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ScienceDirect
Trans. Nonferrous Met. Soc. China 24(2014) 3324−3331
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Fractional distribution and risk assessment of heavy metal contaminated soil in vicinity of a lead/zinc mine

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Received 28 October 2013; accepted 24 March 2014

Abstract: The pollution characteristics of Pb, Cd, Zn, Cu and Ni in soil of lead−zinc mining area were studied. The results indicate that the contamination degree followed the sequence of Cd>Pb>Zn>Ni>Cu and concentrations of Pb, Cd and Zn exceeded corresponding limits of the Chinese National Soil Environmental Quality Standard III. The soil was extremely polluted by Cd (I_{ego}=5.26), moderately to heavily polluted by Zn (I_{ego}=2.38), heavily to extremely polluted by Pb (I_{ego}=4.13). The results of BCR three-step sequential extraction procedure show that the active Cd, Pb and Zn were relatively high and might exert adverse effects on the plants grown in the soil, while Cu and Ni existed in soil with a relatively stable form. Potential ecological risk results indicate that soils were engaging in a high potential ecological risk by pollution of Cd and should be given rise to concern.

Key words: heavy metals; chemical speciation; geoaccumulation index; potential ecological risk

1 Introduction

Heavy metals are regarded as serious pollutants of soil because of their toxicity and environmental persistence [1]. They can be migrated from the soil to other ecosystem components, such as groundwater and plants, thus affecting human health through drinking water and food chain. Health problems can be developed as a result of excessive dietary accumulation of heavy metals in human body [2]. Mining and smelting operations are important causes of heavy metal contamination in the environment due to activities such as mineral excavation, ore transportation, smelting and refining [3]. Moreover, wastewater, waste gas and solid waste generated in the process of mining and smelting activities will lead to the release and migration of heavy metals thus cause heavy metals pollution of soil near the mining area [4,5]. Nowadays, heavy metal contamination of soils in the vicinity of mining areas has been regarded as a great environmental concern [6,7]. Studies have shown that water [8], vegetables [9] and crops [10] are often contaminated by heavy metals dispersed from mining and smelting operations.

The evaluation of heavy metal pollution in soil is very important. The index of geoaccumulation (I_{geo}) has been used as a measure of bottom sediment contaminations since 1970s and numerous researchers have employed it to assess the contamination of soils and sediments [11−13]. The impacts of the background of natural geological process and the human activities on the heavy metal pollution are considered [14]. Potential ecological risk index advanced by HAKANSON [15], according to the characteristics of heavy metal and its environmental behavior, is a method to evaluate the heavy metal contamination. Heavy metal levels in the soil and ecological and environmental effects with toxicology were considered, and pollution was evaluated using comparable and equivalent property index grading method [16].

It is well known that metals in soil are presented in different chemical forms, which influence their reactivity and hence their mobility and bioavailability. Evaluating metal pollution of soils on the basis of total metal content provides little information on the mobility and bioavailability of heavy metals and thus gives poor

Foundation item: Project (2012FJ1010) supported by the National Science and Technology Major Project, China; Project (2012BAC09B04) supported by the National “Twelfth Five-Year” Plan for Science & Technology, China; Project (2012AA06202) supported by the National High-tech Research and Development Program of China
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DOI: 10.1016/S1003-6326(14)63473-7

guidance for the selection of appropriate remediation strategies for contaminated soil. A sequential chemical extraction technique fractionates heavy metals into forms of different solubilities and mobilities, and can therefore furnish potentially valuable information for predicting metal availability and metal movement in the soil [17]. Among the variety of sequential extraction procedures, the three-step scheme proposed by the community bureau of reference (BCR) and the modified version have become very popular in recent years [18]. This method can provide very useful information on metal speciation when assessing the availability of potentially toxic elements in soil [19].

In this work, the heavy metal pollution in soil of Qingjiang lead−zinc mining area in Zixing of Chenzhou, Hunan province, was studied. The concentrations of Pb, Cd, Zn, Cu and Ni were determined to study the pollution levels of heavy metals. Also, the geological evaluation of the cumulative index and potential ecological risk index were adopted to evaluate the pollution levels of Pb, Cd, Zn, Cu and Ni in the soil. Moreover, the sequential extraction of heavy metals in soil samples was performed in accordance with the modified BCR procedure, and the fraction of heavy metals consisted of the weak acid soluble, reducible, oxidizable and residual fractions. This study was to assess the levels of metals contamination and their ecological risk in soil of smelting mining area and provide a scientific basis for heavy metal pollution control.

2 Materials and methods

2.1 Sample collection and preparation

The geography coordinate of the survey site is longitude 113°17′17.76″E−113°17′36.07″E, latitude 25°45′39.29″N−25°46′1.13″N, located at Zixing, Chenzhou, Hunan province, China. Study area is located in the vicinity of a lead−zinc mine. Soil sampling was carried out in September 2012. The locations of sampling areas (*A*−*E*) are shown in Fig. 1. A total of 83 topsoil samples were collected from the 5 sampling areas. Samples were collected from a depth of 5−20 cm. Each sample was picked out from a mixture of 3−5 subsamples. The collected samples were neatly packed in polyethylene bags and transported to the laboratory. At the laboratory, any foreign adhered material was manually removed. All samples were dried at room temperature, disaggregated and sieved through 2 mm sieve for subsequent analysis.

2.2 Measuring method of total heavy metal concentrations and pH

The contents of heavy metals were analyzed by atomic adsorption (WFX200). For the determination of pH, 5 g of soil was taken in a clean and dry beaker (50 mL). Then 25 mL distilled water was added to the beaker and was thoroughly stirred with a glass rod for 0.5 h. The pH of the suspension was determined with an electrical digital pH meter.

2.3 Assessment methods

1) Geoaccumulation index (I_{geo})

The contamination levels of heavy metals in soils were assessed by geoaccumulation index (I_{geo}) [20].

$$I_{geo}=\log_2^{[C_n/(1.5B_n)]}$$

where C_n is the measured concentration of the heavy metal n in the soils, B_n is the geochemical background

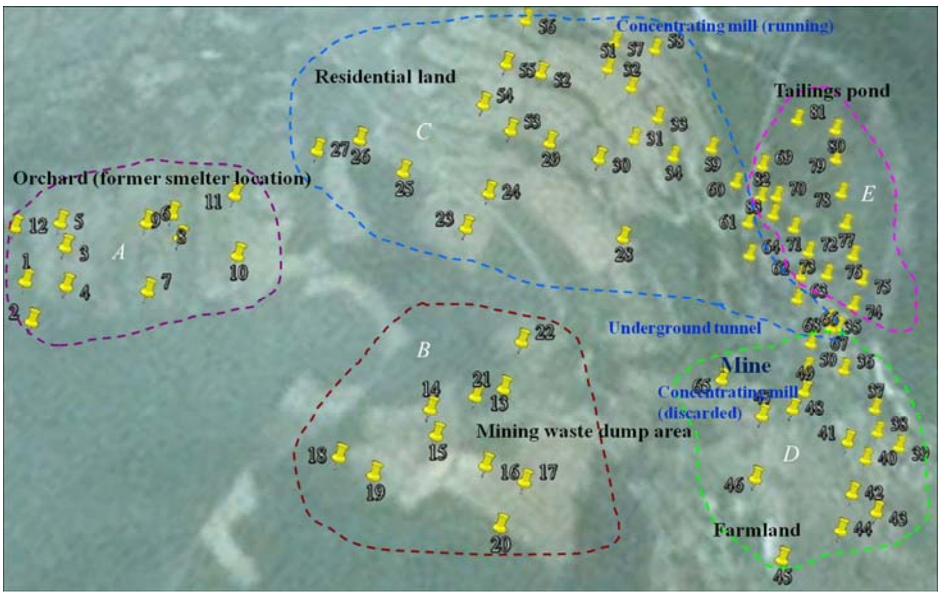

Fig. 1 Sampling areas and sampling locations

concentration of metal n, and 1.5 is the background matrix correction factor due to lithogenic effects. In this study, the heavy metals background values of soil in Hunan province were chosen as the background values for calculating the I_{geo} values.

2) Contamination factor

Assessment of soil contamination is performed by the contamination factor (C_f^i) and degree of contamination (C_d) [15,21].

$$C_f^i = C_s^i / C_n^i, \quad C_d = \sum_i^m C_f^i$$

where C_s^i is the content of metal i, and C_n^i is the reference value, baseline level, or national criteria of metal i. The concentration of the Chinese National Soil Environmental Quality Standard III was used as reference baseline in this study.

3) Ecological risk factor

An ecological risk factor (E_r^i) to quantitatively express the potential ecological risk of a given contaminant also suggested by HAKANSON [15] is

$$E_r^i = T_r^i C_f^i$$

The toxic-response factor T_r^i of heavy metals i are: T_r^{Zn} =1; T_r^{Pb} =5; T_r^{Cd} =30; T_r^{Cu} =5; T_r^{Ni} =5. The following terminologies are used to describe the risk factor: E_r^i<40, low potential ecological risk; 40≤E_r^i<80, moderate potential ecological risk; 80≤ E_r^i <160, considerable potential ecological risk; 160 ≤E_r^i<320, high potential ecological risk; and E_r^i≥320, very high ecological risk.

$$I_r = \sum_{i=1}^n E_r^i = \sum_{i=1}^n (T_r^i C_f^i) = \sum_{i=1}^n T_r^i C_s^i / C_n^i$$

The following terminology is used for the potential ecological risk index (I_r): I_r<150, low ecological risk; 150≤I_r<300, moderate ecological risk; 300≤I_r<600, considerable ecological risk; I_r>600, very high ecological risk.

2.4 Sequential extraction procedure

1) Weak acid soluble fraction

0.500 g of soil sample was treated with 20 mL of 0.11 mol/L HAc solution. This mixture was shaken in a mechanical shaker at (22±5) °C for 16 h. The extraction was separated from the soil residue by centrifugation for 20 min. The supernatant was decanted, collected and stored in bottles for analysis. The residue was washed with 20 mL deionized water, shake and centrifuge. The supernatant was decanted and discarded, taking care not to discard any of the solid residue.

2) Reducible fraction

20 mL of 0.5 mol/L $NH_2OH \cdot HCl$ solution was added to the residue from the first step. The pH value was kept at 2.0. The extraction and the residue were treated as the previous step.

3) Oxidizable fraction

5 mL of 30% H_2O_2 solution was added to the residue from the second step. The mixture was digested at (22±5) °C for 1 h and at (85±2) °C for 1 h, and the volume of liquid was reduced to less than 1.5 mL. A second aliquot of 5 mL of 30% H_2O_2 was added, the mixture was digested at (85±2) °C for 1 h, and the volume of liquid was reduced to about 0.5 mL. Finally, 25 mL of 1 mol/ L NH_4Ac (pH was adjusted to 2.0) solution was added. The extraction and the residue were treated as the previous step. The concentration of heavy metals in the extraction was determined.

4) Residual fraction

The residual fraction was calculated by the difference between the total content and all other fractional content.

The concentrations of Pb, Zn, Cd, Cu and Ni in the various extracts were determined by atomic adsorption (WFX200).

3 Results and discussion

3.1 pH

The relationship between frequency distribution of soil and pH value is given in Fig. 2. Only 2.41% of samples had pH above 8.0 and 10.84% of samples had pH less than 5.0. The pH value of soil ranged between 4.36 and 8.26 and the average pH of soil was 6.0, which indicated that the majority of soil samples were neutral to slightly acid.

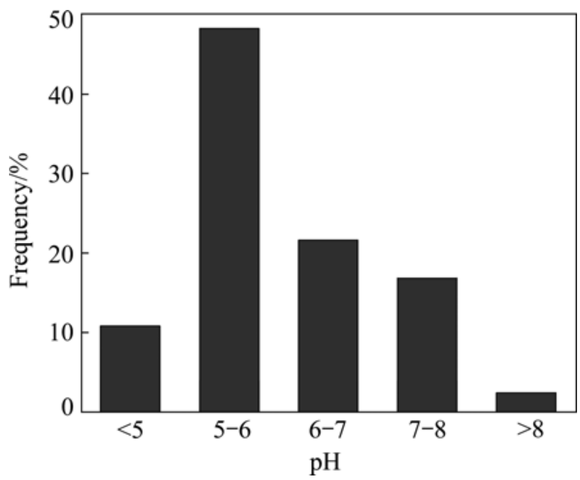

Fig. 2 Frequency distribution of soil with pH value

3.2 Heavy metal concentrations

The heavy metal concentrations of soil samples are presented in Table 1. Total concentrations of heavy metals ranged as follows: Pb 54.60−10053.90 mg/kg, Cd 0.01−114.73 mg/kg, Zn 60.44−4946.59 mg/kg, Cu

Table 1 Heavy metal concentrations in soils of mining areas

Heavy metal	Heavy metal concentration/(mg·kg^{-1})						
	Min	Max	Average	Background value	Standard (III)	Standard deviation	Median
Pb	54.60	10053.90	777.42	89.20	500.00	1545.04	298.09
Cd	0.01	114.73	7.24	0.60	1.00	14.13	2.61
Zn	60.44	4946.59	736.55	103.16	500.00	901.74	326.4
Cu	6.06	120.52	24.18	17.89	400.00	19.86	19.61
Ni	16.35	58.64	25.72	35.67	200.00	6.42	24.72

6.06−120.52 mg/kg and Ni 16.35−58.64 mg/kg. The obtained results show that the maximum concentrations of Cu and Ni in soil samples were below their corresponding limits of the National Soil Environmental Quality Standard III, which indicated that the concentrations of Cu and Ni in the soil are normal. However, high concentrations of Pb, Cd and Zn were observed in most samples. Results of statistic analyses indicated that the average concentrations of heavy metals Pb, Cd, Zn in samples exceeded their corresponding limits of the Chinese National Soil Environmental Quality Standard III with average concentrations of Pb 777.42 mg/kg, Cd 7.24 mg/kg, Zn 736.55 mg/kg. Especially for Cd, its concentration was 6 times that of the National Soil Environmental Quality Standard. This result reflected that the long-term mining and smelting activities led to significant accumulations of Pb, Cd and Zn in soils.

3.3 Assessment of heavy metal pollution

According to MULLER [22], the I_{geo} for each metal is calculated and classified as: uncontaminated ($I_{geo}\leq0$); uncontaminated to moderately contaminated ($0<I_{geo}\leq1$); moderately contaminated ($1<I_{geo}\leq2$); moderately to heavily contaminated ($2<I_{geo}\leq3$); heavily contaminated ($3<I_{geo}\leq4$); heavily to extremely contaminated ($4<I_{geo}\leq5$); extremely contaminated ($I_{geo}\geq5$). Here, 1, 2, 3, 4 and 5 represent Muller class, respectively, as shown in Fig. 3.

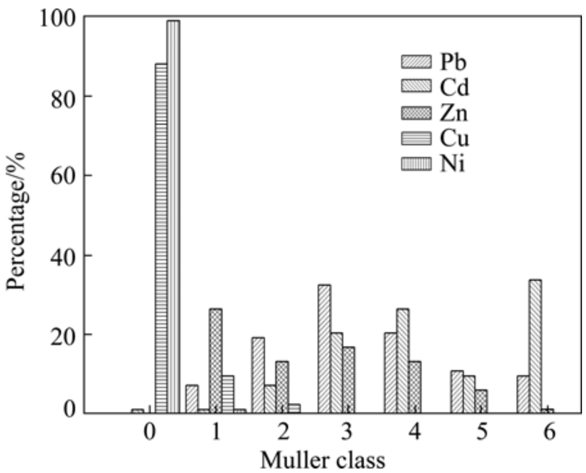

Fig. 3 Percentage of samples in Muller class

It can be found from Fig. 3 that the Cu and Ni contents of surface soil samples were near normal level, since 87.95% and 98.8% of samples are unpolluted by Cu and Ni respectively. The most serious polluted element was Cd, for 90.37% Cd was heavier than moderately polluted and 33.74% Cd was extremely contaminated. As for Pb, contaminated samples accounted for 100% and samples heavier than moderately polluted accounted for 73.49%. Zn in contaminated samples accounted for 77.11% and samples heavier than moderate pollution accounted for 20.48%.

The I_{geo} values were calculated by the heavy metals (Pb, Cd, Zn, Cu and Ni) average concentrations in soil samples. The average values of I_{geo} for each metal and their pollution levels are shown in Table 2.

Table 2 Average values of I_{geo} for each metal

Heavy metal	I_{ego}	Pollution level
Pb	4.13	Heavily to extremely contaminated
Cd	5.26	Extremely contaminated
Zn	2.38	Moderately to heavily contaminated
Cu	−0.76	Unpolluted
Ni	−0.90	Unpolluted

The results indicate that the soils of the study area can be categorized as follows: unpolluted with Cu and Ni, heavily to extremely contaminated with Pb, moderately to heavily contaminated with Zn and extremely polluted with Cd. The assessment results show that the contamination degree from strong to weak in soil is Cd>Pb>Zn>Ni>Cu.

3.4 Potential ecological risk

To further determine the environmental pollution and the ecological damage of heavy metals in the mining area soil, potential ecological risk index method proposed by HAKANSON [15] was employed. The contamination factors of Pb, Cd, Zn, Cu and Ni and their contamination degree values of samples are shown in Table 3. Contamination factors of Pb, Cd, Zn, Cu and Ni varied in the range of 0.11−20.11, 0.01−114.73, 0.12−9.89, 0.02−0.30 and 0.08−0.29, with average values of 1.55, 7.24, 1.47, 0.06 and 0.13, respectively. The contamination factors of heavy metals were ranked in the order of Cd>Pb>Zn>Ni>Cu. Pb and Cd were in a state of very high contamination and Zn was in a state of considerable contamination. The degree of contamination varied from 0.41 to 123.28, with a mean of 10.45.

Table 3 Contamination factors and contamination degree values

Sampling area	Item	C_f^i					C_d
		Pb	Cd	Zn	Cu	Ni	
A	Min	0.21	0.60	0.20	0.03	0.10	1.14
	Max	20.11	17.87	9.89	0.30	0.15	48.32
	Average	4.82	5.66	2.45	0.09	0.12	13.14
B	Min	0.11	0.55	0.14	0.02	0.08	0.90
	Max	10.23	27.01	6.00	0.29	0.29	43.82
	Average	1.87	4.23	1.13	0.09	0.14	7.46
C	Min	0.16	0.26	0.23	0.02	0.09	0.76
	Max	2.49	42.53	5.80	0.12	0.23	51.17
	Average	0.72	5.36	1.00	0.05	0.13	7.26
D	Min	0.17	0.01	0.12	0.02	0.09	0.41
	Max	4.43	114.73	3.75	0.15	0.22	123.28
	Average	0.75	9.30	0.93	0.05	0.12	11.15
E	Min	0.47	0.97	0.71	0.03	0.11	2.29
	Max	2.34	31.17	5.46	0.13	0.17	39.27
	Average	1.29	11.62	2.55	0.05	0.15	15.66
Average		1.55	7.24	1.47	0.06	0.13	10.45

For the description of contamination degree, the following terminologies had been used: C_d<5, low degree of contamination; 5≤C_d<10, moderate degree of contamination; 10≤C_d<20, considerable degree of contamination; C_d>20, very high degree of contamination. As can be seen from Table 3, the contamination degrees of sampling areas *A*, *D* and *E* were greater than 10 and less than 20, which indicated that they were within considerable degree of contamination. The contamination degrees of sampling areas *B* and *C* were greater than 5 and less than 10, which indicated that they were within moderate degree of contamination. The average contamination degree of all soil samples was 10.45, which suggested that they were within considerable degree of contamination. The order of contamination degree of each sampling area was *E*>*A*>*D*>*B*>*C*.

The ecological risk factors and potential ecological risk index are shown in Table 4.

According to Table 4, the potential ecological risk factor of Pb, Zn, Cu, Ni were much less than 40, indicating low ecological risk. The potential ecological risk factor of Cd (E_r^i) was greater than 160 and less than 320, indicating high potential ecological risk. The order of the potential ecological risk factor of heavy metals was Cd>Pb>Zn>Ni>Cu. The potential ecological risk index for each sampling area was in the order of *E*>*D*>*A*>*C*>*B*. In addition, the potential ecological risk index for sampling areas *A*, *C*, *D* and *E* was greater than 150, indicating that the potential ecological risk was moderate and above. Among them, the I_r value of sampling area *E* was greater than 300, indicating high potential ecological risk should be paid close attention to.

Table 4 Ecological risk factors and potential ecological risk index

Sampling area	E_r^i					I_r
	Pb	Cd	Zn	Cu	Ni	
A	24.09	169.94	2.45	0.47	0.59	197.54
B	9.34	126.95	1.13	0.45	0.70	138.57
C	3.59	160.88	1.00	0.25	0.63	166.35
D	3.76	280.98	0.93	0.23	0.61	286.51
E	6.47	348.73	2.55	0.26	0.72	358.73
Average	7.77	217.28	1.47	0.30	0.64	227.46

The average potential ecological risk index of Pb in the studying area was less than 40, which indicated that the ecological risk was low, but there still had the situation of E_r^i >40 in some sampling points. For example, Pb reached considerable potential ecological risk at the most polluted sampling points in the sampling area *A*. Although the content of Zn was high, but as a necessary trace element as well as its low coefficient of biological toxicity, it exerted low potential risk to the ecology. The concentrations of Cu and Ni in the soil were low, so their potential ecological risks were far lower than other heavy metals and exerted no potential harm to environment.

3.5 Fractions distribution of heavy metals

The risks associated with the presence of heavy metals are varied and depend on their chemical forms. The impact of these metals in soils is their possible transfer into water or plants, which is defined by the term of bioavailability. BCR sequential extraction procedure method was used to analyze the chemical speciation and bioavailability of heavy metals in this study. 12 soil samples (numbered S-1 to S-12) with relatively high heavy metal concentrations were selected from 83 topsoil samples to conduct BCR sequential extraction procedure. Data were expressed as different fractions with respect to the total amount in the soil. BCR results of Pb, Zn, Cd, Cu and Ni are shown in Fig. 4.

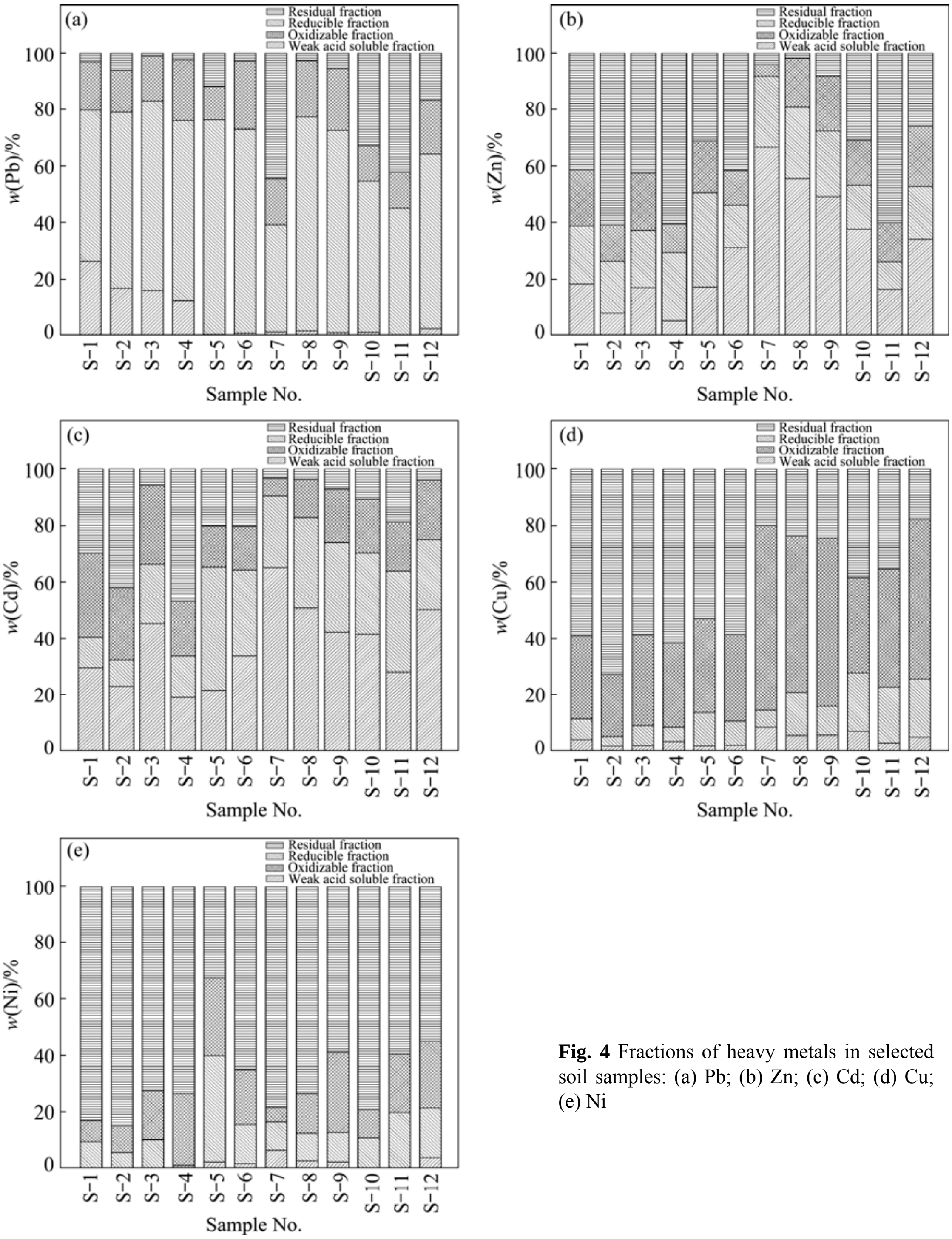

Fig. 4 Fractions of heavy metals in selected soil samples: (a) Pb; (b) Zn; (c) Cd; (d) Cu; (e) Ni

It can be seen from Fig. 4(a) that the predominant form of Pb was reducible fraction, which was in the range from 37.78% to 75.88%. The weak acid soluble fraction (0.26%−26.11%), residual fraction (1.31%−44.47%) and oxidizable fraction (11.82%−23.93%) were the other contributors, but with significantly lower Pb contents compared with the reducible fraction. This reflects the relatively high mobility and bioavailability of Pb in studied soil. The accumulation of Pb in the mobile and bioavailable forms may have increased the Pb concentration in the plants in the contaminated soil, leading to Pb toxicity in the plants.

In the studied soils, the predominant form of zinc was the residual fraction, with an average content of 34.46%. Zn was also found to present in the weak acid soluble fraction (average 29.61%), reducible fraction (average 20.76%) and the oxidizable fraction (average 15.37%), respectively. This implied that under reducing conditions, a part of zinc was unstable and easily released by dissolution. It could be inferred that the potential environmental impact of heavy metals in soils was related not only to their total concentrations and chemical forms, but also to other conditions, such as soil pH and redox potential.

Figure 4(c) shows that the predominant form of Cd was the weak acid soluble fraction, being approximately 37.41% (average) of the total Cd content in the studied soils. Approximately 25.75% (average) of the Cd was associated with the reducible fraction and approximately 19.23% of the Cd was associated with the oxidizable fraction. The smallest part was the residual fraction (17.61% of total Cd). From an environment point of view, it is notable that Cd in weak acid soluble fraction represents serious risk for surrounding ecosystems.

According to Fig. 4(d), Cu was mainly presented in the residual fraction and oxidizable fraction. The content of residual and oxidizable fraction was up to 84.5% (average). The decreasing order of fractions was residual>oxidizable>reducible>weak acid soluble. Less than 4.3% (average) Cu was in weak acid soluble fraction forms, which cannot immediately cause important environmental hazard during mobility. Because the content of Cu in the reducible fraction and weak acid soluble fraction were very low, Cu did not represent a serious environment risk.

Figure 4(e) shows that Ni was strongly associated with the residual fraction, with content ranging from 54.85% to 85.01%. The oxidizable fraction was the next important Ni retention contributor, with an average content of 17.48%. The smallest amount of this element was presented in the weak acid soluble fraction, at a level of about 1.48%. The reducible fraction contained 13.03%. Heavy metals in residual fraction are not likely to be discharged under normal environment conditions. Ni was mainly associated with the oxidizable and residual fractions, so its status in the soils was considered stable.

4 Conclusions

1) Soil samples are neutral to slightly acid. The concentrations of Cu and Ni in the soil are low while the concentrations of Pb, Cd, Zn in samples far exceed their corresponding limits of Chinese Soil Environmental Quality Standard III. Long-term mining and smelting activities lead to the significant accumulations of Pb, Cd and Zn in soils.

2) The I_{geo} values suggest that the soil samples were uncontaminated with Ni and Cu, moderately to strongly contaminated with Zn, heavily to extremely contaminated with Pb, extremely contaminated with Cd. The assessment results show that the contamination degree from strong to weak in soil is Cd>Pb>Zn>Ni>Cu.

3) BCR sequential extraction results show that the active Cd, Pb and Zn are relatively high and might exert adverse effect on the plants grown in the soil, while Cu and Ni exist in soil with a relatively stable form.

4) The potential ecological risk index for each sampling area is in the order of *E*>*D*>*A*>*C*>*B*. The order of the potential ecological risk factor of heavy metals is Cd>Pb>Zn>Ni>Cu. Soils are engaging in a high potential ecological risk by pollution of Cd and should be given rise to widespread concerns.

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某铅锌矿区土壤重金属形态分布及污染风险评价

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摘　要：对某铅锌矿区土壤的铅、镉、锌、铜和镍的污染特征进行了研究。结果表明，5 种重金属的污染程度依次为：镉>铅>锌>镍>铜，其中铅、镉和锌的浓度超过了国家土壤环境质量标准III的浓度限值。土壤受镉污染极大，受锌污染中度至严重，受铅污染严重至极严重。BCR 顺序提取形态分析结果表明，活性镉、铅和锌的含量较高，可能对土壤中的植物生长产生不利影响，而铜和镍以相对稳定的形式存在。潜在生态危害结果表明，镉污染导致土壤潜在着极大的生态风险，应当引起重视。

关键词：重金属；化学形态；地累积指数；潜在生态风险

(Edited by Xiang-qun LI)