Study on Mercury Species in Coal and Pyrolysis-Based Mercury Removal before Utilization

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ABSTRACT: Coal-fired mercury (Hg) pollution control is an important global environmental context. Eight coal samples from different coal fields in China were used to investigate Hg species and the Hg removal effects under different pyrolysis conditions in the presence of nitrogen. These conditions included temperature, particle size, and residence time. The study concludes that the temperature is the most important factor affecting Hg removal from coal, and the mercury release activity at specific temperatures depends on the species and content of Hg present. Large particle size limits the removal rate of Hg, and coal particles smaller than 40 mesh are more favorable for the rapid removal of Hg. For most coal types, pyrolysis of 10–15 min can achieve the ultimate Hg removal effect. Rapid pyrolysis at 600 °C in nitrogen is feasible to remove Hg from coal. Consequently, the Hg removal rate reaches 88–100%, the loss rate of coal calorific value is 2–12%, and approximately 17–58% of S is removed synergistically. HgS, HgSe, HgSO₄, organic matter Hg, and HgO are the main types of Hg species detected in coal, whose thermal decomposition characteristics are the essential criteria for determining the type of Hg removal process. This research will facilitate the improvement of pollution control methods for coal-source Hg pollution.

1. INTRODUCTION

Mercury, due its highly volatile character, high toxicity, bioaccumulation, and strong mobility, has been acknowledged as a global threat to human and environmental health.¹,² Based on data from the "Global Mercury Assessment 2018" released by the United Nations Environment Programme (UNEP), the global inventory of anthropogenic Hg emissions to the atmosphere is estimated to be 2220 t, of which approximately 21% is from coal combustion.³ In China, coal accounts for 47% of anthropogenic Hg emissions.⁴ Despite the dynamic entry of renewable energy sources, it is believed that coal will continue to maintain its dominant role as a primary energy source for global energy generation for many years to come.⁵ Therefore, the clean and safe use of coal has been a priority in energy policies of many countries and organizations. In recent years, increasing attention has been paid to the enhancement of conventional pollution control, determination of combustion transformation, and development of advanced control methods to reduce harmful gases caused by coal combustion, such as Hg, SO₂, and NOₓ.⁶,⁷

The control methods for coal-source Hg pollution are mainly divided into two types: precombustion Hg removal and postcombustion Hg removal. The conventional method for removing Hg before combustion is to improve the coal preparation technology. This is mainly achieved through processes such as deashing and desulfurization (pyrite).⁸ The cost of coal preparation technology is relatively low, but the Hg removal is easily affected by mineral components and coal preparation equipment. As a result, the Hg removal rate varies widely, usually in the range of 23–76%.⁹,¹⁰ Postcombustion Hg removal mainly achieves synergistic removal through pollution control methods.
control equipment such as dust removal and flue gas desulfurization and denitrification. In addition, adsorption technology (activated carbon adsorption,11 calcium-based adsorbent,12 and zeolite13) and corona discharge plasma technology14 have also been applied to remove Hg from flue gas. There are significant differences in Hg emissions among coal-fired power plants, depending on their specific fuel characteristics, existing technology, and operational conditions.15 Furthermore, technology to profitably reduce Hg emissions from coal-fired power plants is not yet commercially available for developing countries like China and India.16

Compared with the high-cost “end treatment” methods and low-efficiency coal preparation methods to remove Hg, coal pyrolysis is a simple and effective method for producing coal with little to no Hg, and this has attracted significant attention. Currently, several important aspects in the pyrolysis process have been explored, such as the Hg release behavior,17−20 effect of atmosphere on Hg removal,21 kinetic analysis,15 and volume reaction model.22 However, there is insufficient research on the mechanism of Hg removal, the applicable conditions of various coal grades, and the loss of calorific value. This study utilized eight types of coal from different coal fields in China to analyze the Hg species in different coal grades; the effect of temperature, particle size, and residence time on the Hg removal from coal; and the Hg removal mechanism during pyrolysis. It can provide a new reference for the research on Hg removal technology based on pyrolysis before coal combustion.

2. MATERIALS AND METHODS

2.1. Samples. Eight types of coal from different coal fields were used in this study. These coal fields are important coal production bases in China, and the coal can be used to verify the applicability of Hg removal by pyrolysis. Late Carboniferous to Early Permian (C2−P2), Late Permian, (P3), and Early to Middle Jurassic (J1−J2) are the three important coal-forming periods in China, and approximately 38.1, 7.5, and 39.6% of mineable coal was produced during these periods, respectively.23 In terms of coal grade, three grades of coal, namely, lignite, bituminous coal, and anthracite, are evaluated. These can be used to compare the difference in the Hg removal effect between different grades of coal. The coal sample information is shown in Table 1.

| name       | coal grade       | coal-forming period | Hg content (μg g−1) | S content (%) | owned coal field        |
|------------|------------------|---------------------|---------------------|--------------|-------------------------|
| HMM        | anthracite       | J1−J2               | 0.330               | 1.60         | Hami coal field, Xinjiang |
| WDM        | anthracite       | J1−J2               | 0.129               | 1.41         | Wuda coal field, Inner Mongolia |
| FSM        | bituminous coal  | C2−P1               | 0.927               | 2.10         | Pinghuo coal field, Shanxi Province |
| YCM        | bituminous coal  | C2−P1               | 0.353               | 1.23         | Yuncheng coal field, Shansi Province |
| PDSM       | bituminous coal  | C2−P1               | 0.240               | 0.89         | Pingdinghe coal field, Henan Province |
| FZM        | lignite          | P3                  | 0.067               | 0.29         | Fuzhou coal field, Fujian Province |
| ZXM        | lignite          | P3                  | 0.414               | 2.57         | Zhengxian coal field, Yunnan Province |
| HZM        | lignite          | P3                  | 0.341               | 0.79         | Hezhou coal field, Guizhou Province |

In this study, the pyrolysis experimental system was mainly composed of a nitrogen supply device (purity ≥ 99.999%), a tube pyrolysis furnace (SK-G04123K), a circulating water-cooling device, and an RA-915 mercury analyzer in series. The RA-915 mercury analyzer (produced by Lumex, Russia) combines Zeeman atomic absorption spectrometry and high-frequency modulation polarized light technology to achieve background correction. The response frequency of Hg signal is 1 s−1, and the detection limits of gas and solid are 0.2 ng m−3 and 0.5 ng g−1, respectively.35,36 The pyrolysis furnace has a self-programming function, and the pyrolysis range is 0−1200 °C with an accuracy of ±1 °C. Two heating modes were involved in the experiment: constant temperature pyrolysis and TPD.

Under constant temperature pyrolysis, the coal carried by quartz boats was pyrolyzed at different temperatures (100, 200, 300, 400, 500, 600, 700, and 800 °C) in N2 to analyze the effect of temperature on Hg removal. The fast pyrolysis experiment was conducted directly at 600 °C. The sample amount was 1.0000 g, and the pyrolysis was performed for 15 min. After the pyrolysis was completed, the sample was immediately transferred to a cooling zone and cooled to room temperature in N2 to be tested. The Hg removal rate (XHg) during coal pyrolysis, is calculated based on the following equation:

\[ X_{Hg} = \frac{H_{g_a} - H_{g_b} (1 - L)}{H_{g_a}} \]  

(1)

Where \( X_{Hg} \) = Hg removal rate, %; \( H_{g_a} \) = Hg concentration in raw coal, ng g−1; \( H_{g_b} \) = Hg concentration in coal after pyrolysis, ng g−1; and \( L \) = weight loss during pyrolysis.

The experimental parameters in the TPD experiment are as follows: pyrolysis range of 30−800 °C, heating rate of 10 °C/min−1, coal sample particle size of 80−100 mesh, a sample amount of 1 g, and a N2 purging speed 1.0 L/min−1. Details of the experiment have been reported in previous studies (Figure 1).28,35

2.2.2. Mercury Content, Sulfur Content, and Calorific Value of Coal. The Hg content of the coal samples was measured using an RA-915 mercury analyzer combined with a Pyro-915+ cracker instrument (produced by Lumex, Russia). The Hg
content results presented are inferred by average values of three duplicate runs, measuring at 0.03 g. The Hg recovery rate of the eight coal samples was tested to be 93–109%, with an average value of 103%, using the standard soil sample GSS-14 (0.089 μg g⁻¹ Hg content) from China.

The total sulfur content in the coal was tested using the Ashka method, according to ASTM (American Society of Testing Materials) D3177-02 and ASTM D423904a standards.

3. RESULTS AND DISCUSSION

3.1. Analysis of Mercury Species in Coal. Figure 2 shows the thermal decomposition curves of the eight coal samples in N₂ by TPD. Except for anthracite coals (HMM and WDM), all other samples showed multiple Hg signal peaks within 30–800 °C, showing evident stage release characteristics. The number and intensity of the Hg peaks indicated differences in Hg species and its content in coal from different regions. To clearly distinguish various Hg peaks in the Hg decomposition curves, overlapping peaks of the curves were deconvolved using Origin 8.0 software. Simultaneously, according to the signal—coverage area ratio of various peaks, the content of different Hg species was inferred (Figure 3).

Based on the deconvolution results, the positions and start-end ranges of Hg peaks presented by different coal samples have

![Figure 1. Schematic diagram of the pyrolysis experiment system.](image1)

![Figure 2. Thermal decomposition curves of mercury in coal](image2)
Figure 3. Content ratio of various mercury species in coal.

good correspondence and reproducibility (Table 2). The Hg peaks always appear near 230, 260, 310, 430, and 540 °C, and the

Table 2. Mercury Signal Peaks for the Coals

| sample name | high peak T (start of peak T−end of peak T) (°C) |
|-------------|-----------------------------------------------|
| HMM         | 319 (280–380) |
| WDM         | 307 (280–400) |
| PSM         | 262 (200–310) 310 (270–380) |
| YCM         | 252 (190–310) 301 (200–400) 439 (370–510) 546 (480–650) |
| PDSM        | 248 (200–300) 313 (250–350) 544 (480–580) |
| FZM         | 240 (200–300) 310 (250–380) |
| ZXM         | 230 (200–250) 306 (260–360) 431 (360–500) 520 (480–600) |
| HZM         | 227 (200–250) 316 (260–370) 524 (480–570) |

start-end ranges are 200–250, 200–310, 250–380, 360–510, and 480–600 °C, respectively. This highly reflects the thermal decomposition characteristics of Hg-OM (organic matter Hg), HgSe, HgS (red), HgO, and HgSO4 (Table 3). Based on the content of various Hg species, HgS occupies the most dominant type in coal, with an average proportion of 63% (27–100%). HgSe occupies a large proportion in bituminous coals (PSM, YM, and PDSM), approximately 41% (11–57%). HgSO4 accounts for 18–25% of the coals, and a small amount of Hg-OM (5–19%) and HgO (0–20%) are reflected in lignite coals (FZM, WXM, and HZM). Guo et al.38 conducted pyrolysis on different specific gravity components of bituminous coal under N2, and proposed that Hg released in 150–400, 500–600, and >750 °C were associated with carbonate minerals and organic materials, sulfide minerals, and aluminosilicate minerals, respectively. Rumayor et al.33 concluded that the main Hg species found in coal from Spain and Australia might be organic matters, pyrite-bound matters (Hg–FeS2), and HgS. Moreover, small amounts of HgO or HgSe may be present in some coal from this region. The results are similar to the results in this study.

Based on the Hg species and its proportions, Hg mainly exists in the form of HgS in coals from China but may coexist with HgSe, HgSO4, Hg-OM, and HgO. In terms of coal grade, only HgS is found in anthracite coals. Bituminous coals are mainly characterized by HgSe and HgS. HgS and HgSO4 are the main forms found in lignite coals, but a small amount of Hg-OM seems to be a typical criterion to identify this type of coal.

3.2. Analysis of Mercury Pyrolysis Removal from Coal.

3.2.1. Effect of Temperature on Mercury Removal. The trend of the $X_{Hg}$ from coal in a nitrogen environment with the temperature is shown in Figure 4. With an increase in the temperature, the $X_{Hg}$ from coal gradually increased and became stable. The Hg release is mainly effective in the range of 200–600 °C. When the temperature reaches 600 °C, the $X_{Hg}$ has reached its peak. The mean value of the correlation coefficient $R^2$ between the $X_{Hg}$ and temperature (T) is 0.82 (0.72–0.96), which indicates that the temperature has an important correlation with Hg removal from coal. The coal pyrolysis was discretely conducted at eight temperature ranges at intervals of 100 °C to calculate the Hg removal rate in each range (Figure 4). The correlation between the Hg removal rate and T in a specific temperature range is extremely poor, with an average $R^2$ value of 0.11 (0.06–0.15). Sotiropoulou et al.15 simulated coal Hg removal during pyrolysis using a first-order reaction rate model and calculated the apparent activation energy of low-temperature (200–300 °C), mild-temperature (300–600 °C), and high-temperature (700–900 °C) regimes as ~3.1, ~6.1, and ~22.4 kJ mol$^{-1}$, respectively. It was explained that rising temperatures contributed to the increase in activation energy; however, it could not provide a strong explanation for the behavior of Hg removal through the simulation results.

The trends of the Hg removal rate in various temperature ranges for HMM and WDM coals are unimodal, and YCM, PDSM, ZXM, and HZM coals are bimodal. The trends are very similar to the thermal decomposition curves of Hg (Figure 2). This indicates that the Hg species in coal and its thermal decomposition characteristics can support the understanding of the Hg release behavior during pyrolysis; Hg will not be removed until the temperature reaches the decomposition temperature of the Hg species itself. When the Hg in coal exists as a single Hg species, the thermal decomposition intensity shows a Gaussian distribution. When multiple Hg species coexist, the stage release characteristics are performed due to the cross decomposition of them. Furthermore, the activity of Hg release at a specific temperature does not necessarily show an increase with the increasing temperature but essentially depends on the species of mercury present.

To improve the understanding of the release behavior of Hg from coal, thermodynamic equilibrium models were used to predict the composition of the chemical species in the gas phase using HSC-Chemistry 6.0 software. Based on the main Hg
species found in the coal, the release process in N₂ is simulated as follows:

\[ \text{HgS} \rightarrow S + \text{Hg(g)} \]  
\[ \text{HgSe} \rightarrow \text{Se} + \text{Hg(g)} \]  
\[ \text{HgO} \rightarrow \text{HgO(g)} \rightarrow \text{Hg(g)} + \text{O}_2 \]  
\[ \text{HgSO}_4 \rightarrow \text{SO}_2(g) + \text{HgO(g)} \]

3.2.2. Effect of Coal Particle Size on Mercury Removal.
Iwashita et al.²⁰ pyrolyzed three different particle sizes of coal: <1 mm, <0.149 mm, and <0.074 mm for 1 h at 300 °C. There was no significant difference in the Hg removal rate among them; consequently, they believed that the Hg removal efficiency had no relation with the particle size. However, a pyrolysis time of 1 h is too long to be suitable for industrial production, and the efficiency difference in the active period of Hg release with different coal sizes may be covered up. Figure 5 shows the Hg removal from coal particles of three different sizes: 80–100 mesh (0.15–0.18 mm), 40–60 mesh (0.25–0.42 mm), and 8–18 mesh (1–2.36 mm) at different temperatures after pyrolysis for 15 min. Comparing the two particle sizes of 80–100 and 40–60 mesh, the deviation of the X_Hg at different temperatures does not exceed 3%. It can be considered that when the coal is in the range of 40–100 mesh, the influence of the particle size on the Hg removal efficiency can be excluded, and this is consistent with Iwashita et al.’s conclusion. However, when compared with these two smaller sizes, the Hg removal efficiency in coal of 8–18 mesh is relatively backward. In addition, the gap is most evident in samples of YCM and PDSM coals. This may be because the release of Hg requires pore channels, and the compact structure of large coal particles will limit the escape of Hg. Taking HMM and WXD coals as examples, the results are quite distinctive. Although the Hg removal efficiency in larger particle coal is significantly reduced at 100–400 °C, Hg is largely removed at 600 °C, reaching a level equivalent to fine coal. This may be related to the Hg species present in the coal. The Hg in HMM and WXD coals exists only in the form of HgS, which is easily removed at lower temperatures. Excessive particle size limits the Hg release efficiency, but it can slowly release Hg over time.

Overall, excessive coal particle size will influence Hg removal during pyrolysis. Pulverized coal smaller than 40 mesh is more conducive to the rapid removal of Hg.

3.2.3. Rapid Pyrolysis for Mercury Removal (600 °C).
3.2.3.1. Effect of Residence Time on Mercury Removal. Figure 6 shows the Hg removal effect at different residence times. This has a similar trend in the eight different coals; with the residence
time prolonged, the $X_{\text{Hg}}$ in the initial stage of pyrolysis increases rapidly then slows down and gradually approaches the limit value. From the perspective of removal efficiency, FZM, WDM, and HMM coals achieved Hg removal earlier than the other coal samples. This may depend on the combined effects of Hg species and total Hg content in coal. The order of the total Hg content of the eight coal samples is as follows: FZM (0.067 μg g$^{-1}$) < WDM (0.129 μg g$^{-1}$) < PDSM (0.240 μg g$^{-1}$) < HMM (0.330 μg g$^{-1}$) < HZM (0.341 μg g$^{-1}$) < YCM (0.353 μg g$^{-1}$) < ZXM (0.414 μg g$^{-1}$) < PSM (0.927 μg g$^{-1}$). In addition, the decomposition order of the Hg species in coal during pyrolysis is Hg-OM < HgSe < HgS < HgO < HgSO$_4$. These three coals (FZM, WDM,
and HMM) have relatively weak thermally stable Hg species (HgS and Hg-OM) and lower Hg content. For the other samples, when $t = 3$ min, the $X_{Hg}$ from coal is relatively low at only 32−58%, which may be caused by the residence time being too short to allow sufficient release of the Hg. Within 10−15 min, the Hg removal curve is basically stable, with a growth rate of less than 7%.

Based on the pyrolysis residence time, under the reactor conditions of this study, reaching the Hg removal rate limit can be satisfactorily achieved for different types of coal by retaining residence time for 10−15 min. Coals with a low Hg content and the presence of readily thermally decomposable Hg species have short residence times.

3.2.3.2. Calorific Value Loss and Synergistic Removal of S−Hg during Pyrolysis. The calorific value of coal is an important control indicator for measuring fuel costs and improving the competitive advantage of power plants. The coal calorific value depends on its carbon content and is a competitive advantage of various types of coal by retaining calorific value is an important control indicator for measuring fuel costs and improving the competitive advantage of power plants. The coal calorific value depends on its carbon content and is affected by moisture and ash. Every 1% increase in moisture or ash will reduce the calorific value by ~260 J/g and ~272 kJ/kg, respectively. In addition, volatile matter is the product of the thermal decomposition of organic matter in coal, and its content is directly proportional to the calorific value of coal.41

Figure 7 shows the calorific value of the coal before and after pyrolysis. After pyrolysis in nitrogen, the calorific value loss rate of the eight coal samples was between 2 and 12%, with an average of 7%. The heat loss was an integrated result of multiple factors, such as the release of volatiles and bound water and energy changes caused by chemical reactions. Based on the test data, the heat retention results were acceptable. This shows that the rapid pyrolysis of coal at 600 °C under the protection of an inert atmosphere can not only effectively and quickly remove Hg from coal but also retain the calorific value.

Furthermore, the synergistic desulfurization effect was also shown during the Hg pyrolysis removal process. Sulfur in coal can be divided into inorganic sulfur (e.g., pyrite sulfur and sulfate sulfur) and organic sulfur (fatty sulfur, aromatic sulfur, and thiophene sulfur). Sulfate is chemically stable and less in coal. Pyrite sulfur is dispersed in the form of crystals in the voids or fissures inside the coal, and it will decompose above 500 °C; FeS $\rightarrow$ FeS + S.32 For organic sulfur, fatty sulfur is the most unstable and will decompose at lower temperatures, aromatic sulfur is second, and thiophene sulfur is the most stable and difficult to remove in low−medium temperature pyrolysis.43,44 From the experimental data, in rapid pyrolysis mode (600 °C), the removal rate of sulfur in the eight coal samples is 17−58%, with an average of 33%. This may be related to the decomposition of pyrite sulfur and organic sulfur components with less thermal stability.

It is generally believed that pyrite in coal is the main carrier of HgS.33,34 Cao et al.38 used time-of-flight secondary ion mass spectrometry (TOF-SIMS) to analyze natural pyrite, and they pointed out that the weathering of pyrite will cause the conversion of HgS to HgSO4. Therefore, the reason for the synergistic removal effect of S and Hg during pyrolysis is probably related to the decomposition of pyrite and its less thermally stable organic components.

4. CONCLUSIONS

Through the pyrolysis analysis of eight types of coal from China in nitrogen, comparatively common results for various types of coal were obtained. Temperature has a significant effect on the Hg removal from coal; however, it has no significant correlation with the Hg release activity at specific temperatures. Pulverized coal smaller than 40 mesh is more favorable to the rapid removal of Hg. For most coal, 10−15 min of pyrolysis can achieve the ultimate Hg removal effect. When the temperature reaches 600 °C, the Hg removal rate can reach 88−100%. HgS is the main form of Hg in Chinese coal and may coexist with HgSe, HgSO4, Hg-OM, and HgO in certain regions. Due to the thermal characteristics of a single Hg species and the cross decomposition of multiple Hg species, the Hg removal from coal shows a stage release characteristic with the increasing temperature. The species and content of Hg in coal are the essential reason for
determining the release characteristics and removal efficiency of Hg through pyrolysis.

Rapid pyrolysis at 600 °C in nitrogen is feasible to remove Hg for different grades of coal. Notably, Hg removal not only achieved the desired effect but also retained the calorific value of coal (the calorific value loss rate was approximately 2–12%), and there was a certain degree of synergistic desulfurization effect in the process. The industrial application based on pyrolysis-based mercury removal is worthy of continued exploration.

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Notes
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