Original Research

Pollution Assessment and Source Apportionment of Heavy Metals in Sediments of Subsidence Section of Luling Mine in the Tuohe River, China

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Abstract

It is crucial to clarify the concentrations and sources of heavy metals in sediment under the influence of coal mining activities. This study surveyed heavy metals (V, Mn, Ni, Zn, Cu, Pb, Cr, and As) in sediments from the mine subsidence section of the Tuohe River in the Huaibei coalfield of China. The Enrichment Factor (*EF*) method and the Nemerow Comprehensive Pollution Index (*NCPI*) method were applied to evaluate the contamination status of heavy metals in sediments. Principal Component Analysis (PCA) and the Positive Matrix Factorization (PMF) model were used to apportion the sources of heavy metals. The results showed that the mean contents of V, Mn, Ni, Zn, Cu, Pb, Cr, and As were 0.93, 1.21, 1.20, 1.06, 1.15, 0.80, 1.18, 1.78 times higher than the background values, respectively. *EF* results showed that the pollution levels of Cr and As were higher than other heavy metals in the study area. *NCPI* results showed that the sites S1-S7 and S10-S11 of the subsidence section were slightly polluted while the sites S8-S9 of the non-subsidence section were moderately polluted. Based on PCA and PMF, three sources were identified: mining source (37.2%), agricultural source (34.8%), and natural source (28.0%). This study provides further insights into the heavy metal pollution treatment of aquatic ecosystems in mining areas.

Keywords: mining subsidence area, sediment, heavy metals, pollution assessment, source analysis

Introduction

In recent years, due to coal mining, large area surface subsidence occurred and then caused the upward seepage of groundwater, forming a special terrestrial aquatic ecosystem, i.e. coal subsidence pool [1, 2]. The structures and compositions of sediments in subsidence pools not only have a significant impact on substance-recycling and energy-flowing in water-sediment systems, but also where the continuously accumulating heavy metals can cause many pollution problems in coal areas. The sediments have the dual

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role of "sink" and "source" for heavy metals in aquatic ecosystems [3]. After heavy metals enter the catchment, they accumulate in sediments through precipitation and flocculation, increasing the concentrations of heavy metals in sediments [4]. When the physical and chemical conditions change (pH, temperature, flow, etc.), the heavy metals in the sediments re-enter the overlying water, causing endogenous pollution and seriously affecting the safety of the water environment [5, 6]. When heavy metals are over-accumulated in sediment, the aquatic ecosystem loses its self-purification capacity [7, 8]. Due to these risks to human health and ecological well-being, the heavy metal pollution in sediments of mining subsidence areas has become a significant environmental hazard, and, thus should be excessively focused.

After the earth's surface collapsed due to coal mining, the accumulation of heavy metals in sediments of subsidence areas was affected by aboriginal soil parent material, the discharge of domestic sewage, and runoff of irrigation water associated with chemical fertilizers and pesticides in the surrounding farmland [9]. Therefore, heavy metal sources in the subsidence area sediments had multi-source characteristics [10]. Some researchers have focused on the pollution status of heavy metals in the water column. After measuring and analyzing the pollution of water and sediment in the coal mining area, these studies evaluated the environmental quality and heavy metal migration ability [11]. Through the determination and evaluation of heavy metal samples in the sediments of subsidence ponds in the Huainan coal mining area, the results showed that the sediment polluted by heavy metals posed a potential threat to the surrounding ecosystem [12]. The evaluation of the water environment quality of the subsidence pond in the Sudong mining area showed the main ions had a cumulative effect and stability along with the increase of subsidence time and were less affected by the environment [13]. Therefore, the heavy metal pollution in subsidence area pools became the study emphasis. However, most studies mainly discussed the subsidence area as a whole and enclosed aquatic ecosystem [14, 15], and few studies focused on the evaluation and source analysis of heavy metal pollution in the sediment of coal subsidence pools connected to rivers.

In this study, the subsidence section of Luling Mine in the Tuohe River, China was taken as the research object. Sediment samples were collected, and eight heavy metals (V, Mn, Ni, Zn, Cu, Pb, Cr, and As) in sediments were determined. Based on the Enrichment Factor method (*EF*) and the Nemerow Comprehensive Pollution Index method (*NCPI*) were used to evaluate the pollution degree of single metal elements and the comprehensive pollution level of these eight heavy metals, respectively. By using Principal Component Analysis (PCA) and Positive Matrix Factorization model (PMF), the main potential sources of heavy metals in the subsidence area were discerned. The study can provide a reference for the treatment of heavy metal pollution in the mining subsidence area and provide data support for the ecological protection of the surrounding environment.

Materials and Methods

Study Area

The subsidence section of Luling Mine in the Tuohe River, with a water depth of 1.5 to 2.0 meters and a water surface width of 20 to 60 meters, is close to the coal mine and railway line of coal transport, surrounded by a large number of farmlands (Fig. 1). During the mining process of Luling Coal Mine, heavy metals were transferred into sediments of the subsidence section along with suspended coal solids. When encountering the scouring of rainwater and surface runoff, the heavy metals from chemical fertilizers remained in surrounding farmlands gradually accumulated in sediments [16] (Fig. 2). Due to the interconnection of water areas between this mining subsidence and the Tuohe River, the distribution of heavy metals in sediment was also probably affected by the discharge of domestic sewage and industrial wastewater from Suzhou City.

Sample Collection

The sediment samples were collected in April 2011 in the subsidence section of Luling Mine in the Tuohe River with latitudes between 33°34'6.72" N and 33°31'23.64"N and longitudes between 117°5'29.58"E and 117°11'43.38"E, showed in Fig 1. By a grab-type silt sampler, surface sediment samples (S2-S12) (0-20 cm) were collected from the subsidence section, and one sediment sample (S1) from the contrast point in the non-subsidence section. After sampling, the sediment samples were cleaned from the plant residuals and other debris, and then transported to the laboratory for further analysis in a sealed bag, and stored at -20ºC.

Sample Processing and Analysis

The collected samples were dried in a freeze dryer, crushed to remove debris such as stones and rhizomes, and filtered through a 200-mesh nylon sieve. Approximately 5 g samples were pressed into a thin sheet with a thickness of about 6 mm from each sampling point using a Tablet machine (FY-15 type). The X-ray fluorescence spectrometer (R-350) was then used to determine the concentrations of heavy metals [17]. Each test lasted 180 s, and the average of three measurements was summarized as the result of each sample. The standard sample should be used for instrument calibration once every analyzing 6 samples.
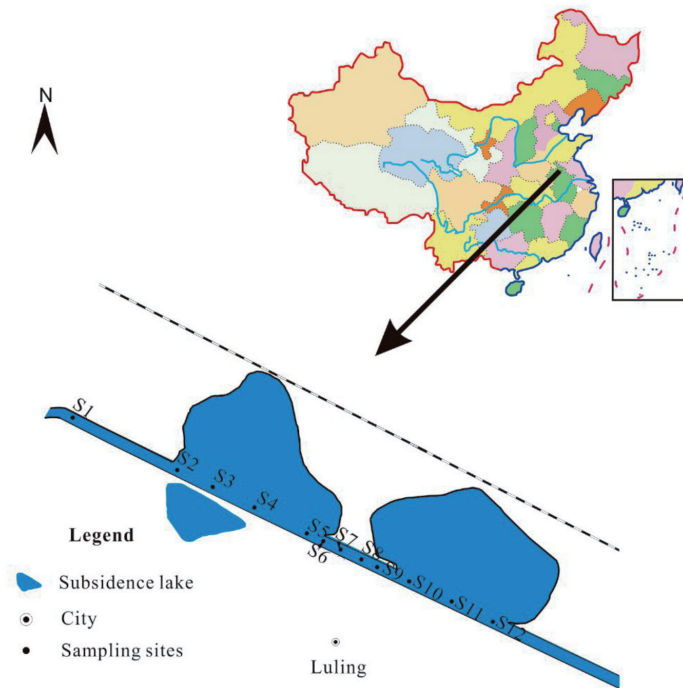
Fig. 1. Sediment sampling sites of the subsidence section of Luling Mine in the Tuohe River, China.
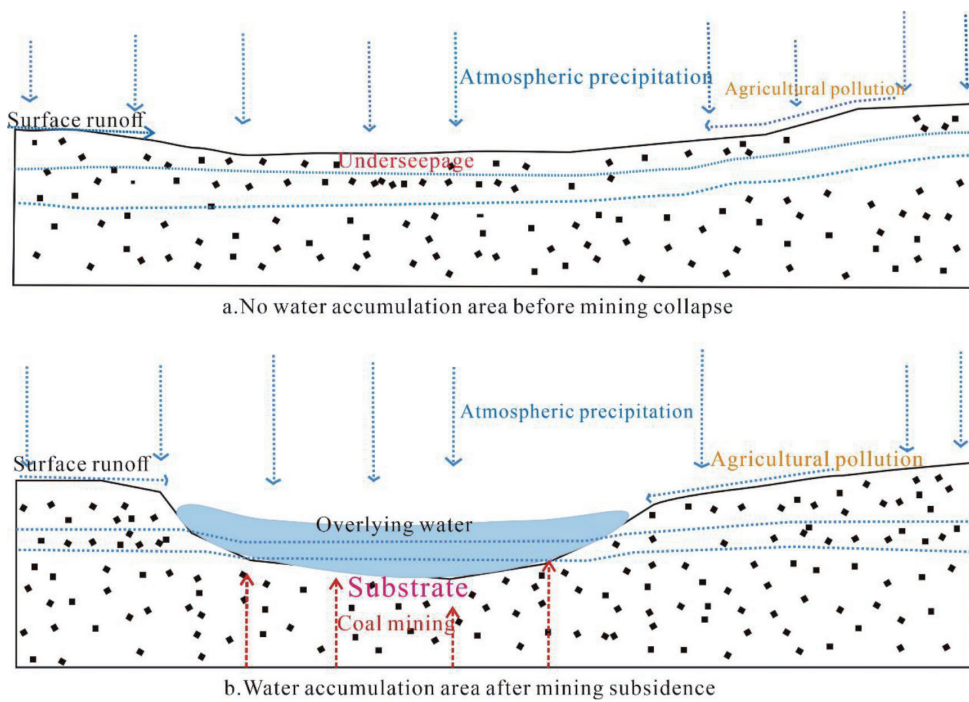

Fig. 2. The formation process and input pathways of potential pollution sources in the mining subsidence section.

Pollution Assessment and Source Apportionment

The *EF* and *NCPI* were effectively applied for assessing the pollution degree of a single element and comprehensive pollution level, respectively [18, 19]. Therefore, to clarify the pollution degrees of heavy metals in the study area, *EF* and *NCPI* have been applied and discussed as follows:

EF Method

The *EF* method has been presented in Equation (1) as follows:

$$EF = \frac{[w(M)/w(M_r)]_{sample}}{[w(M)/w(M_r)]_{baseline}} \qquad (1)$$

Table 1. Pollution level classification based on *EF* and the evaluation result statistics of sediment samples in the subsidence section of Luling Mine in the Tuohe River, China.

EF	Pollution degree	Pollution level	V	Mn	Ni	Zn	Cu	Pb	Cr	As
<1	0	Non-pollution	12	7	8	9	7	5	1	
1~2	1	Slight pollution		5	3	2	4	4	9	11
2~5	2	Moderate pollution			1	1	1	3	2	1
Total samples			12	12	12	12	12	12	12	12

where $w(M)$ denotes the mass fraction of the metal element. The $w(M_r)$, *sample*, and *baseline* mean the mass fraction of the reference element, the sample, and the background value, respectively. In this study, the background value of heavy metals in Anhui Province soil was considered a reference [20-22]. The Iron (Fe) was selected as a reference element. The study conducted by Sutherland divides the pollution degree of elements into 5 grades [23], as shown in Table 1.

NCPI Method

Based on the single factor index, this study applied the *NCPI* method to calculate the comprehensive pollution degree of eight heavy metals at each sampling point (Equation (3)) [24]. The calculation process of the single factor index has been presented in Equation (2).

$$C_f^i = C_i \div C_n^i = P_i \tag{2}$$

where C_f^i, C_i, C_n^i, and P_i denote the pollution index of metal i, the actual content of metal i, the evaluation reference of metal i, and the single-factor pollution index, respectively. In this study, the background value of heavy metals in soil in Anhui Province was used as a reference value for comparison.

$$P_N = \sqrt{\frac{\overline{P_i^2} + P_{imax}^2}{2}} \tag{3}$$

where $\overline{P_i}$ and P_{imax} mean the average single factor index and the maximum single factor index, respectively. P_N denotes *NCPI* value, and its grading standards are shown in Table 2.

Table 2. Classification standard of *NCPI* evaluation method for heavy metals in sediments

P_N	Pollution degree	Pollution level
≤0.7	1	Clean
0.7~1 (≤1)	2	Relatively clean
1~2 (≤2)	3	Slight pollution
2~3 (≤3)	4	Moderate pollution
>3	5	Heavy pollution

Positive Matrix Factorization (PMF) Model

The *PMF* method, as a receptor model, was effectively performed to quantify the pollution sources of heavy metals or other pollutants in aquatic ecosystems [25]. By adopting a robust PMF model, the influence of outliers on the performance of the model was reduced. The principle of this method is to decompose the matrix x_{ij} containing the original test data into two-factor matrices g_{ik} and f_{kj}, and the residual matrix ε_{ij}, which was shown as follows:

$$x_{ij} = \sum_{k=1}^{p} g_{ik} f_{kj} + \varepsilon_{ij} \tag{4}$$

Where x_{ij} represents the concentration of heavy metal j in the i-th sample in the test data set, and g_{ik} means the contribution rate of source k to sample i (source sharing matrix), and f_{kj} denotes the concentration of the j-th heavy metal input by the source k (source composition spectrum matrix), and ε_{ij} is appointed as the random error matrix. The detailed calculation process can be referred to the EPA PMF 5.0 User Guide.

Result and Discussion

Distribution Characteristics of Heavy Metal Contents

The descriptive statistical results of eight heavy metals in the sediments are presented in Table 3. Among all the sampling sites, As content in sediment samples exceeded the soil background value in Anhui Province, while the content of Pb was below the background value. Additionally, 23.08%, 61.54%, 69.23%, 38.46%, 76.92%, and 30.77% of V, Cr, Mn, Ni, Cu, and Zn samples surpassed their corresponding background values, respectively. Among the eight heavy metals, the average contents of Pb and V were lower than the background values, while the average contents of Mn, Ni, Zn, Cu, Cr, and As were higher than the background values. The average contents of V, Mn, Ni, Zn, Cu, Pb, Cr, and as were 0.93, 1.21, 1.20, 1.06, 1.15, 0.80, 1.18, and 1.78 times the background values, respectively, indicating various accumulation levels in the study area.

Table 3. Heavy metal contents in sediments of the subsidence section of Luling Mine in the Tuohe River, China (mg·kg⁻¹).

| Sampling points	V	Mn	Ni	Zn	Cu	Pb	**Cr**	**As**
Collapsed area control point (S1)	89	674	31	59	23	22	65	19
Maximum value	103	1167	96	143	30	24	133	30
Minimum value	82	460	14	50	19	19	58	10
Average (S2 to S12)	91.5	639.8	35.8	66	23.5	21.4	78.3	16
Standard deviation	7.2	187.1	22.5	25.2	3	1.4	19.8	5.4
Coefficient of variation (%)	7.90	29.20	62.80	38.10	12.90	6.70	25.30	33.50
Soil background value [22]	98.2	530	29.8	62	20.4	26.6	66.5	9
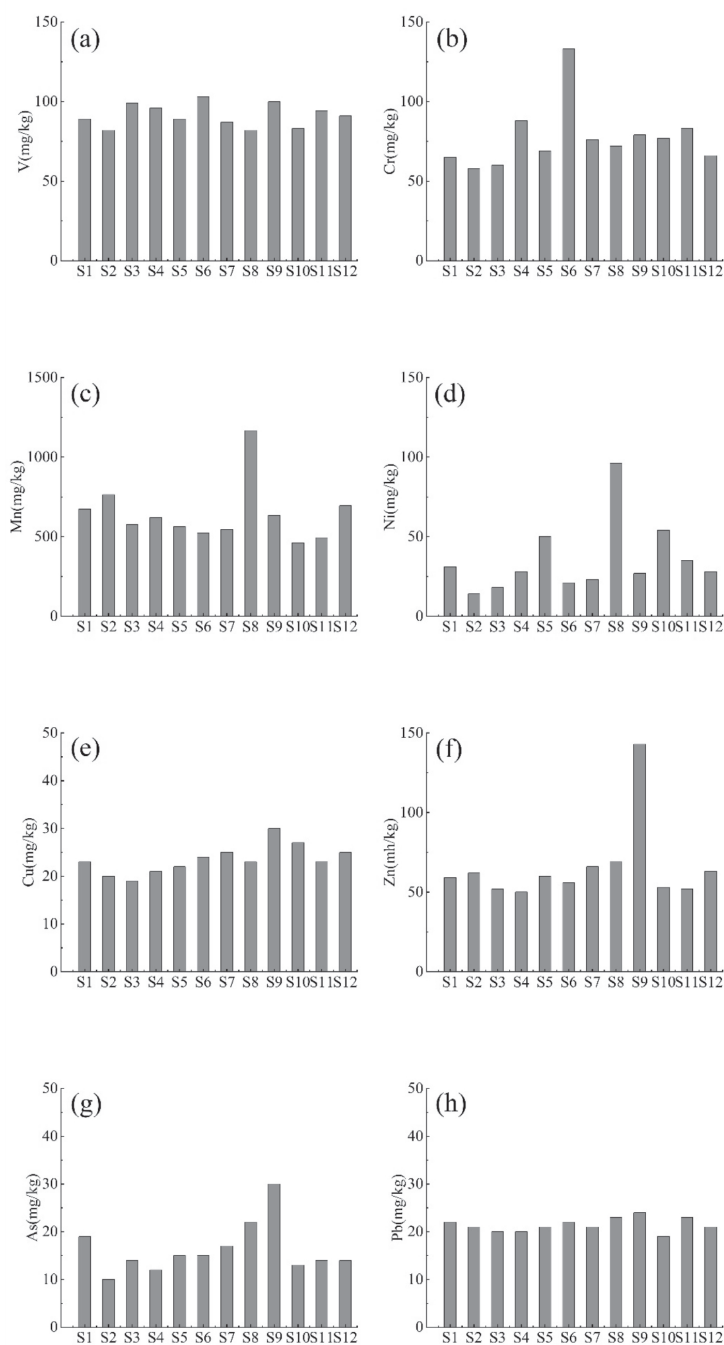
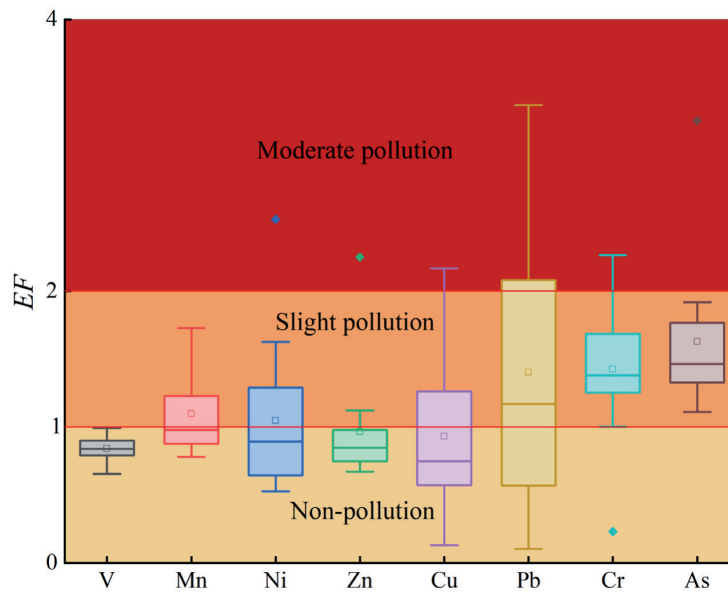Fig. 3. The content distribution of heavy metals in sediments.

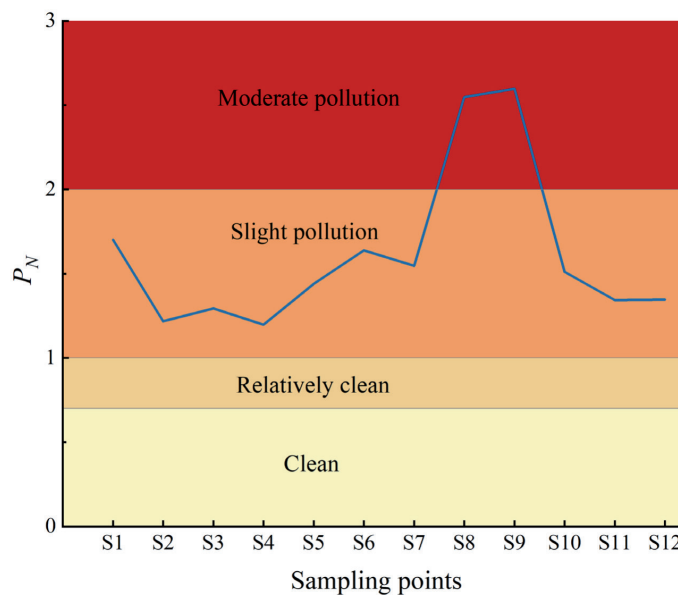Fig. 4. Boxplots of *EF* results of heavy metals in sediments.

Fig. 5. Assessment results of heavy metals in sediment based on *NCPI*.

The coefficient of variation can represent the varying characteristics in the spatial distribution of heavy metals, with a larger coefficient indicating a greater degree of interference from human activities [26]. The coefficients of variation of heavy metals in the sediments of sampling sites were as follows: Ni (62.8%) > Zn (38.1%) > As (33.5%) > Mn (29.2%) > Cr (25.3%) > Cu (12.9%) > V (7.9%) > Pb (6.7%), where the coefficient of variation of Ni is greater than 50%, indicating that Ni has strong and significant spatial heterogeneity. The coefficients of variation of Mn, Zn, Cr, and As were between 20% and 50%, showing strong spatial heterogeneity. The coefficients of variation of V, Cu, and Pb were less than 20%, indicating weak spatial heterogeneity.

The variation of heavy metal contents in the sediments is shown in Fig. 3. In sites S8 and S9, the sudden decrease of the water flow velocity in the river mouth probably led to the accumulation of heavy metals and their deposition in the sediments. Except for Cr, the change trends of other metal elements were basically consistent along with the sampling points.

Evaluation of the *EF* Method

The *EF* values of heavy metals in the sediments are shown in Fig. 4. Combined with Table 1, it can be seen that the average *EF* values of V, Mn, Ni, Zn, Cu, Pb, Cr and As were 0.84, 1.10, 1.05, 0.97, 0.93, 1.40, 1.43 and

Table 4. Correlation analysis among eight heavy metals in sediments of the subsidence section of Luling Mine in the Tuohe River, China

Element	V	Cr	Mn	Ni	Cu	Zn	As	Pb
V	1							
Cr	**0.565**	1						
Mn	-0.437	-0.293	1					
Ni	-0.504	-0.098	**0.617***	1				
Cu	0.085	0.237	-0.156	0.128	1			
Zn	0.240	-0.037	0.145	-0.023	**0.692***	1		
As	0.205	0.023	0.313	0.281	**0.652***	**0.864****	1	
Pb	0.234	0.182	0.367	0.179	0.364	**0.628***	**0.737****	1

Note: * and ** indicate significances at *P*<0.05 and *P*<0.01 levels, respectively.

1.63, respectively. The average *EF* values and less than 1.0 of *EF* results at each site showed that V were non-pollution in the study area. The number of samples with non-pollution and slight pollution for Mn were 7 and 5, respectively, indicating the accumulation of Mn was slightly affected by human activities. Ni, Zn, and Cu displayed *EF* values higher than 2.0 at one sampling site with moderate pollution level but less than 2.0 of *EF* values in other sites with the non-pollution and slight pollution states. The *EF* values of Pb in more than 50% of sampling points were higher than 1.0, especially the sites S2, S3, and S6 (*EF*>2.0) with a moderate pollution level, presenting the obvious enrichment influenced by human activities. The *EF* values of Cr in all sampling sites except site S9 were higher than 1.0 when the *EF* values of As ranged from 1.11 to 3.25, corresponding with their high average *EF* values. Generally, the pollution levels of Cr and As were higher than other heavy metals in the study area. From a perspective of distribution characteristics in sampling sites, the sites S8 and S9 showed high pollution levels of heavy metals, probably because of the influences of hydrodynamic action on migration ability and accumulation of heavy metals.

Evaluation of *NCPI* Method

The *NCPI* values were used to evaluate the comprehensive contamination degree of eight heavy metals in sediments of the study area. As shown in Fig. 5, the *NCPI* values of all sampling points were greater than 1.0. Where, the *NCPI* values in sampling sites S1-S7 and S10-S12 ranged from 1.0 to 2.0, indicating slight pollution according to the grading criteria (Table 2). The *NCPI* values at sites S8 and S9, ranging from 2.0 to 3.0, presented the moderate pollution level. Based on the calculation of the percentage distribution of pollution levels, the sampling sites with slight pollution and moderate pollution accounted for 83.33% and 16.67% of the total sampling sites, respectively. The *NCPI* values of heavy metals in the sediment

of the river section were higher than those in the subsidence pool section (Fig. 5), demonstrating that the hydrodynamic had a great influence on the degree of heavy metal pollution in the study area.

Source Analysis of Heavy Metal Elements in Sediments

Correlation Analysis and Principal Component Analysis of Heavy Metals in Sediments

The correlation analysis of heavy metals is shown in Table 4. As exhibited positive correlations with Cu (r = 0.652), Zn (r = 0.864), and Pb (r = 0.737), and V showed a positive correlation with Cr (r = 0.565). Additionally, there was a positive correlation between Mn and Ni (r = 0.617). The high correlation between these heavy metal elements may be due to their similar sources [27].

The results of the principal component analysis of heavy metals in sediments are shown in Table 5 and Fig. 6. The first Principal Component (PC1) had the highest contribution, accounting for 40.01%. Zn, Cu,

Table 5. The PCA of eight heavy metals in sediments of the subsidence section of Luling Mine in the Tuohe River, China.

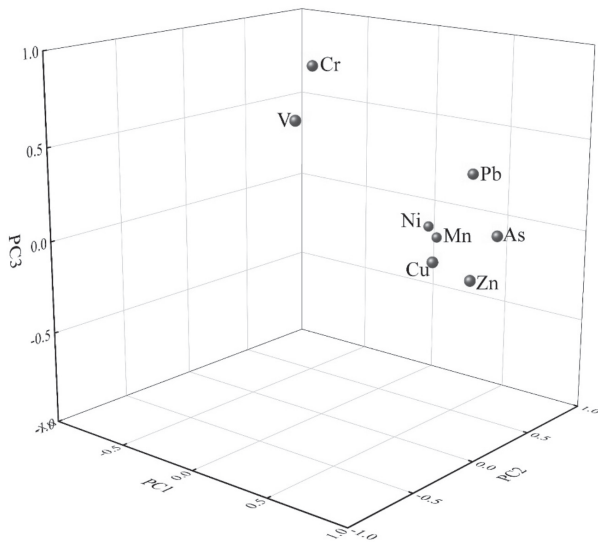| Element	PC1	PC2	PC3
V	0.223	-0.471	**0.726**
Mn	0.105	**0.881**	-0.176
Ni	0.057	**0.867**	-0.117
Zn	**0.971**	-0.029	-0.041
Cu	**0.801**	-0.110	0.039
Pb	**0.687**	0.383	0.376
Cr	0.001	-0.082	**0.918**
As	**0.927**	0.264	0.115
Contribution rate	40.01%	28.95%	12.76%

Fig. 6. Loading distribution of the first three dominant components (PC1, PC2, and PC3) of PCA in sediments.

Pb, and As have higher positive loads. Metals Mn and Ni were loaded in the second principal component (PC2), with a contribution of 28.95%. The third principal component (PC3) explained only 12.76% of the variability and metals Cr and V made relatively large contributions. As shown in Table 5, three principal component factors were extracted, which could explain 81.72% of the total variables.

Source Analysis of Heavy Metals-Based PMF

In order to further analyze the contribution of each factor, The content data of eight heavy metals in 12 surface sediment samples and the uncertainty data related to these contents were input into EPA PMF 5.0 software for analysis. The factor component spectra of heavy metals in surface sediment analyzed by PMF are shown in Fig. 7, with the relative contributions of the

three factors being 34.8%, 37.2%, and 28%, respectively.

The results of the PMF model are shown in Fig. 7. In Factor 1, it can be seen that heavy metals (Zn, Cu, Pb, As) have a high loading, corresponding with PCA results. Combined with correlation analysis, it showed that these heavy metals have a very strong correlation with each other, suggesting that these heavy metals may have the same source [28]. As is generally considered to be a sign element representing agricultural non-point sources [29]. Therefore, it is hypothesized that Factor 1 may be an agricultural pollution source. As shown in Fig. 7, the average proportion of Factor 1 is 34.8%.

In Factor 2, the loading values of Mn and Ni are high, corresponding with PC2 in PCA results. Their average contents were much higher than the background values. Ni is associated with mineral extraction [30]. Therefore, it is hypothesized that factor 2 may be due to coal mining. The contribution of this source to heavy metals was 37.2%.

In Factor 3, the loadings of V and Cr were higher, similar to PC3 in PCA results. Moreover, the contents of V and Cr in the sediments are lower than the soil background values. Therefore, Factor 3 could be regarded as a natural source, such as soil-forming parent material [31]. The average proportion of factor 3 is 28%. Factors 1, 2, and 3 identified by the PMF model are agricultural non-point source pollution (34.8%), industrial sources (37.2%), and natural sources (28%), respectively. This suggested that human activities (agricultural non-point source pollution and industrial source pollution) account for 72%, indicating that heavy metals in sediments in the study area are mainly controlled by human activities.

Conclusions

This study collected sediment samples in sediments of the subsidence section of Luling Mine in the Tuohe River, China, and eight heavy metals (V, Mn, Ni, Zn,
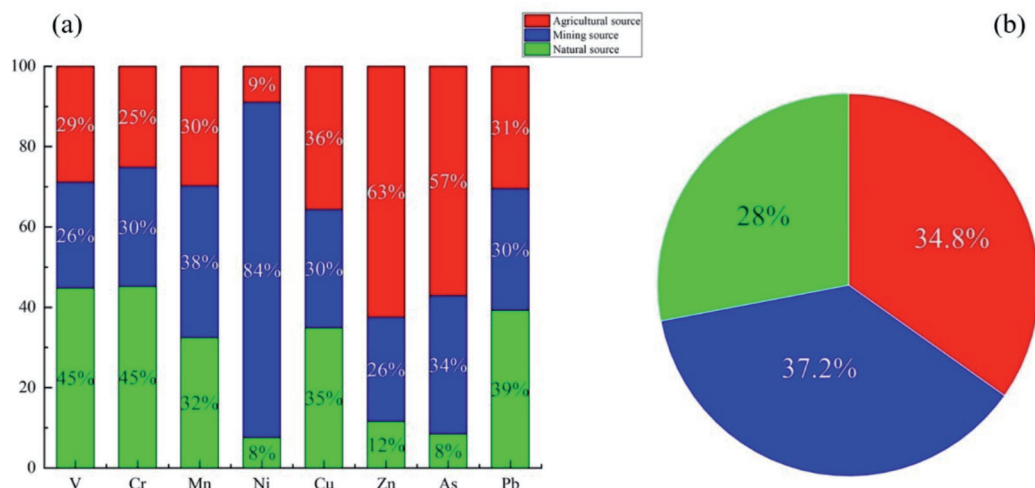

Fig. 7. Contributions from each source: a) to each kind of heavy metal; b) to all heavy metals.

Cu, Pb, Cr, and As) in sediments were determined. The average contents of V, Mn, Ni, Zn, Cu, Pb, Cr, and As were 0.93, 1.21, 1.20, 1.06, 1.15, 0.80, 1.18, and 1.78 times the background values, respectively, with the various accumulation levels in the study area. *EF* results showed that the pollution levels of Cr and As were higher than other heavy metals in the study area. The *NCPI* method showed that the areas located in sites S1-S7 and sites S10-S12 were in a slightly polluted level of eight heavy metals. Only two sampling points (S8 and S9) were in the moderate pollution category. According to the PCA results, there are three sources of heavy metals in the sediment of the study area, including coal mining, agricultural sources, and natural sources. Based on the calculation of the PMF model, the contribution rates of coal mining, agricultural sources, and natural sources were 37.2%, 34.8%, and 28.0%, respectively. The study can provide a reference for the treatment of heavy metal pollution in the mining subsidence area and provided data support for the ecological protection of the surrounding environment.

Acknowledgments

Conflict of interest

The authors declare no conflict of interest.

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