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Speciation and assessment of heavy metals in surface sediments of Jinjiang River tidal reach, southeast of China

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Abstract The concentration and speciation of heavy metals (Cr, Ni, Cu, Zn, Cd, Pb) in surface sediments ($\phi \leq 63$ μm) of Jinjiang River tidal reach are determined to evaluate the metal behavior. A modified BCR three-step sequential extraction procedure is carried out, and the residual fraction is undertaken by microwave-assisted acid digestion. The index of geo-accumulation indicates that Cd appeared highest among all these heavy metals in surface sediments, Cr, Cu, Zn lower, and Ni, Pb the least. The percentage of Zn, Cd is comparatively higher in the acid soluble fraction, Pb and Cu higher in the reductive fraction, indicating larger potential danger to the environment. So it is essential for developing the future remediation plans and pollution control strategies.

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Introduction

Jinjiang River is the third longest river of Fujian Province with the total drainage area of 5,629 km^2, the length of 182 km, and the annual rainfall of 54.25×10^8 m^3. This river flows through many county-cities, such as Yongchun, Anxi, Nan'an, Jinjiang, Quanzhou, and finally infloods into Quanzhou Bay. The main stream of Jinjiang River is about 29 km length, and the tidal reach is about 20 km from the estuary to Nan'an Jinji sluice. According to the characteristics of hydraulic and sediment transportation of tidal reach, the water flow is very complicated and moves back and forth. The main factors affecting the discharge of tidal flow are the tidal range and the upstream inflow. The ecosystem of the tidal reach of Jinjiang River has been destroyed due to the rapid development of urbanization, industrialization, agriculture, and aquatic breeding in the surrounding regions during the past few decades.

Although the pollution of heavy metals in the tidal reach of Jinjiang River has became a serious environmental problem, the study on the heavy metal concentration, distribution, and speciation in surface sediments has not been carried out. The

main objectives of this study were: (a) to analyze the concentration of the heavy metals (Cu, Zn, Pb, Cr, Ni, and Cd) in different geochemical sediment fractions as a means of assessing bioavailability by using a BCR sequential extraction procedure to determine active fractions in surface sediments of the tidal reach of Jinjiang River and (b) to assess the degree of heavy-metal pollution using the geo-accumulation index (I_{geo}) in order to estimate the anthropogenic input and to assess the pollution status on the area.

Materials and methods

Sample collection and preparation

Surface sediments (0–5 cm) were collected from seven stations (Fig. 1) by using a plastic spoon during October and December in 2006. Sediment samples were carefully stored into a clean plastic vessel and kept frozen at −20°C prior to until processing and analysis, when the samples were defrosted at room temperature, and air-dried in a controlled clean environment. The samples were then ground with an agate pestle and mortar and sieved with a nylon 63-μm sieve, and homogenized. The section under the sieve ($\phi \leq 63$ μm) was used to analyze due to the strong association of metals with fine-grained sediments (Chen et al. 2003; Tam and Wong 2000).

Analytical methods

The pH, salinity, and Eh of the sediments were measured by the Orion pH828 meter, the Orion 115A multipurpose salinity meter, and the DW-1 oxidation-reduction potentiometer, respectively. $CaCO_3$ was measured as described by Loring and Rantala (1992), organic matter by Standard methods 209F, APHA (1995), and S^{2-} by Janaki-Raman et al. (2007). A MWD-2 microwave unit was used to determine the residual fraction of heavy metals in the sediments. A modified BCR three-step sequential extraction procedure was employed to obtain the speciation of heavy metals (Cr, Ni, Cu, Zn, Cd, Pb), and the digestion of the residual fraction was undertaken using a microwave-assisted acid digestion procedure (Table 1). After every successive extraction,

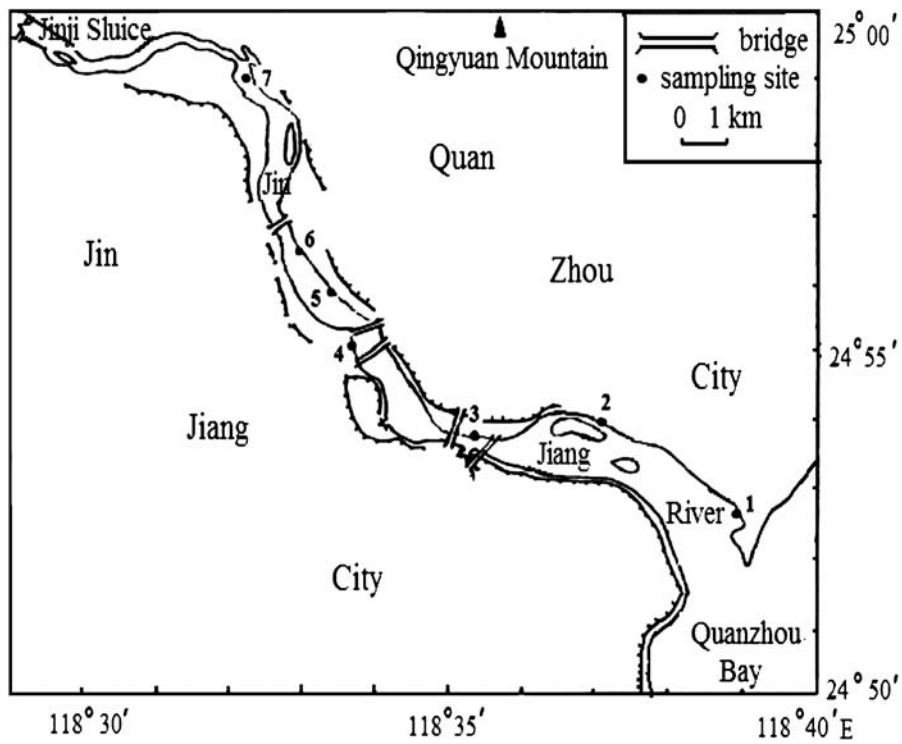

Fig. 1 Sampling sites of surface sediments from Jinjiang River tidal reach

Table 1 Heavy metal sequential extraction schemes of BCR method (Cuong and Obbard 2006; Marchand et al. 2006)

Step	Extractable form	Extraction procedure
I	Acid soluble fraction	Sample 0.5000 g, 20 ml 0.11 mol/L CH_3COOH, Shaking 16 h, centrifuging 6–8 min (4,000 rpm)
II	Reducible fraction	20 ml 0.5 mol/L $NH_2OH{\cdot}HCl$ (pH = 1.5 with HNO_3), Shaking 16 h, centrifuging 6–8 min (4,000 rpm)
III	Oxidizable fraction	5 ml 8.8 mol/L H_2O_2(pH = 2–3 with HNO_3), room temperature 1 h (manual shaking at 10-min interval), 85°C for 1 h, cooling and repeating the above procedure, 25 ml, 1.0 mol/L NH_4COOH (pH = 2 with HNO_3), Shaking 16 h, centrifuging 6–8 min (4,000 rpm)
IV	Residual fraction[a]	0.5 ml deionized water, 2 ml HCl, 2 ml HNO_3, 1 ml HF, room temperature 1 h (manual shaking at 10-min interval), 1 ml H_2O_2, room temperature 2 h (manual shaking at 10-min interval), microwave-assisted acid digestion (160 W 10 min, 240 W 5 min, 320 W 5 min, 480 W 10 min,640 W 10 min, at 5-min interval), 90°C for 2 h, centrifuging 6–8 min (4,000 rpm), 250 ml

[a]Digestion of the residual material is not a step of the modified BCR sequential extraction procedure

the extraction solution was filtered after centrifugation at 3,000 rpm for 10 min. The fractions extracted were as follows: acid soluble fraction (F1), reducible fraction (F2), oxidizable fraction (F3), and residual fraction (F4). The concentrations of heavy metals were determined by ICP-MS (ELAN 9000, Perkin-Elmer). Blanks were included in each step of analysis. Results were expressed as milligram per kilogram dry sediment for total analysis and as percentages for the speciation study. All the reagents used were of analytical reagent grade, the apparatus were all precleaned with deionized water in ultrasonic waves for 30 min.

Results and discussion

Sediment physicochemical characteristics

Table 2 shows the sediment physicochemical characteristics, which were related to the mobility of heavy metals in surface sediments. pH of the sediments was about 6.8–7.8, indicating that the sediments were neutral or weakly alkaline and decreasing from estuary to upstream. The salinity was between 0.1‰ and 3.3‰, higher in the estuary and lower in the upstream. Eh was varied between −105 and 124 mv, which indicated that the sediments were deposited under weakly reduction conditions. Eh was lower at S3 and higher at S7. Organic matter, $CaCO_3$, and S^{2-} are of good correlation with the chemical fractions of trace metals. Metals are easy to combine with organic matter, $CaCO_3$, and S^{2-}. Organic matter, $CaCO_3$, and S^{2-} were within the range of 8.6–10.7%, 0.2–6.2%, 2.0–31.2 $mg{\cdot}kg^{-1}$, respectively.

Speciation concentrations of heavy metals in surface sediments

The recovery of the sun contents of BCR procedure and the total content of heavy metals in surface sediments were listed in Table 3. The concentration of Zn and Cd were higher at S1, while

Table 2 The physicochemical parameters of surface sediments

Sites	pH	Sal (‰)	Eh (mV)	Org. (%)	$CaCO_3$ (%)	S^{2-} ($mg{\cdot}kg^{-1}$)
S1	7.8	3.3	−85	10.0	2.0	6.7
S2	7.8	1.5	−42	10.4	1.8	5.0
S3	7.5	1.2	−105	10.4	0.2	3.2
S4	7.5	0.4	−56	8.6	3.2	4.4
S5	7.1	0.5	65	9.9	6.2	2.0
S6	7.2	0.4	96	10.7	3.4	31.2
S7	6.8	0.1	124	10.2	0.4	2.0

Table 3 The recovery of the speciation contents of BCR procedures and the total content of heavy metals in surface sediments ($mg \cdot kg^{-1}$)

Element	Speciation	S1	S2	S3	S4	S5	S6	S7	Background value[a]
Cr	F1	24.71	23.61	55.48	28.41	60.58	22.72	59.38	32.1
	F2	32.68	164.75	80.03	41.20	51.63	61.24	62.28	
	F3	33.03	54.47	130.68	34.79	114.43	56.64	110.93	
	F4	138.23	392.96	235.18	57.28	208.38	70.71	62.55	
	Total content	251.72	530.35	443.46	177.53	365.30	231.35	309.54	
	Recovery (%)	90.84	119.88	113.06	91.07	119.09	91.34	95.35	
Ni	F1	6.79	12.7	12.32	11.08	8.27	11.43	2.03	9.3
	F2	4.45	8.63	10.42	5.08	6.38	9.32	2.70	
	F3	5.79	6.50	5.00	6.10	4.91	8.26	4.41	
	F4	18.29	14.44	19.29	15.89	20.33	18.20	12.69	
	Total content	36.16	37.28	42.90	39.23	37.65	42.82	19.79	
	Recovery (%)	97.68	113.39	109.63	97.25	105.95	110.25	110.31	
Cu	F1	17.51	44.27	41.11	26.14	23.51	42.19	13.68	18.0
	F2	25.71	67.51	47.09	39.41	29.93	51.30	15.77	
	F3	10.24	14.83	17.93	10.87	31.72	12.95	9.66	
	F4	26.78	33.69	34.21	31.28	31.61	35.85	14.91	
	Total content	85.81	143.19	150.65	110.23	115.22	154.37	53.48	
	Recovery (%)	93.51	111.95	93.16	97.70	101.35	92.17	101.01	
Zn	F1	96.00	117.20	189.71	88.60	45.16	199.80	14.72	69.9
	F2	44.20	200.00	33.70	50.70	21.83	87.50	14.17	
	F3	167.60	102.80	28.31	59.10	57.76	51.50	28.49	
	F4	437.20	243.40	178.27	206.30	198.47	230.70	117.77	
	Total content	628.93	555.65	450.37	432.55	306.41	487.85	184.97	
	Recovery (%)	118.46	119.39	95.47	93.56	105.49	116.74	94.73	
Cd	F1	1.07	1.31	0.47	0.83	0.11	1.56	0.22	0.046
	F2	0.44	1.09	0	0.47	0	0.47	0.04	
	F3	1.15	0.76	0.02	0.84	0.07	0.42	0.33	
	F4	1.91	0.89	0.46	1.32	0.43	1.44	0.56	
	Total content	3.99	3.39	1.18	3.95	0.72	3.25	1.36	
	Recovery (%)	114.54	119.47	80.51	87.57	84.72	119.69	84.56	
Pb	F1	12.22	12.80	39.26	8.46	1.60	12.34	7.05	37.6
	F2	37.85	56.07	54.22	35.70	37.36	52.50	68.42	
	F3	10.04	9.92	16.83	3.15	10.31	4.40	16.00	
	F4	49.20	44.49	65.07	49.56	77.88	42.87	47.46	
	Total content	112.85	110.73	148.52	117.93	115.45	131.21	147.63	
	Recovery (%)	96.86	111.33	118.09	82.14	110.13	85.44	94.11	

F1, F2, F3 and F4 express the acid-soluble, reducible, oxidizable and residual fraction, respectively

[a]Background values of heavy metals in coastal areas soil of Fujian Province (Cheng et al. 2004)

Zn was 9.0 times and Cd was 86.7 times than the background value because S1 is near the estuary where the salty water mixed the fresh water, and the massive mineral particle and the inorganic colloid had the good adsorption performance to the heavy metal ion (Facchinelli et al. 2001); Cr was higher at S2, the concentration of Cr is 16.5 times than the background value, maybe because there is a big sewage outfall; Ni and Pb were higher at S3, the concentration of Ni was 4.6 times and Pb was 4.0 times than the background value, because this site approached the residential areas and near the road and bridge; Cu was higher at S6, the concentration of Cu was 8.6 times than the background value, because a massive construction garbage and a big orchard are located at this place.

The results obtained from the application of the BCR-SEP are summarized for heavy metals

in Fig. 2. The acid soluble fraction is more mobile and dangerous than other fractions. Though the percentage in this fraction is small, the pollution is considerably strong. While the residual fraction presents in the inert phase, being of detritus and lattice origin, which is less dangerous to public health. The reducible and oxidizable fractions are easily available under oxidation-reduction conditions.

Acid soluble fraction This fraction includes exchangeable and carbonate phases and is the most easily changeable, because it is sensitive to changes in pH. The result is very similar to other contaminated sediments of different regions that the extracted percentages for Zn is 8% to 44% and Cd is 18% to 49% (Bird et al. 2003; Guhathakurta and Anilava 2000; Morillo et al. 2008). Zn and Cd trend to be released by the ion

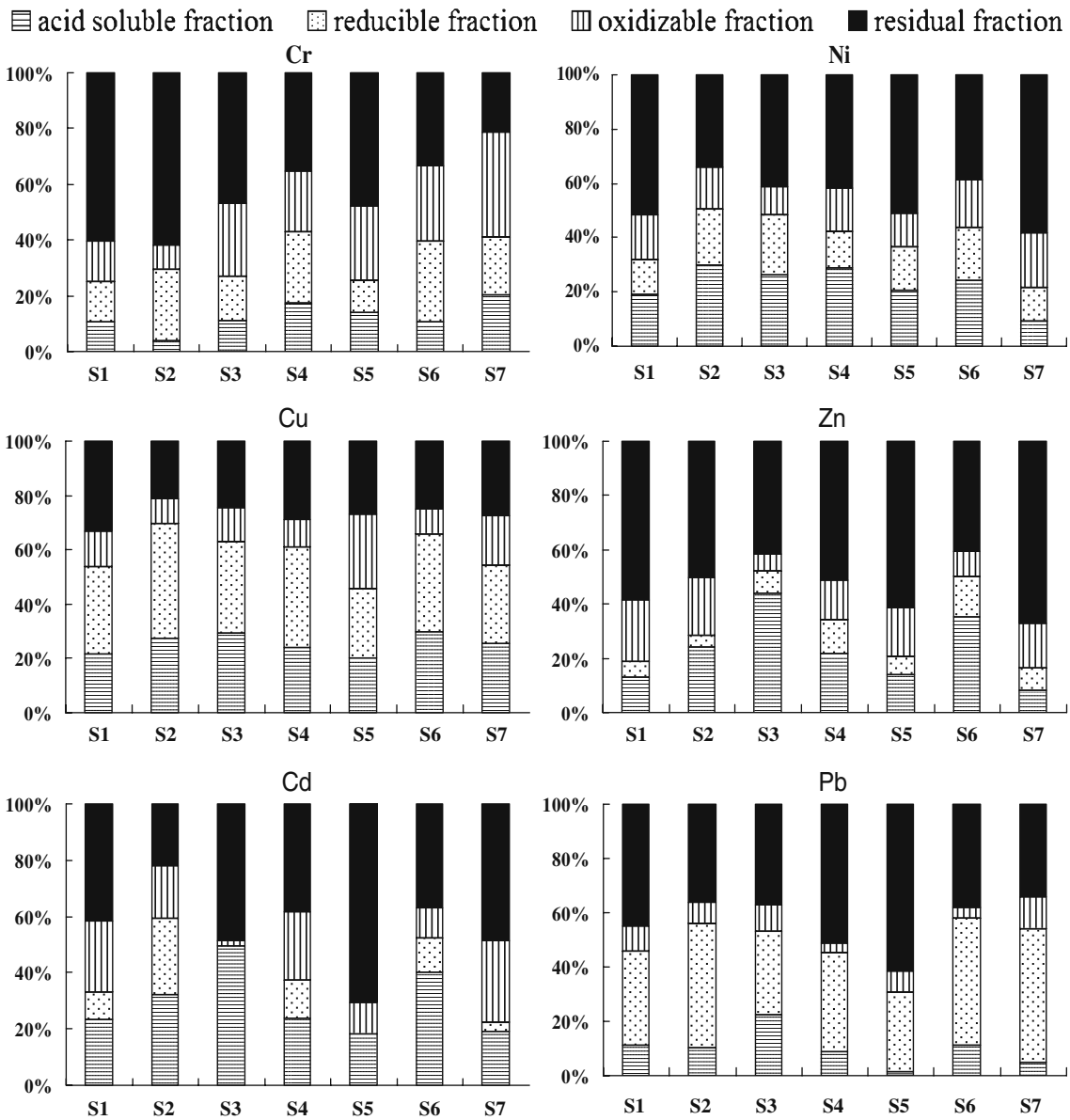

Fig. 2 Chemical speciation of heavy metals (*Cr*, *Ni*, *Cu*, *Zn*, *Cd*, *Pb*) in surface sediments

exchanged mechanism (Prechthai et al. 2008). The behavior of Zn and Cd is not surprising as the chemistry characteristics of Cd and Zn are similar, particularly in their ionic structures, electrovalent bond energies.

Reducible fraction In this fraction, the metals bonded to Fe–Mn oxides are obtained. The percentage of this fraction is relatively high and constituted a large portion of the nonresidual metal. Heavy metals are enwrapped by Fe–Mn oxides or precipitated as hydroxide. Metals bound to Fe–Mn oxides would be released under reductive conditions (Singh et al. 1999) and, therefore, are unstable under anaerobic condition (Flyhammar 1997). Most of Pb was found in the reducible fraction (29–49%). This phenomenon was similar to the results reported by several authors (Guhathakurta and Anilava 2000; Morillo et al. 2008), who found that hydrous Fe–Mn oxides were major scavengers of Pb in sediments. Cu shows similar fraction profile with percentages ranging from 26% to 42%. The sediments collected in this study were from anaerobic environment (Table 1). There is a high percentage of Cu in this fraction, which may be a threat to the aquatic environment under reducing conditions.

Oxidizable fraction Metals bound to organic matter and sulfurs would be re-released under oxidative conditions. In this fraction, the metal ion acts as the central ion, and the active organic matter group acts as the ligand or perhaps through the reaction of the sulfide ion and heavy metals. The organic fraction released in this step is hardly considered very mobile or available because the metals associated with stable high-molecular-weight humic substances that decompose slowly (Singh et al. 1998). A high percentage (9–38%) of Cr was found in the oxidizable fraction, indicating that high organic matter and sulfide absorbed heavy metals and played a significant role in controlling the mobilization of this element.

Residual fraction Metals in this fraction is mainly fixed in the primary and secondary minerals (Wu et al. 2004) and are chemically stable and biologically inactive. The greater the percentage of metals presenting in this fraction, the smaller the risk of the metals because this portion of the metals cannot be re-released to water under normal conditions. The stability of this fraction is controlled by the mineralogy and weathering degree of sediments. The high percentag of Zn (36–67%), Ni (34–51%), and Pb (34–61%) was found in the residual fraction, indicating that Zn and Ni showed little risk to environment.

The sum of the four fractions (F1 + F2 + F3 + F4) was in good agreement with the obtained pseudo-total metal amount by microwave digestion (Table 3). The percentage of nonresidual fractions(F1 + F2 + F3)of all metals decreases in the following order: Cu (74.0%) > Cd (62.5%) > Pb (57.4%) > Ni (56.2%) > Cr (52.8%) > Zn (51.3%), indicating that the mobility of heavy metals decrease in the same order. Therefore, Cu, Cd, Pb, and Ni are more easily released into the water and more bio-available in surface sediments, whereas Cr and Zn are relatively less releasable.

Risk assessment of sediment quality

A number of methods are available for assessing sediment quality, and each has its own advantages and disadvantages (Caeiro et al. 2005). The

Table 4 The index of geo-accumulation (I_{geo}) and classification of heavy-metal pollution

I_{geo}	$I_{geo} < 0$	$0 \le I_{geo} \le 1$	$1 \le I_{geo} \le 2$	$2 \le I_{geo} \le 3$	$3 \le I_{geo} \le 4$	$4 \le I_{geo} \le 5$	$I_{geo} > 5$
Class	0	1	2	3	4	5	6
Pollution degree	None	None to moderate	Moderate	Moderate to strong	Strong	Strong to extreme	Extreme

index of geo-accumulation determines and defines metals contamination in sediments by comparing current concentrations with preindustrial levels. The method requires the total content of heavy metal in surface sediments,considering the human activities, the environmental geochemical background values, and also taking into account the natural diagenesis which can cause the change of background value.

The index of geo-accumulation (I_{geo}) was introduced by Müller (1969):

$$I_{geo} = \log_2 \left[(C_n / k * B_n) \right]$$

where C_n is the measured concentration of the examined metal "n" in the sediments and B_n is the geochemical background concentration of the metal "n." The factor 1.5 is used because of possible variations in background values due to lithological variability. I_{geo} is associated with a qualitative scale of pollution intensity and provides a classification system (Table 4) for the degree of pollution which compared to the background (Table 3; Förstner et al. 1993). The sum of the four fractions was used as the total contents (Table 3) to estimate the heavy-metal pollution in this work.

The calculated values of I_{geo} suggested that the sites are affected differently for different metals (Table 5 and Fig. 3). According to the I_{geo} classification, the sites are highly polluted with respect to most trace metals. Pollution levels of all sites are moderate to strong with the exception of S7, which is moderately polluted; 85.7% of sampling site for Cr, 57.1% for Cu, 42.9% for Zn fall in moderate to strong pollution, and 100% of sampling site for Cd falls in strong pollution.

For the elements, Ni, Cu, and Zn are classified as unpolluted to moderately polluted at S7. Pb and Ni are unpolluted to moderately polluted at all sites, Cu and Zn (except S7) are moderately to strongly polluted, Cr is strongly polluted at S4, while moderately to strongly polluted at other sites, Cd is strongly to extremely polluted at all sites.

In conclusion, based on the I_{geo} classification, the magnitude of heavy-metal pollution in surface sediments of Jinjiang River tidal reach ranks as follow: Cd > Cr > Cu > Zn > Ni > Pb. Based on the intervention values, the sediments are moderately to extremely polluted by heavy metals, which probably originated from ships, industrial wastewater, and domestic activities. Understanding of the mobility of potential toxic metals and how they might transfer under human-induced

Table 5 Geo-accumulation index (I_{geo}) and I_{geo} classes of heavy metals in surface sediments

Elements	S1	S2	S3	S4	S5	S6	S7	Mean
Geo-accumulation index								
Cr	2.2	3.7	3.4	1.7	3.2	2.1	2.6	2.7
Ni	1.3	1.6	1.8	1.5	1.5	1.8	0.6	1.4
Cu	1.6	2.6	2.4	2	2.1	2.4	1	2.0
Zn	2.8	2.7	2	1.9	1.6	2.4	0.7	2.0
Cd	6	5.9	3.8	5.6	3.1	5.8	4.1	4.9
Pb	1	1.1	1.6	0.8	1.2	1	1.3	1.1
Mean	2.5	2.9	2.5	2.3	2.1	2.6	1.7	–
I_{geo} class								
Cr	3	4	4	2	4	3	3	3
Ni	2	2	2	2	2	2	1	2
Cu	2	3	3	2	3	3	1	2
Zn	3	3	2	2	2	3	1	2
Cd	6	6	4	6	4	6	5	5
Pb	1	2	2	1	2	1	2	2
Mean	3	3	3	3	3	3	2	–

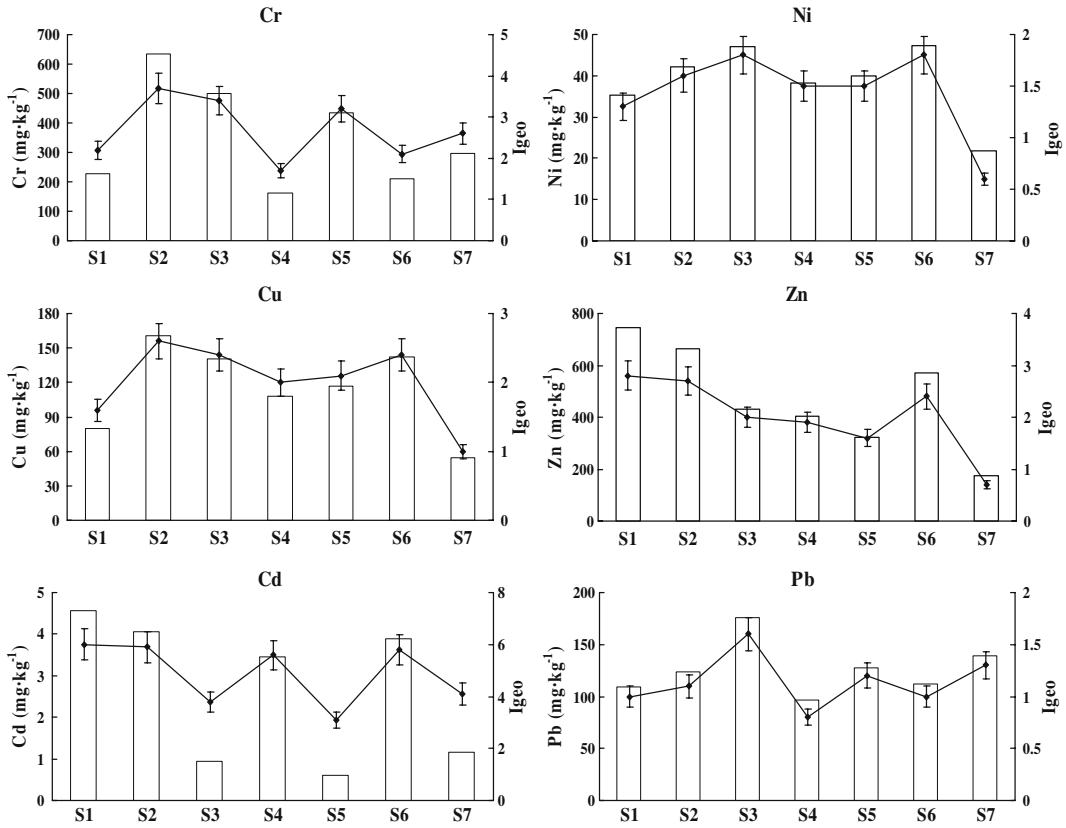

Fig. 3 Heavy metal contents (*bar chart*) and I_{geo} (*scatter plot with connecting line*) distribution in surface sediments

conditions are essential for developing the future remediation plans and pollution control strategies, especially for Cd pollution in surface sediments from the tidal reach of Jinjiang River.

Conclusions

The results show that Zn and Cd presented at comparatively higher percentage in the acid extractable fraction and Pb and Cu relatively higher in the reductive fraction, indicating that these metals have larger potential danger. The highest percentage of Ni, Zn, and Pb was found in the residual fraction, less harmful to the environment. Based on the I_{geo} classification, the magnitude of heavy-metal pollution in surface sediments of Jinjiang River tidal reach ranks as follow: Cd > Cr > Cu > Zn > Ni > Pb. The pollution degree of surface sediments is more serious, which may cause serious environmental problems and risk for human health, so the river should undergo remediation.

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