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Metals Contamination in Soils and Vegetables in Metal Smelter Contaminated Sites in Huangshi, China

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Abstract This study investigated the source and magnitude of metal contamination in soils and vegetables collected in the vicinity of the Daye smelter, China. Results showed that soils and vegetables were heavily contaminated by cadmium (Cd) and lead (Pb). The average levels of Cd and Pb in vegetables were 0.21 and 3.28 mg/kg fresh weight, respectively. Community Bureau of Reference (BCR) operational speciation analysis indicated that the source of metals in soils probably resulted from sewage irrigation and contaminated sediment. Transfer and correlation coefficients were also calculated to evaluate the bioavailability of metals to vegetables. This investigation highlights the potential risk to local residents via consumption of vegetables.

Keywords Metals · BCR-operational speciation · Soil · Vegetable

Metal contamination in agricultural soils is of increasing concern due to food safety issues and potential health risks via consumption of contaminated vegetables (Cui et al., 2004; Wang et al., 2005). Elevated levels of metals in agricultural soils are the result of atmospheric deposition, wastewater irrigation, sludge amendment, and fertilizer application, as well as industrial activities, in particular the metallurgical industries (N et al., 2001; Kachenko and Singh, 2006). Metal pollution in soils and crops from smelter facilities has been studied to a considerable extent. Fernandez-Turiel et al. (2001) found elevated levels of metals in urban soils located within the vicinity of a smelter in Lastenia, Argentina. The metals found at elevated concentrations were lead (Pb) (31–8714 mg/kg), cadmium (Cd) (0.27–30.68 mg/kg), copper (Cu) (21–242 mg/kg) and zinc (Zn) (44–4637 mg/kg). A study of smelter-contaminated soils by Burt et al. (2003) found elevated levels of Cd, Cu, Pb, and Zn in the vicinity of a copper smelter in the Anaconda and Deer Lodge Valley area of Montana, USA. Cui et al. (2004) also found that the soils and vegetables were heavily contaminated by Cd and Pb in the vicinity of the Nanning smelter, China. Large smelters are often located in the suburbs of cities in many countries, and have caused many unfavorable changes in the quality of the soil, water, sediment and crops in the vicinity of the works. Of the pollutants, those most harmful to the environment are metals, including cadmium, lead, copper, chromium, zinc, and nickel. In the present study, we chose the Daye smelter, nationally famous and representative of those in developing countries. The smelter was established in 1953, producing 263,000 t of copper (Cu) and 4 t of gold (Au) annually. Previous studies have found elevated levels of Cd in arable soil, primarily in the nearby areas surrounding the smelter (Bi et al., 2003; Chen et al., 2003), but no information was obtained on cultivated crops and the chemical fractionation of metals in agricultural soils in the study area. The chemical speciation of metal in polluted soil is a good indicator of its bioavailability, mobility or toxicity, because these properties depend basically on the chemical association of different components in soil (Tokalioğlu and Kartal, 2003). To our knowledge, there are few systematic published studies on metal contamination in

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Chinese vegetable-growing regions, particularly in sites contaminated by metal smelters. In reality, the systematic study of metal contamination on the whole water–soil–crop ecosystem is essential in order to understand its detrimental effects and to undertake effective remediation measures.

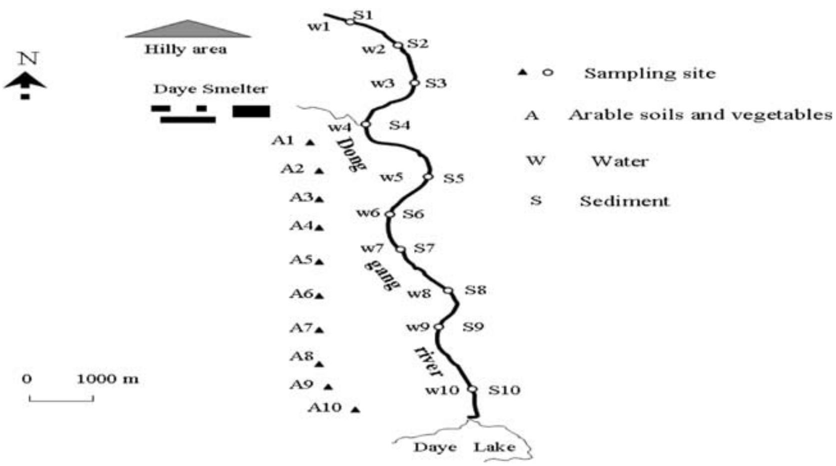
In the present study, samples of soil, water, sediment, and vegetables were collected from a multiple-metal contaminated area. Cadmium, lead, copper, chromium, zinc, and nickel in all samples were determined and the Community Bureau of Reference (BCR)-operational speciations of metals were also analyzed to evaluate the metals' availability from soil to vegetables. The objectives of this study were: (i) to investigate the distribution and concentrations of metals in different media of the water-soil-crop ecosystem and to examine the relationship between them; (ii) to determine the chemical fractions of metals in the agricultural soil, to assess the current and potential risk to the studied area, and to evaluate the metals' bioavailability.

Materials and Methods

The study area is located near Daye smelter in the suburb of Huangshi, the second largest city in Hubei province in central China. Detailed locations of the smelter and sampling sites are shown in Fig. 1. A total of 10 topsoil (0–20cm) samples, 10 subsoil (30–40cm) samples, 10 water samples, 10 sediment samples, and 10 vegetable samples were collected from the study area. Topsoil, subsoil, and vegetable samples were taken from the same sites. From each plot topsoil and subsoil samples were collected with a stainless-steel shovel. Five random samples from each soil plot were taken and bulked together as one composite sample. At the same time, vegetable sample (*Brassica chinensis* L.) was collected on the plot where soil sample was taken. Samples of soil and vegetables were stored in polyethylene bags in the field, and were transferred to the laboratory within three hours for sample processing. Acidified (HNO_3 at 1.0%) water samples were collected in 250 ml polyethylene bottles and sediment samples were also taken from the same sites.

The soil and sediment samples were air-dried at room temperature, and mechanically ground using a wood roller to pass through a 200-mesh sieve. This fine material was used to determine the total metal content and the speciations of metal in the soil and sediment. The total organic carbon in soil and sediment was determined by the modified Walkley and Black method (dichromate oxidation and titration, McCleod, 1975). A 1.0g subsample of soil and sediment was microwave digested in a closed polyvinyl-fluoride vessel with aqua regia using MRAS-5 (CEM, USA). The revised BCR method was performed to analyze the speciations of metals (Rauret et al., 1999) and the residual fraction was digested as for the soil and sediment samples. Vegetables were washed and the inedible parts were removed immediately. The edible parts of the vegetables were washed with deionized water produced by Simplicity-185 deionized water equipment (Millipore, USA), blotted dry with tissue paper and weighed. All the vegetable samples were then dried in an oven at 78°C for 72 h to constant weight, weighed again, and ground using a smash instrument (IKA, Germany) and a homogenizer (IKA, Germany). A 0.5 g subsample of plant powder was also microwave digested with concentrated nitric acid. Concentrations of Cd, Zn, Cr, Cu, Pb, and Ni in all samples were determined by a Perkin–Elmer AA800 atomic absorption spectrometer with an autosampler (combined with flame atomic absorption spectrometer (FAAS) and graphite furnace atomic absorption spectrometer (GFAAS)) according to standard analytical procedures. Samples of soil, sediment, and plant with certified concentration of metals (GSS-15, GSD-8 and GSV-2, respectively, China National Center for Standard Materials) were included for quality assurance. All the experiments were carried out in the Key Lab of Bio-geology and Environmental Geology of the China Ministry of Education.

Fig. 1 Localization of the Daye Smelter and the sampling sites



Results and Discussion

The total concentrations of metals Cd, Zn, Cr, Cu, Pb, and Ni in the topsoil and subsoil from arable land are present in Table 1. The limit values for metals set by the State Environmental Protection Administration of China (SEPAC, 1995) in soils are also shown in Table 1. The analytical results indicated that the total concentrations of metals were elevated in surface layers for all samples relative to the underlying subsoils. Furthermore, Cd contents in all soil samples were 8 to 20 times higher than the limit level; Cu and Pb concentrations slightly exceeded the limit values in some samples from the nearer sample sites to the smelter, while the other samples did not exceed the threshold value; Cr, Ni, and Zn contents in all soil samples were below the limit values. The contamination of topsoil with Cd, Pb, and Cu was more serious than that of the subsoil. This result was similar to previous studies. Storm et al. (1994) found higher concentrations of Cd, Pb, Zn, and Cu in the forest litter of smelter-contaminated soils than in underlying mineral horizons, with these elements persisting after smelting was discontinued. Burt et al. (2003) also found that the total contents of Cu, Zn, Pb, and Cd were elevated in surface layers relative to the underlying subsoils. The decrease in trace element concentration below the surface may result from the humus-enriched surface serving as a sink for trace elements, slowing humification and remobilization of metals in the soil. The effect of organic matter was ubiquitous in the soil. As shown in Table 1, the concentration of total organic carbon (TOC) was significantly higher in the topsoil than the subsoil. Therefore, the decrease in TOC could account for the decrease in trace element concentration below the surface, and the higher proportion of oxidizable fraction (including those bound to organic matter speciation) in the topsoil could also supported it.

The speciations of Cd, Cu, and Pb in all soil samples were analyzed in this study. The recovery values of these three metals range from 86 to 126%, indicating good agreement between the total concentration and the sum of BCR results. There was also good reproducibility between the two replications of the sequential fractionation. These results are in agreement with Tessier et al. (1979) and Burt et al. (2003), who concluded that the precision and accuracy of the sequential extraction procedure was inherently good, the limiting factor being the inherent heterogeneity of the specimen. In the present study, all soil samples were passed through a 200-mesh sieve to guarantee good homogeneity.

Table 1 The total concentrations and standard deviation (in parentheses) of metals and total organic carbon (TOC) of the soil samples

Soil samples		Metals, mg/kg						TOC (%)
		Cd	Cr	Cu	Ni	Pb	Zn	
A1[a]	T[b]	17.9(0.2)	3.3(0.3)	573.4(1.7)	7.9(0.3)	684.5(2.8)	97.3(2.1)	28.7
	S[b]	10.1(0.2)	1.9(0.1)	111.8(2.1)	2.3(0.3)	568.3(3.8)	60.3(3.3)	19.7
A2	T	20.7(0.1)	2.7(0.2)	644.1(2.0)	8.5(0.4)	715.3(2.0)	87.7(2.2)	31.1
	S	14.2(0.1)	1.0(0.1)	102.5(3.0)	3.9(0.2)	500.4(2.0)	44.4(1.9)	20.4
A3	T	17.5(0.2)	2.6(0.2)	529.1(2.0)	9.5(0.3)	694.3(2.4)	94.0(3.0)	19.9
	S	8.9(0.2)	1.1(0.1)	87.7(2.0)	5.6(0.2)	487.5(1.4)	32.5(1.6)	15.6
A4	T	16.8(0.2)	3.1(0.2)	522.7(2.3)	9.8(0.4)	434.5(2.8)	87.6(3.0)	26.8
	S	11.1(0.3)	2.0(0.1)	110.3(3.1)	3.0(0.3)	398.3(3.8)	54.8(2.2)	19.0
A5	T	15.8(0.3)	2.9(0.2)	94.7(1.1)	8.8(0.4)	195.2(1.9)	76.9(3.2)	31.8
	S	13.1 (0.1)	1.5(0.1)	70.3(1.3)	3.5(0.2)	147.4(2.0)	54.5(2.0)	21.3
A6	T	15.5(0.3)	2.5(0.3)	120.2(2.0)	7.9(0.3)	198.3(2.4)	94.1(3.2)	25.5
	S	13.1(0.2)	1.2(0.1)	99.0(1.8)	4.7(0.2)	127.5(1.4)	67.7(2.9)	20.7
A7	T	14.4(0.23)	3.2(0.2)	112.2(2.1)	11.9(0.4)	200.5(2.8)	81.5(2.3)	24.9
	S	12.1(0.2)	2.0(0.1)	96.9(1.9)	6.6(0.1)	188.3(3.8)	62.6(1.9)	19.2
A8	T	15.8(0.3)	2.8(0.2)	101.1(2.1)	6.5(0.3)	275.2(1.9)	88.4(3.3)	25.7
	S	8.1(0.1)	1.7(0.1)	90.0(1.7)	2.5(0.4)	173.4(2.0)	47.5(3.0)	20.2
A9	T	12.2(0.3)	2.5(0.2)	108.1(1.3)	6.0(0.3)	310.4(2.6)	91.4(3.3)	26.6
	S	9.1(0.1)	1.4(0.2)	88.9(2.0)	3.6(0.4)	188.5(1.8)	56.6(2.1)	18.2
A10	T	11.4(0.2)	2.9(0.3)	114.2(2.1)	5.7(0.4)	294.7(2.1)	89.7(3.0)	32.4
	S	8.2(0.1)	1.9(0.2)	100.1(2.4)	4.6(0.5)	270.5(2.6)	57.4(2.2)	21.3
Limit[c]		1.0	300	400	200	500	500	

[a] sampling site. [b]T-topsoil, S-subsoil. [c]limit values in soil

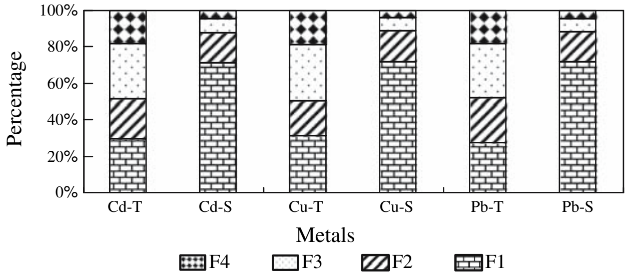


Fig. 2 Average percentages of Cd, Cu, and Pb associated with the acid soluble fraction (F1), reducible fraction (F2), oxidizable fraction (F3), and residual fraction (F4) in topsoil (T) and subsoil (S)

As shown in Fig. 2, the proportions of Cd, Cu, and Pb fractions were remarkably comparable either in the topsoil or in the subsoil, but were quite different between them. The proportion of the acid soluble fraction (F1) was about 25% in topsoil, while F1 was the predominant fraction over the other three fractions within the subsoil. In addition, the sum of reducible (F2) and oxidizable (F3) fractions exceeded the content of residual fraction (F4) in both the topsoil and subsoil, although the proportion of residual fraction in the topsoil was higher than that in the subsoil. For instance, the percentages of the four fractions (F1, F2, F3, and F4) for Cd were 27–33%, 17–26%, 28–41%, and 9–19% of the total Cd in the topsoils, respectively. Nevertheless, the acid soluble fraction accounted for over 70% in most of the subsoils. The difference in fractions between the topsoil and subsoil did not correlate with pervious studies. In general, the acid soluble fraction was elevated in surface samples and decreased abruptly below this depth. The residual fraction is relatively low in the topsoil but increases significantly with depth (Kabala and Singh, 2001; Burt et al., 2003). Nevertheless, in the current study, the proportion of the acid soluble fraction was much higher in the subsoil than the topsoil, but the residual fraction was higher in the topsoil than in the subsoil. This implied that complex sources of metals Cd, Cu, and Pb might exist. It was reported that local farmers mixed the arable soil with sediment from the Donggang River during the 1970s. At that time, smelting sewage was directly poured into the river and the sediment accumulated metals. The sediment and the irrigation water samples collected from Donggang River were also analyzed and the results (Table 2) indicated that the concentrations of Cd, Cu, and Pb were significantly higher in the sediment and water than in the background levels. Consequently, long-term irrigation with polluted water would lead to the contamination of the soil.

The chemical speciation analysis (Fig. 3) also confirmed that the proportion of fractions in the sediment was significantly similar to the subsoil. Therefore, it could be inferred that the contaminated sediment mixed with the soil was applied to a depth where it was far from weathering and anthropogenic disturbance, and where the acid soluble fraction of metals remained. The difference in residual fraction between the topsoil and the subsoil requires further study.

The concentrations of Cd, Cu, and Pb in vegetable (*Brassica chinensis* L.) samples (Fig. 4) were also investigated in this study. The metal contents were expressed on the basis of fresh weight (FW). The levels of Cd, Cu, and Pb in the edible parts of the vegetables ranged from 0.15 to 0.25 mg/kg (with an average as 0.21 mg/kg), 0.54 to 0.98 mg/kg (with an average as 0.80 mg/kg), and 2.44 to 3.98 mg/kg (with an average as 3.28 mg/kg), respectively. Both Cd and Pb in all samples exceeded the Chinese tolerance limit levels for food (SEPAC, 1994) and posed a potential risk to local residents.

The transfer coefficient quantifies the relative differences in the bioavailability of metals to plants and is a function of both soil and plant properties. In this study, the coefficient is calculated by dividing the concentration of a metal in the vegetable matter (DW) by the total metal concentration in the topsoil. Higher transfer coefficients

Table 2 The total concentrations and standard deviation (in parentheses) of metals in the sediment and water samples

No.	Cd		Cu		Pb	
	Sediment mg/kg	Water μg/L	Sediment mg/kg	Water μg/L	Sediment mg/kg	Water μg/L
1	7.3(0.1)	0.3(0.004)	1245.4(12.3)	0.1(0.002)	3473.3(12.6)	0.1(0.003)
2	8.3(0.1)	0.3(0.005)	1431.7(21.7)	0.2(0.005)	3599.7(20.2)	0.1(0.004)
3	9.3(0.2)	0.3(0.002)	1688.6(28.4)	0.2(0.001)	4587.3(13.5)	0.1(0.002)
4	18.7(0.1)	2.7(0.035)	2452.2(25.6)	2.0(0.012)	5860.6(23.3)	0.1(0.003)
5	31.4(0.2)	2.4(0.019)	2836.4(19.8)	1.4(0.026)	6500.2(17.4)	0.2(0.012)
6	38.4(0.1)	2.4(0.025)	3166.3(32.2)	1.2(0.032)	7683.5(25.6)	0.2(0.006)
7	29.7(0.2)	1.7(0.016)	2560.6(24.7)	0.7(0.003)	7300.8(20.3)	0.2(0.007)
8	26.3(0.1)	1.3(0.010)	1873.1(18.8)	0.2(0.006)	7124.4(18.6)	0.1(0.003)
9	20.1(0.2)	0.1(0.002)	1200.8(17.5)	0.1(0.001)	5411.2(20.5)	0.2(0.005)
10	11.2(0.2)	0.1(0.001)	783.5(11.4)	0.1(0.001)	3067.6(13.3)	0.1(0.005)

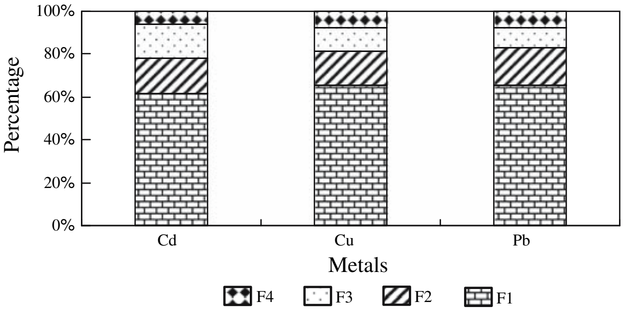


Fig. 3 Average percentages of Cd, Cu, and Pb associated with the acid soluble fraction (F1), reducible fraction (F2), oxidizable fraction (F3), and residual fraction (F4) in sediment from Donggang River

reflect relatively poor retention in soils or greater efficiency of plants to absorb metals. Low coefficients reflect the strong sorption of metals to the soil colloids (Cui et al., 2004; Kachenko and Singh, 2006). Table 3 outlines the range of transfer coefficients (Cd, Cu, and Pb) calculated for the vegetable samples. The table also contains a range of generalized transfer coefficients, which have been suggested by Kloke et al. (1984). Kloke's transfer coefficients are based on the root uptake of metals, with no consideration given to aerial deposition and foliar adsorption of elements. Most of the soil–plant transfer coefficients from the study area were below the suggested range reported by Kloke et al. (1984). Only two vegetable samples exceeded the suggested coefficient range for Pb, and the transfer coefficients in the vegetable samples were similar to the results reported by Cui et al. (2004). As shown in Table 3, the mean value of the transfer coefficient for Cd was higher than for Cu and Pb, which may indicate that Cd in soil was more available to vegetables.

The main pathway of trace elements into plants is via their roots. The degree of contaminating metal uptake by plant roots is dependent on many factors, including the magnitude and chemical form of the trace elements present; soil pH, moisture, aeration, temperature, organic matter, and phosphate content; the presence or absence of competing ions; the plant species, rooting depth, age; and seasonal growth effects (Tokalioğlu and Kartal, 2003). One of the most important applications of chemical fractionation of metals in soils is to estimate bioavailability to plants. For this purpose, a correlation analysis is performed to evaluate the relationship between the extractable fractions of the metals from soils and the metal concentrations in plants. If the accumulation of an element by a plant correlates significantly with the extractable fraction in soils, it can be assumed that the extractable fraction is readily available to plants. The correlation coefficients between extractable fractions of the metals from the topsoil and the metal contents in vegetables are based on the root uptake of metals, with no consideration given to aerial deposition and foliar adsorption of elements. The correlation coefficients are presented in Table 4, where the subscripts 1 and v denote the metals found in fraction 1 of the BCR procedure and in vegetables, respectively. A significant positive correlation is observed between the metal concentrations obtained from soil extracts (from fraction 1) and vegetables for cadmium, e.g., 0.801, at the 99% confidence level. The lack of correlation for copper and lead may be explained by metal species differences in plant uptake, because the bioavailability was different for various metals.

As shown in Table 4, the higher the acid soluble fraction of Cd in soil,the greater the plant uptake of Cd. Generally, the acid soluble fraction was easily absorbed by plants, while the reducible and oxidizable fractions could potentially be absorbed by crops. In the study area, the topsoil contained a considerable proportion of the reducible and oxidizable fractions of metals, which varied from 45% to 67%. These less-bioavailable forms may transform into more-bioavailable forms when the chemical and physical

Fig. 4 Metal concentrations (mg/kg, FW) in vegetable samples: (a) cadmium, (b) copper, (c) lead; the limit levels are indicated by broken lines. VA1 reprents the vegetable sample at site A1

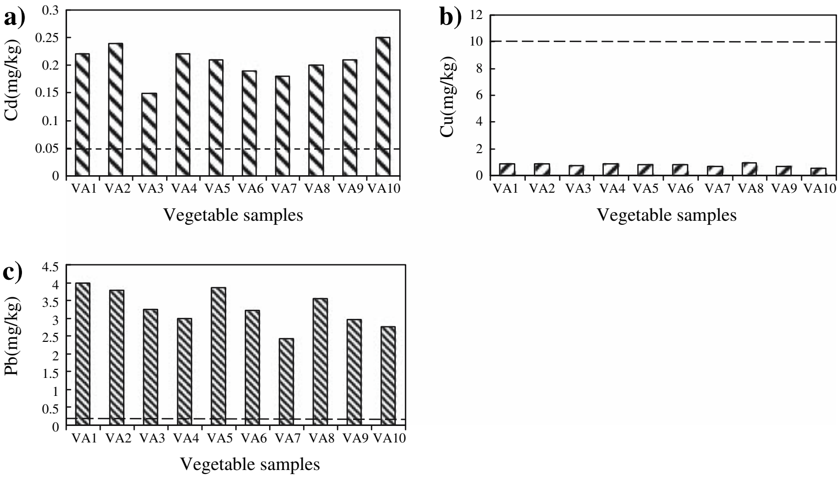


Table 3 The range of transfer coefficients of metals and their suggested coefficient range

Metals	Range Vegetable samples (*n* = 10)	Suggested range[a]
Cd	0.057–0.146(0.090)[b]	1–10
Cu	0.009–0.065(0.032)	0.01–0.1
Pb	0.031–0.132(0.068)	0.01–0.1

[a] Kloke et al. (1984)

[b] The value in brackets indicates the mean level of transfer coefficient

Table 4 Correlation matrix between the metal concentrations in the vegetable (FW) and soil samples (found in fraction 1)

	Cd_v	Cu_v	Pb_v	Cd_1	Cu_1	Pb_1
Cd_v	1					
Cu_v	0.870**	1				
Pb_v	–0.764*	–0.897**	1			
Cd_1	0.801**	0.686*	-0.460	1		
Cu_1	0.375	0.363	-0.109	0.658*	1	
Pb_1	0.425	0.392	-0.103	0.800**	0.817**	1

* and ** mean that the correlation coefficients are significant at the 0.05 level and 0.01 level, respectively

properties of the studied soils altered. Consequently, some measures such as using farmyard manure in farming crops should be taken to delay and slow the bioavailability of metals by changing their chemical forms.

In summary, the total concentrations of metals in the topsoil are generally higher than those in subsoil. It was found that Cd levels in the soil samples were 8 to 20 times higher than the Chinese limit, and Cu and Pb concentrations exceeded the limit values in some samples from the sample sites closer to the smelter. For the speciations of metals, the proportions of Cd, Cu, and Pb fractions were comparable in the topsoil and subsoil, but the topsoil was quite different from subsoil. This probably resulted from the complex processes occurring due to metals added to the soil from sewage irrigation and contaminated sediment. The vegetables are also heavily polluted by metals from the smelter with Cd and Pb concentrations far exceeding the edible limit. In addition, the higher the acid soluble fraction of Cd in the soil, the greater the vegetable uptake of Cd. If effective measures are not taken, the situation will worsen.

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