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# Distribution and enrichment of acid-leachable heavy metals in the intertidal sediments from Quanzhou Bay, southeast coast of China

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**Abstract** The article presents the distribution and enrichment of acid-leachable heavy metals (ALHMs) Cu, Zn, Pb, Cr, Mn, and Fe in the intertidal sediments collected from Quanzhou Bay, southeast coast of China. The contents of ALHMs along with sediment texture, total organic carbon, $S^{2-}$, and $CaCO_3$ in surface sediments were analyzed to identify the input of heavy metals from various sources. The enrichment of ALHMs in the sediments is mainly attributed to the intense industrial activities around Quanzhou Bay and to the serried activities of intertidal breed aquatics along the seacoast. The results also illustrate the association between the ALHMs with the finer fractions, organic matter, and Fe oxyhydroxides in the sediments. The above results were very supported by the multivariate statistical analyses, including correlation, principal component analysis, and hierarchical clustering analysis. Comparative results of ALHMs in the intertidal sediments from Quanzhou Bay with those in other domestic bays and estuaries indicate that the study area has been enriched with heavy metals, especially with Zn, Cu, and Pb, during the past few decades. The results of the present study suggest that the authorities should pay attention to the current status and take some measures to control the heavy metal pollution in the study area.

**Keywords** Acid leachable · Heavy metals · Enrichment · Intertidal sediment · Quanzhou Bay

## Introduction

Tidal beach, an important interface where ocean and land interact, is a transition zone where there are intense hydrodynamic action, sediment transportation, frequent erosion–deposition variation, complicated physicochemical conditions, such as Eh and pH, and abundant of various biological species. This area is also typically frail and sensitive and easy to be damaged by anthropogenic activities, and it is one of the main sinks for the heavy metals along the seashore (William et al. 1994). The heavy metals enriched in the tidal beach not only endanger tidal organisms directly and finally threaten human health through various food chains but also may release into the water column again to cause "secondary pollution" when the environmental conditions change, and

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these sediments are present in the bottom as a main source of heavy metal contamination for a fairly long time.

The mobility and bioavailability of heavy metals in the environment are predominated by their existing fractions. There are two methods to extract the active fraction of heavy metals in sediments. One is to adopt the total content of the nonresidual fractions using multiple-sequential extraction procedure. The other is to choose a certain selective reagent to extract the active fraction directly, named single step extraction procedure. The selective reagents generally include dilute acids, chelating agents, neutral salts, buffer reagents, and so on. The extraction procedure using dilute hydrochloric acid is one of the most widely used methods in single reagent extraction, and it is considered to be a rapid, effective means to assess the mobility of heavy metals in sediment and to evaluate whether the sediment has been polluted by anthropogenic activities (Jayaprakash et al. 2007). After a series of environmental evolution, the acid-leachable heavy metals in sediment are easy to release into water column and bring “secondary pollution” (Ayyamperumal et al. 2006).

In this paper, the acid-leachable heavy metals (ALHMs), which are leachable in 0.5 mol $L^{-1}$ hydrochloric acid, along with sediment texture, total organic carbon (TOC), $S^{2-}$, and $CaCO_3$ in the intertidal surface sediments collected from Quanzhou Bay were determined to identify the input of heavy metals from various sources. The distribution characteristics of ALHMs and their associations with sediment texture, organic matter, $S^{2-}$, and $CaCO_3$ were analyzed. The anthropogenic sources of the active heavy metals and the potential danger to environment were preliminarily discussed. The results will provide basic data and important reference for the environmental protection of the intertidal sediments.

## Materials and methods

### Study area

Quanzhou Bay (24°46′ ∼24°58′ N, 118°38′ ∼ 118°47′ E), the famous jumping off point of the sea route of silk and porcelain road in ancient China (especially in Song and Yuan Dynasties), lies in the west of Taiwan Straits and in southeast of Fujian Province and is a semi-enclosed bay with its mouth opening toward Taiwan Straits, with the largest water depth of 25 m, the total area of 136.42 $km^2$, and the intertidal area of 89.80 $km^2$. The bay is adjacent to the intensely industrialized cities of Quanzhou, Jinjiang, Shishi, and Hui’an. Extensive intertidal flats in the innermost reaches of the bay have been reclaimed and converted into container and industrial complexes, and this development is ongoing. Quanzhou Bay acts as an important estuary of two mainly considerable rivers, namely Jinjiang River, the third largest river with most sand in Fujian Province, and Luoyang River, an important estuarine swamp ecosystem, respectively. The intertidal zone is the transition belt between ocean and land, where many halo bios spawn, incubate, and capture. The ecosystem of intertidal zones has been destroyed due to the rapid development of industrialization, agriculture, mining, and aquatic breeding in the regions surrounding Quanzhou Bay during the past few decades. Recently, the government of Quanzhou City has decided to restore the ecosystem of Quanzhou Bay and its adjacent areas.

### Sampling and preparation

Sixteen surface sediment samples (0–5 cm) from the coastal tidal beach of Quanzhou Bay were collected using a Van Veen grab sampler in November 2007 (Fig. 1). After sampling, sediments were carefully stored into clean plastic vessels and kept frozen at −20°C prior to the further processing and analysis. In the laboratory, the frozen sediment samples were defrosted at ambient temperature and air-dried in a controlled clean environment. Then the samples were ground with an agate pestle and mortar and sieved with a 63-μm nylon sieve. The section under the sieve (<63 μm) was kept in sealed plastic vessels at 4°C for future analysis. The <63-μm fraction was used for analyses in this study due to strong association of metals with fine-grained sediments (Goh and Chou 1997; Tam and Wong 2000). Many previous studies have been completed on the sequential extraction of metals from sediments using

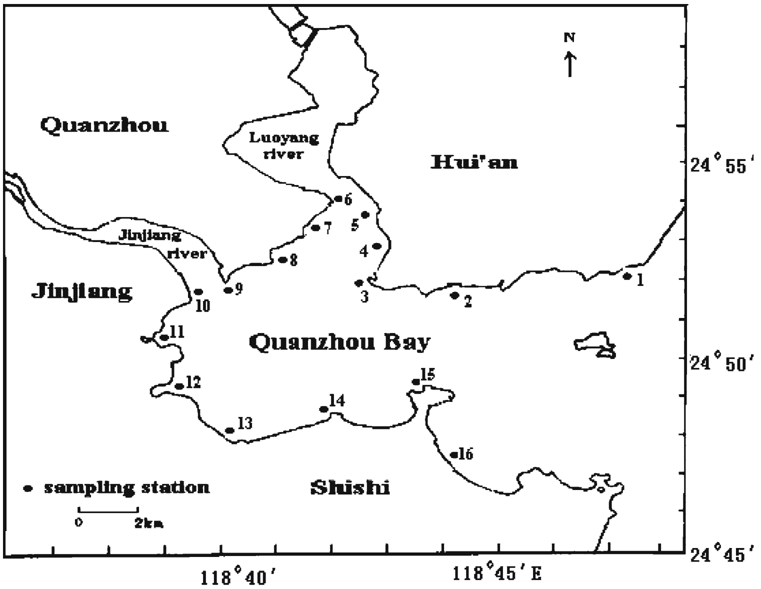

**Fig. 1** Map of sampling sites in Quanzhou Bay

this size fraction (Morillo et al. 2004; Guevara-Riba et al. 2004; Yuan et al. 2004). The moisture content of the dried sample was calculated by heating a portion of sediment in an oven at 105 ± 2°C to a constant weight. Sediment data in this study are reported on a dry weight basis.

## Analytical methods

Textural studies were performed for sediment samples to estimate sand, silt, and clay contents (Ingram 1970). The results are presented as sand and mud (silt and clay) due to the high level of fine particles. The grain size distribution was determined with a Winner 2000 Laser Particle Granularity Analyzer. The values of Eh, pH, and salinity of sediments were determined by an FJA-15 oxidation–reduction potential meter, an Orion 828 pH meter, and an Orion 115A+ salinity meter, respectively. The Analytikjena HT1300-TOC equipment was used to determine the TOC. The contents of $CaCO_3$ and $S^{2-}$ of sediments were measured using the methods described by Loring and Rantala (1992) and Janaki-Raman et al. (2007), respectively.

All chemicals are of analytical reagent grade and contain very low concentrations of trace metals in this study. Normal precautions for trace metals analysis were observed throughout. Double deionized water from a MILLI-Q system supplied was used for preparing the solutions and all dilutions. All the glassware and the Teflon vessels used in the study were previously soaked overnight with 20% $HNO_3$ and then rinsed thoroughly with deionized water.

Extraction of acid leachable metals was performed by taking 1.0 g of dry sediment sample in a 50-mL Teflon centrifuge tube in which 15 mL of 0.5 mol $L^{-1}$ hydrochloric acid was added. After being mechanically shaken for 24 h at 22 ± 5°C, the mixture was centrifuged at 4,000 rpm for 10 min and then filtered with Whatman Grade "A" filter paper (0.45 μm). The filtrate was transferred into a 100-mL volumetric flask. The residue was washed twice with deionized water, and the filtrates were decanted into the same flask, then diluted to 100 mL with 3% $HNO_3$, and stored at 4°C prior to analysis.

The filtered solution was analyzed for acid-leachable Cu, Zn, Pb, Cr, Mn, and Fe with an inductively coupled plasma atomic emission spectrometer (Perkin-Elmer Optima 2000 DV). Background correction and matrix interference were monitored throughout the analysis. All the sediment samples were analyzed in triplicates. The analytical precisions were better than 10%.

## Results and discussion

### Main physical–chemical parameters of sediments

The main physical–chemical parameters, which are sometimes related to the mobility of heavy metals, of the intertidal surface sediments are listed in Table 1. The values of pH, salinity, and Eh were obtained at the sampling sites, and the other parameters were determined with <63 μm sediments in laboratory.

The pH values vary from 6.3 to 7.6, indicating weakly acidic, neutral, or weakly alkaline nature of the studied sediments. The salinity of the surface sediments varies from 0.5‰ to 16.4‰ in different sites because of the frequent mixing of sea water and the fresh drainage. The range of Eh from −248 to 227 mV means comparatively reducible environment in the studied sediments, especially site 8 which shows strong reductive condition. The contents of organic matters in the sediments are characterized with TOC values which vary from 0.81% to 1.88%, indicating moderate level compared with those in other domestic estuaries, with the exception of site 16, whose TOC presents 0.18%. The contents of $S^{2-}$ and $CaCO_3$ range from 0.18% to 1.40% and from 0.52% to 9.49%, respectively. The fine particle (whose diameter is less than 10 μm, i.e., fine silt + clay) contents in sediments are commonly higher, ranging from 56.7% to 84.6%, with the exception of site 16, whose fine particle content accounts for 23.8%. The textures of most sediment studied are clayey silt and silty clay, with the exception of site 16, which only presents sandy.

### Distribution of acid-leachable heavy metals in sediments

The distribution of ALHMs in the intertidal surface sediments from Quanzhou Bay is illustrated in Fig. 2. The contents of ALHMs in different sampling sites are different, probably resulted from the interaction of rock weathering, soil erosion, pollutant emission, and the properties of sediment such as pH, Eh, and organic matter, etc. (Lu et al. 2005). The above factors may change the toxicity of heavy metals by affecting the activity and bioabsorption and metabolic process of heavy metals (Chen et al. 1992).

From the overall distribution characteristic (Fig. 2), the ALHMs contents in the intertidal sediments of the south coast of Quanzhou Bay are commonly higher than those in the north coast mainly because that the famous developed industrialized cities Jinjiang and Shishi are located in the south coast, while the industrialization of Hui'an county located in the north coast started

**Table 1** Main physical–chemical parameters of intertidal surface sediments from Quanzhou Bay

| Site | pH | Salinity (‰) | Eh (mv) | TOC (%) | $S^{2-}$ (%) | $CaCO_3$ (%) | Fine silt + clay (%) |
|---|---|---|---|---|---|---|---|
| 1 | 7.6 | 16.4 | 207 | 1.48 | 0.63 | 9.49 | 65.2 |
| 2 | 6.5 | 10.6 | −136 | 1.12 | 0.55 | 4.36 | 56.7 |
| 3 | 6.5 | 14.3 | −27 | 1.19 | 0.30 | 1.73 | 69.5 |
| 4 | 6.4 | 11.3 | 74 | 1.23 | 0.39 | 3.19 | 77.3 |
| 5 | 6.6 | 11.0 | 227 | 1.45 | 0.63 | 2.44 | 84.6 |
| 6 | 6.3 | 1.7 | −29 | 1.32 | 0.34 | 3.28 | 71.9 |
| 7 | 6.3 | 3.7 | 155 | 1.43 | 0.22 | 8.16 | 67.1 |
| 8 | 6.3 | 1.7 | −248 | 0.81 | 0.56 | 1.42 | 74.1 |
| 9 | 6.9 | 8.4 | −98 | 0.99 | 0.18 | 2.41 | 77.1 |
| 10 | 7.2 | 8.6 | 7 | 1.88 | 0.79 | 5.59 | 79.4 |
| 11 | 7.1 | 4.3 | 10 | 1.43 | 0.30 | 0.52 | 68.9 |
| 12 | 7.1 | 3.8 | −47 | 1.25 | 0.46 | 4.38 | 75.5 |
| 13 | 7.1 | 6.8 | −101 | 1.62 | 1.40 | 6.34 | 80.8 |
| 14 | 7.0 | 4.2 | 198 | 1.50 | 0.28 | 4.15 | 80.3 |
| 15 | 7.4 | 4.2 | 198 | 1.19 | 0.38 | 7.24 | 78.4 |
| 16 | 7.0 | 0.5 | 184 | 0.18 | 0.90 | 6.82 | 23.8 |

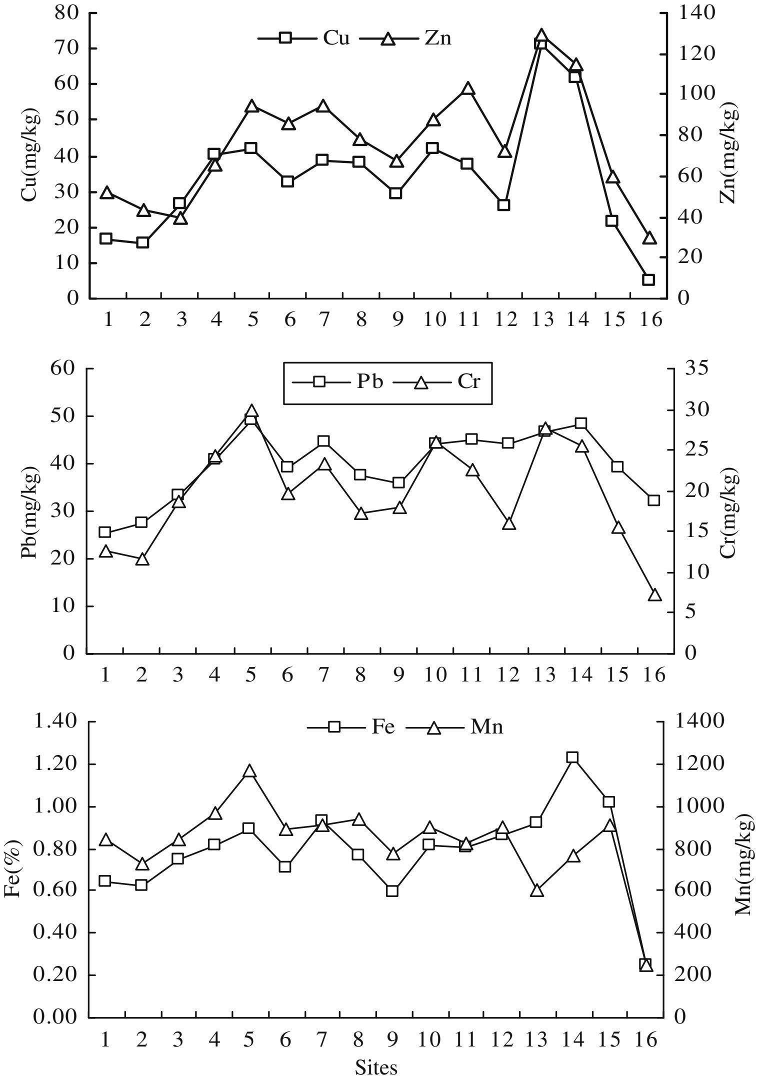

**Fig. 2** Distribution of acid-leachable heavy metals in the intertidal sediments of Quanzhou Bay

comparatively late. The contents of ALHMs in the north coastal intertidal sediments sequentially decline from the estuary of Luoyang River to the bay-mouth (from site 5 to site 1) mainly because that the sea- and freshwater mix in the vicinity of the estuary, which would accelerate the condensation and deposition of the fine particles in which enrichment heavy metals may be adsorbed (Wang and Zhang 2002). Except for site 16, there are large or small sewage outfalls in the vicinity of each sampling site in the south coast; consequently, no accordant variation characteristic of ALHMs contents is found.

The acid-leachable contents of Cu, Zn, Cr, and Pb are significantly higher in site 13 and site 14. The main reasons are that there is Jin-Ji-Nan-Zha sewage outfall in the vicinity of site 13, through which the industrial and domestic sewage from Shuitou town and Xibin town flowed into Quanzhou Bay, and that there are large areas of intertidal breed aquatics around site 14 and several duckeries and henneries opposite to the out dike. Disinfection and sterilization, using pesticide containing metals, as well as breed aquatics drains may affect the contents of heavy metals in the adjacent sediments. The content of acid-leachable Mn in site 5 is the highest (1,167 mg/kg), for the site is the farthest from the coast. This indicates that the marine autogenerating ability of Mn is considerably higher (Zhao 1994). The acid-leachable contents of all heavy metals in site 16 is commonly lower, for the sand domination in the sediment texture and the contents of TOC and fine particle are relatively low (Table 1), which indicates to some extent that the contents of acid-leachable heavy metals are affected by the sediment textures such as granularity, organic matter, and so on.

## Statistical analysis

Statistical analyses based on acid-leachable elements were effectively utilized for the interpretation of geochemical processes of heavy metals (Jonathan et al. 2004; Selvaraj et al. 2004; Jayaprakash et al. 2007). The correlation coefficients among ALHMs and sediment properties in Quanzhou Bay are listed in Table 2.

On the whole, the ALHMs show positive correlation one another, meaning that the heavy metals in this study may have the common pollution source-industrial and agricultural sewage emission and tidal beach breeding. All the acid-leachable metal contents are positively correlated with Fe, but no significantly correlation with Mn, indicating that the ALHMs in the surface sediments are mainly combined with Fe oxyhydroxides but not Mn oxyhydroxides. The ALHMs contents are positively correlated with TOC, which indicates that the organic matter is one of the important factors controlling the potential perniciousness of heavy metals in the sediments and that the organic particles are mainly derived from the industrial effluents as suspended particulate matter and terrestrially derived organic material. The content of acid-leachable Cu is significantly correlated with $S^{2-}$, which indicates that parts of the sulfides fraction of Cu have been extracted by 0.5 mol $L^{-1}$ hydrochloric acid. All the contents of ALHMs are negatively correlated or uncorrelated with $CaCO_3$, which indicates that the studied heavy metals are hardly combined with carbonates. All the ALHMs contents are positively correlated with the contents of fine particles, indicating that the available fractions of heavy metals in the sediments mainly exist in the fine particles and that ALHMs are enriched in sediments mainly due to water-borne pollution of different types.

In order to evaluate the results obtained, principal component analysis and hierarchical clustering analysis (HCA) have been applied on a data set of 16 sampling sites and six heavy metals (Fe, Mn, Cu, Pb, Zn, Cr). Three principal components were extracted, which covered 93.4% of the total variance. The scatter plot of scores on the first two principal components PC1 and PC2 shows a separation among the stations with different geographical position (Fig. 3). In fact, the sites with

**Table 2** Correlation coefficients among ALHMs and sediment properties in Quanzhou Bay ($n = 16$)

| Element | Cu | Zn | Cr | Mn | Fe | Pb | TOC | $S^{2-}$ | $CaCO_3$ | Fine silt + clay |
|---|---|---|---|---|---|---|---|---|---|---|
| Cu | 1.00 | | | | | | | | | |
| Zn | 0.91[a] | 1.00 | | | | | | | | |
| Cr | 0.87[a] | 0.82[a] | 1.00 | | | | | | | |
| Mn | 0.25 | 0.27 | 0.53 | 1.00 | | | | | | |
| Fe | 0.71[a] | 0.69[a] | 0.68[a] | 0.55[b] | 1.00 | | | | | |
| Pb | 0.77[a] | 0.83[a] | 0.83[a] | 0.36 | 0.70[a] | 1.00 | | | | |
| TOC | 0.62[b] | 0.64[b] | 0.72[a] | 0.55[b] | 0.67[b] | 0.49 | 1.00 | | | |
| $S^{2-}$ | 0.57[b] | 0.49 | 0.32 | −0.32 | 0.14 | 0.24 | 0.24 | 1.00 | | |
| $CaCO_3$ | −0.18 | −0.12 | −0.27 | −0.30 | −0.04 | −0.23 | 0.09 | 0.21 | 1.00 | |
| Fine silt + clay | 0.68[a] | 0.62[b] | 0.75[a] | 0.78[a] | 0.78[a] | 0.58[b] | 0.74[a] | 0.16 | −0.28 | 1.00 |

[a] Correlation is significant at the 0.01 probability level
[b] Correlation is significant at the 0.05 probability level

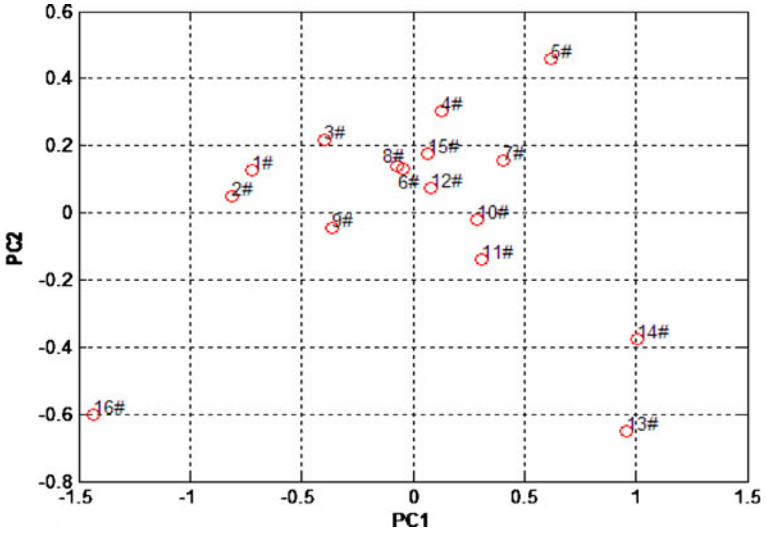

**Fig. 3** Scatter plot of scores on the first two principal components

positive scores on the PC1 show relatively high contents of ALHMs, while the sites with negative scores show relatively low contents of ALHMs. Loading of the variables on the first two principal components shows that Fe, Cr, Pb, Zn, and Cu are the dominant variables on PC1, while Mn is the dominant variable on PC2 (Fig. 4). HCA was carried out by using Ward's method procedure, applied on the Euclidean distances and the dendrogram of the variables. There are five clusters indeed (Fig. 5). The first three clusters include Cu, Pb, Cr, and Zn, indicating that these metals are mainly anthropogenic and are present as excess in concentration apart from the geochemical process. Mn and Fe each can be identified as a cluster. Fe mainly originates from natural weathering and erosion, while Mn mainly originates from natural weathering and marine autogeny.

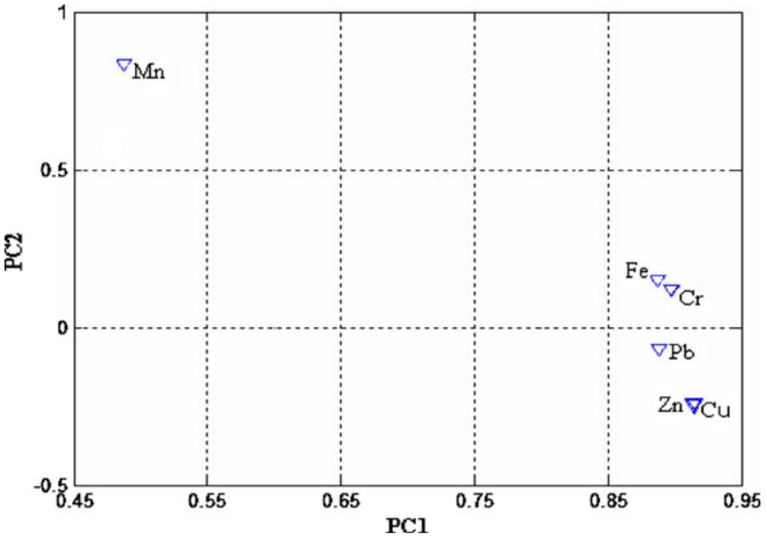

**Fig. 4** Loading of the variables on the first two principal components

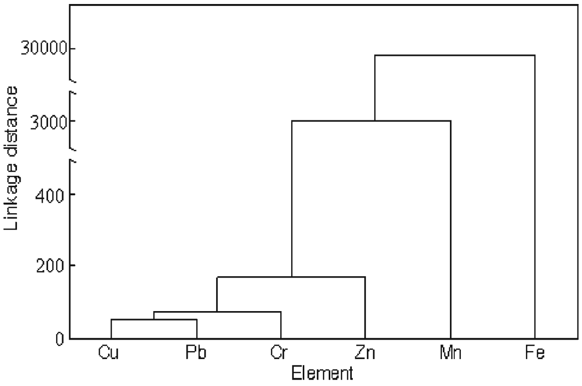

**Fig. 5** Dendrogram of the variables

## Comparison of ALHMs contents with some domestic bays and estuaries

Contents of ALHMs in sediments from Quanzhou Bay and other domestic bays and estuaries are listed in Table 3. The average acid-leachable fraction contents of Zn, Pb, and Cu in the sediments from Quanzhou Bay are significantly higher than those in other domestic bays and estuaries, which maybe greatly affected by the frequently anthropogenic activities. Cu and Zn are mainly affected by the industrial, agricultural, and domestic sewage emission. Pb is mainly affected by the sewage emission and atmospheric dust from the industrial and agricultural process. Xu et al. (1993) found that the contents of Pb, Zn, and Cu were higher along Xiamen Harbor Jiulong Port–Quanzhou Bay–Xinghua Bay, probably for the considerable metallic mines of Pb, Zn, and Cu distributed along the southeast coast. The acid-leachable content of Mn is also higher than those in other domestic bays and estuaries except Yangtze River Estuary. Xu and Li (1989) found that the content of Mn in the sediments of Meizhou Bay was significantly higher than Jiaozhou Bay, Taiwan Strait, East Sea, and some other sea areas when they studied the geochemical characteristics of the sediments in Meizhou Bay and considered that this was related to the high content of MnO in continental-derived rocks of the back and west of Meizhou Bay. The content of MnO in Quanzhou Bay is basically close to that in Meizhou Bay, and it does not change much among the bays in Fujian (Huang and Zeng 1993). So the enrichment of Mn in the sediments of Quanzhou Bay may be the interaction of the higher content of Mn in adjacent continental-derived rocks, regional geochemical features, and biogeochemical procedures in the inner bay. The acid-leachable content of Fe in the sediments from Quanzhou Bay is significantly lower than those in other domestic bays and estuaries, which indicates that Fe mainly originates from natural weathering and erosion in the studied area. The acid-leachable content of Cr in the sediments from Quanzhou Bay is commonly lower than those in other domestic bays and estuaries.

**Table 3** Contents of ALHMs in sediments from Quanzhou Bay and other domestic bays and estuaries

| Element | Quanzhou Bay | Zhifu Bay[a] | Longkou Bay[a] | Rizhao offing[a] | Yellow River Estuary[a] | Yangtze River Estuary[a] |
|---|---|---|---|---|---|---|
| Pb | 39.6 (25.5–49.2) | 24.3 (13.3–58.1) | 18.9 (7.8–30.8) | 18.9 (6.8–36.1) | 22.3 (6.4–38.5) | 26.5 (7.2–41.6) |
| Cu | 34.0 (4.9–71.2) | 32.3 (9.2–214.1) | 6.7 (1.6–21.1) | 9.1 (2.0–26.3) | 12.5 (2.9–20.8) | 16.7 (2.6–28.1) |
| Zn | 76.2 (30.1–129.0) | 75.5 (37.1–244.5) | 20.7 (3.2–43.6) | 20.5 (5.3–42.7) | 23.1 (7.2–35.9) | 48 (21.8–63.8) |
| Cr | 19.8 (7.3–29.8) | 19.0 (7.6–70.3) | 20.3 (15.4–30.3) | 25.0 (15.4–46.3) | 30.5 (17.4–44.2) | 31.2 (18.4–40) |
| Fe | 0.79 (0.25–1.23) | 1.12 (0.48–2.44) | 1.32 (0.31–2.44) | 1.50 (0.31–3.11) | 1.85 (0.89–2.61) | 3.1 (1.58–4.33) |
| Mn | 825 (250–1,167) | 248 (68–477) | 214 (53–457) | 359 (177–684) | 363 (184–578) | 867 (258–4,967) |
| Sample number | 16 | 20 | 13 | 14 | 20 | 14 |

The unit is milligrams per kilogram except percent for Fe. The data in and out of the brackets are the content ranges and average contents separately

[a] The data originate from Zhang et al. (1998)

## Conclusions

The acid-leachable contents of heavy metals (Cu, Zn, Pb, Cr, Mn, and Fe) in the intertidal sediments of the north coast of Quanzhou Bay decrease from the estuary to the bay-mouth, while the variation law is not significant because of the frequent sewage outfalls in the vicinity of most sampling sites along the south coast. The contents of acid-leachable Zn, Pb, and Cu are higher than those in other domestic bays and estuaries and are affected probably by anthropogenic activities. The positive correlation among the ALHMs contents reveals that they may have the common pollution source-industrial and agricultural sewage emission and tidal breeding. The ALHMs in the sediments mainly exist in the fine particles and are mainly combined with Fe oxyhydroxides. Organic matter is one of the factors controlling the potential harm of heavy metals. The ALHMs in sediments may release again under certain environmental condition and cause the secondary pollution of the above water column. The authorities should pay attention to the current status and take some measures to control the heavy metal pollution in the study area.

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