Geochemistry of Mercury in the Permian Tectonically Deformed Coals from Peigou Mine, Xinmi Coalfield, China

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Abstract: As the mercury emitted from coal combustion can lead to serious environmental issues, researchers pay more attention to the content, distribution and occurrence of mercury in coal. In this paper, the content, distribution, and occurrence of mercury in the Permian tectonically deformed coals from Peigou Mine, Xinmi coalfield, Henan Province were investigated. A total of 18 bench samples were taken from No.2-1 coals seam in Peigou Mine, including 15 coal bench samples, two roofs and one floor. The mercury concentration, mineral composition, and main inorganic element content of 18 samples were determined by DMA-80 direct mercury analyzer, XRD, and XRF respectively. The results show that the mercury content ranges from 0.047 ppm to 0.643 ppm, with an average of 0.244 ppm. Though the coal seam has turned into typical tectonically deformed coal by the strong tectonic destruction and plastic deformation, the vertical distribution of mercury has remarkable heterogeneity in coal seam section. By the analysis of correlation between mercury and the main inorganic elements and the mineral composition in coal, we infer that majority of mercury mainly relates to pyrite or kaolinite.

Key words: mercury in coal, minerals in coal, tectonically deformed coal, modes of occurrence

1 Introduction

Mercury is one of the most harmful volatile trace elements in coal. The total mercury capacity of global emissions is 1960 tons due to human activity in 2010, 24% of which comes from coal-fire (Pirrone et al., 2010), so mercury has been defined as a priority control pollutants by China and the United Nations environment program (UNEP), the world health organization (WHO), the European Union, and the United States environmental protection agency (EPA). In 2005, United States have formulated relevant laws and regulations to limit the mercury content ranges from 0.047 ppm to 0.643 ppm, with an average of 0.244 ppm. Though the coal seam has turned into typical tectonically deformed coal by the strong tectonic destruction and plastic deformation, the vertical distribution of mercury has remarkable heterogeneity in coal seam section. By the analysis of correlation between mercury and the main inorganic elements and the mineral composition in coal, we infer that majority of mercury mainly relates to pyrite or kaolinite.

Mercury pollution caused by coal-fire has become non-point source pollution in 1970s, and the mercury from coal combustion release has become the main source of mercury pollution in China (Streets et al., 2011; Liang Yanci et al., 2016).

The average content of mercury in the earth’s crust is 0.103 ppm (Li Tong, 1992), the mercury concentration in coal from North China ranges 0.01–0.5 ppm, with an average of 0.17 ppm (Tang Xiu et al., 2004). The geometric mean content of mercury in main coal basins of China based on coal resources distribution is 0.579 ppm, which is higher than the average of American coal and the world coal (Ren Deyi et al., 2006). The occurrence of mercury in coal determines its volatilization, migration and transformation behavior in combustion and other utilization process, so it is necessary to investigate the content and occurrence of mercury in environment assessment and pollution of mercury in coal. Compare with the main inorganic elements such as aluminium, silicon, et al., the concentration of mercury in coal is extremely low, and its concentration and occurrence varies

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greatly in different regions or coal basins, so many research have been done about it’s concentration and occurrence in coal. Some scholars found mercury may exist in coal in pyrite (Feng et al., 1999; Swaine, 2000; Diehl et al., 2004). Some indirect experiments show that mercury exist in coal in clay mineral or organic component (Wang Qichao et al., 2001; Zhou Yiping, 1994).

Coal resources is abundant in North China, the coal production in this region accounts for roughly 40% of the national output. Tang Xiuyi, Ren Deyi, Dai Shifeng, Huang Wenhui, Liu Dameng, Liu Guijian, Wang Wenfeng, and et al. had investigated the concentration, distribution, and occurrence of mercury and other harmful trace elements in coal in North China coal basin. The results showed that even in North China coal basin, the content and occurrence of mercury in coal varied in different coalfield or mine (Tang Xiuyi et al., 2004; Ren Deyi et al., 2006; Dai Shifeng et al., 2003; Huang Wenhui and Yang Yichun, 2002; Liu Dameng et al., 2000; Liu Guijiang et al., 2003; Wang et al., 2005; Wang T., et al., 2008; Song et al., 2007; Wang et al., 2004; Yang, 2011; Yang et al., 2015; Yang et al., 2016; Zhao et al., 2015; Zheng et al., 2016). In recent years, the coal production is about 150 million tons per year in Henan province, and most of the coal is typical tectonically deformed coal. Few research have been done on the geochemistry of mercury in Permian coal, Henan Province, especially in deformed coal caused by intense tectonic movement and plastic deformation, so in this paper, the content, distribution, and occurrence of mercury in Permian No.2–1 coal seam, Peigou Mine, Xinmi coalfield have been investigated, based on channel sampling, mercury determination, XRD, and XRF test. The article also discussed the influence of deformation of coal seam on the mercury distribution.

2 Geologic Setting

Peigou Mine is located in Xinmi coalfield in Henan Province, North China (Fig. 1). The strata in the Peigou Mine include the Ordovician Majiagou Formation (O2m), the Late Carboniferous Benxi Formation (C3b) and Taiyuan Formation (P1t) (underpart), the Early Permian Taiyuan Formation (P1t) (upper part), Shanxi Formation (P2sh), Lower Shihezi Formation (P2x), Upper Shihezi Formation (P2sh), Sunjiagou Formation (P3y), and Quaternary. There are six coal groups from Taiyuan Formation to Lower Shihezi Formation, and No.2–1 Coal of Shanxi Formation is the major minable coal bed in the Xinmin coalfield, with a thickness that varies from 1.14 to 25.86 m (7.16 m on average) (Fig. 2). The No.2–1 coal seam is typical tectonically deformed coal caused by tectonic movement since Mesozoic era.

The tectonically deformed coal widespread distributes in North China basin, and mortar and mylonitic texture coal mainly distribute in the southern margin of the North China plate, the western margin of Ordos basin, and the Eastern side of Taihang Mountains, where underwent strong tectonic movement (Fig. 1a). Xinmi coalfield is located in the south of North China plate. It belongs to the northeast side of Mianchi-Yima-Yiyang-Lushan-Wuyang thrust nappe tectonic belt in north margin of Qinling orogenic, and controlled by Qinling-Dabie mountain orogenic belt (Fig. 1a). After late Paleozoic coal accumulating period, the tectonic evolution of Xinmi coalfield can be divided into two stages, Mesozoic extrusion and Cenozoic extension. In Mesozoic extrusion stage, NW fold and NNE-NE thrust nappe were formed, which is the main regional tectonic pattern (Cao Daiyong et al., 1994, 2002; Zhang Nianmao et al., 1988). In Cenozoic extensional stage, stretching structure marked by development of tilted fault block and gravity gliding tectonic along weak layer in stratum were formed (Wang Enyng et al., 2010). The No.2-1 coals seam in the bottom is main sliding surface of gravity sliding structure, which has the characteristics of multi-level and multi-period sliding (Hou et al., 2012; Cao et al., 2000; Lei et al., 2010.). Peigou Mine was mainly controlled by Yangjiawa fault and Fushanzi fault. Most faults in mining area are EW normal faults with high angle fault plane, which is high in south and low in north (Fig. 1b). The No.2–1 coal seam of Peigou Mine is a typical “three soft” coal seam (roof, floor and coal seam are broken and the combination) (Fig. 1c). Faults and gravity sliding structures developed strongly, which led to strong rheological deformation and brittle fracture No.2-1 coal seam. For instance, the thickness of No.2–1 coal seam ranges 1.14 to 25.86 meters with sudden thickening and thinning phenomenon. The mylonitic texture coal accounts for more than 65% in Peigou Mine.

3 Sample Collection and Test

3.1 Sample collection and processing

A total of eighteen samples were collected from No.2–1 coal seam, Peigou Mine, following Chinese Standard Method GB/T482—2008, including one floor (No.0), fifteen coal benches (No.1–No.15), and two roofs (No.16 and No.17) (Fig. 2). The original bedding structure of the No.2–1 coal seam was destroyed completely, and the coal with primary structure turned into tectonically deformed coal of type IV—mylonitized coal (Fig. 3). All samples were collected from the heading end of the coal roadway, because the coal roadway walls were shotcreted and bolt
supported. It is impossible to distinguish the lithotype or recognize the bedding in the section for the mylonitized coal, the thickness of each bench is about 18–22 cm, and weight about 1.5 kg. Each bench sample was kept in the sealed plastic bag, avoiding weathering or polluted.

Twenty grams is enough for each sample, for mercury concentration determination, proximate analysis, mineral and inorganic elements composition analysis, and the distribution of mercury in coal seam is heterogeneous, so the picking and crushing of samples for the following test impact influence the test results greatly. Testing samples was cut over an area 1 cm wide and 1 cm deep from the big block sample, and were crushed and ground to pass 75 µm for major and trace element analysis and other tests.

3.2 Experiment and test

Proximate analysis was conducted using national standard GB/T212–2008. The total sulfur was determined following national standard GB/T214–2007.

In order to determine the minor inorganic elements in coal, samples were ashed completely under 815°C following national standard GB/T30725–2014. X-ray fluorescence spectrometry (ARL Quant'X) was used to determine the oxides of major elements in the coal ash, including Al₂O₃, SiO₂, Fe₂O₃, K₂O, CaO, Na₂O, MgO, P₂O₅, TiO₂, and SO₃.

The main minerals in coal samples were identified by powder X-ray diffraction (XRD). Because the content of most minerals is low relatively, low-temperature ashing of
the coal samples was performed on a tube furnace in 100% oxygen and less than 370°C condition, in order to recognize and quantify the minor minerals. Most minerals do not change in low-temperature ashing process (Finkelman et al., 1990). XRD analysis of the low-temperature ashes (LTAs) and the parting samples was performed on a Bruker D8 Advance powder diffractometer with graphite monochromator filtered Cu-Kα radiation and a scintillation detector. Each XRD pattern was recorded over a 2θ interval of 5°–80°, with a step size of 0.02° and a scanning speed of 1 s/step by step scanning mode. X-ray diffractograms of the LTAs were subjected to quantitative mineralogical analysis using RockJock software, which is a free Program for determining quantitative mineralogy from power X-ray diffraction data developed by Eberl (Omotoso et al., 2009) based on the principles for diffractogram profiling set out by Rietveld (Rietveld, 1969). The minerals in coal particle were also observed and identified with FEI field emission scanning electric microscope-energy dispersive X-ray (FSEM-EDX).

The national standard method for mercury determination includes cold-vapor atomic absorption spectrophotometry and atomic fluorescence absorption spectrophotometry. Both methods require sample digestion and further wet chemistry, and sample digestion can be problematic because of mercury volatility. A direct mercury analyzer (Milestone DMA-80) was used for mercury determination following the ASTM method D6722-11. DMA-80 is a direct mercury analyzer, which uses the principle of thermal decomposition, amalgamation and atomic absorption.

Samples are automatic dried in oxygen flow, and then thermally decomposed in an oxygen-rich furnace. A continuous flow of oxygen carries the decomposition products through a catalyst bed where interferences are trapped. All mercury species are reduced to elemental Hg and are then carried along to a gold amalgamator where the mercury is selectively trapped. The system is purged and the amalgamator is subsequently heated which releases all mercury vapors to the single beam, fixed wavelength atomic absorption spectrophotometer.

![Stratigraphic column of Peigou Mine and profile samples in No.2-1 coal seams.](image-url)
Absorbance measured at 253.7 nm is proportional to mercury content in the sample. The DMA-80 can analyze solid samples in 5 minutes with mercury concentrations ranging from 5 ppb to over 25 ppm and does not require any sample preparation. The DMA-80 is fully compliant with US EPA method 7473 and with ASTM method D-6722-11.

0.0500 gram sample is weighed into a quartz boat, which is heated in muffle furnace under 650°C for 30 min to eliminate the background influence, and the sample weight is transferred from the analytical balance to the DMA-80. Each sample was determined for three times, and the average value was used.

4 Results and Discussion

4.1 Coal chemistry and texture

Table 1 summarizes the proximate analyses and total sulfur data for the 18 coal benches from Peigou Mine. The ash yield of No.3 and No.4 samples exceeds the average value of the whole coal seam greatly, partly because of the thin-bedded partings. According to the national standard of coal classification and coal quality classification, the No. 2–1 coal is low-medium ash yield and ultra-low sulfur content meagre coal.

Observing the texture of the massive coal sample, we can find that the primary bedding structure was destroyed, it is very hard to distinguish the micro-lithotype. The micro-bedding deformed and broke greatly, and schistose, wrinkle structure formed. Many sliding structure planes appeared in the coal seam (Fig. 3). The hardness of coal samples decreased significantly, and can be broken into detritus with hands.

4.2 Minerals and main inorganic elements in the No. 2–1 coal

4.2.1 Minerals

The proportion of each crystalline phase identified from

<table>
<thead>
<tr>
<th>Sample</th>
<th>M afl (%)</th>
<th>Afl (%)</th>
<th>Vdaf (%)</th>
<th>Sdaf (%)</th>
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<td>17</td>
<td>1.52</td>
<td>92.35</td>
<td>17.00</td>
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<td>16</td>
<td>1.62</td>
<td>89.72</td>
<td>16.75</td>
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<td>15</td>
<td>0.91</td>
<td>17.74</td>
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<td>14</td>
<td>1.12</td>
<td>10.77</td>
<td>13.63</td>
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<td>13</td>
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<td>13.17</td>
<td>12.93</td>
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<td>4.62</td>
<td>13.32</td>
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<tr>
<td>11</td>
<td>1.12</td>
<td>8.72</td>
<td>12.93</td>
<td>0.29</td>
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<tr>
<td>10</td>
<td>1.10</td>
<td>14.13</td>
<td>12.04</td>
<td>0.59</td>
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<tr>
<td>9</td>
<td>1.17</td>
<td>5.83</td>
<td>12.52</td>
<td>0.33</td>
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<tr>
<td>8</td>
<td>1.13</td>
<td>5.79</td>
<td>12.54</td>
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<td>7</td>
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<td>6.92</td>
<td>13.22</td>
<td>0.31</td>
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<td>1.23</td>
<td>8.52</td>
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<tr>
<td>0</td>
<td>1.25</td>
<td>84.34</td>
<td>18.78</td>
<td>0.44</td>
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the diffractograms of the LTAs, floor and roof is given in Table 2. The percentages of the main inorganic elements oxides (SiO$_2$, Al$_2$O$_3$) calculated from the XRD patterns of LTAs were roughly compared to the XRF results.

The minerals in bench samples include clay minerals such as kaolinite illite, and smectite, and non-clay minerals such as quartz, calcite, ankerite, tobelite, anatase, and magnetite (Table 2). Although the coal seam deposited successively in a relative short geological period, the mineral constitution of different benches varied greatly. Based on their XRD patterns, all bench samples can be divided into three groups. Group 1 consists of No.0, No.6, No.8, No.16, and No.17 bench samples, their main minerals includes quartz, kaolinite and illite (Fig. 4a). Group 2 consists of No.1, No.2, No.3, No.11, No.12, No.13, No.14, and No.15 bench samples, their main mineral includes kaolinite, illite and quartz, calcite only appears in No.3 samples (Fig. 4b). Group 3 consists of No.4, No.5, No.7, No.9, and No.10 bench samples, their main mineral includes illite, kaolinite, smectite, and tobelite(Fig. 4c).

### 4.2.2 Main inorganic elements

The abundances of main inorganic element oxides in the floor, coal bench samples, and proofs are listed in Table 3. Chemical analysis data for the samples were recalculated to provide normalized percentages of the main element oxides in the inorganic fraction. The chemical composition result was derived from high temperature ash. The LTA yield of the bench samples is higher than the high temperature ash yield from the corresponding sample (Fig. 5). The difference is partly due to dehydration of the clay minerals, and carbon dioxide release from the carbonate minerals during the high temperature ashing process.

### 4.3 Mercury content in coal seam

The mercury concentrations of all bench samples of Peigou Mine are shown in table 3. In order to evaluate of mercury content in coal seam, table 4 also lists the average concentration of America coal, Greece coal, Australia coals, as well as other region coals in China. The mercury concentrations of Peigou coal (including the roof and floor) range from 0.047 to 0.643 ppm, with an average value of 0.244 ppm, which is higher slightly than that of...
Fig. 4. XRD patterns and mineral composition of floor, LTAs, and roofs.
North China Permian coal, China coal, America coal, Australia coal and Greece coal. Compare with coals from other regions in China, the average mercury concentration of Peigou Mine exceeded slightly the mercury concentration of Southwest of Guizhou and Yunnan Province coals (Zhang Junying et al., 1999; Li Dahua et al., 2006), and is similar with Huaibei coal (Zheng Liugen et al., 2007). The concentration coefficient (CC, mercury content in the Peigou coals vs. mercury content in the world hard coal) is 2.4 (Yudovich et al., 2005), which indicate mercury enriched in No.2–1 coal, Peigou Mine.

4.4 Distribution of mercury in coal seam

The histogram of mercury content in floor, coal benches and roofs reflects the variation of mercury in the coal seam and adjacent strata (Fig. 6). There is no significant difference between coal seam and floor, roofs in mercury content, the mercury in floor slightly higher than the average content of coal seam, and the mercury in roofs slightly lower than the average content of coal seam. In the coal seam, the minimum mercury content is only 0.065 ppm (No.9) and the maximum content is 0.64 ppm (No.10), the difference up to 10 times. According to the variation of mercury content in vertical section, all samples were divided into three groups: group 1 (No.0–No.9), group 2 (No.10), and group 3 (No.11–No.17). Group 2 (No.10) locates in middle part of the coal seam and with the maximum mercury content. Mercury content in group 1 and group 3 show the variation — decrease from middle to the top and bottom. The mercury variation in floor, coal benches and roof indicates that the mercury distribution in coal seam is relatively stable, for example No.9 sample with the minimum mercury content close to No.10 sample with the maximum mercury content. The geologic processes after coal seam formation didn’t affect mercury distribution greatly in coal section. Although the coal seam has been damaged by brittle deformation and ductile rheology, the mercury distribution in coal seam is inhomogeneity.

4.5 Occurrence of Mercury

The trace element occurrence in coals from different coal basins or coal-forming periods is different, so it is difficult to confirm the mercury occurrence in difference coal samples. The determinations of trace elements occurrence in coal include sequential chemical extraction, scanning electron microscopy with energy dispersive X-Ray Analysis (SEM-EDX), float-sink test, and correlational analysis method (Wang W.F., et al., 2008; Zhang et al., 2015). Finkelman determined the trace elements occurrence in Illinois (United States) coals by float-sink test, and found the mercury exists in inorganic minerals, such as sulfide (sphalerite) or selenide (Finkelman, 1981). Vassilev found that most mercury in Donets Basin coals occurred in pyrite in solid solution (Vassilev, et al., 2001). Junying Zhang and Deyi Ren

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<tr>
<td>Mercury concentration</td>
<td>0.244</td>
<td>0.172</td>
<td>0.19</td>
<td>0.263</td>
<td>0.17</td>
<td>0.10</td>
<td>0.10</td>
<td>0.20</td>
<td>0.17</td>
<td>0.10</td>
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Fig. 5. Relationship between high-temperature ash (A₉; 815°C) and low temperature ash (LTA; 370°C).

Fig. 6. Vertical variation of Hg content in the floor, coal seam and roofs of Peigou Mine.
studied the distribution characteristics of mercury in Southwest of Guizhou coals and found that the clay mineral and hydrothermal calcite were the main carrier of the mercury (Zhang Junying et al., 1999). In this paper, the mercury occurrence was studied by correlational analysis between mercury content and ash yield, relative proportion of minerals, inorganic elements content.

Fig. 7 is the scatter diagram between mercury content and LTAs. It is hard to find the relationship between mercury content and LTAs if all the samples were all considered. If the floor (No.0), roof (No.16 and No.17), outliers (No.10 with the highest mercury content, No.4 with the highest ash yield) are not considered, we will find with the increase of LTAs the mercury content also increase.

In order to evaluate the relationships between mercury and different minerals, correlation analysis between mercury content and mineral percentages such as kaolinite, illite, smectite, total clay minerals, quartz and calcite was shown in the Fig. 8. Except floor (No.0), roof (No.16, No.17), and No.10 (triangle in Fig. 8), mercury in the Peigou coals is positively correlated with kaolinite, indicating a possible kaolinite affinity, and there is no obvious correlation between mercury content and other minerals, such as quartz, calcite, smectite, and illite.

Besides kaolinite, quartz, calcite, and illite, there are also some other minor minerals in floor, coal benches and

![Fig. 7](image-url)  ![Fig. 8](image-url)
roofs. But it is very difficult to identify the minor minerals whose percentage less than 1% or were decomposed in low temperature ashing process. For example, the iron oxides in some high temperature ash samples exceed 3.0%, but only magnetite was identified in some LTAs by XRD. In order to understand the affinity of mercury with inorganic parts in coal, the relationship between mercury and inorganic elements was analyzed by correlation analysis.

Table 5 shows the correlation between mercury and ten inorganic elements such as calcium, silicon, etc. Only the correlation coefficient of sulfur, iron and potassium with mercury is higher than 0.50, their correlation coefficients were 0.70, 0.63 and 0.64, respectively. The potassium mainly exists in illite or is adsorbed in clay minerals, and its content is maximum in floor, No.4 coal, and roofs, in which the mercury content is the minimum, so most mercury maybe exists in pyrite. Pyrite was not identified in LTAs by XRD, the possible reasons are: (1) the pyrite content is too low to be identified by XRD, (2) the pyrite was decomposed to iron and sulfur oxides by oxidation in low temperature ashing process. A few pyrite mineral particles were also be identified by FSEM-EDX (Fig. 9).

5 Conclusions

(1) The mercury content of No.2–1 coal, Peigou Mine ranges from 0.047 to 0.643 ppm, with an average of 0.244 ppm, which is higher than that of north China Permian coal, China coal, United State coal, Australia coal and Greece coal. The mercury enriched slightly in No.2–1 coal Peigou Mine.

(2) The mercury content in floor, coal benches, and roof varies greatly, and shows two circles in vertical section increasing at first and then decreasing. The No.10 sample with the maximum mercury content locates in the middle of the coal seam. There are evidences of volcanic activity, magmatic fluid or other geological process causing extreme enrichment of mercury. The formation of tectonic deformed coal did not cause the local enrichment or redistribution of mercury in coal seam.

(3) The correlation analysis between mercury content and ash yield, minerals percentage, main inorganic elements content showed that most mercury may exist in inorganic part in coal, and was possibly associated mainly with kaolinite and pyrite.

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