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ORIGINAL ARTICLE

Multivariate analysis of heavy metals in surface sediments from lower reaches of the Xiangjiang River, southern China

Longjiang Mao · Duowen Mo · Yuanyuan Guo · Qiang Fu · Jinghong Yang · Yaofeng Jia

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Abstract The concentrations of heavy metals (Cr, Mn, Co, Ni, Cu, Zn, Cd, and Pb) in 16 samples collected from the lower reach (Changsha–Xiangtan–Zhuzhou section) of the Xiangjiang River in southern China were determined by high-resolution inductively coupled plasma mass spectroscopy (HR-ICPMS). Multivariate analysis, such as principal component analysis and cluster analysis, coupled with correlation coefficient analysis, was used to analyze the analytical data and to identify possible pollution sources of heavy metals. The results showed that the eight studied heavy metals accumulated in the sediments from the lower Xiangjiang River, especially Mn, Cu, Zn, Pb and Cd, which were 2.0–2.6, 1.7–2.6, 3.5–3.8, 3.2–3.6 and 189.5–152.8 times the soil trace element background for Hunan Province and UCC background values, respectively. Principal component analysis and cluster analysis, coupled with correlation coefficient analysis, revealed that the sediments from lower Xiangjiang River were mainly influenced by two sources: Cr, Co, Ni, Cu, Zn, Cd and Pb mainly originated from industrial sources, whereas Mn was derived from both industrial and natural sources, but mainly from natural sources due to weathering and erosion.

Keywords Heavy metals · Sediment · Multivariate analysis · Xiangjiang River

L. Mao (✉) · Q. Fu
School of Marine Sciences, Nanjing University of Information Science and Technology, Nanjing 210044, China
e-mail: mlj1214@163.com

D. Mo · Y. Guo
College of Urban and Environmental Sciences, Peking University, Beijing 100871, China

J. Yang
Department of Earth Sciences, Nanjing University, Nanjing 210093, China

Y. Jia
School of Management, North University for Nationalities, Yinchuan 750021, China

Introduction

River sediments play an important role as pollutants and provide a reasonable history of pollution in the river (Gibbs 1977; Filgueiras et al. 2002; Jain 2003; Davutluoglu et al. 2011). Sediments act as both carriers and sinks for contaminants in aquatic environments (Soares et al. 1999; Li et al. 2009). Trace elements, especially the heavy metals, are among the most common and significant environmental pollutants (Davutluoglu et al. 2011). Due to rapid urbanization and industrialization, heavy metals are continuously introduced into the river, estuarine and coastal sediments (Dassenakis et al. 1996; Jha et al. 2003; Xia et al. 2011). Heavy metals are added to an aquatic system by natural or anthropogenic sources (Förstner and Wittmann 1979; Zarazua et al. 2006). Natural sources mainly include weathering of soil and rock, erosion, forest fires and volcanic eruptions; whereas urban and industrial discharge, mining and refining and agricultural drainage caused by anthropogenic activities also discharge into the rivers (Zarazua et al. 2006; Pardo et al. 1990; Baughriet et al. 2007; Klavins et al. 2000). Knowledge of the distribution and concentrations of the heavy metals in the sediments will help detect the source of pollution in the aquatic systems (Förstner and Wittmann 1979; Davutluoglu et al. 2011). Over the past few decades, the pollution of rivers with toxic metals has been attracting considerable public attention (Facchinelli et al. 2001; Huang and Saulwood 2003; Singh Kunwar et al. 2005; Suthar et al. 2009; Mao et al. 2011).

The Xiangjiang River is one of the major and important rivers in Hunan Province, southern China. This river has a length of approximately 856 km (with 660 km in Hunan Province) and a catchment area of approximately 94,660 km^2 (Zhang et al. 1989). In Hunan Province, there are abundant reserves of non-ferrous metals, and most of the ores used for mining, mineral processing and smelting of non-ferrous and rare metals are found in the middle and lower reaches of the Xiangjiang River; and the effluents from these intensive mining and industrial activities are heavily loaded into the river (Zhang et al. 2009). Moreover, many large cities, such as Changsha, Xiangtan and Zhuzhou, are located in the middle and lower reaches of the Xiangjiang River. With the development of industrial production and enlargement of the cities, the middle and lower reaches of the Xiangjiang River have been polluted seriously day by day in recent years (Dong et al. 1992; Zeng et al. 2006). In this study, the Changsha–Xiangtan–Zhuzhou sections of the lower Xiangjiang River were investigated. The objectives were to determine the concentrations of heavy metals in the sediments from the lower reaches of Xiangjiang River and to analyze their potential sources.

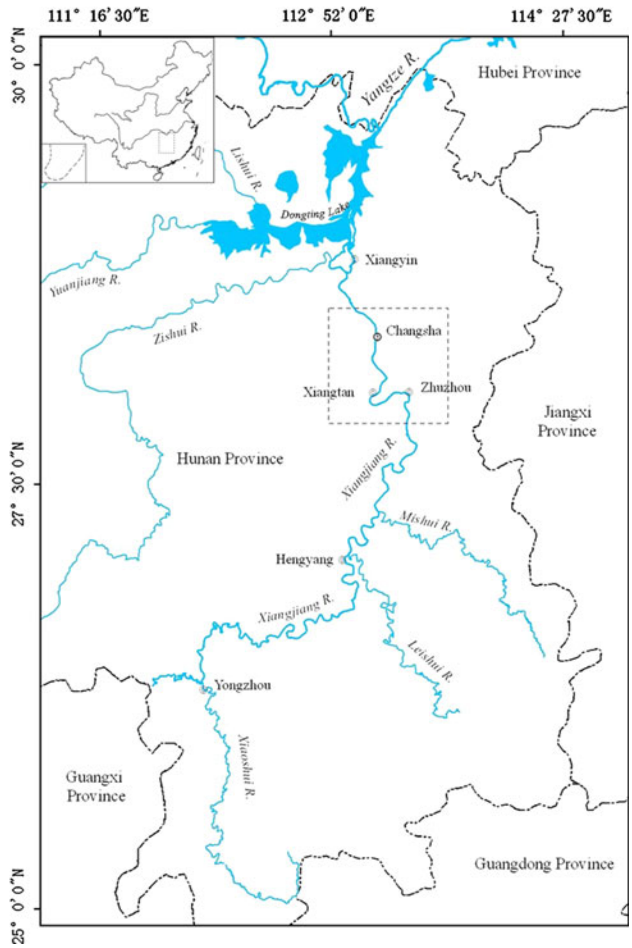

Fig. 1 Location of Xiangjiang River, southern China

Materials and methods

The study area

The Xiangjiang River, one of the main tributaries of the Changjiang River, originates in the Hai-Yang Mountains in Guangxi Zhuang Autonomous Region, and runs across Hunan Province from south to north and finally enters Dongting Lake (Fig. 1). The Xiangjiang River basin is exposed to a sub-tropical monsoon climate. The annual temperature averages between 17 and 18 °C. The mean annual precipitation varies from 1,200 to 1,700 mm; however, 60–70 % of the annual precipitation occurs in the rainy season from April to September, especially from April to June (Zhang et al. 1989; Dong et al. 1992). Due to the relatively warm and humid climate, the vegetation is dense with relatively intensive biological activities. Red soil, which is relatively rich in aluminum, minerals and some elements, such as Fe, Al and Ti, is the main type of soil in the Xiangjiang River drainage basin because of subtropical monsoon climate. The Xiangjiang River yields an average annual sand content of only 0.102–0.173 kg/m^3 and is lower than that of the rivers in northern China (Dong et al. 1992).

Sampling and experimental methods

River floodplain sediments in the Xiangjiang River are usually the finest grained (silt and clay, <63 μ) sediments and, therefore, can potentially enrich most of the pollutants. To avoid sediment grain-size effect, 16 sites of modern floodplain were randomly selected for sediment sampling. The location of the sampling sites is plotted in Fig. 2. Sediment samples were collected along the Changsha–Xiangtan–Zhuzhou section of the lower Xiangjiang River during 10–16 November 2010 using a pre-cleaned and acid-washed PVC spade, and the top 3–5 cm samples were immediately kept in acid-washed polythene bags. All the samples were transported to the laboratory where they were air dried for 2 weeks at ambient temperature for the heavy metal analysis of the sediments.

All of the samples for the chemical analysis were powdered in an agate mortar. About 100 mg fractions of powdered sediment were digested to a mixture of 10 ml HCl ($\rho = 1.19$ g/ml), 10 ml HNO_3 ($\rho = 1.42$ g/ml), 10 ml $HClO_4$ ($\rho = 1.68$ g/ml) and 10 ml HF ($\rho = 1.49$ g/ml) at

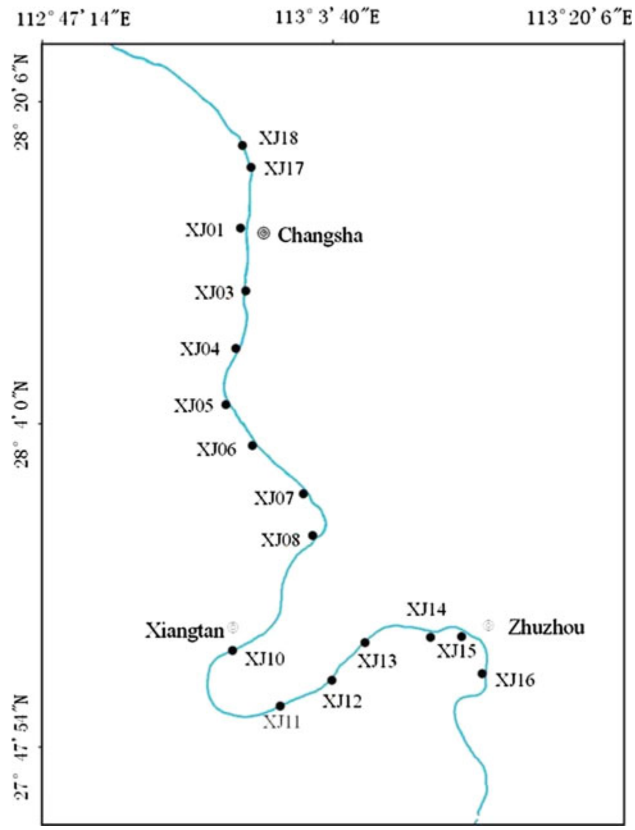

Fig. 2 Sampling locations in Xiangjiang River, southern China

180 °C in a microwave oven (ETHOS TOUCH CONTROL, Milestone Inc., Italy) (Song et al. 2011). Heavy metal element analysis of Cr, Mn, Co, Ni, Cu, Zn, Cd and Pb was determined by high-resolution inductively coupled plasma mass spectroscopy (HR-ICPMS) at the State Key Laboratory for Mineral Deposits Research of Nanjing University. Analyzed data were assessed for accuracy and precision using a quality assurance and quality control (QA/QC) program, which included reagent blanks, duplicate samples with 8 % of sediment samples and certified geochemical reference materials (GSS-2, GSS-3, GSS-4, GSS-6) with deviation <5 % (Jiang et al. 2007; Song et al. 2011).

Multivariate statistical analysis

Multivariate statistical analysis approaches, such as principal component analysis (PCA) and cluster analysis (CA), have been widely applied to assess the level of heavy metals in sediments (Tahri et al. 2005), soil (Facchinelli et al. 2001; Zheng et al. 2008), street dust (Han et al. 2006; Lu et al. 2010), etc. The PCA is used to reduce data and to extract a small number of latent factors for analyzing relationships among the observed variables (Han et al. 2006; Tokalioglu and Kartal 2006; Lu et al. 2010). It is also employed to identify the source of heavy metals in the sediments (natural or anthropogenic) (Facchinelli et al. 2001; Han et al. 2006; Lu et al. 2010). The CA is applied to identify different geochemical groups, clustering the samples on the basis of the similarities of their chemical properties (Han et al. 2006). In addition, the Pearson correlation coefficient (R) can be used to measure the strength of a linear relationship between the concentrations of various metals. In this study, PCA and CA, as well as Pearson's correlation coefficient analysis (R), were performed using the SPSS software (version 12.0 for Windows). Furthermore, the values for KMO (Kaiser–Meyer–Olkin) and Bartlett test of sphericity were also calculated.

Results and discussion

Heavy metal concentrations in surface sediments

Concentrations of eight heavy metals and their mean, standard deviation, minimum and maximum values in different stations of the Xiangjiang River are listed in Table 1. The total concentrations showed wide variations with Cr 12.27–87.99 μg g^{-1}, Mn 159.5–2414 μg g^{-1}, Co 2.21 23.14 μg g^{-1}, Ni 4.71–42.45 μg g^{-1}, Cu 5.31–188.89 μg g^{-1}, Zn 38.41–1250.47 μg g^{-1}, Cd 0.92–81.79 μg g^{-1} and Pb 18.85–198.0 μg g^{-1} (Table 1). The mean values of the heavy metal contents are arranged in the following decreasing order: Mn > Zn > Pb > Cr > Cu > Ni > Cd > Co for the sediments from the Xiangjiang River (Table 1). The mean values of Mn, Zn, Pb, Cr, Cu, Ni, Cd and Co are 1190.05, 266.57, 71.10, 51.99, 43.01, 24.57, 14.97 and 11.55 μg g^{-1}, respectively. These showed that Mn and Zn presented higher levels in Xiangjiang River sediments, whereas Cd and Co presented the lowest values.

In this study, soil trace element background for Hunan Province (CNEMC (China National Environmental Monitoring Center) 1990) and the upper continental crust (UCC) values (Taylor and McLennan 1985) are selected as the background values. The mean values of all the heavy metal concentrations for Xiangjiang River sediments are higher than soil trace element background for Hunan Province (CNEMC (China National Environmental Monitoring Center) 1990) other than Cr and Ni, but also higher than UCC values (Taylor and McLennan 1985). The average Cr, Co and Ni concentrations for the Xiangjiang River sediments are 0.8, 1.2, 0.8 times the soil trace element background for Hunan Province and 1.5, 1.2, 1.2 times UCC background values, respectively (Table 1), which imply that the Xiangjiang River sediments have been unpolluted or slightly polluted by Cr, Co and Ni. The mean of Mn, Cu, Zn and Pb concentrations was 2.0, 1.7, 3.0 and 2.6 times the soil trace element background for Hunan Province and

Table 1 Heavy metal contents in surface sediments collected from 16 sites of the Xiangjiang River

Stations	Cr ($\mu g\ g^{-1}$)	Mn ($\mu g\ g^{-1}$)	Co ($\mu g\ g^{-1}$)	Ni ($\mu g\ g^{-1}$)	Cu ($\mu g\ g^{-1}$)	Zn ($\mu g\ g^{-1}$)	Cd ($\mu g\ g^{-1}$)	Pb ($\mu g\ g^{-1}$)
XJ-01	16.52	334.5	4.11	8.34	6.73	66.30	1.27	29.66
XJ-03	50.87	1429.4	9.08	24.31	36.13	231.01	12.61	59.55
XJ-04	48.28	2012	11.42	24.93	29.37	242.55	11.31	93.36
XJ-05	76.02	2414	15.87	36.10	46.04	211.99	12.33	79.37
XJ-06	57.01	1916	13.60	29.66	30.02	179.47	9.66	59.72
XJ-07	79.72	2167	16.43	39.07	46.59	224.57	12.96	85.23
XJ-08	55.33	401.7	9.95	25.31	24.53	87.90	1.90	71.69
XJ-10	85.86	1872	19.44	40.05	61.35	526.94	26.32	121.8
XJ-11	48.69	1255	14.90	23.64	44.01	309.26	16.36	86.13
XJ-12	82.04	1523	18.87	40.80	60.47	461.10	37.43	92.61
XJ-13	87.99	1966	23.14	42.45	188.89	1250.47	81.79	198.0
XJ-14	22.40	453.3	6.32	11.43	11.86	114.37	5.26	45.92
XJ-15	25.88	456.2	5.81	13.12	16.43	107.81	4.77	35.21
XJ-16	19.49	229.1	3.48	7.39	9.50	65.28	1.46	26.53
XJ-17	62.51	452.5	10.20	21.81	70.91	147.67	3.21	33.95
XJ-18	13.27	159.5	2.21	4.71	5.31	38.41	0.92	18.85
Maximum	87.99	2414	23.14	42.45	188.89	1250.47	81.79	198.0
Minimum	13.27	159.5	2.21	4.71	5.31	38.41	0.92	18.85
Mean	51.99	1190.05	11.55	24.57	43.01	266.57	14.97	71.10
SD[a]	26.12	810.71	6.25	12.80	43.97	296.29	20.39	44.81
BV[b] [33]	44.00	450.00	10.30	21.2	20.00	76.00	0.33	22.00
UCC [34]	35.00	600.00	10.00	20.00	25.00	71.00	0.098	20.00

[a] Standard deviation

[b] Background values of soil in Hunan Province

2.0, 1.7, 3.8 and 3.6 times UCC background values, respectively. These suggest that a significant portion of the Mn, Cu, Zn and Pb metal originated from non-crustal or anthropogenic processes. Although the total Cd concentrations for the soil trace element background for Hunan Province and UCC are lowest, with mean values of 0.079 and 0.098 $\mu g\ g^{-1}$, respectively, higher Cd concentration with a mean of 14.97 $\mu g\ g^{-1}$ presented in the Xiangjiang River sediments, which is 189.5 times the soil trace element background for Hunan Province and 152.8 times the UCC background values, respectively.

Correlation coefficient analysis

The Pearson's correlation coefficients for heavy metals in the Xiangjiang River sediments are listed in Table 2. All the metal pairs show positive relations with each other at 99 % confidence level. Elements Cr, Co, Ni, Cu, Zn, Cd and Pb show significantly positive correlation with each other (>0.6) at $P < 0.01$, and some elemental pairs Cr–Co (0.946), Cr–Ni (0.982), Co–Ni (0.965), Cu–Zn (0.945), Cu–Cd (0.925), Zn–Cd (0.988), Zn–Pb (0.933) and Cd–Pb (0.909) show highly significantly positive correlation at 99 % confidence level. This may imply that elements Cr, Co, Ni, Cu, Zn, Cd and Pb have a common origin, such as industrial effluents. However, a relatively weaker correlation was found between the elemental pairs Mn–Cu (0.477), Mn–Zn (0.518) and Mn–Cd (0.514). This demonstrates that Mn is mainly of natural origin due to weathering and erosion.

Table 2 Pearson's correlations matrix for the heavy metal concentrations in Xiangjiang River sediments

	Cr	Mn	Co	Ni	Cu	Zn	Cd
Mn	0.786						
Co	0.946	0.815					
Ni	0.982	0.850	0.965				
Cu	0.711	0.477	0.773	0.678			
Zn	0.658	0.518	0.788	0.673	0.945		
Cd	0.649	0.514	0.782	0.676	0.925	0.988	
Pb	0.773	0.691	0.887	0.809	0.862	0.933	0.909

Significant at $P < 0.01$ (two tailed)

Principal component analysis

PCA was widely applied to identify sources of heavy metals in river sediments by applying varimax rotation

Table 3 Rotated component matrix for data from Xiangjiang River sediments

Elements	Component		Communalities
	Factor 1	Factor 2	
Cr	**0.89**	0.36	0.93
Mn	**0.76**	**0.54**	0.86
Co	**0.96**	0.23	0.97
Ni	**0.90**	0.4	0.98
Cu	**0.90**	−0.35	0.93
Zn	**0.92**	−0.38	0.99
Cd	**0.91**	−0.38	0.97
Pb	**0.96**	−0.11	0.93
Eigenvalue	6.5	1.06	
% Of variance explained	81.29	13.22	
% Cumulative	81.29	94.51	

PCA loadings >0.5 are listed in bold

Table 4 KMO and Bartlett's test

Kaiser–Meyer–Olkin measure of sampling adequacy	0.724
Bartlett's test of sphericity	
Approx. Chi-square	227.252
df	28
Sig	0.000

with Kaiser normalization. By extracting the eigenvalues and eigenvectors from the correlation matrix, the number of significant factors and the percent of variance explained by each of them were calculated by using the software package of SPSS v12.0. The results of the factor loadings with a varimax rotation, the eigenvalues and communalities are listed in Table 3. The calculated values for the KMO (Kaiser–Meyer–Olkin) and Bartlett test of sphericity) are shown in Table 4. The results show that two eigenvalues explain 94.51 % of the total variance. The first factor explains 81.29 % of the total variance and loads heavily on Cr, Mn, Co, Ni, Cu, Zn, Cd and Pb. Factor 2 is loaded primarily by Mn and accounts for 13.22 % of the total variance (Table 3). The values for KMO and Bartlett's test of sphericity are 0.724 and 222.252 (Table 4), respectively, and the significant level is 0.000 (<0.001). These show that the factor analysis for this study is suitable. Factor 1 should be industrial, which is also evident from the presence of various metal processing industries in the area. In China, the Xiangjiang River basin contains a number of large deposits of nonferrous metals, which have led to the establishment of an extensive metallurgical and chemical industry in the region, especially in the middle and lower reaches (from Zhuzhou to Changsha) of the river (Zhang et al. 2009). Changsha City, the capital of Hunan Province, is located primarily in the downstream of Xiangjiang River. Zhuzhou City, the largest industrial city, also lies in the downstream of Xiangjiang River. With long-time mining and smelting activities of non-ferrous metals, much wastewater has been discharged to the surrounding environment, and the levels of heavy metals in the Xiangjiang River have been significantly enhanced (Chen et al. 2004). In addition, the elevated wastewater, produced by urban development (Fig. 3a) and agricultural activities (Fig. 3b), discharge into the lower reaches of the Xiangjiang River. The source of factor 2 is natural, including weathering and erosion. However, it is found that Mn metal may not only be a natural source entering into the Xiangjiang River basin by weathering and erosion, but also has anthropogenic origin from to industrial wastes.

Cluster analysis

The variables were standardized by means of *z* scores prior to CA, and then Euclidean distances for similarities in the variables were calculated. Finally, hierarchical clustering by applying Ward's method was performed on the standardized data set (Tokalioglu and Kartal 2006). The CA

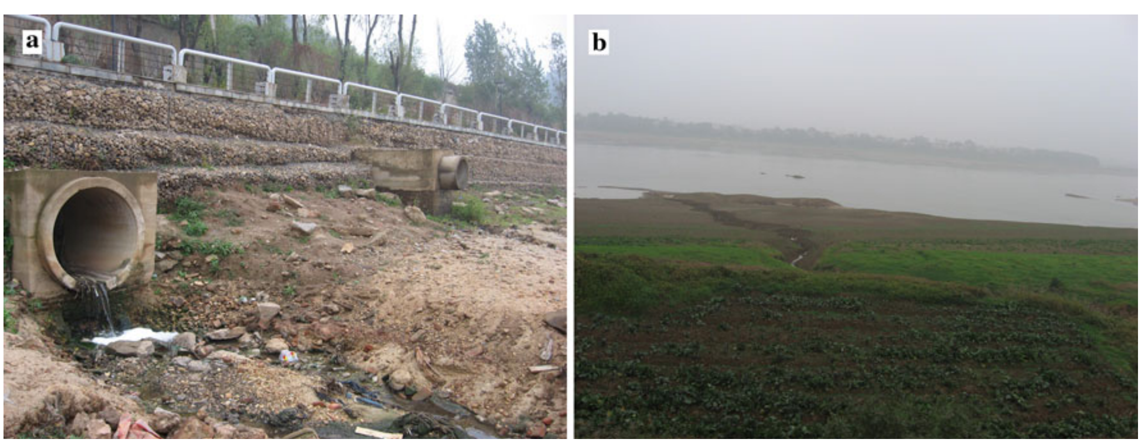

Fig. 3 The wastewater from urban and agriculture activities' discharges into the lower Xiangjiang River

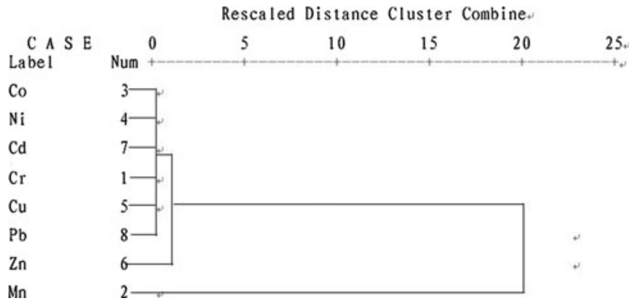

Fig. 4 Dendrogram results from Ward method of hierarchical cluster analysis for the eight elements in the Xiangjiang River sediments

results for the heavy metals in Xiangjiang River sediments are demonstrated in Fig. 4 as a dendrogram. Figure 4 displays two clusters: (1) Co, Ni, Cd, Cr, Cu, Pb and Zn; (2) Mn. Cluster 1 may have anthropogenic origin, whereas cluster 2 may be from weathering and erosion. In summary, the analysis results are in concordance with those of PCA.

Conclusions

The concentrations of heavy metals (Cr, Mn, Co, Ni, Cu, Zn, Cd and Pb) in samples collected from the lower reach (Changsha–Xiangtan–Zhuzhou section) of the Xiangjiang River in southern China have been studied in this work. Heavy metals studied have accumulated in the sediments. The mean concentrations of these heavy metals in the Xiangjiang River sediments are higher than the corresponding natural background values for Xiangjiang River sediments and UCC background values.

Multivariate analysis such as PCA and CA, coupled with correlation coefficient analysis, has proved to be an effective tool to identify sources of these heavy metals in river sediments. Heavy metals, Cr, Co, Ni, Cu, Zn, Cd and Pb, mainly have anthropogenic sources, such as industrial activities and urban development, whereas Mn has mixed sources, derived from both industrial and natural sources, but mainly from natural sources due to weathering and erosion.

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