

Accumulation Characteristics and Chemical Speciation of Cd, Zn and Pb in Soils Impacted by a Pb-Zn Mining Area

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Abstract

An exploratory study on soil contamination of Cd, Zn, Pb was carried out in the surroundings of a historical, abandoned Pb-Zn mining area in Hunan Province, China. The accumulation in soils and representative plants of Cd, Zn, Pb and their chemical speciation were investigated. The obtained results indicated that Cd, Zn and Pb presented a significant contamination compared with Environmental Quality Standards for Soils in China (GB 15618-1995). The geoaccumulation index suggested the degree of contamination: Pb > Cd > Zn. The modified BCR sequential extraction results showed that Cd, Zn, and Pb exist in soil in a relatively unstable form, and will exert a bad effect on the plants grown in the soil. Heavy metals in oranges collected in the sampling area were tested to identify the extent of pollution. The results confirmed that the sampled oranges were polluted with Pb, which exceeded the national food safety standard by 3.4–6.3 times. Heavy metals in branches and leaves showed different accumulation characteristics than the fruits.

Keywords

Heavy metals, chemical speciation, geoaccumulation index, oranges, accumulation

1 Introduction

Heavy metal pollution in soil has caused great concern since heavy metals are permanent toxic pollutants that cannot be biodegraded.¹ Heavy metals in soil may cause fatal diseases through crops and water due to easy accumulation in internal organs.² Cd and Pb are considered potential carcinogens and are associated with etiology of a number of diseases, especially cardiovascular, kidney, blood, nerve, and bone diseases.³ Serious systemic health problems can develop as a result of excessive dietary accumulation of Cd and Pb in the human body. Although Zn is an essential element, its excessive concentration in food and feed plants is of great concern because of their toxicity to humans and animals.

The metal content in soil is the sum of metals originating from natural processes and human activity. Anthropogenic activities such as metal ore mining and smelting have increased the prevalence and occurrence of heavy metal contamination in the Earth's surface, thus affecting food safety and health of humans and animals.⁴ Mining and smelting generates large amounts of tailings and other waste, and has serious environmental impact on the quality of soils. Heavy metals may be released from the mine wastes into soil and surface water systems, while the geological environment contributes to their solubility and mobility.⁵ It is essential to identify the heavy metal pollution which affects the people who live near the contaminated site. Numerous papers have assessed the risk of heavy met-

al pollution in Pb-Zn mining areas and obtained certain results. However, not much literature reports systematically the accumulation of heavy metals in soils. To be specific, S. J. Lu *et al.*⁶ discovered that the high ecological risk of the area was most likely related to acid mine drainage and mining complexes in the soil around the Pb-Zn mine in Huize County, China. J. Y. Qi *et al.*⁷ evaluated the health risks of heavy metals in soils collected from a Pb-Zn mining area with exploitation history of 60 years in southern China, and discovered that Cd and Zn were found mainly in the upper 60 cm of the soil, whereas Pb was mainly in the upper 40 cm, and concluded that there was a high health risk, especially for children. S. C. Obiora *et al.*⁸ assessed the level of heavy metal contamination of soils caused by Pb-Zn mining activities in Enyiba, southeastern Nigeria, and they revealed that the soil quality had deteriorated around the Pb-Zn mines. Insight must also be obtained into the heavy metal accumulation in soils and flora of contaminated areas. Many researchers have assessed heavy metal contamination of soil and plants around mining and smelting areas in Hunan Province.^{9–13} J. Dong *et al.*¹⁴ assessed the potential dietary risk of heavy metals in vegetables at a Pb-Zn mine site, China. Their results indicated that mining activities caused heavy metal contamination of vegetables and severe Cd health risk for the local people. B. Li *et al.*¹⁵ conducted a field survey to investigate the health risk of heavy metals in soils and vegetables around an ancient Zn-smelter in northeastern China. They found that Cd showed higher accumulation capacity in edible parts of vegetables than other metals, leaf and root vegetables were more contaminated with Cd than fruit vegetables. In addition, they claimed that cabbage, Chinese cabbage, tomato, cucumber, and green beans were suitable for growing in

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their studied area. To the best of our knowledge, there is little literature systematically focused on the contamination of the Pb-Zn mining area that we chose to investigate in the Hunan Province.

The objectives of this research were to analyse the total concentration of several heavy metals (Pb, Zn and Cd) in polluted soils near an abandoned Pb-Zn area, to analyse the chemical partitioning of heavy metals, and to carry out an assessment of the environmental risk associated with heavy metal pollution in the soil from the study area. The study aimed to provide a scientific basis for remediation of the heavy metal polluted soil in order to protect the health of the residents.

2 Materials and methods

2.1 Sample collection and preparation

The study area is located in a lead-zinc mining area, as shown in Fig. 1. The sampling area consisted of two parts: an orange orchard nearby an abandoned smelter (soil samples 1–6); uncultivated land of a tailing pond (soil sample 7–12), overgrown with grass and weeds. Sub-samples were collected from a depth of 5–20 cm. Five sub-samples were thoroughly mixed to form a composite sample. The collected samples were neatly packed in polyethylene bags and transported to the laboratory. At the laboratory, any foreign adhered material was removed manually. All samples were dried at room temperature, disaggregated and sieved through 2 mm sieve for subsequent analysis. The mass fractions of Cd and Pb were tested using the Chinese National Standard method (GB/T 17141-1997), Zn mass fractions was tested using the Chinese National Standard method (GB/T 17138-1997).

Furthermore, six representative plant samples were collected for this study. Plant samples 1–4 were representative orange samples (fruits, branches and leaves included) from the orchard. Plant samples 5 and 6 were branches and leaves of representative plants collected from the covering soil of the tailing pond. All plant samples were gently washed with tap water, rinsed with distilled water, and then sent to testing organizations with appropriate qualifications.



Fig. 1 – Sampling map
Slika 1 – Karta uzorkovanja

2.2 The assessment method

The contamination levels of heavy metals in soils were assessed using the geoaccumulation index (I_{geo}) introduced by Müller. The method has been widely employed in European trace metal studies since the late 1960s.

The geoaccumulation index (I_{geo}) is expressed as follows.

$$I_{geo} = \log_2 \left(\frac{w}{1.5 w_b} \right) \quad (1)$$

where w is the measured mass fraction of the heavy metal in the soils, w_b is the geochemical background mass fraction of metal, and 1.5 is the background matrix correction factor due to lithogenic effects.¹⁶ In this study, the heavy metals background value of soil in the Hunan Province was chosen as the background value for calculating the I_{geo} values.

According to Muller, the I_{geo} for each metal is calculated and classified as: uncontaminated ($I_{geo} \leq 0$); uncontaminated to moderately contaminated ($0 < I_{geo} \leq 1$); moderately contaminated ($1 < I_{geo} \leq 2$); moderately to heavily contaminated ($2 < I_{geo} \leq 3$); heavily contaminated ($3 < I_{geo} \leq 4$); heavily to extremely contaminated ($4 < I_{geo} \leq 5$); extremely contaminated ($I_{geo} \geq 5$).

3 Results and discussion

3.1 Mass fractions of heavy metals in soils

The mass fractions of heavy metals in the soil samples are shown in Table 1, as well as the heavy metal background value of the soil in Hunan Province (V_b), and the National Soil Environmental Quality Standard II (GB15618-1995) (V_n) limits. According to data from Table 1, the average mass fractions of Pb, Cd and Zn are much greater than V_b and V_n , even the minimum mass fraction of the heavy metals. The average mass fraction of Cd was as high as 86.93 times the V_n . The minimum value of Cd mass fraction in the study area was as much as 8.70 mg kg^{-1} , more than 28 times the V_n . The average mass fraction of Zn and Pb were 12.18 and 12.29 times the V_n , respectively. The coefficient of variation for Pb and Cd were relatively high. Especially for Pb, the coefficient of variation was as high as 1.10. This showed that the distribution content of Pb and Cd are uneven in the study area, and the contents of heavy metals in different sampling points are quite different.

3.2 Pollution assessment of heavy metals in soils

The I_{geo} values for Cd, Zn and Pb in soil samples are presented in Table 2. The geoaccumulation index revealed that soils near the abandoned Pb-Zn smelter were moderately to strongly contaminated with Zn, and extremely contaminated with Pb and Cd. The assessment results indicated that the contamination degree of three heavy metals follow the sequence of $\text{Cd} > \text{Pb} > \text{Zn}$.

Table 1 – Mass fraction of heavy metals in topsoils
 Tablica 1 – Maseni udjeli teških metala u površinskom sloju tla

	Cd	Zn	Pb
maximum mass fraction/mg kg ⁻¹ najveći maseni udjel/mg kg ⁻¹	114.73	4 946.59	10 053.90
minimum mass fraction/mg kg ⁻¹ najmanji maseni udjel/mg kg ⁻¹	8.70	1 420.26	770.61
average mass fraction/mg kg ⁻¹ prosječni maseni udjel/mg kg ⁻¹	26.08	2 436.43	3 071.63
standard deviation/mg kg ⁻¹ standardno odstupanje/mg kg ⁻¹	28.74	909.35	2 929.92
coefficient of variation/% koeficijent varijacije/%	1.10	0.37	0.95
background value of soil in Hunan/mg kg ⁻¹ pozadinska koncentracija u tlu u Hunanu/mg kg ⁻¹	0.06	95	27
National Soil Environmental Quality Standard II/mg kg ⁻¹	0.30	200	250

Table 2 – I_{geo} values for each metal in the study area
 Tablica 2 – I_{geo} metala u istraživanom području

Soil samples Uzorci tla		I_{geo}		
		Cd	Zn	Pb
1–6	minimum minimum	7.18	4.30	4.83
	maximum maksimum	10.90	5.70	6.36
	average prosjeak	7.93	4.85	5.16
	contamination level razina kontaminacije	EC*	MC**–SC***	EC*
7–12	minimum minimum	7.91	3.90	6.87
	maximum maksimum	9.02	4.85	8.54
	average prosjeak	8.78	4.36	7.40
	contamination level razina kontaminacije	EC*	MC**–SC***	EC*
1–12	average prosjeak	8.36	4.61	6.28
	contamination level razina kontaminacije	EC*	MC**–SC***	EC*

* EC – extremely contaminated / ekstremno kontaminirano

** MC – moderately contaminated / umjereno kontaminirano

*** SC – strongly contaminated / jako kontaminirano

Specifically, soil samples (1–6) near the smelter showed the same contamination level as samples collected from the topsoil of the tailing pond (7–12), as well as the whole study area. The most serious pollution of soil with Cd and Zn appeared in the area near the abandoned smelter, while the tailing pond was most seriously contaminated with Pb. This indicated that Cd and Zn were the most possible pollutant elements in the soils near the Pb-Zn smelter plant, and Pb was higher in the tailing pond topsoil probably because the terrain is lower than the smelter plant.

3.3 Chemical forms of heavy metals in soils

Four soil samples (S-2, S-4 in the orchard area, and S-7, S-11 in the tailing pond area) were picked out to analyse the chemical forms of Cd, Zn and Pb through BCR sequential extraction procedures. S-4 and S-7 represented the highest Cd concentration in corresponding area, while S-2 and S-11 were chosen for their lowest Cd concentration in the respective sampling area. The results are shown in Fig. 2.

It can be seen from S-2 and S-4 in Fig. 2 that the distributions of fractions of Cd, Zn and Pb in the same sampling area were similar to a certain extent. Cd and Zn existed mainly in residual form, while Pb existed mainly in reducible fraction both in S-2 and S-4 near the abandoned smelter. Their fractions of weak acid soluble form plus reducible form were less than 40 %, indicating that Cd and Zn existed in the soil for a long time, and gradually transformed into a more stable form. Unlike Cd and Zn, the contents of residual Pb were very low, with a fraction of less than 7 %. Their reducible forms were predominant, accounting for a high fraction (53.54 % in S-4 and 62.16 % in S-2) of total concentration. This indicated that the active Pb was relatively high, and might exert a harmful effect on plants grown in the soil.

Different from samples near the abandoned smelter, the samples (S-7, S-11) collected from the top of the tailing pond showed disparities in Cd and Zn speciation, and similarity in Pb speciation. Fig. 2 shows that the percentages of reducible fraction and residual fraction of Pb were incredibly close both in S-7 and S-11, and they had hardly any weak acid soluble fraction of Pb, meaning that Pb in the soils would not express toxicity in normal circumstances. However, Cd and Zn in S-7 mainly existed in weak acid soluble fraction (over 60 %) and reducible fraction (over 20 %), with the aggregate percentages of over 90 %, suggesting that Cd and Zn in S-7 were highly active, and might cause serious environmental pollution as the concentrations of Cd and Zn in S-7 were extremely high. While the

percentage of weak acid soluble fraction and reducible fraction of Cd in S-11 was reduced to 63.76 %, it is still toxic, however. On the contrary, the dominating form for Zn in S-11 was residual fraction (60.19 %).

Overall, the chemical speciation of Cd, Zn and Pb were relatively toxic and active in the study area. Samples near the smelter showed remarkably higher percentages of weak acid soluble fraction and reducible fraction than that of samples in the tailing pond. In addition, Cd and Zn in soils from the tailing pond showed dramatically higher activity than that of soils near the smelter. In particular, the speciation of heavy metals in soils near the smelter manifested similarity regardless of pollution conditions. Conversely, the percentage of active fraction of Cd and Zn in the soils from the tailing pond was higher as the concentration was higher.

3.4 Accumulation of heavy metals in plants

3.4.1 Heavy metals in fruit

Heavy metals in oranges collected in the sampling area were tested to identify the extent of pollution. This study examined the concentrations of heavy metals from the fruit of the orange trees grown in the study area, and the results for the four samples (Z-1–Z-4) are shown in Table 3. In

order to compare the concentration of heavy metals to the national standard, the dry weight of oranges was used.

Table 3 – Content of heavy metals in orange samples
Tablica 3 – Teški metali u uzorcima naranči

Samples Uzorci	mass fraction / mg kg ⁻¹ maseni udjel / mg kg ⁻¹		
	Cd	Zn	Pb
Z-1	0.020	10.46	0.73
Z-2	0.008	4.29	0.44
Z-3	0.015	10.34	0.72
Z-4	0.014	1.70	0.64
average prosjeak	0.014	6.70	0.63
(GB 2762-2005)	0.05	–	0.10

According to Table 3, the content of Cd in four orange samples are far less than the permissible level of fruits prescribed by the Limit Content of Pollutants in Food

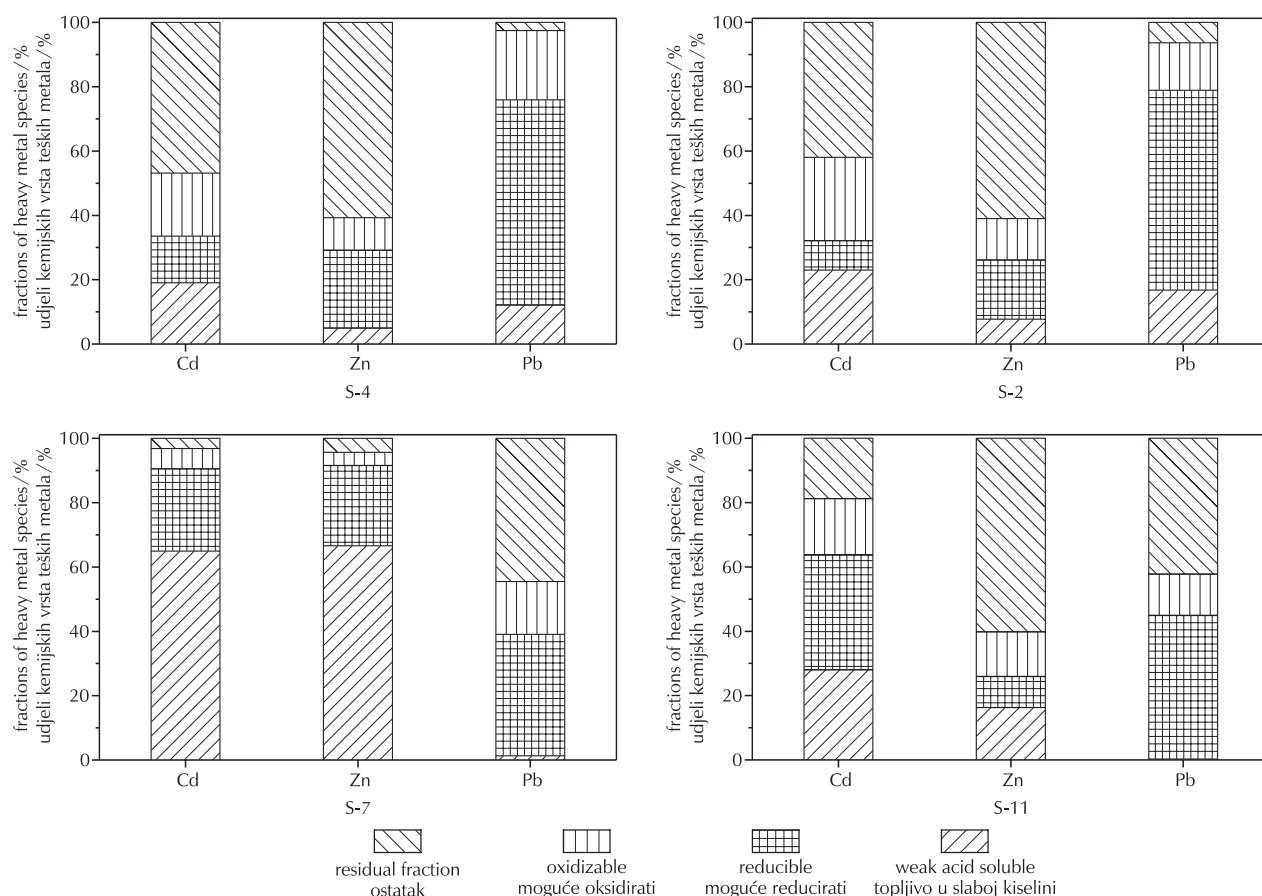


Fig. 2 – Chemical forms of heavy metals in representative soil samples
Slika 2 – Kemijski oblici teških metala u reprezentativnim uzorcima tla

(GB2762-2005), indicating that the orange samples were not polluted by Cd. The reason for this may be that the fruit is not the organ that can accumulate Cd. The Cd content in the orange samples followed the sequence: Z-1 > Z-3 > Z-4 = average value > Z-2. The content of Pb in the oranges was very high, exceeding the national food safety standard by 3.4–6.3 times. Even the content of Pb in the Z-2 (Z-2 has the lowest content of Cd in all samples) is more than four times the level prescribed in the national standard. The Pb content in the orange samples followed the sequence: Z-1 > Z-3 > Z-4 > average value > Z-2. Since Zn is a necessary element for the growth of plants, the relevant standard of Zn according to the Limit Content of Pollutants in Food (GB2762-2005) is absent. The Zn content in the orange samples followed the sequence: Z-1 > Z-3 > average value > Z-2 > Z-4.

3.4.2 Heavy metals in branches and leaves

Heavy metal accumulation characteristics in plants was studied by examining Cd, Zn, Pb concentration in fresh branches and leaves of the plant samples. The results are shown in Table 4. The Cd, Zn and Pb content followed the sequence: Z-1 > Z-6 > Z-4 > average value > Z-1 = Z-2 = Z-4; Z-3 > Z-1 > average value > Z-5 > Z-2 > Z-4 > Z-6; Z-3 > Z-4 > average value > Z-1 > Z-6 > Z-5 > Z-2, respectively. It can be concluded that the plants in the tailing pond accumulated more Cd and less Zn/Pb than had the branches and leaves near the smelter. Compared with the fruits, it was found that heavy metals showed different sequences in samples. To be specific, the content of Pb in fruit sample Z-1 was the highest, followed by Z-3 and Z-4, while the content of Pb in branches and leaves in Z-1 was followed by Z-3 and Z-4.

Table 4 – Content of heavy metals in branches and leaves
Tablica 4 – Teški metali u granama i lišću

Sample Uzorak	mass fraction / mg kg ⁻¹ maseni udjel / mg kg ⁻¹		
	Cd	Zn	Pb
Z-1	0.1	214.2	2.7
Z-2	0.1	110.5	2.2
Z-3	0.2	370.9	19.5
Z-4	0.1	107.8	9.0
Z-5	3.5	140.8	2.5
Z-6	2.8	80.1	2.6
average prosjek	1.34	182.76	7.26

4 Conclusion

In summary, Cd, Zn, Pb present significant soil contamination in the historical, abandoned Pb-Zn mining area. The

concentrations of Cd, Zn, Pb in soils are much higher than the level prescribed by the Environmental Quality Standards for Soils in China. The pollution assessment indicated that the soil in the study area was moderately to strongly contaminated with Zn, and extremely contaminated with Pb and Cd. The chemical forms of heavy metals suggested that Cd, Zn and Pb exist in the soil in a relatively unstable form, and will have an adverse effect on the plants grown in the soil. The oranges grown in the research area proved to not be polluted with Cd but polluted with Pb, exceeding the national food safety standard by 3.4–6.3 times. Heavy metals in plant branches and leaves showed different accumulation characteristics that that of fruits. Greater attention to food safety in the sampling area should be paid.

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List of abbreviations and symbols Popis kratica i simbola

- BCR – European Community Bureau of Reference
– Ured za standardizaciju Europske zajednice
- I_{geo} – geoaccumulation index
– geoakumulacijski indeks
- V_b – heavy metals background value of soil in Hunan Province
– pozadinski sadržaj teških metala u tlu u pokrajini Hunan
- V_n – heavy metals limit II value in National Soil Environmental Quality Standard (GB15618-1995)
– granična vrijednost sadržaja teških metala prema standardima GB15618-1995
- w – measured mass fraction of heavy metal in soils
– mjereni maseni udjel teških metala u tlu
- w_b – geochemical background mass fraction of heavy metal in soils
– pozadinski geokemijski maseni udjel teških metala u tlu

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SAŽETAK

Akumulacija i specijacija kadmija, cinka i olova u tlu na području rudarenja olova i cinka

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Istražena je kontaminacija kadmijem, cinkom i olovom oko napuštenog područja rudarenja olova i cinka u kineskoj pokrajini Hunan. Proučena je akumulacija u tlu i reprezentativnim biljkama te kemijska specijacija teških metala. U usporedbi sa standardima GB15618-1995 zabilježena je značajna kontaminacija kadmijem, cinkom i olovom. Prema geokemijskom indeksu razina kontaminacije slijedi niz Pb > Cd > Zn. Modificirana sekvencijska ekstrakcija prema BCR-u pokazuje da se teški metali u tlu nalaze u obliku relativno nestabilnih oblika i da će nepovoljno utjecati na biljni svijet. Zagađenje je ispitano na uzorcima naranči iz istraživanog područja. Naranče su kontaminirane olovom, a sadržaj olova veći je od 3,4 do 6,3 puta od razine koju dopuštaju nacionalni standardi za sigurnost hrane. Akumulacija teških metala u lišću i granama pokazuje drugačije karakteristike.

Ključne riječi

Teški metali, kemijska specijacija, geoakumulacijski indeks, naranče, akumulacija

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