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Characterization of heavy metal concentrations in the sediments of three freshwater rivers in Huludao City, Northeast China

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Sediment in Wuli River, Cishan River, and Lianshan River has been contaminated by heavy metals and adverse effects would be expected frequently in Wuli River and Cishan River.

Abstract

Wuli River, Cishan River, and Lianshan River are three freshwater rivers flowing through Huludao City, in a region of northeast China strongly affected by industrialization. Contamination assessment has never been conducted in a comprehensive way. For the first time, the contamination of three rivers impacted by different sources in the same city was compared. This work investigated the distribution and sources of Hg, Pb, Cd, Zn and Cu in the surface sediments of Wuli River, Cishan River, and Lianshan River, and assessed heavy metal toxicity risk with the application of two different sets of Sediment Quality Guideline (SQG) indices (effect range low/effect range median values, ERL/ERM; and threshold effect level/probable effect level, TEL/PEL). Furthermore, this study used a toxic unit approach to compare and gauge the individual and combined metal contamination for Hg, Pb, Cd, Zn and Cu. Results showed that Hg contamination in the sediments of Wuli River originated from previous sediment contamination of the chlor-alkali producing industry, and Pb, Cd, Zn and Cu contamination was mainly derived from atmospheric deposition and unknown small pollution sources. Heavy metal contamination to Cishan River sediments was mainly derived from Huludao Zinc Plant, while atmospheric deposition, sewage wastewater and unknown small pollution were the primary sources for Lianshan River. The potential acute toxicity in sediment of Wuli River may be primarily due to Hg contamination. Hg is the major toxicity contributor, accounting for 53.3–93.2%, 7.9–54.9% to total toxicity in Wuli River and Lianshan River, respectively, followed by Cd. In Cishan River, Cd is the major sediment toxicity contributor, however, accounting for 63.2–66.9% of total toxicity.

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Keywords: Sediment; Sediment quality guidelines; Huludao City; Freshwater

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1. Introduction

Sediments represent the largest pool of metals in aquatic environments (Daskalakis and O'Connor, 1995). More than 90% of the heavy metal load in aquatic systems is bound to suspended particulate matter and sediments (Calmano et al., 1993). Sediments polluted with various kinds of hazardous and toxic substances have been found, including trace elements which accumulate in sediments via several pathways: disposal of liquid effluents, terrestrial runoff, and leachate carrying chemicals originating from numerous urban, industrial, and agricultural activities, as well as atmospheric deposition (Rivail Da Silva et al., 1996; Karageorgis et al., 2002; Mucha et al., 2003). The distribution of heavy metals in sediments adjacent to populated areas can provide researchers with evidence of the anthropogenic impact on ecosystems and aid in assessing the risks associated with discharged human waste. The build-up of metals in sediments has significant

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environmental implications for local communities, as well as for river water quality (Demirak et al., 2006).

Sediment quality guidelines (SQGs) are very useful to screen sediment contamination by comparing sediment contaminant concentration with the corresponding quality guideline (Caeiro et al., 2005). These guidelines evaluate the degree to which the sediment-associated chemical status might adversely affect aquatic organisms and are designed to assist the interpretation of sediment quality (Wenning and Ingersoll, 2002). SQGs, including sediment quality criteria, sediment quality objectives and sediment quality standards, have been developed by various federal and provincial agencies in North America for both freshwater and marine ecosystems (Pekey et al., 2004; Caeiro et al., 2005). Such SQGs have been used in numerous applications, including designing monitoring programs, interpreting historical data, evaluating the need for detailed sediment quality assessments, assessing the quality of prospective dredged materials, conducting remedial investigations and ecological risk assessments, and developing sediment quality remediation objectives (Long et al., 1995; Smith et al., 1996; Long and MacDonald, 1998).

Huludao City is an industrial base in Liaoning Province. Wuli River, Cishan River, and Lianshan River are three freshwater rivers which flow through Huludao City, and reach the sea at Jinzhou Gulf. The ecosystems of the three rivers are impacted significantly by historic and current anthropogenic loadings of a variety of pollutants (Na Zheng et al., 2007), including heavy metals. Mercury (Hg) is a contaminant of primary concern in the three rivers (Na Zheng et al., 2007). This is due in part to continued inputs of metals from point sources and atmospheric deposition to the watershed, as well as its ability to be transformed to monomethylmercury (MMHg) via natural processes (Gilmour and Henry, 1991; Benoit et al., 2003). In this study, in order to compare the characterization of heavy metal contamination in sediments in the three rivers, the distribution of Hg, Pb, Cd, Zn and Cu were investigated in the surface sediments and typical sediment profiles of Wuli River, Cishan River and Lianshan River. The ecological risk of sediment was assessed with the application of two different sets of SQG indices. The characteristics of combined contamination were also studied using the toxic unit approach.

2. Materials and methods

2.1. Study area

Huludao City (40°56′ N, 120°28′ E) is in Liaoning Province in northeast China, near Liaodong Gulf (Fig. 1). The production of petrochemicals and nonferrous metal smelting are the primary industries of Huludao City. Jinxi Chemical Factory and Huludao Zinc Plant (the largest zinc smelting plant in Asia) are the main industrial enterprises in Huludao City. Wuli River has a long history of chlor-alkali producing impacts from Jinxi Chemical Factory, while a large amount of waste from Huludao Zinc Plant was discharged into Cishan River, resulting in serious contamination in the two rivers (Zhao and Yan, 1997; Zheng et al., 2007). By contrast, there is no obvious pollution source found along Lianshang River. According to historical data, wastes from Jinxi Chemical Factory and Jinxi Petroleum Chemical Factory were discharged into Wuli River near site W2. However, the use of mercury cathodes in Jinxi Chemical Factor ceased after 1998, and wastewater discharged to Wuli River has been greatly reduced since then. Large amounts of waste from Huludao Zinc Plant were discharged into the Cishan River estuary. There was no large pollution source along Lianshan River, except waste from villagers who lived along it.

2.2. Sediment sampling

Grain size plays a significant role in determining elemental concentrations in sediments (Szefer et al., 1996). It is recommended that a particle size fraction of <63μm should be applied for analysis because this is most nearly equivalent to materials carried in suspension, the most important system for transport of sediments (Salomons and Förstner, 1984; Chen et al., 1994). In this study, the contamination of sediments with particle size fractions <63μm were investigated.

Surface sediment samples were collected from 10 sites in Wuli River, 6 sites in Cishan River and 4 sites in Lianshan River in May 2005 (Fig. 1). Typical sediment profiles from the W7 (Wuli River) and C4 (Cishan River) sites are shown in Fig. 2. These sites were selected because sediment samples at different depths could be easily collected and are not affected by estuary and pollution sources. Sediment samples at depth intervals of 0–5, 5–10, 10–15, 15–20, 20–25, 25–30 and 30–35 cm were collected at these sites. Samples were placed in dark-colored polyethylene bags, refrigerated and returned to the laboratory immediately. Samples were air dried at 4 °C, crushed, passed through 0.063-mm mesh sieve and stored at 4 °C in the dark before analysis of properties and concentrations of heavy metals.

2.3. Heavy metal analysis and quality control

Samples were digested using the method of $H_2SO_4-HNO_3-V_2O_5$ (GB/T 17136-1997) and $HClO_4-HNO_3-HF$ (GB/T 17138, 17141-1997), respectively. The cold atomic absorption technique was used to determine the concentration of total Hg in samples and blanks using F732-V Hg detector. Concentrations of Pb, Cd, Zn, Cu and Fe were analyzed by inductively coupled plasma atomic emission spectrometry, ICP/AES (ICPS-7500, Shimadzu, Japan). For those samples to which ICP/AES was insufficiently sensitive, total metals were determined by a graphite furnace AAS (GFAAS, GBC932AA, Australia). Analytical reagent blanks were prepared with each batch of digestion set and then analyzed for the same element of the samples. The accuracy and precision of the analytical method was estimated by analyzing a sediment Standard Reference Material (GBW 07304(GSD-4)). Accuracy of the analytical method was given as percent recoveries for each of the elements. Results are reported in Table 1.

2.4. Sediment quality guidelines

Two sets of SQGs developed for freshwater ecosystems (MacDonald et al., 2000) were applied in this study to assess the ecotoxicology of trace element concentrations in sediments: (a) the effect range low (ERL)/effect range median (ERM) and (b) the threshold effect level (TEL)/probable effect level (PEL) values. Low range values (i.e., ERLs or TELs) are concentrations below which adverse effects upon sediment dwelling fauna would be infrequently expected. In contrast, the ERMs and PELs represent chemical concentrations above which adverse effects are likely to occur (MacDonald et al., 2000). Furthermore, toxic units were used to normalize the toxicity of the various metals to allow comparison of their relative effects, defined as the ratio of the determined concentration to PEL value (Pedersen et al., 1998).

3. Results and discussion

3.1. Pattern of heavy metal contamination along rivers

3.1.1. Heavy metal distribution in surface sediment

The distributions of Hg, Pb, Cd, Zn and Cu in surface sediments of Wuli River, Cishan River, and Lianshan River are

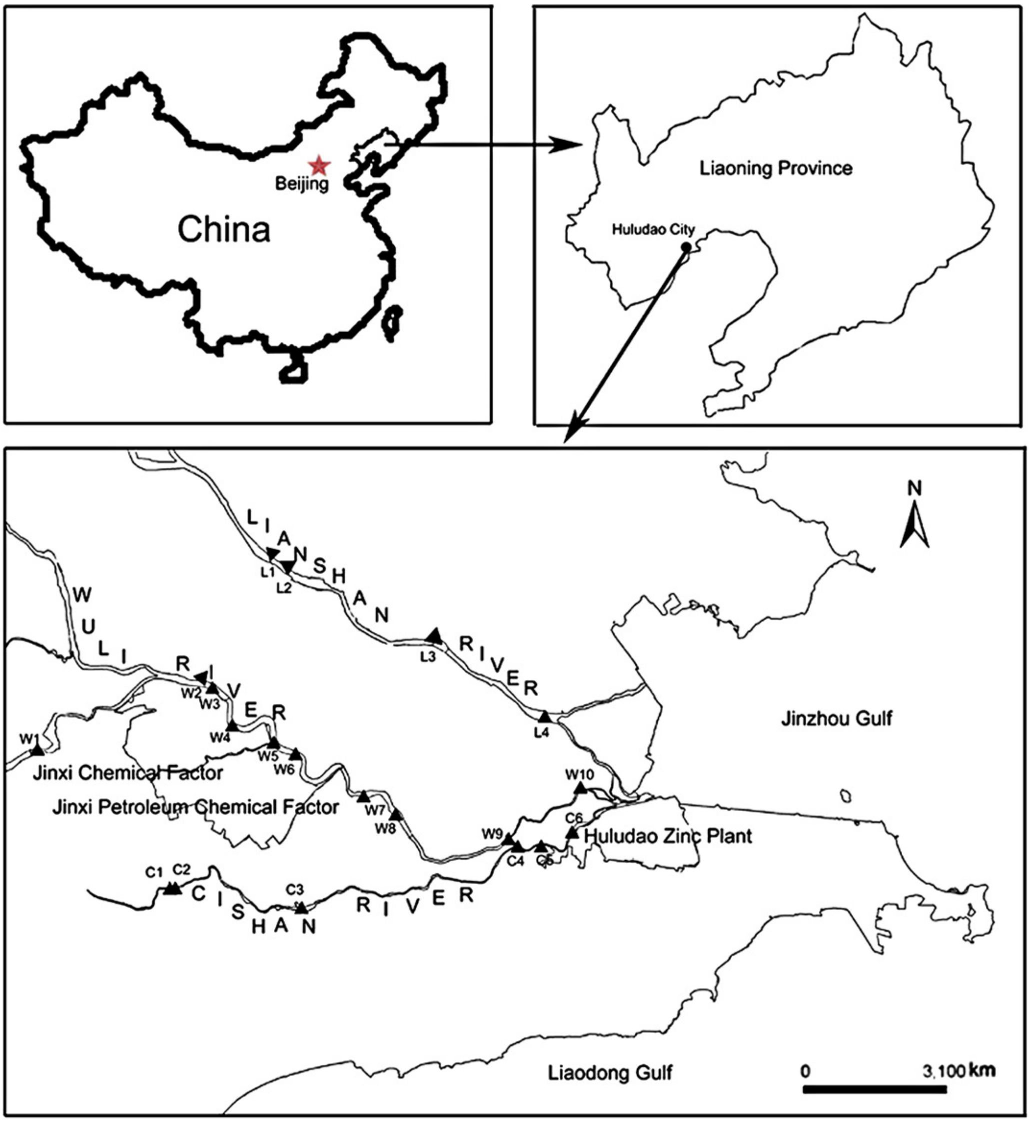

Fig. 1. Sampling sites in sediment of Wuli River, Lianshan River and Cishan River in Huludao City.

shown in Table 2. In Wuli River, Pb, Cd, Zn and Cu concentrations at sampling sites W1–W8 are clearly lower than at sites W9 or W10, and their concentrations in sediments of W7 and W8 sites are lower than at site W6. However, Hg concentration increases along Wuli River, and the maximum Hg concentration is at sampling site W7. The maximum concentrations of Hg, Pb, Cd, Zn and Cu in Wuli River are 15.37, 198.7, 30.85, 2077 and 107.4 mg kg^{-1}, exceeding background sediment values (Sun, 1992) in Liaoning Province of 438.1, 6.9, 262.7, 30.0 and 3.7 times, respectively.

In Cishan River, sediment heavy metal concentrations at sites C4, C5 and C6 are much higher than at sites C1, C2 and C3. The maximum concentrations of Hg, Pb, Cd, Zn and Cu are 132.5, 1551, 1463, 19789 and 1072 mg kg^{-1}, exceeding background values in Liaoning Province by 3784, 60.8, 12503, 293 and 45 times. In Lianshan River, maximum Hg, Pb, Cd, Zn and Cu concentrations at site L3 are 5.39, 164.9, 19.40, 797, 106.8 mg kg^{-1}, exceeding background sediment values in Liaoning Province by 153, 5.6, 164, 10 and 3.6 times.

3.1.2. Heavy metal pollution source

There is no direct pollution source to Lianshan River. Heavy metals in sediment of Lianshan River might originate from atmospheric deposition or wastewater from the residents.

Wuli River has a long history of contamination from the Jinxi Chemical Factory, and Wuli River sediments were contaminated by Hg derived from chlor-alkali production prior to 1998, when the use of mercury cathodes ceased. A correlation matrix shows that the Pb, Cd, Cu and Zn in Wuli River sediments are highly correlated with each other showing a strong positive association, but not with Hg (Table 3). This indicates that Pb, Cd, Zn and Cu may have originated from similar pollution sources, differing from Hg. At sites W1–W8, Pb, Cd, Zn and Cu average concentrations (64.7, 3.951, 348.7 and 51.34 mg kg^{-1}) are clearly lower than at sites W9

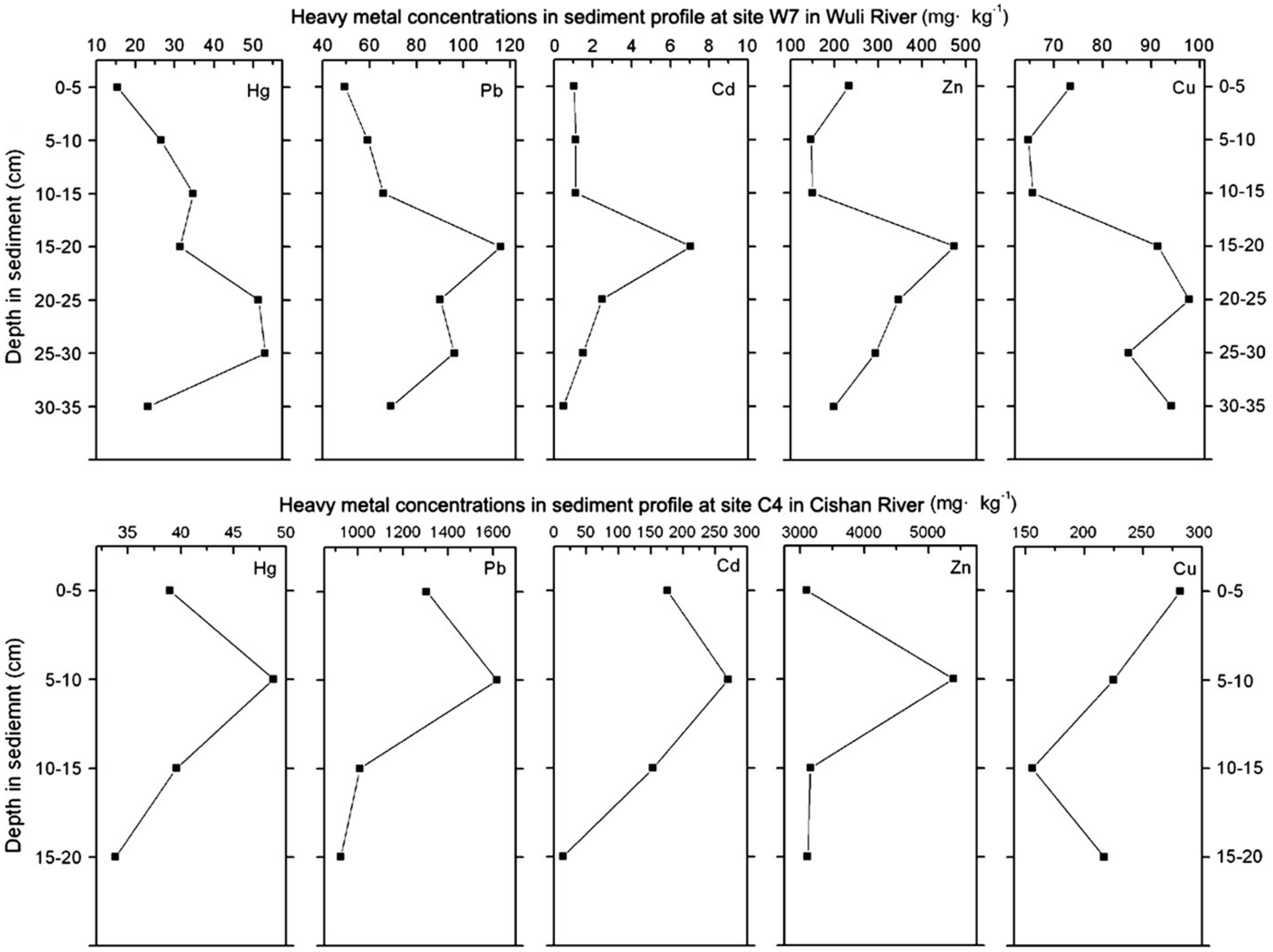

Fig. 2. Heavy metal concentrations in typical sediment profiles from Cishan and Wuli Rivers.

and W10 (177, 28.51, 1496, 99.6 mg kg^{-1}), and similar to Lianshan River and sites C1–C3 in Cishan River. So Pb, Cd, Zn and Cu contamination to sediment of Wuli River at sites W1–W8 may originate from atmospheric deposition or wastewater from the residents, similar to Lianshan River. Relatively higher heavy metal contamination at sites W9 and W10 might be influenced by Huludao Zinc Plant which is near sites W9 and W10, or by other unknown pollution sources. However, current Hg contamination at sites W1–W8 is occurring due to previously contaminated sediments.

Table 1
Observed and certified values of elemental concentrations in standard reference material (concentration units are in mg kg^{-1}, dry weight)

Element	Observations	Blank (μg L^{-1})	Observed ± SD	Certified ± SD	% Recovery
Hg	12	0.00016	0.047 ± 0.005	0.044 ± 0.008	106
Pb	12	0.021	9.68 ± 2.47	10 ± 5	96
Cd	12	0.004	0.20 ± 0.03	0.19 ± 0.02	105
Zn	12	0.053	96 ± 7.25	101 ± 10	95
Cu	12	0.004	36.8 ± 0.96	37 ± 2	99

In Cishan River, heavy metal concentrations in sediments at sites C1–C3 are similar to Lianshan River, and there is no significant difference for heavy metal concentrations between Lianshan River and C1–C3 sites in Cishan River (Table 2). So heavy metals in these sites may also originate from atmospheric deposition. However, sediment heavy metal concentrations at sites C4–C6 are much higher than Lianshan River. Huludao Zinc Plant is situated in the lower reaches of Cishan River (C4–C6). We conclude that these heavy metals at sites C4–C6 originated from Huludao Zinc Plant.

3.2. Heavy metal distribution in typical sediment profiles

Sediment profiles at site W7 in Wuli River and site C4 in Cishan River were selected to investigate the distribution of heavy metals. Hg, Pb, Cd, Zn and Cu concentrations at sites W7 and C4 are significantly higher than background concentrations of 0.035 ± 0.014, 25.10 ± 7.04, 0.117 ± 0.048, 67.12 ± 20.98, 22.99 ± 6.35 mg kg^{-1} recorded in Liaoning Province (Sun, 1992).

At site W7, the maximum Hg concentration is at a depth of 25–30 cm. Maximum concentrations of Pb, Cd, and Zn are at

Table 2
Heavy metal concentrations in surface sediments of Wuli River, Cishan River and Lianshan River (mg kg^{-1})

Sampling sites		Hg	Pb	Cd	Zn	Cu
Wuli River	W1	0.151	81.0	7.44	640	53.9
	W2	0.315	86.7	3.09	144	61.3
	W3	1.10	48.7	2.10	153	36.7
	W4	7.23	51.7	5.39	342.9	31.3
	W5	14.7	48.8	2.74	287	40
	W6	12.6	121	8.40	525	89.4
	W7	15.4	49.5	1.03	394	45.1
	W8	12.6	30.0	1.41	301	53.0
	W9	15.1	155	26.2	914	91.8
	W10	10.0	198	30.9	2077	107
Average W I	W1–W8	8.03	64.7	3.951	348.7	51.34
Average W II	W9,W10	12.535	177	28.51	1496.2	99.6
Average	Total	8.668	80.5	7.947	525.2	56.63
Cishan River	C1	1.16	45.7	1.68	132	40.0
	C2	0.344	72.1	32.9	881	59.1
	C3	0.838	32.7	0.374	106	36.9
	C4	44.0	1551	177	4857	339
	C5	84.6	396	1463	18180	1072
	C6	132	1465	297	19789	131
Average C I	C1–C3	0.7797	50.19	11.65	373.8	45.33
Average C II	C4–C5	87.01	1137	645.9	14275	514.2
Average	Total	33.07	454.1	250.3	5595	217
Lianshan River	L1	0.215	59.3	1.25	114	29.0
	L2	0.349	64.5	1.85	383	64.7
	L3	5.39	131	16.4	797	91.8
	L4	0.394	164	19.4	508	106
Average	Total	1.587	104.9	9.727	450.9	73.08
ERL		0.15	35	5	120	70
ERM		1.3	110	9	270	390
TEL		0.174	35	0.596	123	35.7
PEL		0.486	91.3	3.53	315	197

ERL, effect range low; ERM, effect range median; TEL, threshold effect level; PEL, probable effect level.

depths of 15–20 cm, and maximum Cu concentrations are at a depth of 20–25 cm (Fig. 2). Maximal Hg, Pb, and Cd concentrations are significantly elevated compared to the top sediment. This indicates that Hg, Pb, Cd contamination to sediment in Wuli River has been reduced due to reduced loadings. Cu and Zn contamination, however, is higher in recently deposited sediments. Pb, Cd, Zn distributions at site W7 sampling plot are similar, but different to Hg and Cu. This also suggests that Hg contamination to Wuli River differs from that of the other metals. At site C4, Hg, Pb, Cd and Zn distribution in sediment profiles is similar, but different to Cu, and the maximum Hg, Pb, Cd and Zn concentrations are at a depth of 5–10 cm. This suggests that heavy metal contamination is decreasing.

Table 3
Pearson correlation (PC) coefficient matrix between heavy metals in sediment of Wuli River at sites W1–W8

	Pb	Cd	Zn	Cu	Hg
Pb	1	0.786*	0.437	0.830*	−0.213
Cd		1	0.717*	0.547	−0.229
Zn			1	0.400	0.145
Cu				1	0.096
Hg					1

*Correlation is significant at the 0.05 level (2-tailed).

3.3. Toxicity assessment based ERL/ERM and TEL/PEL

Hazardous waste site evaluations often involve the collection of substantial quantities of sediment chemistry data, and these data are frequently used to support screening-level ecological risk assessments (USEPA, 2005). To evaluate such data, numerical sediment quality guidelines (SQGs) are often used, such as threshold effect levels (TELs) and probable effect levels (PELs), effect range low (ERL) and effect range median (ERM) (USEPA, 2005; Long et al., 1995; Long and MacDonald, 1998; MacDonald et al., 2000).

Low range values (i.e., ERLs or TELs) are concentrations below which adverse effects upon sediment dwelling fauna would be expected infrequently. In contrast, the ERMs and PELs represent chemical concentrations above which adverse effects are likely to occur (Long and MacDonald, 1998). The incidence of toxicity was determined among samples in which none of the substances equaled or exceeded the ERL concentrations, in which one or increasing numbers of substances exceeded ERL concentrations, but none exceeded any ERM, and in which one or increasing numbers of substances exceeded ERM concentrations (Pekey et al., 2004).

Based on ERL/ERM and TEL/PEL comparisons (Table 2), adverse effects would be expected frequently in most surface sediments of Wuli River. Sediments from site W2 are less toxic due to no metal exceeding ERM values, while other sites are most toxic because the ERM for at least one metal is exceeded, especially at sites W9 and W10 which are heavily toxic due to four metals exceeding ERM values.

Sediments from sites C1 and C3 in the Cishan River are less toxic, as no metal exceeds ERM values, while other sites are more toxic because the ERM for at least one metal is exceeded, especially at sites C4, C5 and C6, where four metals exceed ERM values.

3.4. Toxic unit analysis

Potential acute toxicity of contaminants in sediment samples can be estimated as the sum of the toxic units, defined as the ratio of the determined concentration to PEL value (Pedersen et al., 1998). Toxic units (TU) for heavy metals in surface sediments of Wuli River, Cishan River and Lianshan River are shown in Figs. 3–5. Heavy metal toxic units in Wuli River decrease in the order Hg > Cd > Zn > Pb > Cu. Hg toxic units in Wuli River sediments are much higher than other heavy metals at sites W3–W10, even exceeding the sum of Pb, Cd, Zn and Cu toxic units. Cd and Zn in site W1 have relatively high toxic units. In the surface sediments of Cishan River, heavy metal toxic units decrease in the order Hg > Cd > Zn > Pb > Cu. Toxic units at site C4 are significantly elevated compared to C1–C3. Hg and Cd toxic units at sampling sites C4–C6 are much higher than for other metals. Toxic unit values for each metal at sites C1 and C3 are similar to values for sites W1–W3 of Wuli River. Toxic units of individual heavy metals at sites C4–C6 are much higher than for Wuli River.

The sum of toxic units (∑TUs) and relative contributions of all the heavy metals in Wuli River are shown in Fig. 3. The ∑TUs increase from the source to lower reaches of a river. At sites W1 and W2, Hg contribution to ∑TUs is from 5% to 20%. At other sites, Hg is the major contributor to toxicity

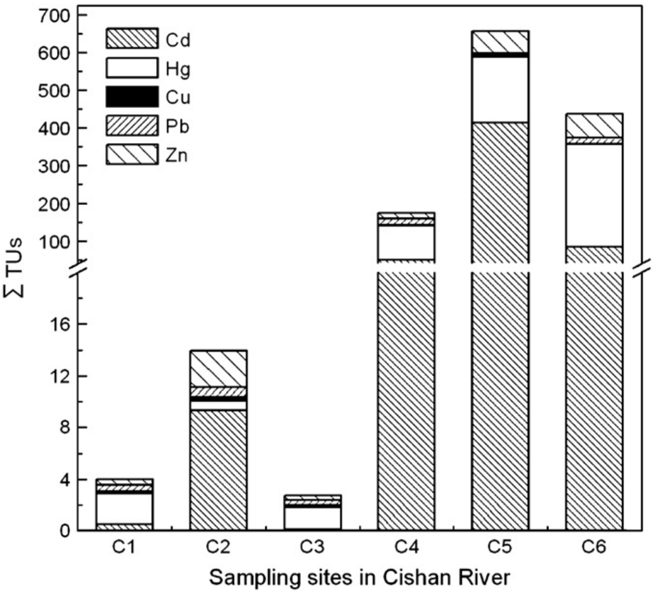

Fig. 4. Estimated sum of the toxic units (∑TUs) in Cishan River surface sediments.

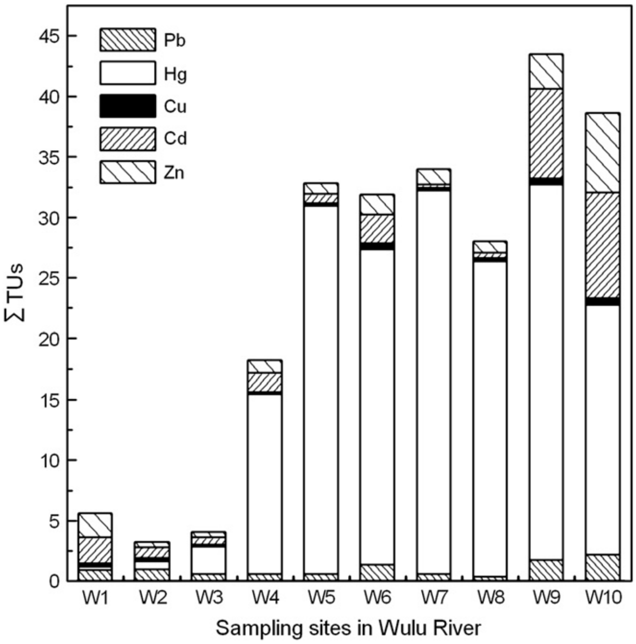

Fig. 3. Estimated sum of the toxic units (∑TUs) in Wuli River surface sediments.

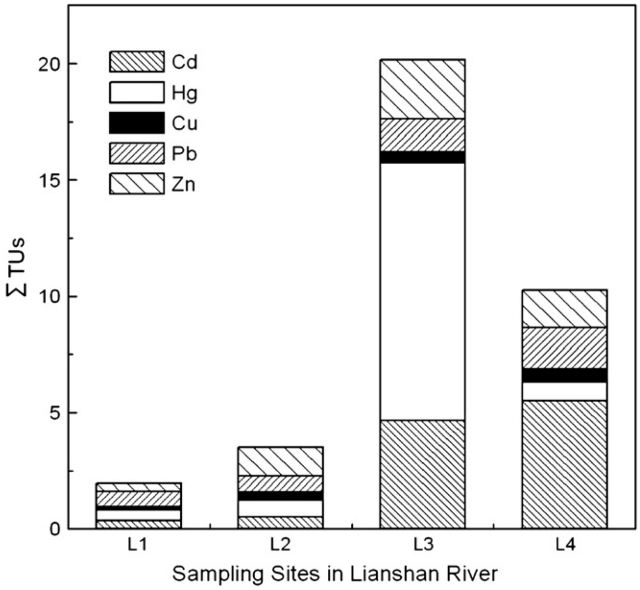

Fig. 5. Estimated sum of the toxic units (∑TUs) in Lianshan River surface sediments.

(53.3–93.2%). The contributions of Cd, Zn, Pb and Cu to $\sum$TUs are 0.8–37.6, 2.8–36.2, 1.2–29.3 and 0.6–9.6%, respectively.

The $\sum$TUs and relative contributions of all the heavy metals in Cishan River are shown in Fig. 4. At sites C2 and C5, the contributions of Hg and Cd toxic units to the sum of TUs are 5.1–26.5% and 63.2–66.9%, respectively, while at other sites on Cishan River the contributions are 51.7–63.5% and 3.9–28.8%, respectively. The contributions of Pb, Zn and Cu toxic units to $\sum$TUs of Cishan River are 0.7–13.2%, 8.8–20.1% and 0.2–6.9%, respectively. It is suggested that the potential exists for acute toxicity in Cishan River sediments due to heavy metal combined contamination, especially Hg and Cd contamination.

The sum of toxic unit ($\sum$TUs) and relative contributions of all the heavy metals in Lianshan River are shown in Fig. 5. Hg, Pb, Cd, Zn and Cu contributions to $\sum$TUs are 7.9–55%, 7.1–33.2%, 15.0–53%, 12.5–34.8% and 2.3–9.4%, respectively.

Heavy metal contamination decreases in the order Cishan River > Wuli River > Lianshan River. Heavy metal contamination to Lianshan River, upstream of Wuli River and Cishan River exists but is relative low, with acute toxicity occurring occasionally. At the downstream end of Cishan River, and downstream of site W4 on Wuli River, heavy metal contamination is higher, with acute toxicity occurring frequently. Wuli River, Cishan River and Lianshan River flow into the sea at Jinzhou Gulf. Estuary sediments were also found to be heavily contaminated. Fan et al. (2006) reported that high acute toxicity would occur in sediments at sites where Wuli River, Cishan River and Lianshan River flow into the sea. The highest acute mortality, 100%, occurred in the amphipod species *Ampetisca abdita* at the mouth of Wuli River and Cishan River by conducting 10-day flow-through sediment acute toxicity tests (Yan et al., 1999).

4. Conclusions

Serious combined heavy metal contamination occurred in the sediments of Wuli River, Cishan River and Lianshan River. Sediment Hg contamination in Wuli River originates from previous sediment contamination related to chlor-alkali production. Heavy metal contamination to Cishan River sediments originates from Huludao Zinc Plant. Heavy metal contamination to the sediment in Lianshan River originates from atmospheric deposition and wastewater. The potential acute toxicity in sediment of Wuli River is observed to be mainly due to Hg contamination. Hg is the major toxicity contributor accounting for 53.3–93.2% of the total toxicity in Wuli River and for 7.9–55% in Lianshan River, followed by Cd. Cd is the major toxicity contributor in Cishan River, however, accounting for 63.2–66.9%.

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