mercuric ion with excess sulfite, is believed to be  $Hg(SO_3)_2^{2-}$  [13]. We can conclude that the intermediate product  $Hg(SO_3)_2^{2-}$  was not stable indefinitely, they decreased as the reaction proceeded, for reduction of  $Hg^{2+}$  in the presence of excess hydrogen sulfite involves in dissociation of sulfite from  $Hg(SO_3)_2^{2-}$ , followed by decomposition of HgSO<sub>3</sub>. Rate constant can be obtained from non-linear least-squares fits to the first-order integrated rate equation [13].

Based on the above experiments, we know that the reaction mechanisms of Hg<sup>2+</sup> absorbed in the wet FGD are complex, the main pathways are through mercuric-sulfite and chloro-mercuric-sulfite species, and it could be described as the reactions shown in Fig. 10. Formations of HgSO<sub>3</sub>, Hg(SO<sub>3</sub>)<sub>2</sub><sup>2-</sup>, ClHgSO<sub>3</sub><sup>-</sup> and Cl<sub>2</sub>HgSO<sub>3</sub><sup>2-</sup>, with their different characters of generating Hg<sup>0</sup> (the sequence of the tendency in Hg<sup>0</sup> re-emission was HgSO<sub>3</sub> > Hg(SO<sub>3</sub>)<sub>2</sub><sup>2-</sup> > ClHgSO<sub>3</sub><sup>-</sup>, Cl<sub>2</sub>HgSO<sub>3</sub><sup>2-</sup> does not decompose to Hg<sup>0</sup>), can explain how excess sulfite and chloride lead to a slower reduction of mercury. The observation agrees very well with a report previously published by others that Chang and Ghorishi suggested that only a portion of Hg<sup>2+</sup> species absorbed in the scrubbing liquor functioned as the precursor of Hg<sup>0</sup> re-emissions, while the remaining portion was relatively stable and would not serve as the precursor of Hg<sup>0</sup> re-emissions, like represented by Eq. (7) [13,15]. In addition, byproducts of Hg(OH)<sup>+</sup> or Hg(OH)<sub>2</sub> may form in the presence of

OH<sup>-</sup>, which greatly weaken the Hg<sup>2+</sup> reduction. Preliminary interpretation of modeling results is that re-emissions rate is controlled by mercuric chloro-sulfite complexes at low pH but gradually by mercuric-sulfite complexes alone at higher pH.

$$HgCl_2 + S(IV) → Hg.S(IV) complexes → Hg0 ↑ → other stable complexes and byproducts (7)$$

#### 4. Conclusion

An evaluation of the influence of different experimental parameters on mercury removal efficiency was carried out. The experimental results indicate that flue gas  $Hg^0$  re-emission across a wet FGD scrubber can be reduced by increasing the initial pH value, concentration of S(IV), or lowering the temperature. Under simulated wet FGD conditions,  $Hg^{2+}$  reduction efficiency increased with the concentration of  $Ca^{2+}$  and  $Mg^{2+}$  increasing, and decreased with the increasing concentration of  $NO_3^-$ ,  $F^-$  and  $CI^-$ . The S(IV) induced  $Hg^{2+}$  reduction and  $Hg^0$  re-emission mechanism is complex, the main pathways are through Hg.S(IV) complexes. Chloride slows re-emissions dramatically through changing reaction mechanism, with formation of new intermediate:  $ClHgSO_3^-$ , which can decompose to  $Hg^0$ , but much more slowly than  $Hg(SO_3)_2^{2-}$  or  $HgSO_3$ . The present findings could be valuable for industrial application of characterizing and optimizing mercury control in wet FGD systems.

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