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Distribution and chemical fractionation of heavy metals in recent sediments from Lake Taihu, China

Ji Shen · Enfeng Liu · Yuxin Zhu · Shouyun Hu · Wenchuan Qu

Abstract Lake Taihu is one of the most contaminated lakes in China. Surface sediment data show that the northern area of the Lake has the worst heavy metals pollution, and high heavy metal concentrations were attributed to discharge of untreated and partially treated industrial waste water from cities to the north of the lake. To study geochemical features and pollution history of heavy metals, total content and chemical fractionations of Cu, Fe, Mn, Ni, Pb, and Zn were analyzed for core sediments from western Lake Taihu using the speciation extraction procedure, proposed by the Commission of the European Communities Bureau of Reference (BCR), together with grain size and organic carbon measurements. Results show that sediments are composed of organic-poor clayey-fine silts for Cores MS and DLS, and have similar geochemical features shown by heavy metals. Cu, Fe, Ni, and Zn mainly are associated with the residue fraction, Mn is concentrated in the exchangeable-carbonate and residue fractions, and Pb is concentrated in the Fe–Mn oxide fraction and organic-sulfide fraction. The fractions of Ni, Pb, and Zn bound to Fe–Mn oxide show significant correlations with Mn from the Fe–Mn oxide fraction, and the organic-sulfide fractions of Cu, Mn, Ni, Pb, and Zn are correlated with TOC. The increase of Cu, Mn, Ni, Pb and Zn content and percentage of extractable fractions in the upper layers of the sediments are correlated with anthropogenic input of heavy metals due to rapid industrial development. This coincides with rapid economic development in the Taihu basin since late 1970s. Heavy metals in the surface sediments have certain potential biological toxicity as shown by the higher SEM/AVS ratio.

Keywords Heavy metals · Sediments · Distribution · Fractionation · Lake Taihu

Introduction

Lake Taihu, the third largest fresh water lake in China, is situated in the center of the Changjiang River Delta (Qin et al., 2007) (Fig. 1). With intensive development of agriculture and industry in this densely populated area since the 1950s, especially since 1978, untreated or partially treated wastewater from industry, agriculture and sewage increasingly has been discharged into Lake Taihu, which induced degeneration of lake ecology and water quality. The quality of Lake Taihu

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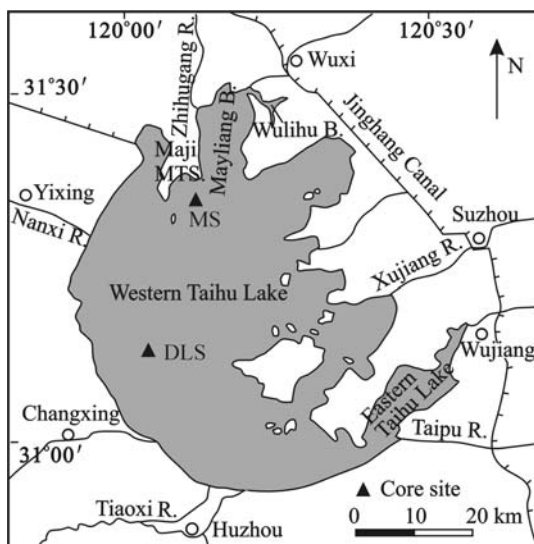
Fig. 1 Location of Lake Taihu and core site

water degraded 2–3 levels from the 1970s to 1990s (Lu, 1998), which caused about 1 billion RMB economic loss per year during the late 1980s, accounting for 3.12% of the local GNP (Ge, 1992).

As one of many environmental problems in Lake Taihu, heavy metal pollution has drawn great attention in recent years. A few studies have been conducted on heavy metal pollution of surface sediments (Dai & Sun, 2001; Qu et al., 2001; Yuan et al., 2002a), which mainly address heavy metal total concentration. However, work on chemical fractionation of heavy metals and pollution history of the sediments are rare (Wang et al., 2002; Rose et al., 2004). There is a lack of systemic investigation on distribution and chemical forms of heavy metals in sediment cores, which are an important record of historical pollution, biogeochemical recycling and toxicity for heavy metals (Luoma, 1983).

In the present study, two sediment cores in western Lake Taihu were obtained. Total concentration of heavy metals and their chemical forms deduced from the BCR method (proposed by the Community of the European Bureau of Reference) (Quevauviller et al., 1997) in the sediment cores are presented. The aim of this study is to understand geochemical characteristics of the heavy metals and pollution features in the sediments and their correlations with economic development and human activities in the Lake Taihu catchment.

Material and methods

Sample collection

According to sedimentary features and the degree of pollution in the sediments (Sun & Huang, 1993; Qu et al., 2001), two sediment cores (MS and DLS) were obtained in northern and southern Lake Taihu, respectively, in October 2002 using a gravity corer (Fig. 1). The sediment exhibited no bioturbation and can be used to reconstruct environmental evolution. Sediments were sliced into sections at 0.5 cm intervals. Samples were freeze dried using a LABCONCO freeze drier. Physical and chemical data from the upper 13 and 7 cm sediments for Cores MS and DLS, respectively, were used.
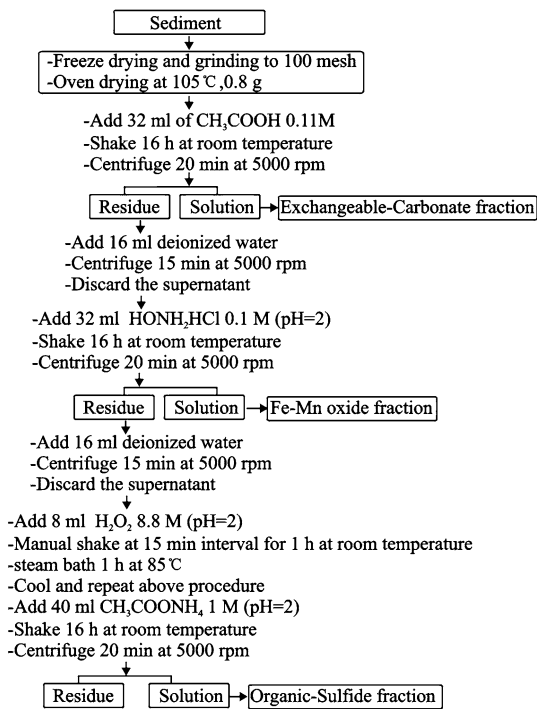
Analysis methods

TOC concentration in samples was determined using a CNHO-S elemental analyzer (Leeman Company). Particle size was measured with a Mastersizer–2000 laser diffraction particle size analyzer (Malvern Instruments Ltd.). Analysis errors for experiments were within ±5%.

For heavy metal analysis, the $HCl–HNO_3–HF$ digestion method was applied. About 125 mg of sediment, ground in an agate mortar to pass 100-μm sieve, was digested with 6.0 ml HNO_3, 0.5 ml HCl, and 3.0 ml HF at 180 ± 5°C for 15 min in a microwave oven (Berghof MWS–3 Digester). Residue was dissolved with 0.5 ml $HClO_4$ by braising in a heating block at <200°C and diluted to 25 ml with double-distilled deionized water. The solution was analyzed for heavy metals and major elements using inductively coupled plasma-atomic emission spectrometry (ICP-AES).

Chemical fractionations of Cu, Fe, Mn, Ni, Pb and Zn were determined using the BCR sequential extraction procedure (Quevauviller et al., 1997; Tokalioğlu et al., 2000) (Fig. 2). Only the three extractable fractions (exchangeable-carbonate, Fe–Mn oxide, and organic-sulfide fraction) were analyzed. Heavy metals concentrations in the extracted solution were analyzed by ICP-AES.

Sediments were dated using the ^{137}Cs methods described by Rosales-Hoza et al. (2003).

Fig. 2 Heavy metals sequence extraction procedure of BCR method

Accuracy of heavy metals analysis method

Four parallel analyses of standard sediment reference material (GSD–9 from Institute of Geophysical and Geochemical Exploration, Chinese Academy of Geological Sciences) were conducted to verify the accuracy of the heavy metal digestion method. The maximum error of all results was <3% of the standard value, showing that the HCl–HNO$_3$–HF digestion method and ICP-AES analysis have reasonable precision and reproducibility.

Three lacustrine samples from Lake Taihu were measured in parallel analyses to verify the accuracy of the BCR procedure due to the lack of suitable standard reference material. Blanks were also used for background correction. Results are shown in Table 1. Maximum errors of the results are within 10%, except 23.0 and 18.6% for the exchangeable-carbonate and organic-sulfide fractions of Pb in samples MS–5 and TH-2, respectively, and 24.4% for the Fe–Mn oxide fraction of Zn in sample MS-5. Errors for Pb and Zn may be related to contamination of certain procedure during the sequential extraction process, such as losing of the liquid during shaking etc (Tokalioğlu et al., 2000).

Table 1 Error analysis of BCR sequential extraction method for sedimentary samples of Lake Taihu

Elements		MS-5				TH-1				TH-2			
		AC	S.D.	CV	ME	AC	S.D.	CV	ME	AC	S.D.	CV	ME
Cu	F1	3.05	0.07	2.19	2.52	2.01	0.05	2.35	2.71	3.54	0.03	0.75	0.85
	F2	3.62	0.01	0.16	0.18	2.97	0.05	1.52	1.57	3.21	0.14	4.21	4.36
	F3	5.28	0.16	3.08	3.35	5.05	0.12	2.28	2.31	10.0	0.30	3.00	3.35
Fe	F1	250	10.4	4.17	4.79	595	14.2	2.38	2.45	1590	17.4	1.10	1.17
	F2	5070	41.2	0.81	0.90	5570	66.7	1.20	1.23	8390	246	2.93	3.38
	F3	989	53.4	5.40	5.81	1310	46.7	3.55	3.77	2190	175	7.99	8.52
Mn	F1	302	4.48	1.48	1.71	192	2.78	1.45	1.52	823	4.34	0.53	0.59
	F2	92.1	3.10	3.37	3.83	49.3	0.50	1.01	1.11	208	4.01	1.93	1.97
	F3	36.2	1.35	3.73	4.09	33.1	1.00	3.03	3.37	64.8	4.74	7.31	7.32
Ni	F1	3.39	0.06	1.62	1.67	2.10	0.07	3.45	4.00	2.96	0.01	0.39	0.45
	F2	4.00	0.06	1.38	1.58	2.86	0.06	1.92	2.21	3.49	0.19	5.48	6.11
	F3	4.59	0.11	2.30	2.40	3.46	0.04	1.17	1.35	4.12	0.06	1.38	1.54
Pb	F1	1.59	0.34	21.3	23.0	0.82	0.07	8.15	9.89	0.42	0.00	0.81	0.55
	F2	8.97	0.51	5.72	6.39	3.74	0.08	2.09	2.41	2.96	0.09	3.04	3.04
	F3	14.19	0.51	3.62	4.13	9.08	0.24	2.59	2.61	11.9	1.95	16.41	18.6
Zn	F1	12.3	0.56	4.56	5.19	3.27	0.07	2.04	2.21	4.12	0.15	3.52	3.56
	F2	16.8	3.59	21.4	24.4	4.48	0.12	2.60	2.83	5.49	0.20	3.70	4.25
	F3	9.10	0.48	5.30	5.86	3.68	0.33	8.89	9.96	6.40	0.21	3.21	3.28

AC, average content (mg kg^{-1}); S.D, standard deviation; CV, coefficient of variation; ME, maximum error

F1, exchangeable-carbonate fraction; F2, Fe–Mn oxide fraction; F3, organic-sulfide fraction

Due to good precision and reproducibility for the digestion and BCR methods, the residue fraction concentration of heavy metals can be represented by the difference of total concentration and extractable fractions of each heavy metal.

Results

Dating results

The chronological framework of Cores MS and DLS were determined by ^{137}Cs dating. Average sedimentation rates were 1.7 mm year^{-1} and 0.9 mm year^{-1}, respectively, for the two cores (Liu et al., 2005). These results are similar to those from other researchers (Sun & Huang, 1993). The ages for Core MS and DLS are shown in Fig. 3.

Heavy metals, grain size and TOC in Cores MS and DLS

Grain size and TOC in the cores are shown in Fig. 3. Sediments were dominated by silt (4–64 μm) and clay (<4 μm). The grain size becomes finer gradually from 14 to 4 cm depth and coarser from 4 cm to the surface in Core MS; and it becomes finer gradually from 7 to 2.5 cm and coarser from 2.5 cm to the surface in Core DLS.

TOC content was <2% for most of the surface sediments (Sun & Huang, 1993). TOC was around 0.62–1.35% in Cores MS and DLS and increases near the surface (Fig. 3).

Concentrations of the major elements (Al, Mg, K and etc) increased from 14 to 4 cm and decreased from 4 cm to the surface in Core MS, having consistent variation with the finer fraction (clay and fine silt). For Core DLS, major elements increased from 7 to 2.5 cm and decreased from 2.5 cm to the surface, also having consistent variation with the finer fraction.

Heavy metals, such as Mn, Ni, Pb and Zn, also had similar variation patterns as major elements in the 14–4 cm interval for Core MS, and 7–2.5 cm for Core DLS, except for higher Mn and Fe in the middle part of the core. In contrast to major elements and the finer fraction, heavy metals generally increased in the top 4–0 cm and 2.5–0 cm for the two sediment cores.

Chemical forms of heavy metals in Cores MS and DLS

Chemical fractionation compositions of Cu, Fe, Mn, Ni, Pb and Zn determined by BCR are illustrated in Fig. 4. Cu, Fe, Ni and Zn were
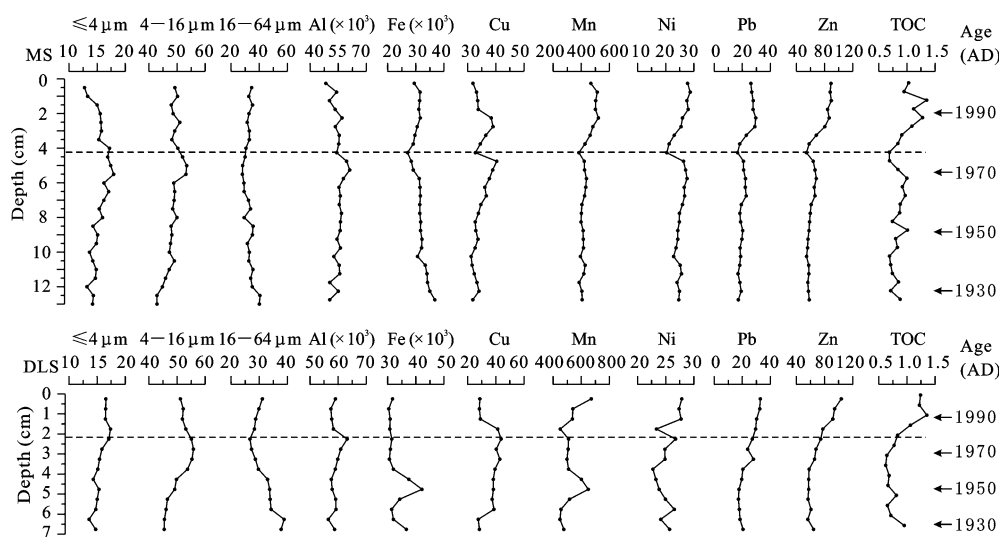

Fig. 3 The variation of grain size (%), heavy metals (mg kg^{-1}) and TOC (%) in Cores MS and DLS
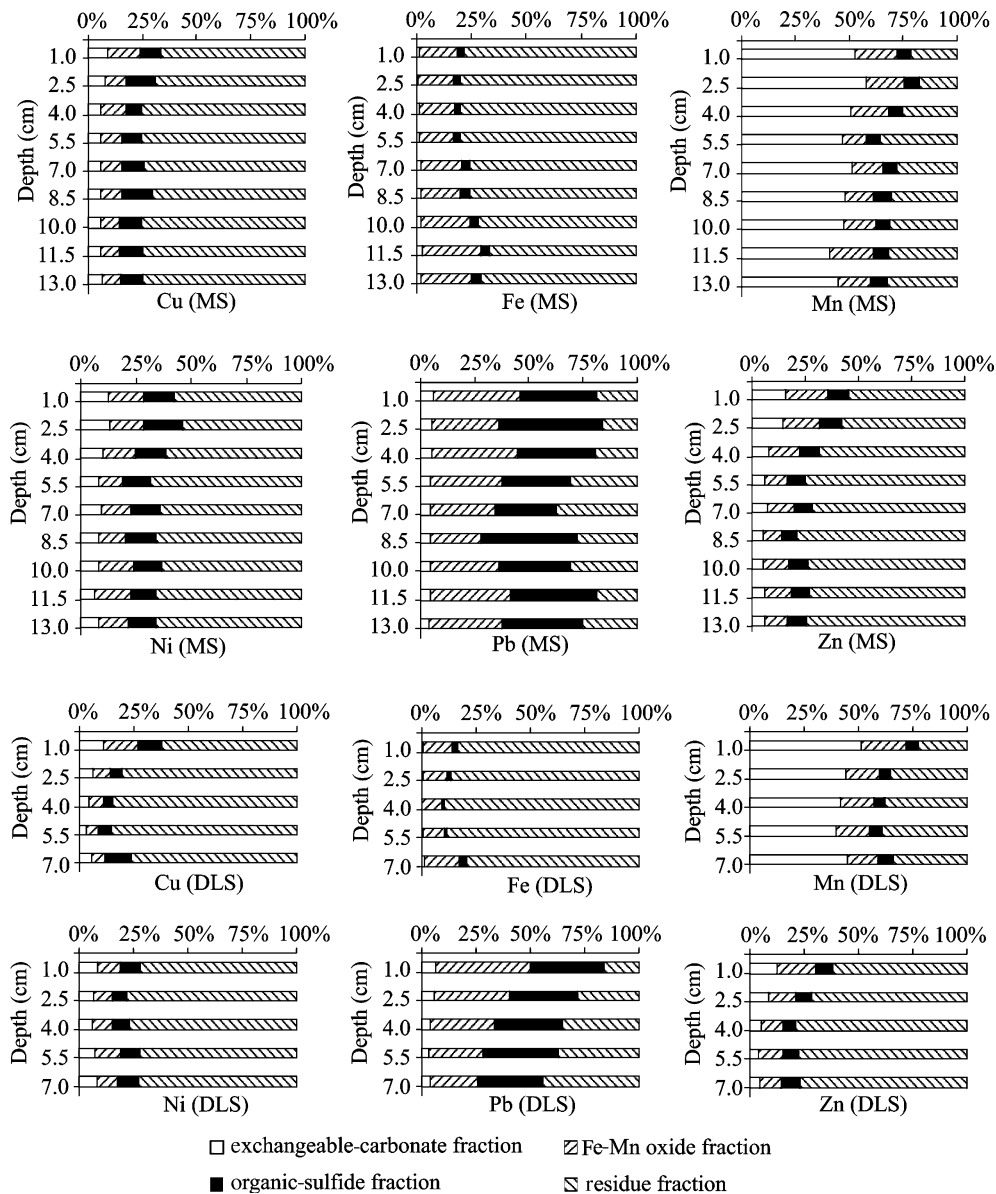
Fig. 4 The chemical fractionation percentages of Cu, Fe, Mn, Ni, Pb and Zn in the sediments of Cores MS and DLS (with 1.5 cm interval)

concentrated in the residue fraction, and average percentages were 72.9%, 75.6%, 62.9% and 69.7% in Core MS, and 77.8%, 85.4%, 72.9% and 73.3% in Core DLS, respectively. Mn was present in the exchangeable-carbonate fraction, and its percentages were 48.9 and 44.3% in Cores MS and DLS, respectively, whereas Pb was present in both the Fe–Mn oxide fraction and organic-sulfide fraction.

Percentages of extractable fractions of Cu, Mn, Ni, Pb and Zn, including exchangeable-carbonate, Fe–Mn oxide and organic-sulfide fractions, had a consistent variation in the vertical sequence for both cores, which were stable at 14–4 and 7–2.5 cm, but increased from 4 to 0 cm and from 2.5 to 0 cm for Cores MS and DLS, respectively (Fig. 4). The percentages of extractable fractions of Cu, Mn, Ni, Pb and Zn in the top 4 and 2.5 cm
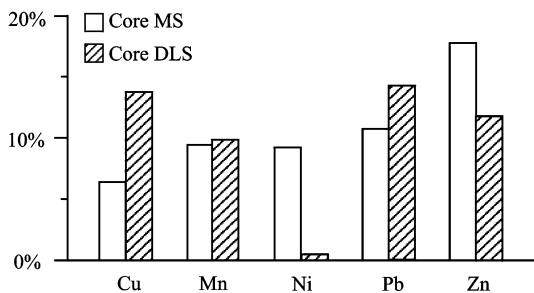
Fig. 5 The increased percentages of Cu, Mn, Ni, Pb and Zn in extractable fractions in the top 4 and 2.5 cm of Cores MS and DLS compared with that in the deeper layers

of Cores MS and DLS increased 0.5–17.8% comparing with that in the deeper layers (Fig. 5). The average percentage of Pb, Ni, Cu, Zn and Mn in the organic-sulfide fraction were 35.8%, 13.5%, 10.3%, 9.3% and 6.8% in Core MS, and 32.3%, 8.7%, 7.9%, 7.6% and 5.9% in Core DLS.

Discussion

Pollution features of the surface sediment

There are two main sources for heavy metals in sediments: natural sources and anthropogenic sources. Heavy metals content commonly has good correlation with major elements and finer fractions in unpolluted sediments, but human pollution can alter this relationship (Abraham, 1998; Covelli & Fontolan, 1997). Therefore, variation patterns of major elements, heavy metals and their correlation with grain size composition can be used to evaluate heavy metals pollution (Rubio et al., 2000). Al, Fe, Mg and K mainly come from natural versus anthropogenic sources and can be used as reference elements for heavy metal pollution evaluation (Covelli & Fontolan, 1997; Soto-Jiménez & Páez-Osuna, 2001).

Heavy metals in surface sediments of Core MS and DLS and the other 12 sites from Qu et al. (2001) were analyzed for spatial distribution of heavy metals in Lake Taihu. Contents of Fe, Mg and other major elements in the sediments of the
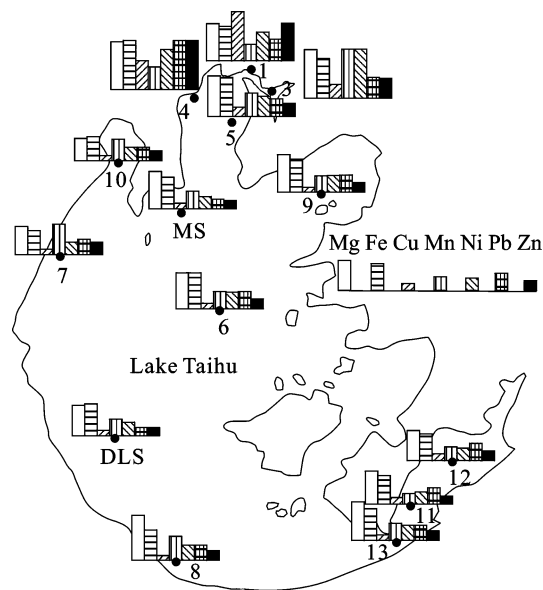
Fig. 6 The relative concentration of heavy metals in each site compared with the average of the 14 sites of Lake Taihu surface sediments (the data of site 1–13 from Qu et al., 2001)

14 sites are similar, but Cu, Mn, Ni, Pb and Zn in the sediments of sites 1–5 have higher concentrations versus Fe and Mg (Al and K not determined at sites 1–13) than other sites (Fig. 6). Concentrations of the three extractable fractions of Cu, Ni and Zn in surface sediments also were higher in the northern lake compared with other areas (Wang et al., 2002). These indicate that sediments in northern Lake Taihu, such as Meiliang Bay and Wuli Bay, are polluted with heavy metals. Pollution in the southern lake is much lighter (Qu et al., 2001; Wang et al., 2002). Larger amounts of waste water with higher concentrations of heavy metals from industries in Changzhou and Wuxi discharged into Wuli and Meiliang Bays via the Zhihugang and Liangxihe is the source of heavy metal pollution in the northern Lake Taihu (Huang et al., 2001; Qu et al., 2001).

Geochemical features of the core sediment

For total heavy metal concentrations, Cu, Mn, Ni, Pb and Zn have similar variation with major elements (Al, Mg and K) and finer fractions before the late 1970s. However, there is a sharp

contrast between Cu, Mn, Ni, Pb and Zn with major elements and finer fractions after the late 1970s for Cores MS and DLS. This indicates that heavy metals in the upper sediments may come primarily from anthropogenic sources (Covelli & Fontolan, 1997; Abraham, 1998). High content of Fe and Mn in the lower part of Core DLS may be related to redox conditions since little industrial waste water was discharged into southern Lake Taihu during the 1950s.

Chemical fractionation compositions of Cu, Fe, Mn, Ni, Pb and Zn were similar for both cores, and similar to those in surface sediments (Wang et al., 2002), but different from other sediments (Tokalioğlu et al., 2000; Tüzen, 2003). Percentages of exchangeable-carbonate fraction of Cu, Fe, Ni, Pb and Zn were <10% and are influenced by physico-chemical properties of the sediments. The exchangeable fraction of heavy metals in sediments generally is low and has little contribution to the percentage of the exchangeable-carbonate fraction (Alvarez et al., 2001). The percentage of the exchangeable-carbonate fraction mainly is influenced by adsorption quantity of the carbonate fraction since the carbonates in sediments contain significant heavy metals (Tokalioğlu et al., 2000). However, the carbonate content of Lake Taihu is low (Yuan et al., 2002b), which may explain the relatively low percentage of the exchangeable-carbonate fraction of Cu, Fe, Ni, Pb and Zn in the sediments. But, the percentage of Mn in the exchangeable-carbonate form was up to 40.9–57.9% (Wang et al., 2002). This may be related to the chemical features of Mn (Liu et al., 1984; Tokalioğlu et al., 2000). Mn can

easily be adsorbed on the surface of fine granules than other ions; the oxidation of Mn^{2+} is more relaxed than Fe^{2+} and can be precipitated until higher pH (8.5–10), which is 1ower than 8.1 in Lake Taihu sediments (Sun & Huang, 1993). So Mn can be easily associated with carbonates (Liu et al., 1984). Another feature is that the radius of Mn^{2+} (0.91) is similar to that of Ca^{2+} (1.08) and Mg^{2+} (0.8), so the replacement of Ca^{2+} and Mg^{2+} by Mn^{2+} is easier. A1l these may be the reasons for higher Mn content in the exchangeable-carbonate forms in the sediments.

The Fe–Mn oxide fraction of heavy metals, particularly those bound by Fe–Mn oxide or precipitated as hydroxide, is an electrovalent bond form and can be re-released into the water under reductive conditions. The average percentage of Cu, Fe, Mn, Ni and Zn in this fraction is 10–19.4%, but Pb is as high as 34.2%. The carbonate form of Pb is loosely bound and may change with environmental conditions (Li et al., 2001), but it can form compounds with Fe–Mn oxide easily. The higher content of Pb in the Fe–Mn oxide fraction in Lake Taihu sediment versus others (Tokalioğlu et al., 2000; Tüzen, 2003) may be related to the better oxidative environment in Lake Taihu sediments. The relationship between Cu, Ni, Pb, Zn of the Fe–Mn oxide fraction and content of Fe and Mn in the Fe–Mn oxide fraction are presented in Table 2. Ni, Pb and Zn display significant correlation with Mn for Core MS, and Cu, Pb and Zn also show good correlation with Mn for Core DLS. No significant relationship between Ni, Pb, Zn and Fe were found. This is consistent with (Oakley et al., 1981), where hydrous Mn oxides

Table 2 Correlation coefficients of the heavy metal chemical forms and with TOC

Correlation coefficients	Core MS					Core DLS				
	Cu	Mn	Ni	Pb	Zn	Cu	Mn	Ni	Pb	Zn
r_1	–0.77[*]	0.04	0.24	–0.52	–0.41	–0.25	–0.12	0.33	–0.07	0.08
r_2	0.42	1.00	0.89[**]	0.77[*]	0.85[**]	0.87	1.00	–0.03	0.89	0.95[*]
r_3	0.74	0.81[**]	0.89[**]	0.90[**]	0.81[**]	0.80	0.68	0.76	0.77	0.94[*]

[**] Correlation is significant at 0.01 level; [*] Correlation is significant at 0.05 level

r_1, the correlation coefficients of Fe–Mn oxide fraction concentrations of Cu, Mn, Ni, Pb, Zn and that of Fe

r_2, the correlation coefficients of Fe–Mn oxide fraction concentrations of Cu, Mn, Ni, Pb, Zn and that of Mn

r_3, the correlation coefficients of organic-sulfide fraction concentrations of Cu, Mn, Ni, Pb, Zn and TOC

exhibited more extensive isomorphic substitution than amorphous Fe oxides and showed greater conditional equilibrium constants for heavy metals than Fe oxides.

Heavy metals bound with sulfide can be re-released into the water under oxidizing conditions. Sulfide in Lake Taihu sediments is low due to seasonal variation of redox conditions and the generally weak oxidation or reduction conditions in surface sediment (Sun & Huang, 1993). The correlation between TOC and the organic-sulfide fractions of Cu, Mn, Ni, Pb and Zn was high for both cores (Table 2). This indicates that contents of the organic-sulfide fraction of Cu, Mn, Ni, Pb and Zn are influenced mainly by organic content in Lake Taihu. In aquatic systems, organic matter exhibits high selectivity for divalent ions. The average percentages of Pb, Ni, Cu, Zn and Mn in the organic-sulfide fraction are consistent with the order of binding strength for metal ions onto organic matter (Cu > Pb > Ni > Zn > Mn) (Föstner & Wittmann, 1988). Copper mainly exists in sulfide form in nature (Li et al., 2001; Hlavay & Polyák, 2002), and the low percentage in the organic-sulfide fraction in the cores may be due to low sulfide content.

Seasonal variation of redox conditions between weak oxidation or reduction and mineralization of organic matter in Lake Taihu surface sediment can influence the chemical forms of heavy metals in the exchangeable-carbonate, Fe–Mn oxide and organic-sulfide fractions (Saeki et al., 1993; Tack et al., 1996). Higher content of the extractable fractions of Cu, Mn, Ni, Pb and Zn in the upper part of the sediment may have potential toxicity. The ratio of SEM (amount of simultaneously extracted metals when AVS is extracted) to AVS (acid volatile sulfides, the concentration of sulfide extracted by 1 M cold hydrochloric acid) can be used to evaluate heavy metals toxicity in sediments (Edward, 1998; Li et al., 2004). SEM/AVS in northern Lake Taihu sediment ranges from 1.3 to 5.9, so heavy metals have potential toxicity (Edward, 1998; Li et al., 2004). This also indicates that, although the organic-sulfide fraction of the heavy metals mainly is influenced by organic content, sulfide also has some influence on heavy metal activity

due to seasonal variation of redox conditions at the water-sediment interface.

Pollution history of the sediment

Chemical fractionation of heavy metals in the cores also can be used to evaluate pollution in the lake. Extractable forms of heavy metals from anthropogenic sources have a higher proportion of heavy metal content than natural sources (Föstner & Wittmann, 1988; Li et al., 2001). Lower concentrations and percentages of extractable forms of Cu, Mn, Ni, Pb and Zn in sediments before the late 1970s suggests that heavy metals mainly came from natural sources, which is consistent with underdeveloped industry in the catchment; higher ones in recent years suggest that heavy metals were anthropogenic partially.

Wuxi is a typical city representative of economic development in the catchment. The gross domestic product of Wuxi increased annually by 30% since the 1980s, accompanied by a large amount of waste water discharge. The main industries in the region are chemical, paper, textile, leather and electroplating, which account for 93.8% of the wastewater discharge (Huang et al., 2001). Wastewater from the industries may be the main source of Cu, Mn, Ni, Pb and Zn in the sediments. Pollution history in the two cores is coincident with distinct heavy metals pollution in Meiliang Bay (Rose et al., 2004). Pollution history of heavy metals recorded in the sediments of Lake Taihu also is consistent with rapid economic development characteristics of the Taihu lake catchment.

Conclusion

Cu, Mn, Ni, Pb and Zn have higher content in surface sediments of northern Lake Taihu and were anthropogenic more than other areas of the lake. The main chemical form for Cu, Fe, Ni and Zn in the sediments was the residual fraction. Mn is mainly present in the exchangeable-carbonate and residue fractions. The organic-sulfide and Fe–Mn oxide fractions were important for Pb. Organic matter and Mn in the Fe–Mn oxide

fraction play an important role in binding heavy metals in the sediments. High concentrations of Cu, Mn, Ni, Pb and Zn, and their extractable fractions in recent sediments indicate that heavy metals were anthropogenic and have potential toxicity. Heavy metals pollution history was recent for both cores, which coincides with rapid economic development in the Taihu catchment.

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