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Multivariate analysis of heavy metal leaching from urban soils following simulated acid rain

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ABSTRACT

Heavy metal pollution in urban soils and its effect on urban ecological environment and human health are of increasing concern. In this study, a leaching experiment was conducted to assess the effect of simulated acid rain (SAR) on the migration of Cd, Pb, Cu, and Zn and their transformation and speciation in nine natural urban soils from Guangzhou, China, combined with two-dimensional hierarchical cluster analysis (2-D HCA) heat map and principal component analysis (PCA). The maximum concentrations of Zn and Cu in leachates were lower than Grade III of Chinese Environmental Quality Standards for Ground and Surface water, although being seriously contaminated with Zn (up to 19,570 mg/kg) and Cu (322.4), thereby suggesting that Zn and Cu leached from urban soils receiving SAR pose little risk of water pollution. However, the maximum concentrations of Pb and Cd in leachates exceeded Grade III and V of the standards, respectively. The proportions of metals in the non-residual fractions were generally decreased after the leaching test for S(oil)5 (with high concentrations of metals in leachate), representing the decreased contamination risk in the soil–water system. A similar trend was observed for S(oil)6 (with low concentrations of metals in leachate) in terms of Zn and Cu, whereas the proportions of Cd and Pb in the non-residual fractions of S6 increased after leaching. Significantly positive relationships were observed between the concentration of heavy metal in leachate and all fractions of heavy metal, as well as the total concentration of soil heavy metals. In terms of Zn, Cd, and Pb, there were negative relationships between the concentration of heavy metal in leachate and clay and silt, respectively, while the inverse relationships were observed for Cu. The release of heavy metals from soil was strongly associated with their fractions and such soil properties as pH, soil texture, and organic matter.

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1. Introduction

Urban soil is a dominant component in urban ecological systems and acts as the source and/or sink for contaminants [1,2]. As urban areas are densely populated, the environmental quality of urban soil has significant effect on urban environment and health of urban inhabitants [3]. Heavy metal contamination of urban soils has attracted considerable attention in the world, particularly in China with rapid urbanization [1,3]. The elevated heavy metal concentrations are almost universally reported in urban soils, although often with high variances [4].

In China, acid rain has been considered another major environmental problem since the late 1980s, especially in southern China [5]. Approximately half of the precipitation in Guangdong Province is acid rain and the direct economic loss due to acid rains is estimated at $4.4 billion for 1 year [6]. Acid rain has a severe effect on the environmental behavior of heavy metals in soil mostly due to the cation exchange in soil with major cations (e.g., H^+, Ca^{2+}, Mg^{2+}, and NH_4^+) accompanied by acid deposition [7,8]. Acid rain falling on land may enhance the release of heavy metal from soils, hence contaminating the groundwater and deteriorating the terrestrial and aquatic ecosystems [9]. Previous studies [10,11] emphasized that the pH of acid rain was the primary factor affecting the mobility and speciation of heavy metals in soil, but the impact of acid rain was also closely related to soil properties [9,12,13].

Guangzhou is the largest city in southern China, with around 80% of total population (14 million as of 2013) living in urban area. With the rapid development of urbanization and industrialization, Guangzhou has suffered from serious acid rain pollution and extensive heavy metal contamination of soil. Effects of acid rain on the mobility and speciation of heavy metals based on naturally contaminated urban soil have rarely been reported. Hierarchical cluster analysis (HCA) is a multivariate technique for sorting individuals with similar characteristics into groups [14]. Principal component analysis (PCA) is used to reduce the dimensions of data, thereby visualizing overall patterns and trends within the dataset [15], and identify which parameters are most

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Table 1
Concentrations of Zn, Cu, Pb, and Cd and physico-chemical properties for studied soils.

Sample	Zn	Cu	Pb	Cd	OM	TP	pH	Particle size distribution (%)		
	mg/kg	mg/kg	mg/kg	mg/kg	g/kg	g/kg		2–0.05 mm	0.05–0.002 mm	<0.002 mm
S1	154.6	14.1	101.5	0.58	25.11	0.25	7.58	45.84	38.12	16.04
S2	334.6	252.8	272.6	2.6	70.88	0.43	6.55	25.48	45.04	29.48
S3	262.8	176.1	219.1	1.26	76.43	0.38	6.81	33.21	40.48	26.31
S4	203.1	20.4	51.6	0.28	26.74	0.28	6.57	36.76	38.84	24.4
S5	19570	322.4	3805	193.8	93.69	0.48	8.41	62.71	24.42	12.87
S6	358.2	114.3	183.2	0.72	78.24	1.16	8.06	63.43	23.59	12.97
S7	416.8	182.9	192.9	1.78	84.85	0.79	7.01	31.32	42.44	26.24
S8	119.1	37.6	115.5	0.33	82.42	0.32	8.33	57.48	25.48	17.04
S9	157	62.5	80.7	0.36	82.22	0.23	6.94	57.48	26.36	16.16

S1 from commercial sites, S2 and S6 from roadsides, S3 and S4 from public building areas, S5 and S7 from industrial sites, S8 from university campus, S9 from public park; OM represents organic matter, TP represents total phosphorus.

important in separating classes [16]. PCA also allows for the evaluation of relationships between observed variables [17,18]. These statistical techniques have been applied to data from a variety of research fields, including environmental and soil sciences [14], but with almost no report on their use to characterize releases of heavy metals after simulated acid rain (SAR), especially the evaluation of the relationships between variables and the application of two-dimensional (2-D) HCA heat map. In this study, we conducted batch column experiments with SAR solutions and nine natural urban soils from Guangzhou. The aims of this study were to: (1) assess the risk of heavy metals (Cd, Pb, Cu, and Zn) in urban soils for water quality affected by SAR; (2) use 2-D HCA heat map and PCA to cluster soils in terms of heavy metal releases from soils after SAR; (3) investigate the changes in speciation fractions of heavy metals in urban soils affected by SAR; and (4) identify how soil properties affect the releases of heavy metals in response to SAR.

2. Materials and methods

2.1. Soil collection and analysis

A total of nine soil samples were collected to represent different land use types, namely urban parks, residential areas, roadsides, and industrial areas from the central urban districts of Guangzhou, where acid rain is a concern. The area has a typical subtropical monsoonal climate, characterized by a mean annual temperature of 21.4 °C to 21.8 °C and a mean annual rainfall of 1782.9 mm [19]. The composite soil samples were collected to a depth of 10 cm by mixing five subsamples within 2 m^2 for each sampling site. Soil samples were air-dried, ground to go through a 2 mm nylon sieve, and homogenized, before preparing the soil column [3].

Sub-samples were further ground with an agate grinder to pass through a 0.15-mm nylon sieve, which were then used to determine selected heavy metals, soil organic matter, and total phosphorus. The concentrations of heavy metals (Cd, Pb, Cu and Zn) in soil, as well as such selected soil properties as pH, organic matter, and particle size distribution were determined according to the methods described by Li et al. [19]. Soil Cd, Pb, Cu, and Zn were digested with a mixture of concentrated nitric acid, hydrofluoric acid, and hyperchloric acid in a polyvinylfluoride crucible, followed by graphite furnace atomic absorption spectrophotometry (GFAAS-AA800, PerkinElmer Inc.) for Cd and Pb and flame atomic absorption spectrophotometry (Hitachi Z-5000) for Cu and Zn, respectively. The results of analyzed metals in a standard soil reference material were in good agreement with the expected values within 10%, and the relative standard deviation of replicates was less than 10%. Soil pH was measured at a ratio of 1:2.5 (weight/volume, soil:distilled water), organic matter was determined using the wet oxidation method, particle-size distribution was determined using the pipette method, and total phosphorus was determined by the molybdenum blue method [20]. The basic physico-chemical properties and total concentrations of heavy metals for nine soil samples prior to leaching are summarized in Table 1.

The chemical partitioning of heavy metals in soils was performed using the modified four-step BCR procedure as previously described [21]. According to the sequential extraction method [22], four operationally defined chemical forms of metals were isolated HOAc extractable, reducible, oxidizable, and residual fractions. The values of four fractions summed up were compared with the total concentration to check the recovery, and the recovery values were found satisfactory (data not shown).

2.2. Preparation of simulated acid rain

In order to reflect the characteristics of real acid rain in southern China, SAR was prepared according to the study of Huang et al. [23] who studied the acid rain in Guangzhou, with a molar ratio of SO_4^{2-} and NO_3^- at 4:1, with the pH adjusted to 4.0. SAR used in the experiments was made of $CaSO_4$, $(NH_4)_2SO_4$, $MgSO_4$, NaF, NH_4NO_3, and KNO_3 at the respective amounts (Table 2).

2.3. Leaching experiment

Leaching experiments were performed using PVC cylinders (4.5 cm inner diameter, a 20 cm height), and 200 g of air-dried soil (<2 mm) was added to each column (Fig. 1). A 2-cm depth quartz sand and a piece of 150 μm nylon mesh were applied at the bottom to retain soil. A piece of quantitative filter paper and another quartz sand layer were placed on top of soil column. The column was slowly saturated with deionized water from the bottom until field-holding capacity was reached and then stabilized for 24 h prior to leaching tests. SAR solution (100 mL) was slowly pumped in to percolate through columns at a rate of about 12.5 mL/h at the top of the column in every 48 h with the total application of 12 times, i.e., each column was flushed with 1200 mL of SAR solution. Nine leaching treatments were carried out in triplicates. Leachate samples were collected at 4-day intervals from the bottom of columns and six leachates were collected from each column. After thoroughly mixing and drying, soils in the column were ground to measure the total and chemical speciation concentrations of Cd, Pb, Cu, and Zn.

Table 2
Components of simulated acid rain (in mg/L).

Component	$CaSO_4 \cdot 2H_2O$	$(NH_4)_2SO_4$	$MgSO_4 \cdot 7H_2O$	NaF	NH_4NO_3	KNO_3
Concentration	11.696	0.859	1.725	0.756	1.926	0.606

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