Mercury removal from coal combustion flue gas by pyrite-modified fly ash adsorbent

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Received: 1 October 2021 / Accepted: 26 January 2022 / Published online: 31 January 2022
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Abstract
Pyrite and fly ash have certain advantages in adsorption and mercury oxidation. The pyrite-modified fly ash (PY + AC-FA) mercury adsorbent was prepared by mixing pyrite (PY) with acid-modified fly ash (AC-FA), which has better mercury removal effect than AC-FA. The experimental results of mercury adsorption show the following: when the reaction temperature is 50 °C, the best doping proportion of modified fly ash is 20 wt%, the mass proportion of pyrite to acid-modified fly ash is 4:1, and the flue gas flow rate is 1.0 L/min; the adsorbent has the best performance, and the adsorption rate of mercury reaches 91.92%. It was also found that the quasi-second-order kinetic model could describe the entire process of adsorption, and its adsorption process was mainly influenced by chemisorption. XRF, BET, SEM, XRD, and TG-DSG were used to characterize these adsorbents, and the mechanism of mercury removal of pyrite-modified fly ash adsorbent is inferred: Hg0 is first adsorbed on the surface of the adsorbent, and then oxidized to HgS by the active component FeS2 in pyrite-modified fly ash.

Keywords Fly ash · Pyrite · Hg0 removal · Kinetic model

Introduction
Coal-fired power plants emit large amounts of mercury during coal combustion (Pirrone et al. 2010). Mercury has high toxicity, non-degradability, and bioaccumulation, which can pose a great threat to human health and the ecological environment (Lin et al. 2014). Therefore, it is important to reduce mercury emissions from coal-fired power plants.

Post-combustion mercury removal technology is the mainstream technology at present. There are two kinds of mercury removal technologies after combustion: one is to spray activated carbon or other kinds of solid adsorbent to remove mercury (Olson et al. 2000; Presto and Granite 2006a, b); the other is the oxidation of mercury through catalysts in denitration equipment. Oxidized mercury is absorbed by wet desulfurization equipment (Yang et al. 2016; Luo et al. 2011; Zhou et al. 2016; Li et al. 2015; Li et al. 2018b, a). Relevant experiments (Han et al. 2016) showed that the modified activated carbon adsorbent could reach more than 92% efficiency of mercury removal. However, the cost of this method is relatively high, and the application in practice needs to reduce the operating cost by recycling.

Fly ash has the advantages of wide source and low price, which makes it have a large market in practical application (Presto and Granite 2006a, b; Wang and Wu 2006). The fly ash consists of inorganic components and organic components. The inorganic components account for about 98%, mainly including aluminosilicates, silicates, sulfates, calcium oxide, and iron oxides (Xia et al. 2015). The adsorption efficiency of mercury from flue gas by fly ash can reach 23.1–26.9%; the removal mechanism was physical and chemical adsorption (Xu et al. 2013). In order to improve the removal efficiency of mercury from fly ash and meet the standard of practical application, the current research mainly focuses on modifying fly ash to improve its adsorption performance for pollutants. Tian et al. (2017) used NaCl and NaBr for loading modification of coal-fired power plant fly ash. The results showed that the adsorbent modified with 5% NaCl solution for 3 h has a better performance of mercury removal. Under certain temperature conditions, the efficiency of mercury removal was above 85%, with the highest
being 92.6%. Therefore, the modified fly ash can be used as a cost-effective substitute for activated carbon.

Pyrite is a sulfide mineral rich in nature, containing a large amount of Fe and S elements (Duan et al. 2016; Han et al. 2014). The reduced sulfur ligands can be complexed with mercury to form the insoluble and potentially less hazardous HgS (Sun et al. 2017; Stein et al. 1996; Willet et al. 1992). In this study, pyrite-modified fly ash (PY+AC-FA) adsorbent with good performance of mercury removal was prepared by doping the modified fly ash with pyrite. The effects of the modification method, reaction temperature, nitrogen flow rate, and doping proportion of fly ash were investigated. BET, SEM, XRD, XRF, and TG-DSC were used to characterize the internal structure and the mechanism of mercury removal, which provided a new idea for the adsorption of mercury from fly ash. Adsorption kinetic study on pyrite-modified fly ash (PY+AC-FA) was performed and the test results were fitted with quasi-secondary kinetics and intra-particle diffusion models, respectively.

**Methods and materials**

**Adsorption performance evaluation system**

In order to evaluate the adsorbent performance, a fixed bed reactor was constructed as shown in Fig. 1. The performance of various adsorbents and the mechanism of mercury removal were investigated. The fixed bed reaction system consists of three main components: the mercury vapor generation unit, fixed bed reaction unit, and tail gas treatment unit. Because of the instability of pyrite in air (Bower et al. 2008), N₂ was used as the carrier gas throughout the experiment. The experimental pipeline was connected by PTFE tube, and the entire pipeline was wrapped with heating cable to prevent the mercury from liquefying and remaining in the pipeline.

The mercury vapor is emitted from the mercury permeable tube in the U-shaped tube. When the external temperature is constant, the mercury in the U-shaped tube reaches vapor–liquid equilibrium; the mercury vapor steadily escapes at a certain rate, and then is blown into the gas path by N₂. The initial mercury concentration can be adjusted by changing the water bath temperature and nitrogen flow rate. The fixed bed reactor is composed of a quartz reaction tube and a temperature-controlled heating tape. To prevent leakage during aeration, the adsorbent is placed between two layers of glass wool. In this experiment, KMnO₄/H₂SO₄ absorption method (Wang et al. 2013) was used to determine the concentration of Hg⁰. After the reaction, the tail gas may contain a certain amount of mercury, and the tail gas needs to pass through the first acidic potassium permanganate absorption bottle and the activated carbon adsorption treatment, and finally be collected by the tail gas treatment device.

**Materials**

**Reagents**

Sulfuric acid (H₂SO₄), hydrochloric acid (HCl), nitric acid (HNO₃), potassium permanganate (KMnO₄), and stannous chloride (SnCl₂) were all analytical reagent.

**Instruments**

The BET of the adsorbents was determined by Quadrasorb EVO™. The specific surface area, pore volume, and pore size distribution of the material to be measured were determined by nitrogen physical adsorption method. The microstructure of adsorbents was characterized by scanning electron microscopy (SEM). The crystal structure of adsorbents was determined by X-ray diffraction spectroscopy (XRD). The element composition of adsorbents was determined by X-ray fluorescence probe (XRF). The thermogravimetric...
analysis (TG-DSC) instrument model was STA-8000 to analyze the adsorbents on the reacted material. X-ray fluorescence spectrometer (XRF) was used to analyze the chemical composition of fly ash and pyrite, as shown in Table 1.

**Adsorbent preparation**

The fly ash used in this experiment was taken from a power plant in North China, marked as FA. Weigh an appropriate amount of FA into a conical flask, add \( \text{H}_2\text{SO}_4 \) or \( \text{NaOH} \), and stir for half an hour at 25 °C. After filtration and drying, acid-modified fly ash (AC-FA) or alkali-modified fly ash (AL-FA) was obtained.

The group with better mercury removal performance in AC-FA and AL-FA was selected to be doped with pyrite (PY), and the effects of fly ash doping amount, reaction temperature, doping proportion, and carrier gas flow rate on mercury removal performance were investigated.

**Adsorption performance experiment**

Before starting the experiment, turn on the fume hood, mercury analyzer, water bath, and heat belt for preheating. Weigh 2.00 g of adsorbent accurately; the adsorbent is placed in the reactor with the glass wool. The mercury concentration in the \( \text{H}_2\text{SO}_4/\text{KMnO}_4 \) solution in this experiment was measured. Each sample was measured three times to take its average value. After the overall experiment, the ventilation should be stopped first, the U-shaped tube was removed, and then, the lower wall of the mercury permeation tube was separate from the glass beads. The mercury permeation tube should be removed after cooling.

The mercury removal efficiency was measured by the concentration of mercury before and after the reaction. The mercury concentration is determined by the absorbance of the samples.

**Data analysis processing**

**Removal efficiency**

The mercury removal efficiency of the adsorbent was measured according to the following equation Eq. (1):

\[
\eta = \frac{\text{Hg}_{\text{in}} - \text{Hg}_{\text{out}}}{\text{Hg}_{\text{in}}} \times 100\%
\]

where \( \text{Hg}_{\text{in}} \)—initial concentration of gaseous elemental mercury, μg/L; \( \text{Hg}_{\text{out}} \)—concentration of gaseous elemental mercury after adsorption, μg/L; \( \eta \)—mercury removal efficiency, %.

**Adsorption amount**

The formula for calculating the adsorption amount of mercury at time \( t \), Eq. (2).

\[
Q_t = \frac{(\text{Hg}_{\text{in}} - \text{Hg}_{\text{out}})Lt}{m}
\]

where \( Q_t \)—the amount of mercury adsorbed at time \( t \), μg/g; \( L \)—gas flow rate, L/min; \( t \)—reaction time, min; \( m \)—the amount of samples, g.

**Adsorption kinetic analysis**

The adsorption kinetics experiments were carried out by using the intra-particle diffusion Eq. (3), and the quasi-second-order kinetic Eq. (4).

\[
\frac{dQ_t}{dt} = k_p Q_{t}^{1/2}
\]

\[
\frac{dQ_t}{dt} = k_2 (Q_e - Q_t)^2
\]

where \( Q_t \)—the amount of mercury adsorbed at time \( t \), μg/g; \( Q_e \)—the amount of mercury adsorbed at adsorption equilibrium, μg/g; \( k_p \)—the intra-particle diffusion rate constant, μg·(g·min\(^{1/2}\))\(^{-1}\); \( k_2 \)—the secondary adsorption rate constant, μg·(g·min\(^{-1}\))\(^{-1}\).

**Results and discussions**

**Characterization results and discussion**

**BET**

The BET characterization results are shown in Table 2.
As shown in Table 2, the specific surface area of FA was 1.610 m²/g, and the specific surface area of AC-FA and AL-FA was 2.612 m²/g and 3.969 m²/g, respectively, which increased by 62.24% and 146.52%. The XRD patterns show the differences between the FA samples before and after the processing. It can be seen from the Fig. 3 that the diffraction peaks of FA before and after modification were many and complex. The peaks marked in the Fig. 3 were the diffraction peaks of mullite (AS), followed by quartz and other substances (Li et al. 2017a, b). By comparison, there was little difference before and after modification except diffraction peak. The peak of modified fly ash was narrower and sharper, and the peak width was smaller. According to Scherrer formula ($\beta = 0.89\lambda /D\cos\theta$, where $\lambda$ is the x-ray wavelength, $D$ is the average crystal size.), the particle size of modified fly ash was smaller and had a larger specific surface area, which was also consistent with the BET measurement results in the “BET” section. It can be seen that sulfuric acid and sodium hydroxide only change the physical structure of the surface of FA, and the overall crystal and material structure of FA did not change.

As can be seen from Fig. 4, the main diffraction peaks of the PY + AC-FA were mullite and FeS₂. The diffraction peak response values of mullite and FeS₂ change with the proportion of AC-FA, which was consistent with the actual situation.

The adsorbents before and after the adsorption reaction were analyzed by XRD to study the reaction mechanism of PY + AC-FA removal of mercury. Figure 5 shows the comparison results.

It can be seen from Fig. 5 that the diffraction peak types of PY + AC-FA before and after the reaction were basically unchanged, and only the diffraction peaks of FeS₂ were different. The intensity of FeS₂ diffraction peaks of PY + AC-FA after the reaction was obviously weaker than that before the reaction. To make the comparison more obvious, the two images were overlapped on the same baseline. As shown in Fig. 5, the response value of the FeS₂ diffraction peaks of PY + AC-FA before the reaction was higher than that after the reaction, which indicates that FeS₂ was involved in the reaction and was consumed by reduction in the process of mercury removal (Dyrssen and Wedborg 1991; Yang et al. 2018).

### TG-DSC

TG-DSC characterization verified that FeS₂ in PY + AC-FA was reduced and consumed in the process of mercury removal. Figure 6 shows the TG-DSC patterns of PY + AC-FA when heated at various temperatures up to 800 °C. As shown in Fig. 6, the weight loss peak of surface adsorbed water on PY + AC-FA appeared between 27 and 110 °C. As the temperature increases, the weight dropped slowly and finally reaches 15.213 mg at 420 °C. This indicates that Hg⁰ (ad) adsorbing on the surface of PY + AC-FA begins to evaporate above its boiling point. The pattern decreases significantly after 420 °C, which indicates that PY + AC-FA began to lose weight substantially. This can be interpreted as the chemical adsorption of FeS₂ in PY + AC-FA with Hg⁰ produces HgS (Li et al. 2017a, b; Liao et al. 2016; Zhao et al. 2016; Wu et al. 2008), and when

| Sample  | Specific surface area (m²/g) | Pore volume (cc/g) | Average pore size (nm) |
|---------|-----------------------------|-------------------|------------------------|
| FA      | 1.610                       | 0.004             | 4.737                  |
| PY      | 0.953                       | 0.003             | 4.173                  |
| AC-FA   | 2.612                       | 0.008             | 4.168                  |
| AL-FA   | 3.969                       | 0.010             | 4.435                  |
| PY + AC-FA | 6.276                  | 0.011             | 2.523                  |
Fig. 2 SEM characterization of samples
the temperature is higher than 420 °C, HgS starts to decompose into Hg and S (Bhargava et al. 2009; Yang et al. 2017). Therefore, the TG-DSC analysis indicates that PY + AC-FA contains surface adsorbed water, Hg, and HgS.

**Mercury removal performance of modified fly ash**

Figure 7 shows the mercury removal efficiency of FA, AC-FA, AL-FA, PY, and PY + AC-FA. With the increase of reaction time, the mercury removal efficiency of the adsorbents increased slowly and then decreased gradually. The maximum mercury removal efficiency of FA was 45.85% when the reaction time was 5 min. Compared with AL-FA, the mercury removal efficiency of AC-FA is higher. When the reaction time was 10 min, the mercury removal efficiency of AC-FA and AL-FA both reached the maximum, and the maximum mercury removal efficiency was 63.97% and 52.09%, respectively. The experimental results showed that both acid–alkali modification could effectively improve the removal capacity of fly ash for mercury (Geng et al. 2021). The maximum mercury removal efficiency of pyrite PY was 74.39%, which was significantly higher than that of FA, AC-FA, and AL-FA, indicating that chemical action was greater than physical adsorption in the reaction system of this test.

PY + AC-FA was prepared by mixing AC-FA and PY in a certain proportion. Compared with AC-FA and PY,
PY + AC-FA offer greater improvements in mercury removal efficiency and persistence, especially the persistence. The mercury removal efficiency of the pyrite adsorbent doped with AC-FA increased from 74.39 to 84.70% at 10 min. After 10 min, PY + AC-FA could still maintain about 75% of the mercury removal efficiency, and the efficiency could last until 30 min. The reason for the better adsorption performance of PY + AC-FA was that PY + AC-FA had a large specific surface area according to the BET and SEM characterization in the “BET” and the “SEM” sections. Such a structure was favorable for the adsorption capacity.

PY + AC-FA showed the best efficiency of mercury removal; therefore, subsequent experiments were conducted to characterize PY + AC-FA and investigate the adsorption properties.

**Mercury removal performance of PY + AC-FA**

**Adsorption kinetics of mercury by PY + AC-FA**

Figure 8 shows the adsorption kinetics and fitting curve of mercury by PY + AC-FA. When the nitrogen flow rate was 1.2 L/min, the inlet mercury concentration was 10 μg/L, and the amount of PY + AC-FA was 2 g; the mercury adsorption amount showed a trend of rapid increase and then slow decrease with the increase of adsorption time. As shown in Fig. 8, when the adsorption time was 60 min, the amount of mercury adsorbed by PY + AC-FA was 191.59 μg/g, and the removal efficiency was 53.22%, which still maintained more than half of the efficiency. In the test time range (0–120 min), the maximum mercury adsorption capacity of PY + AC-FA was 239.26 μg/g, which showed good adsorption capacity.

**Reaction conditions** The nitrogen flow rate is 1.2 L/min; the mercury permeation tube water bath temperature is 60 °C; the reaction temperature is 50 °C; the mercury concentration is 10 μg/L; the mass proportion of AC-FA and PY is 1:1.

The adsorption kinetic process of PY + AC-FA was fitted and analyzed by the intra-particle diffusion model and quasi-second-order kinetic model, respectively. The specific fitting curves and fitting parameters were shown in Fig. 8 and Table 3. As can be seen from the fitted graphs, the curve of quasi-second-order kinetic model had a higher degree of fitting with the measured value. According to the specific fitting parameters in Table 3, the correlation coefficients $R^2$ of intra-particle diffusion and quasi-second-order kinetic model were 0.9500 and 0.9954, respectively, which show extremely significant correlation. Obviously, the quasi-second-order kinetic model showed a higher correlation. In summary, the kinetics of mercury adsorption by PY + AC-FA was consistent with the quasi-second-order kinetic model, and its adsorption process was mainly influenced by chemisorption (Yu et al. 2020).

**Effect of reaction temperature on adsorbent**

Figure 9 shows the curve of the mercury removal efficiency with reaction temperature for PY + AC-FA. As the test temperature increased from 30 to 110 °C, the mercury removal efficiency increased rapidly and then stabilized. When the reaction temperature was 50 °C, the reaction system reached the best state, and the mercury removal efficiency reached 84.70%. The mercury removal efficiency of PY + AC-FA exceeded 75% from 40 to 90 °C, and still maintained a high level. When the reaction temperature was less than
40 °C, the chemical reaction activity of the adsorption system was insufficient, which affected the reaction between PY + AC-FA and mercury, resulting in low efficiency (Zhang et al. 2016). After the experimental temperature exceeded 90 °C, the mercury removal efficiency decreased significantly. The effect of reaction temperature on the Hg0 removal by the adsorbent was mainly manifested in two aspects: on the one hand, the increase of temperature would promote the removal of Hg0 from the surface of PY + AC-FA, so that Hg0 would be carried away by nitrogen before it was oxidized; on the other hand, higher temperature could improve the activity of PY + AC-FA (Li et al. 2018b, a), but as the temperature continued to increase, it would cause the release of elemental sulfur from PY + AC-FA, resulting in lower removal efficiency (Zhang et al. 2019).

**Reaction conditions** The nitrogen flow rate is 1.2 L/min; the mercury permeation tube water bath temperature is 60 °C; the reaction time is 10 min; the mass proportion of AC-FA and PY is 1:1.

**Effect of nitrogen flow rate on adsorbent**

The flow rate of nitrogen had three main effects on the efficiency of mercury removal: the concentration of imported mercury, the reaction time of Hg0 with the adsorbent, and the driving force of nitrogen in the adsorption process. With the increase of flow rate, the gas driving force would increase, and the reaction time between adsorbent and Hg0 would decrease. The higher the imported mercury concentration, the more active sites and empty spaces were required for adsorption (Hsi et al. 2001). Therefore, the relative adsorption space and active sites in the adsorbent were reduced, which led to a decrease in the efficiency of mercury removal. As shown in Fig. 10, the mercury removal efficiency reached the highest when the nitrogen flow rate was 1.0 L/min. The driving force of the flow rate was the main factor affecting the adsorption when the flow rate was small. However, when the flow rate was too high, the decrease of reaction time and the increasing concentration of mercury became the dominant factors, which reduced the performance of the adsorbent in removing mercury.

**Reaction conditions** The mercury permeation tube water bath temperature is 60 °C; the reaction temperature is 50 °C; the reaction time is 10 min; the mass proportion of AC-FA and PY is 1:1.

**Effect of AC-FA doping proportion on adsorbent**

In order to investigate the effect of the percentage of AC-FA on the efficiency of the adsorbent, PY + AC-FA adsorbents with modified fly ash percentages of 20wt%, 25wt%, 33.3wt%,
50wt%, 66.7wt%, 75wt%, and 80wt% were prepared, respectively. The variation of mercury adsorption efficiency with the proportion of AC-FA is shown in Fig. 11.

As shown in Fig. 11, when the proportion of AC-FA was 0, the mercury removal efficiency of PY + AC-FA was 74.39%. With the increase of the proportion, the mercury removal performance of adsorbent was greatly improved to 91.92%. However, when the proportion of AC-FA was more than 20wt%, the mercury removal efficiency decreased from 91.92 to 59.1%. When the proportion of AC-FA was less than 20wt%, the incorporation of AC-FA increased the specific surface area of PY + AC-FA and enhanced its physical adsorption capacity. However, with the increase of the AC-FA proportion, the pyrite content decreased and the adsorbent surface mainly undergone physical adsorption, which was not sufficient to remove mercury from the flue gas, thus leading to a gradual decrease in the efficiency of mercury removal.

**Reaction conditions** The nitrogen flow rate is 1.2 L/min; the mercury permeation tube water bath temperature is 60 °C; the reaction time is 10 min; the reaction temperature is 50 °C.

**Mechanism discussion**

Based on the BET, SEM, XRF, XRD, TG-DSC characterization of adsorbents, the Hg⁰ removal mechanism was proposed, as shown in Eqs. (5)–(7).

Firstly, Hg⁰ was adsorbed on the surface of the adsorbent,

\[ \text{Hg}^0_{\text{g}} + (\text{PY} + \text{AC} - \text{FA}) \rightarrow \text{Hg}^0_{\text{ad}} \]  \hspace{1cm} (5)

Secondly, Hg⁰ reacted with the active component FeS₂ in PY + AC-FA,

\[ \text{Hg}^0_{\text{ad}} + \text{FeS}_2 \rightarrow \text{HgS}_{\text{ad}} + \text{FeS} \]  \hspace{1cm} (6)

\[ \text{Hg}^0_{\text{ad}} + \text{FeS} \rightarrow \text{HgS}_{\text{ad}} + \text{Fe} \]  \hspace{1cm} (7)

Finally, most of the mercury was adsorbed on the surface of the adsorbent as HgS.

**Conclusion**

In this paper, the sulfuric acid-modified fly ash (AC-FA) was mixed with pyrite (PY), and the pyrite-modified fly ash (PY + AC-FA) adsorbent for mercury removal was successfully prepared. The characterizations results showed that PY + AC-FA had large specific surface area, pore diameter and successfully loaded FeS₂ on the surface. When the reaction temperature was 50 °C, the doping proportion of AC-FA was 20 wt%, and the nitrogen flow rate was 1.0 L/min; the removal rate of Hg⁰ could reach 91.92%. It was also found that the quasi-second-order kinetic model could describe the entire process of adsorption, and its adsorption process was mainly influenced by chemisorption. Based on the XRF, BET, SEM, XRD and TG-DSC characterization of adsorbents, the mercury removal mechanism of pyrite-modified fly ash (PY + AC-FA) was proposed: first, Hg⁰ was adsorbed on the surface of the adsorbents; then, Hg⁰ was oxidized by the active component FeS₂ in pyrite-modified fly ash (PY + AC-FA) to HgS, and finally, most of the mercury was adsorbed on the surface of the adsorbents in the form of HgS.

**Author contribution** Xu WANG: Conceptualization, Data Curation, Writing-Original Draft. Liqiang QI: Resources, Writing-Review&Editing, Funding acquisition. Wen WANG: Formal analysis. Jingxin LI: Supervision. Yan HUANG: Methodology.

**Funding** This work was supported by the Natural Science Foundation of Hebei Province (Grant No. B2019502067) and the Fundamental Research Funds for the Central Universities (Grant No. 2020MS152).

**Declarations**

**Ethics approval and consent to participate** Not applicable.

**Consent for publication** Not applicable.

**Competing interests** The authors declare no competing interests.
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