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Original Research

Quantitative Contributions of the Sources, Contamination and Ecological Risk of Heavy Metals in Soils from a Closed Coal Mine of Huaibei Coalfield, Eastern China

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Abstract

With the increasing number of closed mines, the geological environment problems around closed mines have become increasingly prominent, especially in terms of soil pollution. In this paper, a total of 32 topsoil samples were collected from Qianling closed coal mine and the contents of Cu, Zn, Co, Cr, Mn, As, Cd and Pb were analyzed. The results indicated that the contents Zn and As exceeded the surface soil background values of Anhui Province. Compared with the soil of production mines, the contents of Pb and Zn were higher. The assessment results of pollution and ecological risk of soil based on Nemerow index, Geological accumulation index and potential ecological risk assessment method showed that the closed coal mine was in the mild to moderate pollution and mild hazard ecological risk. The Pearson correlation and positive matrix decomposition model were applied to identify the sources of soil heavy metals, which included atmospheric dustfall (22.47%), natural factors (22.51%), agricultural activities (4.40%), transportation (8.90%) and industrial emissions (42.08%), respectively.

Keywords: source analysis, ecological risk, soil heavy metals, closed coal mine, quantitative contributions

Introduction

China's energy structure was low in oil and gas, and relatively rich coal. Coal had always held a dominant position in China's primary energy, and this situation can not be changed for a long time [1-2]. Due to the long-term high-intensity mining of coal resources in China, some old mining areas tended to be exhausted and closed [3]. It was estimated that by 2030, the number of closed/abandoned mines in China will reach 15000 [4]. During the mining, selection by washing, storage and transportation of coal resources,

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some pollutants (such as waste water, coal dust and coal gangue, etc.) were easily discharged into the surrounding environment. The pollutants entered the soil through runoff, sedimentation and leaching, which would lead to the enrichment of heavy metals in the soil of the coal mine area [5], resulting in the soil around the closed/abandoned mine safety and environmental issues [6]. Once the soil was contaminated by heavy metals, it would not only inhibit and poison the growth and development of plants, but also had a serious impact on the closure of the originally fragile ecological environment around coal mining areas. It may also endanger human health through the food chain and restrict local economic development [7].

In recent years, predecessors have carried out a large number of studies on heavy metal pollution and environmental effects of coal mine soil, which mainly focus on the content characteristics of heavy metals in coal mine soil, pollution (impact) assessment [8-11], spatial differentiation [12-13], enrichment characteristics [14-15] and pollution source analysis etc. The method of soil pollution source analysis is considered a central issue and it is essential to select appropriate approaches to identify the source. Several approaches that integrated by Unmix model, Ensemble model, Isotropic model and PMF model [16-18]. In general, the authors have mostly studied soil heavy metal pollution on production coal mines, and relatively few studies on closed/abandoned mines.

The purpose of the present study was to provide scientific basis for environmental supervision, early warning and treatment of soil in closed mining area, which using quantitative contributions of the sources, contamination and ecological risk of heavy metals.

Materials and Methods

General Situation of the Study Area

The Qianling Coal Mine was located in the Huaibei coal field of northern Anhui Province. It was built in 1971 and put into operation in 1983. It had a design capacity of 300,000 tons of raw coal per year [19] The mine was closed in August 2015. The regional profile and the location of the study area were shown in Fig. 1a) and Fig. 1b).

The soil types in the study area were mainly tidal soil, mortar black soil, silt black soil, green loess, etc.

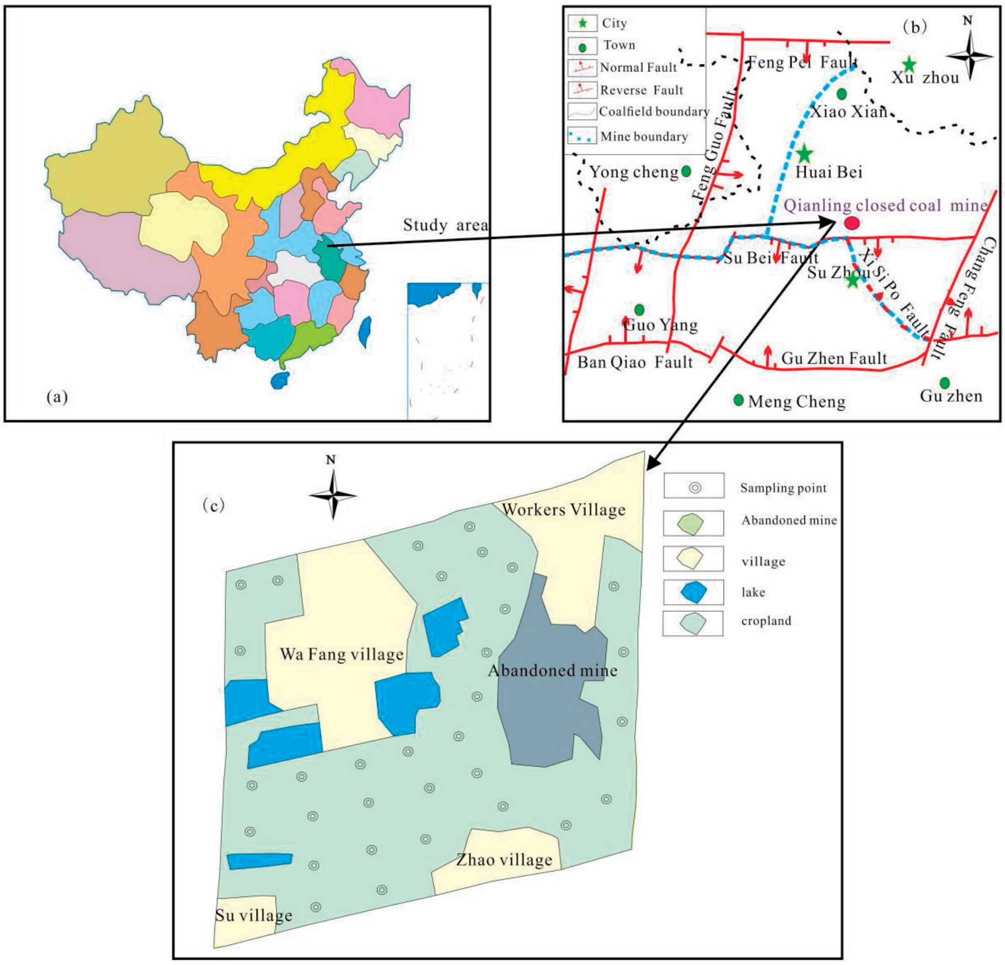

Fig. 1. Geographical location and sampling point distribution map of the study area.

The soil organic matter content was low, the structure was poor, and the arable property was good, which belonged to the loose accumulation layer of Cenozoic. The study area had four distinct seasons, mild climate and suitable rainfall. It had the typical warm zone and semi-humid monsoon climate characteristics of mid-latitude regions. The rainfall was mainly concentrated in summer, and the winter was dry. The temperature changed greatly in spring and autumn. The annual distribution of rainfall was uneven, mainly concentrated in July to September, accounting for 75% of the annual precipitation. The dominant wind direction in summer was southeast wind, and the dominant wind direction in winter was northeast wind [20].

Sample Collection, Processing and Testing

The grid distribution method was mainly used for the layout of sampling points, taking into account the surface water system and traffic roads in the mining area. A total of 32 sampling points were arranged. The distribution of sampling points was shown in Fig. 1c). The surface soil of 0-10 cm was collected with stainless steel shovel, and the surface debris was removed, and then put it into a clean sealing bag, labeled and the sampling point was positioned with GPS instrument. After the samples were transported back to the laboratory, they were dried by natural air, crushed, passed through 60, 80, 100 and 200 mesh wooden nylon sieves in turn, and then the samples were reduced to about 1kg by quartering method. The 5 g soil samples to be measured were accurately weighed by the analytical balance, and the samples to be tested were taken out after being pressed by 20 t tablet press.

The content of Cu, Zn, Co, Cr, Mn, As, Cd, and Pb elements in the sample was determined by X-ray fluorescence spectrometer (ICP-2000), and the soil component analysis standard material (GBW07430, GSS-16) was detected, and the recovery rate ranged from 86% to 115%, the relative deviation between samples is <5%, and the test results met the quality control requirements.

Data Processing and Analysis Methods

Statistical Analysis

The IBM SPSS statistics 19.0 software was used to process and analyze the data, including descriptive statistical analysis of heavy metal elements and Pearson correlation analysis. The EpaPMF5.0 software was used to analyze the sources of heavy metals in soil. The box diagram and histogram were drawn by Origin 8.0 software.

Pollution Assessment Method

Nemero Comprehensive Pollution Index Method

Nemero comprehensive pollution index method takes into account the average value and maximum value of single factor pollution index. It is an evaluation method that comprehensively considers the impact of various metals in soil on environmental quality [21-23]. Its calculation formulas are (1) and (2). The evaluation standard of pollution degree is shown in Table 1.

$$P_i = C_i / S_i \tag{1}$$

$$P_n = \sqrt{\frac{(P_{i,ave})^2 + (P_{i,\max})^2}{2}} \tag{2}$$

...where P_i is a single environmental quality index; P_n represents the Nemeiro comprehensive pollution index; C_i is the measured element content; S_i is the element reference standard content; $P_{i,ave}$ is the average value of the element environmental quality index of the sampling point; $P_{i,\max}$ is the maximum value of the environmental quality index of the sampling point.

Geological Accumulation Index Method

The Geological accumulation index was proposed by the German scientist Muller in 1969 to quantitatively evaluate the degree of heavy metal pollution in sediments [24]. The classification standard of heavy

Table 1. Classification Standard of soil heavy metal pollution.

Geological accumulation		Nemero comprehensive pollution				Potential ecological hazard			
I_{geo}	Level	P_i	Level	P_n	Level	E_r^i	Level	RI	Level
≤0	Non	≤1	Clean	≤0.7	Clean	<40	Mild	<150	Mild
0~1	Non-Mild	1~2	Mild	0.7~1	Warning line	40~80	Moderate	150~300	Moderate
1~2	Mild	2~3	Moderate	1~2	Mild	80~160	Strong	300~600	Strong
2~3	Mild-Strong	>3	Serious	2~3	Moderate	160~320	Very Strong	≥600	Very Strong
3~4	Strong-Extreme			>3	Serious	≥320	Extreme Strong		
>5	Extreme								

metal pollution is shown in Table 1, and the calculation formula is (3).

$$I_{geo} = \log_2\left[C_n / K \bullet B_n\right] \tag{3}$$

...where C_n is the content of the element in the soil; B_n is the reference value of the element. In this study, the soil background value of Anhui Province is taken as the reference value [25], and K represents the variation coefficient of background value caused by diagenesis (generally K = 1.5).

Potential Ecological Hazard Index Method

The potential ecological risk index was used to evaluate the potential risk, ecological sensitivity and toxicity of heavy metal concentration, reflecting the pollution degree of single pollutant and even the pollution level of mixed pollutants [26]. The formula is (4).

$$RI = \sum_{i=1}^{n} E_r^i = \sum_{i=1}^{n} T_r^i C_r^i = \sum_{i=1}^{n} T_r^i C_o^i / C_n^i \tag{4}$$

...where RI is the potential ecological hazard index of multiple heavy metals; E_r^i represents the potential ecological hazard index of a single heavy metal; C_o^i means the actual measured value of elements; C_n^i is the reference value of elements; T_r^i represents the toxicity response coefficient of elements.

Pollution Source Analysis Method

Positive matrix decomposition is a multi-element analysis technique that decomposes sample data into two matrices of factor contribution and factor distribution. Its main advantage is to correlate the sample size and estimated uncertainty with the sample data to weight a single point, and to easily manage missing data [27]. The PMF model uses the weighted least squares method to limit and iteratively calculate, and continuously decompose the matrix. The optimization goal is to minimize the objective function. The calculation formulas for the minimum value of the objective function Q are (5) and (6).

$$Q = \sum_{i=1}^{n} \sum_{j=1}^{m} \left(\frac{x_{ij} - \sum_{k=1}^{p} g_{ik} f_{kj}}{u_{ij}}\right)^2 \tag{5}$$

$$X_{ij} = \sum_{k=1}^{p} g_{ik} f_{kj} + e_{ij} \tag{6}$$

In addition, the establishment of the PMF model requires the uncertainty u_{ij} of the sample species concentration, and the calculation formulas are (7) and (8).

When c≤MDL, $u_{ij} = \frac{5}{6} \times MDL$ (7)

When c>MDL, $u_{ij} = \sqrt{(EF \times c)^2 + (0.5 \times MDL)^2}$ (8)

...where c is the concentration of elements in the sample; MDL is the detection limit of the determination method; EF is the determination precision.

Results and Discussion

Characteristics of Soil Heavy Metal Content

The statistical characteristics of the contents of Cu, Zn, Co, Cr, Mn, As, Cd, and Pb of the 32 soil samples were listed in Table 2. It can be seen that the order of the average content of heavy metals in the study area was Mn>Zn>Pb>Cr>As>Cu>Co>Cd. Except the average contents of Cu, Co, and Cr were lower than the background values of Anhui Province, the average contents of Zn, Mn, As, Cd, and Pb were 89.37 mg/kg, 544.29 mg/kg, 12.63 mg/kg, 0.10 mg/kg, 31.90 mg/kg, exceeding the soil background values of Anhui Province, respectively 1.44, 1.03, 1.40, 1.03, and 1.20 times, indicating that these heavy metals had accumulated different degrees of pollution accumulation, including Mn and Cd relatively were low, Pb, As and Zn were relatively high. Compared with the soil pollution risk screening value of the currently piloted "Soil Environmental Quality-Agricultural Land Soil Pollution Risk Control Standard GB15618-2018" [28], the average values of Cu, Zn, Co, Cr, Mn and Cd were all lower than the soil pollution risk screening value except for the Pb exceeding standard.

The coefficient of variation can reflect the degree of dispersion of the data. According to Wilding's classification of the degree of variation [29], it could be seen that Zn, Mn, and As (35.64%, 21.05%, 34.31%) were moderate variations (15%<CV<36%); Cu, Co, Cd (175.68%, 91.52%, 45.86%) were high variance (CV>36%); Cr (9.31%) was low variance (CV<15%).

Comparative Analysis of Soil Heavy Metals Content with Production Mining Area

It could be seen from Table 3 that the contents of Zn, As and Pb in the soil of closed mine were higher than those of local ordinary cultivated land [30]. In the study area, the average contents of Zn, Cr, Cu, and Cd were basically consistent with the results of the study on soil heavy metals in the Sunan mining area [31] (production mining area) and Linhuan mining area [32] (production mining area), and the order of contents was Zn> Cr>Cu>Cd, and compared with the heavy metals in the wasteland of Sudong mining area [33] (production mining area), Cr, Zn, Pb are in different order from As and Cu. Compared with the soil in Sudong, Sunan and

Table 2. Statistical characteristics of heavy metal content in soil samples.

Element	Range ($mg \cdot kg^{-1}$)	Mean ($mg \cdot kg^{-1}$)	Standard deviation ($mg \cdot kg^{-1}$)	Coefficient of variation/(%)	Background values of Anhui Province ($mg \cdot kg^{-1}$)	Chinese soil pollution risk screening values/($mg \cdot kg^{-1}$)
Cu	0.58~46.39	5.53	9.72	175.68%	20.4	100.00
Zn	53.65~235.93	89.37	31.85	35.64%	62.00	250.00
Co	0.55~19.15	3.25	2.97	91.52%	16.30	-
Cr	23.58~37.57	31.75	2.95	9.31%	66.50	200.00
Mn	296.34~977.31	544.29	114.55	21.05%	530.00	-
As	5.37~20.92	12.63	4.33	34.31%	9.00	30.00
Cd	0.03~0.20	0.10	0.04	45.86%	0.097	0.30
Pb	23.36~46.98	31.90	4.16	13.04%	26.60	120

Table 3. Comparison of heavy metals content with production coal mine ($mg \cdot kg^{-1}$).

Area	Cu	Zn	Co	Cr	Mn	As	Cd	Pb	Literature sources
Study area (closed)	5.53	89.37	3.25	31.75	544.29	12.63	0.10	31.90	This study
Local cultivated land	22.12	57.63	-	67.70	-	9.71	0.13	17.58	[30]
Southern-Su (production)	28.78	65.43	-	50.89	555.30	-	0.28	12.48	[31]
Eastern-Su (production)	26.16	63.68	9.12	69.33	457.74	14.99	0.08	28.99	[32]
Linhuan (production)	24.77	60.25		50.02			0.28	27.10	[33]

Linhuan mining areas, it is found that the closed mines had lower Cu and Cr, and significantly higher Pb and Zn contents. It was estimated that there were Pb and Zn pollution in the surface soil of the closed mine.

Liu [9] et al. analyzed the heavy metals in the soil of the East Coal Field and showed that the content of heavy metals in the soil around the coal mine was affected by mining years, coal combustion, wind direction, topography, slope direction, soil texture and human activities. The closed coal mine was an old mining area with long mining activities and significant accumulation of heavy metals. The soil in this mining area was dominated by fluvo-aquic soil and sand ginger black soil, with heavy texture, poor permeability, low degree of heavy metal leaching, and more heavy metals attached to it. Considering that the closed mine is located in the Huaibei coal field, the terrain was flat, and the influence of terrain and slope can be basically ignored. The mining years, soil texture, wind direction and human activities had a significant impact on the distribution of heavy metal content in the closed mine.

Assessment of Soil Heavy Metal Pollution

Based on the background values of surface soil in Anhui Province, the Geological accumulation index, Nemerow comprehensive index and potential ecological hazard index of soil samples in the study area were calculated.

According to Table 1, the Nemerow index of all elements in the sample were greater than 0, in which the Nemerow index of Cr was 0.52, ranging from 0 to 0.72, which belonged to the clean; Cu, as, Cd, Pb, Mn, Co exceeded the warning line and were in the light pollution level, in which the Nemerow index of Zn was the highest of 2.88, belonging to moderate pollution. The Nemerow indexes were ranked as Zn (2.88) >As (1.92)>Cd = Cu (1.62)>Pb (1.51)>Mn (1.49)>Co (0.84)>Cr (0.52), as shown in Fig. 2.

According to the classification standard of pollution level in Table 1 and formula (3), the Geological accumulation index of Co and Cr were less than 0, and they were in a Non-pollution state, indicating that the pollution risk of heavy metal elements in Co and Cr was low; Zn , As, Cd had a certain level of accumulation, in which Zn was in Non-pollution, Non-Mild and Mild pollution state, and the proportions of sample points accounted for 78.13%, 18.75%, and 3.13% of the total number of samples respectively. The pollution contribution of As was 46.88%, and that of Cd was 15.63%. The pollution levels of As and Cd belonged to Non-Mild level. For Pb, Cu and Mn, only one abnormal sample point was in Non-Mild pollution level, and the rest were Non-pollution, as shown in Fig. 3. From the average value of the Geological accumulation index, the pollution degree of heavy metal elements on the soil around the closed mine was in order: Zn>As>Pb>Mn>Cd>Cr>Co>Cu, which

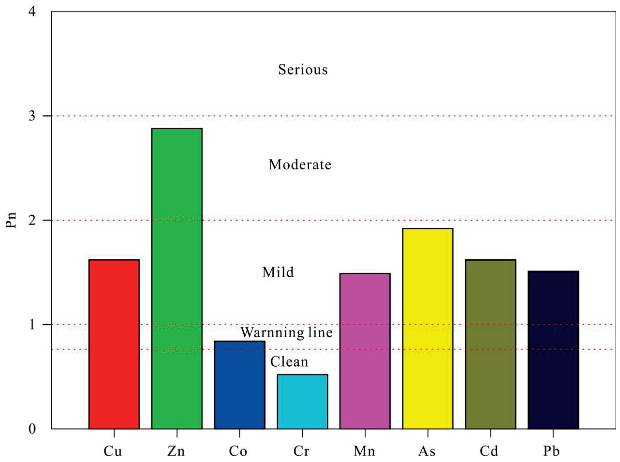

Fig. 2. Histogram of Nemero comprehensive pollution assessment.

was consistent with the analysis results of heavy metal content characteristics, indicating that the pollution accumulation of Zn and As in the closed coal mine soil had occurred to a certain extent.

According to Table 1 and formula (4), the potential ecological risk index of Zn, As and Pb in 100% soil samples was in a mild risk state, and Cd was the most important potential ecological risk factor, with 18.75% and 81.25% of soil samples showing moderate and mild hazards respectively (Fig. 4a, Fig. 4b). In general, the potential risk of soil heavy metals to the ecological environment in the study area was at a mild level (31.60<RI<92.61), as shown in Fig. 4c).

Different evaluation methods have different specific evaluation results. The reason is that Nemeiro index and Geological accumulation index will change due to different evaluation standards, and their results have a linear relationship with the content of heavy metals. Therefore, the two methods are basically consistent with the content analysis results [34]. The potential ecological hazard index is weighted due to the toxic effects of different heavy metals, and the environmental ecological effects are linked with toxicology. The evaluation results will be more focused on toxicological aspects due to the toxicity coefficient. Based on the three evaluation methods, it can be known that the main control objects of heavy metals in the soil of the Qianling closed mine were Zn, As, and Cd, and other heavy metals need to be controlled.

Source Analysis of Soil Heavy Metal Pollution

Correlation Analysis

Studies have shown that there was a significant correlation between the contents of heavy metals, which could reflect that each element has the same source or geochemical process [35]. If there was a significant and extremely significant correlation between the elements, it indicated that the elements generally had a certain homologous relationship or belonged to the compound pollution situation.

Table 4 showed Pearson correlation coefficients of 8 heavy metals in surface soil of closed mine. It could be seen from Table 4 that Mn-Co ($r = 0.654$), Zn-Cu ($r = 0.520$), Pb-Zn ($r = 0.489$) and Pb-Cu ($r = 0.358$) had extremely significant positive correlation ($P<0.01$), while Cu-Mn ($r = 0.332$), Cu-Pb ($r = 0.358$) and Zn-Cr ($r = 0.329$) were positively correlated, indicating that

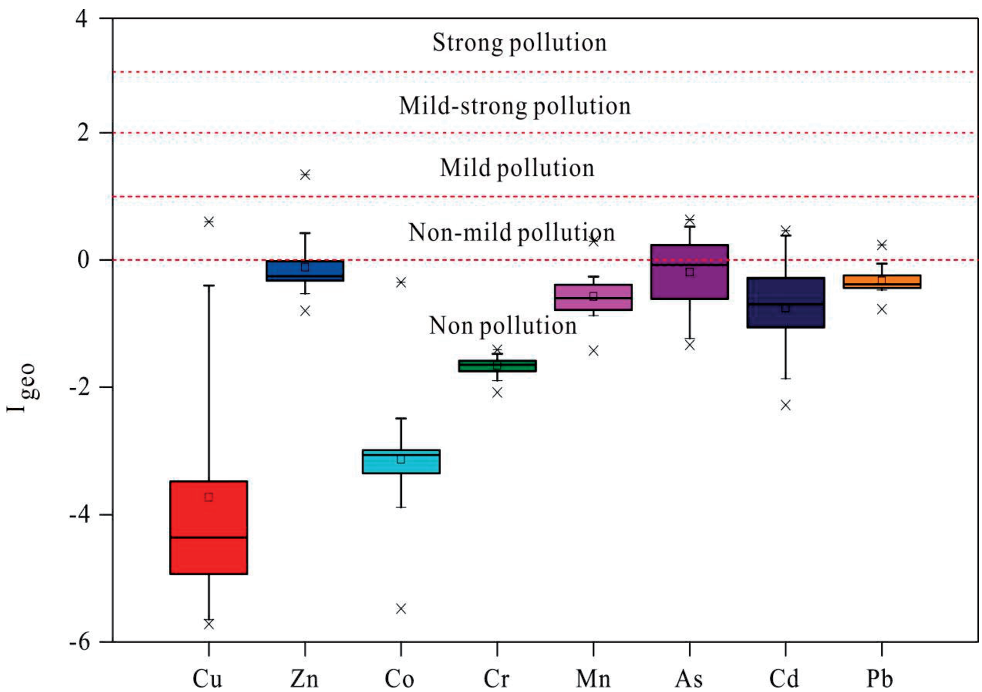

Fig. 3. Box diagram of Geological accumulation index.

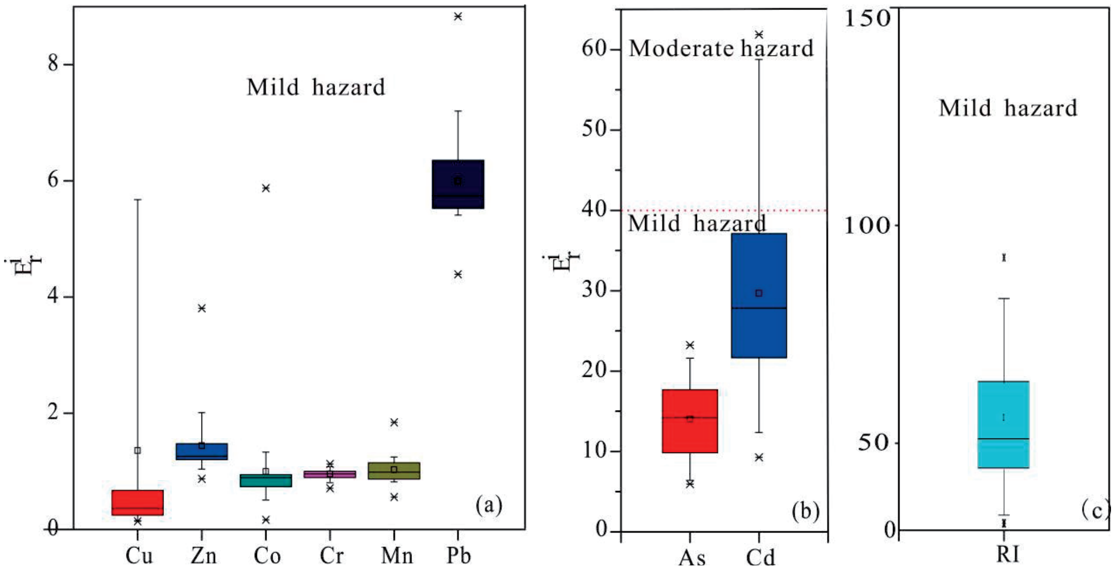

Fig. 4. Box diagram of potential ecological risk assessment.

Pb, Zn and Cu may had the same source or similar geochemical process, while Mn and Co may had the same influence factors. According to the correlation preliminary judgment, the sources of heavy metals in the study area were complex, and different sources of heavy metals need further analysis.

Quantitative Contributions of the Sources Analysis

In order to further analyze the possible pollution sources of heavy metals in the soil of the closed coal mine, the positive rectangular decomposition (PMF) model was used to analyze heavy metal samples, and the contribution ratio of eight elements was evaluated. The pollution sources and contribution ratios of the eight heavy metals were shown in Fig. 5.

The main load element of factor 1 was As, which the load rate was 57.8%. Other elements had lower load rate (Fig. 5a). When analyzing the sources of heavy metals in Zhundong coal mine soil, scholars pointed out that As mainly came from industrial emissions, coal combustion and transportation [9]. The previous studies on emission characteristics of coal fired boilers showed that As element accounted for 84.6% of raw coal content in fly dust from coal combustion [36]. The evaluation of heavy metal pollution in farmland soils in the coal mine-affected area of northern Bangladesh found that coal mine dust containing sulfide mineral particles was deposited on the surface and releases As and other toxic elements after oxidation [37]. The atmosphere is an important carrier of natural and man-made pollutants. Atmospheric dust reduction may be an important way for soil heavy metal enrichment. The Coal mine dust and fly ash deposited on the soil surface of mining area for a long time after closing the mine, which caused the accumulation of soil As in mining area. Therefore, factor 1 represented the source of atmospheric dustfall caused by mixed factors.

Table 4. The Pearson correlation analysis between elements.

Elements	Cu	Zn	Co	Cr	Mn	As	Cd	Pb
Cu	1							
Zn	.520**	1						
Co	0.198	0.016	1					
Cr	0.09	0.329	0.112	1				
Mn	0.332	-0.084	.654**	0.265	1			
As	-0.286	-0.268	-0.177	-0.051	-0.103	1		
Cd	-0.01	0.056	0.037	0.209	-0.014	0.271	1	
Pb	.358*	.489**	-0.198	-0.231	-0.126	-0.289	-0.213	1

** the correlation was significant at 0.01 level (bilateral)
* the correlation was significant at 0.05 level (bilateral)

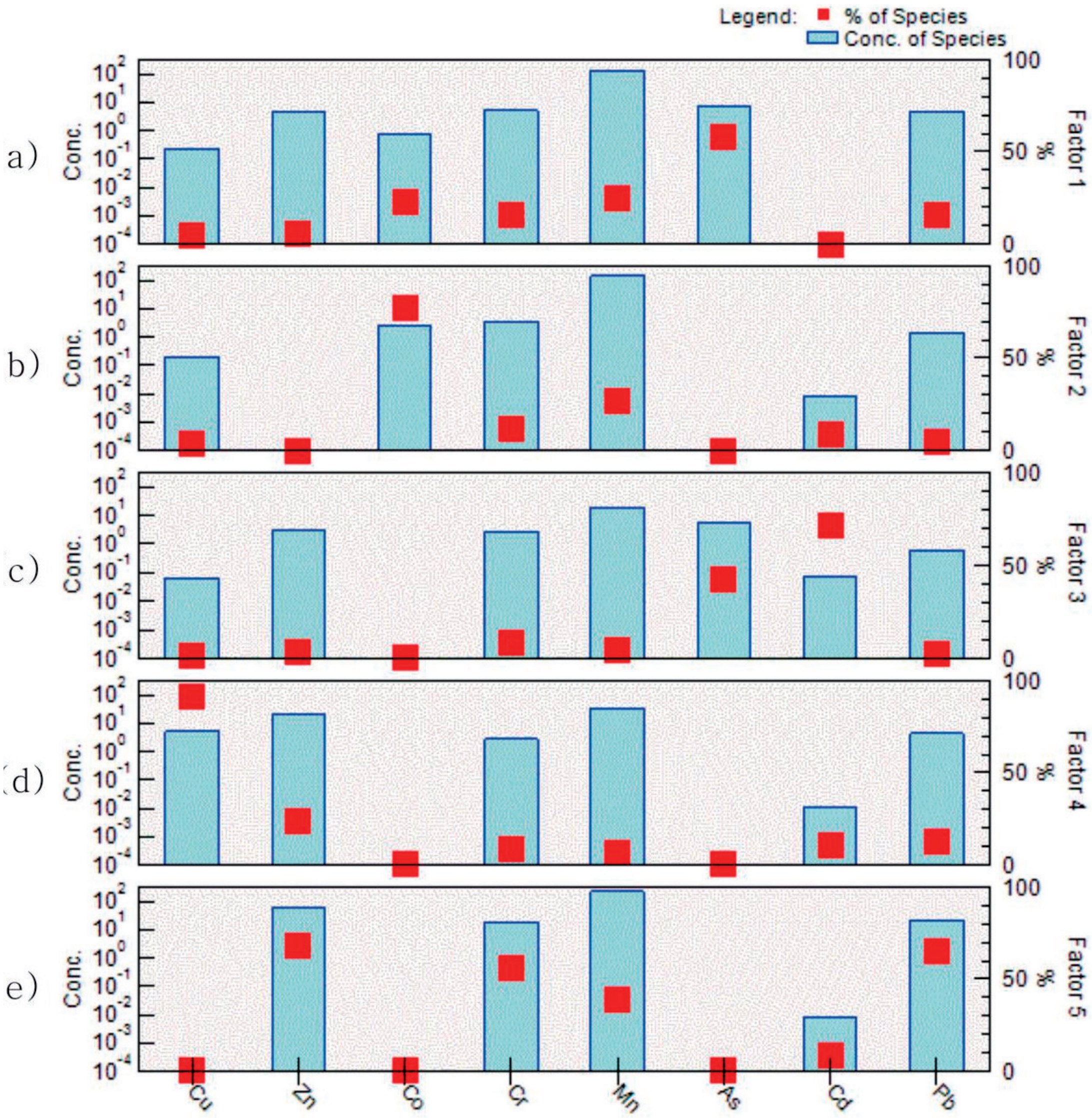

Fig. 5. Source profiles and source contribution of soil heavy metal from PMF.

Factor 2 is dominated by Co (77.6%) and Mn (26.6%) (Fig. 5b). The results of heavy metal pollution in the Sudong mining area found that natural factors such as soil parent material were the main source of Co-Mn enrichment [32]. The basic statistical characteristics and correlation analysis of heavy metals in the soil showed that the Co content was lower than the soil background value in Anhui Province and was not polluted . The Mn content exceeded the standard by only 1.03 times, which belonged to mild pollution, and there was a strong positive correlation between Co and Mn, indicating that natural factors were the main sources of Co and Mn in the study area. Therefore, factor 2 resolved to a natural source.

In factor 3, Cd (72.0%) and As (42.2%) contributed the most (Fig. 5c). Research on the sources of Cd in China's soil show that Cd mainly comes from agricultural production activities [38]. The previous studies have found that 40% of As in the environment may be related to the natural environment and agricultural activities, and the remaining 60% was attributed to traffic and industrial emissions [39]. In the actual agricultural production, irrigation and fertilization was an indispensable part of the farmland around the mining area. The use of a large number of chemical fertilizers led to the accumulation of Cd in the soil. Pollution assessment and correlation analysis showed that Cd and As had a certain accumulation level in the soil of closed mining area, and the correlation between Cd and As was significant. Therefore, factor 3 was resolved as an agricultural pollution source.

The main load elements of factor 4 were Cu (90.6%) and Zn (23.2%) (Fig. 5d). Cu and Zn were the main components of motor vehicle emissions [40-42], and the wear of engine components and fuel or gasoline leakage may be the cumulative source of Cu [43]. As a fuel additive for automobiles, Zn came from tire wear and road equipment [44], which entered the surface soil through atmospheric dust [45]. The railway and road on the west side of the Qianling closed coal mine was

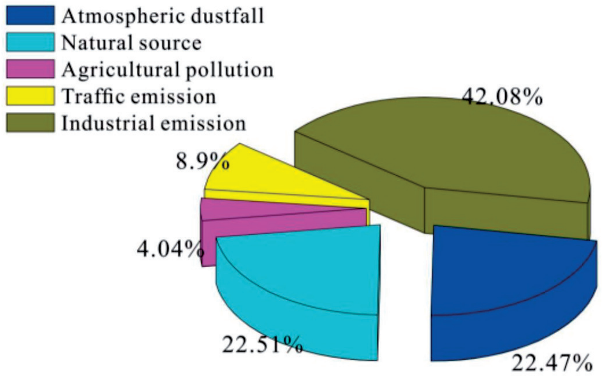

Fig. 6. Pie chart of contribution proportion of pollution sources.

the main hubs of coal transportation. From mining to closure for decades, the frequency of trains and cars was high and the traffic volume was large. Transportation was the main reason for the accumulation of Cu and Zn in the soil of Qianling closed mine. Therefore, factor 4 resolved to the traffic source.

The main load elements of factor 5 were Zn (67.8%), Pb (65.3%), and Cr (55.9%) (Fig. 5e). The vehicle emissions were cumulative sources of Cu and Zn; The fuel combustion, gasoline additives, and engine led to Pb emissions [46]. The high concentrations of Cr were related to industrial activities, and industrial waste treatment, sewage sludge, spills and residues all led to Cr enrichment [47]. The evaluation and correlation analysis of soil heavy metal pollution showed that the pollution level of Zn in the mining area was the highest compared with other elements, and there was a strong positive correlation between Zn and Pb, and a significant correlation between Zn and Cr. Before the mine closure, the emissions from coal combustion, coal chemical industry, coal electricity metallurgy and other industrial activities caused soil Zn, Pb, Cr pollution. Therefore, industrial emissions were the possible pollution sources of Zn, Pb and Cr.

According to the factor fingerprints of each heavy metal element, the contribution proportion of each pollution source was calculated, as shown in Fig. 6. The industrial emissions contributed the most to soil heavy metals (42.08%), followed by natural source (22.51%), atmospheric dust source (22.47%), traffic source (8.90%), and agricultural pollution source (4.04%). In short, human activities were the dominant factors, which 77.49% of the pollution was caused by human factors, especially coal mining, coal combustion, coal chemical industry and other industrial activities were the main sources of heavy metals pollution in the study area.

Conclusions

The conclusions drawn from this research suggested that the average contents of Zn, Mn, As, Cd and Pb exceeded the background values of surface soil in Anhui Province. Compared with the soil in Sudong, Sunan and Linhuan mining areas, it is found that the closed mines had significantly higher Pb and Zn contents. It was estimated that there were Pb and Zn pollution in the surface soil of the closed mine. The assessment results of pollution and ecological risk of soil showed that the closed coal mine was in the mild to moderate pollution and mild hazard ecological risk, in which Zn pollution degree was the largest, followed by As. The Pearson correlation and positive matrix decomposition model were applied to identify the sources of soil heavy metals, which were atmospheric dustfall (22.47%), natural factors (22.51%), agricultural activities (4.40%), transportation (8.90%) and industrial emissions (42.08%), respectively, of which 77.49% were related to human activities.

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Conflict of Interest

The authors declare no conflict of interest.

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