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Original Research Paper

Speciation Distribution Characteristics of Heavy Metals and its Relationships with Soil Acid Chemical Properties in the Chengdu Plain

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ABSTRACT

Based on the field investigation, 197 soil samples were collected to analyse the concentration and speciation distribution characteristics of Cd, Cu, Pb, Cr and Zn. The relationships between the soil acid chemical properties and the speciation of heavy metals were studied in this paper. The results showed that the content of Cd, Cu, Pb, Cr and Zn in the soil significantly increased compared with the Second National Soil Survey. Comparing with the standard of National Soil Environmental Quality, the heavy metals of soil in the Chengdu plain has polluted the soil to certain degree. For the form distribution of soil heavy metal, Cd was mainly presented in the Fe-Mn oxidation bound and Cu, Pb, Cr, Zn mainly consisted in the residual form. There was certain correlation between each active state (exchangeable form, carbonate state, Fe-Mn oxidation state and organic combination state), which could be converted mutually and had strong potential hazards to environment. The acid chemical properties of soil had notable or highly notable impact on the pattern distribution of heavy metals. Soil pH had obvious effect on the liable morphological changes of Cd, Pb and Zn. The soil acid buffer property (soil acid damage capacity) had highly significant positive correlation with exchangeable form, carbonate state, Fe-Mn oxidation state and liable speciation of Cd in the soil, while had highly significant negative correlation with exchangeable form, organic bound and liable speciation of Cr in the soil, and remarkable negative or positive correlation with exchangeable form, organic bound, carbonate state and Fe-Mn oxidation of Zn in the Chengdu Plain.

INTRODUCTION

With the rapid development of social economy, the heavy metal pollution problem becomes increasingly serious. The environmental behaviour and ecological effects are inextricably linked with the soil morphology and attracted more and more attention (Zhao et al. 2014, Chen et al. 2006, Vries et al. 2011, Green et al. 2006). Relevant studies showed that the heavy metal speciation in soil had close relationship with the acidity of soil and that the varying acidification properties in soil were bound to cause the differences of heavy metal speciation changes (Muhammad et al. 2012, Guo & Huang 2003, Mcbride & Blasiak 1979). The heavy metal pollution problem of soil in the Chengdu plain had been attracting the scientists' attention since the 1980s. Some scholars had conducted related researches on its pollution conditions and distribution characteristics (Yang et al. 2008, Yao & Liang 2002, Zhu 1999), finding that the phenomenon of heavy metal accumulation was prevalent in the surface soil in plain areas. Some areas were polluted to varying degrees by heavy metals. However, there were few relevant reports on the distribution characteristics of heavy metal speciation in soil and the acid chemical properties of soil in the Chengdu Plain. Therefore, the heavy metal speciation in soil was analysed to find out its potential hazards to environment and meanwhile a deep research on the impacts of the acid chemical properties of soil to the heavy metal speciation variation in the soil was carried out in this paper.

MATERIALS AND METHODS

Study area: Located in the west of Sichuan basin and with the terrain tilting from the north-west to the south-east, the Chengdu Plain is an alluvial composite fan plain formed by several rivers including the Minjiang River and Tuojiang River. The alluvial fan of Minjiang River is the main part, the soil developed here is mostly the paddy soil formed by the gray and gray-brown alluvial deposits of modern rivers (SPAD 1997). This research primarily focused on the central core part of the Chengdu Plain that located in east longitude 102°542'-104°532' and north latitude30°052'-31°262' with a total area of 5300 km^2, including 11 cities (Counties): Pengzhou, Xindu, Shuangliu, Wenjiang, Pixian, Dujiangyan, Deyang, Guanghan, Qionglai, Chongzhou and Xinjin. As a subtropical monsoon climate zone with rich heat, abundant rainfall, four distinct seasons and hot rainy season, the area develops deep soil which has rich mineral nutrients and higher fertility levels. It yields two crops in one year and the field crops mainly include rice, wheat, corn, rape and vegetables.

Soil sample collection: Based on the basic situation of the Chengdu Plain and the spatial and temporal variation of soil environmental quality, regional social and economic conditions, the distribution of rivers, terrain and parent materials, productivity levels and the changes in cropping systems were taken into consideration, soil samples were collected in the core areas of the Chengdu Plain and the GPS was employed to position the sampling location. The multi-point mixed method was used to collect 197 soil samples (0-20cm) which were pre-processed after being dried naturally.

Test and data analysis methods: Conventional analysis methods were used for the basic physical and chemical properties of soil. The mixed acid of HNO3-HF-HClO4 was employed to digest the soil samples and soil heavy metals were analysed by atomic absorption spectrophotometry (AAS) (Lu 1999).

According to the Tessier sequential extraction procedure (Tessier et al. 1979), heavy metal speciation can be divided into exchangeable form, carbonate bound, Fe-Mn oxidation bound, organic bound and residual state. Heavy metal concentration of extract was measured by ICP-MS.

The measurement of soil acid-damage capacity was by the method of Liao & Dai (1991). Excel 2007 and SPSS13.0 were used for the statistical analysis.

RESULTS AND DISCUSSION

Statistical characteristics of soil heavy metals: The content of soil heavy metals in the Chengdu Plain has undergone great changes (Table 1). The average value of Cd, Cu, Pb, Cr and Zn were 0.27 mg/kg, 35.51 mg/kg, 67.04 mg/kg, 106.26 mg/kg and 94.01 mg/kg, respectively. The variation coefficient of Cd was relatively big while that of Cu and Zn were smaller. Compared with the background value of soil heavy metals in the Second National Soil Survey, the content of Cd, Cu, Pb, Cr and Zn in soil increased in a wide margin. In particular, as soil Pb was strongly influenced by human activities, its content was 2.7 times that of the soil background value in 1982.

Pollution assessment of soil heavy metal: Under the influence of alluvial soil forming processes, the background value of soil heavy metal contents in the Chengdu Plain varies greatly across regions. In the meantime, strongly intervened by human activities, the soil heavy metal content in the plain generally displayed an upward trend. With China's Soil Environment Quality Standard as the assessment standard (Chinese Environmental Standards Compilation 2000), the Cd content in soil lied between the first-level and second-level standard, and 58.89% of the collected soil samples reach the first-level standard of National Soil Environment Quality. Cu in soil exceeded the national first-level standard in 74.62% of the samples, and 3.55% of the samples surpassed the national second-level standard. The average content of Pb exceeded the first-level standard accounts for 42.64%. While that of Cr was lower than the first-level standard of national soil environment quality in 28.46%, and exceeded the second-level standard only in 10.15% of the samples. The average content of Zn lied within the first-level standard, and 57.37% of the soil reached the first-level standard and 42.63% was within the second-level standard.

The background value was measured by Yao Tingshen (Yao 1987) and was selected as the benchmark concentration value of assessment. The pollution levels were obtained from the grading standards of geo-accumulation index. Among the collected soil samples, 45.69% of them got cadmium contamination and the pollution level ranged from the first level to the second level with the first level being the main part, which was equivalent to the transitional stage from non-pollution to medium contamination. The vast majority of soil samples did not get polluted by the copper and only 16.24% of the soil was influenced by exogenous copper. Nearly half of the soil got polluted by lead in varying degrees. The pollution levels had a large span, which were distributed from the first level to the fourth level and dominated by the first and second level. Individual samples had approached or reached the strongest pollution. A small number of samples get influenced by the exogenous chromium in varying degrees, and 87.82% and 89.01% of them did not get chromium and zinc contaminated, respectively. The zinc pollution was at the first level, which was equal to the transitional stage from non-pollution to light contamination.

The distribution characteristics of soil heavy metal speciation: The form distribution of Cd in soil was as follows: Fe-Mn oxidation state > residual state > carbonate state > organic bound > exchangeable state (Table 3). The Fe-Mn oxidation state constituted the main part. The content of the residual form Cd was 0.073mg/kg, accounting for 27.34% of the total amount and the active state Cd (the sum of exchangeable state, carbonate state, Fe-Mn oxidation state and organic bound) took up 72.66% of the total amount. The Cu in soil was primarily in the residual state and the organic bound. The average content of the residual state was 26.96 mg/kg, and accounted for 75.92% of the total amount, while the active state accounted for the 24.08% of the total amount. The form distribution of Cu was as follows: residual state > organic bound > Fe-Mn oxidation state

Table 1: Statistical characteristics of Cd, Cu, Pb, Cr, Zn content in soil*.

Item	Minimum (mg/kg)	Maximum (mg/kg)	Average (mg/kg)	Variation coefficient	Background value of 1982 (mg/kg)
Cd	0.04	0.66	0.27	0.65	0.16
Cu	19.00	77.50	35.51	0.31	31.57
Pb	15.51	108.72	67.04	0.49	24.76
Cr	49.30	221.81	106.26	0.42	80.15
Zn	41.77	135.64	94.01	0.23	90.78

*Data in the table were the statistical results of 197 samples.

Table 2: Soil environmental quality standard (mg/kg)*.

	Level	Cd ≤	Farmland Cu ≤	Orchard Cu ≤	Pb ≤	Paddy field Cr ≤	Dryland Cr ≤	Zn ≤
First-level	Natural background	0.20	35	-	35	90	90	100
Second-level	pH < 6.5	0.30	50	150	250	250	150	200
	pH 6.5-7.5	0.60	100	200	300	300	200	250
	pH > 7.5	1.0	100	200	350	350	250	300

*Chinese Environmental Standards Compilation (2000)

Table 3: Descriptive statistics of different forms of soil Cd, Cu, Pb, Cr and Zn (mg/kg).

Item	Average	Exchangeable		Carbonate		Fe-Mn oxidation		Organic		Active state*		Residual	
		Content	%	Content	%	Content	%	Content	%	Content	%	Content	%
Cd	0.267	0.027	10.11	0.053	19.85	0.086	32.21	0.028	10.49	0.194	72.66	0.073	27.34
Cu	35.51	0.24	0.68	0.77	2.17	1.24	3.49	6.30	17.74	8.55	24.08	26.96	75.92
Pb	67.04	2.21	3.30	3.29	4.91	8.53	12.72	13.38	19.96	27.41	40.89	39.63	59.11
Cr	106.26	0.88	0.83	1.03	0.97	1.52	1.43	4.40	4.14	7.83	7.37	98.43	92.63
Zn	94.01	1.37	1.46	1.96	2.08	10.22	10.87	12.62	13.43	26.17	27.84	67.84	72.16

*The active state was the sum of exchangeable, carbonate, Fe-Mn oxidation and organic bound.

> carbonate state > exchangeable state. The Pb in soil was dominated by the residual state and its average content was 39.63 mg/kg, accounting for 59.11% of the total amount. The organic bound Pb accounted for 48.81% of the active state and its form distribution characteristic was consistent with that of Cu. The Cr in soil was mainly composed of the residual state and the active state Cr merely accounted for 7.37% of the total amount. Its form distribution was below: residual state > organic bound > Fe-Mn oxidation state > carbonate state > exchangeable state. The content of the active state Zn in soil was 26.17 mg/kg, accounting for 27.84% of the total amount and the organic bound Zn took up the largest proportion, being 48.22%. The form distribution had the following peculiarity: residual state > organic bound > Fe-Mn oxidation state > carbonate state > exchangeable state. Except for Cd, which was mainly consisted of Fe-Mn oxidation state, the remaining were dominated by the residual state.

The active form of Cd accounted for a great percentage and the correlation between different states of Cd was so strong that it had reached a prominent level (Table 4). These forms could transform from one to another, posing a potential threat to environment. Cu existed mainly in the form of residual in the soil, exchangeable Cu which could cause direct harm to creatures accounted for a small proportion of active forms. And it only had a relatively significant correlation with Fe-Mn oxide. However, the potential damage from Cu was mainly determined by carbonate bound, Fe-Mn oxide and organic matter bound. Active patterns of Pb accounted for 40.89%. Its various forms also displayed a significant correlation, and the amount of potentially available forms which could be absorbed by creature was large, posing a great potential risk to the well-being. Active states of Cr only accounted for 7.37% of the total. Because Cr^{3+} in the soil could be solidly absorbed and precipitated, exchangeable Cr only accounts for 0.83% of the total. Exchangeable Cr had a negative correlation with carbonate and Fe-Mn oxidation bound while it had a significant correlation with organic combined. Besides, its amount of potentially available forms that creatures could absorb was small, so Cr had smaller potential toxicity than others. There were relatively few exchangeable and carbonate bound Zn in the soil, which could easily be absorbed by plants. A negative correlation between exchangeable Zn and carbonate form Zn was expressed, but the correlation between exchangeable Zn and Fe-Mn oxidative Zn was not significant.

Table 4: The correlation of different forms of soil Cd, Cu, Pb, Cr and Zn.

Element	Forms	Exchangeable (Ex.)	Bound Carbonate (Ac.)	Bound of Iron-Manganese oxide (B.I.M)	Bound of organic Matter (B.O.M)	Active (Ac.)
Cd	Ex.	1	0.789**	0.868**	0.335**	0.882**
	B.C.		1	0.844**	0.363**	0.954**
	B.I.M.			1	0.545**	0.954**
	B.O.M.				1	0.516**
	Ac.					1
Cu	Ex.	1	0.146	0.241*	0.196	0.262*
	B.C.		1	0.747**	0.447**	0.595**
	B.I.M.			1	0.638**	0.783**
	B.O.M.				1	0.966**
	Ac.					1
Pb	Ex.	1	0.904**	0.652**	0.280**	0.716**
	B.C.		1	0.658**	0.336**	0.742**
	B.I.M.			1	0.355**	0.872**
	B.O.M.				1	0.705**
	Ac.					1
Cr	Ex.	1	-0.031	-0.004	0.246*	0.376**
	B.C.		1	0.153	0.032	0.167
	B.I.M.			1	0.315**	0.657**
	B.O.M.				1	0.889**
	Ac.					1
Zn	Ex.	1	-0.040	0.025	0.248*	0.182
	B.C.		1	0.705**	0.286**	0.623**
	B.I.M.			1	0.513**	0.915**
	B.O.M.				1	0.791**
	Ac.					1

In the soil, available Zn could only provide plants with what they need for growth and did no harm to the environment of the soil.

Soil acid chemical characteristics: As can be seen from soil acid chemical features (Table 5), the soil displayed a weak variation, and its pH value ranged from 4.47 to 7.80, its amplitude covered 3.33 unit and median was 6.36. Acid-buffering properties reflect its sensitivity to acid. Acid-damage volume means soil's demand of acid per unit when the pH value reaches to 3.5 (plants will then be damaged), and it also represent soil's acid-buffering capability. The acid-damage volume in the Chengdu plain varied greatly from 2.8-1530.60 mmol/kg, and demonstrated a sharp variation. The maximum acid-damage volume corresponded to the pH value of 7.58, but the correspondence between pH and acid-damage volume was not absolute consistency, instead, there was a significant positive correlation between them, and the r was 0.447.

The impact of soil acid chemical characteristics on heavy metal forms: Soil pH has a great impact on the sorption and desorption of heavy metals (Zhang et al. 2005). In light of the characteristics of heavy metal speciation, active fractions could be mutually transformed to each other when affected by the exterior environment, especially when pH changed (Table 6). Exchangeable state would also experience migration and transformation as the environment changed. There was a significant positive correlation between the soil pH and Cd and Pb whereas a negative correlation between pH and Cr, and a significant negative correlation between pH and Zn. When the soil pH was high, Zn could be deposited easily with hydroxide or carbonate, which would reduce exchangeable Zn. In the reductive conditions if pH was high, Cr existed in a less toxic state of Cr^{3+}, and precipitated with hydroxide, so that the other speciation of Cr accounted for a small number, which had a negative correlation with pH. Sediment produced by Cd in a higher pH, and only when pH >10, the hydroxide sediment of Cd may be completely precipitated. The soil pH of Chengdu Plain ranged from slightly alkaline to acidic. Exchangeable Cd had a significant positive correlation with soil pH. Carbonate combined speciation was the most sensitive to pH and there was a significant correlation between soil pH and carbonate state Cd and Zn. Both of them could easily be released into the environment with soil pH decreased; whereas the pH increased could facilitate the generation of carbonate and make carbonate mineral coprecipitate.

Fe-Mn oxide had a vast surface and a strong adsorption capacity of metal ion, and with the pH increased it could benefit the creation of Fe-Mn oxidation state. Elements ex-

Table 5: Descriptive statistical analysis of soil acidification characteristics.

Item	Minimum	Maximum	Mean	Coefficient of variation
pH	4.47	7.80	6.36 (median)	0.10
Acid-damage volume (mmol/kg)	2.80	1530.60	146.32	2.44

Table 6: The correlation between soil pH and Cd, Cu, Pb, Cr, Zn.

Element	Ex.	B.C.	B.I.M.	B.O.M.	Ac.
Cd	0.657**	0.686**	0.643**	0.248*	0.677**
Cu	0.019	0.180	0.179	-0.101	-0.022
Pb	0.292**	0.220*	0.232*	-0.173	0.084
Cr	-0.191	0.152	-0.021	-0.234*	-0.199
Zn	-0.455**	0.371**	0.398**	-0.148	0.190

Table 7: The correlation between soil acid-damage volume and forms of Cd, Cu, Pb, Cr and Zn.

Element	Ex.	B.C.	B.I.M.	B.O.M.	Ac.
Cd	0.698**	0.812**	0.695**	0.038	0.758**
Cu	0.127	0.420**	0.318**	-0.106	0.017
Pb	0.187	0.175	0.226*	-0.493**	-0.060
Cr	-0.313**	0.149	0.050	-0.493**	-0.367**
Zn	-0.361**	0.249*	0.288**	-0.422**	-0.028

cept Cr had a significant positive correlation with pH. With pH increased, the complexation ability of organic bound Cd improved. However, other elements displayed a negative correlation with pH in this regard, which was probably due to the fact that the combination of heavy metal and organic matter had changed from a general complexation to a more stable chelating state, which was difficult to be extracted. Or due to the fact that hydroxy complexes which were produced by heavy metals promoted the dissolution of heavy metals.

The soil acid-buffering capability in soils was significantly different (Ren et al. 2011, Sakala et al. 2004). Different heavy metals had different form distribution characteristics in soil with different acid-buffering capabilities, but they also showed some relevance (Table 7). In accordance with the correlation between pH and heavy metal speciation, acid-damage volume also had a positive correlation with exchangeable, Fe-Mn oxidative and organic bound Cd, but had not remarkable correlation with organic combined speciation. Carbonate bound and Fe-Mn oxidative Cu performed a notable correlation with acid-damage volume, and active speciation showed a positive correlation with acid-buffering capability. The relevance between exchangeable, bound of carbonate Pb and acid-damage volume was not prominent, but the correlation between bound of organic Pb was highly significant. The relationship between active Pb and acid-buffering capability was negative. The form distribution of active Cr demonstrated a remarkable negative correlation, and the exchangeable and bound organic Cr had a significantly negative correlation with acid-damage volume. The correlation between Zn and acid-damage volume differed from that of soil pH, which performed a negative correlation trend. The difference of correlation, between the heavy metal and soil pH, acid-buffering capability, was mainly because of soil acid-buffering capability leading to the discrepancy of soil pH. There was no linear relationship between the soil pH and acid-buffering capability, namely, a huge gap of acid-buffering capability existed in the same soil type or in various regions, and even they had the same soil pH.

CONCLUSIONS

The content of soil Cd, Cu, Pb, Cr and Zn was higher than that of the Second National Soil Survey. The content of these in most soils exceeded the first level of the Soil Environmental Quality Standard, and a few reached the second level. Apart from Cd and Pb, which polluted a large coverage of soil, others had slightly polluted. The Pb pollution degree ranged for extensive, while others lied in the transition degree from non-pollution and moderate pollution.

In terms of heavy metal speciation distribution, Cd existed mainly in the form of Fe-Mn oxide, and its distribution feature was as follows, Fe-Mn oxide bound > residual state > carbonate state > organic combined state > exchangeable state. Cu, Pb, Cr and Zn displayed mainly in the form of residual, and their distribution was as follows, residual > organic combined state > Fe-Mn oxide bound > carbonate state > exchangeable state. Active speciation of different heavy metals was correlated to each other and could be transformed mutually under certain conditions. Cd and Pb posed relatively great risk to the well-being whereas Cr and Zn posed relatively minor danger.

Soil heavy metal speciation was influenced by soil acid chemical characteristics and there was some degree of correlation between them. Under different acidic conditions, the disparity of soil heavy metal speciation distribution varied with the change of soil pH. The correlation between pH and heavy metal speciation differed from that of acid-buffering capability, which might be caused by the non-linear relationship between pH and acid-buffering capability.

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