



Study of heavy metal speciation in branch sediments of Poyang Lake

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Abstract

This study concentrates on the environmental pollution level of sediments in the six branches of Poyang Lake, the biggest fresh water lake in China. This is the first systematic report on the speciation analysis of heavy metals (Cu, Co, Cd, Pb, and Ni) in the six branches of the lake. A reported analytical procedure involving a five-step sequential extraction is used for the partition of particulating heavy metals. The sediment samples are analyzed using flame atomic absorption spectroscopy (FAAS). Experimental results obtained from five replicate samples of fluvial bottom surface sediments at the sampling points demonstrated that the relative standard deviation of the sequential extraction procedure was generally better than 10% (Cd except). The average extracted contents of the five elements, analyzed after all five steps, are found to be (mg/kg) for Cu: 26.89, Co: 16.25, Cd: 1.08, Pb: 37.98, and Ni: 20.46. The content of the exchangeable species was generally lower. Except Cu, the percentage of the species bond to organic matter was lower than 20%. The fractions containing the most metal for Cu, Co, and Ni were the residues (52.26%, 45.28%, and 74.82%, respectively).

Key words: Poyang Lake; speciation analysis; heavy metal; sequential extraction

Introduction

Poyang Lake (28°4′–29°46′N, 115°49′–116°46′E) is located in the north of the Jiangxi Province and it lies on the southern bank of the Yangtze River. It is the biggest fresh water lake (21224.4 km²) in China. Ganjiang River, Fuhe River, Raohe River, Xinjiang River, Xiushui River, and Xihe River are its six main branches. Dexing and Yongping copper mines are at the side of the Poyang Lake. The metal contents are very high at the conjunction of the rivers and Poyang Lake, where the aquatic environment has been contaminated. The results from the determination of heavy metals in sediments reflect the impacts of industrial activities and urban development (Fang *et al.*, 2005). The total contents of heavy metals in the sediment can give an insight into the chemical speciation. The toxicity of the metal particularly depends on their chemical forms rather than on their total contents, and therefore, speciation studies increasingly gain importance (Liu *et al.*, 2007). However, thus far, there has been no article that has given a systematic report about the species analysis of heavy metals in the sediments of the six main branches of the Poyang Lake. It is extremely significant to analyze the heavy metal fractions and to find out the distribution in different fractions. The study is conducive to the study on the fraction background concentration of heavy metals in this area.

There is a great diversity of possible associations of heavy metal elements with the various substrates. They can be adsorbed on the particle surfaces of clays, iron, and manganese oxyhydroxides or organic matter present in the lattice of secondary minerals, such as, carbonates or sulfides; occluded in amorphous iron and manganese oxyhydroxides, sulfides or remains of biological organisms; and present in the lattice of primary minerals (Tessier *et al.*, 1979). Heavy metals in sediments can be classified, on the basis of the primary accumulation mechanism, as adsorptive and exchangeable, bond to carbonate phases and Mn-oxide, bond to organic matter and sulfides, and detrital or lattice metals (Salomons and Förstner, 1980). Tessier set up the five-step sequential extraction method to separate five metal fractions in soil and sediment samples. A number of studies on the metal distribution in river sediments and suspended particles, and on speciation of metals have been performed (Santos *et al.*, 2002; Wisotzky and Cremer, 2003; He *et al.*, 2003; Alonso *et al.*, 2004; Chen *et al.*, 2005; Nowierski *et al.*, 2006).

In this study, six main branches of the Poyang Lake were studied, to estimate their environmental pollution levels of five heavy metal elements. An improved five-step sequential extraction procedure (Akçay *et al.*, 2003) was used to determine the respective distribution of heavy metals (Cu, Co, Cd, Pb, and Ni) in the following fractions: exchangeable species, bond to carbonates, bond to Fe–Mn oxides, bond to organic sulfurs, and bond to residue.

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1 Materials and methods

1.1 Chemicals and reagents

All chemicals used are of analytical grade. All solutions were prepared by double distilled water.

1.2 Sample collection and pretreatment

Sediment samples were collected on 14–18 May, 2005. Six main branches of Poyang Lake, Xiushui River, Xihe River, Raohe River, Fuhe River, and Ganjiang River I and II, were selected to collect sediment samples, (Xinjiang River, another branch of the Poyang Lake, flows rapidly and the riverbed is sandy, hence, the authors had not collected suitable sediment samples). A total of six surface sediment samples were collected from various entrances of the rivers mentioned earlier, which were rushing into the Poyang Lake (Fig.1). Every sample consisted of approximately 300 g of surface layer sediment (0–2 cm). Polyethylene scoops and cans were used exclusively for sampling and storage. The samples were put in ice bags during the transportation to the laboratory and stored in a deep-freeze unit until the drying procedure. Samples were dried at room temperature in the laboratory. The dried sediment samples were sieved through a 73- μ m screen for the analysis.

1.3 Locations of sediments and environmental parameters

The location of each sampling point was confirmed by a 3011 GPS Compass (Thales Navigation Company, America). The environmental parameters of the river water above the sampling points were confirmed by a 5-star portable multi-parameters measuring instrument (Thermo Orion company, America). The results are shown in Table 1.

1.4 Metal analysis

An atomic absorption spectrometer (Model 4129) (Electronic Corporation of India), equipped with an air-acetylene flame was used to monitor metal ions. The analysis lines for Cu, Co, Cd, Pb, and Ni were 324.8, 240.3, 228.8, 283.3, and 232.0 nm, respectively. The detection limits for the elements mentioned earlier were 0.01, 0.03, 0.002, 0.05, and 0.02 mg/L, respectively.

1.5 Speciation process

The five-step sequential extraction procedure (Akca *et al.*, 2003) was applied to determine heavy metal fractions in the collected sediments.

The first fraction that contained exchangeable species

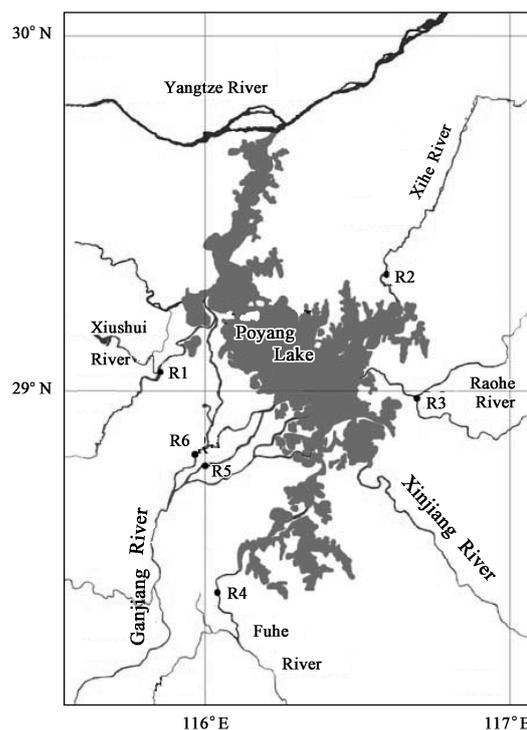


Fig. 1 Working area and sampling points in the six branches of Poyang Lake.

of trace metals (S1) was obtained with the help of the following procedure: each solid sample was extracted at room temperature for 1 h, with 10 ml of magnesium chloride solution (1 mol/L $MgCl_2$ buffered at pH 7.0) with continuous shaking. The heavy metals adsorbed on the sediments were exchangeable and could be in equilibrium with the water's ionic composition, which was typical for sorption-desorption processes.

The second fraction contained the species bond to carbonates (S2). The residue from the first extraction was leached for 5 h at room temperature with 10 ml of 1 mol/L NaAc adjusted to pH 5.0 with acetic acid (HAc) maintained.

The residue from the second extraction was extracted with 20 ml of 0.004 mol/L $NH_2OH \cdot HCl$ dissolved in 25% (v/v) HAc. The latter experiment was performed at $96 \pm 3^\circ C$, with occasional agitation, and the time needed for complete dissolution of free iron oxides was determined and used. The third fraction included trace metal species bond to Fe-Mn oxides (S3).

Three milliliters of 0.02 mol/L HNO_3 and 5 ml of 30% H_2O_2 adjusted to pH 2.0 with HNO_3 , were added to the residue from the third extraction. The mixture was heated at $85 \pm 2^\circ C$ for 2 h, with occasional agitation.

Table 1 Locations of sampling points and environmental parameters

Sampling site	Longitude	Latitude	Water temp. ($^\circ C$)	Dissolved oxygen (mg/L)	pH	Conductivity ($\mu S/cm$)	E_h (mV)
R1	115°48'37.9"E	29°02'12.2"N	27.8	2.9	6.76	728	-249
R2	116°35'14.5"E	29°21'47.4"N	35.2	3.2	6.73	88.0	-215
R3	116°41'25.7"E	28°57'14.2"N	32.8	3.9	6.41	257	-241
R4	116°05'22.7"E	28°21'27.8"N	32.5	3.3	6.45	65.4	-195
R5	116°00'58.1"E	28°46'20.4"N	32.0	3.5	7.37	110.7	-182
R6	115°57'31.6"E	28°49'25.0"N	31.9	4.0	7.34	105.5	-125

A second 3 ml aliquot of 30% H₂O₂ adjusted to pH 2.0 with HNO₃ was then added, and the sample was heated again to 85 ± 2°C for 3 h with intermittent agitation. After cooling, 5 ml of 3.2 mol/L NH₄Ac in 20% (v/v) HNO₃ was added and the sample was diluted to 20 ml and agitated continuously for 30 min. The addition of NH₄Ac was designed to prevent adsorption of the extracted metals onto the oxidized sediment. The fourth extract thus involved the trace metal species bond to organic matter (S4).

Finally, the residue from the fourth extraction was digested with a mixture in a ratio of 4:1:1:1, of HNO₃:HF:HClO₄:HCl for 3 h in a water-bath. The residue dissolved entirely and it was diluted with 100 ml double distilled water (S5).

1.6 Precision of method

The sediment samples collected from the Xiushui River (R1), Xihe River (R2), Raohe River (R3), Fuhe River (R4), Ganjiang River I (R5), and II (R6) were treated repeatedly, five times, by the five-step sequential extraction procedure. The relative standard deviations (RSD) of total contents for heavy metals (Cu, Co, Cd, Pb, and Ni) were calculated, which are shown in Table 2. From the results, it was found that the RSD of heavy metals, except for Cd were lower than 10%. This proved that the method precision was good. The RSD of Cd was large, the reason might be that the quantity of Cd in the sediment samples was very low (only 2.80% of total Pb content). An error in the operation influenced the final result seriously.

Table 2 Relative standard deviation (RSD) of total contents after the sequential extraction procedure

Sampling site	RSD (%)				
	Cu	Cd	Co	Pb	Ni
R1	5.21	5.96	3.16	3.13	2.89
R2	1.03	15.81	3.77	1.76	1.02
R3	2.43	20.02	2.28	7.21	3.67
R4	1.64	17.33	1.29	4.31	1.48
R5	0.55	3.42	1.50	1.26	3.70
R6	0.66	8.78	0.38	3.14	0.79

2 Results and discussion

All the sediment samples collected were estimated, to confirm the fraction contents of heavy metals by the five-step sequential extraction procedure. The arithmetical mean and standard deviation of each heavy metal fraction were calculated and are shown in Table 3 and Fig.2. In the mean percentages of the exchangeable species (S1) in six sediments, for Cu, Co, Pb, and Ni, the main available portions were low (lower than 2%). However, the S1 mean percentage for Cd was very high (11.93%), which proved that the biological availability of Cd was high. The fraction containing the most metal for Pb was the species bond to Fe-Mn oxides (S3), the mean percentage of S3 in the six sediments was 38.61%. The main fractions for Cu, Co, and Ni were the residual species (S5). The biological availabilities of those heavy metals were relatively lower.

2.1 Copper

Copper is a very important element, which could influence the metabolism of the human body and it is also a nutritional element for living beings. But if the intake is too much, it will cause toxicity (Fan *et al.*, 2002). The mean value of total copper in each sediment sample is relatively higher (26.89 mg/kg), which approaches the finger (25.0 mg/kg) in the Fuhe River sediments (Lü, 1994). However, the author's study took the advantage over his by doing fraction analysis. More than half the copper (mean value 52.26%) extracted was leached in the last step of the procedure. The fraction containing most of the copper was also the residual (S5) in the sediment samples of BM and Gediz rivers (Mark and Robert, 2002). The mean percentage of the exchangeable species in six sediments, that is, the most available fraction, was only 1.07%. Many studies showed similar results (Mark and Robert, 2002; Zhu *et al.*, 2001; Bi, 2003; Dong *et al.*, 2004; Wu *et al.*, 2004).

From analysis of the geographical distribution, it was found that the mean content of total copper was the highest in the Fuhe River sediment sample (45.34 mg/kg). The mean contents of total copper in the Raohe River, Xiushui

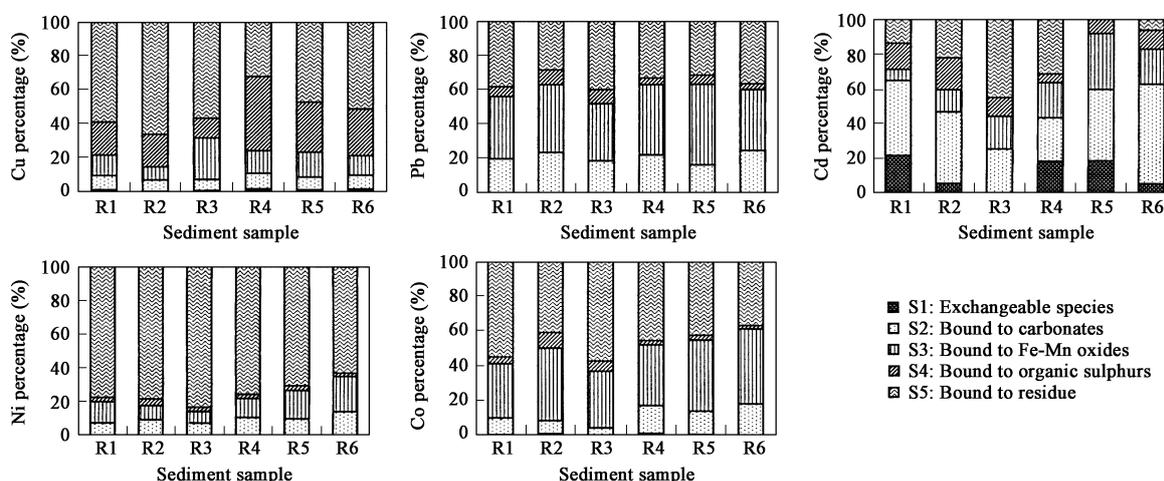


Fig. 2 Percentage distribution of extracted metals after the five-step sequential extraction procedure.

Table 3 Distribution of heavy metals in five fractions

Element	Sampling site	S1 (mg/kg)	S2 (mg/kg)	S3 (mg/kg)	S4 (mg/kg)	S5 (mg/kg)
Cu	R1	0.32±0.05	2.18±0.26	3.27±0.24	5.15±0.28	15.87±1.35
	R2	–	1.17 ±0.03	1.34±0.10	3.30±0.17	11.42±0.06
	R3	0.15±0.06	1.97±0.03	7.24±0.47	3.52±0.58	17.00±0.62
	R4	0.72±0.02	4.15±0.14	5.98±0.41	19.84±0.49	14.70±0.41
	R5	0.28±0.12	1.93 ±0.10	3.63±0.46	7.50±0.27	12.03±0.52
	R6	0.27±0.07	1.36±0.14	1.89±0.10	4.62±0.23	8.58±0.27
Cd	R1	0.19±0.02	0.37±0.04	0.06±0.01	0.13±0.03	0.12±0.03
	R2	0.05±0.06	0.38±0.03	0.12±0.02	0.17±0.06	0.20±0.03
	R3	–	0.30±0.04	0.22±0.08	0.13±0.01	0.52±0.19
	R4	0.25±0.14	0.34±0.07	0.27±0.04	0.07±0.02	0.42±0.15
	R5	0.23±0.02	0.52±0.02	0.40±0.02	0.10±0.01	–
	R6	0.05±0.04	0.54±0.02	0.19±0.03	0.10±0.04	0.06±0.05
Co	R1	0.02±0.02	1.60±0.22	5.09±0.13	0.68±0.06	9.01±0.37
	R2	0.14±0.04	1.26±0.12	7.05±0.10	1.52±0.05	6.91±0.43
	R3	–	0.60±0.08	4.56±0.20	0.86±0.10	8.04±0.23
	R4	0.19±0.08	2.59±0.17	5.64±0.09	0.39±0.08	7.35±0.17
	R5	0.09±0.03	2.39±0.09	7.29±0.16	0.49±0.07	7.58±0.10
	R6	–	2.94±0.14	6.93±0.15	0.35±0.12	5.92±0.20
Pb	R1	–	6.13±0.44	11.41±0.27	1.83±0.17	12.08±0.32
	R2	–	5.92±0.38	9.97±0.59	2.23±0.11	7.23±0.20
	R3	–	4.90±0.11	8.76±1.52	2.15±0.347	10.69±0.95
	R4	–	9.39±1.05	17.55±0.25	1.64±0.238	14.37±1.55
	R5	–	10.27±0.15	29.69±0.72	3.33±0.348	20.25±0.67
	R6	–	9.28±0.435	13.49±0.578	1.25±0.589	14.07±0.86
Ni	R1	–	1.65±0.693	3.08±0.109	0.64±0.096	18.66±0.44
	R2	–	2.02±0.128	1.84±0.359	0.90±0.106	17.60±0.15
	R3	–	1.91±0.228	1.92±0.355	0.77±0.357	23.22±0.72
	R4	–	1.46±0.090	1.60±0.054	0.36±0.036	10.66±0.16
	R5	–	1.68±0.058	3.03±0.179	0.53±0.124	12.46±0.45
	R6	–	2.33±0.090	3.55±0.127	0.35±0.012	10.55±0.24

Data are the mean ± SD ($n = 5$); “–” means data lower than the detection limit. S1: exchangeable species; S2: bound to carbonates; S3: bound to Fe-Mn oxides; S4: bound to organic sulphurs; S5: bound to residue.

River, Ganjiang River I, Xihe River, and Ganjiang River II sediment samples were 29.88, 26.79, 25.37, 17.23, and 16.72 mg/kg, respectively. Except the species bond to organic matter (S4), the mean total contents of copper in the Fuhe River and the Raohe River sediment samples were similar (25.54 and 26.36 mg/kg, respectively). Although the latitude of Fuhe River was lower, the weather was warmer and more humid, which made hydrophytes grow and flourish. A great deal of organic matter was produced and became a portion of the sediment. Therefore, the S4 mean content of the Fuhe River (19.80 mg/kg) was higher than that of the Raohe River (3.52 mg/kg), whose riverbed was sandy. The first three fractions (S1 + S2 + S3), were relatively available fractions, the mean contents of the Fuhe River, Raohe River, Ganjiang River I, Xiushui River, Ganjiang River II, and Xihe River sediment samples were 10.84, 9.36, 5.84, 5.77, 3.52 and 2.51 mg/kg.

2.2 Cadmium

The mean total content of cadmium (1.08 mg/kg) in the sediment samples was the lowest in five heavy metals. It was slightly higher than the background of Poyang Lake (0.75 mg/kg) (Lü, 1994). The S1 mean percentage was higher than 10% (11.63%), the (S1 + S2 + S3) mean percentages for Xiushui River, Xihe River, Raohe River, Fuhe River, Ganjiang River I, and Ganjiang River II reached as high as 71.31%, 59.65%, 44.08%, 63.70%, 92.04%, and 82.94%. The mean percentage of the stable residual fraction (S5) in the six sediments was lower than

20% (19.61%). The results improved when cadmium was very active. The fraction containing the most cadmium (mean value 50.56%) was the species bond to carbonates (S2) in the sediments, where compounds of cadmium in sediments were released by the HAc-NaAc buffer solution (Pickering, 1986). The phenomenon of the reactive fraction containing most of the cadmium also appeared in the sediments of the Volvi and Koronia Lakes (Fytianos and Lourantou, 2004), the oxic estuarine (Gmez-Ariza *et al.*, 1999), the Iberian Pyrite Belt Rivers (Galán *et al.*, 2003), and the Xiaonan Lake (Wang *et al.*, 1999). The mean total amount of cadmium in the sediment samples of each river was 1.35 mg/kg (Fuhe River), 1.26 mg/kg (Ganjiang River I), 1.16 mg/kg (Raohe River), 0.94 mg/kg (Ganjiang River II), 0.91 mg/kg (Xihe River), and 0.86 mg/kg (Xiushui River), respectively. Although the mean total amount of cadmium in the sediments was lower than that of other metals, the amount poured into rivers should be managed. As the amounts increase, the active fractions (more than half of the total amount) would be easily released, and would then influence the environment and human beings through the food chain.

2.3 Cobalt

The mean total cobalt contents in the six sediment samples were not very high (16.25 mg/kg), and approach the finger (17.1 mg/kg) in the Yenshui River sediments (Chang *et al.*, 1998). The species bond to the mean percentage of the Fe-Mn oxide (S3) and the residual fraction

(S5) mean percentage were relatively high, 37.31% and 46.28%, respectively. The percentage of S3 and S5 were also high in the sediment samples of the Suez Canal (El-Azim and El-Moselhy, 2005). Only 0.44 mean percentage of cobalt was extracted in the first step (S1). The result was similar to the S1 percentage (1%) in the sediment samples of rivers in Latvia. In the matter of geographical distribution, the mean total cobalt content (17.835 mg/kg) in the sediment samples of the Ganjiang River was the highest one. The mean values of the other five rivers, Xihe River, Xiushui River, Fuhe River, Ganjiang River II, and Raohe River, were 16.89, 16.39, 16.15, 16.14, and 14.066 mg/kg. There was no obvious diversity in the total cobalt content of each river. The first three reactive fractions (S1 + S2 + S3) mean contents were 9.87, 9.77, 8.46, 8.41, 6.71, and 5.16 mg/kg for Ganjiang River II, Ganjiang River I, Xihe River, Fuhe River, Xiushui River, and Raohe River.

2.4 Lead

The mean total content of lead (37.98 mg/kg) in the sediment samples was the highest in the five determined metal elements. The result was slightly lower than the total value of Pb (39.5 mg/kg) in the sediment of the Raohe River (Lü, 1994). The mean percentage of each fraction was 0.00%, 20.13%, 39.87%, 5.46%, and 34.53% for S1, S2, S3, S4, and S5, respectively. From the results it was seen that the S1 mean content was very low (lower than the detection limit). The fraction containing the most lead was a relative reactive fraction (S3), and the mean percentage of the first three reactive fractions (S1 + S2 + S3) was more than half (60%). S3 was also the fraction containing the most lead in the sediment samples of many rivers and lakes (Relic *et al.*, 2005; Galán *et al.*, 2003; El-Azim and El-Moselhy, 2005; Zhu *et al.*, 2001; Feng *et al.*, 2004). The mean total content of lead in the Ganjiang River I, Fuhe River, Ganjiang River II, Xiushui River, Raohe River, and Xihe River was 63.55, 42.98, 38.09, 31.45, 26.50, and 25.35 mg/kg, respectively. The mean contents of the first three fractions of the above rivers were 39.96, 26.94, 22.76, 17.54, 13.65, and 15.89 mg/kg, respectively. Lead should be managed seriously for large quantity and high percentage of reactive fractions.

2.5 Nickel

The mean total content of nickel was 20.46 mg/kg. The mean percentage of the first three active fractions (S1 + S2 + S3) was lower than one in third (22.34%). Many studies had also reported that the percentage of active fractions of nickel in the sediments was not high (Noverita *et al.*, 2004; Filgueiras *et al.*, 2004; Klavins *et al.*, 2000; Gómez-Ariza *et al.*, 1999). More than two in third nickel (mean value 74.82%) was extracted in the last step (S5). It had also been reported that the largest amount of nickel was associated with the residual fraction in some sediments (Relic *et al.*, 2005). In the analyzed sediments, nickel was associated in decreasing amounts in S5 (74.82%) > S3 (12.92%) > S2 (9.42%) > S4 (2.84%) > S1 (lower than detection limit) fractions of the analyzed sediments. In the matter of geographical distribution, the mean contents of total nickel

in the sediments were 27.82, 24.02, 22.36, 17.70, 16.78, and 14.08 mg/kg for Raohe River, Xiushui River, Xihe River, Ganjiang River I, Ganjiang River II, and Fuhe River, respectively. The mean contents of the first three active fractions (S1 + S2 + S3) for the above-mentioned rivers were 3.83, 4.72, 3.86, 4.71, 5.89, and 3.05 mg/kg. The low percentage active fraction was an advantage, to control the pollution in this area.

3 Conclusions

The five-step sequential extraction procedure was used to fractionate Cu, Co, Cd, Pb, and Ni in the investigated sediments in six branches of the Poyang Lake. Lead is the most important element in environmental pollution. Undoubtedly, lead brings about serious environmental harm. In view of its large mean of total amount, and more than 60% active fraction, it is better to exert a great deal of attention on lead. The content of lead in live sewage is relatively low. The main headwaters are industry wastewater and mineral exploitation; hence, these two aspects should be managed and controlled seriously. The second element of high content, copper, is a concern, coming from copper mine exploitation, but its main chemical fraction is the residue, which has little effect on environment and humans. The total content of cadmium is low, but its mean total percentage of the first three active fractions is very high, therefore, its input also must be managed meticulously. Most amounts of other heavy metals are extracted in inertia fractions. The lower mean percentage of active fractions is an advantage, to control the environmental pollution in the branches of the Poyang Lake.

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