Adsorption of mercury in coal-fired power plants gypsum slurry on TiO$_2$/chitosan composite material

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Abstract. In this study, a simple method was used to prepare a chitosan adsorbent to mix with KI and TiO$_2$. Gravimetric analysis (TG), scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) were used to characterize the samples before and after adsorption of Hg$^{2+}$. A mercury adsorption experiment was also conducted in the gypsum slurry. The results show that using hydrobromic acid as a solvent of adsorbent resulted in a better adsorption effect than using acetic acid alone. Also, the sample (CS-KI/TiO$_2$-HBr) had a maximum mercury adsorption capacity when the pH=5 and the t=50°C. The characterization experiments showed that the thermal stability of composite materials declined and the TiO$_2$ uniformly dispersed in the surface of the samples with a lamellar structure, generating a lot of cracks and recesses that increased the reactive sites. Furthermore, when the TiO$_2$ reacted with CS, it resulted in Ti-C, Ti-O and Ti-N bonds. The Br- can prevent the growth of TiO$_2$ crystal grains and strengthen the ability of I$^-$ to remove mercury. The adsorption isotherm and kinetic results indicated that the adsorption behaviour of CS-KI/TiO$_2$-HBr as it removes Hg$^{2+}$ is an inhomogeneous multilayer adsorption process. The surface adsorption and intraparticle diffusion effects are both important in the Hg$^{2+}$ adsorption process.

1. Introduction

Mercury is a toxic heavy metal element that usually exists in the lithosphere, hydrosphere and atmosphere. It has been a long-term pollution problem because of its bio-accumulation and migration. Coal-fired power plants are the main source of anthropogenic mercury emissions. In the coal-fired flue gas particle state, elemental mercury (Hg$^0$) and oxidized mercury (Hg$^{2+}$) are the main forms of mercury. Wet desulfurization (WFGD), a technology that uses limestone or lime as a sorbent, has been found to be effective to simultaneously remove SO$_2$ and Hg$^{2+}$ from the flue gas, which reduces mercury emission. Especially, the installation of denitrification equipment can enhance the oxidation of Hg$^0$ to Hg$^{2+}$, but results in mercury enrichment in the gypsum slurry. However, this method also has many disadvantages. The enriched Hg$^{2+}$ is converted to Hg$^0$ again by the reducing substances in gypsum slurry (such as sulfite ions), and this converted mercury is then re-released into the atmosphere [1]. The reuse of desulfurizing gypsum will release the mercury again. Any additional mercury that is in the wastewater from the desulfurization process is discharged into the environment, which will cause water pollution [2].

Studies have shown that the mercury in the desulfurization slurry mainly exists in the form of mercury sulfide (HgS) and mercury halide [3]. The desulfurization slurry undergoes a rise in temperature and pH, which promotes the transformation of Hg$^{2+}$ to Hg$^0$ [4, 5]. The research of Raquel [1] shows that Fenton reagent can make sulfite ions into sulfate ions, and the addition of sodium
sulfide (Na$_2$S) and 2,4,6 trimercaptotriazinetrisodium (TMT) can prevent the conversion of Hg$^{2+}$. Sun et al. [5, 6] compared the inhibitory effect of Na$_2$S, TMT and sodium dithiocarbamate (DTCR) for the reduction of Hg$^{2+}$, and found that DTCR has the best inhibition effect.

Polymer chitosan contains a lot of -NH$_2$ and -OH groups, and is a good adsorbent of heavy metal ions. Its amino and hydroxyl groups can act as chelating sites to effectively adsorb Hg$^{2+}$. However, chitosan has a low adsorption capacity and poor selectivity for heavy metal before it is modified. In this paper, a composite chitosan adsorbent was prepared by mixing KI and TiO$_2$ to realize adsorption of Hg$^{2+}$. Its adsorption for mercury in the gypsum liquid was tested. The objective of this work is to synthesize a novel sorbent that is suitable for mercury removal in flue gas after FGD in power plant. The determination of sorption isotherms and kinetics of Hg$^{2+}$ sorption on modified CS sorbent is important to find the controlling steps in uptake kinetics and achieve a complete adsorption mechanism.

2. Materials and methods

2.1. Materials

Chitosan (CS) (degree of deacetylation>85%), hydrobromic acid (HBr), 36% acetic acid (HAc), titanium dioxide (TiO$_2$), potassium iodide (KI), sodium hydroxide (NaOH), deionized water, gypsum.

2.2. Preparation of the adsorbents

20 ml 2% HBr (or HAc) was taken. 1 g CS was added. The mixture was stirred until it was solved; then, 10 ml deionized water (including 0.2g TiO$_2$ and 0.2g KI) was added. The mixture was magnetically stirred for 10 min in a 50°C water bath pot to form sol solution. Next, the sol was added to a 10%wt NaOH solution drop wise. The particles were filtered and washed until neutral, then allowed to dry at 50 °C. Then, the CS-KI/TiO$_2$-HBr (or CS-KI/TiO$_2$-HAc) gel particles were acquired.

2.3. Characterization of the adsorbents

The adsorption capacity of mercury was measured by a solid mercury analyzer (Lumex RA915+, Russia); Thermal stability analysis used a thermogravimetric analyzer (SDTQ-600, TA company in the United States); Microscopic morphology was observed by scanning electron microscopy (Dutch Phenom-Word company PhenomProX); Adsorbent structure was measured by a Fourier transform infrared spectrometer (American PerkinElmer Spectrum 100).

2.4. The experiment of mercury removal in the gypsum slurry

5g dry gypsum and 45 ml deionized water or 45 ml 1ppm mercury standard solution were used. They were configured to 10% gypsum slurry A and B and stirred for 10 min. It was determined that the pH of the solution A was 5. Then, 10% NaOH was added dropwise to the B solution, adjusting it until the pH was identical to solution A. 15 mg samples were placed into the A and B solution, and stirring was continued for 60 min in a 30°C water bath pot for the adsorption experiment. The samples were dried at 50 °C. Finally, the adsorption capacity of a single sample was tested by the mercury analyzer.

2.5. The experiments for mercury removal by CS-KI/TiO$_2$-HBr

The studies show that the adsorption isotherms of chitosan are mainly in line with the Langmuir model. The increase of temperature was helpful for the adsorption of Hg$^{2+}$ within a certain temperature range, and the ability to adsorb Hg$^{2+}$ was influenced by the Ph [7]. From the former study, CS-KI/TiO$_2$-HBr was found to have a better adsorption capacity for Hg$^{2+}$ in the gypsum slurry. So, CS-KI/TiO$_2$-HBr was used as the adsorbent to study the sorption behavior of Hg$^{2+}$. The experimental conditions were: total solution 50 ml, Hg$^{2+}$ concentration 1ppm, water bath temperature 30°C, adsorption time 1 hour and continuous stirring. The adsorption kinetics experiment was carried out at 303 K and pH 5.0 for 6h.
Adsorption isotherms could describe adsorbate distribution when the adsorption process reaches equilibrium. This is therefore effective to clarify the adsorption mechanism. Thus, the experiment was carried out at 303 K and pH 5.0, and the concentrations of Hg$^{2+}$ were 0.5, 1, 3, 5, 10, 20, 40, 60, and 80 ppm, at a volume of 50 ml.

3. Results and discussion

3.1. Mercury removal in the gypsum slurry

The test results are shown in Figure 1. As the figure shows, when the sample is less than 3 mg, especially in the low concentration mercury solution (A), the adsorption capacity is clearly influenced by the sample quality. Although the quality is lower, the adsorption capacity is enhanced. When the quality of the single sample is greater than 3 mg, there is not a significant change to the adsorption capacity. Also, the adsorption capacity of CS-KI/TiO$_2$-HBr is better than CS-KI/TiO$_2$-HAc, proving that the presence of Br$^-$ can enhance the Hg$^{2+}$ adsorption of CS.

![Figure 1](image.png)

**Figure 1.** (a) The original gypsum slurry (b) Add 1 ppm Hg$^{2+}$ to gypsum slurry.

3.2. Effects of solution pH and reaction temperature

Figure 2 shows the experimental results under different pH conditions. As can be seen from the figure, CS-KI/TiO$_2$-HBr had the largest adsorption capacity when the pH was between 5 and 7. At the same time, when the pH was more than 7 or less than 5, it greatly affected the adsorption capacity. The reason maybe that H$^+$ makes the -NH$_2$ of the CS protonate, and then it loses reactive activity. Also, it is easy for the OH$^-$ in the alkaline solution to react with Hg$^{2+}$ to produce Hg(OH)$_2$, which then decomposes into HgO precipitation. Thus, the concentration of Hg$^{2+}$ in the solution will decrease and affect the Hg$^{2+}$ adsorption on Chitosan. The reaction equations are as follows:

\[
\text{Hg}^{2+} + 2\text{OH}^- \rightarrow \text{HgO} \downarrow + \text{H}_2\text{O}
\]

Figure 3 shows that the adsorption capacity of CS-KI/TiO$_2$-HBr increased with an increase in temperature when it was under 50°C; however, when the temperature was over 50°C, the adsorption capacity reduced sharply. This is consistent with the findings in other literature [7]. In contrast, it is clear from Figures 2 and 3 that the absorption capacity of CS-KI/TiO$_2$-HBr in the gypsum slurry was lower at 1/3 than in water. It may be that other metal ions or nonmetallic ions in the gypsum slurry occupy the active site of the adsorbent, thus reducing the adsorption of Hg$^{2+}$. Additionally, the insoluble particles in the gypsum slurry occupy the micropores of the adsorbent, which can reduce the adsorption capacity of the adsorbent.
3.3. Heat stability experiment

A 12 mg sample was placed into the thermogravimetric analyzer, and the temperature was increased from 30 °C to 800 °C at a rate of 20 °C/min, used N₂ as shielding gas. As shown in Figure 4, the thermal stability of the two samples decreased, and there was no obvious weightlessness peak of the physical adsorption of water. The decomposition temperature of CS-KI/TiO₂-HAc is between 248°C and 331°C. The maximum weight loss rate was at 279°C. The thermal stability of CS-KI/TiO₂-HBr is better than that of CS-KI/TiO₂-HAc. Its decomposition temperature is from 235°C to 355°C, and at 288°C, its decomposition rate was the fastest.

The decrease in thermal stability was likely due to the CS taking a hydrolysis reaction by acid catalysed in the process of preparation. With the molecular chain of CS degradation, fracture, and...
shortening, the crystallinity and the thermal stability will decrease [8]. As compared to CS, the DTG diagrams of the composite adsorbents showed two adjacent weight loss peaks. The first weightless peak maybe formed by the pyrolysis of iodine. The presence of Br- reduced the pyrolysis of the iodine.

3.4. Scanning electron microscopy (SEM)

Figure 5 shows the microscopic morphology of the CS and the modified CS surface magnified about 20000 times by scanning electron microscopy. As Figure 5 (a) demonstrates, the fold and reticular formation are obvious on the surface of CS. As can be seen from Figures 5 (b) and (c), TiO$_2$ distributed well in the surface of the sample by the sheet structure, and the sample’s surface formed a large number of cracks and grooves, which provide more contact area to absorb the mercury. Also, many KI, which are much smaller than TiO$_2$, joined together to provide a new reactive site of absorbing the mercury. A comparison of Figures 5 (b) and (c) demonstrates that the grain size of TiO$_2$ layer of CS-KI/TiO$_2$-HBr is smaller than the CS-KI/TiO$_2$-HAc. It can increase the adsorbent’s surface area and promote adsorption of mercury, indicating that the presence of HBr can inhibit the growth of TiO$_2$ grains and that the use of HBr is better than HAc.

![Figure 5. SEM of original (a) CS, (b) CS-KI/TiO$_2$-HAc, (c) CS-KI/TiO$_2$-HBr.](image)

Figure 5. SEM of original (a) CS, (b) CS-KI/TiO$_2$-HAc, (c) CS-KI/TiO$_2$-HBr.

Figure 6 shows the scanning electron micrographs of Hg$^{2+}$ loaded adsorbents. Agglomerate and spherical particles were loaded on the adsorbents’ surface to reduce the porous structure. This was especially true of the CS-HAc (as Figure 6(d) shows); it has no co-ions, but has a multi-hole surface. The white grain of HgCl$_2$ is full of the porous surface of CS-HAc. Figures 6(e) and 6(f) are the SEM of CS sorbents which are modified by different acids and supplemented by KI and TiO$_2$. Though the existence of TiO$_2$ and KI influences observation of mercury adsorption, it can be clearly seen that many tiny white grains are well distributed on the surface of the sorbents. The cracks and grooves of the sorbents are full of white tiny spherical particles. This can be explained by inhomogeneous multilayer adsorption on the surface of the sorbent.

![Figure 6. SEM of Hg$^{2+}$ loaded adsorbents (d) CS-HAc, (e) CS-KI/TiO$_2$-HAc, (f) CS-KI/TiO$_2$-HBr.](image)

Figure 6. SEM of Hg$^{2+}$ loaded adsorbents (d) CS-HAc, (e) CS-KI/TiO$_2$-HAc, (f) CS-KI/TiO$_2$-HBr.

3.5. Fourier transform infrared spectroscopy (FTIR)

The infrared spectrum can be used to analyze molecular structure and chemical bonding. The infrared spectrum can reflect the change of the samples after modification, and can be used to analyze the effective functional groups of adsorbents. As shown in Figure 7, after modification, the O-H stretching vibration peak of the CS was at 3373cm$^{-1}$, the C-O stretching vibration peak of the CS was at 1084cm$^{-1}$.
and the C-H stretching vibration peak of CS was at 2918 cm\(^{-1}\) [9, 10]. The O-H, C-O and C-H stretching vibration peak of CS all have undergone place migration. The peaks became sharp, perhaps because the addition of TiO\(_2\) produced Ti-O bonds or Ti-C bonds. Also, the amide I band at 1661 cm\(^{-1}\) migrated to low wave about 5cm\(^{-1}\), which perhaps formed the Ti-N bonds. Besides a new peak appeared at 1450 cm\(^{-1}\) in the modified samples. It is possible that TiO\(_2\) and the CS form ed the Ti-O bonds. At the same time, the C-O vibration peak at 1084 cm\(^{-1}\) of C\(_3\) moved to 1071 cm\(^{-1}\) and the peak intensity greatly increased, which is a typical TiO\(_2\) characteristic peak. The modified samples also produced double I- peaks, which replaced H+ at 900 cm\(^{-1}\). All the samples formed C-Br absorption peaks near 667cm\(^{-1}\) because the KBr had a reaction with the sample during the process of making the tablet.

The sorbent structure underwent significant changes, as shown by the contrast between Figures 7 and 8. The large band around 3410 cm\(^{-1}\) is commonly attributed to the combination of stretching vibrations of OH and NH\(_2\) groups that shift to higher wave numbers after adsorption of Hg\(^{2+}\), especially CS-KI/TiO\(_2\)-HBr. The peaks at around 1661cm\(^{-1}\) and 1076 cm\(^{-1}\) are assigned to –NH\(_2\) and –C–OH [11, 12], which all shift to lower wave numbers after adsorbing Hg\(^{2+}\). Because of the chelating effects, new chemical bonds are formed between CS and Hg\(^{2+}\) [13-16]. The bands at 2922 and 2818cm\(^{-1}\) are attributed to C-Ti disappearing in CS-KI/TiO\(_2\)-HBr. This indicates that Hg\(^{2+}\) is covered by HgI\(_2\). The band at 1071 cm\(^{-1}\), which is a typical TiO\(_2\) characteristic peak, is weak in CS-KI/TiO\(_2\)-HBr. Combined with Figure 6(f), it leads to the conclusion that the TiO\(_2\) surface maybe covered with a large number of mercury compounds like HgI\(_2\) or HgBr\(_2\). The chemical reaction mechanism is as follows:

\[
\text{Hg}^{2+} + 2\text{Br}^- \rightarrow \text{HgBr}_2
\]

(2)

\[
\text{Hg}^{2+} + 2\text{I}^- \rightarrow \text{HgI}_2
\]

(3)

The Hg\(^{2+}\) adsorption on the composite sorbent is inhomogeneous multilayer adsorption. The surface adsorption and intraparticle diffusion effects in the Hg\(^{2+}\) adsorption process are both important.

**Figure 7.** FTIR of the adsorbents.

**Figure 8.** FTIR of the adsorbents after adsorption Hg\(^{2+}\).

### 3.6. Adsorption equilibrium study

#### 3.6.1. Adsorption kinetics

Adsorption kinetics is a fundamental and significant tool to evaluate adsorption behaviour [9, 10, 17, 18]. Typical kinetic adsorption results are shown in Figure 9.

From above, the metal ions are chelated onto the adsorbents during the adsorption process. On the basis of the pseudo-first order, pseudo-second order and intraparticle diffusion kinetic models, the kinetic data simulation was taken to further study the adsorption mechanism. The equations are expressed in Table 1, where k1 is the pseudo-first order rate constant(min\(^{-1}\)) of sorption, qe and q(t) (mg/g) are the amounts of Hg\(^{2+}\) sorbed at equilibrium and time t, k2 is the pseudo-second order rate constant (g mg\(^{-1}\)min\(^{-1}\)), and Ki is the intraparticle diffusion rate (mg g\(^{-1}\)min\(^{0.5}\)).
Figure 9. Adsorption kinetics of CS-KI/TiO$_2$-HBr for removal of Hg$^{2+}$ (PH 5; T: 303K).

After fitting by the kinetic models, the parameters and the correlation coefficients ($R^2$) are listed in Table 2. The $R^2$ value of the intraparticle diffusion model is closer to 1.0 than the other two, indicating that is the most suitable model to describe the adsorption of CS-KI/TiO$_2$-HBr for Hg$^{2+}$. However, all three models had very similar $R^2$ values. The pseudo-second order equation was slightly better than the pseudo-first order model. In addition, the intraparticle diffusion is very important because the sorbent is a composite material. The addition of TiO$_2$, KI and HBr made the adsorbents own a special surface morphology that had large number of cracks and grooves. That will make the adsorption of Hg$^{2+}$ easier because of the surface adsorption and intraparticle diffusion effects.

Table 1. Kinetics model equations.

| Type                        | Non-Linear form       | Linear form                         | Plot                        |
|-----------------------------|-----------------------|-------------------------------------|-----------------------------|
| Pseudo-first order model    | $q_t = q_e (1-e^{-K_1 t})$ | $\log(q_t-q_e) = \log q_e - \left( \frac{K_1}{2.303} t \right)$ | $\log(q_t-q_e)$ vs. $t$    |
| Pseudo-second order model   | $q_t = \frac{K_2 t}{1+K_1 q_t}$ | $\frac{t}{q_t} = \frac{1}{K_2 q_t} + \frac{1}{q_i}$ | $\frac{t}{q_t}$ vs. $t$   |
| Intraparticle diffusion model| $q_t = K_i t^{0.5} + C$ | $q_t = K_i t^{0.5} + C$ | $q_t$ vs. $t^{0.5}$        |

Table 2. Kinetic parameters for Hg$^{2+}$ sorption.

| Adsorbent   | $q_{max}$ (mg/g) | $Pseudo$-first order model | $Pseudo$-second order model | Intraparticle diffusion model |
|-------------|------------------|-----------------------------|-----------------------------|-------------------------------|
|             | $K_1$ (min$^{-1}$) | $q_e$ (mg/g) | $R^2$ | $K_2$ (min$^{-1}$) | $q_e$ (mg/g) | $R^2$ | $K_i$ (mg/g·min$^{-0.5}$) | $C$ (mg/g) | $R^2$ |
| CS-KI/TI0$_2$-HBr | 1.95 | 0.0077 | 1.97 | 0.934 | 0.0035 | 2.39 | 0.939 | 0.085 | 0.14 | 0.952 |

3.6.2. Adsorption equilibrium analysis. The results of the adsorption isotherms are shown in Figure 10. From Figure 10, the equilibrium Hg uptake of CS-KI/TiO$_2$-HBr is approximately 1.98mg/g.

The Langmuir and Freundlich models are also used to further study the adsorption process. The former is based on the assumption of monolayer adsorption on a homogeneous surface. The latterised based on the assumption that adsorption energy decays exponentially with coverage rise. This condition is usually present in inhomogeneous multilayer adsorption on the surface [9 10]. Their equations are expressed in Table 3.
### Figure 10. Adsorption isotherms of CS-KI/TiO$_2$-HBr for removal of Hg$^{2+}$ (PH 5; T: 303K).

### Table 3. Adsorption isotherm equations.

| Type       | Non-Linear form | Linear form | Plot          |
|------------|-----------------|-------------|---------------|
| Langmuir   | $q_\text{eq} = \frac{q_{\text{m,L}} K_C C_{\text{eq}}}{1 + K_C C_{\text{eq}}}$ | $\frac{C_{\text{eq}}}{q_\text{eq}} = \frac{1}{q_{\text{m,L}}} K_C + \frac{1}{q_{\text{m,L}}} C_{\text{eq}}$ | $C_{\text{eq}}$ vs. $C_{\text{eq}}$ |
| Freundlich | $q_\text{eq} = K_C C_{\text{eq}}^{1/n}$ | $\ln q_\text{eq} = \ln K_F + \frac{1}{n} \ln C_{\text{eq}}$ | $\ln q_\text{eq}$ vs. $\ln C_{\text{eq}}$ |

The fitting results are listed in Table 4. Evidently, the correlation coefficient (R$^2$) of the Freundlich model is closer to 1.0, which is much higher than the Langmuir model. That is to say that the mercury ions first interacted with the adsorbent surface at sites with strong binding forces, only then moving to sites where the binding force is weak. As the adsorption process progresses, the binding force between the adsorbent and the molecule becomes increasingly weak. The Hg$^{2+}$ adsorption on the composite sorbent is inhomogeneous multilayer adsorption, as Figure 6 demonstrates.

### Table 4. Parameters of the adsorption isotherm.

| Sorbent               | $q_{\text{max}}$ (mg/g) | $q_{\text{m,L}}$ (mg/g) | $K_L$ (L/mg) | $R^2$ | $n$ | $K_F$ | $R^2$ |
|-----------------------|--------------------------|--------------------------|--------------|-------|-----|-------|-------|
| CS-KI/TiO$_2$-HBr     | 1.95                     | 1.98                     | 0.24         | 0.950 | 3.39| 0.58  | 0.963 |

### 4. Conclusion

A novel Chitosan adsorbent compound for mixing with KI and TiO$_2$ was developed for use in mercury adsorption experiments on gypsum slurry. The results show that the composite adsorbents successfully adsorbed both low and the high concentration Hg$^{2+}$ in the gypsum slurry. HBr as a solvent in composite adsorbent for gypsum slurry Hg$^{2+}$ adsorption performed better than HAc in preparing the adsorbent. The presence of Br$^-$ can prevent the growth of TiO$_2$ crystal grain and strengthen the ability of I$^-$ to remove mercury and enhance the adsorption ability of chitosan for Hg$^{2+}$. Also, TiO$_2$ may form Ti-C, Ti-O and Ti-N bonds with CS. The lamellar structure of TiO$_2$ promotes the adsorption of Hg$^{2+}$ by the chitosan. The adsorption isotherms and kinetics simulation indicated that the adsorption behaviour of CS-KI/TiO$_2$-HBr to remove the Hg$^{2+}$ was an inhomogeneous multilayer adsorption process. The surface adsorption and intraparticle diffusion effects both are important to the Hg$^{2+}$ adsorption process. However, the inhibition effect of Hg$^{2+}$ into Hg0 was not verified for the composite materials in this work. Therefore, further research should focus on this inhibition effect.
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