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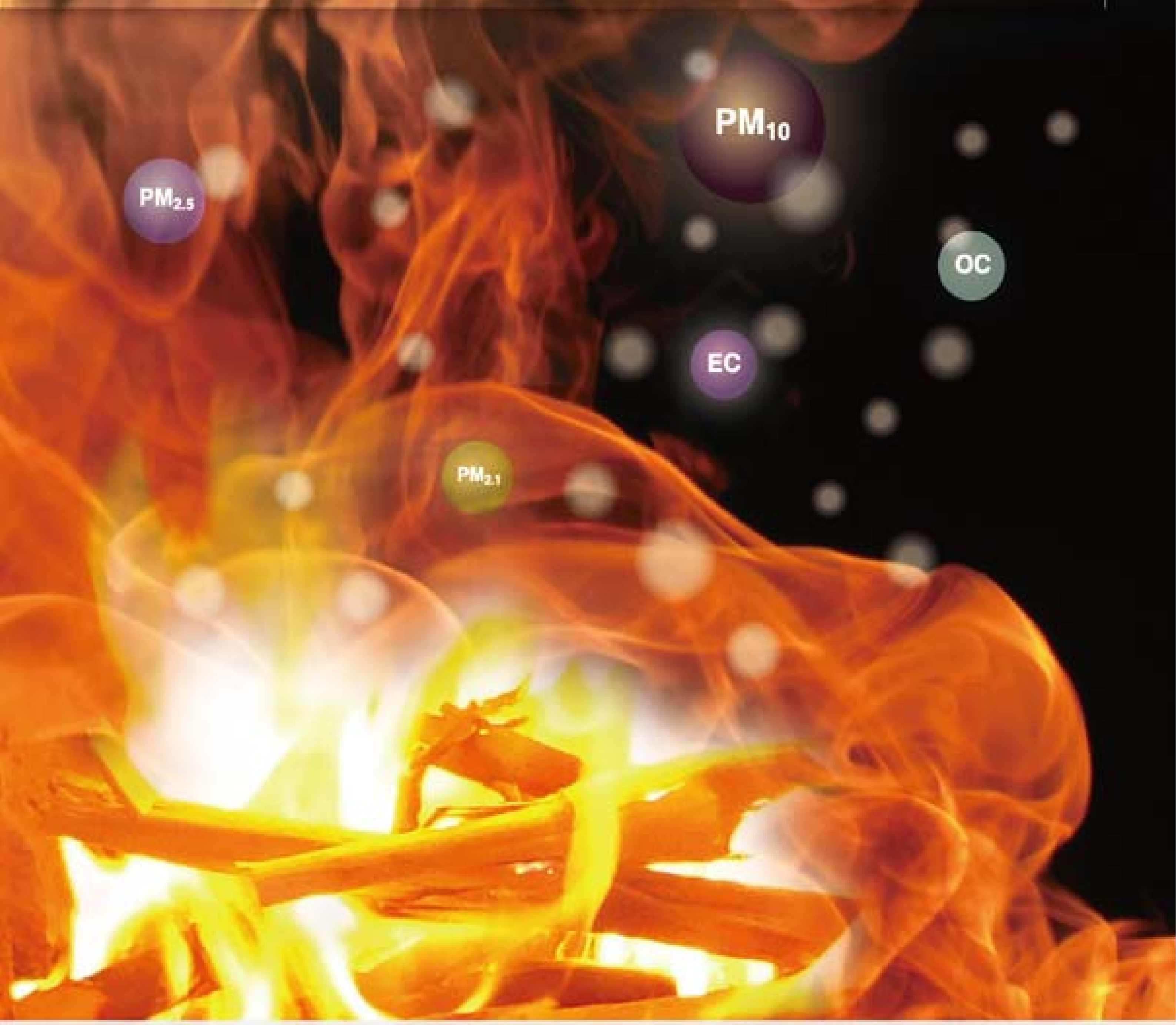

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Factors influencing the contents of metals and As in soils around the watershed of Guanting Reservoir, China

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Abstract

Topsoil samples from 61 sites around the Guanting Reservoir, China, were measured for Cu, Zn, Cr, Ni, Cd, Pb and As concentrations. The mean concentrations of Cu, Zn, Cr, Ni, Cd, Pb and As were 16.8, 59.4, 37.8, 18.3, 0.32, 20.1 and 8.67 mg/kg dry weight, respectively. Factors that influence the dynamics of these metals in soils around the watersheds of Beijing reservoirs were examined. The influence of atmospheric deposition, land use, soil texture, soil type and soil chemical parameters on metal contents in soils was investigated. Atmospheric deposition, land use and soil texture were the important factors affecting heavy metal residues. Soil type and soil chemical parameters were also involved in heavy metal retention in soils. The data provided in this study are considered crucial for reservoir remediation, especially since the Guanting Reservoir will serve as one of the main drinking water sources for Beijing in the foreseeable future.

Key words: heavy metals; atmospheric deposition; land use; soil texture; watershed

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Introduction

Over the past several decades, the presence of metals and As in the environment has been of world-wide concern because of their known environmental properties such as ubiquity, persistence, and toxicity (Spry and Wiener, 1991; Du Laing et al., 2009; Lee and Pandey, 2012). Heavy metal pollution not only degrades the quality of soil, aquatic and atmospheric environments, but also affects crop growth. It even enters the agricultural food chain through uptake by plants and thus affects human health (Li et al., 2008b).

Among all the environmental media, soil is the critical interface in the ecosystem, because it can be a source of pollution to surface and ground waters, living organisms, and sediments (Facchinelli et al., 2001). The metal and As pollution in soils could come from natural and anthropogenic sources, such as parent material, atmospheric deposition and agricultural activities (Xu et al., 2011). Different sources have different characteristics. For example, patterns of heavy metal contamination in soils can vary depending on the land use pattern (Gaw et al., 2006). Soil type and soil physical and chemical parameters can also affect the residue levels of elements significantly. Comprehensive understanding of factors that affect metal and As residues in soils will help in understanding the fate of trace metals in soils and in the examination of possible methods of remediation of contaminated soils.

Historically, the Guanting Reservoir was one of the five major water systems used for agriculture, industry, and municipalities in Beijing (Zhang et al., 2005). Because of industrial pollution and intensive farming along its watershed, the quality of water deteriorated. As a result, the Guanting Reservoir has no longer been used as a source of drinking water for Beijing, since 1997. This is a serious issue because Beijing currently has a severe shortage of water. Since the Beijing municipal government and the national government have launched the Beijing water environmental protection initiative, one of the plans has been the resumption of use of the Guanting Reservoir as a drinking water source (Hu et al., 2010). Over the past few years, several investigations on metals in different media around the Guanting Reservoir have been conducted (Zhang et al., 2002; Luo et al., 2007a, 2007b). Earlier studies were focused on the spatial distribution and ecological risk of

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heavy metals. However, there is limited information on how various soil and environmental factors affect the metal residues in the watershed of Guanting Reservoir. Thus, the elucidation of temporal trends of metal concentrations and factors affecting metal and As residues in soils around the reservoir is important for developing strategies for reservoir conservation and management.

The objectives of this research were to: (1) determine concentrations of cadmium (Cd), copper (Cu), nickel (Ni), lead (Pb), chromium (Cr) and zinc (Zn) and the metalloid As (As) in surface soils around the Guanting Reservoir; (2) to identify the sources of metal and As residues; and (3) to elucidate how the environmental factors affect metal and As contents in soils. The collected information will facilitate a better understanding of the residue characteristics and factors affecting metal and As contents in soils around the watershed.

1 Materials and methods

1.1 Study area

The Guanting Reservoir is located northwest of Beijing City (115.43°–115.97°E, 40.19°–40.50°N). The study site covers about 920 km^2, including 98 km^2 of water and 820 km^2 of surrounding land. The climate in the region is a cool temperature continental monsoon climate, with a mean annual temperature between 3 and 9°C and an average annual precipitation between 370 and 480 mm. The primary rock types are intermediate and acidic igneous; and the soil types are fluvo-aquic soil (FAS), calcareous-cinnamon soil (CCS), fluvo-cinnamon soil (FCS) and meadow-wind sand soil (MWSS). The types of land use around the Guanting Reservoir include woodland, orchard, farmland, and fallow, which together account for about 90% of the total area. Agriculture is the major land-use in the area around the Guanting Reservoir. Agrochemicals are applied to the farmland around this area, with an average chemical fertilizer application of approximately 107 kg/ha and pesticide usage of approximately 4.5 kg/ha. There are also several highways across this area with heavy traffic flow in the past several years.

1.2 Soil sampling

In May 2009, 61 soil samples were collected around the watershed of the Guanting Reservoir. Throughout the survey, a global positioning system was used to locate the sampling sites (**Fig. 1**). Each sample was made from a mixture of five sub-samples collected from the center and at the four corners of an area of about 10 m × 10 m. All soil sub-samples were collected at a depth of 0–20 cm using a stainless steel shovel. Therefore, the 61 soil samples were composed from a total of 305 soil sub-samples collected. Grass and other sundries were removed from the surface of each sampling location before the sample was collected. Soil was air-dried, crushed in an agate mortar, passed through a nylon 100-mesh sieve, and then stored at 4°C in the dark before analysis.

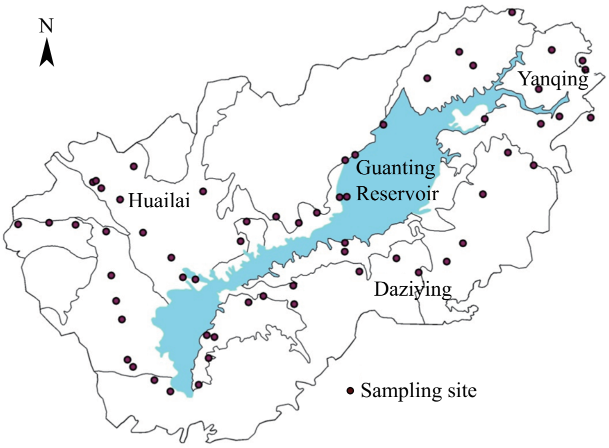

Fig. 1 Map of sampling sites around Guanting Reservoir.

1.3 Soil sample analysis

Soils were digested with a mixture of concentrated HCl-HNO_3-HF-$HClO_4$. Concentrations of the metals Cd, Cu, Ni, Pb, Cr and Zn in the digestion solution were determined by ICP-MS (7500CX, Agilent, USA). For As determination, samples were digested using aqua regia (HCl:HNO_3, *V/V*, 3:1), as recommended by the State Environmental Protection Administration of China (GB/T 22105-2008) (Liu et al., 2008). Standard reference materials, GSS-1 soils, obtained from the Center of National Standard Reference Material of China, were analyzed as part of the quality assurance and quality control procedures. Metal concentrations for GSS-1 were within the range of the certified values and the replicate analysis of each batch of samples showed that the analytical precision was within 10% variability. Samples were carefully handled to avoid introduction or loss of trace elements during preparation and analysis. All materials used during analytical determinations were kept in Teflon or other metal-free containers. Concentrations are expressed on a dry weight (dw) basis. The pH and total organic carbon (TOC) values were measured using the standard methods (Liu, 2001). Soil pH was determined from a 1:2.5 water-soil slurry by a Delta 320-s pH meter (Mettler-Toledo, Greifensee, Shanghai, China). Soil TOC was determined by titration with $FeSO_4$ after digestion with $K_2Cr_2O_7$-H_2SO_4 solution.

1.4 Statistical analyses

Statistical analyses were conducted using Microsoft Excel and SPSS 13.0 statistical software on a personal computer. The distribution of concentrations was tested with the Kolmogorov-Smirnov method to determine if they approximated the normal probability distribution. To evaluate the relationships between concentrations of elements and soil chemical characteristics, correlation analysis was used.

The Pearson correlation coefficient (for normally distributed concentrations), *r*, was used to measure the strength of a linear relationship between metals and soil chemical parameters.

2 Results and discussion

2.1 Preliminary data description

The concentrations of metals and As in soils from the vicinity of the Guanting Reservoir are shown in **Table 1**. The soil pH in the study area was between 6.88 and 8.03 with an average of 7.68. The TOC content in the soil ranged from 0.38 to 1.90 g/kg with a mean of 0.95 g/kg. The Kolmogorov-Smirnov test indicated that concentrations of Cr, Cd, Pb, Ni, As and Zn followed normal distributions and that Cu was log-normally distributed (*K-S*, $P > 0.05$). Because most of the Guanting Reservoir was located in Hebei Province, the concentrations of metals and As in our soils were compared with background values reported for Hebei Province. The mean concentration of Cd (0.32 mg/kg dw) was greater than its background value (0.08 mg/kg dw) as well as the recommended threshold limit value (0.30 mg/kg dw). It could thus be concluded that surface soils in the vicinity of the Guanting Reservoir were contaminated with Cd. Cd is relatively mobile in soils (Babic et al., 1998), and thus, could migrate to the Guanting Reservoir and degrade its water quality.

The mean concentration of Cu in the soils was 16.8 mg/kg dw. The Cu concentration values had a coefficient of variation of 64.1% and skewness of 3.11, which were the greatest of the elements studied. This observation is the result of the heterogeneity of Cu contents, with a few locations having elevated concentrations. The mean concentrations of Ni and Cr were 18.3 and 37.9 mg/kg dw, respectively. The numbers of samples exceeding the corresponding reference value for both of them were 1. Thus, it can be concluded that Ni and Cr contaminations in soils around the Guanting Reservoir are minimal.

The mean concentrations of Zn, Pb and As were 59.4, 20.1 and 8.67 mg/kg dw, respectively. The numbers of samples exceeding the corresponding reference values for Zn, Pb and As were 12, 26 and 6, respectively. Thus, Zn, Pb and As contamination in soils around the watershed of the Guanting Reservoir should be given attention. Ratios of the mean concentrations in soils around the watershed of the Guanting Reservoir divided by the corresponding reference values decreased in the order of Cd > Pb > Zn > Cu > As > Ni > Cr. Multiplying the ratios by the number of values significantly exceeding the respective reference values provides a combined estimation of both the magnitude and extent of contamination around the watershed of the Guanting Reservoir. This weighted result suggested that the magnitude of heavy metal pollution in soils around the watershed of the Guanting Reservoir declines in the order of Cd > Pb > Zn > Cu > As > Ni > Cr.

2.2 Metal concentrations affected by atmospheric deposition

A comparison of the metal and As contents in 2009 (our results) and 2003 (Luo et al., 2007b) is shown in **Table 2.** It is clear that levels of metal residues are mostly higher in our study in 2009, although it should be noted that an accurate comparison of metal residues cannot be made unless the sampling sites and points were all the same. One important reason for the increase in the concentration of elements was atmospheric deposition from sources originating from transportation, energy production, mining, metallurgical industries, manufacturing processes and waste incineration (Nriagu, 1990). The rate of atmospheric

Table 1 Descriptive statistics of heavy metal concentrations and soil properties from the Guanting Reservoir

Metal	Mean	Median	Min.	Max.	CV (%)	Skew -ness	Kurtosis	*K-S*, *P*	Background value[a]	Number exceeding values
Cu (mg/kg dw)	16.8	14.9	5.87	73.6	64.1	3.11	13.02	0.28[b]	21.7	8
Zn (mg/kg dw)	59.4	57.9	29.2	120.3	30.2	1.18	2.07	0.14	68.0	12
Cr (mg/kg dw)	37.9	38.5	23.4	98.4	29.5	2.65	13.51	0.22	63.9	1
Ni (mg/kg dw)	18.3	18.3	9.58	52.7	34.0	2.87	15.05	0.08	30.8	1
Cd (mg/kg dw)	0.32	0.30	0.15	0.74	31.3	1.59	4.15	0.15	0.08	61
Pb (mg/kg dw)	20.1	19.2	12.7	34.6	19.9	1.25	2.77	0.27	20.0	26
As (mg/kg dw)	8.67	8.76	4.63	15.9	26.5	0.51	0.61	0.97	12.0	6
pH	7.68	7.65	7.25	8.03	0.02	–0.03	–0.10	0.72	–	–
TOC (g/kg)	0.95	0.87	0.38	1.90	36.8	0.78	0.29	0.28	–	–

CV: coefficient of variation.

[a] Background values of soil heavy metals in Hebei Province (Wang and Wei, 1995); [b] dataset is ln-transformed. –: no value.

Table 2 Metals and As concentrations in 2009 and 2003

Year	Cu (mg/kg)	Zn (mg/kg)	Cr (mg/kg)	Ni (mg/kg)	Cd (mg/kg)	Pb (mg/kg)	As (mg/kg)	Reference
2009	16.80	59.40	37.80	18.28	0.32	20.13	8.67	This study
2003	13.54	54.28	32.35	15.81	0.68	8.21	6.88	Luo et al., 2007b

deposition depends on the proximity to point sources of pollution, such as heavy industry or major roads (Hu and Balasubramanian, 2003; Nicholson et al., 2003). Several major freeways for coal transportation were distributed in the study area. The total amount of coal transportation in this area increased greatly from 2003 to 2009 because of the rapid economic development of China and the discovery of coal mines in Inner Mongolia. Due to the large volume of vehicular traffic for coal transportation, traffic jams are a very common phenomenon. As a result, the discharge of pollutants in vehicle exhaust has increased.

One of the important sources of Pb was vehicle exhaust (Feng et al., 2011), which also contains Zn and Cu (Madrid et al., 2002). In addition, the braking system of vehicles contains high concentrations of Zn and Cu (Martín et al., 1998; Lindström, 2001). The coal-related industries, such as iron and steel plants, power stations and coal yards, have rapidly developed (including quantity and scale) during the past 7 years. Most of these companies were constructed along the major roads and distributed in the northwest part and upstream of the Guanting Reservoir. Due to the combustion of coal and smelting of iron and steel plants, metals including Cr, As, Pb and Ni could be discharged into the air (Dauvalter, 2003; Wang and Stuanes, 2003). Considering the role of wind and terrain, most of the metals in the air were deposited onto the surface soil around the Guanting Reservoir and contribute to overall soil concentrations (Steinnes et al., 1997), because the north, east and south boundaries of the Guanting Reservoir are mountains and the long-term wind direction of this area was from northwest to southeast.

Cadmium is the only metal whose concentration decreased 0.36 mg/kg dw from 2003 to 2009. This suggests that the output of Cd was much greater than the input. One of the reasons could be that atmospheric deposition contributes little to the accumulation of cadmium in soil. Luo et al. (2007b) reported that the main source of Cd was wastewater in the year of 2003. Presumably, the wastewater which contains Cd might have been controlled by the local government in the past several years. Furthermore, Cd is relatively mobile in soils (Babic et al., 1998).

2.3 Metal concentrations affected by land use patterns

Land use is an important factor that could affect metal distributions because different land uses could contribute to differences in metal sources, farming practices, and soil properties (Kim et al., 2003). Soils collected from different land-use patterns were analyzed to determine the effects of land use on metal and As residues. Four types of land use (woodland, fallow, farmland and orchard) were identified in the Guanting Reservoir area. Descriptive statistics of metal concentrations in soils from different land uses are shown in **Table 3**. In general, concentrations of metals and As in soils under different land uses varied greatly.

The results in **Table 3** indicated that the largest average values for Zn (63.5 mg/kg dw), Cr (39.0 mg/kg dw), Ni (20.1 mg/kg dw), Cd (0.36 mg/kg dw), Pb (21.1 mg/kg dw) and As (9.67 mg/kg dw) were found in the farmland. This might be related to the large-scale application of phosphate fertilizers and sludge that contained Cd, Zn and As (Taylor, 1997; Folkes et al., 2001; Nan et al., 2002) in farmlands. The elevated concentrations of As and Pb in farmland soils have been shown to be most likely due to the use of lead-arsenate insecticides to control insects (Kober et al., 1999; Wang et al., 2007). In addition, wastewater irrigation could be mainly responsible for the great concentrations of Cr and Ni in farmland soils around the Guanting Reservoir (Sedlak et al., 1997; Sun and Hao, 2004).

The largest average values for Cu (19.4 mg/kg dw) was found in orchard soils. To identify which kind of fruit cultivation had the largest impact, the orchard soils were categorized into four types of cultivation patterns as vineyard, peach, apple and apricot. The mean concentrations of Cu determined in vineyard, peach, apple and apricot were 29.4, 11.7, 13.7 and 15.1 mg/kg dw, respectively. In addition, the highest levels of Cu were all found in vineyard soil, which confirmed that grape cultivation contributed to high levels of Cu. There are more than ten large grape vine plantations (including the famous Great Wall Grape Vine Plantation) located in this area. In recent years, the grape cultivation area in the County has been expanding. Cu pollution in some soils around the Guanting

Table 3 Concentrations (mg/kg dw) of heavy metals in soils from different land use around the Guanting Reservoir

Land use	*n*	Parameter	Cu	Zn	Cr	Ni	Cd	Pb	As
Woodland	4	Mean	11.0	42.8	37.1	17.0	0.27	18.0	7.65
		SD	4.48	10.2	7.08	4.50	0.08	3.04	1.68
		Median	11.4	42.8	39.3	17.7	0.27	17.5	7.16
Fallow	7	Mean	12.1	56.5	33.0	15.7	0.28	18.7	8.79
		SD	2.86	24.6	4.85	1.86	0.09	3.24	3.17
		Median	11.5	49.5	32.1	15.2	0.26	18.6	7.81
Farmland	19	Mean	15.5	63.5	39.0	20.1	0.36	21.1	9.67
		SD	3.58	16.3	8.81	4.63	0.12	3.84	1.81
		Median	15.6	58.4	40.7	19.9	0.31	19.4	9.55
Orchard	31	Mean	19.4	59.7	36.0	17.9	0.31	20.1	8.16
		SD	14.2	17.4	12.8	7.60	0.09	4.29	2.30
		Median	15.8	58.5	35.7	18.2	0.30	19.2	8.41

n: the number of sampling sites; SD: standard deviation.

Reservoir might be the result of historical application of Cu-based fungicides in vineyards or orchards (Hollnd and Solomona, 1999). This result is in agreement with those reported in the literature (Deluisa et al., 1996; Besnard et al., 2001).

Fallow land soils have the lowest mean values for Cr (33.0 mg/kg dw) and Ni (15.7 mg/kg dw), while woodland soils have the lowest mean values for Cu (11.0 mg/kg dw), Zn (42.8 mg/kg dw), Cd (0.27 mg/kg dw), Pb (18.0 mg/kg dw) and As (7.65 mg/kg dw). The lower values in both fallow and woodland soils, as compared to farmland and orchard soils, may be due to less common application of fertilizers and pesticides. In addition, the lower metal concentrations detected in fallow and woodland soils might be due to the drift of metals and atmospheric deposition from the nearby areas (de Vries et al., 2003). Since metals and As in the woodland soils can be reduced by plant uptake (MacFarlane et al., 2003), most metal contents in the woodland soils are lower than those in the fallow land soils.

2.4 Metals concentrations affected by soil type and soil texture

Soil types are expected to influence heavy metal accumulation in agricultural soils. Four general soil types dominated in the study area: CCS, FAS, FCS, and MWSS. However, because only two soil samples were collected for the MWSS, metal and As contents for this soil type are not listed due to uncertainty. The concentrations of metals and As in different soil types are given in **Fig. 2a**. The highest levels of As, Ni, Zn, Cu and Cr were found in the CCS. The mean concentrations of Ni, Cr and Cu decrease in the order of CCS > FCS > FAS, while the average contents of As and Zn decreased in the order of CCS > FAS > FCS. Different soil types were constituted by various soil parent matrixes, which were very important for the levels of metals and As. Navas and Machín (2002) found that soil type determined to some extent the variation of metal and As concentrations in the soils of Aragón Spain. Lu et al. (2012) also found that there were significant differences in As and Pb concentrations between the Ustalfs and the Ustochrepts in the agricultural soils in Shunyi, Beijing, China.

Compared to FAS and FCS, the CCS has high concentrations of $CaCO_3$, and calcium cations compete with metals ions and restrain the assimilation of metals by organisms, and finally lessen the bioaccessibility of elements and increase the contents of elements in the soil (El-Enany, 1995; Li et al., 2008a). Moreover, metal contents in FAS and FCS are more or less affected by groundwater. The groundwater table can change with season and weather conditions. Because both soils are distributed on smooth plains, the depth of groundwater is shallow, which makes it easy for the groundwater to reach the surface by capillary action (Zhang, 2002) and leach the metals.

Soil texture is a classification tool used in both the field and laboratory to determine classes for soils based on their physical texture (Shirazi and Boersma, 1984). Soil textures are classified by the fractions of each soil component present in a soil. In this study, soil samples were categorized into four types of textures (loam, light loam, sandy loam, and sand) to explain the influence of soil texture on metals and As residue levels. The concentrations of metals and As for different soil textures are given in **Fig. 2b**. The highest levels of As, Pb, Cd and Cr were found in loamy soil, and the largest concentrations of Ni, Zn and Cu were found in light loamy soil. The lowest levels of As, Pb, Cd, Ni and Zn were found in sandy soil, and the lowest contents of Cr and Cu existed in sandy loam soil.

The differences may be explained as follows. Compared to sandy and sandy loam soils, loam and light loamy soils can have larger surface-area-to volume ratios and surface energy, which can have an effect on the ability of the soil particles to adsorb metals, as well as the ability to retard the bioavailability of these elements (Zibilske and Risser, 1986; Wang et al., 2006). Meanwhile, the inherent characteristics of metals play an important role in the adsorptive capacity of soil as well. Of the seven elements, the lowest relative difference in concentrations among the four soil textures is for Cu and the biggest relative difference value for Cd. This may be due to the high chelating ability of Cu, resulting in its low degradation and

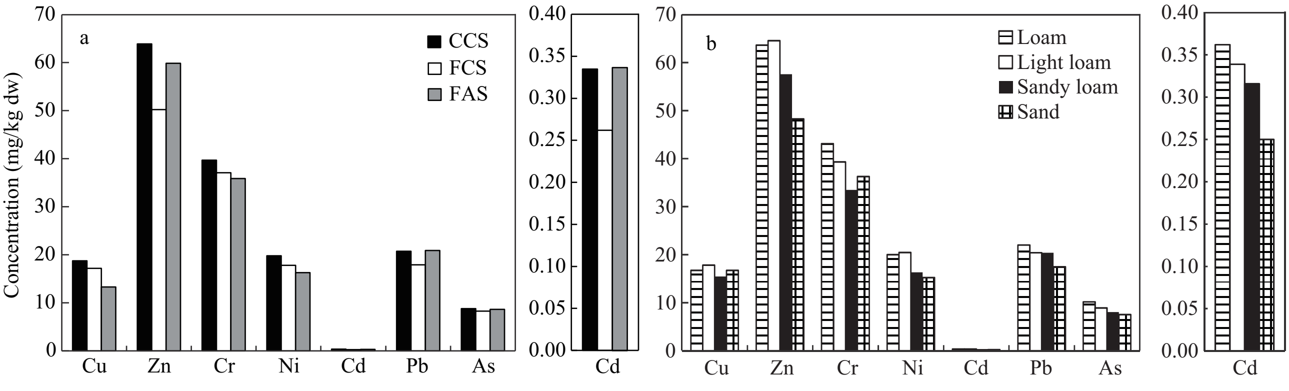

Fig. 2 Concentrations of metals and As in different soil types (a) and different textures (b). CCS: calcareous cinnamon soil; FCS: fluvo cinnamon soil; FAS: fluvo-aquic soil.

Table 4 Correlations between metals and As concentrations and soil chemical parameters in soils around Guanting Reservoir

	Cu	Zn	Cr	Ni	Cd	Pb	As
pH	0.05	–0.37**	0.08	–0.03	–0.36**	–0.29*	–0.20
TOC	0.14	0.63**	0.09	0.30*	0.58*	0.49**	0.50**

** Correlation is significant at the 0.01 level (2-tailed); * correlation is significant at the 0.05 level (2-tailed).

bioavailability (Huang et al., 2009). The large variation in Cd contents may be explained by its high mobility and relatively weak chelating capacity (Babic et al., 1998).

2.5 Effects of pH and total organic carbon

Correlation analysis was conducted to determine the extent of association between element concentrations and soil chemical parameters around the watershed of the Guanting Reservoir (**Table 4**). TOC played an important role in determining the concentrations of pollutants. Surface soil rich in organic matter could offer much more functional groups (like COO– groups) that could complex and deposit metals ions (Baker et al., 1995). Hence, as the amount of organic matter present in soil increased, the opportunity for forming stable metal-organic matter complexes increased. The TOC values were significantly correlated ($P < 0.01$) with concentrations of Zn, As and Pb, and less correlated ($P< 0.05$) with concentrations of Ni and Cd. In this study, there was no positive correlation found between TOC and concentrations of Cu and Cr. It is therefore impossible to use a single soil geochemical factor to predict the distribution of metal and As residues in soils on a regional scale.

The metal speciation, solubility and adsorption to solid phases are intimately connected to the pH. Numerous studies have found a significant effect of soil pH on metal bioavailability (Cavallaro and Mcbride, 1984; Sauvé et al., 2000). The pH values were significantly negatively correlated ($P < 0.01$) with concentrations of Zn and Cd, and less negatively correlated ($P < 0.05$) with concentrations of Pb. This could be explained by the fact that metals competed with H^+ for positions on the exchange sites when soil pH was low, thereby increasing the solubility of metals in the soil solution (McBride et al., 1997; Sauvé et al., 1997). This process increases the concentrations of metals in the bioavailable fraction and ultimately reduces metal contents in soil. The soil pH had no significant correlations with the rest of the elements studied, indicating that soil pH had no obvious effect on Cu, Cr, Ni and As residues in the study area.

3 Conclusions

An extensive investigation of metals and As in the vicinity of the Guanting Reservoir was carried out. The average concentrations of Cu, Zn, Cr, Ni, Cd, Pb and As in soils around the Guanting Reservoir were found to be (16.8 ± 10.77), (59.4 ± 17.94), (37.8 ± 11.17), (18.3 ± 6.21), (0.32 ± 0.10), (20.1 ± 4.00) and (8.67 ± 2.30) mg/kg dw, respectively. The magnitude of contamination in soil declined in the order of Cd > Pb > Zn > Cu > As > Ni > Cr.

Except for cadmium, an important source of metals in this area was atmospheric deposition. Land uses, soil types and chemical parameters accounted for considerable differences in metal and As accumulation in the soils. The metal residues in different land use categories varied greatly. In general, metal pollution in the four land use categories decreased in the order of farmland > orchard > fallow > woodland. Compared to sandy loam soil and sandy soil, metal and As levels were inclined to accumulate more in loam and light loam soil. As, Ni, Zn, Cu and Cr residues were highest in calcareous cinnamon soil, while Pb and Cd residues were highest in fluvo cinnamon soil. There were significant correlations between TOC and Zn, As and Pb suggesting a strong influence of organic matter on these elements' residue levels. Soil pH levels were significantly negatively correlated with concentrations of Zn and Cd, and had less or no obvious effect on the other metals.

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